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Structural and Optical Properties of ZnO Nanorods Thin Films Prepared Using Hydrothermal Technique and Effect of growth time

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ABSTRACT

This paper describes, Synthesis of zinc oxide nanorods (ZnO NRs) using hydrothermal technique at different growth time. The structural and morphological properties were characterized by X-ray diffraction (XRD), Energy Dispersive X-Ray (EDX) and Field Emission Scanning Electron Microscope (FE-SEM). The ZnO NRs were obvious hexangular wurtzite structure and preferentially oriented along the c-axis (002) and growth vertically to the substrates. The optical properties were studied. From UV-Visible spectrophotometer and Photoluminescence (PL), the optical band gap energy of all ZnO NRs samples (S1, S2 and S3) were calculated to be (3.425 eV, 3.4 eV, 3.425 eV) respectively. Also, the effect of growth time on ZnO nanorods was studied.

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الخواص التركيبية والبصرية للاغشية الرقيقة لقضبان اوكسيد الزنك النانوية (ZnO Nanorods) المحضرة بالطريقة الحرارية المائية (Hydrothermal) وتأثير زمن الانماء

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الـــخُـــلاصـــة

تـم تحضـير قضـبان اوكسـيد الزنـك النانويـة (ZnO Nanorods) بالطريقـة الحراريـة المائية (Hydrothermal) في زمن انمـاء متغير. تمت دراسـة الخـواص التركيبيـة والسـطحية بواسـطة حيـود الاشـعة السـينية (XRD) ومطيافيـة تشـتت الطاقـة بالاشـعة السـينية (EDX) والمجهر الالكتروني الماسح تأثير المجال (FE-SEM). قضبان اوكسيد الزنك النانوية (ZnO Nanorods) تمتلـك التركيـب السداسـي ونمـو بلـوري بأتجـاه المحـور(c) بأتجـاه المسـتوي البلوري(002) عموديا على القاعدة. تمت دراسة الخواص البصرية بواسطة مطياف الاشعة المرئيـة وفـوق البنفسـجية (UV-Visible Spectroscopy) ومطيـاف انبعـاث الضـوء (Photoluminescence Spectroscopy (PL)) وحساب فجوة الطاقة البصرية لجميع العينات (S1, S2 and S3) لتكن(3.425 eV, 3.4 eV, 3.425 eV) على التوالي، ايضا تمت دراسة تأثير زمن الانماء على قضبان الزنك النانوية(ZnO Nanorods).

الكلمات المفتاحية:

اغشية رقيقة
أوكسيد الزنك
قضبان نانوية
الطريقة الحرارية المائية

1. INTRODUCTION

Nanotechnology is an emerging, highly interdisciplinary field. It has the ability to manipulate matter at an atomic scale. Nanotechnology can be useful in diagnostic techniques, drug delivery, sunscreens, antimicrobial bandages, disinfectant, and a friendly manufacturing process that reduce waste products [1,2]. Nanorods and nanowires have recently attracted considerable attention towards scientific community because of their novel properties and potential technological applications. It is widely used in the field of catalyst [1], gas sensor [2], solar cell materials [3], antimicrobial materials [4], optoelectronics devices [5,6].

Zinc oxide (ZnO) is a self-activated crystal of hexagonal wurtzite structure with direct wide band gap energy of 3.37 eV at room temperature [2-4]. ZnO has strong excitonic emission in the ultraviolet range even at room temperature due to its larger excitonic binding energy of 60 meV. Which is significantly larger than other materials [5]. ZnO has attracted much interest as one of the multifunctional inorganic nanoparticles due to its unique combination of superior chemical, physical, optical, biological, electrical, long-term environmental stability, biocompatibility, low cost and non-toxic properties [3,4]. Therefore, nano-ZnO can potentially be applied to catalysts, gas sensors, semiconductors, varistors, piezoelectric devices, field-emission displays, ultraviolet (UV) photodiodes, surface acoustic wave (SAW) devices, UV-shielding materials, rubber, medical and dental, pigments and coatings, ceramic, concrete, antibacterial and bactericide, and composites [6-15]. Various morphology of ZnO nanostructures have been realized. This includes nanoparticles, nanorods, nanowires, nanobelts, nanotubes, nanobridges, nanowalls, nanohelixes, mesoporous single-crystal nanowires, and polyhedral cages [9,16]. On the other hand, one-dimensional (ID) nanostructures such as rods, wires, belts and tubes have attracted noticeable attention in recent years. This is due to their many unique properties and the possibility that may be used as building blocks for future electronics and photonics, as well as for life-science applications. ID nanostructures are expected to play an important role as both interconnects and functional units in fabricating electronic, optoelectronic, electrochemical and electromechanical Nano devices [17].

