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Effect of nitrogen flow ratio on nano-mechanical properties of tantalum nitride thin film

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ABSTRACT

In this research, nanostructured tantalum nitride (TaN) thin films were deposited on 316L stainless steel substrates by reactive magnetron sputtering. The effect of nitrogen flow ratio (N2/(Ar + N2)) on the structural, mechanical and surface properties of deposited films was studied by means of XRD, SEM, Nano-indentation and AFM. The results revealed a strong correlation between the crystal structure and properties of TaN film, where the deposited film transforms from hexagonal γ-Ta_2N to hexagonal ε-TaN and cubic TaN with increasing nitrogen flow ratio from 10% to 25%. In addition, it was found that increasing the nitrogen flow ratio will result in the target poisoning and lower the deposition rate, which causes the grains growth and roughening of the surface.

1. Introduction

Metal nitrides are of great interest for structural applications owing to their excellent properties [1–3]. In recent years, tantalum nitride is more considered among other metal nitrides due to its promising properties, such as good resistance to wear and oxidative corrosion, super hardness, high strength and toughness even at elevated temperature, high thermal stability and superior thermal conductivity [4–6]. These properties make this material attractive for various applications such as diffusion barrier, wear and corrosion-resistance material, high speed thermal printing head, stable resistor used in silicon-based integrated circuits and micro-electronics industries [7–10].

As tantalum is a refractory metal with high melting point, sputtering is an attractive method to deposit tantalum and tantalum nitride films, since it can be easily scaled up from laboratory sized targets to large-scale industrial applications [11,12]. Therefore, it is important to predict the effect of deposition process on the surface morphology and mechanical properties of deposited film.

Sputtering process is controlled by many parameters which may affect the properties of deposited film [13]. One of the most important parameters affecting the properties of produced film is the reactive gas imposed to the sputtering media which can produce a variety of compounds with stoichiometric and non-stoichiometric compositions. Tantalum nitride shows different stable: Ta(N), hexagonal γ-Ta_2N and hexagonal ε-TaN, and meta-stable phase structures such as F.C.C- δ-TaN and other nitrogen rich compounds such as Ta_5N_6, Ta_4N_5 and Ta_3N_5 with various properties [14,15]. On the other hand, phase and microstructure of the film are important factors by which friction and wear can be manipulated. Moreover, the thinner films are appropriate in improving the surface properties (such as hardness and scratch resistance of small devices) due to lower intrinsic stress [16]. Because of the complexity of structures and properties, this research focused on the effect of N_2 flow rate introduced to the system during sputter deposition of tantalum nitride thin films and investigation of subsequent structures and properties of produced films. In this regard, tantalum nitride thin films were deposited using reactive magnetron sputtering technique on the stainless steel substrate.

Mechanical properties of materials at micro and nano-scale are of interest due to their unique properties associated with the small volumes [17]. In this regard, the nano-mechanical properties of the films were measured using load-displacement curves provided by

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Table 1
Elastic-plastic work of deformation.

Specimen	N_2 flow rate (SCCM)	N_2/N_2+Ar (%)	Elastic work (pJ)	Plastic work (pJ)	Total work (pJ)	Plasticity (%)
TaN10	2	10	5.0	5.2	10.2	58
TaN15	3	15	4.3	6.8	11.1	58
TaN20	4	20	5.8	8.3	14.1	66
TaN25	5	25	5.1	13.9	19.0	80

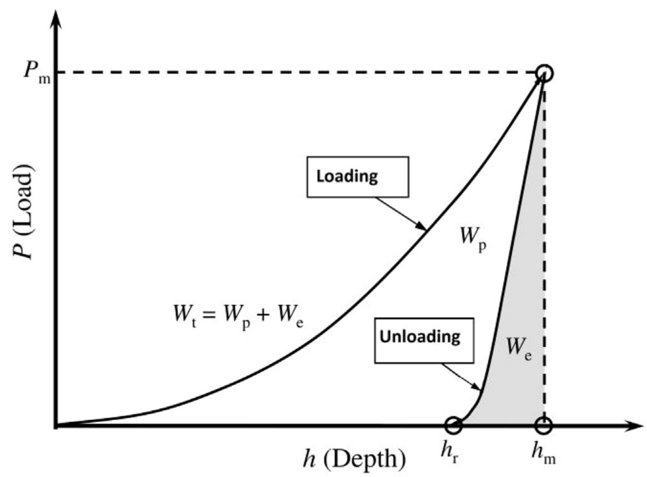

Fig. 1. Measuring the work of plastic (pl) and elastic (el) deformation using force-displacement curve [35].

nano-indentation method which is a common method for investigation of mechanical properties in sub-micron regime [18,19]. The surface morphology of the specimens was investigated using atomic force microscope. In addition, structural properties of the films were studied using X-ray diffraction (XRD) analysis and Scanning Electron Microscopy (SEM) micrographs.

