INFLUENCE OF SPUTTERING POWER ON PROPERTIES OF TITANIUM THIN FILMS DEPOSITED BY RF MAGNETRON SPUTTERING

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ABSTRACT

Titanium (Ti) thin films were deposited on glass substrates by radio-frequency (RF) magnetron sputtering under various sputtering power (75-150W) at a relatively low temperature (200 ºC). The influence of sputtering power on the structural, optical and electrical resistivity properties of the films were studied. X-ray diffraction (XRD), field emission-scanning electron microscopy (FE-SEM), atomic force microscopy (AFM), UV-Vis-NIR spectrophotometer and four-point probe system were employed to characterize the deposited films. XRD results exhibited only a single prominent peak corresponding to Ti (002) orientation of hexagonal close-packed (hcp) structure. Ti thin film deposited under sputtering power of 75W has amorphous nature. As the crystallinity of the Ti films increased with sputtering power, the grain size and surface roughness of Ti films increased, however, a decrease in optical transmittance and electrical resistivity were found. Moreover, the film deposited under 120W sputtering power demonstrated the highest optical reflectance in the visible and near infrared wavelength regions.

Keywords: titanium, thin films, RF magnetron sputtering, surface topography, transmittance, electrical resistivity.

INTRODUCTION

Titanium (Ti) has been widely used in several applications such as aerospace, motor science, chemical engineering, microelectronics, biomedical devices and functional thin layers in semiconductor devices, due to its good mechanical properties, for examples high specific strength, low resistivity and remarkable resistance to corrosion (Benard *et al.* 1998), (Hyun *et al.* 2003), (Resnik *et al.* 2007).

In thin film synthesis, there are a number of different techniques that can be used to grow Ti thin films such as electron beam evaporation (Cai *et al.* 2005), (AkgÜL, 2014), chemical vapor deposition (Ahn *et al.* 2003), (Hofmann *et al.* 2003), template stripping (Vasilescu *et al.* 2000) and magnetron sputtering (Arnell and Kelly, 1999), (Kelly and Arnell, 2000), (Savaloni *et al.* 2004), (Musil *et al.* 2005). In (Vossen, 1976), the authors had pointed out that magnetron sputtering techniques are more preferable because it can be performed under lower temperature than the other techniques as also discussed in (Chen *et al.* 2011) that industries require lower processing temperatures in device and product manufacturing. Magnetron sputtering techniques are also most extensively used because of the ability to control the sputtering conditions and produce very high purity films (Meng and dos Santos, 1993) (Einollahzadeh-Samadi and Dariani, 2013). Furthermore, magnetron sputtering techniques are considered as clean environmental friendly techniques as the whole process is performed in a closed system (Navinšek *et al.* 1999). As a result, these techniques have been applied in several applications including coatings with specific properties (Rossnagel, 1999). Magnetron sputtering techniques include direct-current (DC) magnetron sputtering and radio-frequency (RF) magnetron sputtering have been employed to customize films characteristics and study the effect of deposition parameters such as substrate temperature, sputtering power, thickness and gas flow rate. Several studies have been done to understand the influence of deposition parameters on the characteristics of magnetron sputtered Ti thin films (Henderson *et al.* 2003), (Jeyachandran *et al.* 2006), (Jeyachandran *et al.* 2007), (Chawla *et al.* 2008), (Jin *et al.* 2009), (Einollahzadeh-Samadi and Dariani, 2013). For example, (Martin *et al.* 1998) studied the influence of bias power on some properties of Ti films. Effect of cathode power, sputtering pressure and base vacuum conditions on properties of Ti thin films were also studied (Jeyachandran *et al.* 2006). (Jeyachandran *et al.* 2007) also researched the influence of film thickness on the electrical, structural, optical and surface properties of sputtered Ti films. In a paper of (Jin *et al.* 2009), the authors investigated the effect of sputtering power on surface topography of DC magnetron sputtered Ti films. In (Chawla *et al.* 2009), the authors reported the effect of DC magnetron sputtering power (50W, 100W and 150W) on structural properties of the deposited Ti thin films. (Einollahzadeh-Samadi and Dariani, 2013) researched the effect of substrate temperature and deposition rate on the morphology and optical properties of deposited Ti films by DC magnetron sputtering process.

There are some literatures studied the influence of deposition parameters on structure, morphology and optical properties of Ti films using DC magnetron sputtering. However, only a few studies reported on RF magnetron sputtering.

In this paper, we report the structural, optical and electrical properties of RF magnetron sputtered Ti films under different sputtering power (75W – 150W) at 200 ºC.