Among the (ID) nanostructures, ZnO nanorods (NRs) and nanotubes (NTs) have been widely studied because of their easy formation and device applications [17,18]. Recently different synthesis methods have been devised for ZnO nanostructures such as vapor transport process [19, 20], spray pyrolysis [21, 22], thermal decomposition [23], chemical bath deposition(CBD) and hydrothermal synthesis [24,25], sol-gel processing [26], direct precipitation and co-precipitation [27-29]. Among these methods, vapor deposition (PVD) and chemical vapor deposition (CVD) have been developed to synthesize ZnO nanoparticles into complex structures such as flower-like and web-like agglomerates. However, in order to obtain the final structure these methods, this usually requires multiple steps, sophisticated equipment and high temperature. By contrast, low-temperature wet chemical processes such as, hydrolysis, precipitation and hydrothermal process are cost-effective and scalable and have been used for preparation a wide variety of ZnO nanostructures [30]. Hydrothermal technique is a promising alternative synthetic method for nanostructure materials because of the low process temperature and very easy to control the particle size. The hydrothermal process has several advantage over other growth processes such as use of simple equipment, catalyst-free growth, low cost, large area uniform production, environmental friendliness and less hazardous. The low reaction temperatures make this method preferable one for microelectronics and plastic electronics. It has also been successfully

employed to prepare nanoscale ZnO and other luminescent materials [31]. In brief, the particle properties such as morphology and size can be controlled via the hydrothermal process by adjusting the reaction temperature, time and concentration of precursors.

In this paper to prepare of ZnO NRs thin films using two-step hydrothermal technique was studied. Influence of reaction time were studied on nanorods diameter. The structural and optical properties of the ZnO NRs were investigated using X-ray diffraction(XRD), Energy Dispersive X-Ray Spectrometer (EDX), field-emission scanning electron microscopy (FE-SEM), UV-visible spectrophotometer, and photoluminescence (PL).

Experimental

2.1 preparation of the ZnO seed solution

The glass substrates were cleaned by the Acetone, Ethanol and Isopropanol for 5min in an ultrasonic bath respectively. After that Alcohol and the Deionized (DI) water were used for 5 min. The substrates were fully cleaned dried, and then kept in suitable container.

Homogenous and uniform ZnO nuclide were deposited using the sol–gel spin coating method [31]. Before seed layer deposition, the ZnO solution was prepared using zinc acetate dihydrate as a precursor and monoethanolamine (MEA) as a stabilizer. In this study, 2-methoxyethanol (2-ME) was used as solvent. Precursor solution was obtained by mixing of (0.05 M) zinc acetate dihydrate with 100 ml of solvent. To ensure that the zinc powder was completely dissolved in the solvent, the mixed solution was stirred on a hot plate stirrer at 70°C for 20 min. Then,10ml of MEA was gradually added to the precursor solution, while stirring constantly at 70°C for 2 h. The milky solution was then changed into a homogenous and transparent solution. The solution was stored for 24 h at RT before deposition.

2.2 Preparation of the ZnO seed layers

conventional spin coater was used to deposit the precursor solution on the cleaned substrates at 3,000 rpm for 30 s. A drying process was then performed on a hot plate at 200°C for 5 min. The same coating process was repeated thrice to obtain thicker and more homogenous ZnO films. The coated films were annealed at 350°C for 2 h to remove the organic component and solvent from the films. The annealing process was conducted in the conventional furnace. The preparation of the ZnO thin films is shown in fig. (1).

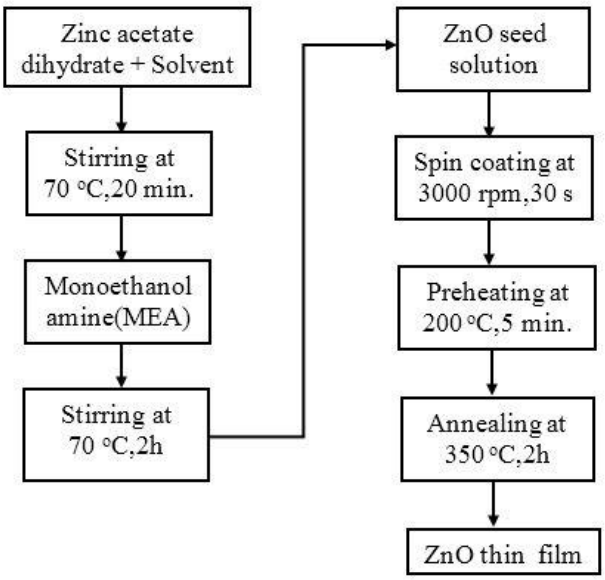


Fig. (1): ZnO thin film preparation process flow.

2.4 Preparation of the ZnO NRs

After the uniform coating of the ZnO nanoparticles on the substrate, the ZnO NRs were obtained through hydrothermal growth. The growth solution consisted of an aqueous solution of zinc nitrate hexahydrate, which acted as the Zn^{2+} source, and hexamethylenetetramine (HMTA). The concentration of the Zn $(NO_3)_2$ was maintained at 25 mM, and the molar ratio of the Zn $(NO_3)_2$ to HMTA was 1:1. For the complete dissolution of the Zn $(NO_3)_2$ and HMTA powder in (DI) water, the resultant solution was stirred using a magnetic stirrer for 3 min at RT. the suspension was transferred into a Teflon lined stainless steel autoclave with a volume of 100 ml, The

