

Study the Influence of Thermal Oxidation on the Structures of Titanium Very Thin Layers

Saeid Rafizadeh, Haleh Kangarlou

Abstract— Ti films with the same thickness, deposition angle (near normal) and deposition rate were deposited on glass substrates at room temperature under UHV conditions. Different annealing temperatures 120, 270 and 320 degree Celsius with $7 \text{ cm}^3/\text{sec}$ uniform oxygen flow, were used for producing titanium oxide layers. Thin film structures were studied by AFM and XRD methods. During annealing processes roughness of the films changed due to the formation of titanium oxide. Titanium oxide formation at various annealing temperature can play an important role on the structure and property of the films.

Index Terms— Titanium, Titanium dioxide, Thin Films, AFM, XRD

I. 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. TiO_2 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. The applications of TiO_2 thin films include gas and humidity sensors, solar energy converters or reflecting and protective coatings on optical elements, structural ceramics and biocompatible materials [7, 8]. On the other hand, the high k property of TiO_2 as well as magneto- doping processes were investigated for applications as gate dielectric material and electron spin based nano devices [9,10].

TiO_2 has three main crystal phases: anatase, rutile and brookite [11,12]. Among these phases, anatase phase, which is a meta-stable phase, is also chemically and optically active and suitable for photo- catalyst [13].

In this work the influence of annealing temperature and oxygen flow on the structure and roughness of produced layers and also crystallographic directions and their dependence to mentioned parameters has been studied.

II. METHOD

Titanium films of 20 nm thickness were deposited on glass substrates at room temperature. The residual gas was composed mainly of H_2 , H_2O , CO and CO_2 as detected by the quad ro pole mass spectrometer. The substrate normal was at 8.5 degree to the direction of the evaporated beam and the distance between the evaporation crucible and substrate was 40 cm. Just before use all glass substrates were ultrasonically cleaned in heated acetone, then in ethanol. Other deposition conditions were the same during coating. Vacuum pressure was about 10^{-6} torr and deposition rate was $2 \text{ \AA}/\text{sec}$. Thickness of the layers were determined by quartz crystal microbalance. The Ti thin films were heated at temperatures (120, 270, 320 Celsius degree) under uniform oxygen flow $7 \text{ cm}^3/\text{sec}$ for about 2 hours to change the structure of layers and to produce titanium dioxide layers. The structure of these films was studied 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 was obtained by means of AFM (Dual Scope™ DS 95-200/50) analysis.

III. RESULT AND DISCUSSION

Figure 1(a-d) shows the morphology of the produced layers (AFM). Figure 1(a) shows the AFM image of as deposited Ti film at room temperature with 20 nm thickness. The surface is smooth and the shape of uniform glass substrate in this layer, can be seen. By annealing at 120 °C in presence of uniform oxygen flow($7 \text{ Cm}^3/\text{sec}$) oxygen will penetrate into the structure and titanium oxide starts to form as tiny needle like grains (Figure 1(b)). In Figure 1(c), at the annealing temperature of 270°C, it can be seen that the titanium oxide grain sizes has increased. This is because of high surface diffusion and coalescence of the grains to decrease the surface area. Figure 1(d) shows the produced layer with 320°C annealing temperature. It can be seen that, because of surface diffusion and bulk diffusions in high temperature and coalescence of the grains, titanium oxide has further increase at the size of grains.

Saeid Rafizadeh is with Department of Electronic, I.A.U Urmia Branch (phone: 0098-441-3440680; fax: 0098-441-3440680; e-mail: saeidrafizadeh@gmail.com).

Haleh Kangarlou is with the Department of Physics, I.A.U Urmia Branch (e-mail: h.kangarlou@iaurmia.ac.ir).

