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Structural, Optical, and Morphological Study of the Zinc Oxide Nano-Thin Films with Different Thickness Prepared by Pulsed Laser Deposition Technique

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Abstract

The goal of this investigation is to prepare zinc oxide (ZnO) nano-thin films by pulsed laser deposition (PLD) technique through Q-switching double frequency Nd:YAG laser (532 nm) wavelength, pulse frequency 6 Hz, and 300 mJ energy under vacuum conditions (10^{-3} torr) at room temperature. (ZnO) nano-thin films were deposited on glass substrates with different thickness of 300, 600 and 900 nm. ZnO films, were then annealed in air at a temperature of 500 °C for one hour. The results were compared with the researchers' previous theoretical study. The XRD analysis of ZnO nano-thin films indicated a hexagonal multi-crystalline wurtzite structure with preferential growth lines (100), (002), (101) for ZnO nano-thin films with different thicknesses of un-annealed samples and after annealing. While the UV-Visible spectrum manifested that the ZnO has a high absorption at UV range and wide energy gap values of (3.4, 3.42, and 3.46 eV) for the three thicknesses. The surface topography of the films evinced a rough surface which increased with increasing thickness, whereas the grain size decreased, and the average grain size was about 56.68 nm. Furthermore, the nano-thin films showed a granular morphology with a tendency to form smaller particles with increasing thickness.

Keywords: Zinc oxide, nano-thin films, PLD, Optical Energy Gap, XRD

دراسة تركيبية, بصرية و مورفولوجية لأغشية اكسيد الزنك الرقيقة–النانوية بسمك مختلف محضرة بتقنية الترسيب بالليزر النبضي

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الخلاصة

الهدف من هذا البحث هو تحضير أغشية رقيقة–نانوية من أكسيد الزنك (ZnO) بتقنية الترسيب بالليزر النبضي (PLD) من خلال Q–switching مزدوج التردد Nd: YAG (532 نانومتر) الطول الموجي، تردد النبض 6 هرتز، و 300 مللي جول من الطاقة تحت ظروف الفراغ (10^{-3} تور) في درجة حرارة الغرفة، على قواعد زجاجية مختلفة السمك 300, 600, 900 نانومتر. تم بعد ذلك تلدين أغشية ZnO في الهواء عند درجة حرارة 500 درجة مئوية لمدة ساعة

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واحدة. تمت مقارنة النتائج بالدراسة النظرية السابقة للباحثين. أشار تحليل XRD لأغشية ZnO الرقيقة النانوية إلى بنية wurtzite سداسية متعددة التبلور مع خطوط نمو تفضيلية (100)، (002)، (101) لأغشية ZnO النانوية بسمك مختلف بدون تلدين و بعد التلدين. بينما أظهر طيف الاشعة المرئية–فوق البنفسجية أن أكسيد الزنك لديه امتصاص عالي في نطاق الأشعة فوق البنفسجية وقيم فجوة طاقة واسعة (3.4، 3.42 و 3.46 الكترون.فولت)، على التوالي. أظهر الشكل الطوبوغرافي السطحي للأغشية سطح أكثر خشونة، وزادت خشونته مع زيادة السمك، بينما انخفض حجم الحبيبات، وكان متوسط حجم الحبيبات حوالي 56.68 نانومتر. بالإضافة لذلك اظهرت الأغشية الرقيقة النانوية شكلاً حبيبيا يميل الى تكوين جسيمات اصغر مع زيادة السمك.

1. Introduction

ZnO is an important semiconductor material of the II-VI group and has a wide band gap with a direct transition of about 3.3 eV, and a large value of excitons binding energy around 60 meV; moreover, it is non-toxic and widespread in nature [1]. With these properties, ZnO has potential use in many optoelectronic applications, such as ultra-violet light-emitting diodes (LED) [2], thermal, optical temperature sensors [3], gas sensors [4], and dye sensitive solar cells [5]. In solar cell device technology, ZnO is a promising transparent conducting oxides (TCO) material replacing the traditional indium tin oxide. Because of its low financial cost and excellent material properties, it is commonly used as a window layer [6]. ZnO thin films have been deposited by many methods, including spray pyrolysis [7], hydrothermal growth [8], molecular beam epitaxy [9], chemical vapour deposition [10], and pulse laser deposition(PLD) [11, 12, 13, 14]. Among numerous deposition techniques, the PLD technique has been considered as a promising approach for fabricating ZnO thin films because it permits the deposition of high-quality thin films at high oxygen ambient pressure and low growth temperature. PLD can successfully transfer the stoichiometry of a complex molecular structure compound, which is hard to obtain with other deposition techniques [15]. Several studies were devoted to the parameters affecting the particle size of zinc oxide via the PLD technique. Thermally activated deposition parameters, like desorption, adsorption, nucleation, surface diffusion, crystallization/re-crystallization, substrate surface roughness, and morphology of the deposited thin films of ZnO may be regulated by several factors which include background gas pressure, substrate temperature, laser energy density (i.e. laser impact), repetition rate, target to substrate distance, oxygen partial pressure in the chamber of deposition, post-growth annealing, and thickness of ZnO thin films formed leading to various property modifications [16]. Film thickness is an important factor to control the optical, morphological, and structural characteristics of the ZnO films. Longer deposition time leads to an enhanced thickness of the deposited ZnO thin films. Many researchers have studied the thickness effects on the ZnO thin films prepared by PLD [17, 18, 19, 20, 21].