ZnO NRs were grown by immersing the substrate with the seed layers that was placed vertically in the prepared aqueous solution. the autoclave was sealed and kept at 110°C and different synthesis time (3 h, 5 h and 7 h). After that the autoclave cooled down to room temperature. After the growth process, the obtained samples washed several times in ethanol and distilled water and dried at 80°C for 30 min then the thin film were annealed at 350°C for 2 h to remove residual organic materials [26]. The chemical reaction can be stated below [32]:

$$C_6H_{12}N_4 + 6H_2O \leftrightarrow 6CH_2O + 4NH_3 \quad (1)$$

$$(CH_2)_6N_4 + Zn^{2+} \rightarrow [Zn(CH_2)6N4]^{2+} \quad (2)$$

$$NH_3 + H_2O \leftrightarrow NH^+_4 + OH^- \quad (3)$$

$$Zn^{2+} + 4NH_3 \rightarrow Zn\,(NH_3)^{2+}_4 \quad (4)$$

$$Zn^{2+} + 4OH^- \rightarrow Zn(OH)^{2-}_4 \quad (5)$$

$$Zn(NH_3)^{2+}_4 + 2OH^- \rightarrow ZnO + 4NH_3 + H_2O \quad (6)$$

On the initial growth of the ZnO nanorods, the (HMTA) started to breakdown into ammonia (equation (1)) and produce hydroxide ion, namely (OH^-) (equation (3)). The (Zn^{2+}) Cation further reacted to (NH_3) and (OH^-) anion forming ZnO core (equation (4) and equation (5)). The crystal core would develop, and further degraded into the ZnO core nanorods under the influence of more(OH) ion at a specific temperature, according to equation (6) [33]. Hence, as the time passed by, the ZnO core would keep growing. Subsequently, the(Zn) on the surface of the substrate would oxidize and form ZnO nanorods on the substrate. In the meantime, the oxidation process resulted in the lateral growth of the ZnO core during the attachment of (Zn) atom on the ZnO nuclei core [23]. The growth process of the ZnO NRs is presented in fig. (2).

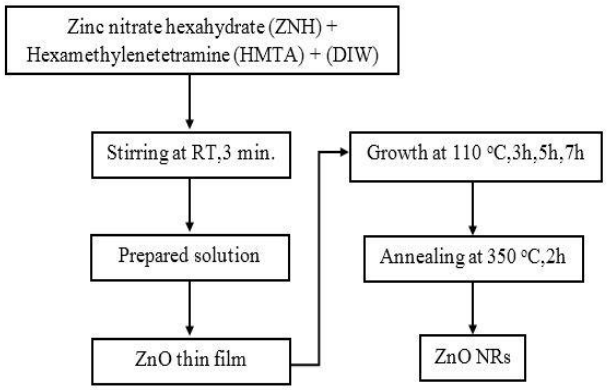


Fig. (2): ZnO NRs growth process.

2.4 Characterization

The morphology of the prepared ZnO NRs were analyzed using a field-emission scanning electron microscopy (FE-SEM) (Nova 450) operating at 10.0 KV. The crystallization and structural properties of ZnO NRs characterized by X-Ray Diffraction (XRD, Philips X'Pert PRO) with a Cu Kα radiation (λ = 1.54 Å). The composition of ZnO NRs films was studied by energy dispersion X-ray spectroscopy (EDX). A UV-visible spectrophotometer (1800 SHIMADZU) was used to study the optical properties of the ZnO NRs at RT. In addition, the optical and luminescence properties of the ZnO NRs was studied through photoluminescence (RF-5301 SHIMADZU).

3. RESULTS AND DISUCUSSION

3.1 XRD analysis

The crystal structure and microstructure of the as synthesized ZnO NRs and seed layer were studied through XRD. Fig. (3) shows the XRD pattern of the ZnO seed layer grown on glass substrate(So). The sample(So) had a random ship, meanwhile, the severity of the diffraction peaks corresponding to the directions (100), (002), and (101) weak due to use one layer. It is reported that the growth of NRs can be induced by the seed layer as nuclei centers on substrate surface [34]. The XRD patterns of the grown ZnO NR on glass substrate at pH10 and deposition time of [3h(S1),5h(S2),7h(S3)] using hydrothermal method show a strong

diffraction peak at 2θ value of (34.53°, 34.56°, 34.53°) respectively along (002) direction indicating the orientation along the c-axis, which consequently indicating the preferential orientation of ZnO NR (JCPDS card no. 36-1451). This would, suggest that the prepared ZnO nanorods have the wurtzite structure [35]. The films show an additional peaks around (31.86°, 36.33°, 47.63°, 56.66°,62.96°) are indexed to (100), (101), (102), (110), (103) orientation of ZnO materials. In respect to ZnO peaks, no other residual peaks were observed (JCPDS no. 75-0576) [36].

