

The tribological characteristics of titanium nitride coatings Part I. Coating thickness effects

Yeong Yan Guu^a, Jen Fin Lin^{a,*}, Chi-Fong Ai^b

^a Department of Mechanical Engineering, National Cheng Kung University, Tainan 70101, Taiwan

^b Physics Division, Institute of Nuclear Energy Research, P.O. Box 3-1, Lung-Tan 325, Taiwan

Received 20 September 1994; accepted 1 March 1995

Abstract

The tribological behavior of specimens coated by two layers, titanium nitride film as the top layer and titanium film as the underlayer, is studied. The coating layers of the bottom specimens were deposited using the cathodic arc ion plating process technique. Experiments were carried out on a wear test machine using a thrust-washer adapter to simulate the surface contacts between the steel ring (the upper specimen) and the titanium nitride coated washer (the bottom specimen). We address the subject about the influence of the thickness of the two coating layers on tribological behavior, wear mechanism, specimen's hardness and adhesive strength. A thin titanium nitride film in combination with a thick titanium film attenuates the adhesive strength, resulting in a significant increase in wear rate. The influence of the titanium nitride or titanium thickness on the friction coefficient is quite limited at higher sliding speeds. The specimen's hardness is increased by thickening the titanium nitride layer, but is lowered by increasing the titanium thickness. When the specimen's sliding speed is increased, both the wear rates and the friction coefficients show significant decline.

Keywords: Titanium nitride; Wear; Adhesion; Cathodic arc

1. Introduction

Titanium nitride (TiN) forms an excellent protective coating because of its hardness, refractory nature and corrosive resistance. In addition, its resistance to wear makes it an excellent tribological coating. However, the tribological behavior of TiN film is found to vary with substrate, deposition method, coating film thickness, stoichiometry, heat treatment and the type of wear. Like most ceramic materials, TiN films are very brittle; they have a great tendency to fracture and spall from the substrate during wear, especially on softer substrates. These tendencies increase with increasing applied load and the film thickness.

In several studies, the effects of the coating layer thickness on both friction and wear behavior were investigated. Bowden and Tabor [1] studied the coefficient of friction; they showed it to decrease as the film thickness decreases, until the film is very thin. For even thinner films, Finkin [2] proposed a theory to explain the phenomenon of increasing friction with the further decrease of the film thickness. His theory uses the applied mechanics of frictional contact approach

instead of the popular explanation of film penetration. Posti and Nieminen [3] studied the influence of coating thickness on the lifetime of titanium-nitride physical vapor deposition (PVD)-coated high speed steel cutting tools. The thickness of the coating was found to have a significant effect on tool life. In the study of Shimura et al. [4], the structural changes and frictional properties of sputtered TiN films on a glass plate were investigated as a function of film thickness, load and the influence of oxidizing. It was concluded that the friction coefficient for TiN under friction is more controlled by the gases absorbed on the interface than by the formation of oxides.

Sherbiny and Halling's study [5] gave experimental results of the tribological behavior of soft metallic films on hard substrates. They showed that the adhesion strength of the film to the substrate is a critical factor determining the film life time span, which demonstrates the value of ion-plated films. Sirvio et al. [6] studied the abrasive wear of ion-plated TiN coatings on both hardened tool steel and on hardened and plasma-nitrided tool steel. They suggested a wear mechanism for hard coatings on softer substrates under abrasive conditions. The wear rate of coated steel increases with increasing initial substrate surface roughness.

* Corresponding author.

Various applications of coating technologies were reviewed concerning wear resistance, corrosion resistance and decorative coatings. Idanowski et al. [7] reviewed the ion plating method for TiN deposition; the basic physical and mechanical features of TiN layers were presented along with the advantages and applications of TiN layers. Randhawa and Johnson's study [8] focused on the cathodic arc plasma deposition process, emphasizing the role of the plasma environment for the reactive deposition of nitrides, carbides, carbonitrides and oxides.

Hedenqvist et al. [9] conducted a new laboratory wear test to study the effects of TiN coating on HSS cutting tool wear. The TiN coating greatly enhanced tool wear resistance. Huang et al. [10] studied the microstructure of Ti/TiN multilayer films, which were deposited by hollow cathode discharge (HCD) ion plating using transmission electron microscopy and X-ray diffraction (XRD). The grain size of multilayer films was observed to closely correlate with multilayer film indentation behavior.

Ai et al. [11] analyzed the characteristics of TiN films, deposited by the means of the HCD ion plating technique, on the surfaces of tungsten, 304 stainless steel and 2014-T6 aluminum alloy, mainly by using high resolution pulsed-laser atom-probe (PLAP) field-ion microscopy (FIM). The XRD detections indicated that the films consisted predominantly of the TiN phase; depth profiling disclosed the existence of a rather thin interface of about 15 atomic layers. The XRD detections also revealed that the impurity, TiO, concentrated not only in this interface but also near the top film surface. Musil et al. [12] studied the properties of TiN, ZrN and ZrTiN coatings prepared by a cathodic arc (CA) ion-plating process. Cathodes made of Ti and Zr were evaporated in an N₂ atmosphere. Special attention was devoted to the coating properties, namely to the microstructure, surface morphology, phase and chemical composition, microhardness and adhesion. Lin and Horng [13] used a thrust-washer adapter to simulate the surface contacts between SKD 61 steel and the TiN coating, which was deposited by the CA plasma technique. The effect of the coating layers on tribological behavior was evaluated for different thickness combinations: the TiN film as the top layer and the electroless nickel film as the underlayer.

