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Investigation of Structural Properties of Titanium Thick Layers Produced by PVD Method under Heat Process

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Abstract: Titanium thin layers with the same deposition conditions were deposited on glass substrates at room temperature. Different annealing temperatures namely, 400 K, 500 K and 600 K with uniform 9 cm^3/sec oxygen flow were used to produce titanium oxide layers. The nano-structure of layers were studied using AFM and XRD methods. Optical Reflectance were studied by spectrophotometer method. Correlation between nano-structures and optical reflectance versus annealing temperature and penetrating oxygen were Annealing temperature and getting property of Titanium atoms play an important role on structural properties and Reflectance of layers.

Key words: AFM · XRD · Annealing process

INTRODUCTION

Thin films have been finding more and more applications in various areas. The oxide films are stable, strongly adherent to the substrate, mechanically hard and resistant to moisture and acids [1]. Recently, titanium oxide (TiO_2) thin films have been emerged as one of the most promising oxide materials owing to their optical, electrical and photoelectrochemical properties [2]. Amorphous TiO_2 layers are mainly used as high refractive optical layers in layer stacks with antireflection properties or in filters [3]. For its high refractive index, wide band gap and chemical stability, polycrystalline TiO_2 films are used for a variety of applications such as optics industry, dyesensitized solar cells, dielectric applications, self-cleaning purposes and photocatalytic layers [4]. Previous studies indicate that the properties of TiO_2 films appear to strongly depend on the process conditions and starting materials used in the processes [2]. There are many different methods to prepare TiO_2 films, including sol-gel method [5], Chemical Bath Deposition [1], spin coating-pyrolysis process [2, 6], sputtering [7, 8], PECVD [9], liquid deposition method and so on.

MATERIALS AND METHODS

Titanium films of 266 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 7 degree to the direction of the evaporated beam and distance between evaporation crucible and substrate was found to be 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} tour and deposition rate was 0.7 A°/sec. Thickness of the layers were determined by using quartz crystal microbalance. The Ti thin films were heated at temperatures (400, 500 and 600 Kelvin degree) under uniform oxygen flow 9 cm^3/sec for about 2 hours to change the structure of layers and to produce titanium oxide layers. The structure of these films was studied using a Philips XRD X'pert MPD Diffractometer ($CuK_{\acute{a}}$ 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. Reflectance of the films were measured by using UV-VIS spectrophotometer (Hitachi U - 3310) instrument.

RESULTS AND DISCUSSION

Figure 1 (a-d) shows, AFM images for the layers produced in this work. Figure 1 (a), shows the topography of as deposited Ti film, at room temperature and 266 nm thickness. As it can be seen, surface is full of domed grains.

