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Physical properties of dc magnetron sputtered titanium oxide thin films

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ABSTRACT:
We investigated on compositional, structural, surface morphology and optical properties of titanium dioxide (TiO_2) thin films deposited on glass substrates at different substrate temperatures by dc magnetron sputtering technique. The crystallinity of films increases with increasing the substrate temperature from 303K to 698K. The films deposited at substrate temperature of 303K exhibited smooth surface compare to higher substrate temperature deposited films. The absorption edge of the films shifted towards visible light region as increasing the substrate temperatures from 303K to 698K. The contact angle of films decreasing with increasing the substrate temperature.

Keywords: Titanium dioxide, Thin films, Sputtering, Substrate temperature

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I. INTRODUCTION

Titanium dioxide (TiO_2) films have extensive applications in several fields like solar cells [1], heat mirrors [2], pigments [3], cosmetics[4], antifogging surfaces[5] and photocatalysis[6], due to its unique properties such as high refractive index [7], high relative dielectric [8], and strong mechanical, catalytic, hydrophilic/hydrophobic andoptical properties[6]. However, the properties of TiO_2 thin films are strongly dependent on the deposition technique. Various thin films deposition methods like sputtering [6], thermal oxidation [9], spin coating [10], chemical vapor deposition[11] and sol-gel [12] have been used. Among these techniques, dc magnetron sputtering is one of best technique due to its good adherent to substrate, no direct heating, uniformity, high deposition rates and easy to control the chemical composition of the film. In this investigation, TiO_2 films are deposited by dc magnetron sputtering technique and studied the effect of substrate temperature on the compositional, structural, microstructural, morphological and optical properties.

II. EXPERIMENTAL

TiO_2thin films deposited onglass substrates at different substrate temperatures ranging from 303 to 698K by using dc reactivemagnetron sputtering. A high purity (99.99%)Ti target with 3mm thick and 100mm dia was used as sputtering target. The distance between target to substrate was 65mm and the substrate rotation was fixed at10rpm. Before deposition, the vacuumchamber was evacuated to a base pressure of $5x10^{-4}$ Pa bythe combination of a rotary pump and diffusion pump.Sputtering was performed in pure argon(Ar)and oxygen (O_2) ambient through mass flow controllers. The target was pre-sputtered for 15min in Ar environment to removecontaminants on the target. The deposition pressure was 2.5 Pa. The chemical composition of the films was analyzed by energy dispersive spectroscopy (EDS). The films thickness was about 190 nm and was measured with step method. The structural analysis of deposited films was carried out by X-ray diffractometer (XRD). The microstructure and surface morphology of the films were observed using a scanning electron microscopy (SEM) and atomic force microscopy (AFM), respectively. Optical transmittance spectra in the UV-Visible range were measured bydouble-beam UV-Vis-NIR spectrophotometer.

III. RESULTS AND DISCUSSION

The chemical composition of the films at different substrate temperature was determined byEDS and are listed in Table 1. EDS results revealed that films consist Ti and O_2 only. The atomic percentage of Ti increased with increasing of substrate temperature, whereas, atomic percentage of oxygen decreased. EDS spectra of TiO_2films at different substrate temperaturesis shown in Fig.1.

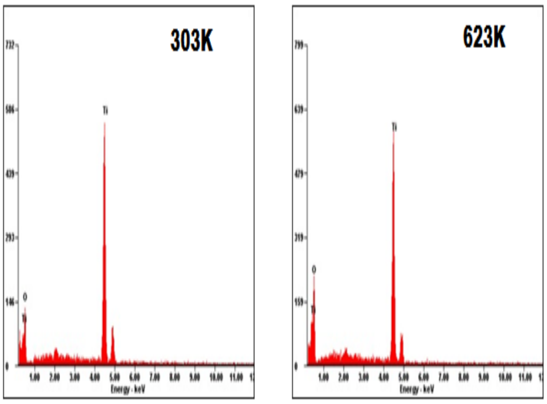

Fig.1.EDS spectra of TiO_2 films at different substrate temperatures.

Table 1. Elemental composition of TiO_2 films at different substrate temperatures.