In this study, the effects were examined, using two coating layers with different thicknesses, on both the tribological behavior and the adhesion strength. The experiments were carried out using a thrust-washer adapter in a wear test machine. The upper specimens, made of the SKD 61 steel, were tempered to elevate the material hardness. The bottom specimens, which used the SKD 61 steel as the substrate, were coated by various thicknesses of TiN as the top layer and Ti as the underlayer. The coating layers were formed by PVD using the CA ion plating process technique. The tribological behaviors reflecting the changes of the coating layers are expressed in terms of these parameters: wear volume, friction coefficient, wear mechanism, and adhesion strength.

2. Experimental details

2.1. Coating deposition

The coating layers were deposited using the CA ion plating process method [14]. For the coating process, the specimens were heated to about 300 °C in a vacuum deposition chamber at 10^{-5} – 10^{-6} Torr (1.33×10^{-3} – 1.33×10^{-4} Pa). The steered CA plasma source with a titanium arc target was operated with an arc current between 80 and 100 A. After the titanium ion bump at –1000 V bias voltage, the Ti film was deposited with the substrate bias voltage at –200 V. Finally, nitrogen gas with a partial pressure of 2×10^{-3} torr (2.66×10^{-1} Pa) was fed into the vacuum deposition chamber. The TiN film was deposited on the specimens at the temperature of 400 °C. The end-process surface roughness of the bottom specimens was about 0.3–0.4 mm Ra.

2.2. Friction and wear tester and dimensions of specimens

The friction and wear tests were carried out in a multi-specimen wear testing machine using a thrust-washer adapter. The ring, which was rotating at a constant sliding speed during the wear process, was pressed against a stationary washer to simulate the tribological behavior of surface contacts. No lubricant was used in the experimental process. Fig. 1 shows a schematic diagram of the thrust-washer adapter assembly.

The substrates of the upper and bottom specimens were both made of the same material, JIS G4404 SKD61 steel. The steel rings for the upper specimens were tempered at a temperature of 825 °C to obtain a hardness of 20–24 HRC. The substrates of the bottom specimens were quenched first at 1020 °C; then tempered at 560 °C so that the hardness was increased to 50–55 HRC. Before depositing the two coating layers, the bottom specimens were finely polished to a surface roughness of 0.3 mm Ra. The dimensions of the upper and lower specimens were prepared according to the ASTM D3702-78 standard test method [15]. These specimens were tested under the same load of 222.4 N, but at two different sliding speeds, 0.705 m s^{-1} and 1.41 m s^{-1} , respectively. The sliding motion terminated at a distance of 2023 m.

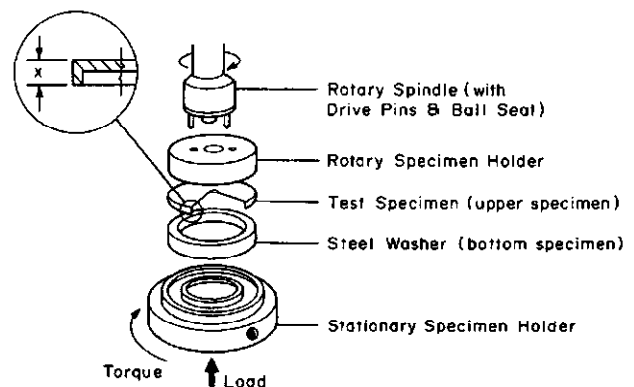


Fig. 1. Schematic diagram of the thrust-washer adapter.

2.3. Hardness and adhesion tests

The hardness of the coating specimens could be found using a Vickers microhardness tester under a load of 100 gf (1 N) for a duration of 15 s. The indentation method was used to evaluate the adhesion strength of the coating layers [16,17]. Simple hardness tests were carried out on a Rockwell hardness tester under a load of 150 kg (1500 N), applying a diamond of the C scale to the sample surfaces. The resulting surface indentation fractures were examined using optical microscopy.

3. Results and discussion

3.1. The influence of titanium nitride thickness on tribological behavior

The experimental data shown in Table 1 reveals the influence of TiN thickness on the bottom specimen's wear rates. The wear rate of the bottom specimen is evidently related to the combined effects of three controlling factors: the TiN coating thickness (as the top layer), the Ti film thickness (as the underlayer), and the sliding velocity. The wear rates for the thinnest TiN films (at 1 mm), at the sliding speed of 0.705 m s^{-1} , were the largest for all three Ti thicknesses (0, 0.1 and 0.5 mm); particularly when compared with the other two TiN layer thicknesses of 3 and 5 mm. When the sliding speed was increased to 1.41 m s^{-1} , the bottom specimens with a TiN film thickness of 1 mm and the Ti film of 0.5 mm yielded a particularly large wear rate. The experimental results show that an extremely thin TiN film, in combination

with a thick Ti film, greatly attenuates the adhesive strength which bonds the substrate to the TiN film, resulting in a significant increase in wear rate. Similarly, a thick TiN film (5 mm), for the purpose of lowering the wear rate, has to have a thin titanium underlayer.

A thick TiN film (5 mm) as top layer over a thick Ti underlayer (0.5 mm) somewhat reduces the wear rate, although the reduction is less than for the case of a thinner TiN film (3 mm) combined with a thick titanium layer (0.5 mm).

For the coating specimens without the Ti underlayer, the wear rate is evidently related to the adhesion strength of the TiN film. If the TiN layer thickness is too thin (1 mm), the specimens cannot bear the high shear force for the sliding motions of 0.705 m s^{-1} , thus resulting in a large wear rate (of over 12 times that of the 3 mm film). However, when the thickness of the top layer is very high (5 mm), the adhesive strength is also attenuated substantially, thus causing the wear rate to increase (of over twice the 3 mm value). There exists an optimum thickness (around 3 mm) for the top layer such that the least wear rate is created.

