Synthesis Of Nanostructured TiO_2 Thin Films By Pulsed Laser Deposition (PLD) And The Effect Of Annealing Temperature On Structural And Morphological Properties

Sarmad Sabih Al-Obaidi
Ali Ahmed Yousif
Dept.of Physics/ College of Education/University of Al-Mustansiriyah

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Abstract

In this work, nanostructured TiO_2 thin films were grown by pulsed laser deposition (PLD) technique on glass substrates. TiO_2 thin films then were annealed at 400-600 °C in air for a period of 2 hours. Effect of annealing on the structural and morphological were studied. Many growth parameters have been considered to specify the optimum conditions, namely substrate temperature (300 °C), oxygen pressure (10^{-2} Torr), laser fluence energy density (0.4 J/cm^2), using double frequency Q-switching Nd:YAG laser beam (wavelength 532nm), repetition rate (1-6 Hz) and the pulse duration of 10 ns. The results of the X-ray test show that all nanostructures tetragonal are polycrystalline. These results show that grain size increase from 19.5 nm to 29.5 with the increase of annealing temperature. The XRD results also reveal that the deposited thin film, annealed at 400 °C of TiO_2 have anatase phase. Thin films annealed at 500 °C and 600 °C have mixed anatase and rutile phases. Full Width at Half Maximum (FWHM) values of the (101) peaks of these films decrease from 0.450° to 0.301° with the increase of annealing temperature. Surface morphology of the thin films have been studied by using atomic force microscopes (AFM). AFM measurements confirmed that the films have good crystalline and homogeneous surface. The Root Mean Square (RMS) value of thin films surface roughness are increased with the increase of annealing temperature.

Keywords: Titanium Dioxide, Pulsed Laser Deposition, Structural, Morphology, TiO_2 Films

Introduction

Nanocrystalline titanium dioxide thin films have important applications in the field of optoelectronic materials. TiO_2 is an important photocatalytic material [1], which can be used in the form of thin films in dye-sensitized solar cells [2] and anti-reflection coatings [3]. It is known that the TiO_2 films have excellent photocatalytic and photoinduced hydrophilic properties for environmental applications, such as air purification, sterilization, antifogging, and self-cleaning [4, 5]. Titanium dioxide occurs in three crystalline polymorphs: rutile, anatase and brookite [6]. Anatase has attracted attention for its prominent photocatalytic activity [7]. It is a metastable phase of TiO_2 and converts to the rutile phase, which is thermodynamically stable, on high temperature annealing [8]. Anatase and rutile phases crystallize in tetragonal structure while brookite crystallizes in orthorhombic structure. Many deposition methods have been used to prepare TiO_2 thin films, such as electron-beam evaporation [9,10], ion-beam assisted deposition [11,12], DC reactive magnetron sputtering [13], RF reactive magnetron sputtering [14,15], sol-gel dip coating method [16,17], sol-gel spin coating method [18], chemical vapor deposition [5], plasma enhanced chemical vapor deposition [19] and pulsed laser deposition (PLD) [20]. The laser ablation technique is what makes PLD attractive for thin film deposition, because the ablation of the target preserves its stoichiometry in the thin film. The PLD process is simple and versatile, these characteristics have made it possible to successfully deposit high quality thin films of various materials such as oxides, high-temperature superconductors, magnetoelectrics, and ceramics [21]. Pulsed laser deposition proved to be a favorable technique for the deposition of titanium dioxide at different technological conditions on different substrates. That supposed to result in the different structural and micro structural properties, different surface morphology of the nanostructures to be obtained [22]. For practical applications, deposition parameters have to be optimized to achieve the desired structure and properties in the films. The pulsed laser deposition technique can produce adherent and uniform film over wide areas. The deposit stoichiometry can also be well-controlled. A number of studies on the deposition of TiO_2 films by pulsed laser have appeared in the literature recently [23- 26]. Thin film properties such as crystallinity, particle size, degree of homogeneity, etc. depend largely on annealing temperature and substrate topography [27].

In this study, the influence of annealing over the range 400 to 600 °C on the structural properties measured by XRD technique, and morphological properties measured by AFM properties of TiO_2 thin films was investigated.

