

Thermal stability and microstructure characterization of CrN/WN multilayer coatings fabricated by ion-beam assisted deposition

Yan-Zuo Tsai, Jenq-Gong Duh *

Department of Materials Science and Engineering, National Tsing Hua University, Hsinchu, Taiwan

Available online 13 September 2005

Abstract

CrN/WN multilayer coatings were deposited on both silicon (100) and stainless steel substrate by the ion-beam assisted deposition. The bilayer periods were designed and controlled in the range from 3 nm to 30 nm. The CrN/WN films were annealed from 750 °C to 850 °C for 1 and 4 h in vacuum to evaluate the thermal stability of these coatings. The difference of microstructural characterization between the CrN/WN multilayer coating and the CrN single layer coating was investigated by scanning electron microscopy. The phase transformation after annealing was probed by X-ray diffraction. The hardness of as-deposited CrN/WN coating with 8 nm bilayer period was much higher than that of CrN and WN single layers. The thermal stability of CrN/WN multilayer coatings with different bilayer periods was also discussed in correlation to the associated microstructural evolution.

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Keywords: CrN; CrN/WN; IBAD; Superlattice; Nanolayer; Hardness

1. Introduction

Due to the excellent performance in hardness, wear and corrosion resistance, the transition metal nitrides have been taken for hard protective coating [1,2]. In the nitride systems, the chromium nitride (CrN) films have been investigated for years, and proved to exhibit good mechanical performance, thermal properties and anti-oxidation behaviours [3,4]. Recent studies also show that multilayer coatings composed of two kinds of transition nitride films exhibit superior mechanical strength, such as hardness, adhesion, and wear resistance, as compared to single layer nitride coating due to their specific interfaces [5–7]. Several new material systems, including TiN/AlN, TiN/NbN, CrN/TiN and CrN/AlN, exhibit evident enhancement of micro-hardness [7–10]. Theoretically, the enhanced hardness could be explained by dislocation blocking between interfaces, due to the shear moduli difference, and by coherency strain from lattice mismatch of the two different

material systems [11]. The multilayer coatings sustain high hardness at the room temperature, however, after heat treatment at elevated temperature in vacuum, the strength of multilayer coatings would be degraded rapidly, which was caused by the vanishing of nanolayered structure due to the inter-diffusion of atoms. The thermal stability is thus a critical issue and plays an important role on performance of tools operated at the elevated temperature in vacuum. In this study, the combination of chromium nitride and tungsten nitride coating was proposed to form a nanostructure coating system. Efforts were concentrated on the structural characterization and thermal stability of both CrN and CrN/WN coatings with bilayer periods of 3 nm to 30 nm in vacuum environment. The microstructure of CrN and CrN/WN multilayer coatings were analyzed by X-ray diffraction and SEM. In addition, the strength of the deposit under various conditions was investigated by nanoindentation.

2. Experimental details

The CrN single layer coating and CrN/WN multilayer coatings were fabricated on the silicon (100) and AISI

* Corresponding author. Fax: +886 3 5712686.

E-mail address: jgd@mx.nthu.edu.tw (J.-G. Duh).

Table 1

Layer configuration and microhardness of the CrN/WN multilayer coatings

Sample name	Bilayer period (nominal) (nm)	Bilayer period (calculated) (nm)	$\Delta 2\theta$ (deg)	Microhardness (GPa)
041126	30	29.4	0.30	25.0
041028	10	11.4	0.77	24.5
041105	8	8.4	1.05	29.1
041116	5	5.6	1.57	24.5
041207	3	3.2	2.76	25.6

420 tool steel substrate by ion-beam assisted deposition (IBAD). Metallurgical finishing and polishing with SiC sandpapers of #120, #240, #400, #600, #800, and #1200 were used to remove the contamination on the surface of steel substrate. To obtain an adequate surface condition for the following tests and property evaluation, the ground substrates were then polished with a 1.0 μm diamond powder. After grinding and polishing treatment, the substrates were cleaned by ultrasonic vibration cleaning in acetone to remove all contaminates. Both chromium and tungsten target of 3 in. in diameter were 99.99 wt.% in purity. After loading of the substrates and targets, the vacuum chamber was degassed down to 2.1×10^{-4} Pa, followed by the inlet of argon and nitrogen gases as plasma source and reactive gas, respectively, to a working pressure of 1.2×10^{-1} Pa. The target-to-substrate distance was fixed at 100 mm from sputtering target sources and ion gun. Before deposition, both chromium and tungsten targets were pre-sputtered for 2 min to clean the target surface, and then Cr interlayer was deposited with a power of 300 W for 2 min. An assisted ion source (Mark II Gridless ion source, Veeco) was adopted during sputtering.

