Optik 124 (2013) 107–112

Contents lists available at SciVerse ScienceDirect

Optik

journal homepage: www.elsevier.de/ijleo

Deposition angle dependence of optical and structural properties of titanium nano-layers

Haleh Kangarlou*, Maryam Motallebi Aghgonbad, Zahra Barjisi

Faculty of Science, Urmia Branch, Islamic Azad University, Urmia, Iran

ARTICLE INFO

Article history:
Received 9 June 2011
Accepted 14 November 2011

Keywords:
Titanium
Thin films
Kramers–Kronig
Effective medium approximation

ABSTRACT

Titanium nano-layers on glass substrates were prepared by physical vapor deposition method, under HV conditions, with different deposition angles of 0°, 10° and 15° at 373 K temperature. The layers thickness is measured 21 nm, by quartz crystal technique. The nano-structures of the films were obtained, using X-ray diffraction (XRD), and atomic force microscopy (AFM) methods, and their optical properties were determined by spectrophotometer in the spectral range of 400–800 nm. The optical functions were calculated by using Kramers–Kronig relations. The effective medium approximation analysis was employed to establish the relationship between the nano-structure and (EMA) predictions. The aim of this research is to use different deposition angles to produce titanium nano layers, and investigate the relation between nano-structures and optical properties. Nano-structure of the layers showed tapered crystallites separated by voids (porous structure), that is in agreement with, Zone I of structure zone model. There was no clear crystallographic direction for the layers produced in this work.

The optical data, show dependence to deposition angle, and there is a good agreement between them and Johnson and Christy's data [17].

1. Introduction

Titanium and its alloys, due to good mechanical properties, a very high strength, excellent corrosion resistance and good biocompatibility have attracted considerable interest in several industries, such as aerospace engineering, motor science, chemical processes and biomedical industries [1,2]. Because of their good biocompatibility, they have been used widely as an implant material in human body and also in dental and orthopedic fields [3,4]. The chemical stability of titanium results the presence of a thin but stable oxide film, typically a few nanometers thick. Recently, preparing good titanium films with surfaces more defined than those of polished bulk metal is easier because of the advances in the evaporation technique [5]. Thin films have been finding more and more applications in various areas. Due to a larger area to volume ratio, thin films properties are different from their bulk material properties [6]. Pure titanium is a highly reactive metal and it is normally protected by an oxide film consisting mainly of TiO_2 which is very stable [1,2]. Titanium oxide is one of the transparent conductive oxides. The oxide films are stable, strongly adherent to the substrate, mechanically hard and resistant to moisture and acids [7].

For optimal performance of devices, these films should have special properties. Different properties of thin films are strongly influenced by the nano-structure of films such as grain size, nano-strain, crystallographic orientation and other features. It is shown that the nanostructure of thin films is strongly affected by film preparation procedures and deposition condition. For example substrate temperature [8–10], angle of incidence [11–13], deposition rate [14,15] and film thickness [16] have important effects on the morphology and nano-structure of thin films. Ti thin films despite their importance in different technologies are only reported by Johnson and Christy [17] and of bulk Ti samples by Lynch et al. [18] and Wall et al. [19]. Therefore, it is of interest to find the relationship between different theories [20–22] given for optical parameters and the structural changes described by variation of film thickness and substrate temperature (structure zone model, SZM) [23–25] for Ti thin films produced under UHV conditions.

2. Experimental details

Titanium films at three angles were deposited on glass substrates (18 mm × 18 mm × 1 mm cut from microscope slide) by resistive evaporation, from tungsten boats at 373 K temperature. The purity of titanium powder was 98%. An ETS 160 (Vacuum Evaporation System User Manual) coating plant with a base pressure of 10^{-6} mbar was used. Prior to deposition, all glass substrates were ultrasonically cleaned in heated acetone then in ethanol.

* Corresponding author.
E-mail addresses: h.kangarlou@iaurmia.ac.ir (H. Kangarlou), m.motallebi89@gmail.com (M.M. Aghgonbad), barjisi@gmail.com (Z. Barjisi).

0030-4026/$ – see front matter © 2011 Elsevier GmbH. All rights reserved.
doi:10.1016/j.ijleo.2011.11.089

The substrate holder was a disk of 36.5 cm in diameter with adjustable height up to 50 cm and also adjustable keepers for replacement any kind of substrate on it. The distance between the center of the evaporation boat and the center of the substrate was 45 cm. The layers thicknesses were determined by quartz crystal technique. Other deposition conditions such as deposition rate, vacuum pressure, and substrate temperature were the same for all layers. The layers with known 0°, 10° and 15° were produced at 373 K. The nano-structures of these films were obtained by 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^{TM} DS 95-200/50) analysis. Transmittance of the films were measured by using UV–VIS spectrophotometer (Hitachi U-3310) instrument. The spectra of the layers were in the range of 400–800 nm wavelength, and by using Kramers–Kronig relations [13], we extrapolated the rest of reflectivity curves with Johnson and Christy's [14] data. The optical properties such as n, k, ε_1, ε_2, α, Band-gap energy, void fraction, energy loss function and plasma oscillations were calculated and compared with Johnson and Christy's results [14]. The correlation between Ti thin film nano-structure and its optical properties, for different film deposition angles, can be achieved through using the effective-media approximation (EMA). The changes in the fraction of voids can be attributed to change of the nano-structure in the evolution of film nano-structure by substrate temperature, film thickness, deposition angle, and deposition rate. There was a good agreement between nano-structures, optical properties and predictions of EMA method.