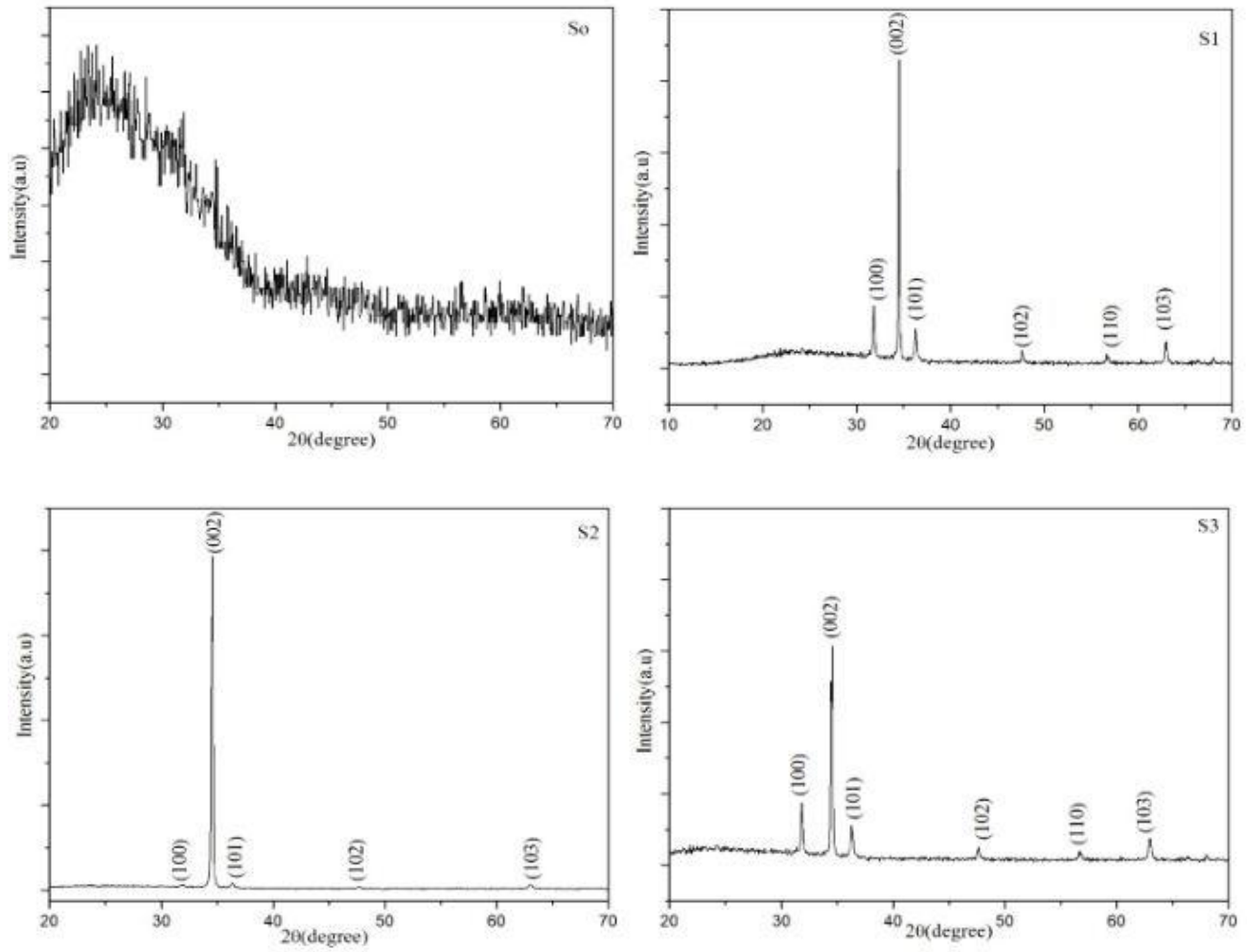


Fig. (3): (So) XRD pattern of ZnO seed layer, (S1) ZnO NRs grown on seed layer(3h), (S2) ZnO NRs grown on seed layer(5h), (S3) ZnO NRs grown on seed layer(7h).

The unit cell "*a*" and "*c*" of the crystalline ZnO nanorods with (002) preferred orientation is calculated using the equation (7) [35,37].

$$\frac{1}{d_{hkl}^2} = \frac{4}{3}\left(\frac{h^2 + hk + k^2}{a^2}\right) + \frac{l^2}{c^2} \qquad (7)$$

The values obtained for (S2) the unit cell *a* = 3.19 Å and *c* = 5.209 Å are in a good agreement with those reported in the JCPDS standard data (JCPDS card no. 36-1451). The calculated parameters are given in table 1.

Table 1. Lattice parameters of the ZnO nanorods

sample	*a* (Å)		*c* (Å)		c/a	
	Standard	Calculated	Standard	Calculated	Standard	Calculated
S1	3.253	2.99	5.215	5.19	1.603	1.735
S2		3.19		5.209		1.632
S3		2.99		5.19		1.735

3.2 EDX analysis

The (EDX) spectrum analysis was conducted to analyze the ZnO nanorods samples, as shown in fig. (4) graphs, in figures can notice the two main peaks were for Zn and O elements. To illustrates, the approximate atomic ratio of zinc and oxygen (Zn/O) were (0.87, 0.92 and 0.85) for samples S1, S2 and S3, respectively, which demonstrate the elemental composition of ZnO. Besides this, the (EDX) shows the presence of (Zn & O) only, which indicating for high purity. On the other hand, carbon that appeared in graphs were attributed to carbon conductive tape that used to attach the samples to the specimen holders, sodium and silicone that appeared in graphs were attributed to substrate used.

