

# ULTRATHIN TaN<sub>x</sub> FILMS PREPARED BY DC MAGNETRON SPUTTERING

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**Abstract.** Ultrathin Tantalum nitride (Ta<sub>N<sub>x</sub></sub>) films were prepared by dc magnetron sputtering on glass substrates at different nitrogen partial pressures. The crystal structure, compositional, microstructure, surface morphology, and optical properties were investigated. The deposition rate of the films was strongly influenced by the nitrogen partial pressure and it was continuously decreased from 3.1 to 1.8nm/min with increase of nitrogen partial pressure from 1x10<sup>-2</sup>Pa to 8x10<sup>-2</sup>Pa. The films show very fine grains and homogenous microstructure when the films deposited at nitrogen partial pressure of 4x10<sup>-2</sup>Pa. The optical transmittance of the films increased from 83 to 91% in the visible region by increasing of nitrogen partial pressure from 1x10<sup>-2</sup> to 4x10<sup>-2</sup>Pa.

**Keywords:** Tantalum nitride, ultrathin films, Sputtering, Nitrogen partial pressure

## I. INTRODUCTION

Tantalum nitride (Ta<sub>N<sub>x</sub></sub>) has become a promising candidate material in many applications such as structural elements in integrated circuits, film resistors, diffusion barrier, anti-corrosive layers, high-speed thermal printing head due to its unique properties like high corrosive resistivity, good optical properties, good chemical and thermal stability, high hardness, high melting point, high electrical and thermal conductivity [1-6]. The Ta<sub>N<sub>x</sub></sub> films were prepared by various thin films deposition techniques such as atomic layer deposition, cathodic vacuum arc deposition and sputtering etc. [7-9]. Among these techniques, sputtering is one of best techniques to prepare stoichiometry films with uniform thickness and good adhesion to substrates. The properties of Ta<sub>N<sub>x</sub></sub> films are strongly depends on the deposition parameters. In the present work ultrathin Ta<sub>N<sub>x</sub></sub> films were prepared on glass substrates under different nitrogen partial pressure using dc magnetron sputtering technique and studied their structural, compositional, microstructural, surface morphology and optical properties.

## II. EXPERIMENTAL

Ultrathin Ta<sub>N<sub>x</sub></sub> films were deposited on well cleaned glass substrates using dc magnetron sputtering. Prior to the deposition, the process chamber was evacuated until the base pressure reached under 7.8x10<sup>-4</sup>Pa. The pure metallic tantalum (99.99%) was used as target. The pure nitrogen and argon were used as reactive and sputtering gases, respectively. The flow rate of argon and nitrogen were controlled by mass flow controllers. Before deposition of each film the tantalum (Ta) target was pre-sputtered in pure argon atmosphere for 20min in order to remove oxide layers formed if any on the target. A shutter was incorporated below the sputtering target to isolate the substrate during the pre-sputtering process. This is essential in the reactive sputtering to obtain the stoichiometry films with reproducible properties. Ultrathin Ta<sub>N<sub>x</sub></sub> films were deposited at different nitrogen partial pressures of 1x10<sup>-2</sup>, 4x10<sup>-2</sup> and 8x10<sup>-2</sup>Pa by keeping the other deposition parameters such as substrate temperature(303K), sputtering power(100W) and sputtering pressure(2Pa) as constant. During the films deposition, the substrate holder was rotated at 15rpm to obtain homogeneous film thickness. The structural properties were characterized using X-ray diffraction (XRD). The microstructures and surface morphology were analyzed by field emission scanning electron microscope (FE-SEM) and atomic force microscope (AFM), respectively. X-ray photoelectron spectroscopy (XPS) was used to determine the chemical states of the films. The transmittance of the films was recorded by UV-Vis-NIR spectrometer. The films thickness was measured by Ellipsometer measurements and it was around 15nm. Target to substrate distance was about 80mm.