The influence of TiN thickness on the friction coefficient is rather limited, even when the coating thickness varied between 1 mm to 5 mm. That is, most of the friction coefficients varied in a narrow range regarding the friction contacts sliding at a fixed speed. Relatively higher friction coefficients exist only for the specimens with a TiN thickness of 3 mm, with a Ti underlayer, sliding at the speed of 0.705 m s^{-1} , and all TiN thickness of 5 mm, sliding at the speed of 1.41 m s^{-1} ; these specimens were coated by the same titanium thickness of 0.5 mm.

Table 1

The wear rates of the disk specimens and the friction coefficients varying with the thicknesses of two coating layers

Case No.	Thickness of TiN film (μm)	Thickness of Ti film (μm)	Load(N)	Sliding speed (m s^{-1})	Sliding distance (m)	Friction coefficient	Wear rate $\times 10^{-3}$ ($\text{mm}^3 \text{m}^{-1}$)
1	1					0.50–0.58	1.704
2	3	0	222.4	0.705	2032	0.49–0.58	0.125
3	5					0.52–0.64	0.262
4	1					0.48–0.52	0.993
5	3	0.1	222.4	0.705	2032	0.48–0.58	0.257
6	5					0.46–0.54	0.129
7	1					0.47–0.54	4.394
8	3	0.5	222.4	0.705	2032	0.57–0.63	0.187
9	5					0.47–0.58	0.144
	1					0.37–0.42	0.040
	3	0	222.4	1.41	2032	0.38–0.44	0.065
	5					0.39–0.44	0.050
	1					0.35–0.46	0.032
	3	0.1	222.4	1.41	2032	0.36–0.43	0.032
	5					0.35–0.44	0.021
	1					0.40–0.46	0.217
	3	0.5	222.4	1.41	2032	0.35–0.41	0.014
	5					0.34–0.40	0.022
				0.705		0.46–0.53	3.742
	0	0	222.4	1.41	2032	0.42–0.47	0.324

The significant rise in material hardness enhances the brittleness of the coating layer and thus increase the stress required to shear the asperity contacts. The friction coefficients for the coating specimens are normally confined to a narrow range regardless of the thickness of the coating layers. However, those coefficients are slightly greater than for metal surfaces without any coatings. From analysis of our results, the friction coefficient seems to be affected slightly by the hardness of the substrate when the hard TiN film is deposited as the sole coating layer and when the wear depth is smaller than the coating thickness. It is noticeable that the friction coefficients corresponding to the specimens with TiN film only yield an appreciable increase at the sliding speed of 0.705 m s^{-1} when the thickness of the TiN film is increased from 3 mm to 5 mm. A reasonable interpretation for this behavior is that the brittleness of the coating layer is enhanced as the TiN thickness is increased. The higher shear strength of the coating layers encourages a slight elevation in the friction coefficient. The friction coefficient increase becomes trivial when the sliding speed is increased to 1.41 m s^{-1} .

3.2. Influence of titanium thickness on tribological behavior

The influence of the Ti thickness on the wear rate depends upon the thickness of TiN and the sliding velocity of the specimens (see Table 1). At the sliding speed of 0.705 m s^{-1} , the experimental results on wear rate show that the specimens coated by a 1 mm TiN film may cause an increase in wear rate when the Ti layer is thickened from 0.1 to 0.5 mm. Also, a thicker TiN film (5 mm) incorporating a Ti layer (0.1–0.5 mm) reduces the wear rate of the bottom specimen. The wear rates for the 1 mm TiN film are higher than for the 5 mm film. The lowest wear rates were for the specimens with a TiN thickness of 3 mm (of the four thicknesses tested) and a Ti thickness of 0.5 mm (the largest of the three thicknesses tested).

The same effect of Ti thickness on wear rate is reflected from the results with 1.41 m s^{-1} as the sliding speed; however, the wear rate is considerably less than for the 0.705 m s^{-1} sliding speed. Regardless of the sliding speeds, the highest wear rates exist with the specimens which combine a thin TiN film (1 mm) with a thick Ti layer (0.5 mm).

Generally, when the workpieces in frictional contact operated at a fixed sliding speed, most of the friction coefficients varied in a narrow range, even when the Ti layer was thickened from 0 mm to 0.5 mm.

Notable rises in the friction coefficient, owing to the increase in Ti coating thickness, appear in the specimens with the same 0.5 mm Ti thickness but with two different sliding speeds, with the TiN thicknesses being 3 mm and 5 mm, respectively. Our investigation showed that the friction coefficients are reduced slightly if the thickness of the Ti film is increased from 0 to 0.1 mm. Normally, both the hardness of the coatings and the substrate are the two controlling factors governing surface fracturing, so when the softer titanium film

is deposited as the underlayer, the brittleness of the outer coating layer is attenuated. Lower shear strength in the softer material helps to lower the friction coefficient.

3.3. Influence of sliding speed on wear rate and friction coefficient

The specimen's sliding speed is an influential factor for determining its wear rate and friction coefficient. As the sliding speed is elevated from 0.705 m s^{-1} to 1.41 m s^{-1} , both the wear rates and the friction coefficients show significant declines. The reductions in wear rate become particularly pronounced for the cases of having large wear rate at the sliding speed of 0.705 m s^{-1} , especially for a TiN thickness of 1 mm. The friction coefficients drop from the range of 0.46–0.63 for 0.705 m s^{-1} to 0.34–0.46 for 1.41 m s^{-1} (see Table 1).

The tribological behavior related to the friction coefficient decline is attributable to the shearing of the thin TiN film, and to the extremely high rates of strain that may occur within the film itself. When specimens slide at higher speeds, we can view the frictional hot spots as adiabatic; here the small contact regions are rapidly sheared away, so heat is generated at a rate perhaps much faster than the rate at which it can be conducted away. Such intense localized heating will substantially reduce the friction coefficient. If softening and plastic flow of asperities start to occur at a lower speed, both the friction and the wear rates would decrease while increasing the sliding speed.

