Surface & Coatings Technology 259 (2014) 123–128

Contents lists available at ScienceDirect

Surface & Coatings Technology

journal homepage: www.elsevier.com/locate/surfcoat

Fabrication and tribological behavior of sputtering TaN coatings

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ARTICLE INFO

Available online 25 March 2014

Keywords:
TaN
Amorphous
Crystalline
Tribological behavior

ABSTRACT

Tantalum nitride, TaN, coatings with crystalline and amorphous microstructure features are fabricated by magnetron reactively sputtering through Ar/N_2 gas flow control. The high Ar/N_2 flow ratio of 18/2 sccm/sccm enhances the formation of crystalline fcc TaN phase, while amorphous structure with excess N content in the TaN coating is obtained under a low ratio of 12/8. The multilayer coating with alternating stacking of the amorphous and crystalline TaN nanolayers, i.e., heterostructural a-TaN/c-TaN, is deposited for comparison. The c-TaN exhibits higher hardness and modulus around 21 and 232 GPa, respectively, as compared to the a-TaN. The a-TaN/c-TaN multilayer coating possesses hardness and modulus between those of a-TaN and c-TaN layers. Tribological behavior, including scratch and wear, of various TaN coatings are evaluated. The single TaN coatings, either with amorphous or crystalline structure, show an early stage severe cracking and chipping in scratch results. The brittle failure along the scratch scars of the single-layer TaN coatings is suppressed by the multilayer feature. Similar brittle failure modes are found for the a-TaN and c-TaN single-layer coatings under the pin-on-disc wear test. Smooth wear scar edge and limited delamination of the multilayer a-TaN/c-TaN coating are observed, indicating a tougher mechanical behavior and stronger adhesion of the multilayer coating as compared to single TaN layers.

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1. Introduction

The multilayer coating system has been intensively developed and adopted for practical protective coating applications due to its superior mechanical properties as compared to the single-layer film [1–6]. With an increasing demand for industry practice, further investigation of hard coatings in multilayer feature with modulation of different phases has attracted much attention in past few years. The combination of metal/nitride and nitride/nitride to form multilayer composite coatings with different crystal structures and phases is highly focused [7–9]. Transition-metal nitride films, such as, CrN, TiN, TaN, etc., have been utilized for cutting and drilling tools due to their excellent hardness, wear, and corrosion resistance [10–13]. Tantalum nitride film in particular is attracting increasing attention for thin film resistors, diffusion barriers in microelectronics [14–23], and surface protective coatings as well [24–26]. There are valuable investigations on the mechanical properties and thermal stability of TaN films, indicating its potential application as hard coatings [24–26]. However, the overall published reports for TaN are far less than other nitrides, such as TiN and CrN. In the physical vapor deposition, PVD, TaN film shows a variety of compound solutions, including BCC α-Ta(N), hexagonal γ-TaN, hexagonal ε-TaN, Ta_2N, and WC structure θ-TaN, cubic NaCl δ-TaN, hexagonal Ta_5N_6, tetragonal Ta_4N_5, and orthorhombic Ta_3N_5 [27,28]. The structures of TaN coatings prepared by PVD method depend intimately on the deposition technique and the process parameters. Especially for sputtering gas control, the coatings can be made with nanocrystalline and amorphous features [29]. Multilayer structure and related mechanical and corrosion behavior were also intensively studied [4,30]. It would be a potential niche to provide a multilayer nitride coating by one simple element with distinct microstructure combination though parameter control during fabrication. In this study, the single and multilayer TaN coatings are fabricated by reactive magnetron sputtering technique. The single-layer TaN coating, manipulated by Ar and reactive N_2 gas inlet ratio, forms crystalline or amorphous structure. The multilayer TaN coating is then deposited with alternating layers of different TaN microstructures. The phase, microstructure, and tribological properties of the coatings are analyzed and discussed.

2. Experimental procedures

The AISI 420 and Si (111) wafer were used as substrates in this study for various analyses. The AISI 420 substrates were ground with SiC sandpaper from 200# to 1200#, followed by 1 μm Al_2O_3 slurry polishing to obtain a surface roughness Ra below 30.0 nm. The commercially as received Si(111) wafer exhibited an Ra less than 15.0 nm. The substrates were cleaned in acetone and followed D.I. water ultrasonically for a suitable surface condition before sputtering deposition. Single-layer

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http://dx.doi.org/10.1016/j.surfcoat.2014.03.024
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and multilayer TaN coatings were deposited by radio frequency (RF) magnetron reactive sputtering technique with a commercial sputtering coater (MGS-350, JunSun Tech Co., Taiwan). A Ta target with 99.995% and 50.4 mm in purity and diameter, respectively, was applied as deposition source. During reactive sputtering, Ar and N_2 were applied as plasma source and reacting gases, respectively. A vacuum environment down to 1.06×10^{-4} Pa and a substrate temperature of 350 °C were fixed before deposition. The RF input power on Ta target for both TaN single and multilayer coatings was fixed at 100 W. The working distance, the target-to-substrate distance, was kept 100 mm during sputtering. The presputtering of the Ta target was carried out for 5 min to clean the Ta source surface. The working pressure was controlled at 6×10^{-1} Pa under a total gas flow of 20 sccm. The Ar to N_2 flow ratio, i.e., Ar/N_2, was controlled at 18/2 and 12/8 to form TaN coatings with different composition and microstructure, as designated in Table 1. TaN multilayer coatings were then fabricated by sequential stacking of crystalline, c-TaN ($Ar/N_2 = 18/2$), and amorphous, a-TaN ($Ar/N_2 = 12/8$), layers with 20 nm bilayer thicknesses. The sputtering of TaN was shuttered during the change of Ar/N_2 ratios of 18/2 and 12/8 sccm/sccm and reoperated when a stable flow ratio was reached in multilayer TaN deposition. The overall coating thickness for each sample was controlled around 1 μm. The deposition intervals for c-TaN and a-TaN single layers were 72 and 90 min. The calculated deposition rates for c-TaN and a-TaN layers were 13.9 and 11.1 nm/min, respectively. The composition of the coatings was determined with an electron probe microanalyzer (FE-EPMA, JAX-8800, JOEL, Japan). The cross-sectional images, detailed microstructure, and phase analysis were carried out through field emission scanning electron microscopy (FESEM, JSM-6700 F, JEOL, Japan) and transmission electron microscopy (TEM, JEM-2100, JEOL, Japan). The hardness and Young's modulus of the coatings were measured and analyzed through nano-indentation technique with a nano-hardness tester (TriboIndenter, TI900, Hysitron, USA). The scratch test was carried out with a microscratch tester (Scratch Tester, J&L Tech, Korea) with progressive loading from 0 to 50 N. The wear test was conducted with a pin-on-disc wear tester (Tribometer, J&L Tech, Korea). The rotation radius was 4 mm and the total wear distance was 500 m.