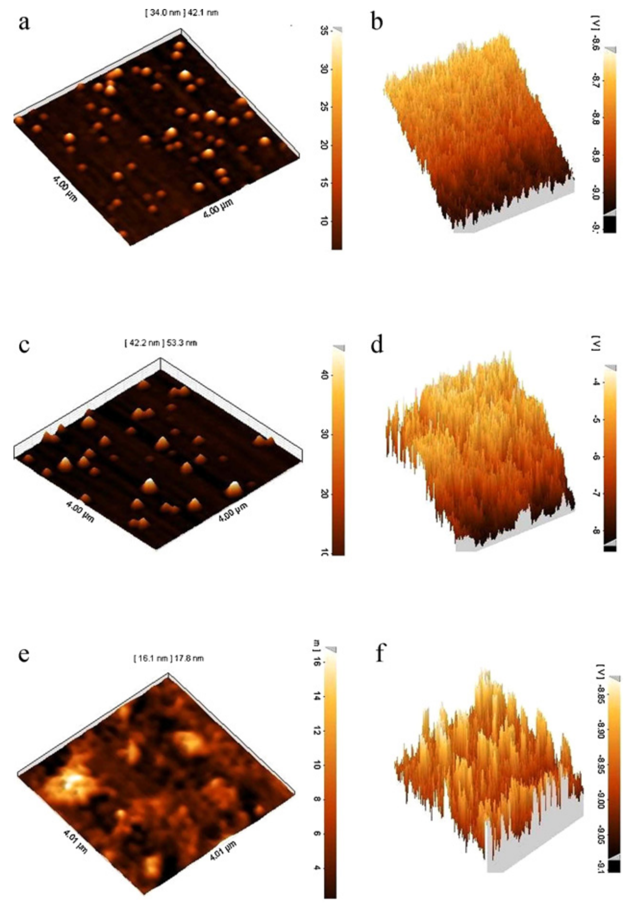

Fig. 1. AFM images of titanium/glass layers with different deposition angles.

3. Results and discussions

3.1. Structural analysis

Fig. 1(a–f) shows topographic and phase images, for titanium/glass nano layers produced in this work. As it can be seen from AFM images (Fig. 1), by increasing deposition angle, topography of the layers will change. For normal deposition angle, the surface has more titanium grains and less voids, because of best condition for coating. By increasing deposition angle for the same layers, the surfaces have less titanium grains, and more voids form on them. According to structure zone model [23] for 373 K temperature T_s/T_m is 0.17, that T_s and T_m are the substrate temperature and the melting point of titanium, respectively. This is in agreement with AFM images and we are encountered with Zone I that consists of, tapered crystallites separated by voids (porous structure).

Fig. 2 shows the X-ray diffraction (XRD) for the layers produced in this work. As it can be seen, because of nanometric thickness of the layers (d = 21 nm) and low deposition temperature (T = 373 K), all the layers are amorphous, and there is no crystallographic direction, for them.

3.2. Optical analysis

Fig. 3 shows the reflectance curves of very thin layers of titanium powder deposited on glass substrates with the same deposition conditions and different deposition angles. Johnson and Christy's results for titanium layers are also included for comparison. Thickness of the layers in this research is about 21 nm and they were prepared at the same 373 K temperature. The layers are too thin so they cannot be seen with naked eye. As we can see, Reflectance for the layers produced in this work is very low, that is because of nano metric thickness of the layers and also by increasing deposition angle, reflectance decreases. That is because deposition conditions become undesirable and formation of voids increases. The general trend of our results is similar to those of Johnson and Christy's. The transmittance curves of titanium layers prepared with different deposition angles are shown in Fig. 4. Johnson and Christy's results are also included for comparison. As it can be seen, the general trend of our results is the same as Johnson and Christy's results. With increasing deposition angle transmittance increases. This increase is because of the formation of more fraction of voids on the layers. In our results a high transmittance can be seen, because of the nanometer thickness (d = 21 nm) of the layers, which cannot be seen with naked eye. Reflectance and transmittance spectra of the layers are measured experimentally in visible light wave length range (400–800 nm). In Fig. 5 we can see the real part of refractive index (n). Johnson and Christy's results are also included for comparison.

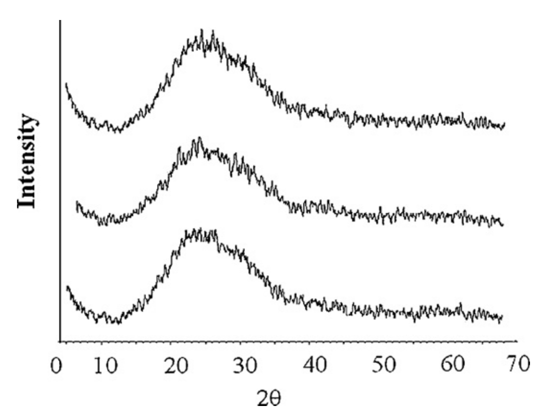

Fig. 2. XRD patterns of titanium/glass layers with different deposition angles.