3.4. Influences of the thickness of top layer and underlayer on specimen's temperature

The variations of the thermal conductivities with temperature and the thicknesses of two coating layers obviously dominate the specimen's temperature. Table 2 shows the temperature measures at the depth of 2.5 mm beneath the substrate surface. The elevations in specimen temperature are significant when the sliding speed is increased, especially for the titanium nitride layer with a thin thickness of 1 mm. An interesting characteristic is demonstrated in the data for the titanium nitride layer with a fixed thickness. Irrespective of the sliding speed, the differences between the maximum and minimum temperatures which result from the changes in titanium thickness are enhanced if the top layer is formed by a thin TiN film. That is, the effect of the underlayer thickness

Table 2
The measured temperatures (in °C) at the depth of 2.5 mm beneath the substrate surface*

	TiN = 1 μm	TiN = 3 μm	TiN = 5 μm
Ti = 0 μm	230	307	300
Ti = 0.1 μm	276	288	309
Ti = 0.5 μm	296	316	292

*Load, 222.4 N; sliding speed, 0.705 m s^{-1} ; sliding distance, 2032 m.



Fig. 2. The topography of the disk with the titanium nitride as the top layer.

on specimen temperature becomes significant only in the specimens with a thin top layer. Both the complicated behavior in the thermal conductivities of the two coating layers and the friction coefficient during the wearing process are responsible for the random variations in specimen temperature with the titanium thickness.

3.5. Wear mechanisms and debris

Fig. 2 shows a specimen photograph before wear tests. The white particles exhibited on the surface were identified as the microparticles formed during the deposition process.

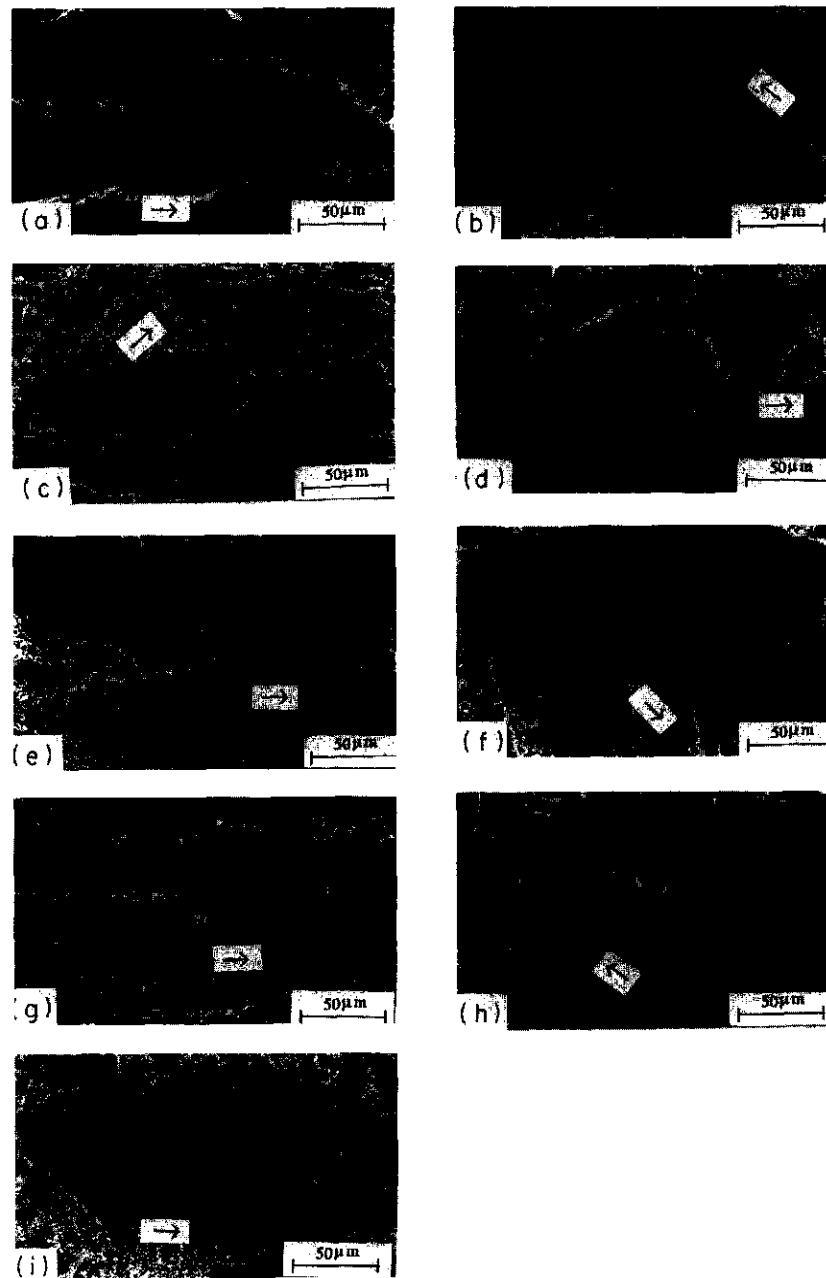


Fig. 3. Scanning electron micrographs of the wear mechanisms of the bottom specimens varying with the thickness combinations of two coating layers. The operation conditions include: the sliding speed, 0.705 m s^{-1} ; the applied load, 222.4 N; the sliding distance, 2032 m. (a) TiN: $1 \mu\text{m}$, Ti: $0 \mu\text{m}$; (b) TiN: $3 \mu\text{m}$, Ti: $0 \mu\text{m}$; (c) TiN: $5 \mu\text{m}$, Ti: $0 \mu\text{m}$; (d) TiN: $1 \mu\text{m}$, Ti: $0.1 \mu\text{m}$; (e) TiN: $3 \mu\text{m}$, Ti: $0.1 \mu\text{m}$; (f) TiN: $5 \mu\text{m}$, Ti: $0.1 \mu\text{m}$; (g) TiN: $1 \mu\text{m}$, Ti: $0.5 \mu\text{m}$; (h) TiN: $3 \mu\text{m}$, Ti: $0.5 \mu\text{m}$; (i) TiN: $5 \mu\text{m}$, Ti: $0.5 \mu\text{m}$. The arrows indicate the sliding direction.

