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PHYSICAL PROPERTIES OF TITANIUM NITRIDE THIN FILM PREPARED BY DC MAGNETRON SPUTTERING

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Abstract. The physical properties of the titanium nitride thin film have been prepared on p-type silicon (100) substrates by at room temperature by reactive DC magnetron sputtering technique using pure Si target with varying oxygen partial pressure during growth at reported. The oxygen partial pressure in the growth chamber is varied between (97 % argon) and (3 % oxygen). The X ray diffraction (XRD) analysis showed that all the films were polycrystalline.

1. Introduction

Titanium nitride (TiN) has been widely used as a coating material, ranging from diffusion barrier in microelectronic industry, to the hard and protective coatings on mechanical tools and decorative coatings [1, 2]. Because of the well-established application history, TiN thin films are good choices to meet the requirements. To improve the characteristics of TiN thin film, many studies have been presented to understand the relationships between the processing parameters, the film structure and properties [3, 4]. The previous research shows that the mechanical properties of TiN film are strongly affected by its preferred orientation, residual stress, packing factor, and grain size. Also the resistivity of TiN thin films is correlated to the packing factor [5]. DC magnetron sputtering is presently used for the deposition of a wide variety of thin films, especially oxides, nitrides and carbides [6-8]. The presented models do not include the effect of different reactivities of the reactive sputter process [9]. DC magnetron sputtering from elemental targets is a suitable technique for the preparation of compounds. In the past decade, DC magnetron sputtering technique has been used especially for hard coating.Presently, semiconducting nitrides (GaN, InN) are very important. DC magnetron sputtering is a cheap and well developed deposition technique that should be applied also for the preparation of these nitrides. The reactive sputtering of nitrides, especially that of the refractory nitrides (TiN, TaN, etc.), has been investigated greatly over the past years. Hofmann was the first to make a systematic study formation of nitrides by sputtering titanium targets in Ar/N2 mixtures [10]. In his model he discovered a parameter that took into account the different reactivities of metals to nitrogen. But he could not find a relationship between the heat of formation of the nitrides and the discharge characteristics. Mientus and Ellmer have investigated the changes of nitrogen partial pressure for formation of the nitrides and they discovered that both the discharge voltage and the deposition rate show a significant change when the nitrogen partial pressure increased. The discharge voltage increases or decreases depending on a decreased or increased secondary electron emission coefficient of the nitride target surface. For Ti and TiN, the discharge voltage increases by about 20–30 % when nitrogen partial pressure increased from zero to 3.7×10^{-3} Torr. In

addition, in this paper it was concluded that deposition rate decreases when nitrogen partial pressure increases [11].

TiN thin films were deposited by DC magnetron sputtering method in Ar + N2 atmosphere. The reactive sputter gas was a mixture of Ar (99.999 %) and N2 (99.999 %) with the ratio Ar (97 %) and N2 (3 %) by volume and effect of temperature on the structural properties of the films was investigated. Topography and atomic structure were investigated by atomic force microscopy (AFM) and X-ray diffraction (XRD), respectively. XRD showed that TiN nanocrystal is formed on these films. Average particle size nanoparticles were estimated by Scherrer formula, also we were compared with AFM images.

2. Experiment

The titanium nitride thin film is deposited on p-type silicon (100) substrates by reactive DC magnetron sputtering technique. This system is consisted of planer Ti metal (99.99 % pure) with 5 cm diameter. The distance between wafer and target was 3 cm. The growth chamber is initially evacuated up to a pressure of 1×10^{-5} mbar by a rotary pump (ALCATEL) and defusing pumps. Then Gas mixture of pure 97 % Argon and 3 % Nitrogen is introduced into the chamber. Pressure of sputter chamber in during growth for all samples was constant 2×10^{-2} Torr, and density current is 10 mA/cm^2. Deposition process has been done in 3 states with different substrate temperature(300 °, 400 °, 500 °C) respectively, and deposition time for all samples is 120 min. The crystal structure of the films was characterized using STOE SIADI MP X-ray Diffractometer. The Cu Ka line at 1.5405 Å was used as the source of X-ray radiation. Morphology and roughness of the surface film is measured by the Park Scientific scanning probe microscopy (SPM) system and for the calculated of grain size was used WSXM 4 software.

