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Structural Changes of UHV Deposited Titanium Thin Films in Presence of Oxygen Flow and Temperature

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Abstract

Titanium dioxide thin layers on glass substrates, were produced by annealing method, different annealing temperatures as, 393K, 493K and 593K were used. The initial layers were Ti/glass, with same thicknesses, near normal deposition angle and same deposition rate that produced in room temperature, an uniform oxygen flow with 6 cm^3/sec , were used for oxidation. The nano structure of the layers were determined by AFM and XRD methods. Roughness of the films changed due to annealing process. topography and crystallography of produced layers also changed during annealing processes.

Key Words: AFM, XRD, Crystallographic Direction, Titanium Dioxide

INTRODUCTION

The ultimate goal for the human beings living in 21st century is to establish environmentally harmonized technologies for sustainable society and energy systems. These technologies include the environmental protection the pollution control, 3R (reduce, reuse and recycle) of materials, the renewable and/or clean energy systems, and so on. Under the NITech 21st Century COE program on 'World Ceramics Centre for Environmental Harmony', we have conducted research to contribute to the designate. technologies through material science and applied physics.

Since the finding of the photo-induced decomposition of water on $Ti0_2$ electrodes by Fujishima and Hond [l] in 1971, TiO2 has become a promising material as photo catalyst[2] to decompose and oxidize various organic and or inorganic chemicals in waste and emission by its strong oxidation activity based on the created super oxide anion radical (0_2) and OH radical. Its high chemical stability and inexpensive cost are the other benefits for the use of this material as photo-catalyst. This material has additionally great potential for the application to dye-sensitized photovoltaic cells [3,4], energy efficient windows and/or other optical coatings [5-7], and capacitors in LSI [8]. The wide band gap semiconductor TiO_2 exists in three different crystalline polymorphs: rutile, anatase and brookite. Among them, rutile (R-titania) and anatase (A-titania) are the most common and widely used phases in applications.Motivated by those excellent applications of $Ti0_2$, an increasing number of, researchers have engaged in the fabrication of $Ti0_2$. We have recently achieved: (1) the simultaneous growth of epitaxial thin films of both A-titania on $SrTi0_2$ (STO) substrate and R-titania on sapphire substrates, and growth of polycrystalline A and/or R-titania thin films on silicon and/or glass, by helicon RF magnetron sputtering [9-11]; (2) the determination of optical properties of the fabricated polycrystalline and/or epitaxial A and/or R-titania thin films [12] and interpretation of the observed wider band-gap values [13]; (3) the evaluation of bactericidal ability by killing Escherichia coli [14]; and (4) the improvement of optical reactivity by N_2-H_2 plasma surface-treatment [15]. 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 .

MATERIALS AND METHODS

Titanium films of 73.3 nm thickness were deposited on glass substrates on 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 8 degree to the direction of the evaporated beam and the distance between the 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 3×10^{-6} torr and deposition rate was 0.7 A/sec. Thickness of the layers were determined by quartz crystal technique .we used annealing oven and different annealing temperatures (393K, 493K, 593K) and uniform oxygen flow 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 was obtained by means of AFM (Dual Scope ™ DS 95-200/50) analysis.