The amount of surface deterioration exhibited, in terms of wear mechanisms, is somewhat dependent upon the thickness variations of two coating layers. Fig. 3(a)–(i) show photographs of the bottom specimen's worn surfaces using different combinations of the two coating layers with different thicknesses. The photos were selected for specimens with TiN thicknesses of 1 μm and 5 μm , in combination with various Ti underlayer thicknesses.

The micrographs for cases 1 and 7 (Fig. 3, (a) and (g)) show the worn surfaces with deep grooves, parallel to the sliding direction; the grooves were produced in the underlying material after the coating layers had been removed. Fig. 3(g) shows the grooves created on a specimen of case 7. Most of the worn surfaces are strongly metallic and part of the steel substrate was exposed during frictional contacts. Intersecting cracking would cause segments of the TiN coating to be plucked from the surface. Once the coating films had been removed by cracking and plucking mechanisms, then hard particles ploughed grooves. The presence of the grooves caused larger wear losses.

The micrographs in Fig. 3, parts (b)–(i), of the worn surfaces, all show a common feature; instead of deep grooves, the dominant wear mechanisms formed only in to a shallow depth beneath the contact surface, were the surface delaminations which result from the plucking mechanism, and microcracks. Fig. 4 shows the wear mechanism for the specimen of case 8. Entire regions of the worn surface are basically covered by TiN and Ti films; Nearly no metallic evidence could be detected. Obviously, the wear behavior takes place in the coating layers. The wear losses corresponding to this kind of surface fracture are relatively smaller compared to the cases with deep grooves.

The adhesive wear appearing within the metal–ceramic contacts is strongly dependent upon the sliding speed. In this study, severe adhesions were formed in the bottom specimens at the sliding speed of 1.41 m s^{-1} . They are marked by the letter A in the photograph of Fig. 4. The weight changes of the bottom specimens in some cases were measured as posi-

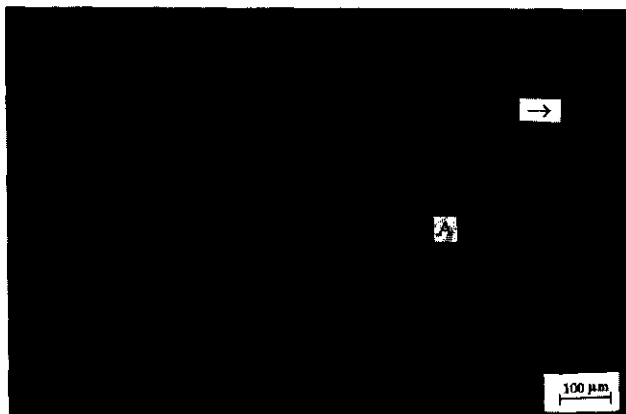


Fig. 4. The photograph showing the wear mechanism of the bottom specimen with a TiN thickness of $3 \mu\text{m}$ and a Ti thickness of $0.5 \mu\text{m}$. The operation conditions include: the sliding speed, 1.41 m s^{-1} ; the applied load, 222.4 N ; the sliding distance, 2032 m . Mark A, adhesive material.

tive as shown in Fig. 5. That is, the material adhesions from the upper specimens caused the weight of the bottom specimen to increase after wearing. However, when the sliding speed was reduced to 0.705 m s^{-1} , the amount of adhesive wear was greatly lowered; an increase in the weight of the bottom specimen did not happen in all five cases.

The size of debris produced by the specimens offers another way to observe relation of debris with specimen's wear loss. Fig. 6(a) and Fig. 6(b) show the debris from the specimens corresponding to cases 4 and 7. The debris is the product related to the smaller and larger wear losses, respectively. The debris shown in Fig. 6(a) from case 4 is shown

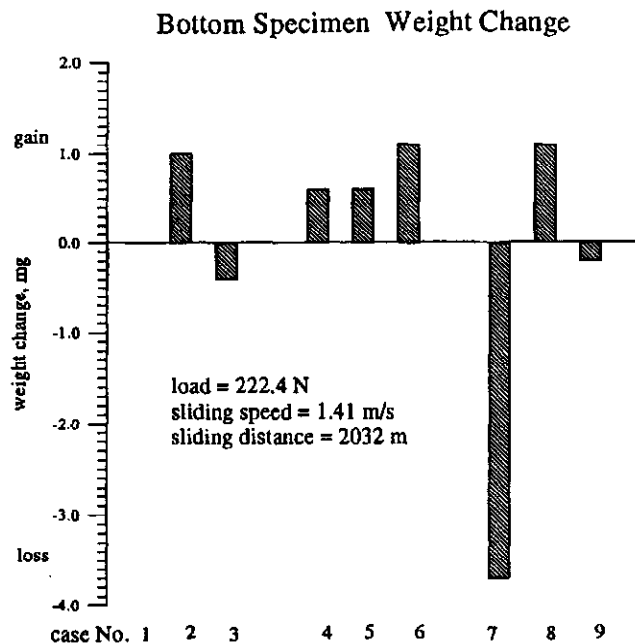


Fig. 5. The weight change of the bottom specimens under the conditions: the sliding speed, 1.41 m s^{-1} ; the applied load, 222.4 N ; the sliding distance, 2032 m .

