Available online at www.sciencedirect.com

SCIENCE DIRECT®

Surface & Coatings Technology 198 (2005) 432–436

SURFACE & COATINGS TECHNOLOGY

www.elsevier.com/locate/surfcoat

Unbalanced magnetron sputtered Ti–Si–N:MoS_x composite coatings for improvement of tribological properties

Xing-zhao Ding[a,*], X.T. Zeng[a,b], Takashi Goto[b]

[a]*Surface Technology Group, Singapore Institute of Manufacturing Technology, 71 Nanyang Drive, Singapore 638075, Singapore*
[b]*Institute for Materials Research, Tohoku University, Sendai 980-8577, Japan*

Abstract

Development of nanocomposite coatings attracted great interest in the past few years. The Ti–Si–N nanocomposite coatings demonstrated superhigh hardness and thermal stability. The tribological performance of these coatings, however, was often under expectation in comparison with that of conventional coatings such as TiN, due to their high friction coefficients. It is desired to incorporate lubricant elements into these coatings without significantly sacrificing their mechanical properties.

In this work, Ti–Si–N composite coatings incorporated with MoS_x were deposited using unbalanced magnetron sputtering. The MoS_2 was co-sputtered and served as lubricant element to reduce the wear friction coefficient. The results showed that with a small amount of MoS_2 incorporation (less than 5 at.% of Mo), the tribological properties of the as-deposited composite coatings were significantly improved with little influence on their superhigh hardness. Both the friction coefficient and wear rate decreased monotonously with increase of the Mo content. It is interesting to note that for the coating with only about 1 at.% of Mo content, the friction coefficient and wear rate were abruptly reduced to about 1/2 and 1/3 of those without MoS_2 incorporation, respectively. When the Mo content was increased up to about 5 at.%, the wear rate of the coating could be reduced to about one tenth in comparison with the coatings without MoS_2 co-deposition.

PACS: 81.15.Je; 81.15.Cd; 62.20.Qp
Keywords: Hard coatings; Magnetron sputtering; Ti–Si–N; Tribological properties; Friction

1. Introduction

In the past few years, Ti–Si–N nanocomposite coatings have attracted great attention over the world because of their superhigh hardness which can be more than twice of that of the commercially available titanium nitride coatings [1–5]. In practical applications, however, high hardness is not the only decisive factor, other key properties that determine the performance of a coating include friction coefficient, thermal and oxidation resistance, toughness and adhesion. It has been reported [6] and also observed in the present work that the tribological properties of the Ti–Si–N nanocomposite coatings showed insignificant improvement in wear test, in comparison with those of titanium nitride, due to their high friction coefficients. Therefore, how to reduce the friction coefficient of such superhard coatings becomes an interesting research topic.

MoS_2 is well known as a dry lubricant material. In high speed machining and some other dry cutting applications, it was used as the overlayer for hard coatings such as TiAlN to reduce the friction coefficient of the coated cutting tools [7,8]. However, owing to the fact that these overlayers are soft, they are consumed very fast, resulting in a short service lifetime. Alternatively, it would be ideal if such lubricant elements can be incorporated into the hard coatings without significantly sacrificing their mechanical properties, thus the lubricating effect can be maintained throughout the entire lifetime of the hard coating. In this case, the matrix hard phase provides wear, scratch and abrasion resistance and load-bearing capacity for the coated surface, while the soft lubricant phase, on the other hand, lowers the friction coefficient between the coated surface and counterpart. As the hard phase wears down and the lubricant is removed

* Corresponding author. Tel.: +65 6793 8528; fax: +65 6792 2779.
E-mail address: xzding@SIMTech.a-star.edu.sg (X. Ding).

0257-8972/$ - see front matter © 2004 Elsevier B.V. All rights reserved.
doi:10.1016/j.surfcoat.2004.10.053

from the tribo-contact, new reservoirs of the lubricant are gradually exposed.

It has been successfully demonstrated [9–12] that MoS_2 can be added to hard, wear-resistant ceramic coatings such as TiN, CrN, TiB_2, and Al_2O_3, by either chemical vapor deposition (CVD) or physical vapor deposition (PVD) processes. Recently, it was shown [13] that the TiN films incorporated with small amounts of 8% MoS_x exhibited both high hardness and low friction coefficient and were especially suited to dry cuttings. It was demonstrated that the lifetime of the drills coated with TiN–MoS_x was increased for about 1.5–2 times in comparison with the pure-TiN coated ones.

In this work, Ti–Si–N composite coatings incorporated with MoS_2 were deposited using unbalanced magnetron sputtering. The mechanical and tribological properties of these composite coatings were characterized and compared with the Ti–Si–N coating.