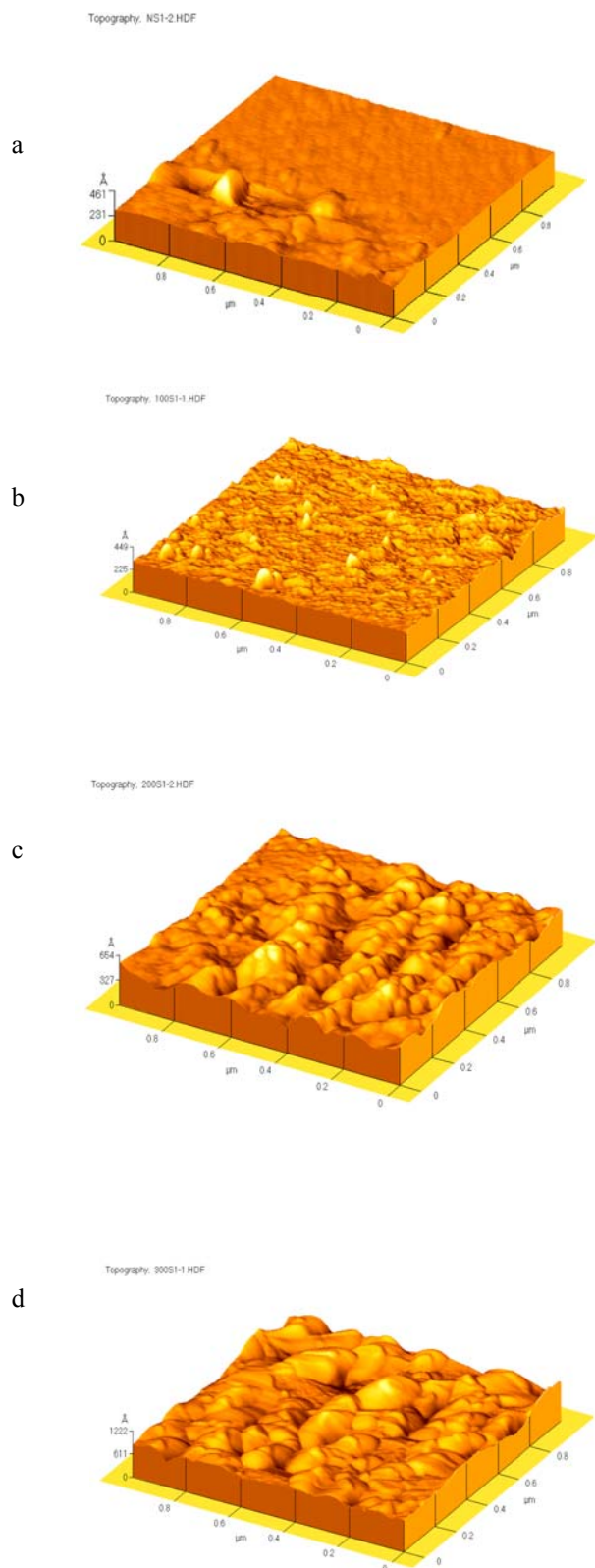


Fig.1. The AFM images of (a) deposited Ti, with annealing at (b) 120 °C ; (c) 270 °C; (d) 320°C with oxygen flow.

Figure 2 shows the roughness of layers versus annealing temperature. By increasing annealing temperature roughness also increases due to penetration of oxygen into the titanium thin film and subsequent titanium oxide formation. So by

increasing annealing temperature (from 120 °C to 320 °C), roughness will increase from 10 nm to 20 nm which is in agreement with AFM images. Ti has a high solubility for oxygen and also has a high reactivity with oxygen to titanium dioxide.

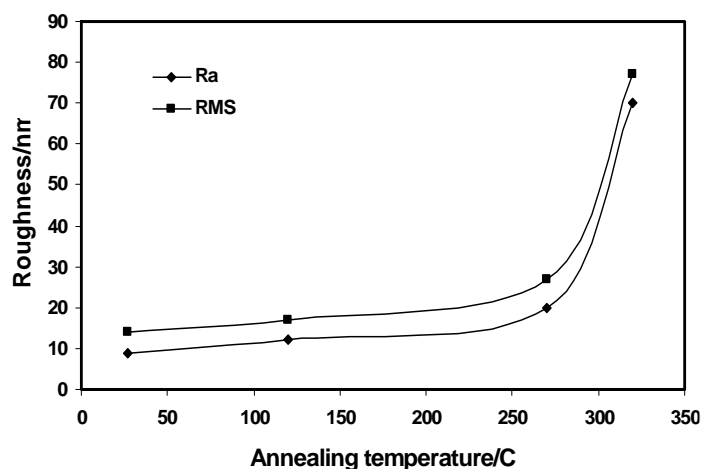


Fig.2. The roughness diagram of deposited Ti, with annealing at 120 °C, 270 °C and 320 °C and oxygen flow.

Figure 3 (a-d), shows XRD pattern of the layers in this work. As it can be seen all layers are amorphous, and there is no sharp peaks for layers in this work. This is because of two reasons, the first one is low annealing temperature as the layers need higher temperature for crystallization and second, the low thickness (20 nm) of the layers.