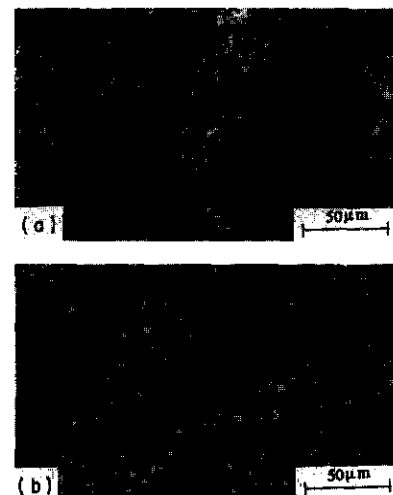


Fig. 6. The wear debris yielded in the specimens under the load of 222.4 N , at the sliding speed of 0.705 m s^{-1} , and the sliding distance 2032 m . The thicknesses of two coating layers are (a) TiN: $1 \mu\text{m}$, Ti: $0.1 \mu\text{m}$ and (b) TiN: $1 \mu\text{m}$, Ti: $0.5 \mu\text{m}$.

to be fine particles; the wear loss is thereby ascertained to be low. Conversely, some chunks which came from the specimen surface grooving of case 7 are present in Fig. 6(b), the wear loss is greatly increased.

3.6. Hardness tests

The hardness of the specimens with two coating layers were determined by using Vickers microhardness tester under the load of 100 gf (1 N) for the duration of 15 s. It must be noticed that the measured hardness is a mixture of the hardness of the top layer, the underlayer and the softer substrate. The data in Table 3 shows the average hardness of the specimens with various combinations of the two coating layers. The specimen's hardness is certainly increased by thickening the harder TiN layer. Conversely, increasing the thickness of the softer Ti layer causes the hardness to decline. Consequently, the specimens with a thick TiN layer in combination with a thin Ti layer normally possess a higher hardness. The hardness of the specimens with the 5 mm thick TiN film without the Ti layer has the greatest hardness. Generally speaking, an increase in specimen hardness probably helps to lower the wear loss, but increasing the hardness of a specimen does not always mean it will have a smaller wear loss.

3.7. Adhesion characteristics

In the present study, titanium film is deposited as the underlayer. The choice of titanium as the underlayer is based on the viewpoint that: (1) the depositions of both the top layer (titanium nitride film) and the underlayer (titanium film) can be directly completed in a chamber without moving the specimens out of the chamber in order to coating the underlayer; this technique saves the cost of coating rig; (2) the plating of titanium as the underlayer alleviates the significant difference in material properties between the titanium nitride film and the steel substrate when the specimen is subject to thermal stress effect during the frictional contacts; (3) the thermal conductivity of the titanium film is quite lower, this

feature encourages the lowering of thermal stress effect in the deposition process.

The force which is needed to separate the coatings from the substrate is considered to be the adhesive strength. The indentation tests were conducted on a Rockwell hardness tester, applying a diamond of the C scale to the surface of the sample. The resulting damage to the coating around the indentation was examined using optical microscopy. The indentation depths measured from the microhardness tests are certainly affected by the specimen's hardness. The greater the specimen's hardness is, the smaller the indentation depth will be. Increasing the thickness of the softer Ti underlayer reduces the hardness, resulting in an increased indentation depth. Conversely, increasing the thickness of the top TiN layer decreases the indentation depth.

Table 3 shows the indentation depth values for workpieces with various coating thicknesses. The experimental results for indentation depth revealed that the indenter penetrated through the coating layers to the steel substrate when the specimens were coated by a TiN film only 1 mm thick as the top layer. However, the indenter still stayed within the coating layers under the same load when the thickness of the top layer was either 3 mm or 5 mm. The correlation of the indentation depth with the formation of surface cracks will be discussed in following paragraphs.

The quantity of surface fractures in the coatings resulting from indentation force is strongly dependent upon both the thicknesses of the two coating layers and the indentation depth. As shown in Fig. 7, parts (a), (e) and (g) for cases 1, 4 and 7, respectively, if the indentation depth is larger than the combined thickness of two coating layers, the surface cracks scatter sparsely within an annulus around the indentation circle.

The above phenomena seemingly contradicts the anticipation that the specimens with a thin Ti layer thickness should have more severe surface cracks. A reasonable interpretation for this characteristic is that since the indenter penetrated through the coating layers reaching to the steel substrate, the substrate bears part of the direct indentation vectored force,

Table 3
The hardness of disk specimens and the indentation depths^a

Case No.	Ti film (μm)	TiN film (μm)	Average Vickers hardness (Hv)	Indentation depth (μm)
1	0	1	1087	2.6
2	0	3	1381	2.3
3	0	5	2045	1.9
4	0.1	1	1084	2.6
5	0.1	3	1328	2.4
6	0.1	5	1865	2.0
7	0.5	1	931	2.9
8	0.5	3	1161	2.6
9	0.5	5	1711	2.1

^aIndentation load, 100 gf (1 N).