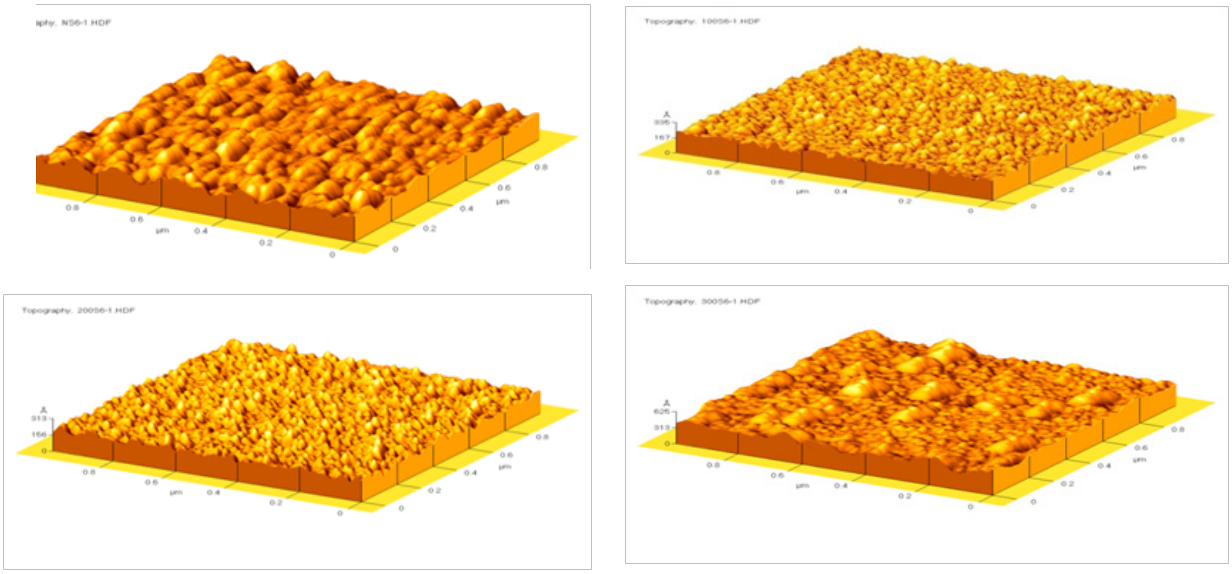

Fig1. AFM images (a) as deposited Ti;(b) 393K ; (c) 493K; and(d) 593K, annealing temperature and oxygen flow.

RESULTS

Figure1(a-d) show the morphology of the produced layers (AFM).Figure1(a) ,shows the AFM image of as deposited Ti film, in room temperature and with 73.3 nm thickness. As it can be seen , the surface is full of domed grains.

By increasing annealing temperature to 393K and in presence of uniform oxygen flow 6(Cm^3/sec), oxygen will penetrate to the grain structure and brake them down to tiny needle like grains (Figure1(b)). In Figure1(c) annealing temperature increases to 493K, and as it can be seen the oxygen penetrates more and more to grain structures and brake them down to tinier grains and in the other hand by increasing annealing temperature surface diffusion will happen, so roughness will increase in this sample. Figure1(d) shows the produced layer with 593K annealing temperature in presence of uniform oxygen flow, as it can be seen because of surface diffusions In high temperature there are big domed grains and small domed grains between them, it seems there are two different kind of nano structures in this layer.

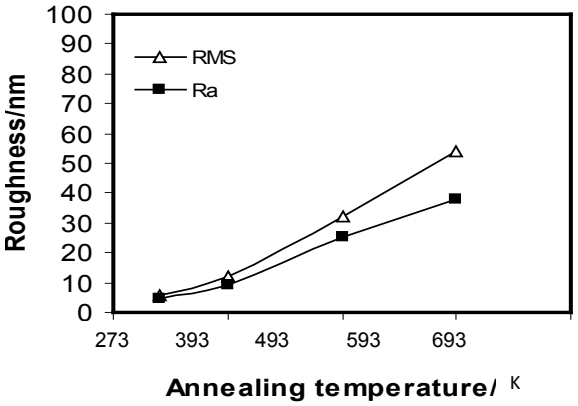

Fig2. The roughness diagram of , as deposited Ti, (b) 393K ;(c) 493K and (d) 593K, annealing temperature and oxygen flow.

Figure 2 shows the diagram of the roughness for layers 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 in higher temperatures.

As we know Ti is a getter metal and in presence of oxygen and of course heat will convert to titanium dioxide. Figure 3(a-d), shows XRD images for $Ti0_2$ /glass, layers 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 gettering property of Titanium, Figure3(b) shows XRD pattern of Titanium dioxide on glass substrate at 393K annealing temperature and 6(cm^3/sec) oxygen flow, as it can be seen there is a wide peak that relates to A(103), and A(004) crystallographic directions, by increasing annealing temperature to 493K in same uniform oxygen flow, same peaks are presented on layer (Fig 3 (c)), and in Figure 3 (d) XRD pattern for $Ti0_2$ /glass, in 593K and 6(cm^3/sec) oxygen flow, shows A(103) crystallographic direction and seems to begin phase transition, to rutile phase R(211) crystallographic direction. So by increasing annealing temperature we can see more peaks and also anatase phase will convert to rutile phase.

DISCUSSION

The influence of annealing temperature and uniform oxygen flow on titanium layers with same thickness are obtained. This is accomplished by the studying the relationship between AFM and XRD results. The morphology of the layers changes by increasing heat and in presence of oxygen. By increasing annealing temperature and in presence of oxygen flow, at the first step oxygen penetrates to grain structures and brake them down to needle like grains, by increasing heat, the oxygen penetration will continue and because of surface diffusion, roughness will change. There are still needle like grains on surface. By increasing annealing temperature to 593K, two different kinds of nano structures configure(small domed grains between bigger domed grains) and roughness of layers increases. Because of gettering property of Ti and presence of oxygen flow, titanium dioxide layers produce. XRD patterns

