



Effect of coating architectures on the wear and hydrophobic properties of Al–N/Cr–N multilayer coatings

Yu-Sen Yang^{a,*}, Ting-Pin Cho^{b,c}, You-Chang Lin^a

^a Department of Mechanical and Automation Engineering, National Kaohsiung First University of Science and Technology, 2 Juoyue Rd., Nantz District, Kaohsiung 81164, Taiwan, ROC

^b Institute of Engineering Science and Technology, National Kaohsiung First University of Science and Technology, 2 Juoyue Rd., Nantz District, Kaohsiung 81164, Taiwan, ROC

^c Metal Industries Research & Development Centre, 1001 Kaonan Highway, Kaohsiung 811, Taiwan, ROC

ARTICLE INFO

Available online 4 February 2014

Keywords:

Reactive sputtering
Al–N/Cr–N coatings
Multilayer coating
Water contact angle
Wear

ABSTRACT

Two coating architectures with various deposition times (Dts) for each layer and N₂ gas flow rate ratio η (N₂/(N₂ + Ar)) of Al–N/Cr–N multilayer coatings were prepared using unbalanced magnetron reactive sputtering. Using the same coating process, two coating architectures were deposited on the high speed tool steel and Si wafer, one starting with an Al–N layer designed as the Al–N/Cr–N/.../Cr–N/Al–N (architecture A) and the other starting with a Cr–N layer designed as the Cr–N/Al–N/.../Al–N/Cr–N (architecture C). During the coating process, the N₂ gas flow rate ratio η was set at 15%, 20% and 25% to control the Cr–N and Al–N layer phases. This study investigates the effects of architectures, Dt and η on the wear and hydrophobic properties of the coatings. The X-ray diffraction results show that Cr(N), Cr(N) + Cr₂N and Cr₂N + CrN phases respectively form in the Cr–N layer given η = 15%, 20% and 25%, and only the hcp-AlN phase forms in the Al–N layer. The layer thickness ratio (λ) of the Cr–N layer to the Al–N layer increases from 2.7 to 6.4 as η increases from 15 to 25%. The wear resistance of the coatings is strongly related to the coating architecture. The architecture A coatings exhibit better wear resistance than the architecture C coatings. The number of layers and layer thickness ratios λ also affects the wear behavior. In terms of the hydrophobic property, the coating architecture C with the Cr₂N + CrN phase (η = 25%) on the top layer shows higher water contact angle measurements of 95°–97°.

© 2014 Elsevier B.V. All rights reserved.

1. Introduction

Physical vapor deposition (PVD) processes have long been used in the preparation of hard coatings [1–3]. PVD nitride coatings exhibit good wear resistance and chemical stability and have been successfully used on industrial protective usage [4–11]. The first generation of hard PVD coatings consisted of monolayer metal nitrides such as TiN, CrN and ZrN. These PVD coatings are still used extensively, but suffer from insufficient tribological properties. CrCN and TiCN exhibit lower wear rates than CrN and TiN [1,3]. Incorporating Al into the TiN and CrN structure serves to increase oxidation resistance as well as the tribological properties [8,12–15]. Superior tribological performance has been achieved using multilayer structures such as TiN/TiCN, TiN/CrN, TiN/AlN, and CrN/CrCN [16–21].

In addition to wear resistance, a higher WCA is also an important property for plastic injection molds [22]. The coating's anti-sticking property is related to the polar components of surface energy, with lower polar components corresponding with increased water-repellency as well as a higher water contact angle (WCA) [23,24]. WCA measurement is a handy method to study the water-repellency behavior of coatings. A

higher WCA corresponds with improved release performance for plastic injection molds.

Previous research has focused on nano-structured CrN/AlN multilayer coatings [25,26], but the effect of architecture and layer thickness ratio on wear and hydrophobic properties has been neglected. The Al–N/Cr–N multilayer coatings were deposited in Ar/N₂ plasma with various N₂ gas flow rate ratios by reactive magnetron sputtering. The effects of architecture, N₂ flow rate ratio and layer thickness ratio on wear resistance and WCA were investigated. In particular, the different behaviors corresponding to various architectures of Al–N/Cr–N/.../Cr–N/Al–N and Cr–N/Al–N/.../Al–N/Cr–N are explored.