The current of assisted ion beam and electron beam were 4.0 and 2.7 A, respectively. Sputtering of Cr and W was proceeded alternately to form the sequential CrN/WN multilayer coating. Both the input power on Cr and W target were fixed at 300 W. The deposition time of individual nitride layer of the multilayer CrN/WN coating during sequential sputtering was modified from 8 to 93 s. The total thickness of every multilayer coating with different bilayer periods was controlled around 1.0 μm . The CrN/WN multilayer coatings were then annealed at 750, 800 and 850 $^{\circ}\text{C}$ in a vacuum chamber with a pressure of 6.7×10^{-4} Pa for 1 h and 4 h.

The multilayer structure and crystallographic phases of the thin film were identified by low-angle and high angle diffraction, respectively, with an X-ray diffractometer (Shimadzu, XRD6000) under $\theta/2\theta$ mode. The coating thickness and cross-section image were observed with a field emission scanning electron microscope (FESEM, JSM-6500, JEOL, Japan). The microhardness of the coatings was analyzed with a nanoindentation apparatus (TriboScope, Hysitron, Minneapolis, MN) equipped with a Berkovich indenter. The maximum load adopted for all the coating was fixed at 3000 μN .

3. Results and discussion

3.1. Low-angle X-ray diffraction

The CrN/WN multilayer coatings were fabricated with total thickness about 1.0 μm by IBAD process. By controlling the deposition time of individual nitride layer,

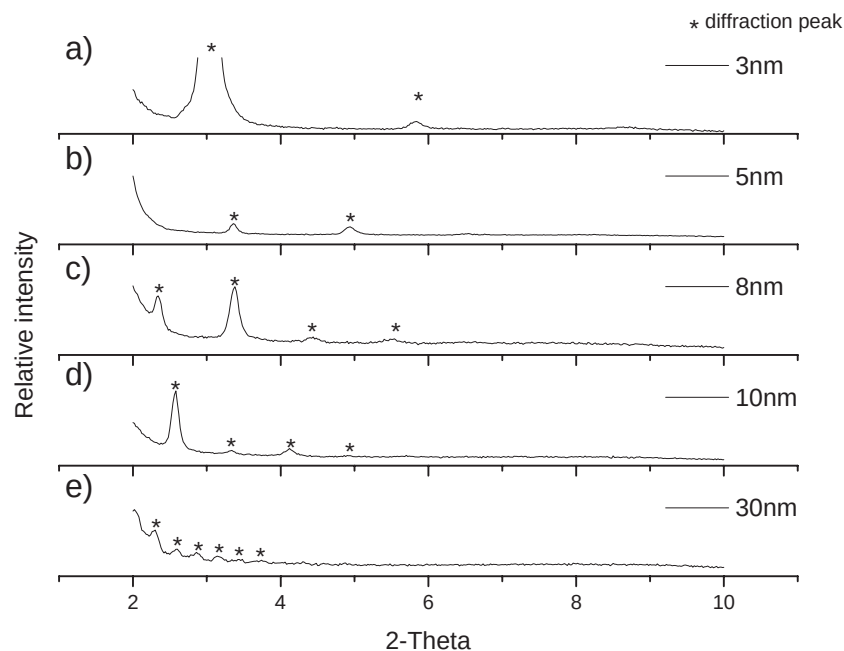


Fig. 1. Low-angle X-ray diffraction spectra of CrN/WN multilayer coatings with adjusted bilayer period (a) $A=3$, (b) $A=5$, (c) $A=8$, (d) $A=10$ and (e) $A=30$ nm.

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