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EXPERIMENTAL DETAILS

Film Preparation

Ti thin films were deposited on cleaned glass substrates by using a RF magnetron sputtering system with a 76 mm diameter x 5 mm thick Ti target of 99% purity. Before deposition, the glass substrates were cleaned in an ultrasonic bath with ethanol for 15 mins, rinsed in distilled water, dried with nitrogen gas of 99.9% purity and clamped on the substrate holder in the chamber. The sputtering chamber was evacuated to 10-6 Torr by using a turbo molecular pump. During deposition, the working pressure and substrate temperature were kept constant at 5.4 x 10-3 Torr and 200 ºC, respectively. Commercial argon (Ar) of 99.9% purity was used as the sputtering gas and kept constant at gas flow rate of 20 sccm that can be controlled by a mass flow controller. Ti films with different sputtering power (75W, 100W, 120W and 150W) were deposited after pre-sputtering the Ti target in an Ar atmosphere for 10 min in order to remove oxide layers. For uniform film thickness, all substrates were rotated at 10 rpm. The film thickness during deposition was monitored with a quartz thickness meter supplied by INFICON. The film thickness was kept at 50 nm.

Film Characterization

The structural properties of deposited Ti films were characterized by using X-ray diffractometer (XRD), field emission-scanning electron microscope (FE-SEM) and atomic force microscope (AFM). The X-ray diffraction of the films were obtained using a Shimadzu X-Ray Diffractometer 7000 with a monochromatic high-intensity CuKα radiation (λ = 1.54056Å) radiation operated at 40kV and 30mA. The patterns were recorded at a scanning rate of 2º/min in the angular (2Θ) ranges from 30º to 80º with an incident angle at 1.0º. The surface topography of the films were characterized by a commercially available AFM system from CSM instruments. The measurements were performed in contact tapping mode with a constant load force on the cantilever of 2.0 nN, and hence a constant force was applied on the samples. The FE-SEM images of the films were studied by using a JEOL JSM-7610F field emission scanning electron microscope and taken at different acceleration voltages.
The optical properties of the films were measured by using a CARY 5000 UV-Vis-NIR spectrophotometer in wavelength range of 300nm to 2500nm for optical transmittance and 300nm to 800nm for optical reflectance. The scanning rate was 240nm/min.

Electrical resistivity measurement were performed with a four-point probe system (Scientific Equipment Roorkee, India) at room temperature with a constant applied current of 8.15 mA. Four probes were placed in contact with the films and lied in a straight line with equal spacings. In order to reduce anisotropy defect, the measurements were carried out by taking the current and voltage values along the sample length, normal to the length and in hexagonal directions.

RESULTS AND DISCUSSION

Structural Properties

Figure-1 presents the XRD diffraction patterns of the deposited Ti thin films. The film deposited at sputtering power of 75W exhibited amorphous structure as shown in Figure-1(a). As the sputtering power increased at 100W and 120W, the crystallinity in the films increased. In the 2Θ range under investigation, only single prominent peak can be observed that corresponding to the (002) orientation of hexagonal close-packed (hcp) structure. These results also reported by (Jeyachandran *et al.* 2006), the crystallinity in the films increased with increase in the sputtering power, i.e. at low sputtering power, the films were amorphous and at high sputtering power, crystalline films were observed. However, in this work, as the sputtering power increased to 150W, the peak broadened and weakened. This might be due to a change in its phase or crystal structure as deposition rate varied. The similar phenomenon was also observed by (Hofmann *et al.* 2003), in which the change in the structural property of Ti films was due to the deposition rate.

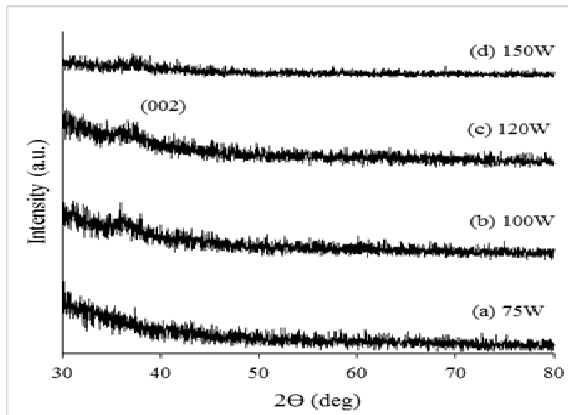

Figure-1. X-ray diffraction patterns of the titanium films deposited at different sputtering power, i.e. (a) 75W, (b) 100W, (c) 120W and (d) 150W.

