

On the dependence of the structural, electrical, mechanical, and tribological properties of Ta₂N thin films on film thickness: Application in microelectronic devices

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ABSTRACT: The mechanical and tribological properties play a vital role in the production of the microelectronic device because the long-term stability of the device depends on them. In the present research, the structural, electrical, mechanical, and tribological characteristics of Ta₂N thin films were studied for the development of microelectronic devices. The samples were grown by DC reactive magnetron sputtering technique on (400)-oriented Si and SiO₂/Si substrates at different thicknesses (80-200 nm). X-ray diffraction technique was used to identify the phase and crystallographic structure while the electrical behavior was investigated by a four-point probe instrument. Nanoindentation and scratch tests were also employed to study the mechanical and tribological characteristics, respectively. All samples represented decent irreversibility of resistivity. A decrease in the film thickness increased the resistivity and developed the nano-strain in the film structure. A decrease in film thickness also improved the hardness as well as resistance to wear and abrasion damage of the samples. The investigations showed that the mentioned behaviors were due to the reduction of crystallite size which in turn caused a denser structure.

Keywords: Hardness, Resistivity, Ta₂N, Thin film, Thickness.

INTRODUCTION

Transition metal nitrides (TMNs) have been the subject of various research projects worldwide due to their decent hardness, adherence, abrasion, and corrosion resistance [1-5]. They are also used as protective coatings with functional optical properties and in microelectronic devices [6]. Tantalum nitride is one of the TMNs that has been used effectively in the semiconductor industry because of its high melting point, good resistivity uniformity, low-temperature coefficient of

resistivity (TCR), low voltage coefficient of resistivity (VCR), and good thermal stability [7, 8]. Nevertheless, there are very few works on tantalum nitride thin films, presumably due to the high cost and more stringent deposition conditions, compared to other TMNs. Table 1 lists a summary of some researches about this material [2, 6, 9-13]. As can be seen, most of the reports investigated separately the electrical or mechanical properties of the samples as the main dependent variable. But, it is well-known that for the development of new materials

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as a microelectronic device, the combination of electrical and mechanical characteristics should be noticed. Mechanical properties play a vital role in the production of the microelectronic device because the stability of the device depends on them. Furthermore, internal stress in the film and insufficient adhesion to the substrate may lead to cracks. Therefore, for long-term reliability, a microelectronic device should present the desired electrical behavior with high hardness, decent elasticity, fine adhesion on Si, and low interface stress [14, 15].

Variations in stoichiometry are common in tantalum nitride thin films because of their structure [2]. Exper-

imentally, BCC-TaN_{0.05}, FCC-TaN, hexagonal-TaN, hexagonal-Ta₂N, tetragonal-Ta₄N₅, orthorhombic Ta₄N, hexagonal-Ta₅N₆, and orthorhombic-Ta₃N₅ have been synthesized by controlling growth methods and conditions [13, 16]. In the first section of the research, Ta_xN thin films were fabricated using DC reactive magnetron sputtering technique [17]. The phase and structure of the samples were tuned by the nitrogen/argon ratio [N₂/(N₂+Ar)] and deposition power. It was deduced that the tantalum nitride deposited at a nitrogen/argon ratio of 0.05 can be formed in the phase of Ta₂N with the hexagonal structure. The investigations showed that the mentioned phase with near-zero TCR,

Table 1. A summary of previously published works on Tantalum nitride thin films.

Deposition technique	Substrate	Independent Variable	Depended variable	Ref.
Reactive DC magnetron sputtering	SiO ₂ /Si	N ₂ partial pressure	Resistivity	[9]
			TCR*	
			Phase/structure	
Reactive DC magnetron sputtering	SiO ₂ /Si	Film thickness	Surface morphology	[10]
			Resistivity	
			TCR*	
Reactive RF magnetron sputtering	SiO ₂ /Si	N ₂ partial pressure	Phase/structure	[11]
			Surface morphology	
			Resistivity	
Reactive DC magnetron sputtering	Si(111)	N ₂ partial pressure	Resistivity	[12]
	Glass			
	Phase/structure			
Reactive RF magnetron sputtering	Si(111)	Sputtering power	Surface morphology	[2]
	AISI 420 steel			
	Microstructure			
DC reactive magnetron sputtering	SKD11 tool steel	N ₂ partial pressure	Hardness	[6]
			Wear resistance	
			Phase/structure	
Plasma assisted bias sputtering	Ti-6Al-4V	Ar/N ₂ flux ratios	Surface morphology	[13]
			Hardness	
			Wear resistance	
			Microstructure	
			Mechanical properties	

*) Temperature coefficient of resistance

as well as decent electrical, mechanical, and tribological characteristics can be introduced as a stable phase of tantalum nitride for the development of microelectronic devices compared with TaN and other phases. In the second section of our research (current work), based on the knowledge obtained from the previous work [17], we study the effect of film thickness on the structural, electrical, mechanical, and tribological properties of the Ta₂N thin films. It should be noted that the data of the sample with thickness of 140 nm is related to our previous work [17].