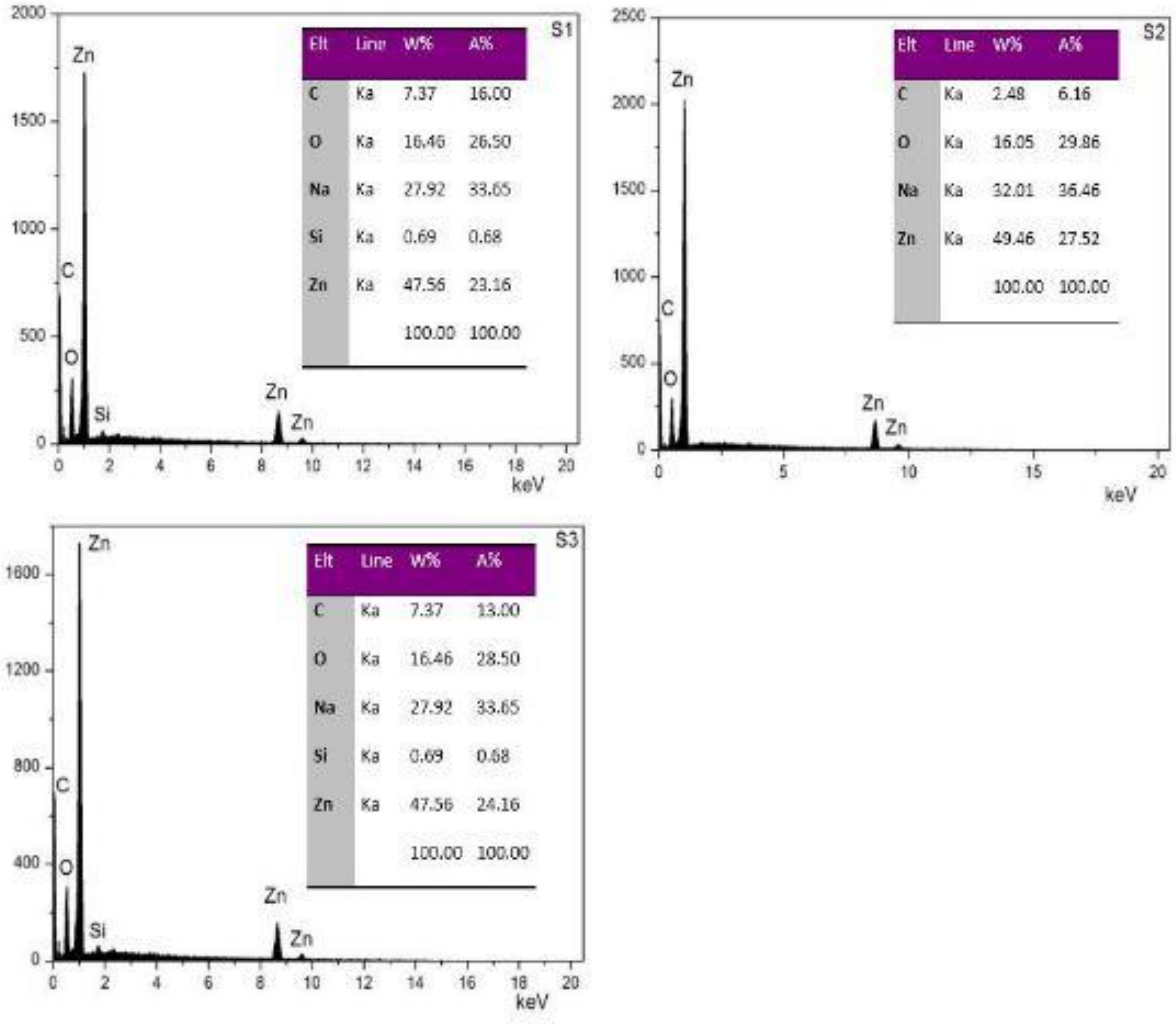


Fig. (4): The EDX of the ZnO NR

3.3 FE-SEM analysis

Fig. (5) (So) is the (FE-SEM) image of the ZnO seed layer grown onto a glass substrate at (200 nm) scale. The FE-SEM image shows a number of nano-sized particles on the surface of the substrate of these structures which were formed by small cluster of particles in size range of (20-37) nm in diameter. The seed layer surface observed is smoother and high density. Fig. (5) (S1, S2 and S3) shows the surface morphology of ZnO NRs grown on the seed layer by hydrothermal method at optimized pH 10 grown at 1μm and 200 nm scale. It indicates that all the as-grown ZnO nanorods are hexagonal facets and aligned preferentially along the c-axis direction. The growth of ZnO NRs by hydrothermal method on seed layered glass substrate leads to a vertical growth with the diameter ranging from (20-240) nm with perfect hexagonal shaped. The dimensional values of the nanorods diameter (D) can be seen in Fig. (5).