The aim of this work is to investigate experimentally the structural, optical, topographical and morphological characteristics of different thicknesses of ZnO nano-thin film deposit by PLD approach and to compare the results with our previous theoretical study [22].

2. Experimental work

ZnO nano-thin films were deposited in a PLD system on glass substrates. The target was prepared from pure zinc oxide powder (purchased from Thomas Baker (chemicals) Pvt. Ltd.) (of (99%) purity) in the form of pellets (with 10 mm diameter, 2mm thickness, and 1gram weight) by employing pressing of 6 Ton. The glass substrate, after ultrasonically cleaned with ethanol, was placed on a holder at a 3 cm distance above the target (ZnO pellet) in parallel with the target surface. A Q-switched (2^{nd} harmonic) Nd: YAG laser (λ = 532 nm energy = 300 mJ) was utilized and operated at a repetition pulse frequency of 6 Hz under vacuum conditions (10^{-3} torr) using a rotary pump at the room temperature. ZnO nano-thin films were

prepared with different thicknesses of (300, 600, and 900 nm) by increasing the number of laser shots. ZnO films were then annealed in air at a temperature of 500 °C for one hour. The films thickness was measured approximately using the interferometer fringes method, which is an application of Fizeau fringes equal spacing using a He-Ne laser of 632.8 nm wavelength, as shown in Figure 1. The thickness of the 300 nm samples were measured and the other samples thicknesses were estimated empirically according to number of laser pulses (1 pulse = 3 nm). X-ray diffraction (XRD) spectra were obtained with an X-ray diffractometer (XRD6000 Labx, supplied by Shimadzu) using X-ray source (Cu-Kα) radiation of λ=1.5406Å. The optical properties of the ZnO nano-thin films at room temperature were investigated with UV-Vis spectrophotometer (Shimadzu 1800). The surface topography was studied with Atomic Force Microscope (AFM) (CSPM AA-3000 AFM supplied by Angstrom Co.), and surface morphology was studied with Field Emission Electron Microscope (FESEM) (INSPECT F50 (HR-SEM)).

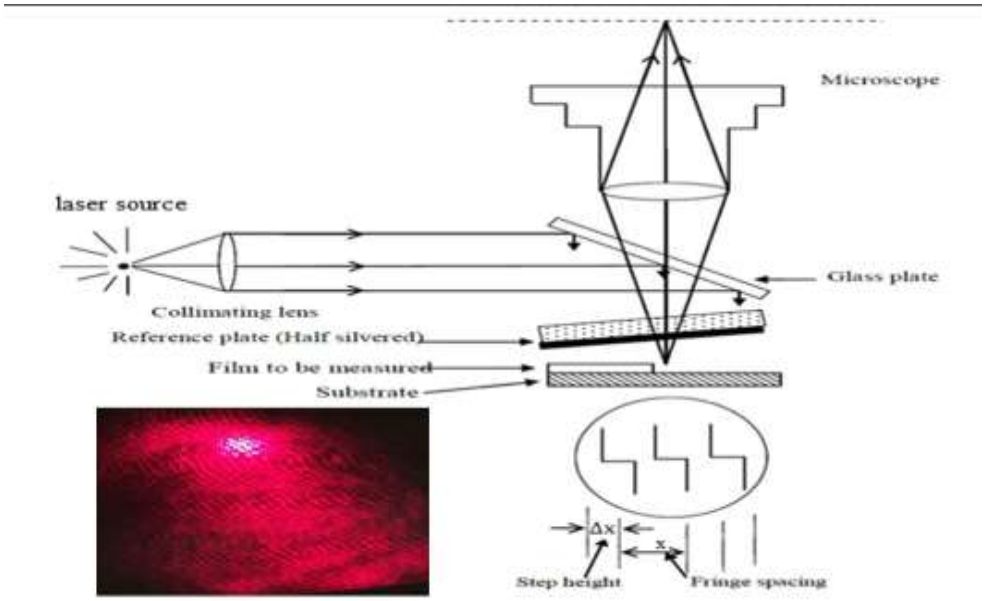


Figure 1: Interferometer arrangement to produce reflection Fizeau fringes.

