Investigation of Optical and Structural Properties of Titanium Very Thin Layers 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, 120 ºC, 270 ºC and 320 ºC with uniform 7 cm3/sec oxygen flow were used to produce titanium oxide layers. The nano-structure of layers were studied using AFM and XRD methods. Optical properties and optical constant of layers were calculated using kramers-kronig method. Correlation between nano-structures and optical properties were investigated. Fraction of voids and band gap energy were also estimated. Annealing temperature and getting property play an important role on both structural and optical properties of layers.

Key words: Titanium Dioxide, AFM, XRD, Kramers-Kronig.

INTRODUCTION

Applications, involving low-loss, low-scatter optical coatings for visible and near infrared optics (K.A. Vorotilov, et al., 1992; M.G. Krishana, K. Narasimha, 1983; J.A. Dobrowski, 1987) and for electrical devices (P. Babelon, et al., 1998; Y. Leprince-Wang, et al., 1997), have simulated a considerable amount of activity in fabrication of dielectric films with high refractive index and low absorption. Titanium oxide thin layers exhibit many attractive characteristics. Thanks to their high refractive index and dielectric constant, to their transparency in a wide spectral range, and to their stability, they are more and more investigated.

It is worthy to note that the optical properties of titanium oxide thin layers are generally different from the bulk properties, and optic researches need to relate these properties to the thin layer stoichiometry and morphology (packing, crystallisation, and structure of crystals).

There are many methods for the preparation of titanium thin oxide films such as sputtering, chemical vapor deposition, ion beam assisted deposition, reactive vaporization, laser-assisted evaporation, sol-gel process, and spray pyrolysis (P. Lobl, et al., 1994; N.C. Da Cruz, et al., 2000; H.K. Jang, et al., 2000; Q Tang, et al., 1999; M. Rinner, et al., 2000). However, the magnetron sputtering technique seems to be the most favorable because the material can be supplied to the growing surface layer in the correct proportions and with sufficient energy to ensure the formation of a dense structure and the deposition parameters can be controlled easily (T. Ohwaki, Y. Taka, 1989; L. Meng, M.P. Dos Santos, 1993; T. Pawlewicz, 1982; J.M. Bennet, 1989).

MATERIALS AND METHODS

Titanium films of 18 nm thickness were deposited on glass substrates at room temperature. The residual gas was composed mainly of H2, H2O, CO and CO2 as detected by the quad rotor mass spectrometer. The substrate normal was at 5 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 torr and deposition rate was 0.5 A/sec. Thickness of the layers were determined using quartz crystal microbalance. The Ti thin films were heated at temperatures (120, 270, 320 Celsius degree) under uniform oxygen flow 6 cm3/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 (CuKα 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.

RESULTS AND DISCUSSION

Structural Properties:

Figure 1(a-c) shows the morphology of produced layers obtained by means of AFM. By annealing at 120 ºC in presence of uniform oxygen flow (6 Cm3/sec), oxygen will penetrate into the structure and titanium oxide...
begin to form as tiny needle like grains (Figure 1(a)). In Figure 1(b), at the annealing temperature of 270 °C, it can be seen that the grain’s size of titanium oxide has been increased. This is because of high surface diffusion and coalescence of grains. Figure 1(c) shows the produced layer with 320 °C annealing temperature. Because of surface diffusion at high temperature and coalescence, grain’s size increases and more voids form on the layer.

Fig. 1: The AFM images of TiO\textsubscript{2} layers with annealing at (a) 120 °C ; (b) 270 °C ; (c) 320 °C.

Figure 2 shows the roughness of the layers vs annealing temperature. By increasing annealing temperature, roughness is also increasing due to oxygen penetration into the titanium thin film and subsequent titanium oxide formation. So by increasing annealing temperature (from 120 °C to 320 °C), roughness increase from 10 nm to 20 nm in accordance with AFM images. Ti has a high solubility for oxygen and also has a high reactivity with oxygen to produced titanium dioxide.

Fig. 2: The roughness diagram of TiO\textsubscript{2} layers, at 120 °C, 270 °C and 320 °C annealing temperature.

Figure 3 (a-c), shows XRD pattern of the layers produced in this work. As it can be seen all the layers are amorphous, and there is no sharp peak for the 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 (18 nm) of the layers.

Fig. 3: The XRD patterns of TiO\textsubscript{2} layers with annealing at (a) 120 °C; (b) 270 °C; (c) 320 °C.
**Optical Properties:**

Figure 4 shows the real and imaginary parts of refractive indices. The general trends of all curves are the same. By increasing annealing temperature, for the real part of refractive index, curves cross each other and there is no specific trend, that is because of comparison between increasing temperature and also oxygen penetration to grain structure in one hand and very low thickness of the layers (d = 18 nm) on the other hand (Figure 4(a)).

Figure 4 (b) shows the imaginary part of refractive index (k). The general trends of all curves are the same. As it can be seen, extinction coefficient curves cross each other, that is because of comparison between increasing annealing temperature and oxygen penetration to grain structures, in this very low thickness of produced layers in this work.

**Fig. 4:** The Refractive index of deposited TiO$_2$ films with different annealing temperatures (a) Real part; (b) Imaginary Part.

Figure 5 shows the real and imaginary parts of dielectric constant. The trends of all curves are the same in both 5 (a) and 5 (b) figures. $\varepsilon_1$ and $\varepsilon_2$ curves also cross each other as we discussed before. By increasing annealing temperature, reaction between Ti atoms and oxygen increases, so dielectric property increases.
Figure 5 shows the dielectric constants of deposited TiO$_2$ films with different annealing temperatures. (a) Real part; (b) Imaginary Part.

Figure 6 shows the absorption coefficient curves for the layers produced in this work. By increasing annealing temperature, surface diffusion happens which tends to formation of more voids. Therefore transmittance increases and absorbance decreases. High temperature is a good reason for more oxygen absorption by Ti atoms. Due to very low thickness of the layers produced in this work, they all tend to incoordination in results.

Figure 7 shows the fraction of voids for the layers produced in this work. As it can be seen, by increasing annealing temperature, surface diffusion happens that tends to formation of more voids on the layers which is in agreement with AFM images and structural analysis that discussed before.
Figure 8 shows the band gap energy for titanium dioxide layers produced in this work, with very low thickness (d=18 nm). By increasing temperature, surface diffusion happens that tends to formation of more voids and oxygen absorption by titanium atoms, so band gap energy increases. The estimated band gap energy for 120 °C annealing temperature is about 3.7 eV and for 270 °C annealing temperature is about 3.73 eV and for 320 °C annealing temperature is about 4.4 eV.

Conclusions:
Optical properties of titanium dioxide layers were determined using kramers-kronig method. All curves cross each other, that is because of comparison between increasing annealing temperature and oxygen penetration to grain’s structure. The point is the low thickness of the layers produced in this work that tends to inconsistency in results.

By increasing annealing temperature, surface diffusion happens and the reaction rate between titanium atoms and oxygen increases. Bigger grains with more void fractions formed which is in agreement with AFM results. Dielectric property and band gap energy increases for the layers produced in this work.

REFERENCES