3. Results and discussion

3.1. Microstructure

The controlled Ar/N_2 ratios in manufacture process exhibited strong influence on composition and phase of the TaN coatings. As listed in Table 1, the composition ratio of Ta/N for the coating deposited under $Ar/N_2 = 18/2$, denoted as c-TaN, was close to 1. On the other hand, an excess of N content was obtained for the coating generated under $Ar/N_2 = 12/8$. The higher input of N_2 in the process lowered down the Ar^+ ion intensity and energy in the plasma; thus, a lower Ta content in the coating was expected. Fig. 1 shows the cross-sectional images of the TaN single and multilayer coatings under different Ar/N_2 inputs. A columnar crystalline structure was found for the c-TaN coating, while smooth and featureless image was obtained for the deposit formed under $Ar/N_2 = 12/8$, designed as a-TaN. For a detailed insight of the microstructure and configuration, TEM was conducted for various TaN single and multilayer coatings. Fig. 2 indicates the TEM images and selected area electron diffraction patterns, SAED, of the c-TaN, a-TaN, and a-TaN/c-TaN multilayer films. A columnar structure for the c-TaN film could be observed in Fig. 2a, and a fully crystalline fcc δ-TaN

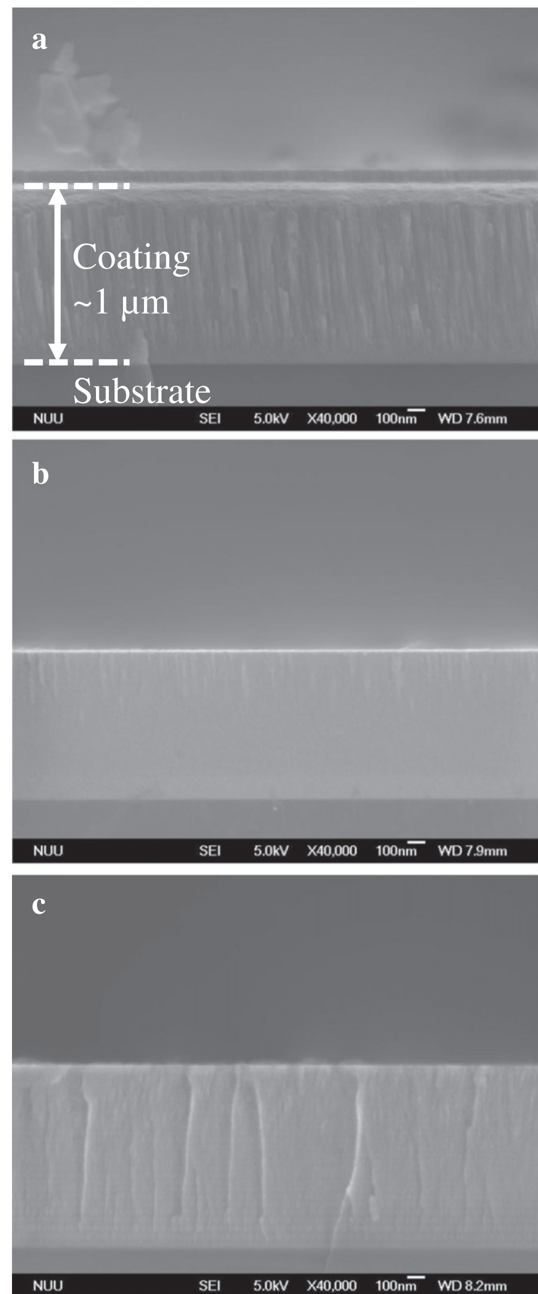

Fig. 1. Cross-sectional SEM images of samples (a) c-TaN, (b) a-TaN, and (c) multilayer TaN coatings.

Table 1
The deposition condition, composition, hardness, and modulus of various TaN coatings.

Coating designation	Gas flow (sccm)		Chemical composition (at%)		Indentation hardness (GPa)	Modulus (GPa)	H/E	H3/E2
	Ar	N2	Ta	N				
c-TaN	18	2	47.8 ± 2.9	52.2 ± 2.9	21.1 ± 1.1	232.7 ± 7.6	0.091	0.173
a-TaN	12	8	39.3 ± 0.8	60.7 ± 0.8	14.1 ± 0.2	190.3 ± 2.3	0.074	0.078
Multilayer	–	–	–	–	16.7 ± 0.5	206.9 ± 3.2	0.081	0.109

*Input power of Ta for all coatings is fixed at 100 W.

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