EXPERIMENTAL DETAILS

Film preparation

Ta₂N thin films were deposited by a DC reactive magnetron sputtering system on the unheated Si and SiO₂/Si substrates under different thicknesses (80, 140, and 200 nm). High purity circular Ta (99.98%) of 76 mm diameter and 1 mm thickness was used as the target while the target to substrate distance was 10 cm. Initially, the chamber was evacuated to a base pressure of 4×10⁻⁶ mbar by a turbo molecular pump and changed to 8×10⁻³ mbar during the deposition. The ratio of [N₂/(N₂+Ar)] and the flow rate of Ar+N₂ were 0.05 and 10 standard cubic centimeter per minute (sccm), respectively.

The sputtering power was also set at 150 W. Before the deposition, the target was sputter etched for 3 minutes with a shutter between the target and the substrate. Four samples were produced in each run. The samples deposited on Si substrate were used for structural, mechanical, and tribological tests while those deposited on SiO₂/Si substrate were used for electrical measurements.

As the substrate, n-type Si(400) wafers with the dimension of 20×20 mm² were ultrasonically cleaned in acetone and ethanol. For electrical investigations, the substrates were oxidized in a horizontal tube furnace (Exciton, 1200-30/6, T.H, Iran equipped to Shinko temperature programmable controller-PCD 33A) with an oxygen gas flow of 200 (sccm) at 1100 °C to obtain SiO₂ layer with a desired thickness [18,19]. The deposition rate and the thickness of samples were controlled using in-situ quartz crystal monitor.

Film characterization

The crystallographic structure of the Ta₂N samples was analyzed using a Philips XRD X'pert MPD Diffractometer (Cu Kα radiation) with a step size of 0.02° and a count time of 1 s per step. A four-point probe instrument was employed to measure the resistivity at room temperature. The mechanical and tribological tests were done by a Hysitron Inc. TriboScope Nano-mechanical Test Instrument with a 2D transducer, complete software, and Berkovich diamond indenter. An applied load of 600 μN with loading time, dwelling time, and unloading time of 30 s, 10 s, and 30 s was employed in the indentation test. To get accurate mean and standard deviation, all samples were tested over 5 times and averaged. The distance between two indentations was not less than three times of the minor diagonal to prevent stress-field effects from nearby indentations. Also, the scratch test parameter including force, scratch length, and scratch time were 600 μN, 4 μm, and 35 s, respectively.

RESULTS AND DISCUSSION

Crystallographic structure

Fig. 1 depicts the XRD patterns of the tantalum nitride thin films deposited under different thicknesses while the numerical data are summarized in Table 2. There is no significant variation in the crystallographic structure of the deposited samples under different thicknesses. Contradistinguished to JCPDS Card No.: 26-0985, two diffraction peaks are recognized for all samples attributed to hexagonal structure (Ta₂N (100) and Ta₂N (002)). The observed structure is in agreement with the previous reports that used the same nitrogen/argon content [2, 7, 11]. For a closer investigation of the crystallographic structure, full width at half maximum (FWHM), crystallite size (D), texture coefficient (TC), plan spacing (d), and nano strain (ε) are evaluated. The FWHM of the samples, presented in column 5 of Table 2, is used to calculate the crystallite size by the Scherrer formula [20]. It can be seen that an increase in film thickness causes the increasing of the crystallite size (column 6 of Table 2). This behavior agrees well with the Structural Zone Model (SZM) predictions proposed by Messier [21]. Polycrystalline thin films deposited on different substrates

generally show preferred orientation, depending on the film material as well as deposition methods and parameters [22]. To examine the preferred orientation of the samples the texture coefficient is calculated using the Harris method [23]:

$$TC_{(hkl)} = \frac{\frac{I_{(hkl)}}{I_{0(hkl)}}}{\left(\frac{1}{N}\right) \sum \frac{I_{(hkl)}}{I_{0(hkl)}}} \quad (1)$$