3. Results and Discussion

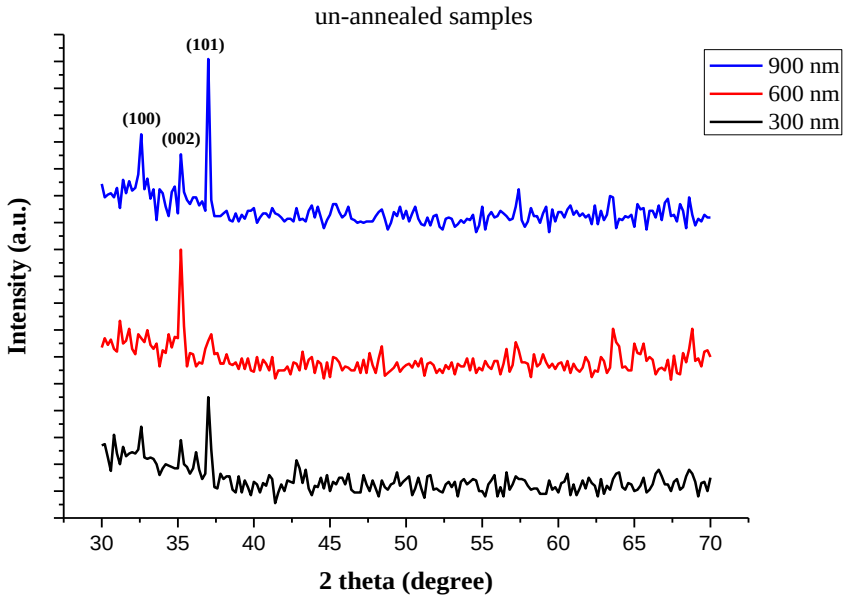
3.1 Structural Properties

XRD analysis was conducted on the crystalline structure of the ZnO nano-thin films as depicted in Figure 2. The diffractograms evince a polycrystalline films with a hexagonal wurtzite structure as judged by ICCD (card no: 50664), which matched well with those reported elsewhere [23, 24] and has a preferred growth along the c-axis orientation. The c-axis of a hexagonal cell is normal to the substrate which results in the columnar crystallites perpendicular to the surface of the substrate. The indexed peaks position preferential growth lines are (100), (002), (101) for all ZnO films. A small shift in the peaks position and peaks of higher FWHM were noticed, as compared with spectrum of a standard bulk which is attributed to the formation of more stoichiometric ZnO films. The small particle size of ZnO nano-thin films leads to broadened diffraction peaks. The crystal size of the films decreased with increasing the film thickness and the annealing temperature. These results matches those of Al-Assiri et al.[14] and of Zhu et al.[20]. It can be noticed that the nano-thin films polycrystalline structure tend to form nanoparticles with the increase of film thickness and annealing temperature. Annealing at this high temperature reduces the localized states and crystal defects. Also, it supports the diffusion of atoms absorbed on the substrate and accelerates the migration of atoms to the energy favorable positions, resulting in the

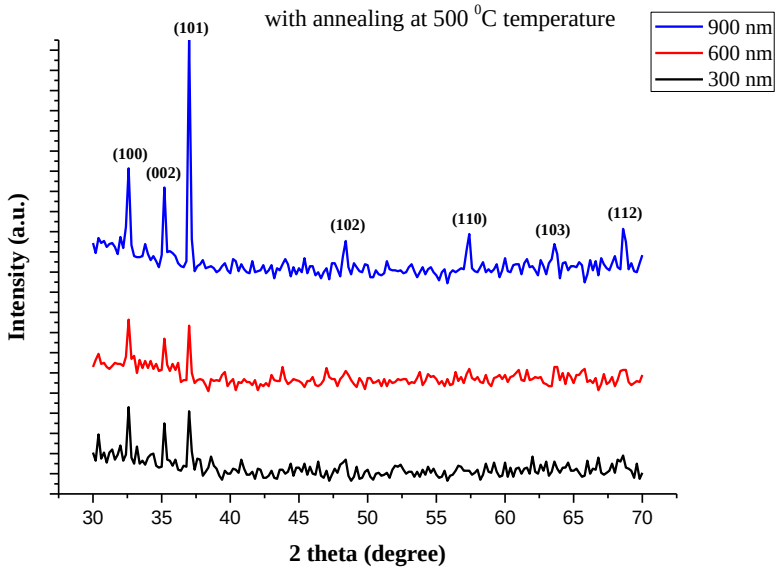
enhancement the crystallinity of the ZnO nano-thin films. The evaluated mean crystallite size (D) was determined using the Debye-Scherrer formula (Equation 1) [25], and the results are listed in Table 1:

$$D = \frac{0.9\,\lambda}{\beta cos\,\theta}\,, \tag{1}$$

Where: λ = the X-radiation wavelength, (1.5418 Å); β = the Full Width at Half Maximum (FWHM), (in the units of radians); and θ = the diffraction angle of maximum intensity, degree.



(a)



(b)

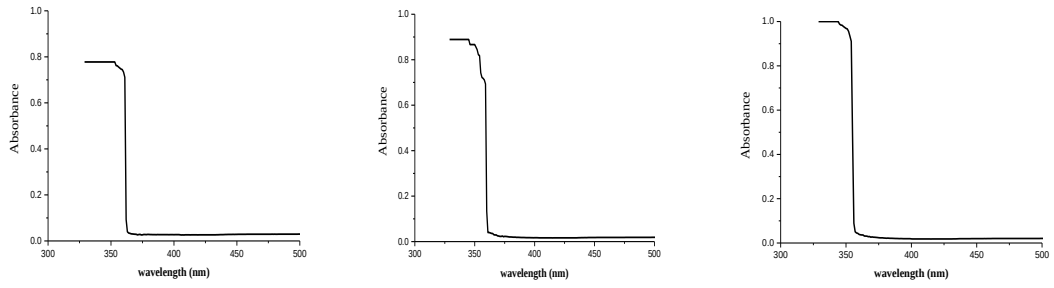
Figure 2: XRD patterns of (a) ZnO nano-thin films of (300, 600, and 900 nm) thicknesses, and (b) ZnO nano-thin films of (300, 600, and 900 nm) thicknesses after annealing temperature at 500 °C.

