

Study the Influence of Different Annealing Temperature on the Nanostructures of TiO₂ Thin Films

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Abstract: Ti films of the same thicknesses and near normal deposition angle and with the same deposition rate were deposited on glass substrates, at room temperature, under UHV conditions. Different annealing temperatures as 410 °K, 510 °K and 610 °K with uniform 8 cm³/sec oxygen flow, were used to produce titanium oxide layers. Their nanostructures were determined by AFM and XRD methods. Roughness of the films changed due to annealing process. The gettering property of Ti and annealing temperature can play an important role on the nanostructure of the films.

Key words: Titanium Dioxide • Thin Films • AFM • XRD

INTRODUCTION

Thin metal oxide films over metal and semiconductor surfaces are probably the most widely studied structures in basic physics and technology. Applications, involving low-loss, low-scatter optical coatings for visible and near infrared optics [1-4] and for electrical devices [5,6], have stimulated a considerable amount of activity in fabrication of dielectric films with high refractive index and low absorption. TiO₂ attracts the interest of the scientific community due to its special properties such as high chemical stability, mechanical resistance and high optical transmittance in the visible-IR spectral range [7]. The applications of TiO₂ thin films include gas and humidity sensors, solar energy converters or reflecting and protective coatings on optical elements, structural ceramics and biocompatible materials [8, 9]. On the other hand, the high k property of TiO₂, as well as magneto-doping processes were investigated for applications as gate dielectric material and electron spin based nano devices [10,11].

TiO₂ has three main crystal phases: anatase, rutile and brookite [12, 13]. Among these phases, anatase phase, which is a meta-stable phase, is also chemically and optically active and suitable for photo-catalyst [14].

In this work we want to study the influence of annealing temperature and oxygen flow on nanostructure and roughness of produced layers and also crystallographic directions and their dependence to mentioned parameters.

Experimental Details: Titanium films of 65.7 nm thickness were deposited on glass substrates at room temperature. The residual gas was composed mainly of H₂, H₂O, CO and CO₂ as detected by the quad ro pole mass spectrometer. The substrate normal was at 10 degree to the direction of evaporated beam and the distance between evaporation crucible and substrate was 45 cm.

Just before use all glass substrates were ultrasonically cleaned in heated acetone, then ethanol. Other deposition conditions were same during coating. Vacuum pressure was about 10⁻⁶ torr and deposition rate was 0.9 Å/sec. Thickness of layers were determined by quartz crystal technique. Annealing oven with different annealing temperatures (410 °K, 510 °K, 610 °K) and uniform oxygen flow was used, to change nanostructure of layers and produce titanium dioxide layers. The nanostructure of these films was obtained using a Philips XRD X'pert MPD Diffractometer (CuK_α radiation) with a step size of 0.03 and count time of 1s per step, while the surface physical morphology and roughness were obtained by means of AFM (Dual Scope™ DS 95-200/50) analysis.

RESULTS AND DISCUSSION

Figure1(a-d), shows the morphology of produced layers (AFM).Figure1(a), shows the AFM image of as deposited 65.7 nm thickness Ti film at room temperature. As it can be seen, the surface is full of domed grains.