3.4 Optical Properties of ZnO Nanorods

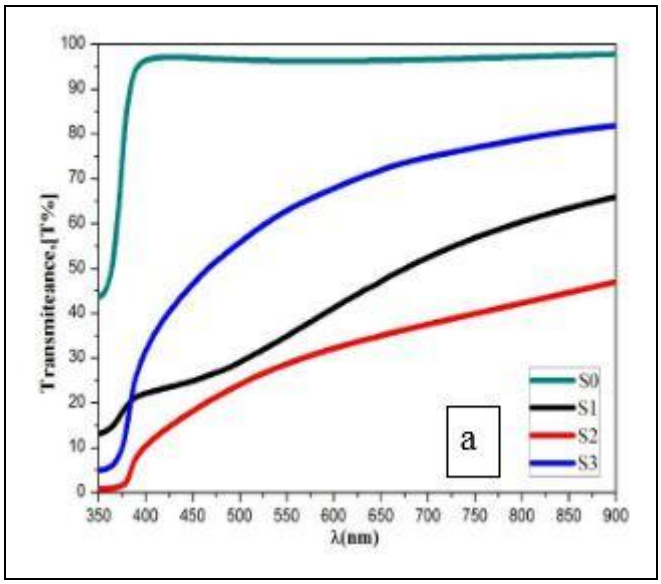
Optical band gap of ZnO NRs arrays films were evaluated from transmittance data through the well-known Tauc relation, for direct band gap semiconductors transition (equation (8)) [38]:

$$(\alpha h\nu)^n = B(h\nu - E_g) \quad (8)$$

where is the(α) absorption coefficient; B is an energy-independent constant; n is a constant that determines the type of the optical transition ($n = 2$ for allowed direct transitions and $n = 1/2$ for allowed indirect transitions), E_g is the optical band gap, h is the Planck constant, and ν is the frequency.

The optical band gap was obtained by extrapolating $(\alpha h\nu)^2$ *vs* $h\nu$. and the results are presented in fig. (6). The insets show the optical transmittance of the ZnO seed layers and the ZnO NRs arrays. The ZnO nanostructured films were found to be transparent in the visible range, with an average transmittance 95%. This high transmittance of the ZnO seed layers may be a consequence of its low thickness and small and highly connected ZnO grains. After the growth of ZnO NRs, the transmittance decreases to 40%. The few transparency (40%) of ZnO NRs array is due to their high columnar oriented grains and for being well-aligned perpendicular to the substrate, these results corroborate the FE-SEM surface images.

The estimated band gaps of ZnO NRs arrays were found to be (S1 and S3 (3.425eV), S2 (3.4eV)), for annealing temperature of 350 ^{o}C. It is noted that the band gap for ZnO is 3.37 eV. The increases of the band gap for ZnO NRs arrays can be explained due to the effect of quantum confinement. However, there are several parameters that may influence the optical band gap of a semiconductor material, like the residual strain, defects.



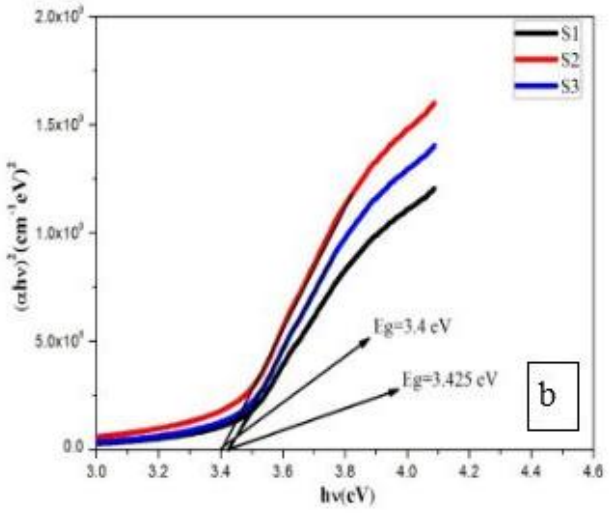


Fig. (6): (a)Optical transmission of seed layer and ZnO NRs films with different growth time, (So) ZnO seed layer, (S1) ZnO NRs grown on seed layer(3h), (S2) ZnO NRs grown on seed layer(5h), (S3) ZnO NRs grown on seed layer(7h). (b)Touch plot$(\alpha h\upsilon)^2$ versus photon energy ($h\upsilon$).

3.5 Photoluminescence (PL) analysis

The study of the luminescent properties of these ZnO nanostructures can shed some light on defects in the ZnO crystals and their potential as photonic materials. Normally ZnO nanostructures can exhibit a UV emission and visible emission (defect emission). In our case, a sharp and strong UV peak dominates the PL spectrum. Fig. (7) PL measurements were carried out at room temperature with excitation of a xenon lamp at 325 nm. To examine the effect of the growth time on the optical properties of the ZnO nanorods. All of the samples exhibit three different emission peaks are observed. One is the strong UV region and the other is the weak deep-level emission (DLE) at visible region. The UV emission is the exciton recombination related near-band edge emission (NBE), was due to the recombination of free exciton through an exciton-exciton collision process and the DLE usually accompanies the presence of structural defects and impurities. [12,33,35]. The photoluminescence spectra demonstrated a strong UV emission at 381nm and a visible emission at 462 nm,544 nm. The intensity of visible peak of sample 2 is lower as compared to the intensity of sample 1 and 3. The decrement in the intensity of visible region as well as higher UV/Vis. ratio for sample 2 suggests that sample 2 has good crystallinity.