Where I is the measured intensity, I_0 is the standard intensity, and N is the number of the peak. The results are given in column 7 of Table 2, and indicate that the (002) plane can be introduced as a preferred crystal growth orientation of Ta_2N samples with an increase in thickness. The investigation of the diffraction line positions (column 3 of Table 2) shows a tendency to shift toward lower diffraction angles as the thickness increases. This tendency provides the necessary information for evaluating the plane spacing (d) and nano-strain (ε) in the samples. The Bragg law was employed to calculate the plane spacing (d) [24] while the nano-strain (ε) was evaluated using the following equation [25]:

$$\varepsilon = (d - d_0) / d_0 \quad (2)$$

where d (column 8 of Table 2) and d_0 are the plane spacing of the sample and standard powder sample, respectively. The obtained results that are listed in column 9 of Table 1 suggest the compressive strain for all

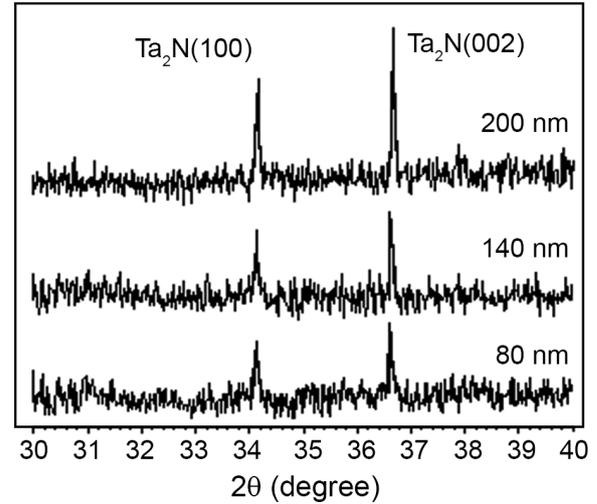


Fig. 1. X-ray diffraction pattern of the tantalum nitride thin films deposited at different thicknesses.

samples. However, an increase in film thickness limits the mentioned nano-strain in the film's structure. It is well-known that the crystallites/grains size increases in three ways [26]: i) Ostwald ripening, ii) coalescence due to mobility, and iii) coalescence by the growth. Therefore, it is expected that an increase in the film thickness causes the coalescence of the smaller crystallites (in the preferred orientation) by growth process. An increase in the number of incoming atoms on the substrate causes the improvement of the surface energy which in turn can limit the nano-strain in the film structure.

Electrical Properties

The reliability of the thin film resistors is strongly

Table 2. Detail of the structural parameters obtained from XRD analysis.

Thickness (nm)	(hkl)	2 θ (deg.)	I (a.u.)	FWHM (2 θ) (deg.)	D (nm)	TC	d (Å)	ε $\times 10^{-5}$
80	(100)	34.01	29	0.36	25.1	1.00	2.63390	-117.5
	(002)	36.61	36	0.40	20.3	0.99	2.45259	-179.4
140	(100)	34.01	39	0.34	27.5	0.99	2.63390	-117.5
	(002)	36.59	49	0.26	34.8	1.00	2.45388	-126.9
200	(100)	33.99	48	0.30	31.4	0.99	2.63540	-60.6
	(002)	36.57	63	0.22	39.0	1.02	2.45518	-74.0

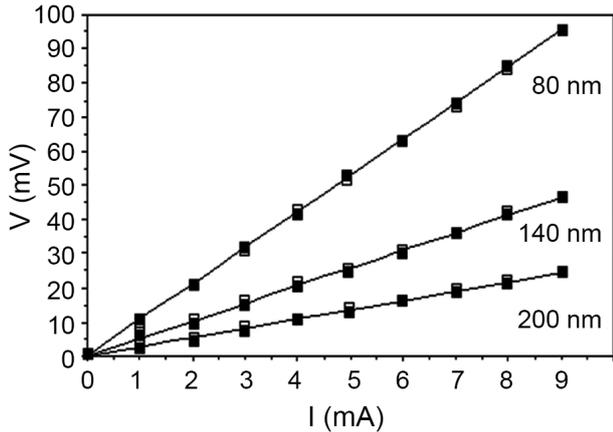


Fig. 2. Dependence of voltage with current of the Ta₂N thin films deposited at different thicknesses (The filled and hollow symbols show the measured values in the increasing and decreasing of increment, respectively).