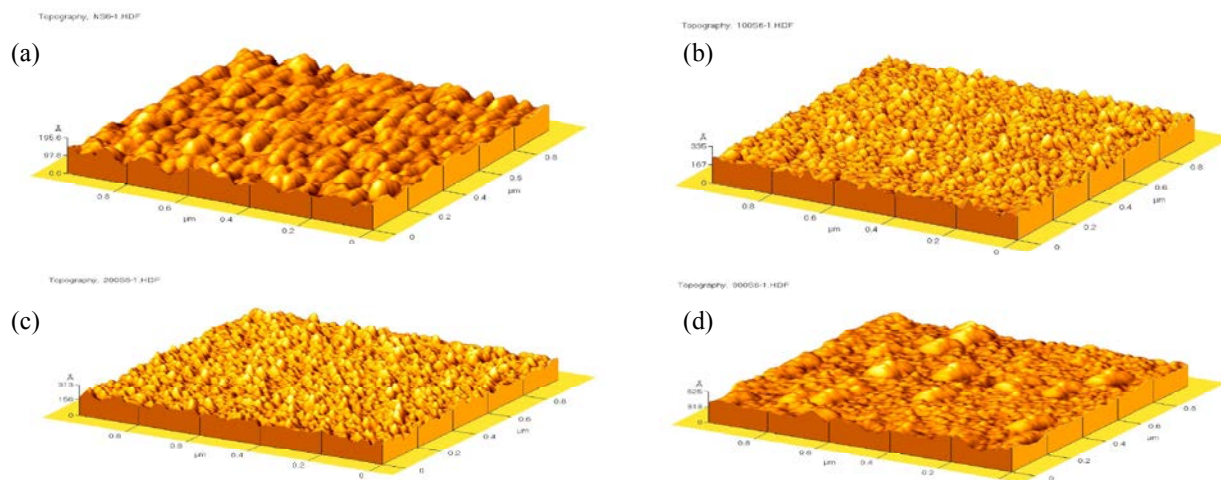


Fig. 1: The AFM images of, as deposited Ti (a); at 410 °K(b); at 510 °K(c) and 610 °K (d), annealing temperatures and in presence of 8 cm³/sec oxygen flow.

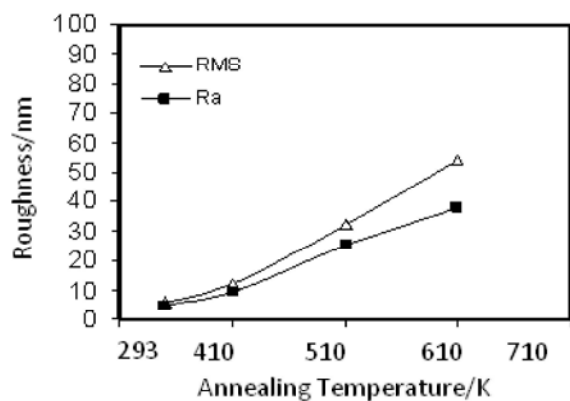


Fig. 2: The roughness diagram of, as deposited Ti (a); at 410 °K(b); at 510 °K(c) and 610 °K (d), annealing temperatures and in presence of 8 cm³/sec oxygen flow.

By increasing annealing temperature to 410 °K and in presence of uniform oxygen flow(8 Cm³/sec), oxygen penetrates to grains structures and brake them down to tiny needle-like grains (Figure1(b)). In Figure 1(c), annealing temperature increases to 510 °K and as it can be seen, oxygen penetrates more and more to grains structures and brake them down to tinier grains and on the other hand by increasing annealing temperature, surface diffusion happens, so roughness increases in this sample. Figure1(d), shows the produced layer at 610 °K annealing temperature in presence of uniform oxygen flow. As it can be seen, because of surface diffusions at high temperature, there are big domed grains and small domed grains between them. It seems there are two different kind of nanostructures in this layer.

Figure 2, shows the diagram of roughness for the layers produced in this work. By increasing annealing temperature, roughness is also increasing and this is because of penetrating oxygen to grain structures and migration of grains at higher temperatures.

As we know Ti is a getter metal and in presence of oxygen and heat will convert to titanium dioxide. Figure 3(a-d), shows the XRD images of TiO₂ /glass layers produced in this work.

As it can be seen in Figure 3(a), there is an anatase A(103) phase with (103) crystallographic direction. This is because of gettering property of Titanium. Figure 3(b) shows XRD pattern of Titanium dioxide on glass substrate at 410 °K annealing temperature and (8 cm³/sec) oxygen flow. As it can be seen there is a wide peak at A(103) and A(004) crystallographic directions. By increasing annealing temperature to 510 °K at the same uniform oxygen flow, the same peaks are presented on layer (Fig 3 (c)). In Figure 3(d), XRD pattern of TiO₂ /glass layer at 610 °K and in presence of 8 cm³/sec oxygen flow, shows A(103) crystallographic direction and it seems to begin phase transition to rutile phase R(211) crystallographic direction. So by increasing annealing temperature we can see more peaks and phase transition from anatase to rutile phase.