Table 1: Structural information from the XRD pattern for ZnO nano-thin films at different thicknesses of un-annealed films and after annealing at 500 °C temperature

Sample	hkl	Peak position (2θ)	FWHM β (deg.)	FWHM β (rad)	Crystal size D(nm)
600 nm thickness	(100)	32.6	0.381	0.0066	21.88
	(002)	35.2	0.81	0.0141	10.31
	(101)	37	0.359	0.0062	23.57
	(100)	32.4	0.693	0.01208	11.948
	(002)	35.2	0.481	0.0084	17.31
	(101)	37	0.723	0.0126	11.59
	(100)	32.6	0.484	0.0084	17.19
	(002)	35.2	0.874	0.0152	9.566
	(101)	37	0.511	0.0089	16.42
600 nm thickness after annealing	(100)	32.6	0.488	0.0085	16.99
	(002)	35.2	0.803	0.0140	10.386
	(101)	37	0.615	0.0107	13.66
	(100)	32.6	0.541	0.0094	15.36
	(002)	35.2	0.938	0.0163	8.92
	(101)	37	0.688	0.0120	12.18
	(100)	32.6	0.607	0.0105	13.75
	(002)	35.2	0.998	0.0174	8.356
	(101)	37	0.723	0.0126	11.6

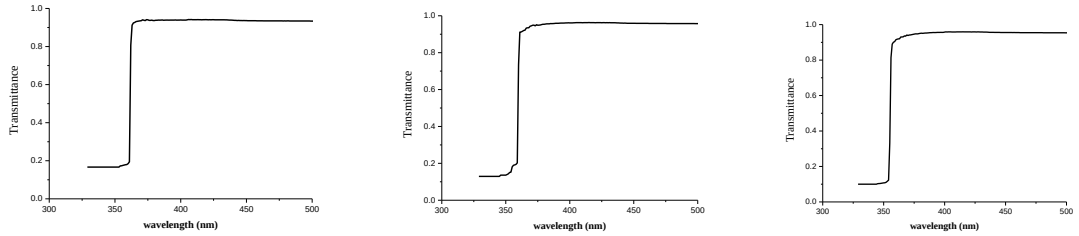
3.2 Optical characteristics

Absorbance, transmittance, and reflectance of the un-annealed ZnO nano-thin films of (300, 600, and 900 nm) thickness are shown in Figure 3, Figure 4, and Figure 5, respectively. These figures depict that the ZnO nano-thin films are of high absorption in the ultraviolet region. They also show a significant increase of absorbance with the increase of film thickness, which is in a good agreement with Beer- Lambert law. In addition the increase of thin film thickness (300, 600, and 900 nm) results in shifting the maximum absorption edge position towards shorter wavelengths of (362, 360, and 356 nm), respectively. This suggests the increase in the band gap energy with the increase of films thickness.



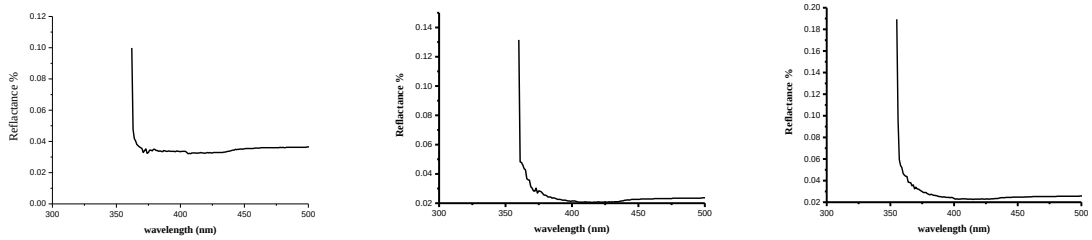
(a) **(b)** **(c)**

Figure 3: The absorbance spectra of ZnO of thickness: (a) 300 nm, (b) 600 nm, and (c) 900 nm.



(a) **(b)** **(c)**

Figure 4: The transmittance spectra of ZnO of thickness: (a) 300 nm, (b) 600 nm, and (c) 900 nm.



(a) **(b)** **(c)**

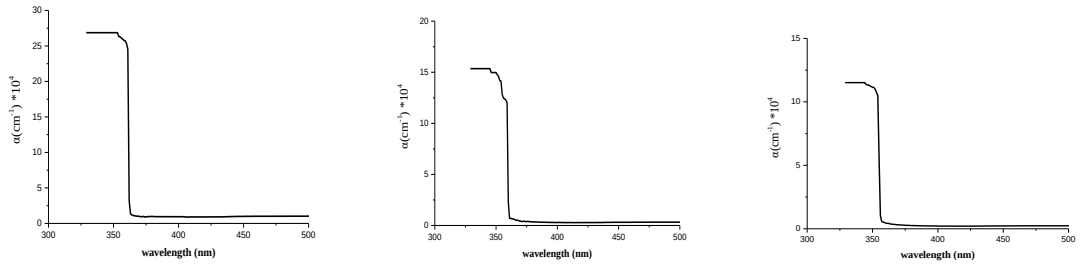
Figure 5: The reflectance spectra of ZnO of thickness: (a) 300 nm, (b) 600 nm, and (c) 900 nm.