3. Results and discussion

Figure shows AFM images and topography curve of the titanium nitride thin films. The topography curve shows the distribution of nanoparticle size. The maximum of curve shows the nonoparticle average radius and width of the curve shows the variance of radius size. Figure 2 shows the abundance diagram as a function of surface topography variation. This information derivation of AFM analysis .The small width of the Gaussian diagram shows uniformity of grain size in surface. But in nitride sample prepared at 500 ° width of the Gaussian diagram increased and shows variation of surface morphology was increased. This proposition shows in 3D images too. The width of Gaussian diagram gives the grain size of thin films 200 nm.

Figure 3 gives the roughness of films RMS (root-mean-square). The calculate result of Fig. 2 gives with increases substrate temperature variation of topography and surface roughness reason agglomeration of particle [12]. Figure 4 presents the X-ray diffraction result on the titanium nitride thin films for different substrate temperature. As may be seen, films with grains preferentially oriented along (101), (200) between 43 and 44 degree. With increases of substrate temperature, intensity of (200) peak decreases and at 500 ° deletion and single crystal Ti_2N (tetragonal) extended. X-ray diffraction peaks shift towards the standard samples (No stress) show the kind of pressure and tensile stress in the crystal lattice structure of the titanium nitride. The more unstable the phase transition temperature in the titanium substrate and the heat is due to the disappearance. According to Scherrer's formula, the diameter of the crystalline grain:

$$d = (0.9\lambda)/(\beta\cos\theta),$$

where λ is the incident wavelength (for Cu($K\alpha$) = 1.54056), β is full width at half maximum (FWHM) of intensity, and θ is the Bragg angle.