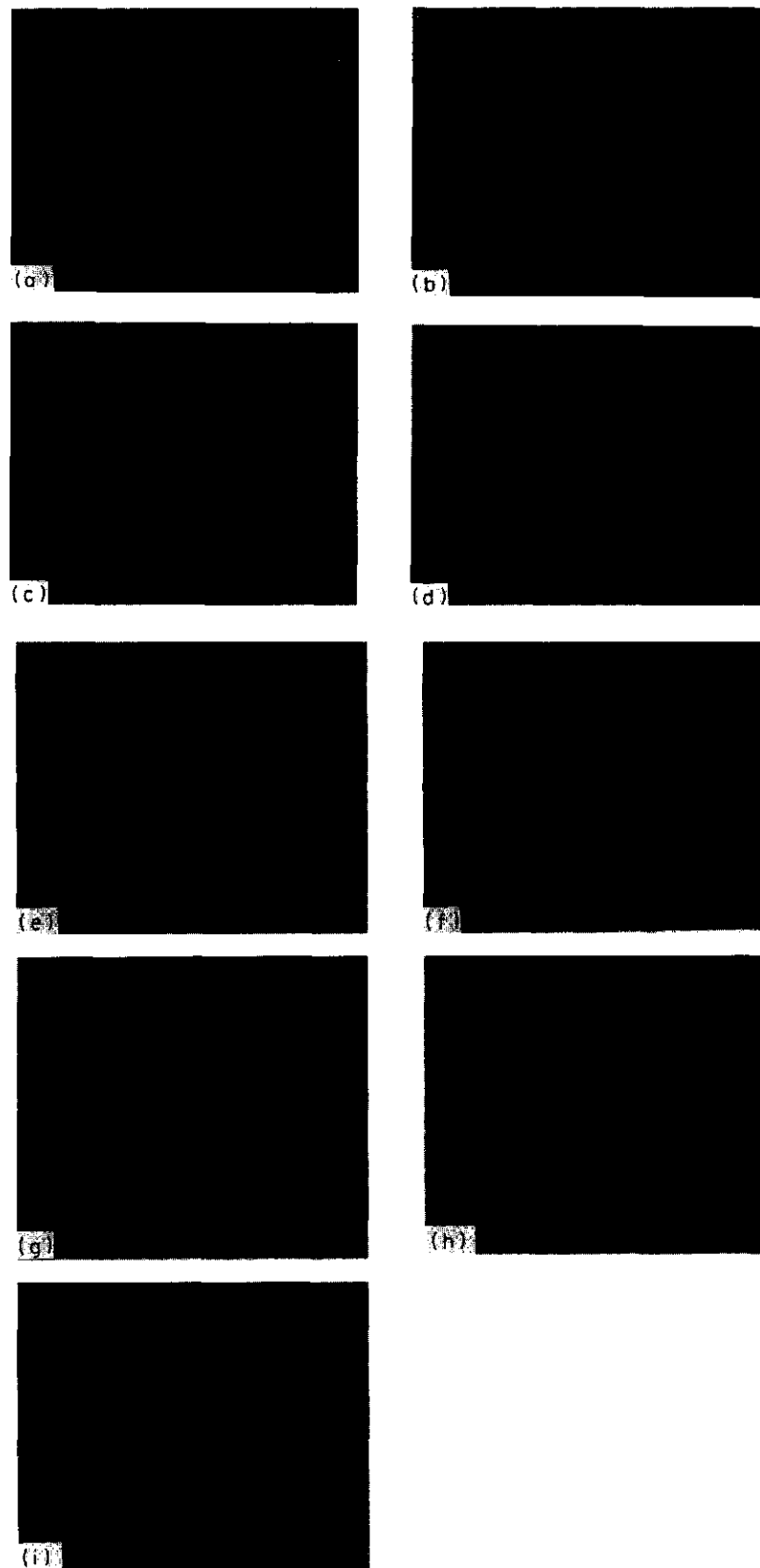


Fig. 7. The surface failures due to indentation tests of the specimens with various thickness combinations of two coating layers. (a) TiN: 1 μm , Ti: 0 μm ; (b) TiN: 3 μm , Ti: 0 μm ; (c) TiN: 5 μm , Ti: 0 μm ; (d) TiN: 1 μm , Ti: 0.1 μm ; (e) TiN: 3 μm , Ti: 0.1 μm ; (f) TiN: 5 μm , Ti: 0.1 μm ; (g) TiN: 1 μm , Ti: 0.5 μm ; (h) TiN: 3 μm , Ti: 0.5 μm ; (i) TiN: 5 μm , Ti: 0.5 μm .

greatly reducing the force vectored upon the coating layers, thus resulting in the sparse quantity of cracks present on the fractured surface.

The cracking fractures exhibited in the specimens with a TiN layer 3 mm or 5 mm thick are much denser than the specimens with a 1 mm TiN thick layer. According to the data shown in Table 3, the penetrating depth of the indenter is actually less than the combined thickness of two coating layers. The normal force owing to indentation was mostly supported by the two coating layers; consequently, the coating layers absorbed most of the indentation energy, resulting in both dense cracking and even some delaminations within the coating layers. The photographs for thick TiN layers are shown in Fig. 7, parts (b), (c), (e), (f), (h) and (i).

We have therefore concluded that the adhesive strength of the coating layers cannot be identified by the cracks formed on the surface only. The penetrating depth of the indenter should also be taken into account before the correlation of adhesion strength with cracking fractures is determined.

Several other additional characteristics about adhesion strength can be observed by differing the thickness of either the TiN layer or the Ti layer. The micrographs of the indentations shown in Fig. 7, parts (b), (c), (e), (f), (h) and (i), are for the specimens with top layer thicknesses: of either 3 mm or 5 mm; the Ti underlayer thickness is 0 mm, 0.1 mm or 0.5 mm. The quantity of cracks is related to the thickness of two coating layers. If the underlayer is held at a constant thickness, and if the TiN top layer thickness is increased from 3 mm to 5 mm, the quantity of cracks increases and the cracking fractures scatter over a wider area. The surface cracking increase becomes especially noticeable for the specimens with a thick Ti underlayer.

It is interesting to note that the surface fracturing observed on the specimens without an underlayer is quantitatively almost the same for both the 3 mm thick TiN layer and the 5 mm one. That indicates the adhesion strength of the coating layer is attenuated by increasing the top layer thickness. No notable difference in adhesion strength appears in the specimens with different thicknesses of the top layer without a Ti underlayer.