The absorption coefficient (α) was determined as follow [23]:

$$\alpha = 2.303 \frac{A}{t}, \quad (2)$$

Where A = absorbance, and t = thin film thickness, nm.

Figure 6 displays the absorption coefficient versus the light wavelength for ZnO nano-thin films with 300, 600, and 900 nm thickness.



(a) **(b)** **(c)**

Figure 6: Absorption coefficient versus wavelength for the ZnO nano-thin films with thickness: (a) 300 nm, (b) 600 nm, and (c) 900 nm.

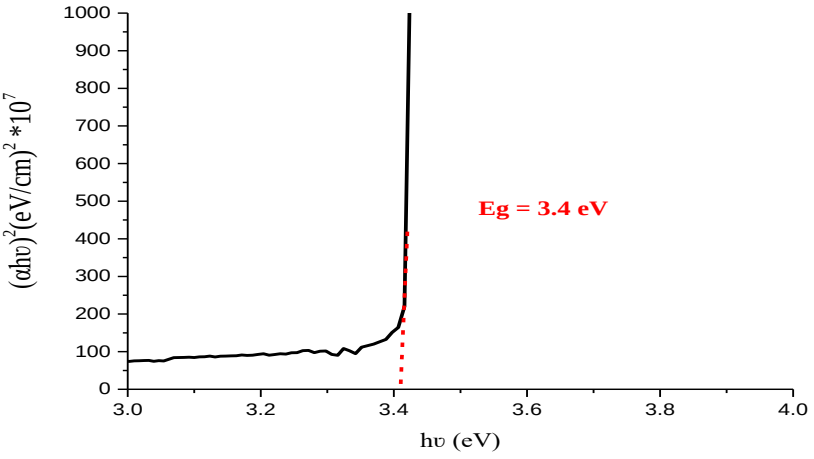
The absorption coefficient (α) maximum magnitudes are ($3.21*10^4$, $2.31*10^4$, and $1.03*10^4$) cm^{-1} for film thickness (300, 600, and 900 nm), respectively. This refers to a high probability of the allowed direct transition.

The optical energy band gap (*Eg*) was calculated using Tauc's equation [21]:

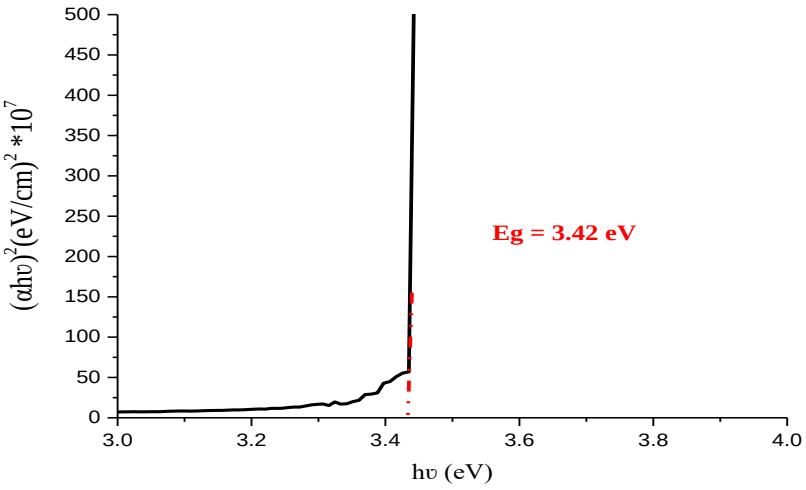
$$\alpha h\nu = B(h\nu - E_g)^n , \quad (3)$$

Where: B is a constant, hν is the incident photon energy(eV); Eg is the optical energy band gap(eV); and n=1/2 for the direct transition.

The values of the optical energy band gap (listed in Table 2) were obtained from extrapolation of the linear part of the plots of $(\alpha h\nu)^2$ vs. $(h\nu)$ (as shown in Figure 7). The estimated values of the energy band gaps are (3.40, 3.42, and 3.46 eV) which were higher than that of the bulk ZnO (3.3 eV) [1]. These results agree very well with our previous theoretical study, where the energy band gap values of (3.35-3.096 eV) decreased with increasing particle size due to quantum confinement [22]. It has been noticed that band gap magnitudes increase with increasing the thin film thickness. This increase is related to the decrease of the particles size (this result agrees with those of Nithya and Rugmini Radhakrishnan[24] and of Kumar et al.[25]), this means that the optical band gap values are directly proportional to the crystallinity of the thin film [26].