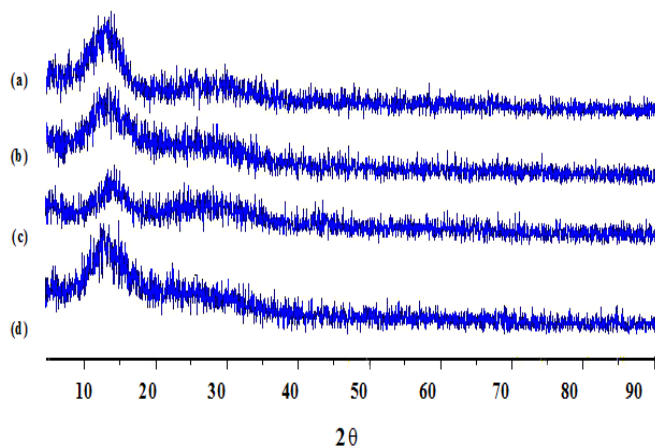


Fig. 3. The XRD patterns of (a) deposited Ti; with annealing at (b) 120 °C ; (c) 270°C; (d) 320°C and oxygen flow.

IV. CONCLUSION

The influence of annealing temperature and uniform oxygen flow on titanium layers with the same thickness was studied using AFM and XRD techniques. The morphology of the layers changes by increasing annealing temperature in the presence of oxygen flow. By increasing annealing temperature in the presence of oxygen flow, at the first step oxygen penetrate into the layer and then titanium oxide

forms as needle like grains. By increasing annealing temperature, because of surface and bulk diffusion and coalescence of grains, the titanium oxide grain size increases. Roughness increases by increasing annealing temperature according to AFM results. XRD patterns of all titanium dioxide layers produced at various temperatures with the same thickness (20 nm) are amorphous because of low temperature and low thickness.

REFERENCES

- [1] K.A. Vorotilov, E.V. Orlova, V.I. Petrovsky. *Thin Solid Films* 207 (1992) 180.
- [2] M.G. Krishana, K. Narasimha Rao. S. Mohan. *J. Appl. Phys.* 73 (1983) 434.
- [3] J. Rancourt, *User's Handbook; Optical Thin Film*. McGraw-Hill. New York, 1987.
- [4] J.A. Dobrowski, in: W Driscoll (Ed.). *Coatings and Filters in Handbook of Optics*. McGraw-Hill, New York, 1987.
- [5] P. Babelon, A S. Dequiedt, H Mostesa-Sba, S. Bourgeois, P. Sibillot. M Sacilotti. *Thin Solid Films* 322 (1998) 63.
- [6] Y. Leprince-Wang. K.-Y. Zhang. V. Nguyen, V. An, D. Souche, J. Rivory, *Thin Solid Films* 307 (1997) 38.
- [7] P. Lobl, M. Huppertz, D. Mergel, *Thin Solid Films* 251 (1994) 72.
- [8] N.C. Da Cruz, E.C. Rangel. B.C.J.J. Wang. B.C. Trasferetti, C.U. Davanzo, S.G.C. Castro, M.A.B De Moraes, *Surf. Coat. Technol.* 126 (2000) 123.
- [9] H.K. Jang, S.W. Whangbo. H B Kim. K.Y. Im, Y.S. Lee. I.W. Lyo. C.N. Whang, C.H. Wang, G. Kim. H.S. Lee, J.M. Lee, *J. Vac. Sci. Technol. A* 18 (2000) 917.
- [10] Q Tang, K. Kikuchi, S. Ogura. A. Macleod, *J. Vac. Sci. Technol. A* 17 (1999) 3379.
- [11] M. Rinner. J. Gerlach, W. Ensinger. *Surf. Coat. Technol* 132 (2000) 111.
- [12] T. Ohwaki, Y. Taga. *Appl. Phys. Lett.* 54 (1989) 1664.
- [13] L. Meng. M.P. Dos Santos. *Thin Solid Films* 223 (1993) 22.
- [14] T. Pawlewicz, P.M. Martin, D.D. Hays, I B. Mann, *The International Society for Optical Engineering. Optical Thin Films* 325 (1982) 105.
- [15] J.M. Bennet, E. Pelletier. G. Albrand, J.P. Borgogno. B. Lazarides. C.K. Carniglia, R.A. Schmell. T.H. Allen, T Tuttle-Hart, K.H. ^ Guenther. A. Saxer, *Appl. Opt.* 28 (1989).