Experimental Details

Titanium dioxide from ASDGF Company with a titanium target of 99.99% purity on glass slides as substrates. The powder was pressed under 5 tons to form a target with 2.5 cm diameter and 0.4 cm thickness. Glass slides each with area 3 x 2 cm^2 were cleaned by alcohol with ultrasonic waves (Cerry PUL 125 device) for 10 minutes in order to remove the impurities and residuals from their surfaces. Thin films were deposited by using pulsed laser deposition in University of Technology by employing a Q switched Nd: YAG laser at wavelength 532 nm with 0.4 J/cm^2 of energy density, pulse width 10 ns and repetition frequency 6 Hz. Uniform ablation ensured by rotating the target at constant speed. The focused Nd:YAG Second Harmonic Generation (SHG) Q-switching laser beam incident on the target surface making an angle of 45° with it. The films were deposited on glass substrate at temperatures 300 °C. The pulsed laser deposition experiment was carried out inside a vacuum chamber (10^{-2} Torr). The substrates deposited at 300 °C with TiO_2 annealed at 400 °C, 500 °C and 600 °C in University of Al-Mustansiriyah using an electric furnace for 2 h in air. The crystallinity of the prepared films was analyzed using X-ray Diffraction (XRD) measurements (Shimadzu 6000 made in Japan) in Ministry of Science and Technology using

Cu Kα radiation (λ=1.5406 Å) and operating at an accelerating voltage of 40 kV and an emission current of 30 mA. Data were acquired over the range of 2θ from 20° to 60°.

The XRD method was used to study the change of crystalline structure. For morphological investigations, Atomic Force Microscopy (AFM) images were recorded by using nanoscope scanning probe microscope controller in a tapping mode (made in USA). AFM images were used to observe the surface roughness and topography of deposited thin films. Measurements were made AFM at the Ministry of Science and Technology.

Experimental Results

Throughout studying the X-ray diffraction spectrum, we can understand the crystalline growth nature of TiO_2 thin films prepared by pulsed leaser deposition on glass substrates at 300 °C at different annealing temperatures (400, 500, and 600) °C with a fixed annealing time of 2 h in air. Figure (1) shows X-ray diffraction patterns for TiO_2 films. We compared deposited film at 300 °C with annealed films at 400 °C, 500 °C and 600 °C as shown in Figure (1). As-deposited TiO_2 film at 300 °C is found to be crystalline and possesses anatase structure as it shows few peaks of anatase (101) and (004), while film annealed at 400 °C having peaks of anatase (101), (004) and (200), film annealed at 500 °C having peaks of anatase (101), (004), (200) and rutile (110) and film annealed at 600 °C having peaks of anatase (101), (004), (200) and rutile (110), (211).

The X-ray spectra show well-defined diffraction peaks showing good crystallinity, it was found that all the films were polycrystalline with a tetragonal crystal structure and no amorphous phase is detected. The diffraction peaks are in a good agreement with those given in JCPD data card for TiO_2 anatase and rutile [28]. It was observed that the intensities of the peaks of few TiO_2 planes increased slightly with the increase of annealing temperature. In addition, the location of the (101) peaks is shifted to lower 2θ angles from 2θ=25.27 ° to 2θ=25.11 °. For a crystalline phase to develop, the depositing atoms should have sufficient energy. High substrate temperatures can achieve the sufficient energy to generate crystalline phases [29]. X-ray diffraction analysis revealed that TiO_2 thin films are amorphous if the temperature substrate is lower than 300 °C [23]. It was found that anatase films were deposited and crystallized effectively for heated substrate at 300 °C and working pressure of 10^{-2} Torr. The reason may be that the kinetic energy of the particle is high enough to initiate crystallization. For the samples annealed at 500 and 600 °C, other characteristic peaks of anatase and rutile phase. These results are in agreement with other reports on the mixed phase TiO_2 by PLD method [26,30,31].The transformation from anatase to rutile occurs at temperatures higher than 500 °C under vacuum.