Figure-2 shows the three-dimensional (3D) AFM images of Ti thin films deposited with different sputtering power. The images were acquired in a 2.0 µm x 2.0 µm area. To study the surface topographies of the Ti films from the AFM images, it is assumed that the dark regions represent areas with zero or near zero height value along the Z axle (positive direction) and the bright regions represent higher areas i.e. the top of bulging grains. Figure-2(a), (b) and (c) show that the grain size and surface roughness of Ti films increased with increase in sputtering power. Particularly, the change of grain microstructure from amorphous to crystalline state can be observed as the sputtering power increased. In (Chawla *et al.* 2009), the authors also observed that the increase in sputtering power contributes in an increase of the adatom mobility and the deposition rate, resulting in the growth of crystallite size and higher surface roughness of Ti thin films. The surface roughness of the deposited Ti films was

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detected to be in the range of 3.11-5.11 nm with increase in sputtering power. Higher surface roughness was resulted due to the formation of deep channels and large bulging grains on the film surface as seen in the Figure-2(c). However, the surface roughness reduced to 4.03 nm for deposited film with sputtering power of 150W as shown in Figure-2(d), which contributes to the smooth film surface. The similar behavior was observed in (Jin *et al.* 2009), in which the decrease in the film roughness was due to the larger hexagonal grains obtained during the transformation of crystal structure.

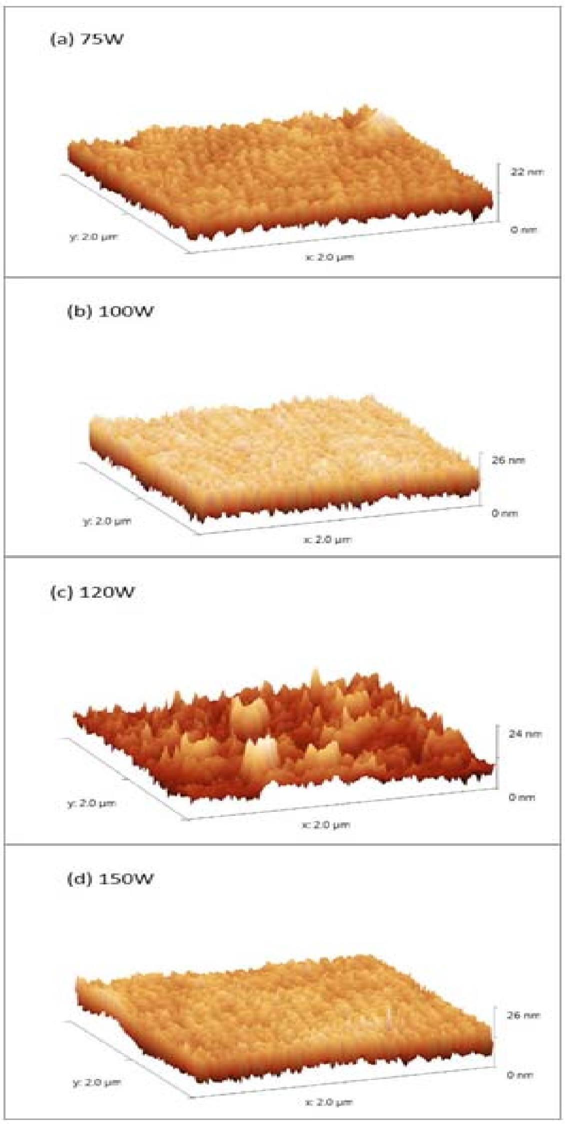

Figure-2. AFM 3D-images of Ti thin films on glass substrates as a function of sputtering power at (a) 75W, (b) 100W, (c) 120W and (d) 150W, in 2μm x 2μm surface area.