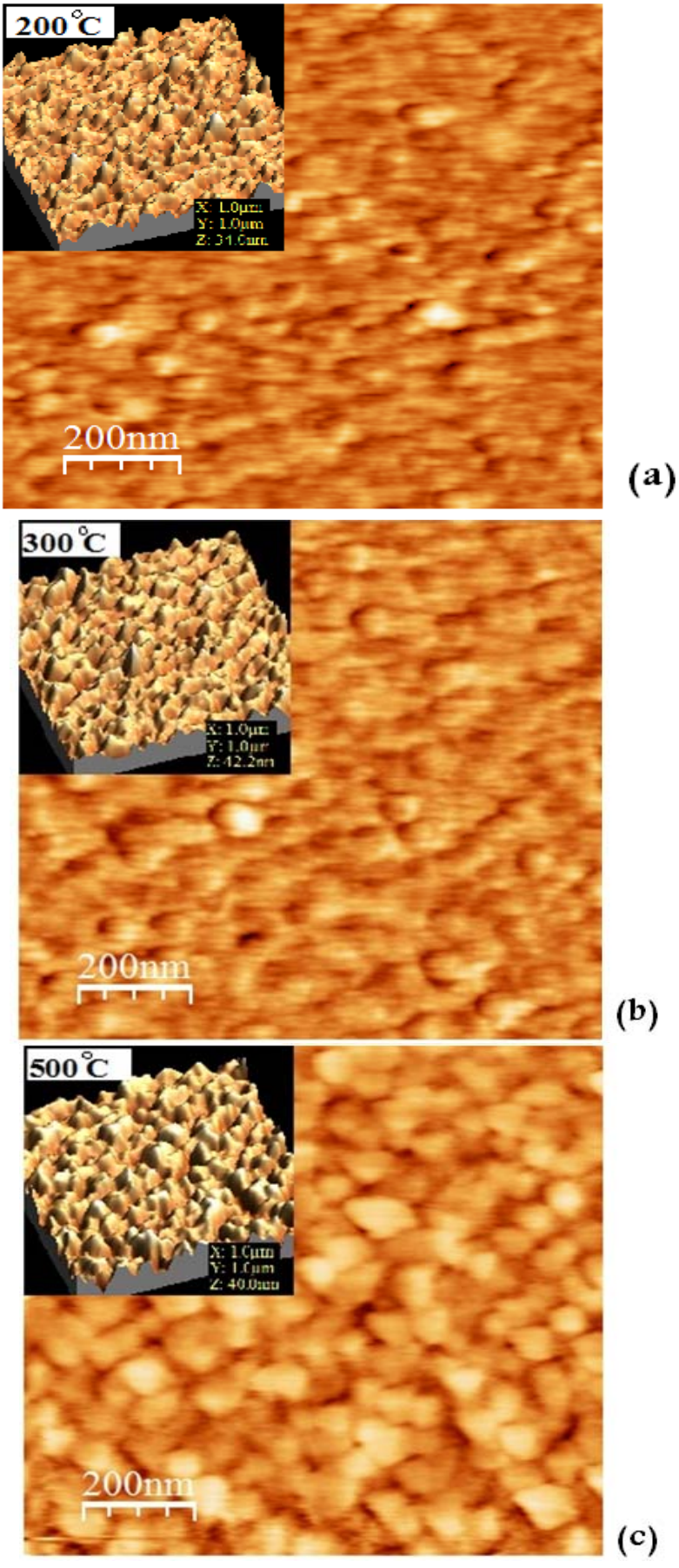

Fig. 1. 2D and 3D AFM images of grown layers: a) 200 ºC, b) 300 ºC, and c) 500 ºC.

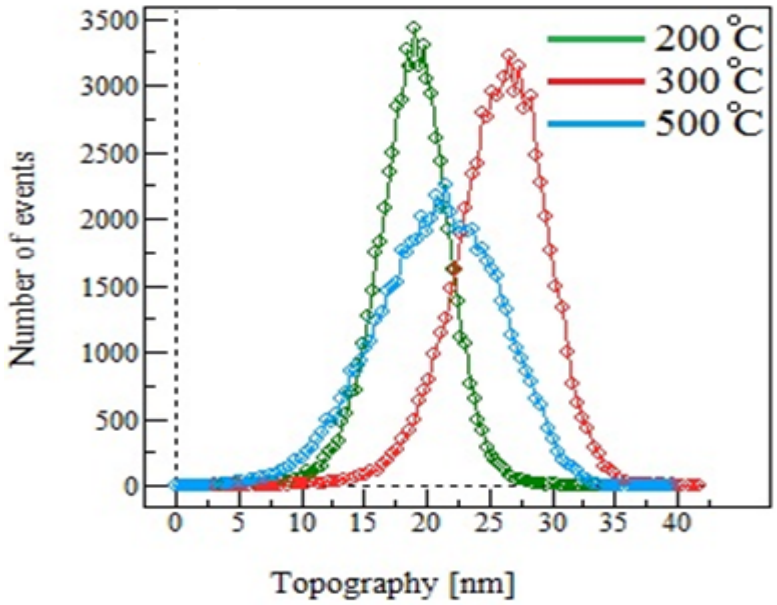

Fig. 2. The abundance diagram as a function of surface topography variation.

Table 1 shows characteristics and size of nano particles using Debye - Scherrer. This result gives with increases of substrate temperature more particles in (101) phase hang together, grain size increased and result of this subject the peak of XRD pattern become sharp. So with contraction of nano particle and create of major particle the grain size was increased and surface roughness and e distance proportion of peak and valley increased. Collation of grain size calculated by scherrer's formula and AFM images was accordant. The XRD spectrum shows variation of substrate temperature influence at intensity of peak and orientation of layers.

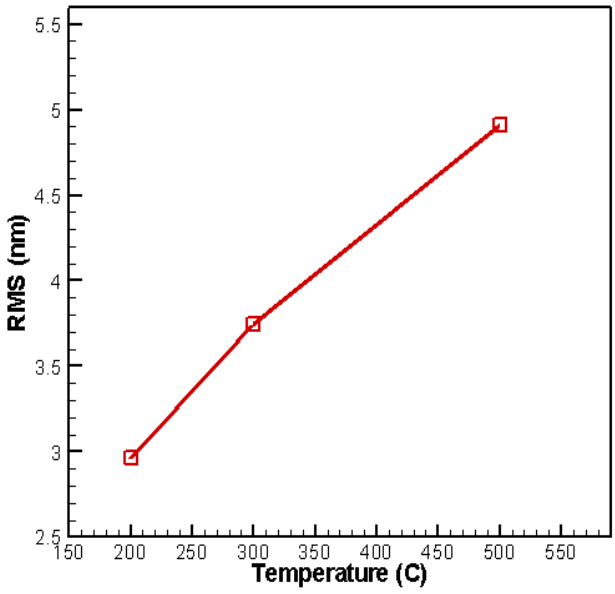

Fig. 3. The roughness of films RMS (root-mean-square) according to temperature.