## III. RESULTS AND DISCUSSIONS

The deposition rate of the films was strongly influenced by the nitrogen partial pressure and it was continuously decreased from 2.1 to 1.2nm/min with increase of nitrogen partial pressure from 1x10<sup>-2</sup> to 8x10<sup>-2</sup>Pa. The decreasing of the deposition rate with increasing of nitrogen partial pressure is often observed in reactive sputtering of tantalum nitride films. At lower nitrogen partial pressures, the deposition rate was high due to the high sputtering yield of tantalum and insufficient availability of nitrogen for reaction with tantalum. The deposition rate of Ta<sub>N<sub>x</sub></sub> films was decreased with increasing the nitrogen partial pressure, due to the target-poisoning effect (i.e. formation of Ta<sub>N<sub>x</sub></sub> phase on the target surface) resulting low sputtering yield. Grosser et al. [10] observed the similar changes in deposition rate by increasing the nitrogen flow rate in dc magnetron sputtered Ta<sub>N<sub>x</sub></sub> films.

Fig.1. shows the XRD patterns of ultrathin Ta<sub>N<sub>x</sub></sub> films at different nitrogen partial pressures. The deposited films exhibited amorphous structure regardless of the nitrogen partial pressures. The films deposited on amorphous glass substrates should have sufficiently thick and there is much more matter which can diffract X-rays and the peak is more intense. In the case of ultrathin films

deposited on amorphous glass substrates, it diffracts less and the noise in the recorded spectra is so important that hides any form of growth orientation [11].

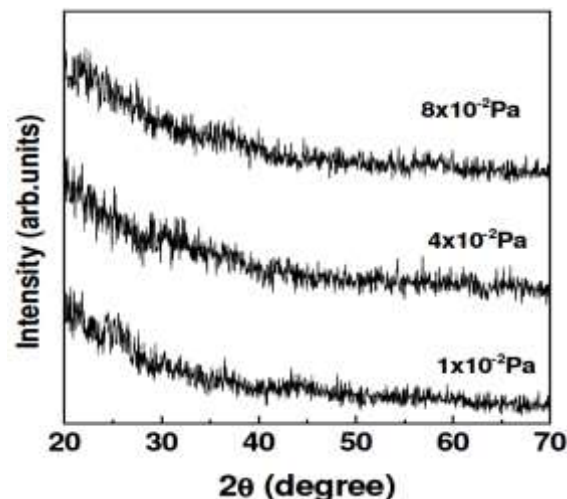


Fig.1. XRD patterns of ultrathin  $\text{TaN}_x$  films at different nitrogen partial pressures.

The XPS spectra of  $\text{TaN}_x$  films at different nitrogen partial pressures are shown Fig.2. The binding energy values of Ta4f in  $\text{TaN}_x$  films are 25.1eV, 26.2eV; 25.17eV, 27.28eV; and 25.09eV, 27.13eV for nitrogen partial pressure of  $1 \times 10^{-2}$ ,  $4 \times 10^{-2}$  and  $8 \times 10^{-2}$  Pa, respectively. The obtained binding energy values of  $\text{TaN}_x$  films are not coincide with Ta (Ta4f=21.9 eV),  $\text{TaN}_x$  (22.2 eV, 24.1 eV) and  $\text{TaO}_x$  (26.4 eV, 28.3 eV) values [12-13].

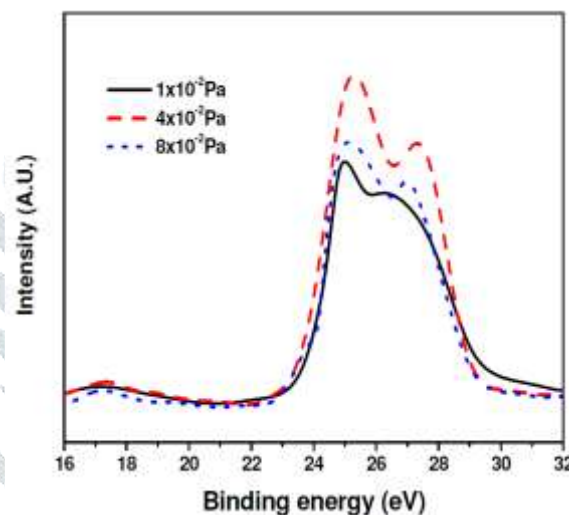


Fig.2. XPS spectra of ultrathin  $\text{TaN}_x$  films at different nitrogen partial pressures.