(a)



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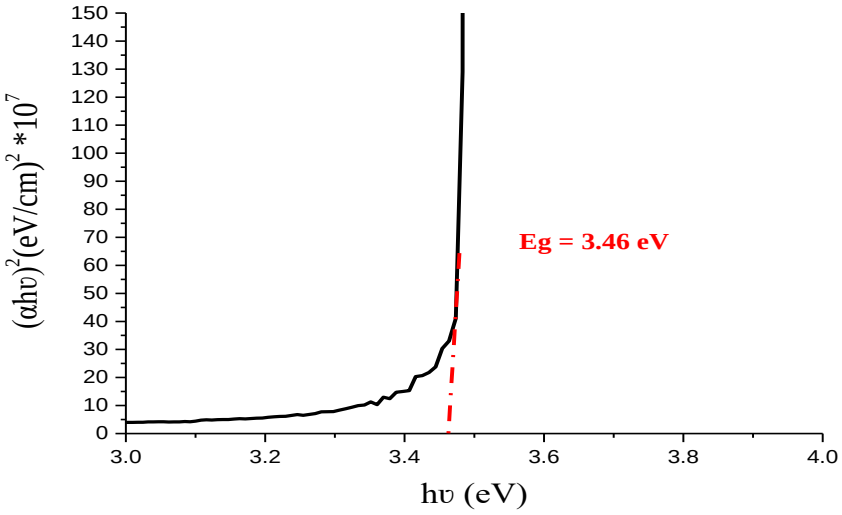


(c)

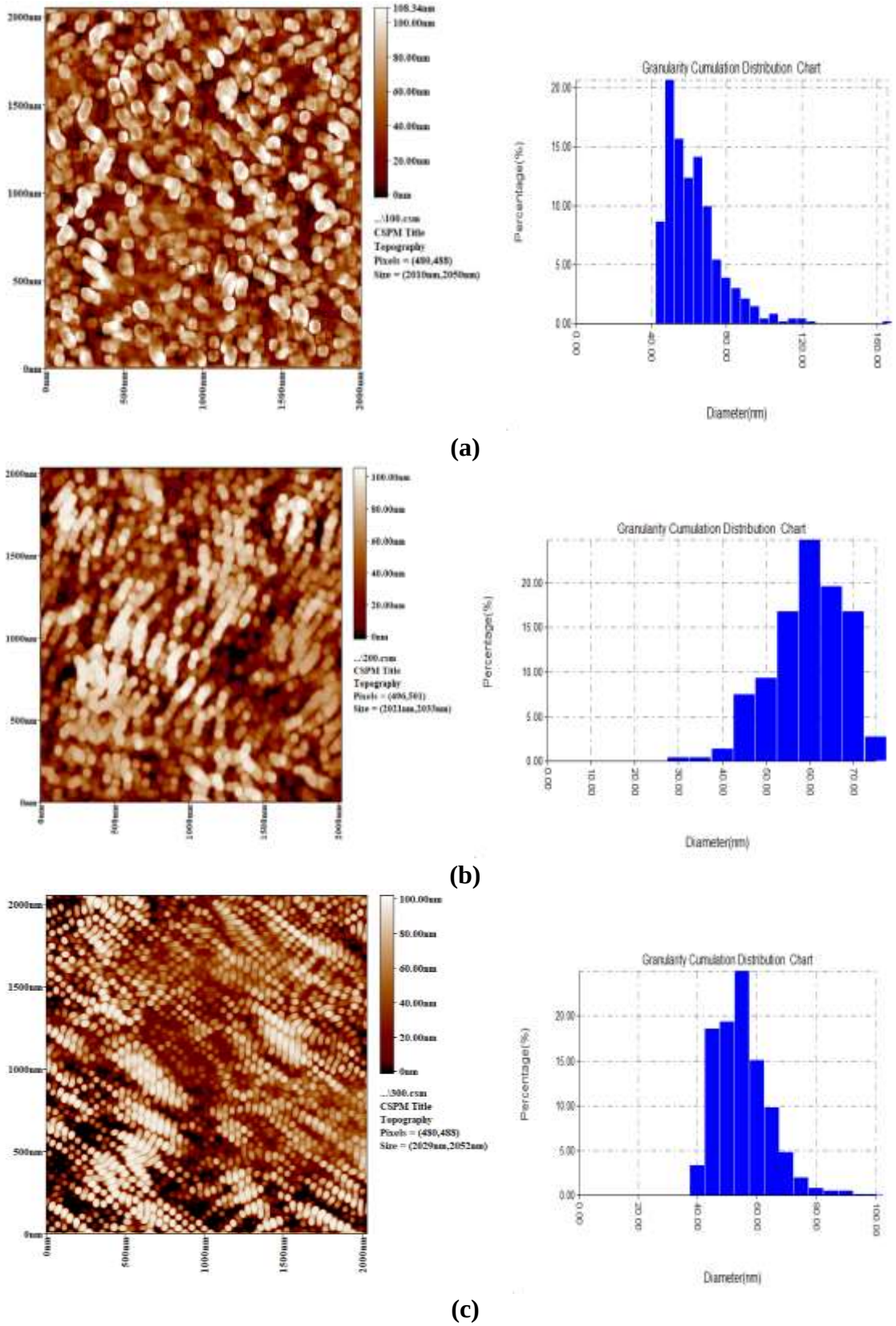
Figure 7: $(\alpha h\nu)^2$ vs. $(h\nu)$ plots for un-annealed ZnO nano-thin film of thickness: (a) 300 nm, (b) 600 nm, and (c) 900 nm.

Table 2: The optical energy band gaps for un-annealed ZnO nano-thin films of different thicknesses compared with the theoretical values of ZnO wurtzoid.