The increase in peak intensity indicates an improvement in the crystallinity of the films. This leads to decrease in Full Width at Half Maximums (FWHM) of peak and increase in grain size. The lattice constants and the relative intensity ratio, in the diffraction pattern of TiO_2 films are given in table (1). The lattice constants obtained are found to be in good agreement with JCPD.

The values of Full Width at Half Maximum (FWHM) of the peaks decreases with annealing temperature, this goes in agreement with the previous work [32]. The average grain size (g) in thin films is calculated using Scherer's formula: [33]

$$g = (0.94\ \lambda) / [\Delta_{(2\theta)} \cos\theta] \quad \ldots\ldots\ldots\ldots(1)$$

Where (λ) is the x-ray wavelength (Å), $\Delta_{(2\theta)}$ FWHM (radian) and (θ) Bragg diffraction angle of the XRD peak (degree).

The values of average grain size listed in table (2) increase with the increase of annealing temperature for TiO_2 thin films. The average grain size and Full Width at Half Maximum (FWHM) of the (101) plane as a function of annealing temperature for TiO_2 thin films are shown in figure (3). The micro strain depends directly on the lattice constant (c) and its value related to the shift from the JCPD standard value which could be calculated using the relation:[34]

$$Strain(\delta) = \frac{|c - c_O|}{c_O} \times 100\% \quad ....(2)$$

Where *(c)* and (c_o) are the lattice parameters of the thin film from experimental worked and TiO_2 thin film obtained from JCPDS respectively.

The film annealed at 600 ºC temperature showed the maximum compressive strain (3.640×10^{-3}), which decreased to nearly zero at 500 ºC, as shown in table (2). The calculation of the film stress is based on the strain model, which could be calculated by using the relation:[34] as shown in table (2).

$$S_s = \frac{2c^2{}_{13} - c_{33}(c_{11} + c_{12})}{2c_{13}} \times \frac{c - c_O}{c_O} \quad ....(3)$$

The values of the elastic constants from single crystalline TiO_2 are used, c_{11}=208.8 GPa, c_{33}=213.8 GPa, c_{12}=119.7 GPa and c_{13}=104.2 GPa [34].

To describe the preferential orientation, the values of texture coefficient (T_c) of the thin films are listed in table (2). The texture coefficient is calculated by using the relation: [35]

$$T_C(hkl) = \frac{I(hkl)/I_0(hkl)}{N_r^{-1}\sum I(hkl)/I_0(hkl)} \quad ....(4)$$

Where (I) is the measured intensity, (I_o) is the JCPDS standard intensity, (N_r) is the reflection number and (hkl) is Miller indices.

For crystal plane (101), the values of texture coefficient decrease with the increase of annealing temperature as shown in figure (2).This is a usual result because the increase of annealing temperature causes an increase in the surface roughness.

The surface morphology of all the TiO_2 films is presented by AFM images in tapping mode. The surface morphology reveals the Nano-crystalline TiO_2 grains. Figure (3) shows the three dimensional AFM images of the TiO_2 thin films deposited at 300 ºC and annealed at different temperatures (400, 500 and 600) ºC. The surface morphology of the TiO_2 thin films changed with the different annealing temperatures, as observed from the AFM micrographs figure (3) proves that the grains are semi uniformly distributed within the scanning area (10 μm × 10 μm), with individual columnar grains extending upwards.

The values of the root mean square (RMS) and surface roughness of TiO_2 films are shown in the table (3), i.e. the root mean square (RMS) and surface roughness increased with the increase of annealing temperature, this result is in agreement with the previous work [36].

In general as the annealing temperature increases, the RMS, roughness of the TiO_2 films and the grain size increase. The surface roughness of the TiO_2 thin films increases with film thickness, annealing temperature, and annealing time [37].

Conclusion

XRD results reveal that the deposited thin film and annealed at 400 °C of TiO_2 have a good Nanocrystalline tetragonal anatase phase structure. Thin films annealed at 500 °C and 600 °C have mixed anatase and rutile phase structure. And it is observed that the TiO_2 films exhibit a polycrystalline having (101), (110), (004), (200), (211) planes, the peak intensities, micro strain (δ), grain size (g) increases and texture coefficient (T_c) decreases with the increase of annealing temperature.