2. Experimental details

2.1. Deposition of Al–N/Cr–N multilayer films

The multilayer coatings were prepared using a closed field unbalanced magnetron sputtering system with four vertical cathodes at intervals of 90°. The deposition chamber was 550 mm in diameter and 500 mm tall, while the dimensions of the target are 300 mm × 109 mm × 10 mm. Two Al (purity 99.999%) targets were powered by a medium-frequency (MF, 40 kHz) power source for the deposition of the AlN layers. A Cr (purity 99.95%) target was powered by a DC power source for the deposition of the Cr–N coating. The gas purity of

* Corresponding author. Tel.: +886 7 6011000x2292; fax: +886 7 6011066.
E-mail address: yusen@nkfust.edu.tw (Y.-S. Yang).

both the Ar and N₂ is 99.999%. For the wear test, JIS SKH51 high-speed steel specimens measuring 40 mm × 40 mm × 4 mm were heat treated to a hardness of 62.5 ± 1 HRC. Morphological anisotropy affects the water contact angle [27] and in this study this impact was minimized using polished Si (100) substrates. Test specimens were cleaned in an ultrasonic cleaner with surfactant for 15 min followed by de-ionized water for 10 min, and then dried at 100 °C for 15 min before the coating deposition. Prior to deposition, the coating chamber was pumped down to 2.6 × 10^{−3} Pa. Substrates were bombarded with argon ions (Ar⁺) at a pressure of 0.57 Pa and a bias of −450 V for 20 min before deposition.

The sputtering process includes three branches which correspond to the well-known metallic, transition and reactive mode of discharge [28]. As shown in Fig. 1, based on the reactive sputtering hysteresis curve of AlN coatings (discharge current 3 A), the N₂ flow rate ratio η (N₂/(N₂ + Ar)) was set at 15, 20 and 25% for the transition and reactive modes.

As the η will seriously affect the deposition rate, the deposition rates of Al–N and Cr–N for each run were measured prior to the experimental stage. The deposition temperature of all coatings was about 200 ± 20 °C. A 60 nm-thick Cr layer was initially deposited as an interlayer for all coatings.

The formulation Z-Dt- η represents the architecture (Z), deposition time (Dt) of individual Al–N or Cr–N layer and N₂ flow rate ratio (η). Table 1 provides a detailed description of the formulation. Two architectures were designed: Al–N/Cr–N.../Cr–N/Al–N (architecture A) and Cr–N/Al–N.../Al–N/Cr–N (architecture C). The Dt was respectively set at 1, 2 and 3 min, to obtain the various layer thicknesses. Table 2 shows all of the deposition parameters for the Al–N/Cr–N multilayer coatings. Fig. 2 shows the jig specially-designed to produce the Al–N/Cr–N multilayer coatings. As shown in the figure, the deposition zone can be separated into two independent zones using a stainless steel plate to avoid cross-contamination of the Al–N and Cr–N coatings. Thus, for example, the specimens A-2-20 and C-2-20 can be deposited in the same batch. First, only the Cr target was powered on to deposit a 60 nm-thick Cr interlayer on specimens A-2-20 and C-2-20 at a jig rotation speed of 3 rpm. Then, rotating the A-2-20 and C-2-20 specimens to respectively face the Al target and Cr targets, Al–N and Cr–N layers were simultaneously deposited with an η value of 20% for 2 min. Then, by rotating the jig by 180° as quickly as possible, the Cr–N layer was deposited on the A-2-20 specimen and the Al–N layer was deposited on the C-2-20 specimen for 2 min at the same time. The process was repeated until the layers were complete to satisfy experimental requirements.