2. Materials and methods

Thin tantalum nitride films were deposited by reactive magnetron sputtering system using 99.999% pure argon plasma with 99.999% pure nitrogen as reactive gas. A 50 mm diameter tantalum with 99.95% purity was used as sputtering target and an austenitic 316 stainless steel disk of 30 mm diameter was applied as substrate which was well polished and cleaned, each one for 15 min, in acetone and methanol ultrasonic bath. The nominal composition of 316L SS substrate in wt% was: Cr: 17.08; Ni: 13.23; Mo: 2.91; C: 0.03; Mn: 0.83; Si: 0.96; N: 0.08; P: 0.04; S: 0.01 and Fe: balance. All specimens were sputter deposited for 15 min in a constant power of 30 W. Target to substrate distance was set to be 10 cm. The base pressure of the chamber was approximately 7×10^{-6} torr and the working pressure was kept constant near 5×10^{-3} torr. In order to investigate the effect of nitrogen flow ratio, ($N_2/(Ar + N_2)$), different nitrogen flow ratios from 10% to 25% were applied which are listed in Table 1.

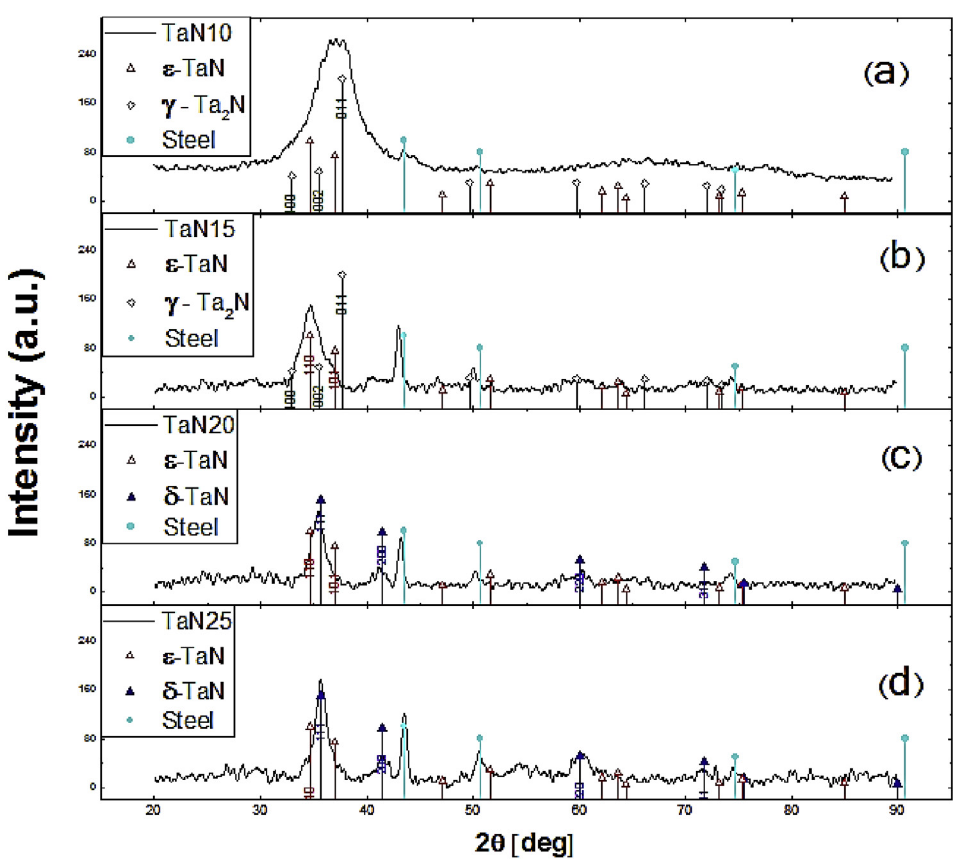

Fig. 2. XRD profiles of tantalum nitride films with different N_2 flow ratio: (a) 10% (b) 15% (c) 20% (d) 25%.