AFM results showed the slow growth of crystallite sizes for as-grown films and annealed films at 400 to 600 °C. The values of the root mean square (RMS) and surface roughness of TiO_2 films increased with the increase of annealing temperature.

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Table No.(1): Lattice constants and interpllanar spacing of TiO_2 thin films.

Temperature (°C)	□□□ □degree)	(hkl)	Interplanar spacing, d Å	Lattice constant Å		c/a
				a	c	
As-deposited at 300	25.27	A(101)	3.15	3.3439	/	2.842
	37.83	A(004)	2.37	/	9.5050	
400	25.2	A(101)	3.53	3.8	/	3.502
	37.81	A(004)	2.37	/	9.5098	
	48.1	A(200)	1.89	3.7803	/	
500	25.17	A(101)	3.53	3.8	/	3.503
	37.79	A(004)	2.38	/	9.5147	
	48.12	A(200)	1.89	3.7788	/	
	27.47	R(110)	3.24	4.5881	/	/
600	25.11	A(101)	3.54	3.8170	/	2.497
	37.72	A(004)	2.38	/	9.5317	
	48	A(200)	1.89	3.7877	/	
	27.41	R(110)	3.25	4.5979	/	0.641
	54.35	R(211)	1.68	/	2.9484	

Table No. (2): Structural properties for TiO_2 thin films.

Temperature (°C)	□□□ □degree)	**(hkl)**	**Stress S_s (GPa)**	**Strain δ (10^{-3})**	**FWHM°**	**Average Grain Size(nm)**	**Texture (T_c)**
As-deposited at 300	25.27	A(101)	0.218	0.9354	0.450	19.5	1.813
	37.83	A(004)			0.440		
400	25.2	A(101)	0.098	0.4225	0.421	21	1.126
	37.81	A(004)			0.412		
	48.1	A(200)			0.410		
500	25.17	A(101)	-0.019	0.084	0.352	24	1.154
	37.79	A(004)			0.400		
	48.12	A(200)			0.349		
	27.47	R(110)			0.334	25.6	
600	25.11	A(101)	-0.435	1.8709	0.301	28.5	0.952
	37.72	A(004)			0.288		
	48	A(200)			0.338		
	27.41	R(110)	0.849	3.640	0.315	29.5	
	54.35	R(211)			0.293		

Table No.(3): Morphological characteristics from AFM images for TiO_2 thin film

Temperature °C	***Roughness average (nm)***	***Root Mean Square (RMS) (nm)***
As-deposited at 300	*46.5*	*60.5*
400	*76.6*	*95*
500	*84.3*	*105*
600	*88.6*	*114*