Substrate temperature	Elemental composition	
	Ti (at%)	O_2 (at%)
303K	23.61	76.39
623K	27.53	72.47
698K	30.40	69.60

Structural properties

The XRD patterns of TiO_2 films deposited at different substrate temperatures are shown in Fig.2. Indeed, the films deposited at room temperature (303K) show an amorphous structure. However, the films deposited at substrate temperature of 623K exhibited a broad and weak peak related to (101) of anatase phase, indicating the beginning of crystallinity of the films. On further increasing the substrate temperature to 698K the peak intensity increased slightly. The films deposited at low substrate temperature sputtered atoms having low surface mobility and it prevents the crystallization. As increasing the substrate temperature, the sputtered atoms acquire enough mobility to achieve better crystallization. In general, the crystallinity of TiO_2 films started at higher temperatures when it deposited on the amorphous substrates, and the growth of anatase phase dominant than rutile phase at low temperatures due to surface Gibbs free energy of anatasepahse is lower than that of rutile phase, consequently, TiO_2 initially prefers to nucleate into anatase phase rather than rutile[13]. The increasing of the crystallinity of TiO_2 films with temperature was observed by Dussanet al.[14] andPjevic et al.[15].

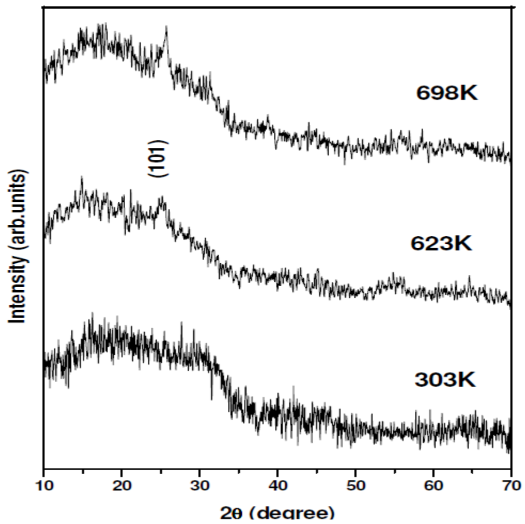

Fig.2. XRD patterns of TiO_2 films at different substrate temperatures.

Microstructure and surface morphology properties

Fig.3. shows the SEM images of TiO_2 films at different substrate temperatures. The films deposited at substrate temperature of 303K shows smooth and dense structure without cracks and voids, whereas the films deposited at substrate temperature of 623K exhibited small grains and the size of grains increased on further increasing the substrate temperature to 698K. The AFM images of TiO_2 films at different substrate temperatures are shown in Fig.4. The surface morphology of films was effected by the substrate temperature. As increasing the substrate temperature from 303 to 698K the grain size increases and grain boundaries reduced and become sharp. The TiO_2 films deposited at substrate temperature of 303 and 623K exhibited good homogeneity.The obtained RMS roughness values are 3.9, 1.9 and 1.5nm for substrate temperature of 303, 623 and 698K, respectively. However, RMS roughness increased with increasing of annealing temperature was observed in rf magnetron sputtered TiO_2 films by Pradhan et al.[16].

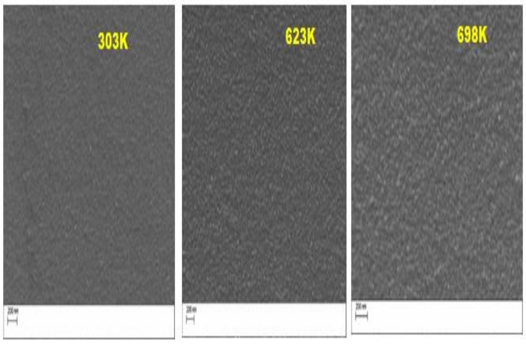

Fig.3. SEM images of TiO_2 films at different substrate temperatures.

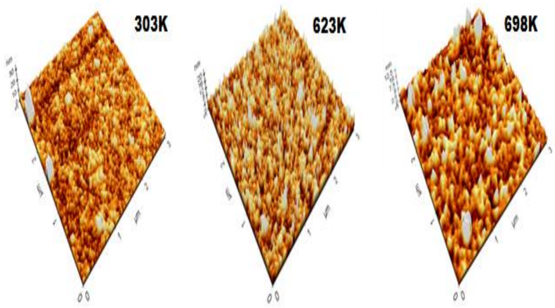

Fig.4. AFM images of TiO_2 films at different substrate temperatures.