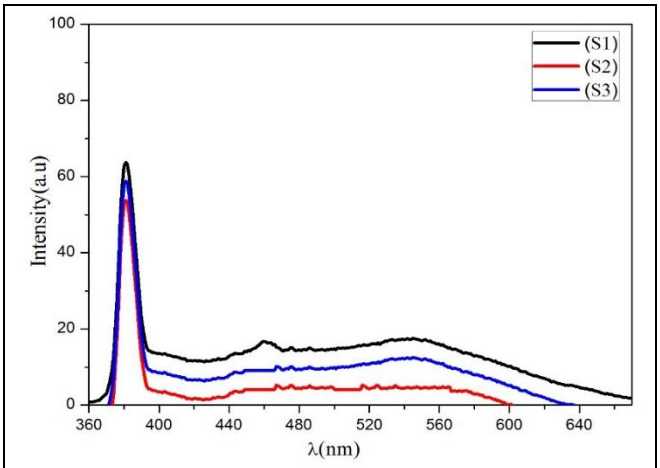


Fig. (7): Room-temperature photoluminescence spectra of ZnO NRs formed on seeds at different growth times, (S1) (3h), (S2) (5h), (S3) (7h).

3.6 Effect of growth time on ZnO nanorods

In order to investigate the effect of reaction time on morphology, three deposition times were selected. Namely, the layers were deposited for 3, 5 and 7 hours at the same temperature of 110 °C. Fig. 8 shows the effect of growth time on the size of NRs at different immersion and fixed annealing temperature 350 °C. Based on fig. 8(d-f), at the immersion and post-annealing temperature of respectively, the size of NRs increases from 46.25 nm to 162.50nm as the growth time increases. Overall, it can be observed that the size of NRs increase as the growth rate increases at fixed immersion temperature.

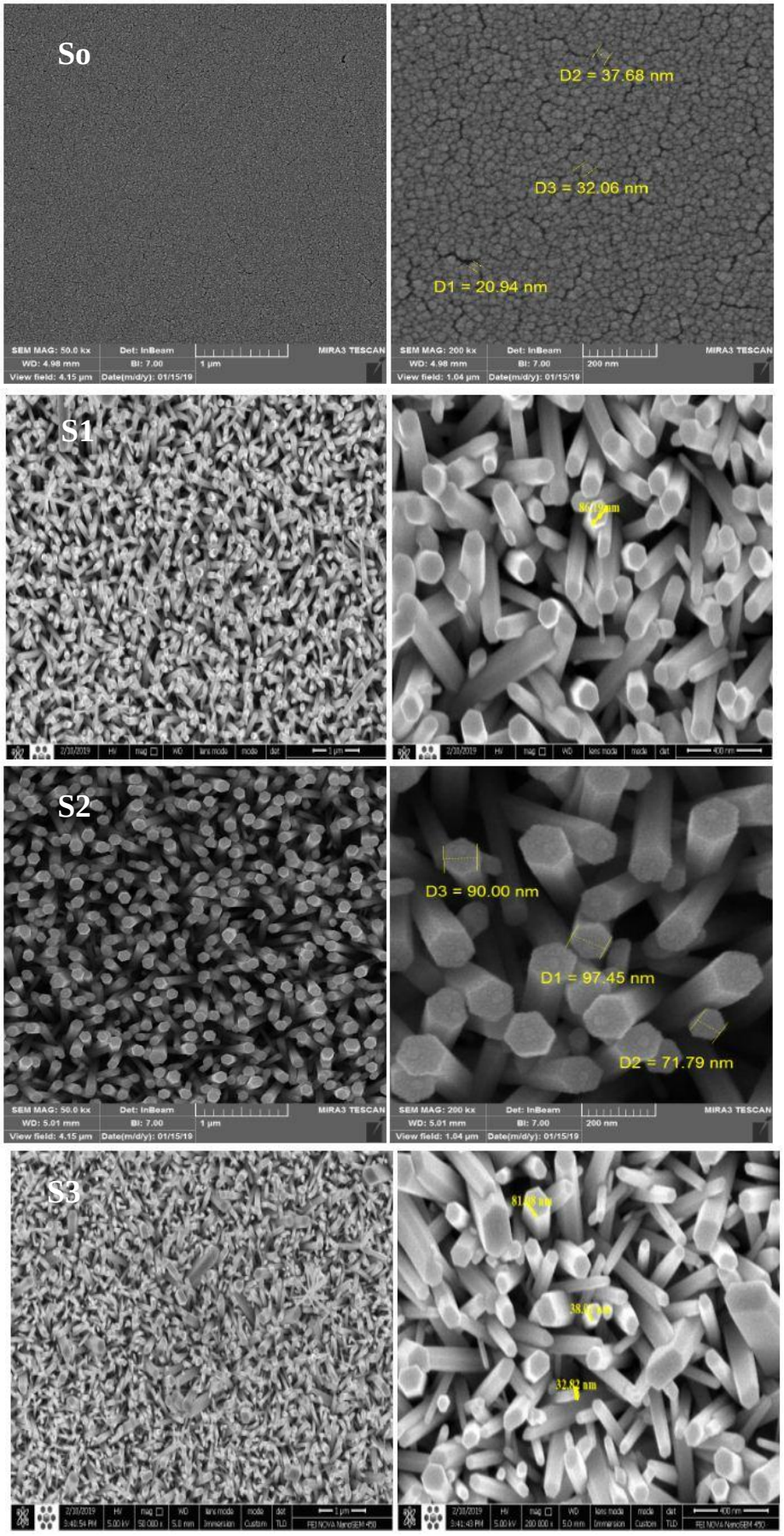


Fig. (5): FE-SEM images of (So) ZnO seed layer, (S1) ZnO NRs grown on seed layer(3h), (S2) ZnO NRs grown on seed layer(5h), (S3) ZnO NRs grown on seed layer(7h).

