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Study the Influence of Thermal Annealing in Presence of Oxygen Flow on the Nano-Structures and Optical Properties of Titanium Oxide Thin Layers

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Abstract: Ti films of the same thickness, were deposited on glass substrate under UHV conditions at room temperature and near normal deposition angle. Different annealing temperatures as 410 ºK and 510 ºK with uniform 7 cm^3/sec, oxygen flow, were used for producing titanium oxide layers. AFM and XRD methods were used to study the nano-structures of produced layers. Due to annealing process, roughness of the layers changed. The optical properties also determined by spectrophotometer in UV-VIS range. The gettering property of Ti and annealing temperature, can play an important role on the nano-structures and reflection curves of the films.

Key words: Titanium dioxide; Thin films; AFM; XRD; spectrophotometer.

INTRODUCTION

TiO_2 is one of the most widely studied ceramic materials used as films. Now-a-days, TiO2 films have a wide range of applications on glass substrates such as heat mirror films on building and automotive glasses, self-cleaning glass, air cleaning lamp, wiperless windshield etc. For these applications, thin TiO_2 films with nanometer thickness are mostly employed (Hasan, 2010). TiO_2 is known to exist in an amorphous form and to crystallize in three distinct structures: two tetragonal phases, anatase (a = 3.785 °A, c = 9.514 °A) and rutile (a = 4.593 °A, c = 2.959 °A), and a third orthorhombic phase, brookite (a = 5.456 °A, b = 9.182 °A,c = 5.143 °A). Among them, rutile is not only the densest, but also thermodynamically the most stable phase, so it is interesting for optical coatings (Rao and S. Mohan, 1990; Balasubramanian, 1993).

The properties of the titanium oxide films depend not only on the preparation techniques but also on the deposition conditions. The influence of different deposition parameters on the nanostructure of metallic films has been reported by many researchers (Movchan, 1969; Thornton, 1975; Messier, 1985; Grovenor, 1984; Hentzell, 1984; Messier, 1984; Messier, 1986; Savaloni, 2002; Barna, 1998; Petrov, 2003; Thompson, 2000; Haleh Kangarlou, 2011).

The aim of this work is to study the influence of annealing temperature and oxygen flow on nano-structure, roughness and optical properties of produced layers and also investigation about crystallographic directions and their dependence to mentioned parameters.

MATERIALS AND METHODS

Titanium films of 75 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 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 the same during coating. Vacuum pressure was about 10^{-6} mbar and deposition rate was 1Aº/sec. Thickness of the layers were determined by quartz crystal technique. Titanium dioxide layers produced using an annealing oven in presence of uniform oxygen flow at two different annealing temperatures (410 ºk and 510 ºk).

The nano-structure 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 1 s per step, while the surface physical morphology and roughness were obtained by means of AFM (Dual Scope ™ DS 95-200/50) analysis. Reflectance of the films were measured using UV-VIS spectrophotometer (Hitachi U – 3310) instrument.

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RESULTS AND DISCUSSION

Figure 1 (a-c) shows, AFM images of the layers produced in this work. Figure 1 (a), shows the topography of as deposited Ti film, at room temperature and 75 nm thickness. As it can be seen, surface is full of domed grains.

During annealing process at 410 ºK and in presence of uniform oxygen flow (7 Cm^3/sec), oxygen penetrates to grain structure and brake it down to tiny needle-like grains (Figure 1 (b)). In Figure 1 (c) annealing temperature increases to 510 ºK, and as it can be seen beside penetrating oxygen, because of high temperature, surface diffusion happens and bigger domed grains appear.