Samples	Optical energy gap (E_g)
300 nm thickness	3.40 eV
600 nm thickness	3.42 eV
900 nm thickness	3.46 eV
ZnO nanostructure wurtzoids [22]	(3.096 – 3.35) eV

3.3 Topography Properties

The surface topography of the un-annealed ZnO samples were examined with a non-contact mode atomic force microscope (AFM). Figures 8 and 9 show the 2 and 3 dimensions AFM images of the deposited ZnO nano-thin films with different thickness values. The average grain size and roughness values corresponding to the nano-thin film thicknesses (300, 600, and 900 nm) are shown in Table 3. As the thickness increased, a decrease in the observed grain size is manifested but with increase of the roughness. The grains are uniformly distributed as the thickness increases due to decrease of the tensile strain in films, which results in a superior quality of the crystallinity. While the roughness increases due to more grain boundaries. These results agree well with those of Christoulakis et al. [18]. The average grain size was estimated from the images to be equal to 56.68 nm.



(a)

(b)

(c)

Figure 8: AFM images of un-annealed ZnO nano-thin films deposited with different thickness values: (a) 300 nm, (b) 600 nm, and (c) 900 nm in two dimensions with granularity cumulative distribution chart

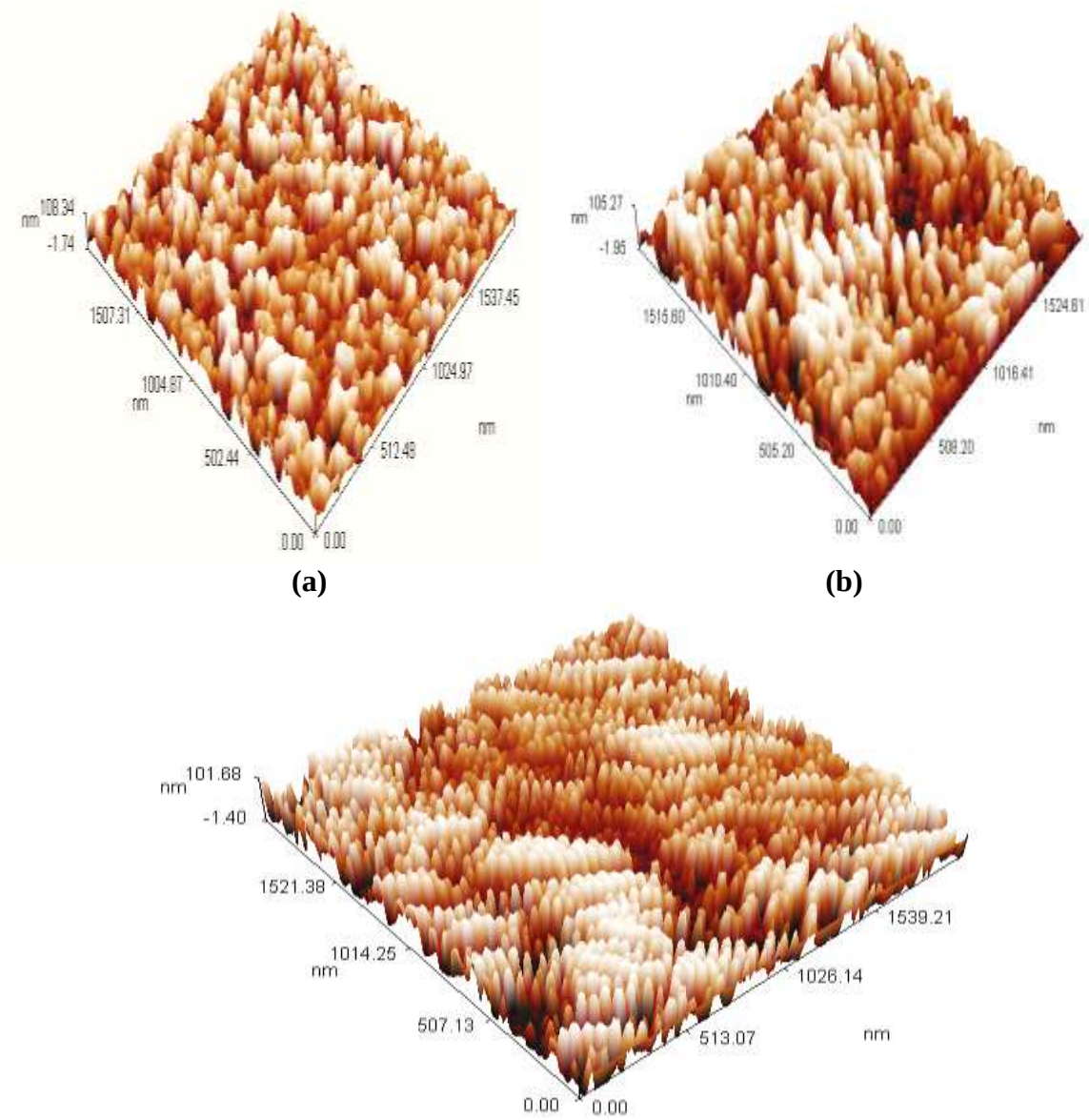


Figure -9: AFM images of un-annealed ZnO nano-thin films deposited with different thickness values: (a) 300 nm, (b) 600 nm, and (c) 900 nm in three dimensions.