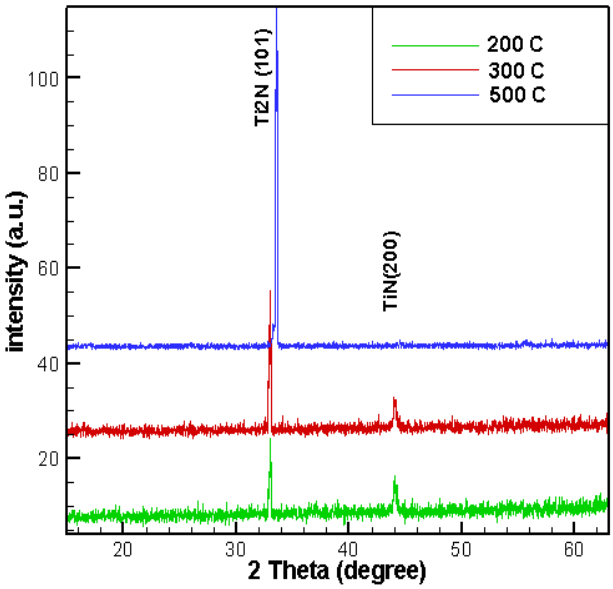

Fig. 4. The X-ray diffraction result on the titanium nitride thin films for different substrate temperatures.

Table 1. Characteristics and size of nanoparticles using Debye - Scherrer.

No	FWHM[°2T](101)	D, nm	FWHM[°2T](200)	D, nm
A200	0.2363	35.1	0.3840	22.3
A300	0.21	39.5	0.3360	25.5
A500	0.14	59.3	----	----

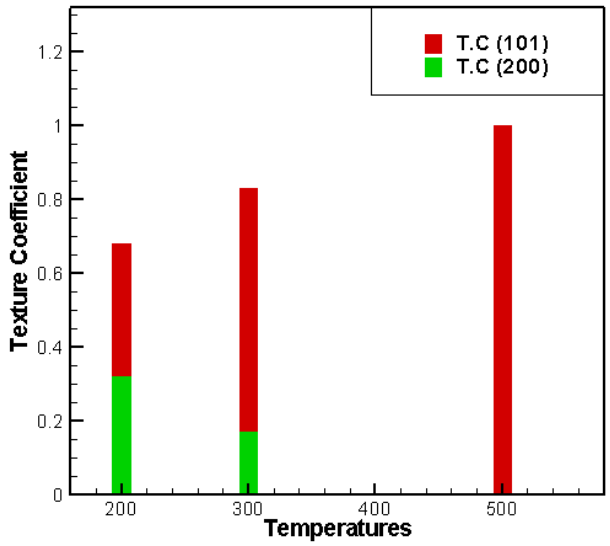

Fig. 5. The tissue coefficient of films.

Figure 5 shows the tissue coefficient of films. Tissue coefficient was respective intensity of XRD pattern peak for orientation of different layers and calculated from:

$$\mathrm{T.C}_{(hkl)} = \frac{I_{(hkl)}}{I_{(101)} + I_{(200)}}.$$

4. Conclusions

Titanium nitride thin films have been prepared at different temperature of substrate by reactive DC magnetron sputtering. Crystal structure and surface morphology of the thin films were evaluated by XRD and AFM analysis. With increased substrate temperature and lattice structure the diffraction peak and crystal structure were translated to single crystal of Ti_2N (phase (101) and tetragonal). AFM images show the creation of nanostructure thin films and with increasing substrate temperature variation of surface topography and increasing of surface roughness. Grain size was evaluated by scherrer's formula and XRD patterns and concord with calculated grain size of AFM images.

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