Raman studies

Fig.5. shows the Raman spectra of TiO_2 films at different substrate temperatures. The films deposited at 303K, there is no peak in the spectra and it implies that films are amorphous [17]. By increasing the substrate temperature to 623K, two broad peaks are appeared at 403 and 642 $cm^{-1}$are from TiO_2anatase phase [18-19]. On further increasing the substrate temperature to 698K, the peaks intensity increased and broadness was decreased slightly. The rutile phase is not appeared in the present study due to as the rutile phase develops at higher temperature i.e beyond 1173K [20]. The increases of the peak intensity with substrate temperature is due to increase in crystallinity of the anatase phase [16].

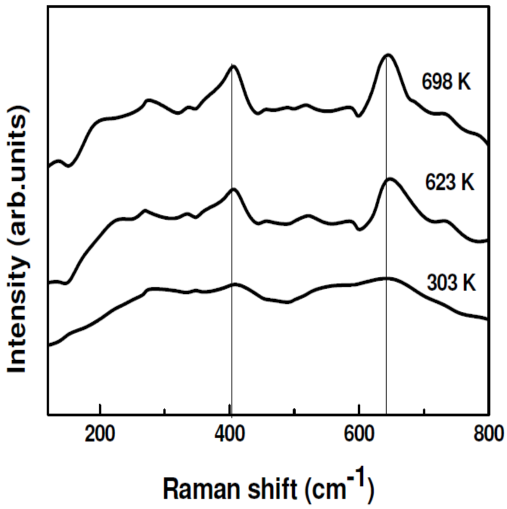

Fig.5. Raman spectra of TiO_2 films at different substrate temperatures.

Optical properties

The absorption spectra of the TiO_2 films at different substrate temperatures are shown in Fig.6. The absorption edge of the films shifted towards the visible light region as increasing the substrate temperatures from 303 to 698K, which is due to increasing of the oxygen vacancies.The optical band gap (E_g) of the films was determined from the extrapolation of the linear portion of the plots of $(\alpha h\nu)^{1/2}$versus ($h\nu$) (α is the absorption coefficient, $h\nu$ is the photon energy). The $(\alpha h\nu)^{1/2}$ versus ($h\nu$) plot for TiO_2 films at different substrate temperatures is presented in Fig.7. It is found that the band gap values of the films decreased with increasing substrate temperature. The obtained band gap values of TiO_2 thin films at different substrate temperatures are 3.33, 3.26 and 3.17eV for 303, 623 and 698K, respectively. The decreasing of band gap values with increases of substrate temperature was due to reduction of structural defects and enhancement of crystallinity of the films.

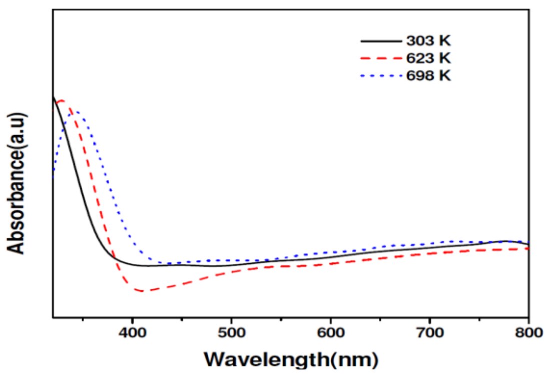

Fig.6. Absorption spectra of the TiO_2 films at different substrate temperatures.

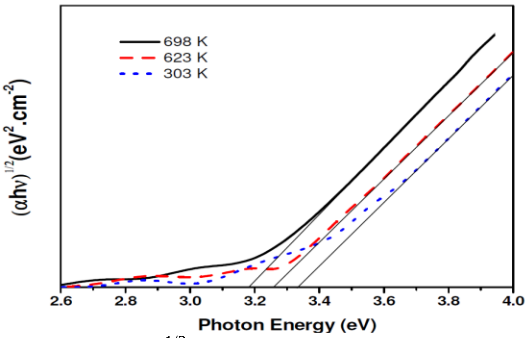

Fig.7. $(\alpha h\nu)^{1/2}$ versus $(h\nu)$ for TiO_2 films at different substrate temperatures.