The microstructural changes of  $\text{TaN}_x$  films at various nitrogen partial pressures are shown in Fig.3. The microstructure of the films was highly influenced by nitrogen partial pressures. The films exhibited inhomogeneous microstructure at low nitrogen partial pressure of  $1 \times 10^{-2}$  Pa due to poor surface mobility of adatoms, consequently, agglomerated and the clusters are nucleated on the substrate. The films show very fine grains and homogenous microstructure when the films deposited at nitrogen partial pressure of  $4 \times 10^{-2}$  Pa. Beyond this nitrogen partial pressure, the surface of the films exhibited inhomogeneous microstructure, which is due to the excess nitrogen might induce defects in the films, and influenced the nucleation and growth of the films at higher nitrogen partial pressures.

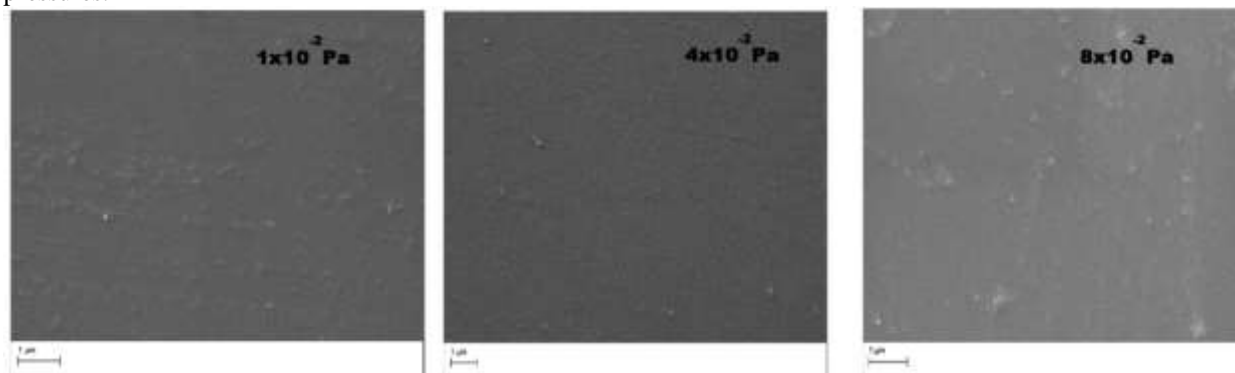


Fig.3. SEM images of ultrathin  $\text{TaN}_x$  films at different nitrogen partial pressures.

The two-dimensional AFM images of ultrathin TaN<sub>x</sub> films at different nitrogen partial pressures are shown in Fig.4. The films surface and RMS roughness was strongly influenced by the nitrogen partial pressures. The films become uniform by increasing of the nitrogen partial pressure from 1x10<sup>-2</sup> to 4x10<sup>-2</sup>Pa, thereafter small islands are formed on the surface of the films. The obtained RMS surface roughness values are 2.5, 0.7 and 1.6nm, for the nitrogen partial pressure of 1x10<sup>-2</sup>, 4x10<sup>-2</sup> and 8x10<sup>-2</sup>Pa, respectively. The RMS roughness decreases with increase of nitrogen partial pressure from 1x10<sup>-2</sup> to 4x10<sup>-2</sup>Pa is due to formation of very fine grains and absence of islands. The RMS roughness increased again at higher nitrogen partial pressure of 8x10<sup>-2</sup>Pa. The similar variation was observed by Koscielniak et al. [14] in rheotaxial growth vacuum oxidation ultrathin In<sub>2</sub>O<sub>3</sub> films.