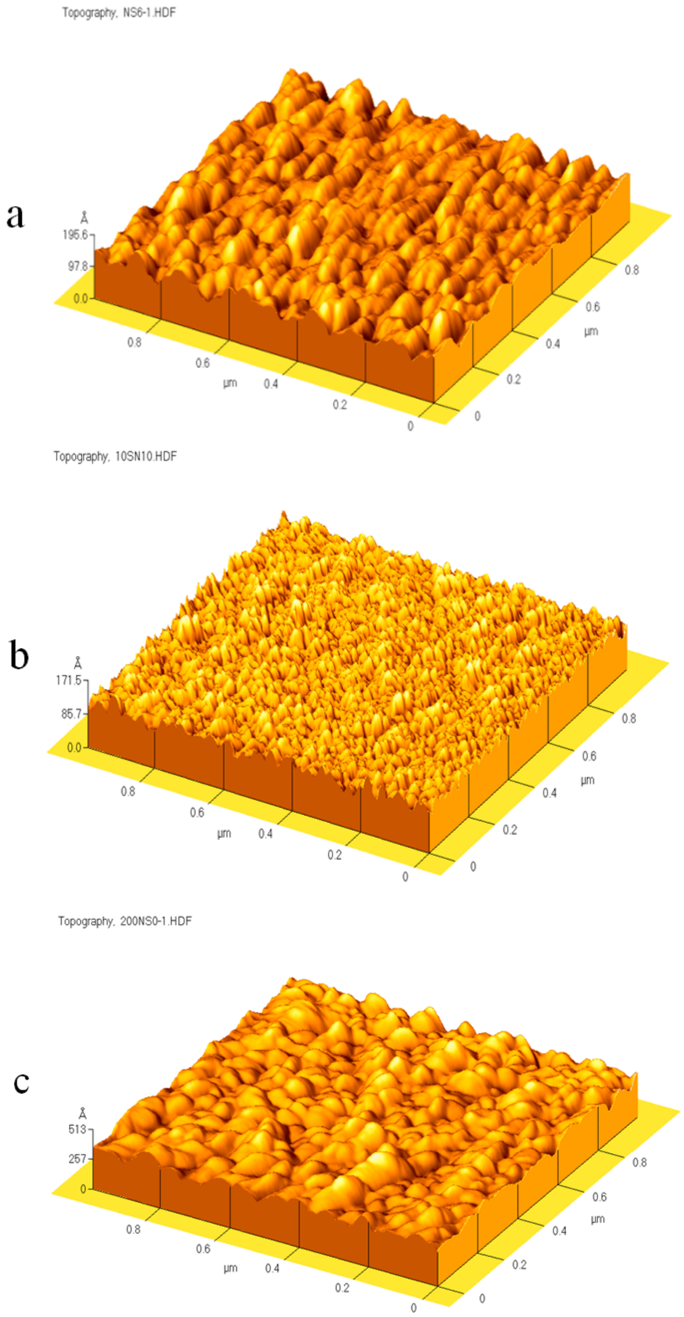

Fig. 1: The AFM images of (a) as deposited Ti; (b) 410 ºK; and (c) 510 ºK, annealing temperature in presence of oxygen flow.

Figure 2 shows, roughness diagram of the layers produced in this work. In presence of annealing temperature and oxygen flow, roughness decreases in first step (450 ºK), by increasing annealing temperature to 510 ºK, surface diffusion is dominant and roughness increases, which is in agreement with AFM results.

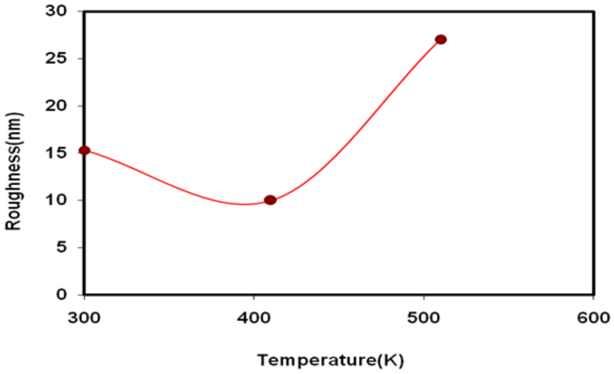

Fig. 2: The roughness diagram of (a) as deposited Ti; (b) 410 ºK; and (c) 510 ºK, annealing temperature in presence of oxygen flow.

Figure 3 (a-c) shows, XRD patterns of the layers produced in this work. As it can be seen, from Figure 3 (a), there is no any clear crystallographic direction which shows that the produced Titanium in room temperature is amorphous. There is a doubt about, anatase A(103) phase with (103) crystallographic direction, that is because of gettering property of Ti. Figure 3 (b) shows XRD pattern of titanium dioxide on glass substrate at 410 ºK annealing temperature and 7 (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, and by increasing annealing temperature to 510 ºK in presence of the same uniform oxygen flow, A(004) crystallographic direction is clear. So by increasing annealing temperature a phase transition for titanium oxide happens.

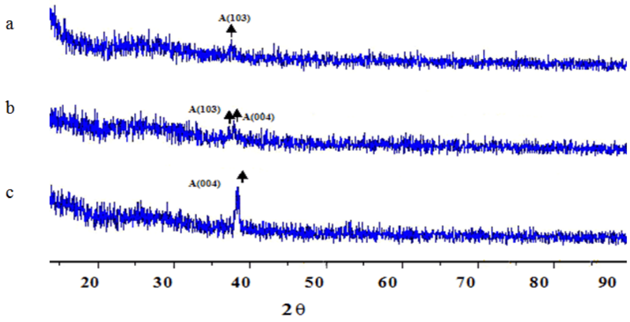

Fig. 3: The XRD patterns of (a) as deposited Ti; (b) 410 ºK; and (c) 510 ºK, annealing temperature in presence of oxygen flow.

Figure 4 shows, reflection of the layers produced in this work, as it can be seen in presence of temperature and oxygen flow there is a competition between gettering property and surface diffusion, but in general because of presence of heat and migration of grains, and decreasing fraction of voids reflection increases.

The structural and optical properties of titanium layers in presence of heat and oxygen flow, were studied. This was accomplished by studying the relationship between AFM and XRD results, in addition to reflection of the layers. The morphology of the layers changed by increasing heat and in presence of oxygen. By increasing annealing temperature and in presence of oxygen flow, in the first step oxygen penetrated to grain structure and broke it down to needle like grains, and in second step by increasing heat, because of dominant surface diffusion bigger domed grains appeared. Roughness of the layers also changed, in agreement with AFM images. By increasing temperature to 410 ºK and 510 ºK, XRD patterns showed anatase structure in A(103) crystallographic direction and A(103) and A(004) crystallographic directions. The produced Titanium layer at room temperature was amorphous. Reflection curves in general show increasing trend because of heat and surface diffusion. Almost two different properties as gettering and surface diffusion were in competition with each other for the layers studied in this work.

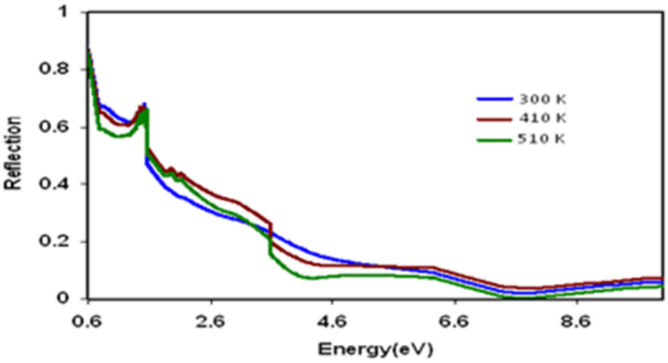

Fig. 4: The Reflection diagram (a) as deposited Ti; (b) 410 ºK; and (c) 510 ºK, annealing temperature in presence of oxygen flow.

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