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Structure, hardness and tribological properties of nanolayered TiN/TaN multilayer coatings

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Abstract

TiN/TaN coatings, consisting of alternating nanoscaled TiN and TaN layers, were deposited using magnetron sputtering technology. The structure, hardness, tribological properties and wear mechanism were assessed using X-ray diffraction, microhardness, ball-on-disc testing and a 3-D surface profiler, respectively. The results showed that the TiN/TaN coatings exhibited a good modulation period and a sharp interface between TiN and TaN layers. In mutilayered TiN/TaN coatings, the TiN layers had a cubic structure, but a hexagonal structure emerged among the TaN layers besides the cubic structure as the modulation period went beyond 8.5 nm. The microhardness was affected by the modulation period and a maximum hardness value of 31.5 GPa appeared at a modulation period of 8.5 nm. The coefficient of friction was high and the wear resistance was improved for TiN/TaN coatings compared with a homogenous TiN coating, the wear mechanism exhibited predominantly ploughing, material transfer and local spallation.

Keywords: TiN/TaN multilayered coating; Hardness; Coefficient of friction; Wear resistance

1. Introduction

Multilayers with bilayer lengths in the nanometer range exhibit significant improvements in hardness, toughness, oxidation resistance and corrosion resistance as compared to single-layered coatings. Among them, transitional nitrides/nitrides have attracted much attention recently due to their substantial strength and hardness enhancements. Mutilayers consisting of very thin (2–10 nm) layers of nitride materials deposited by magnetron sputtering have a hardness in excess of 5000 kg/mm^2 [1]. This hardness is comparable to that of cubic-BN and is second only to diamond. Therefore, the potential for the development of new hard coatings for the machine industry (for example in cutting operations) is great, using materials with good tribological properties as the individual layers of the multilayers. Up to now most of the work has been done on the mechanism for nitride superlattice hardening. A variety of mechanisms have been used to explain the enhancement, including dislocation blocking by layer interfaces, Hall–Petch strengthening and the supermodulus effect [2–4].

In recent years, hard coatings have found wide applications in cutting tools in terms of extending the

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operating life and range of conditions for which they are used. Especially hard and wear resistant TiN coatings deposited by physical vapor deposition (PVD) or by plasma assisted chemical vapor deposition (PACVD) have gained increasing importance in industry, for example in protecting cutting tools, and the tribological properties and wear mechanism have been widely investigated. On the other hand, even though several coating studies have revealed that many nitride multilayers (TiAlN/VN [5], TiN/CrN [6] and TiN/Nb [7]) can outperform the single layered TiN in terms of wear resistance, little is known about the detailed tribological behavior of the nitride multilayered coatings, such as the worn surface profile and the effect of structure and hardness on the wear mechanism. The main purpose of this paper is to investigate the structure and tribological behaviors with particular reference to the morphology of worn surfaces, and establish correlations between hardness and tribological properties of TiN/TaN multilayered coatings deposited by a reactive magnetron sputtering technique.

2. Experimental details

The polycrystalline TiN/TaN nanomultilayers were deposited using a JPG450 magnetron sputtering system, which has three targets including one d.c. and two r.f. magnetron cathodes. The sputtering targets were pure Ti (99.9%) and Ta (99.9%), which were mounted on each of the r.f. cathodes. Ground and polished single-crystal silicon (111) wafers were used as substrate materials, which were chemically cleaned in an ultrasonic agitator in acetone, and absolute alcohol before being mounted in the vacuum chamber. For all coatings, the deposition sequence started with the growth of a thin (approximately 20 nm) Ti interlayer followed by a 200-nm-thick TiN layer. Both the Ti and the TiN layers were obtained with the substrates held stationary above the Ti target. After this, the substrate was rotated to the position above Ta and Ti targets alternately and was held stationary for different times to obtain a compositionally-modulated structure. The modulation ratio was obtained though exact control of the stopping times in front of the Ti and Ta targets. Typically, TiN/TaN multilayers were deposited under a base pressure of 4×10^{-4} Pa and a total Ar+ N_2 gas pressure of 5.0×10^{-1} Pa. The modulation ratio l_{TaN}/l_{TiN} was fixed at 3:1. The source power of Ti and Ta targets were 110 and 70 W, respectively. The total thickness of multilayers was 1.0 μm, and all the substrates were resistively heated to 723 K during deposition.

Reciprocating ball-on-disc sliding experiments were performed in ambient air (60% RH) on a UMT-2 machine. 5 mm diameter hardened 52100 bearing steel balls (61HRC and 0.05 μm roughness) were employed as the counterpart, and the coatings were tested as the disc. The sliding speed was 1.0 mm/s over a stroke (track) length of 10 mm. A load of 4.0 N was used. All the experiments were run for 180 cycles. A Newview5022 3-D surface profiler was used to determine the depth profiles and hence cross-sectional areas of wear tracks. Based on the wear track diameter and depth profiles at several locations, the coating volume removed during the testing was obtained to evaluate the coating wear resistance.

The modulation periods of TiN/TaN multilayers were measured by a low-angle X-ray reflectivity method using a Rigaku X-ray diffractometer (XRD) using Cu K_α radiation under conditions of 40 kV and 30 mA. The coating crystallographic structures were characterized by high-angle X-ray diffraction (XRD). An atomic force microscope (AFM) was used to examine the surface morphology of the coatings. The hardness of the coatings was measured for 15 s at a load of 0.1 N using a DMH-2LS microhardness tester with a pyramidal Knoop diamond tip indenter. The morphologies of wear surfaces of coatings were examined using a Newview5022 3-D surface profiler and a JEOL8600 scanning electron microscope (SEM) with an energy dispersive X-ray spectrometer (EDS) attachment.

3. Results and discussion

3.1. Structure, surface morphology and hardness of multilayered TiN/TaN coatings

Low-angle X-ray reflectivity refers to the reflection of X-rays from the interfaces between layers. Reflection peaks of different orders in the low-angle X-ray reflectivity spectra occur at 2θ positions given by the modified Bragg's law:

$$\mathrm{Sin}^2\theta = (m\lambda/2\Lambda)^2 + 2\delta \tag{1}$$

where m is the order of the reflection, λ is the X-ray wavelength, δ is related to the average reflective index n, and Λ is the modulation period of a multilayer (bilayer period). Fig. 1 represents the low-angle reflective spectra of TiN/TaN multilayers with Λ=2.8 nm and Λ=5.8 nm. The strong superlattice reflections indicate that the multilayered coatings deposited in this study have well-defined periodicity and abrupt interfaces, which are a beneficial characteristic for hardness enhancement. By plotting $\mathrm{Sin}^2\theta$ vs. m^2 curve and fitting the data to a straight line, modulation period values can be determined from the slope of the line. All modulation periods presented in this paper were calculated using this method, and are shown in Table 1.