dependent on the irreversibility of resistance [9]. To confirm the mentioned phenomenon, the variations of the resistance of the sample (I-V curves) were measured during both increasing and decreasing of voltage. The variation of the voltage of the samples versus the current is depicted in Fig. 2. The filled and hollow symbols are attributed to the measured values in the increasing and decreasing of increment, respectively. It can be observed that all samples deposited at different thicknesses show decent irreversibility. The values of the resistivity of the Ta₂N thin films, measured by a four-point probe instrument, are also shown in Fig. 3. It can be seen that the resistivity values decrease with an increase in thickness. The conductivity of the thin films can be limited by the scattering of carriers from the grain boundaries and film surface [26]. So that, the reduction of film thickness and/or the increasing

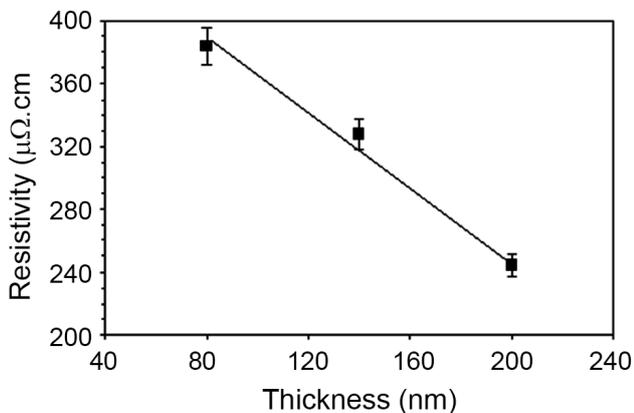


Fig. 3. The resistivity of the Ta₂N thin films as a function of film thickness.

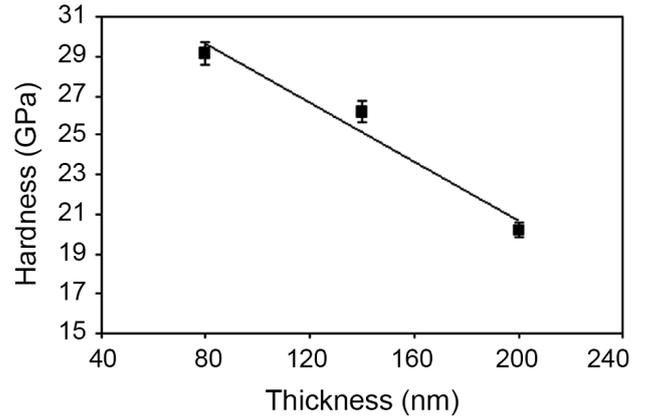


Fig. 4. The hardness values of the Ta₂N thin films as a function of film thickness.

of grain boundaries develop the scattering of the carriers. Therefore, based on the structural analyses, the reduction of carriers scattering due to the increase of film thickness and crystallite size can be the effective factor on resistivity values while the nitrogen concentration and phase are the same for all samples.

Mechanical and Tribological Properties

There is a widespread variance in the previous reports about the mechanical characteristics of tantalum nitride thin films. For instance, the hardness value of 20 GPa for an fcc-TaN thin film with (200) preferred orientation was reported by Bernoulli et al. [27]. They used the DC magnetron sputtering technique to deposit their samples. Nie et al. [28] reported a hardness value of 26 GPa for an fcc-TaN thin film with a strong (200) preferred orientation deposited by RF magnetron sputtering deposition. Their samples had a nonporous structure and a grain size of 50 nm. The

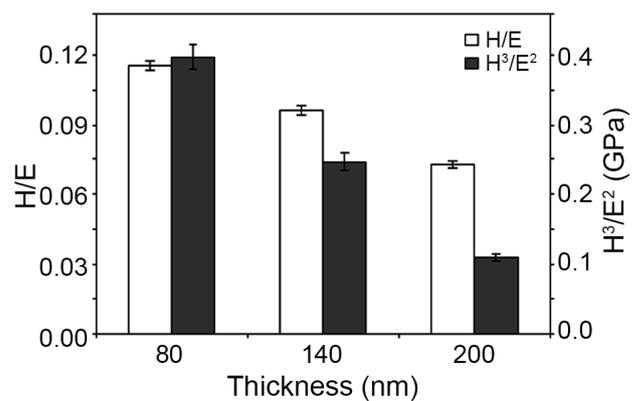


Fig. 5. The parameters of H/E and H³/E² of the Ta₂N thin films deposited at different thicknesses.