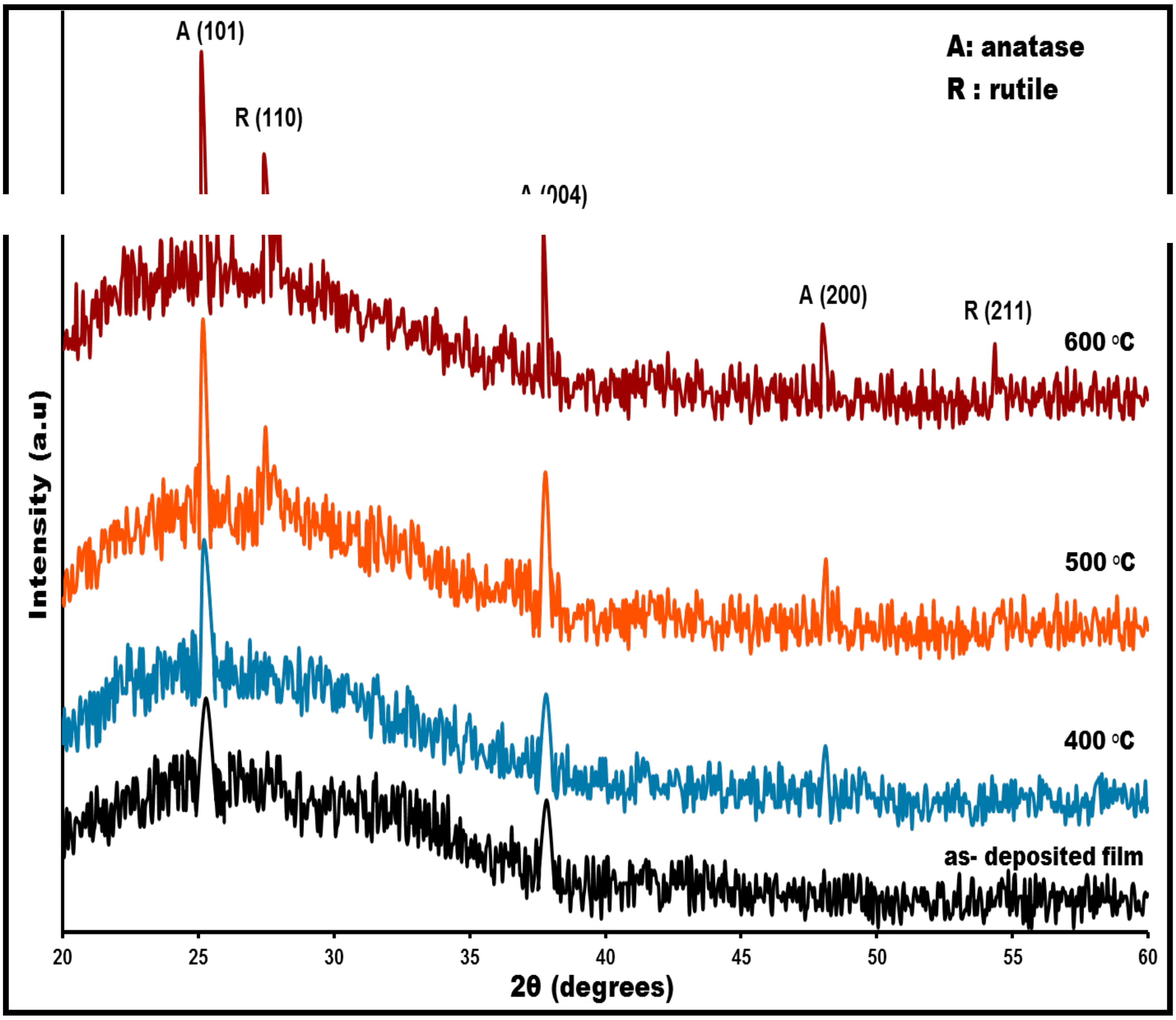

Fig. (1): XRD patterns of TiO_2 films deposited at 300 °C temperature and annealed at 400 °C, 500 °C and 600 °C