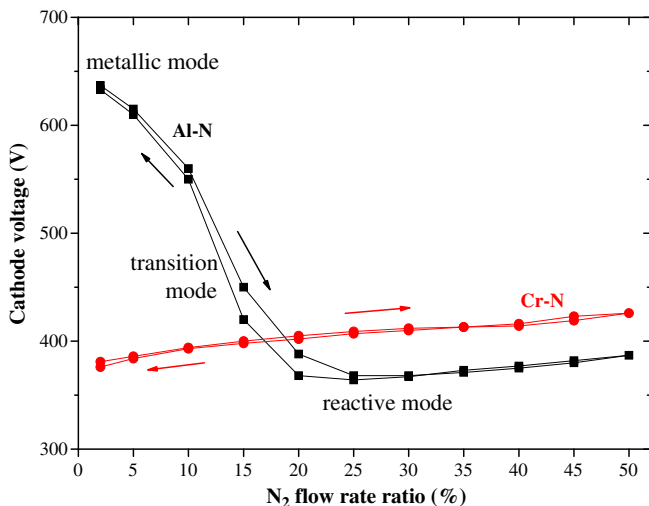


Fig. 1. Reactive sputtering hysteresis curves of Al–N and Cr–N coatings.

Table 1
Detailed description of formulation.

Formulation	Architecture	Deposition time per layer	N ₂ flow rate ratio
	Z	Dt	η
Z-Dt- η	A: Al–N/Cr–N...Cr–N/Al–N	1: 1 min	15: 15%
	C: Cr–N/Al–N...Al–N/Cr–N	2: 2 min	20: 20%
		3: 3 min	25: 25%

2.2. Coating characterization

The thickness and morphologies of the multilayer coatings were observed by field emission scanning electron microscopy (FE-SEM, Hitachi-4700) with an accelerating voltage of 15 kV. The sliding wear behavior of the coatings deposited on the high-speed steel specimens was investigated by means of a ball-on-disk wear testing machine (Freeform, SMT1-25N) with a 6.3 mm diameter Cr steel ball (C:0.95–1.10%, Cr:1.30–1.60%, Si:0.15–0.35%, hardness of 62–65 HRC). The sliding velocity was set at a constant speed of 0.3 ms^{−1}. The rotation cycle diameter, sliding distance and normal load were respectively set at 10 mm, 1000 m and 5 N. The wear rate was observed using a 3D scanning system (TalyScan 150) and a mathematical estimation model [29].

The hardness H and elastic modulus E of the coatings were measured using a nano-indentation instrument (ASMEC, UNAT-M) to calculate the H/E with a force at final contact of 35 mN. The crystal structure of the coatings was characterized by a grazing incidence X-ray diffractometer (Bede, D1 HR-XRD) with Cu K α radiation (λ = 1.5418 Å, 2 θ /min = 3°). The surface roughness Ra of the coatings was measured by a surface profiler (KLA Tencor Alpha-Step IQ) with a vertical resolution of 0.24 Å. The measurement distance and scanning speed were 800 μ m and 50 μ m/s, respectively. The sessile-drop method was used for the WCA measurement by a contact angle measurement device (FTA, Inc., FTA-200). The contact angle is the angle at which a liquid/vapor interface meets a solid surface. The equilibrium of forces among the surface tensions at a 3-phase boundary is described by Young's equation [30]. All of the properties were investigated using Si (100) wafer, except for the wear behavior.

3. Results and discussion

3.1. Architecture and deposition rate

Al–N/Cr–N multilayer coating architectures can be achieved by controlling the N₂ gas flow rate ratio (η) and the deposition time (Dt) per layer. Fig. 3 shows SEM images of two typical architectures. Fig. 3(a)

Table 2
Deposition parameters for Al–N/Cr–N multilayer coatings.

Coatings	Al–N layer	Cr–N layer
Interlayer:		
Total flow rate (Ar) (sccm)	30	30
Target materials	Cr	Cr
Target sputtering current (A)	3	3
Substrate bias frequency (kHz)	50	50
Bias of substrate (−V)	70	70
Thickness of interlayer (nm)	60	60
Multilayer:		
Total flow rate (Ar + N ₂) (sccm)	50	50
Flow rate ratio N ₂ /(Ar + N ₂) (%)	15, 20, 25	15, 20, 25
Target materials	Al	Cr
Working pressure (Pa)	0.40	0.40
Target sputtering current (A)	3	3
Substrate bias frequency (kHz)	100	100
Bias of substrate (−V)	75	75
Duty cycle (%)	15	15
Distance, target to specimen (cm)	7	7

Download English Version:

<https://daneshyari.com/en/article/1657350>

Download Persian Version:

<https://daneshyari.com/article/1657350>

[Daneshyari.com](https://daneshyari.com)