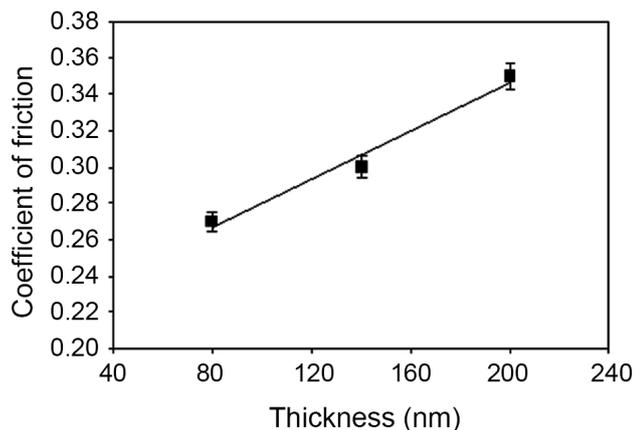


Fig. 6. The coefficient of friction values of the Ta₂N thin films deposited at different thicknesses.

hardness values of about 30-35 GPa are also reported for Ta₂N with hexagonal structure [13, 29, 30]. These results show that the mechanical characteristics of polycrystalline tantalum nitride are strongly affected by the chemical and phase composition as well as growth methods and parameters. The dependence of hardness values of Ta₂N thin films prepared in this work on the film thickness is shown in Fig. 4. It can be observed that the highest value of hardness is related to the sample with a thickness of 80 nm (29.1±0.6 GPa) that decreases with an increase in film thickness.

The elastic modulus values were found to be 250, 270, and 275 GPa for the samples with thickness of 80, 140 and 200 nm, respectively. The obtained values for elastic modulus in this research are smaller than the values of 350-400 GPa for Ta₂N coatings reported by other researchers [29, 31]. It is well-known that the resistance to contact damage depends not only on the hardness of the films but on the H/E and H³/E² ratios too [32]; so that, the higher values of the mentioned parameters (H/E and H³/E²) lead to improved wear resistance and tolerance to yield damage [33, 34]. The parameters of H/E and H³/E² of Ta₂N samples deposited under different thicknesses are represented in Fig. 5. It can be deduced that the Ta₂N sample of 80 nm thickness shows a better resistance to wear and abrasion damage than the thicker samples. To confirm the mentioned result, the scratch test was performed on all samples. The coefficient of friction and scratch volume of the Ta₂N samples as a function of thickness are plotted in Figs. 6 and 7, respectively. The results confirm that an increase in film thickness de-

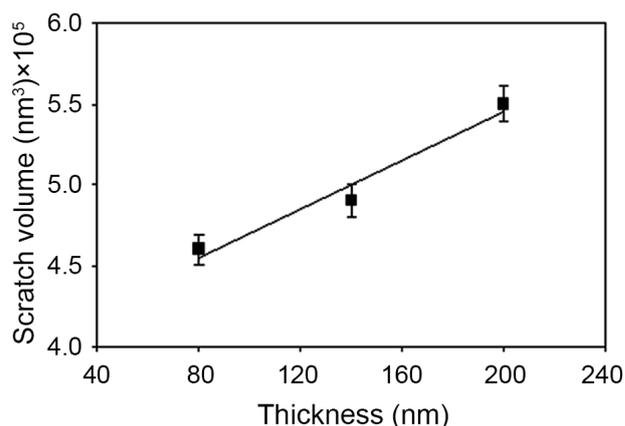


Fig. 7. The scratch volume values of the Ta₂N thin films deposited at different thicknesses.

stroys the resistance to wear and abrasion damage of the samples. This behavior can be attributed to the denser structure of thinner sample. The thin film hardness can be affected by several parameters including phase and crystallographic structure, density, stoichiometry, grain size, and lattice parameters [13, 14, 35-38]. According to the Musil theory, the reduction of grain size results in more grain boundaries which in turn suppresses the mobile and empty spaces and improves the hardness of the samples [39]. Furthermore, based on the inverse Hall-Patch hardening effect, the highest hardness of a nano-crystalline material may be achieved with a grain size of ~ 10-20 nm [40], and our sample with 80 nm thickness had a crystallite size of ~ 20 nm (second row of Table 2).

CONCLUSION

Ta₂N thin films were deposited using DC reactive magnetron sputtering technique under different thicknesses on the unheated Si and SiO₂/Si substrates. Nanostructures, electrical resistivity, as well as mechanical and tribological behaviors were studied as a function of film thickness. An increase in film thickness caused the increase of the crystallite size and the reduction of nano-strain in the film structure. All Ta₂N thin films with different thicknesses showed decent irreversibility of resistance. The highest resistivity and the best mechanical and tribological characteristics were related to the thinnest sample with 80 nm thickness because of its denser and close-packed structure.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interest regarding the manuscript. The authors also declare that no funds, grants, or other support were received during the preparation of this manuscript.

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