2. Experimental details

Ti–Si–N:MoS_x composite coatings, with a thickness around 3 μm, were deposited using a Teer UDP-550 unbalanced magnetron sputtering system. Fig. 1 shows a schematic diagram of the deposition system. One titanium target, one silicon target, and two molybdenum disulfide targets (all of the targets are of rectangular shape with dimensions of 133×330 mm^2) were installed around the cylindrical wall of the deposition chamber. Deposition was conducted in a flowing Ar+N_2 mixture atmosphere. The DC current applied onto the Ti target was fixed at 7 A, and the RF power applied onto the Si target was fixed at 600 W. Without MoS_2 doping, a pure Ti–Si–N film was first deposited, which has a silicon content of about 8 at.% and a microhardness of about 40 GPa. As a benchmark, a pure TiN coating was also deposited under similar conditions.

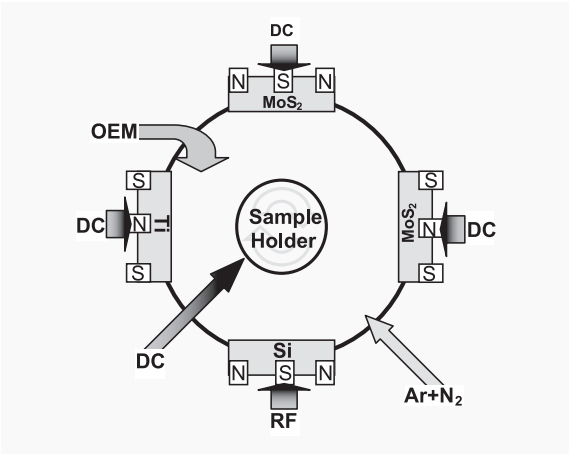

Fig. 1. A schematic diagram of the deposition system for the Ti–Si–N:MoS_x composite films.

For MoS_x incorporation, both the two MoS_2 targets were DC power sputtered. In order to control the MoS_2 incorporation level, the sputtering DC current applied onto the MoS_2 targets was adjusted from 0 to 0.8 A, and correspondingly, the Mo content in the resultant coatings changed from 0 to 13.5 at.%. The unbalanced magnetrons were arranged in a closed-field configuration to provide both a high sputtering rate on the targets and a high plasma density in the zone of deposition [14–18]. High speed steel (HSS) discs of 50-mm diameter were used as substrates, which were ultrasonically cleaned in a series of alkaline solutions and nitrogen blow dried. Then substrates were fixed on a vertical substrate holder which rotates continuously around the central axis at a speed of 10 rpm. Prior to deposition, the vacuum chamber was pumped down to 2×10^{-4} Pa or lower, and the substrates were in situ cleaned with argon plasma at a bias of −500 V for 30 min. During deposition, a negative DC bias of −70 V was applied to the substrate. The argon flow rate was fixed at 18 sccm, while the nitrogen flow rate was automatically controlled through a piezo-valve driven by a computer in order to keep a constant percentage of the optical emission intensity of Ti emission lines at a preset value of 60%. The working pressure was around 0.12 Pa.

The composition of the as-deposited Ti–Si–N:MoS_x coatings was measured by energy dispersive analysis of X-rays (EDX), which is attached to a field emission scanning electron microscopy (Jeol JSM 6340F) system. For crystalline structure analysis, X-ray diffraction (XRD) experiments were performed on a Philips X-ray diffractometer in a θ–2θ scan mode using Cu K_α radiation (40 kV, 30 mA). Microhardness of the coatings was measured using a Nanotest 550 nanoindenter equipped with a Berkovich diamond indenter. The Oliver and Phar [19] method was used for hardness calculation. The maximum indentation depth was set at 200 nm, which is less than one tenth of the coating thickness. The effect of substrate on the hardness was therefore avoided [19]. To evaluate the tribological properties of the Ti–Si–N:MoS_x composite films, sliding wear tests were carried out at ambient atmosphere (relative humidity of 45±10 RH% and temperature of 24±1 °C) using a pin-on-disc tribometer. The tests were performed at a normal load of 5 N using an alumina ball with 9.5 mm in diameter as the wear counterpart. The sliding linear speed and total sliding distance were set at 20 cm/s and 1000 m, respectively. The wear loss and normalized wear rate were calculated from the cross-sectional area of the wear track, as measured by a Talysurf profilometer, and the wear track diameter, i.e.,

$$R = AD/(NL)$$

where R is the measured wear rate in mm^3/N m, A is the cross-sectional area of the wear track in mm^2, D is the diameter of the wear track in mm, N is the normal load applied on the sliding pin in Newton, and L is the total sliding distance during the wear test in meter.