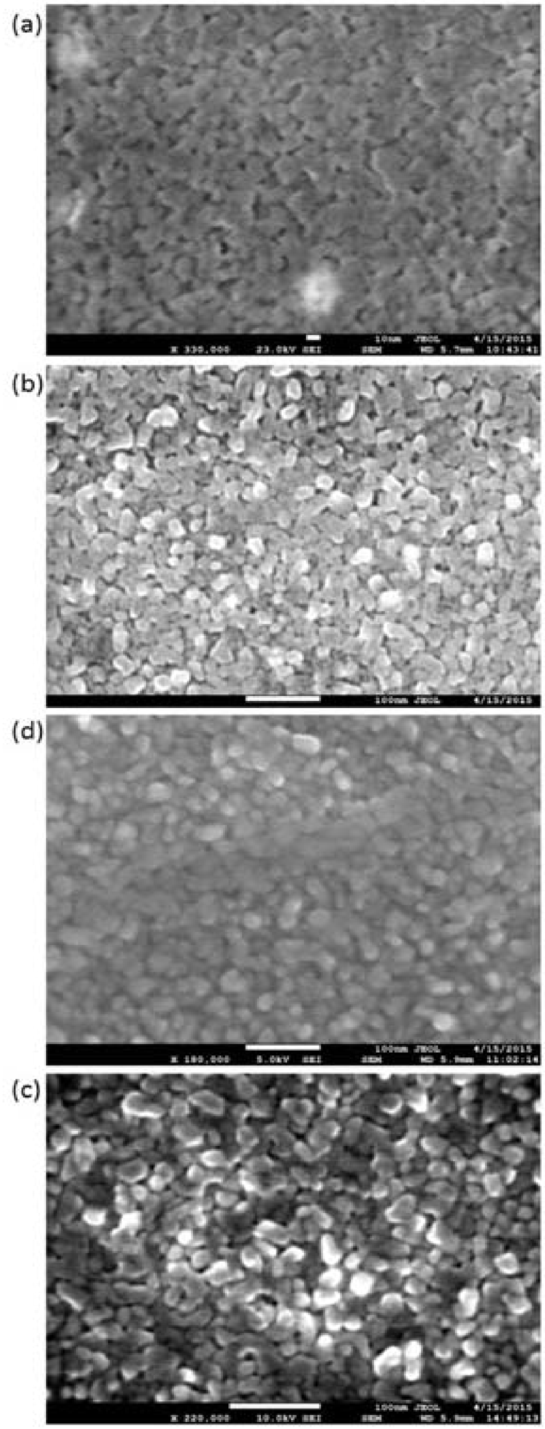

Figure-3. FE-SEM images of titanium films deposited at sputtering power of (a) 75W, (b) 100W, (c) 120W and (d) 150W, with different acceleration voltages, i.e. 23.0kV, 17.0kV, 10.0kV and 5.0kV, respectively.

The FE-SEM photographs of Ti thin films with various sputtering power are shown in Figure-3. These images were taken at different acceleration voltages, hence giving different signal, in order to obtain good resolution

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of the grains. Figure-3(a) was obtained at 10nm magnification for Ti thin film deposited with sputtering power of 75W, showing the amorphous nature of the film. Figure-3(b) and (c) show good crystallite resolution at 100nm magnification. The crystallite dimension increased with sputtering power from 100W to 120W as also observed in AFM measurements. Grain growth can also be seen from Figure-3(d) which refers to Ti film deposited with sputtering power of 150W. According to (Lu *et al.* 2001), at high sputtering power, the Ti atoms are more energetic to reach the substrates resulting in the formation of nuclei. Hence, more nuclei present on the substrate, suggests more sites for grain growth. The average grain size in the deposited Ti films was estimated to be 23.5 nm, 33.0 nm, 38.6 nm, 44.3 nm with increase in sputtering power 75W, 100W, 120W, 150W, respectively.

Optical Properties

The Figure-4 shows the optical transmittance of titanium films deposited with sputtering power of (a) 75W, (b) 100W, (c) 120W and (d) 150W. From these four samples, the film deposited with sputtering power of 75W exhibited high transmittance in the visible and near infrared wavelength regions, which related to its amorphous nature. As studied by (Meng and dos Santos, 1993), porous surfaces may have voids which consent more light to transmit, thus resulting in higher optical transmittance. As the crystallinity of the titanium films increased with sputtering power from 75W to 120W, the transmittance decreased to lower than 10%. The film deposited with sputtering power of 150W showed the lowest optical transmittance. In (Schiller *et al.* 1981), the authors reported that the decrease in the transmittance spectra was caused by the increase in the loss of light-scattering as the grain size increased.

Furthermore, the optical reflectance of the deposited Ti films can be observed in the Figure-5. These spectrums were taken by using specular reflectance which indicates high surface roughness that contributes to high optical reflectance on the surface of the films. The highest optical reflectance in the visible and near infrared wavelength regions was obtained for the film deposited with sputtering power of 120W which has high surface roughness as observed in Figure-2(c) and Figure-3(c). The reflectance in the near infrared region, i.e. at 800nm, decreases slightly from 44.3%, 44.2%, 43.0% and 38.1% for the deposited films with sputtering power of 120W, 150W, 100W and 75W, respectively.