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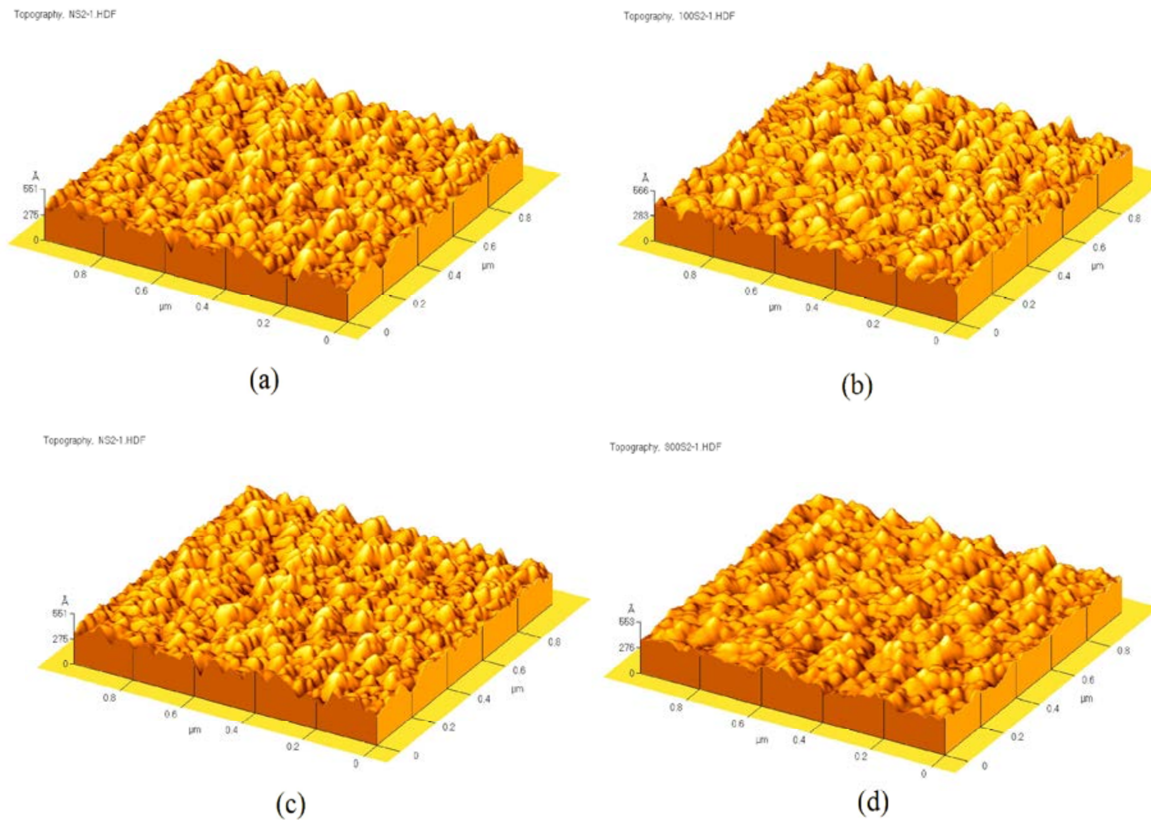

Fig. 1: AFM images of (a) as deposited Ti; (b) 400 K; (c) 500 K; and (d) 600 K, annealing temperature and uniform oxygen flow

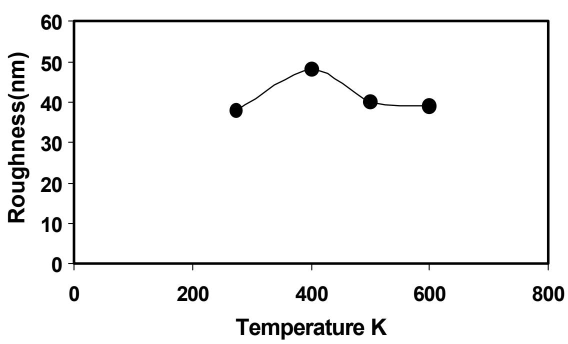

Fig. 2: Roughness diagram of, the layers produced in this work under different annealing temperatures and an uniform oxygen flow

In presence of annealing temperature of 400 K and uniform oxygen flow (9 Cm^3/sec), oxygen will penetrate to the grain structure, but surface is still full of domed grains (Figure 1 (b)). In Figure 1 (c) annealing temperature increases to 500K and as it can be seen beside penetrating oxygen, annealing temperature is also high enough, surface is still full of domed grains, but because of penetration of oxygen grains break down to smaller grains. As it can be seen from Figure 1 (d), annealing temperature increases to 600 K, because of high temperature, except penetration of oxygen grain have surface and bulk diffusion, so they coalescence and smoother layers with small grains produces.

Figure 2 shows, diagram of roughness for the layers produced in this work. In presence of annealing temperature and oxygen flow, roughness increases in first step (400K), by increasing annealing temperature to 500K and 600 K, except penetration of oxygen surface and bulk diffusions happens that tends to a decreasing trend for roughness, that is in agreement with AFM results.

