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The Growth Characteristics of RF-Magnetron Sputtered Nanocrystalline TiO₂ Thin Films

Abstract- In this paper, RF Magnetron sputtered TiO₂ thin films deposited on glass slices at various powers (75,100,125 and 150) Watt for (1.5) hour and different thickness (62.5-88-118 and 132.6) nm, the TiO₂ thin films annealed with 400°C for 2 hour and the morphology and structure of these films are described by X-ray diffraction XRD and atomic force microscopy AFM to show the phase structure. X-ray diffraction investigation uncovered that the crystalline size of the TiO₂ thin films displays an expanding pattern with increasing the sputtering power. The preferred orientation of (101) was watched for the films deposited with sputtering power of (75,100,125 and 150) Watt.

Keywords- Titanium dioxide, Thin films, RF sputtering.

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1. Introduction

The production of TiO₂ thin films is one of major researches in photovoltaic area to limit the optimum conditions for sputtering TiO₂ thin film on different substrates. Many of methods have been used to prepare TiO₂ thin films as DC or RF sputtering, chemical vapor deposition and sol-gel process, etc [1].

TiO₂ can be found in three main crystalline phases Anatase (tetragonal), Rutile (tetragonal) and Brookite (orthorhombic). The first two phases have excellent photocatalytic and antibacterial properties [2]. Rutile TiO₂ has some good properties over Anatase phase like as higher dielectric constant, higher electric resistance, higher chemical stability, higher refractive constant[3].

High chemical and thermal stabilities, higher refractive index (Anatase=2.488, Rutile=2.609, Brookite=2.538), low absorbance and low dispersion in visible and near-infrared spectral area [4]. TiO₂ (Anatase) is an n-type semiconductor, thin films of TiO₂ utilized as optical coating, solar cell, integrate circuits, heat reflecting layer and waveguides have also been shown to be resistance to corrosive and mechanical attack and show stability over lengthy

time span duration.[5]. The application of these thin films include great important areas as renewable energy production represented by solar cell, smart window, gas sensors, photoconductive, hydrogen production, etc. [6].

The aim of this work is preparing the TiO₂ nanostructures thin films by using the RF magnetron sputtering and studying the effect of sputtering power in some physical properties which play a role in solar cells fabrication

2. Experimental

RF sputtering method utilized to product TiO₂ thin films on glass substrates with different working conditions as shown in table (1) where, P₁ is the base vacuum pressure and P₂ is the working argon pressure of vacuum sputtering chamber, T is the substrates temperature which was in the rang (55-65) due to the sputtering processes. The target type is TiO₂ of 99.99% which was powder and formed by pressing and sintering in 600°C in dimension (D=5cm,thickness of 3mm), and the distance between target and substrates holder is 5 cm. In this work TiO₂ thin films thickness is measured by using an optical method by using Phillips BLK-CXR-SR-25 Spectrometer.

Table 1: The working conditions of TiO₂ thin films preparing via RF sputtering method

POWER(Watt)	Thickness Th (nm)	P1(Torr) × 10 – 3	P2(Torr) ×10-3	T°(C)
75	62.5	4.23	7.45	55
100	88	4.13	7.9	60
125	118	5.5	7.12	58
150	132.6	4.15	7.15	65

The crystalline size, roughness and free mean square of thin film surface measured by atomic force microscopy (AFM ntegra NT-MDT). The crystalline structure can be determined for any thin film the X-Ray diffraction (XRD) which is Japanese type (Shimad Zu 7000), the crystalline size (D) was measured using Debye-Scherrer's formula(1):[7]

$$D = \frac{k\lambda}{\beta \cos\theta}$$

(1)

Where the symbol k is mean the shape factor which taken as 0.94, λ is the X-ray wavelength (1.5406Å°) for Cu-k_α, β is the full width at half-maximum (FWHM) and θ is the (Bragg's angle). [7].

3. Results and Discussion

Roughness is the most important parameter because an optimum roughness facilities the photons absorption, Figure 1 shows the two and three dimensions AFM images of the TiO₂ thin films at different sputtering power.

We can notice that the grain size of the deposited films was detected to be in the range (16-71)nm with increasing in sputtering power. Higher roughness was resulted due to the formation of deep channels and large bulging grains on the surface, which contributes to the smooth film s Figure 2 shown XRD pattern of TiO₂ thin films deposited on glass substrate by different power and annealing at 400°C for 2 hour. urface.

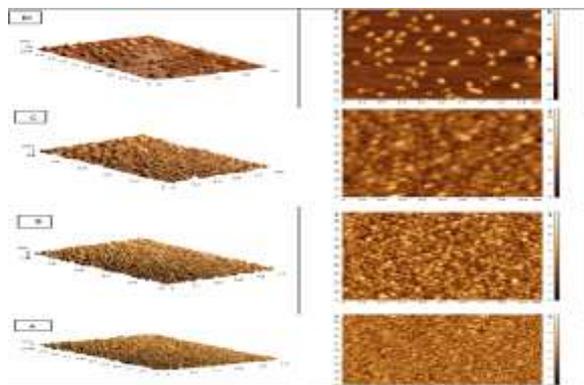


Figure 1: AFM images of TiO₂ thin film at (75, 100, 125 and 150) Watt with sputtering time=1.5hour

It can be observed that the average roughness increases with increasing power. Table (2) shows the values of average grain size, root mean square and roughness with different power, the grain size and surface roughness raise and morphology of the grains become crystalline.