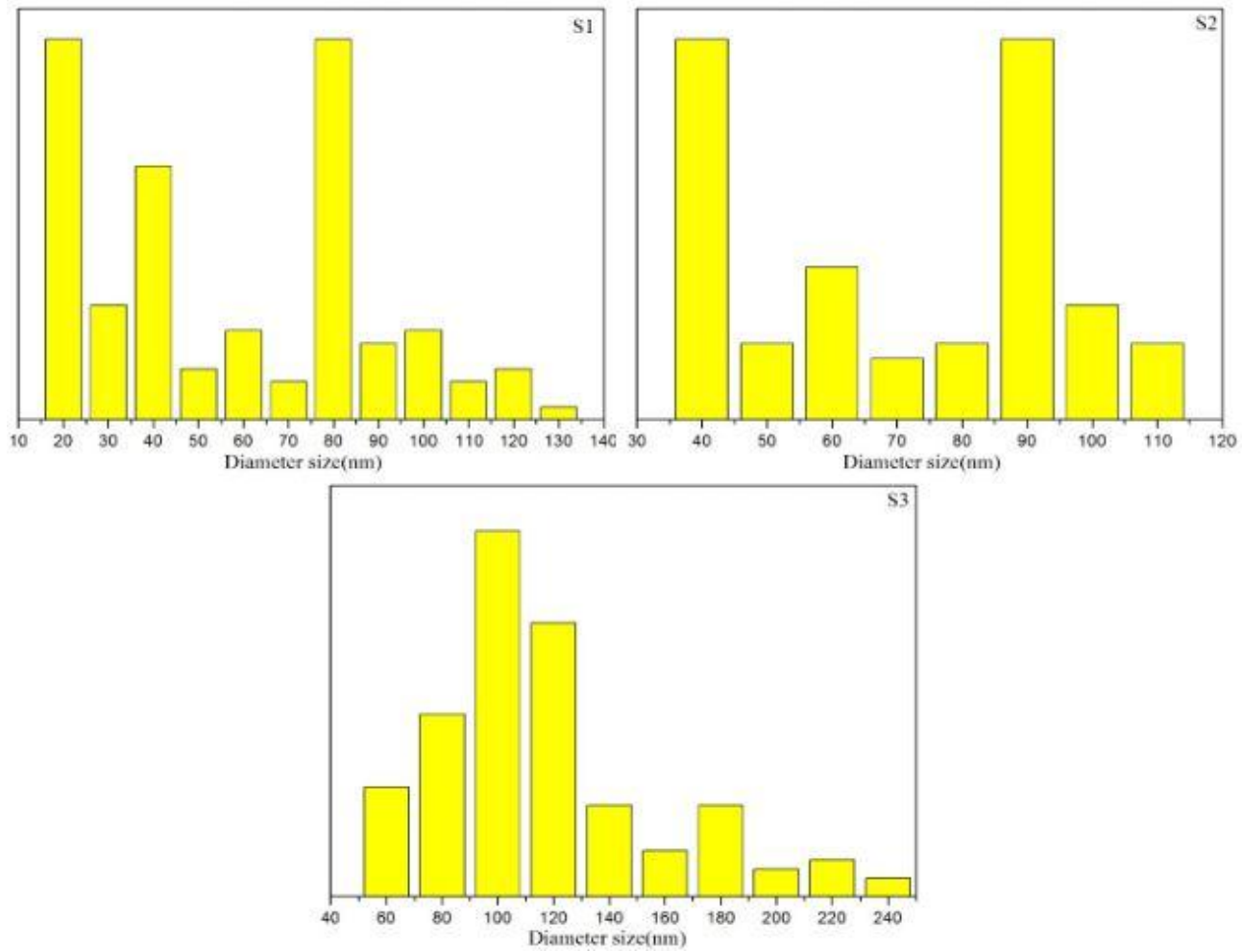


Fig. (8): Diameter distribution of the corresponding ZnO NRs, S1(3h), S2(5h), S3(7h).

4. Conclusions

In conclusion, have successfully synthesized well-aligned ZnO NRs on ZnO seed layer by the hydrothermal process at a new temperature. This method allowed obtaining homogeneous layers of ZnO NRs. The effects of growth time on the of ZnO NRs have been successfully investigated. The relation between diameter and reaction parameters was studied. As time increases, the diameter becomes bigger (up to 240 nm). The growth time of synthesis had an influence on the morphology of the film surface. When time was too low or too high, the ZnO rods were less porous structures were fabricated. For further investigation it is crucial to take into account seed layer morphology. It is also necessary to carry out test of repeatability.

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