Fig.8. shows the water contact angle(CA)on TiO_2 films surfaceat different substrate temperatures. The CA of films decreases with increasing the substrate temperature from 303 to 698K. This behavior suggests that films surface changed from hydrophobic to hydrophilic nature. The films show the high CA at low substrate temperature deposited films was due to low surface energy. The similar behavior was observed by Pradhan et al.[16] in rf magnetron sputtered TiO_2 films.

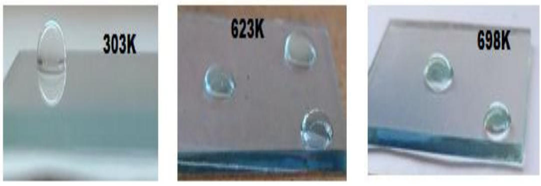

Fig.8. Water droplet on TiO_2 film surface at different substrate temperatures.

IV. CONCLUSIONS

TiO_2 thin films were deposited onto glass substrates by dc magnetronsputtering at varioussubstrate temperatures. The as deposited films at substrate temperature of 303K are amorphous nature and the crystallinity of the films started at higher substrate temperatures. The grain size and RMS roughness of the films increased with increasing the substrate temperature. The optical band gap of films decreases with increasing the substrate temperature. The contact angle of films decreasing with increasing the substrate temperature from 303 to 698K.

REFERENCES:

[1]. Y. Saito, S. Kambe, T. Kitamura, Y. Wada, S. Yanagida, Sol. Energy Mater. Sol. Cells 83 (2004)1-13.
[2]. M. Okada, M. Tazawa P. Jin, Y. Yamada, K. Yoshimura, Vacuum80 (2006) 732-5.
[3]. R. Bardet, MN. Belgacem, J. Bras, Cellulose20 (2013) 3025-37.
[4]. P.Tucci, G.Porta, M.Agostini, D. dinsadle, K. Cain, A.F. Agro, G. Melino, A. Wills, Cell Death Dis4 (2013) e549.
[5]. C.Euvananont, C. Junin, K. Inpor,P. Limthongkul, C. Thanachayanont, Ceram Int. 34 (2008) 1067-71.
[6]. A.R.G.Korpi, S.Rezaee, C. Luna, S. Ta˘lu, A. Arman, A. Ahmadpourian, Results in Physics 7 (2017) 3349-3352.
[7]. E. T. Fitzgibbons, K. J. Sladek, W. H. Hartwig, J. Electrochem. Soc. 119(1972)735-739.
[8]. G. V. Samsonov, The Oxide Handbook (IFI/Plenum, New York, 1982).
[9]. A.T. Zajac, Materials Science in Semiconductor Processing 83 (2018) 159-170.
[10]. C.-P. Lin, H. Chen, A. Nakaruk, P. Koshy, C.C. Sorrell, Energy Procedia 34 (2013) 627- 636.
[11]. H.H. Nguyena, D-J.Kima, D-W.Parkb, K-S.Kima,J. Energy Chemistry 22(2013)375-381.
[12]. C.M. Malengreaux, G.M.-L. Leonard, S.L. Pirard, I. Cimieri,S.D. Lambert, J.R. Bartlett, B.Heinrichs, Chemical Engineering Journal 243 (2014) 537-548.
[13]. K. Ding, Z. Miao, B. Hu, G. An, Z. Sun, B. Han, Z. Liu, Langmuir 26 (2010) 10294-10302.
[14]. A. Dussan, A. Bohórquez, Heiddy P. Quiroz, Applied Surface Science 424 (2017) 111-114.
[15]. D. Pjević, T. Marinković, J. Savić, N. Bundaleski, M. Obradović, M. Milosavljević, M. Kulik, Thin Solid Films 91 (2015) 224-229.
[16]. S.S. Pradhan, S.Sahoo, S.K. Pradhan, Thin Solid Films 518 (2010) 6904-6908.
[17]. N.R. Mathews, E.R. Morales, M.A.C. Jacome, J.A.T. Antonio, Sol. Energy 83 (2009) 1499.
[18]. J. Zhang, M. Li, Z. Feng, J. Chen, C. Li, J. Phys. Chem. B 110 (2006) 927.
[19]. B. Karunagaran, K. Kim, D. Mangalaraj, J. Yi, S. Velumani, Sol. Energy Matter. Sol.Cells 88 (2005) 199.
[20]. S.W. Ryu, E.J. Kim, S.K. Ko, S.H. Hahn, Mater. Lett. 58 (2004) 582.

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