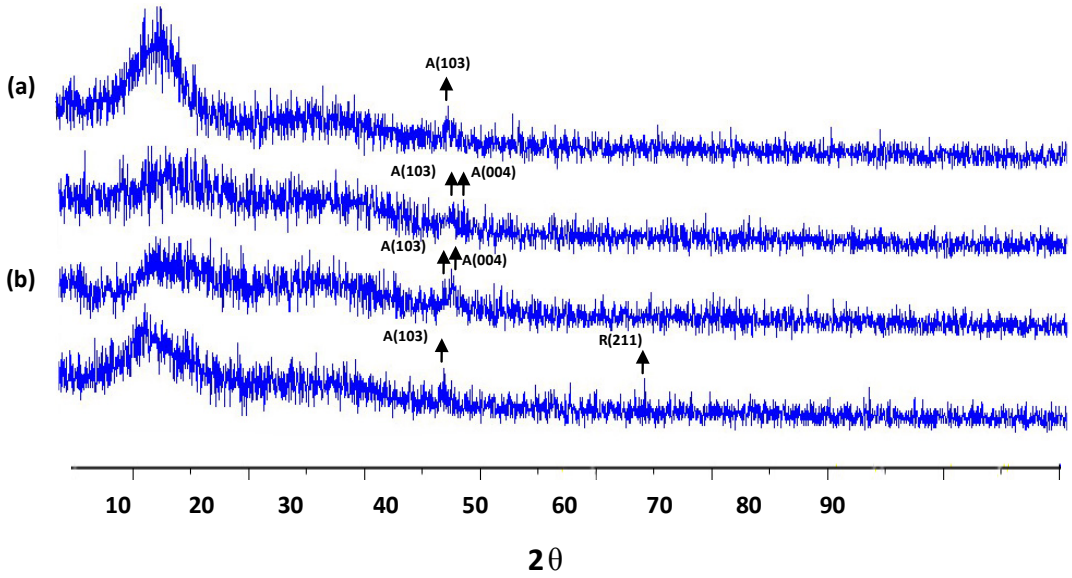

Figure 3(a) there is an anatase A(103) phase with (103) crystallographic direction.

show crystallographic structure. For as deposited Ti layer, A(103) begin to grow. By increasing temperature to 393K and 493K, A(103) and A(004) crystallographic directions are clear, and in higher temperature (593K) there are two different A(103) and R(211) crystallographic directions, and phase transition happened so there is a phase transition from anatase to rutile structure.

REFERENCES

[1] A.Fujishima, K. Honda. Nature 238 (1972) 37.
[2] A.Fujishima, K. Honda, Bull. Chem. Soc. Jpn 44 (1971) 1148.
[3] B.O'Regan, M. Gralzel. Nature (Lond.) 353 (1991) 737.
[4] A.Hagfelt, M. Gratzel, Chem. Rev. 95 (1995) 49.
[5] C.G.Grankvist, Materials Science for Solar Energy Conversion Systems, Pergamon Press. Oxford, 1991
[6] P.Jin. L. Miao. S. Tanemura. G. Xu. M. Tazawa. K. Yoshimura, Appl. Surf. Sci. 212-213 (2003) 775.
[7] G.S.Brady. Materials Handbook, 10th ed.. McGraw-Hill, New York. 1971
[8] Y.H Lee. Vacuum 51 (1998) 503.
[9] L.Miao. P Jin. K. Kaneko, S. Tanemura. in: H.J. Gao. H. Fuclis.D.M. Chen (Eds). Proceedings of Eighth 1UMRS International Conference on Electronic Materials: Advanced Nanomaterials and Nanodevices, IOP, 2002. p. 943.
[10] L.Miao. S Tanemura, P. Jin, K. Kaneko. A. Terai, N. Nabatova- Gabain. J. Cryst. Growth 254 (2003) 100.
[11] L. Miao, P. Jin. K Kaneko, A. Terai, N. Nabatova-Gabain, S. Tanemura. Appl. Surf. Sci. 212-213 (2003) 255.
[12] S.Tanemura. L Miao. P. Jin. K. Kaneko. A. Terai, N. Nabatova- Gabain, Appl. Surf. Sci. 212-213 (2003) 654.
[13] W.Wunderlich, L. Miao. S. Tanemura, M. Tanemura, P Jin. K. Kaneko, A. Terai, N. Nabatova-Gabin, R. Belkada, Int. J. Nanosci 2004; (in press).
[14] L.Miao. S. Tanemura, Y. Kondo, M. Iwata, S. Toh. K. Kaneko. Appl Surf. Sci. 238 (2004) 125.
[15] L.Miao, S. Tanemura, H. Watanabe, Y. Mori. K. Kaneko, S. Toh. J_Cryst. Growth 260/1-2 (2004) 118.