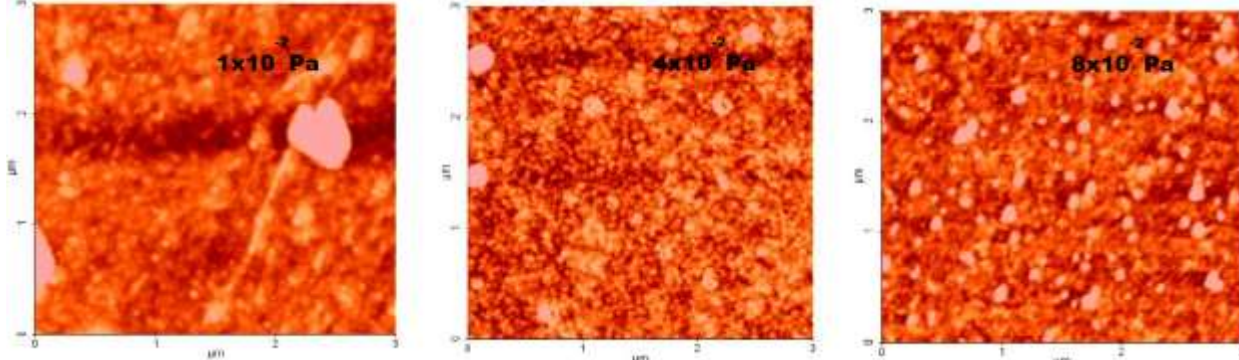


Fig.4. Two-dimensional AFM images of ultrathin TaN<sub>x</sub> films at different nitrogen partial pressures.

Fig.5. shows the variation of the optical transmittance of the ultrathin TaN<sub>x</sub> films with nitrogen partial pressures. The optical transmittance of bare glass was measured and it was around 92% at wavelength of 550nm. The optical transmittance of the films increased from 83 to 91% in the visible region by increasing of nitrogen partial pressure from 1x10<sup>-2</sup> to 4x10<sup>-2</sup>Pa. On further increasing the partial pressure the transmittance was decreased to 87%. The variations in the optical transmittance with nitrogen partial pressure is may due to the changes in surface roughness of the films. The optical band gap of the films was also influenced by the nitrogen partial pressure. The obtained band gap values are 2.52, 2.69, 2.77eV for nitrogen partial pressure of 1x10<sup>-2</sup>, 4x10<sup>-2</sup> and 8x10<sup>-2</sup>Pa, respectively. The present obtained values are close to the reported values of atomic layer deposition ultrathin TaN<sub>x</sub> films by Wu et al. [15]. The obtained values are not near to the band gap values of Ta<sub>2</sub>O<sub>5</sub> (E<sub>g</sub>=4.5eV), TaO<sub>2</sub> (E<sub>g</sub>=3.9-4.5eV), and Ta(NO) (E<sub>g</sub>=4.3eV) [16-18]. These results indicate that the films deposited at different nitrogen partial pressures were not contained oxygen.

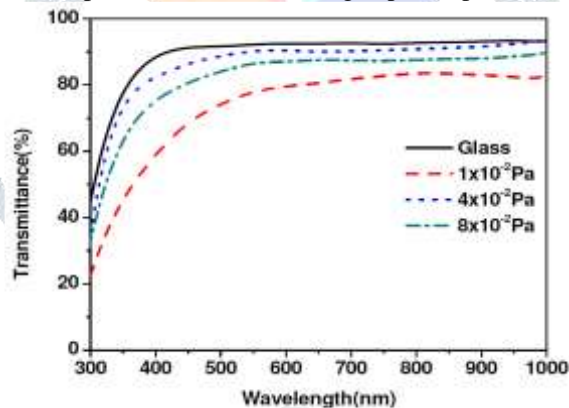


Fig.5. Optical transmittance spectra of ultrathin TaN<sub>x</sub> films at different nitrogen partial pressures.

#### IV. CONCLUSIONS

Ultrathin TaN<sub>x</sub> films were prepared by dc magnetron sputtering on glass substrates at different nitrogen partial pressures. The deposited films exhibited amorphous structure regardless of the nitrogen partial pressures. The film surface and RMS roughness was strongly influenced by the nitrogen partial pressures. The films become uniform by increasing of the nitrogen partial pressure. The films deposited at nitrogen partial pressure of 4x10<sup>-2</sup>Pa exhibited smooth surface with RMS surface roughness of 0.7nm. The optical transmittance of the films increased from 83 to 91% in the visible region by increasing of nitrogen partial pressure from 1x10<sup>-2</sup> to 4x10<sup>-2</sup>Pa. The optical band gap of the films was also influenced by the nitrogen partial pressures.

#### V. ACKNOWLEDGEMENT

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