Table 2: The values of grain size and roughness at different power

Power (Watt)	Average Roughness(nm)	Root mean square(nm)	Average grain size(nm)
75	0.643	0.816	16.4
100	1.044	1.434	19.5
125	1.081	1.469	28.5
150	4.105	6.297	71.5

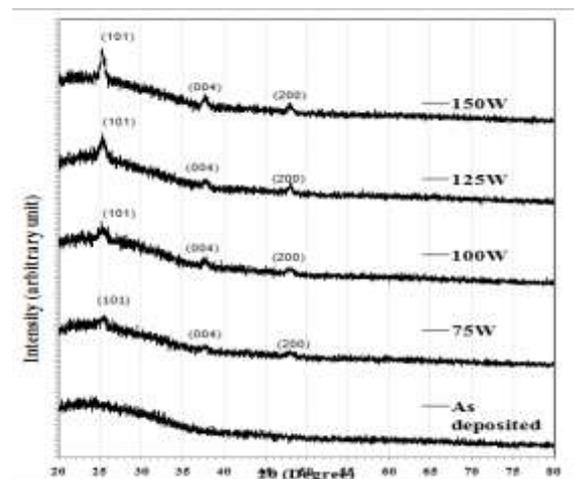


Figure 2: XRD pattern of TiO₂ thin film at (75, 100, 125 and 150)Watt with sputtering time=1.5hour

It is clear from Table 3 which displays results of comparison between annealed and non-annealed films that the TiO₂ film in as deposited case (non annealing) be amorphous structure. In addition, the as deposited specimens included the powers of 75,100,125 and 150 Watt have same pattern before annealing (as deposited),on the other hand, annealing TiO₂ films with 400°C for 2 hour having various values of crystalline size about (6.2 to16.4) nm, i.e the grain size increases with increasing the power and the thickness of these films. The 2θ at peaks 25.30,37.76 and 48.03 confirms the TiO₂ Anatase structure, that agree with [8].

Table 3: Characteristics of TiO₂ thin film at different power estimated from XRD data

Power (Watt)	Thickness (nm)	2θ (Deg.)	FWHM (Deg.)	d _{hkl} Exp.(Å)	G.S (nm)	hkl	Phase	d _{hkl} Std.(Å)																																																																																																								
As deposited	62.5	25.301	1.3200	3.5173	6.2	(101)	Anatase	3.5172																																																																																																								
		2							75	88	25.302	1.2390	3.5171	6.6	(101)	Anatase	3.5172	4	37.766	0.7438	2.3801	11.3	(004)	Anatase	2.3799	2	100	118	48.033	0.7344	1.8926	11.8	(200)	Anatase	1.8925	3	25.303	1.0090	3.5169	8.1	(101)	Anatase	3.5172	6	125	132.6	37.767	0.6322	2.3801	13.3	(004)	Anatase	2.3799	4	48.034	0.6242	1.8926	13.9	(200)	Anatase	1.8925	5	150	132.6	25.304	0.7910	3.5168	10.3	(101)	Anatase	3.5172	8	37.768	0.5374	2.3800	15.6	(004)	Anatase	2.3799	6	150	132.6	48.035	0.5306	1.8925	16.4	(200)	Anatase	1.8925	7																						
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4. Conclusion

The influence of sputtering power on the microstructural morphologies of TiO₂ thin films deposited by using RF-Magnetron sputtering technique were scanned in the present work. XRD studies showed that TiO₂ thin films deposited at (75,100,125 and 150) Watt sputtering power are amorphous. Subsequent annealing during 2h at 400°C. The TiO₂ films deposited on glass slices had a preferred crystal plane orientation in the (101) direction with various sputtering power, strong diffraction peaks indicating TiO₂ in Anatase phase that good agreement with [8]. AFM measurements of the films are effected with growing in the sputtering power due to the growing in the surfacediffusion.

5. References

- [1] P. Kajitvi Chyanukul, J. Ananpattarachai, and S. Pongpom, "Sol-gel preparation and properties study of TiO₂ thin films for photocatalytic reduction of chromium (VI) in photocatalysis process," *Science and Technology of Advanced Materials*, vol.6, no. 4, 352-358, 2005.
- [2] L. Bedlikyan, S. Zakhariyev, and M. Zakhariyeva, "Titanium dioxide thin film: preparation and optical properties," *Journal of Chemical Technology and Metallurgy*, vol. 48, no. 6, 555-558, 2013.
- [3] J. Adawiya Haider, N. Zainab Jameel, and Y. Samar Taha, "Synthesis and characterization of TiO₂ nanoparticales via sol-gel method by pulse laser

ablation," *Engineering and Technology Journal*, vol. 33, no. 5, 2015.

[4] R. Zallen, M.B. Moret, "The optical absorption edge of Brookite TiO₂," *solid State Communications*, vol. 173, no. 3, 2006.

[5] A. Enesca, Andronic, A. Duta, and S. Manolache, "Optical properties and chemical stability of WO₃ and TiO₂ thin film photocatalysts," *Romanian Journal of Information Science and Technology*, vol. 10, no. 3, 269-277, 2007.

[6] Z. Thaira Altayyar, Shubhra Gangopadhyay, "perovskite thin film preparation and energy band-gap determination for solar cell applications," *Engineering and Technology Journal*, vol.34, no. 14, 2016.

[7] A. Muhsin Kudhir, S. Raad Sabry, K. Yousif Al-Haidarie, "Effect of flow rate on characterizations of TiO₂ nano fibers using electro spinning method," *Engineering and Technology Journal*, vol. 33, no. 9, 2015.

[8] M. Ba-abbad muneer, H. Abdul Amir kadhun, A. Bakar mohamad, S. takriff mohd, k. sopian, "Synthesis and catalytic activity of TiO₂ nanoparticles for photochemical oxidation of concentrated chlorophenols under direct solar radiation," *International Journal Electrochemical Science*, Vol. 7, No. 3, 4871-4888, 2012.

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