Summery: The influence of annealing temperature and uniform oxygen flow on titanium layers of the same thicknesses were obtained. This was accomplished by studying the relationship between AFM and XRD results. The morphology of layers changed by increasing heat and in presence of oxygen flow. By increasing annealing

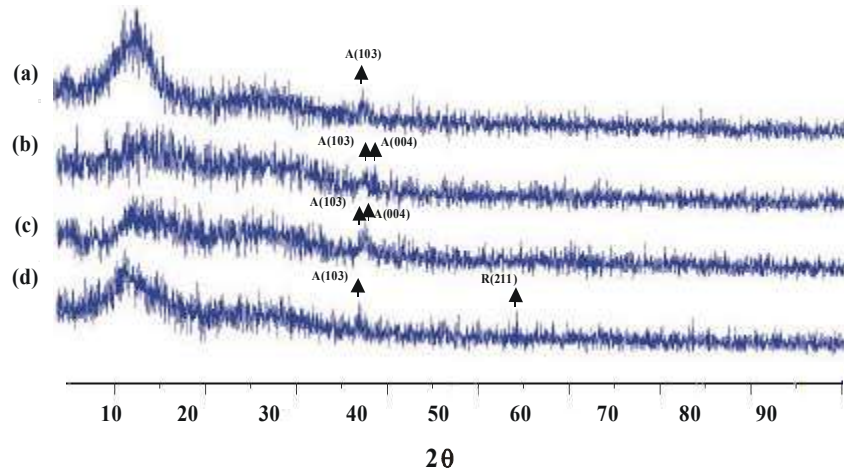


Fig. 3: The XRD patterns of as deposited Ti (a); at 410 °K(b); at 510 °K(c) and 610 °K (d), annealing temperatures and in presence of 8 cm³/sec oxygen flow.

temperature and in presence of oxygen flow, at the first step, oxygen penetrated to grains and broke them down to needle-like grains. By increasing heat, penetration of oxygen continue and because of surface diffusion roughness changed. There were still needle-like grains on the surface. By increasing annealing temperature to 610 °K, two different kinds of nanostructures configured (small domed grains between bigger domed grains). Also by increasing annealing temperature roughness of layers increased. Because of gettering property of Ti and presence of oxygen flow, titanium dioxide layers produced. XRD patterns showed anatase structure at A(103) and A(004) crystallographic directions by increasing temperature to 410 °K and 510 °K. At higher temperature (610 °K) two different A(103) and R(211) crystallographic directions appeared and phase transition happened.

REFERENCES

1. Vorotilov, K.A., E.V. Orlova and V.I. Petrovsky, 1992. *Thin Solid Films*, 207: 180.
2. Krishana, M.G., K. Narasimha Rao and S. Mohan, 1983. *J. Appl. Phys.*, 73: 434.
3. Rancourt, J., 1987. *User's Handbook; Optical Thin Film*. McGraw-Hill. New York.
4. Dobrowski, J.A., 1987. in: W Driscoll (Ed.). *Coatings and Filters in Handbook of Optics*. McGraw-Hill, New York.
5. Babelon, P., A.S. Dequiedt, H. Mostesa-Sba, S. Bourgeois, P. Sibillot and M. Sacilotti, 1998. *Thin Solid Films*, 322: 63.
6. Leprince-Wang, Y., K.Y. Zhang, V. Nguyen And Souche, J. Rivory, 1997. *Thin Solid Films*, 307: 38.
7. Kangarlou, H. And Saeid Rafizadeh, 2011. *World Applied Sciences Journal*, 13: 46.
8. Lobl, P., M. Huppertz and D. Mergel, 1994. *Thin Solid Films* 251: 72.
9. Da Cruz, N.C., E.C. Rangel, B.C.J.J. Wang, B.C. Trasferetti, C.U. Davanzo, S.G.C. Castro and M.A.B De Moraes, 2000. *Surf. Coat. Technol.*, 126: 123.
10. Jang, H.K., S.W. Whangbo, H.B. Kim, K.Y. Im, Y.S. Lee, L.W. Lyo, C.N. Whang, C.H. Wang, G. Kim, H.S. Lee and J.M. Lee, 2000. *J. Vac. Sci. Technol. A.*, 18: 917.
11. Tang, Q., K. Kikuchi, S. Ogura and A. Macleod, 1999. *J. Vac. Sci. Technol. A.*, 17: 3379.
12. Rinner, M., J. Gerlach and W. Ensinger, 2000. *Surf. Coat. Technol.*, 132: 111.
13. Ohwaki, T. And Y. Taga, 1989. *Appl. Phys. Lett.*, 54: 1664.
14. Meng, L. And M.P. Dos Santos, 1993. *Thin Solid Films*, 223: 22.