The amount of surface cracking can be reduced substantially by increasing the thickness of the Ti underlayer while keeping the top layer at a constant thickness. If the top layer is 3 mm, there is an effective reduction in surface cracking. So increasing the thickness of the Ti underlayer enhances the adhesion strength. We concluded while experimentally differing both the thicknesses of the TiN top layer and the Ti underlayer, the adhesion strength of the coating layers is effectively enhanced if increasing the thickness of the underlayer is in conjunction with a decrease in the top layer thickness.

The wear loss corresponding to a specimen with a higher adhesion strength is normally lower. The relatively smaller wear losses for cases 2, 3, 5, 6, 8 and 9 are due to having a higher adhesion strength as assessed by the indentation tests. On the other hand, the larger wear losses of cases 1, 4 and 7

are believed to result from the lower adhesive strength of the coating layers. The specimens for case 7, which is the combination of the thinnest TiN top layer (1 mm) with the thickest Ti underlayer (0.5 mm), evidently causes the largest wear volume of the nine cases.

4. Conclusions

The tribological behavior of the bottom specimens, coated by a titanium nitride film for the top layer and a titanium film for the underlayer, is dominated by the following factors: the thickness of two coating layers, the material of the upper and bottom specimens and the sliding velocity. The frictional and wear performances of the coating layers can be evaluated in terms of wear loss, the friction coefficient, the wear mechanism, wear debris, the specimen's hardness and indentation fracture. The conclusions drawn are as follows.

1. A thin titanium nitride film in combination with a thick titanium underlayer attenuates the adhesive strength, resulting in a significant increase in wear rate. Conversely, a thick titanium nitride film, for the purpose of lowering wear loss, has to incorporate a thin titanium film underlayer.
2. At higher sliding speeds, the influence of either the titanium nitride or titanium thickness on the friction coefficient, is quite limited. However, the friction coefficients at lower sliding speeds are slightly affected by the thicknesses of the two coating layers.
3. The specimen's hardness is increased by thickening the titanium nitride layer, but is lowered by thickening the titanium layer. The significant increase in material hardness will enhance the brittleness of the coating layers; the friction coefficients as to the harder materials are, often but not always, relatively higher. Conversely, the lowering a specimen's hardness results in the decline of the friction coefficient.
4. When the specimen's sliding speed is increased, both the wear rates and the friction coefficients show significant decline.
5. The adhesion strength of the coating layers cannot be measured only by the quantity of cracks formed on the surface; the penetration depth of the indenter should also be taken into account.
6. Adhesive wear becomes severe for the ceramic–ceramic coatings as the sliding speed is increased from 0.705 m s^{-1} to 1.41 m s^{-1} .

References

- [1] F.P. Bowden and D. Tabor, Friction and Lubrication Report No. 1, Bulletin No. 145, Council for Scientific and Industrial Research, Melbourne, 1942; *The Friction and Lubrication of Solids*, Oxford University Press, London, 1954.
- [2] E.F. Finkin, A theory for the friction of sulfide and other thin films, *Wear*, 18 (1971) 231–241.

- [3] E. Posti and I. Nieminen, Coating thickness effects on the life of titanium nitride PVD coated tools, *Mater. Manuf. Process.*, 4(2) (1989) 239–252.
- [4] Y. Shimura, T. Ito, Y. Taga and K. Nakajima, Frictional properties of sputtered TiN film, *Wear*, 49 (1978) 179–193.
- [5] M.A. Sherbiny and J. Halling, Friction and wear of ion-plated soft metallic films, *Wear*, 45 (1977) 211–220.
- [6] E.H. Sirvio, M. Sulonen and H. Sundquist, Abrasive wear of ion-plated titanium nitride coatings on plasma-nitride steel surfaces, *Thin Solid Films*, 96 (1982) 93–101.
- [7] J. Idanowski, J. Markowski and W. Oleszkiewicz, Modification of metal properties by ion plating of TiN, *Vacuum*, 36(1) (1986) 591–594.
- [8] H. Randhawa and P.C. Johnson, Technical note: a review of cathodic arc plasma deposition processes and their applications, *Surf. Coatings Technol.*, 31 (1987) 303–318.
- [9] P. Hedenqvist, M. Olsson and S. Soderberg, Influence of TiN coating on wear of high speed steel tools as studied by new laboratory wear test, *Surf. Eng.*, 5(2) (1989) 141–150.
- [10] R.F. Huang, L.S. Wen, L.P. Guo, J. Gong, B.H. Yu and H. Bangert, Microstructural and indentational characterization of Ti/TiN multilayer films, *Surf. Coatings Technol.* V50(2) (1992) 97–101.
- [11] C.F. Ai, J.Y. Wu and C.S. Lee, Study of HCD ion plated titanium nitride films, *Vacuum*, V44(2) (1993) 99–104.
- [12] J. Musil, I. Stepanek, J. Jr. Musil, M. Kolega, O. Blahora, J. Vyskocil and J. Kasl, Properties of TiN, ZrN and ZrTiN coatings prepared by cathodic arc evaporation, *Mater. Process.*, VA163(2) (1993) 211–214.
- [13] J.F. Lin and J.H. Horng, Analysis of the tribological behavior and wear mechanisms of titanium nitride coating, *Wear*, 171 (1994) 59–69.
- [14] C.F. Ai, Characterization of plasma-aid PVD processes, *J. Vacuum Sci. ROC.*, 6(2) (1993) 37–45.
- [15] ASTM Standard, *Standard test method for wear rate of materials in self-lubricated rubbing contact using a thrust washer testing machine*, D3702, Vol. 5–3, 1983, pp. 417–423.
- [16] T. Arai, H. Fujita and M. Watanabe, Valuation of adhesion strength of thin hard coatings, *Thin Solid Films*, 154 (1987) 387–401.
- [17] P.R. Chalker, S.J. Bull and D.S. Rickerby, A review of the methods for the evaluation of coating–substrate adhesion, *Mater. Sci. Eng.*, A140 (1991) 583–592.