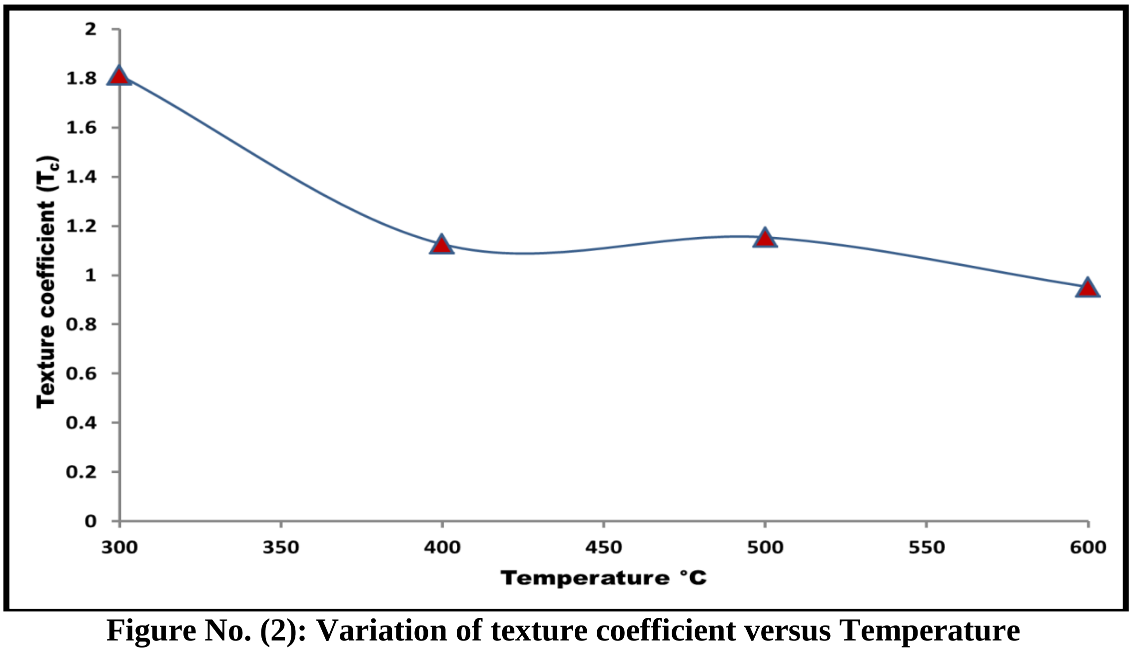

Figure No. (2): Variation of texture coefficient versus Temperature

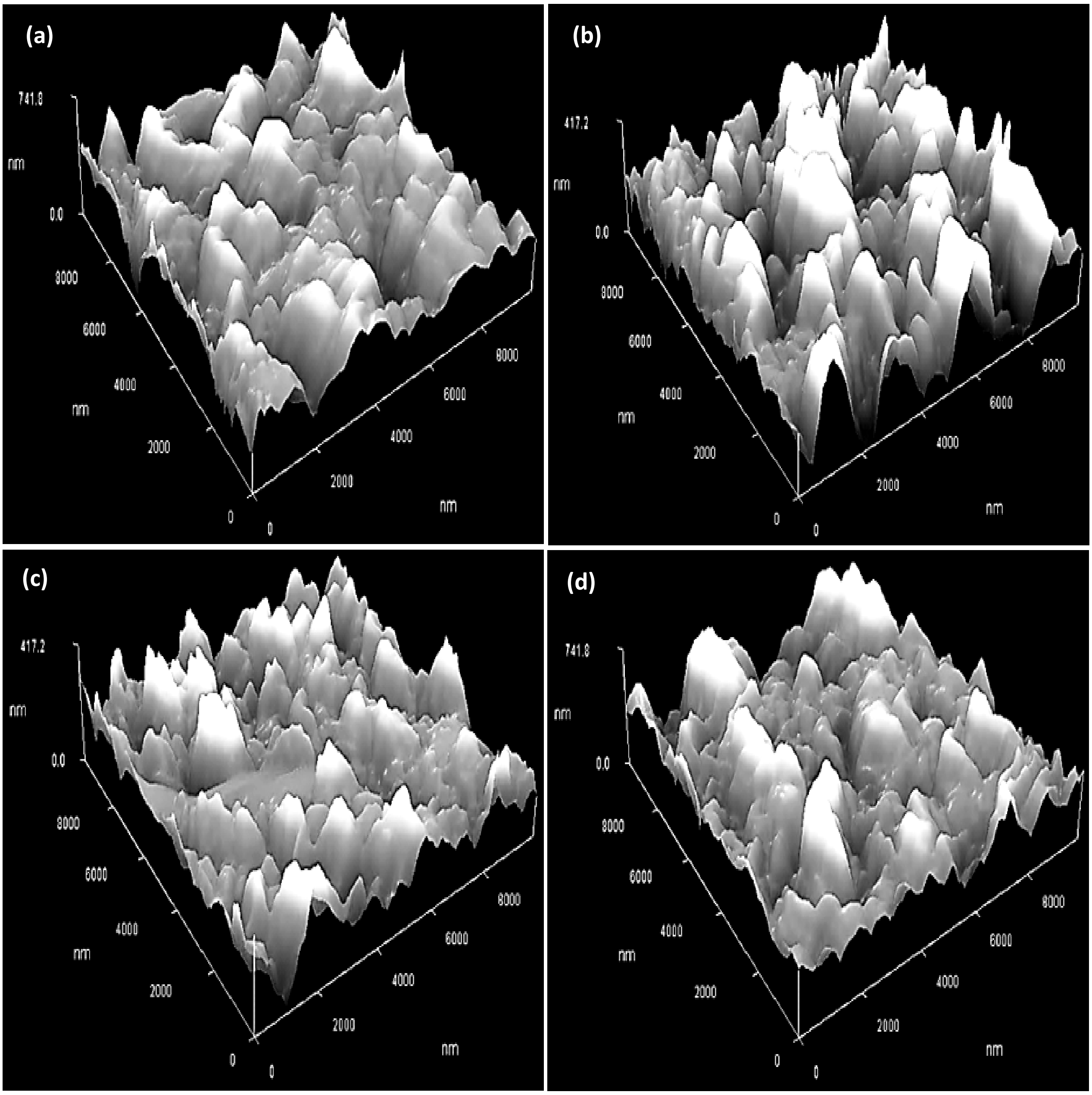

Figure No .(3): AFM images of TiO_2 films deposited at 300 °C temperature and annealed at different temperatures: (a) As-deposited, (b) 400 °C, (c) 500 °C and (d) 600 °C.

تصنيع أغشية TiO_2 الرقيقة ذي التركيب النانوي بتقنية ترسيب الليزر النبضي (PLD) وتأثير درجة حرارة التلدين في الخصائص التركيبية وطبوغرافية السطح

سرمد صبيح العبيدي
علي أحمد يوسف
قسم الفيزياء/ كلية التربية/ الجامعة المستنصرية

استلم البحث في :9تشرين الاول 2012، قبل البحث في: 9 كانون الاول 2012

الخلاصة

في هذا البحث، تم انماء أغشية اوكسيد التيتانيوم (TiO_2) النانوية بوساطة تقنية ترسيب الليزر النبضي (PLD) على قواعد زجاجية. ومن ثم لدنت أغشية TiO_2 الرقيقة من 400 الى 600 درجة مئوية في الهواء مدة ساعتين . ودرس تأثير التلدين في الخصائص التركيبية والطبوغرافية. عوامل عديدة لأنماء الأغشية اخذت بنظر الاعتبار لتحديد الحالة المثلى مثل درجة حرارة القاعدة (300 ºC) ،ضغط الأوكسجين (10^{-2} Torr) ،كثافة طاقة الفيض الليزري($0.4\ J/cm^2$) باستخدام التردد المضاعف لليزر النيديميوم- ياك الذي يعمل بتقنية عامل النوعية عند الطول الموجي 532nm بمعدل تكرارية 1 - (6 هرتز) وامد نبضة 10 نانوثانية. تُظهر نتائج فحوصات الأشعة السينية أن جميع التراكيب النانوية رباعية متعددة التبلور. وان هذه النتائج تظهر زيادة في حجم الحبيبات من 19.5 نانومتر الى 29.5 نانومترمع زيادة درجة حرارة التلدين. نتائج الأشعة السينية اظهرت ايضا ان الغشاء المرسب والملدن في 400 درجة مئوية لثنائي اوكسيد التيتانيوم ذي طور الأناتاس. اما الأغشية الملدنة عند 500 و 600 درجة مئوية فتمتلك خليطاً من طوري الأناتاس والروتيل. ان قيم عرض المنحني عند منتصف القمة لأغشية ثنائي اوكسيد التيتانيوم لأنماط (101) قد صغر من 0.450° الى 0.301° بزيادة درجة حرارة التلدين. ودرست طبوغرافية السطح للأغشية الرقيقة باستخدام مجهر القوى الذرية (AFM) الذي اثبت ان الاغشية المنمات بهذه الطريقة لها تبلور جيد وذو سطح متجانس. وان قيم مربع الجذر المتوسط RMS للأغشية الرقيقة وخشونة السطح تزداد مع زيادة درجة الحرارة التلدين.

الكلمات المفتاحية : ثنائي اوكسيد التيتانيوم، ترسيب الليزر النبضي، التركيبية ، طبوغرافية السطح، اغشية TiO_2.