Table 3: Topography information from the AFM images for un-annealed ZnO nano-thin films

Sample thickness (nm)	Average roughness (nm)	Grain size (nm)	Average grain size (nm)
300	24.7	60.22	56.68
600	24.8	57.13	
900	25.5	52.71	

3.4 Morphology properties

Field emission electron microscope (FESEM) was used to study un-annealed ZnO nano thin films morphology, as shown in Figure 10. The analysis revealed that all grown films with different thickness (300, 600, and 900 nm) have granular morphology with a tendency of

forming smaller particles with increasing thickness. These results which matches those of the XRD, UV- Visible and AFM.

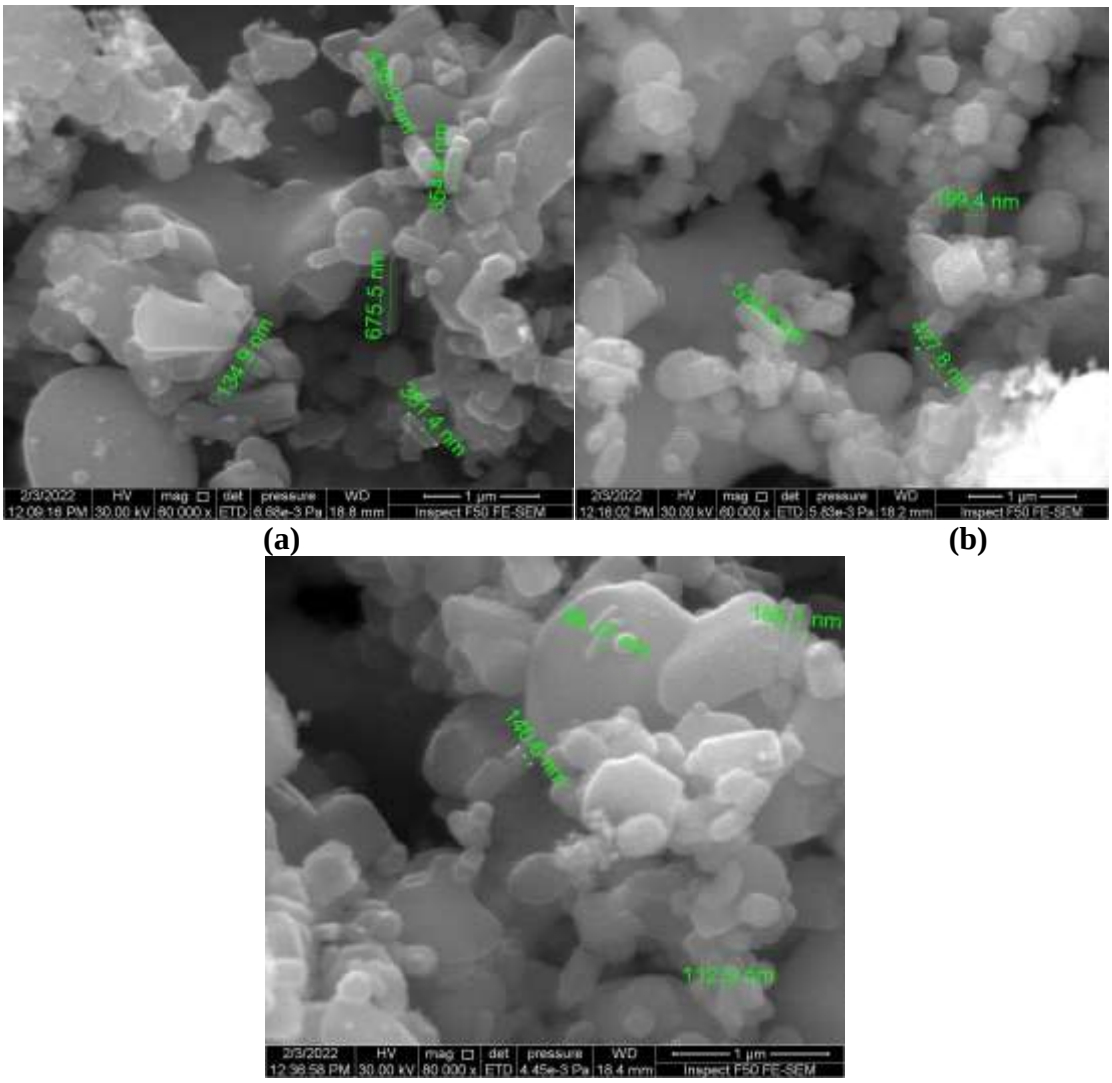


Figure 10: FESEM images of un-annealed ZnO nano-thin films deposited with different thickness values: (a) 300 nm, (b) 600 nm, and (c) 900 nm.

4. Conclusions

ZnO nano thin films can be used in optoelectronic devices, especially in UV-light emitting diode as well as in gas sensors with improved sensitivity applications. The films were successfully deposited with different thicknesses via PLD technique. X-ray diffraction results indicated a wurtzite hexagonal multi-crystalline structure for ZnO nano-thin films with crystal size decreasing with increasing film thickness and with annealing temperature. ZnO thin films showed high absorption at UV range and high transmittance to visible light. With increasing nano-thin films thickness, the optical energy direct band gap values increased and the grains size decreased because of the quantum confinement effect These results matched other experimental results and the researchers' previous theoretical results.

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