Figure 3 (a-d) shows, XRD patterns for the layers produced in this work. As it can be seen, from Figure 3 (a), there is an A(004) crystallographic peak, for as deposited Titanium layer with 266 nm thickness. That is because of gettering property of Titanium atoms. These atoms interact with impurities, specially with oxygen, that tends to preparation of Titanium oxide layer. In presence of 400 K annealing temperature and uniform oxygen flow for same thickness (266 nm), two A(101), A(004) and R(310),

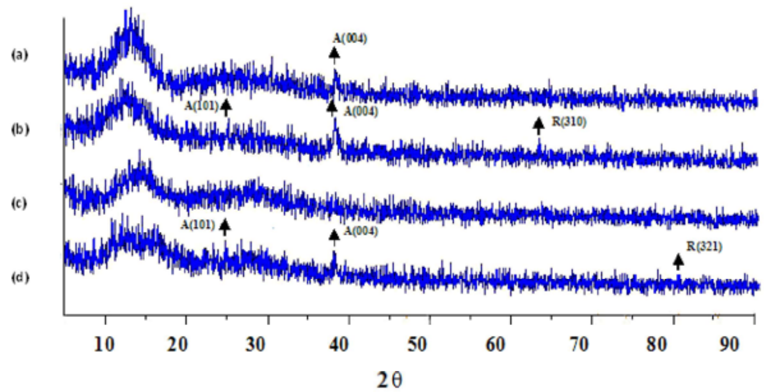

Fig. 3: XRD patterns of (a) as deposited Ti; (b) 400 K; (c) 500 K; and (d) 600 K, annealing temperature and uniform oxygen flow

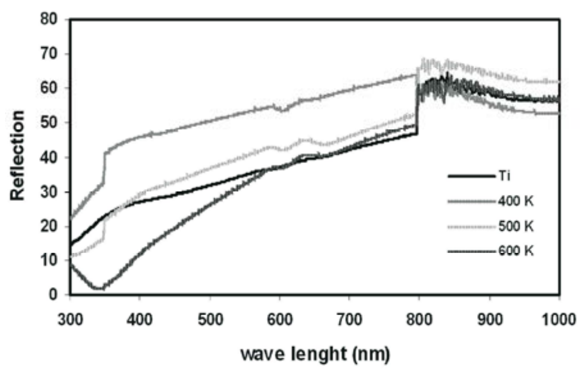

Fig. 4: Reflectance diagrams of, the layers produced in this work under different annealing temperatures and an uniform oxygen flow

Titanium dioxide crystallographic peaks appear (Figure 3 (b)). As it can be seen from Figure 3 (c), there is no crystallographic direction for 500 K annealing temperature, that could be, because of competition between diffusions, gettering property of Ti atoms and penetration of oxygen to grain structure. Figure 3 (d) shows, XRD pattern of Ti layers under 600 K annealing temperature and oxygen flow. As it can be seen Titanium dioxide A(101), A(004) and small R(321) crystallographic peaks form on layer. All XRD patterns are noisy and they have a wide peak at about 15 degree, that is because of glass substrate.

Figure 4 shows, Reflection of the layers produced in this work, as it can be seen in presence of annealing temperature and oxygen flow there is a competition between gettering property, surface and bulk diffusions and penetration of oxygen, but in general, by increasing annealing temperature, we are encountered with migration and coalescence of grains there for bigger grains with more fraction of voids appear on layers, that tends to a decreasing trend for reflectance curves.

Summery: The structural and optical reflectance of Titanium layers in presence of heat and oxygen flow, were obtained. This was accomplished by the studying the relationship between AFM and XRD results, in addition Reflection of layers. The morphology of the Ti and Titanium dioxide layers of 266 nm thickness by increasing heat and presence of oxygen flow is almost the same for the layers produced in this work. This result tends us to a fact that is the high thickness of a layer is very important and there is a limited thickness for penetrating oxygen, for thick layers oxygen couldn't penetrate to depth of substrate, so it couldn't tend to clear changes in morphology of layers. roughness of the layers will change in agreement with AFM images. XRD patterns showed accumulation of anatase and rutile crystallographic peaks. Reflection curves in general show decreasing trend because of heat and diffusions. Almost different properties as gettering, surface and bulk diffusions and penetrating of oxygen, are in competition with each other for the layers investigate in this work.

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