Influence of Annealing Temperature and Oxygen Flow
On the Nanostructures of UHV Deposited Titanium Thin Films

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Abstract: - Ti films of same thickness, and near normal deposition angle, and same deposition rate were deposited on glass substrates, at room temperature, under UHV conditions. Different annealing temperatures as 150, 250 and 350 Celsius degree with uniform 5 cm^3/sec, oxygen flow, were used for producing titanium oxide layers. Their nano structures 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 in the nanostructure of the films.

Key-Words: - Titanium; Titanium dioxide; Thin films; AFM; XRD

1 Introduction
Ti0₂ 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 [1]. The applications of Ti0₂ thin films include gas and humidity sensors, solar energy converters or reflecting and protective coatings on optical elements. Moreover, photocatalytic activity of Ti0₂ thin films has been investigated for environmental purification and self-cleaning applications, governed by the photo-induced decomposition of organic pollutants [2-4]. As known, Ti0₂ crystallizes in three different crystallo-graphic structures: brookite (orthorhombic), rutile (tetragonal) and anatase (tetragonal). Brookite is formed only in extreme preparation conditions, while rutile is the most common Ti0₂ crystal phase in nature. Anatase is metastable and thus it can be synthesized only in the restricted range of growth conditions. Indeed, according to the phase diagram, irreversible anatase to rutile phase transition takes place at temperatures higher than about 700 °C [5]. It is well known, however, that anatase exhibits the highest photocatalytic activity among the crystallographic phases of Ti0₂, being more appropriate for related applications [6,7]. In recent years important scientific activity was dedicated to finding production routes leading to nano-structured materials with tailorable functional properties. As known, control at the nano-scale of material structure during the synthesis process offers the possibility of designing high quality devices with improved and pre-defined performances. Nano-crystalline Ti0₂ thin films have been prepared up to now by a large variety of growth techniques, such as metal- organic chemical vapour deposition (MOCVD) [8,9], reactive magnetron sputtering [10,11 ], sol-gel chemistry [12], filtered cathodic vacuum arc [13] and pulsed laser deposition (PLD) [14-22]. PLD has important advantages over other deposition methods. Through the appropriate adjustments of process parameters such as laser fluence, number of laser pulses, ambient reactive gas pressure and substrate temperature PLD allows for the precise control of the growth process and, thus, the synthesized thin films' crystalline structure, stoichiometry, thickness, as well as their adherence to the substrate surface [23-25]. However, in general the Ti0₂ films prepared by PLD mainly consisted of mixtures of crystal phases [16, 19-21]. It was shown that the formation of pure, monophasic Ti0₂ thin films of either rutile or anatase structure require, besides the adjustment of the process parameters, special substrate materials ensuring small in-plane lattice mismatch at the films- substrate interface [14,22]. In this work we want to study the influence of
annealing temperature and oxygen flow on nano structure and roughness of produced layers and also crystallographic directions and their dependence to mentioned parameters.

2 Experimental Details
Titanium films of 105 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 quadro 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 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 1.3 A/sec. Thickness of the layers were determined by quartz crystal technique. We used annealing oven and different annealing temperatures (150, 250, 350 Celsius degree) and uniform oxygen flow to change nano structure of layers and produce titanium dioxide layers. The nanostructure of these films was obtained using a Philips XRD X’pert MPD Diffractometer (CuKα radiation) with a step size of 0.03 and count time of 1 s per step, while the surface physical morphology and roughness was obtained by means of AFM (Dual Scope™ DS 95-200/50) analysis.

3 Result and Discussion
Figure 1 (a-d) show the morphology of the produced layers (AFM). Figure 1 (a) shows the AFM image of deposited Ti film, in room temperature and 105 nm thickness. As it can be seen, the surface is full of small grains.

By increasing annealing temperature to 150 ºC and in presence of uniform oxygen flow (5 Cm³/sec) oxygen will penetrate to the grain structure and brake them down to tinnier needle like grains (Figure 1 (b)). In Figure 1 (c) annealing temperature increases to 250°C, and as it can be seen the grains are domed and clearly bigger. This is because of surface diffusion in this temperature. Figure 1 (d) shows the produced layer with 350°C annealing temperature, as it can be seen because of surface and bulk diffusions in high temperature all grains have a uniform shape and the layer is smooth.
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 structure and migration of grains. But in 350°C because of the surface and bulk diffusions, the surface gets uniform, that is in agreement with AFM images. So roughness will decrease in 350°C annealing temperature. As we know Ti is a getter metal and in presence of oxygen and of course heat will Convert to titanium dioxide.

![Roughness Diagram of Layers](image)

Figure 3 (a-d), shows XRD images for layers in this work. As it can be seen layers changed to titanium dioxide layers, in most of all anatase A(004) Crystallographic direction in room temperature, 100°C and 200°C annealing temperatures. We have TiO2 layers with anatase structure and A(004) crystallographic direction (Figure 3 (a-c)). In Figure 3 (d), the anatase peak is wide it seems to be additional of A(103) and A(004) crystallographic directions. by increasing annealing temperature the peaks are sharper and there are more crystallized direction.

![XRD Images of Layers](image)

4 Conclusion

The influence of annealing temperature and uniform oxygen flow on titanium layers with same thickness were obtained. This was accomplished by the studying the relationship between AFM and XRD results. The morphology of the layers changes by increasing heat and presence of oxygen. By increasing annealing temperature and presence of oxygen flow, in the first step oxygen penetrate to grains and brake them down to needle like grains, by increasing heat, because of surface diffusion grains get domed and bigger more heat (350°C) causes additionally bulk diffusion and surface become smooth. Roughness increases by increasing annealing temperature and at 350°C decreases that is in agreement with AFM results. Because of gettering property of Ti and presence of oxygen titanium dioxide layers produced, XRD patterns showed anatase structure in A(004) crystallographic direction for all layers and additional A(103) crystallographic direction at 350°C. by increasing annealing temperature the peaks are sharper and there are more crystallized direction.

References:

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