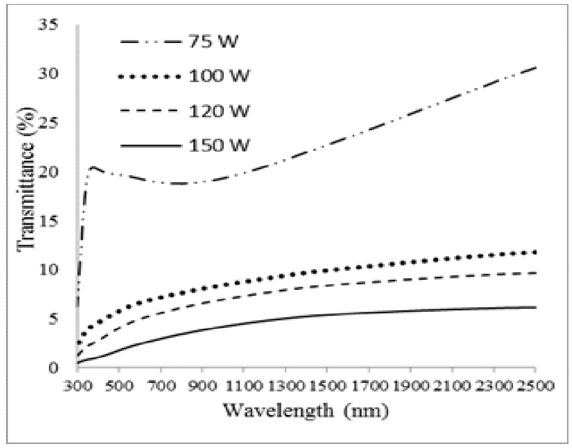

Figure-4. Optical Transmittance of the titanium thin films deposited at different sputtering power versus wavelength ranging from 300 nm to 2500 nm.

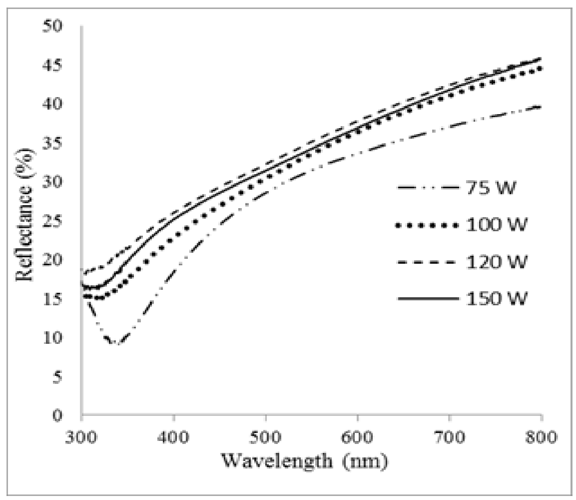

Figure-5. Optical Reflectance of titanium thin films deposited at different sputtering power versus wavelength ranging from 300 nm to 800 nm.

Electrical Property

The electrical resistivity of the deposited Ti films as a function of sputtering power was shown in Fig. 6. The room temperature resistivity of the films decreased with increase in sputtering power. This suggests that the atomic mobility on the growing surface increased (Lu *et al.* 2001), (Andújar *et al.* 2002) and there is a change in metallic characteristics of the Ti films (Jeyachandran *et al.* 2006). In other words, as the sputtering power increasing, the atoms have larger driving force to move to appropriate lattice sites and become more perfect crystals (Lu *et al.* 2001). Therefore, as the films have more crystalline, the resistivity of the films is lower.

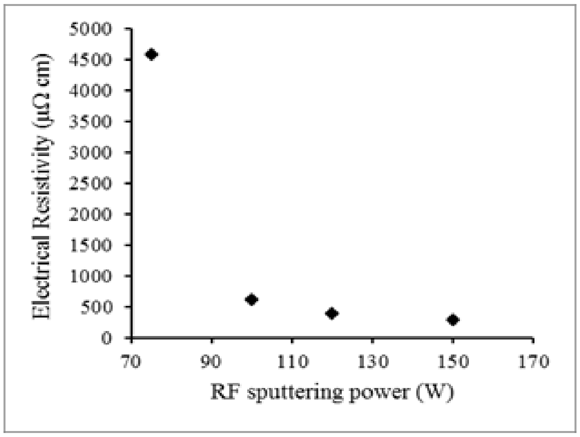

Figure-6. Electrical resistivity of the Ti thin films with respect to various sputtering power of 75W, 100W, 120W and 150W.

CONCLUSIONS

In this paper, we have done several studies on the structural, optical and electrical properties of Ti thin films prepared by using RF magnetron sputtering under sputtering power of 75W to 150W at relatively low temperature (200 ºC). XRD results showed only single prominent peak corresponding to Ti (002) orientation of hexagonal close-packed (hcp) structure. Additionally, the film deposited at sputtering power of 75W exhibited amorphous nature. As the sputtering power increased, the crystallinity, the grain size and the surface roughness of Ti films was found to be increased. However, the surface roughness reduced for deposited Ti thin films with sputtering power of 150W. Moreover, as the crystallinity of the titanium films increased with sputtering power from 75W to 120W, the optical transmittance decreased. The optical reflectance spectrums were taken by using specular reflectance. The highest optical reflectance in the visible and near infrared wavelength regions was obtained for the film deposited with sputtering power of 120W due to its high surface roughness. The significant changes in structural properties with increasing sputtering power lead to the growth of atomic mobility, therefore, the electrical resistivity of the Ti films decreased.

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