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Adhesion and Wear Resistance of Thin Hard Coatings

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Abstract

We investigated adhesion and tribological properties of TiN and TiCN coatings prepared by physical vapour deposition (PVD) on an HSS steel substrate. Adhesion was studied by scratch test while the tribological properties were investigated using a pin-on-disk tribometer. TiCN shows higher adhesion to substrate and better wear resistance than TiN. © 1997 Elsevier Science Limited.

Résumé

Nous avons étudié l'adhérence et les propriétés tribologiques des revêtements TiN et TiCN élaborés en phase vapeur par PVD, sur un substrat en acier rapide. L'adhérence des revêtements a été qualifiée grâce au test de la rayure et les propriétés tribologiques ont été étudiées à l'aide d'un tribométre pion-plan. Le revêtement TiCN montre une meilleure adhérece au substrat et une résistance à l'usure supérieure à celle du TiN.

1 Introduction

Thin hard coatings deposited by chemical vapour deposition (CVD) or physical vapour deposition (PVD) can improve significantly the tribological performance of tools and machine parts. TiN and TiCN coatings produced by these techniques increase greatly tool lifetime.[1-3]

The reactions of CVD deposition takes place at a temperature of about 1000°C and thus its range of application is limited. A much lower deposition temperature can be achieved with PVD processes which operate at a temperature between 400 and 600°C.

Among the PVD techniques for TiN and TiCN coatings, the reactive ion plating is one of the most commonly used. Films with dense structure and more adhesion than the CVD ones may be obtained.[3]

In tribological applications, especially when abrasion is the dominant wear mechanism, TiCN is superior to TiN coatings due to (1) its higher hardness (3000 Hv against 2000 Hv for the nitride) and (2) the presence of carbone which acts as a lubricant leading to reduced friction and wear.[4,5]

In mechanical applications, the coating adhesion to the surface of the substrate is one of the most important properties. Adhesion strength of coating is widely studied using the scratch test. The latter consists of introducing stresses by deforming the surface by means of indentation with a moving diamond tip. The applied load is increased either stepwise or continuously until the film detachment. The smallest load at which the coating is damaged is called the critical load Lc.

In the present paper, we have studied adhesion and tribological properties of TiN and TiCN films deposited on Z85WDCV6542 steel with a hardness of 880 Hv. The friction tests were conducted against two kinds of materials, 100C6 steel and alumina.

2 Experimental Details

The scratch tests were performed using the CSEM-Revetest automatic apparatus fitted with a Rockwell C diamond stylus. Tests were carried out using a loading rate of 100 $N\,min^{-1}$ and a table speed of 10 $mm\,min^{-1}$. The resultant profiles of the acoustic emission (AE) signal intensity and tangential force are plotted versus load (Fig. 1). The critical load Lc for coating to fail (by spalling) corresponds to an abrupt increase of AE signal and is confirmed by scanning electron microscope examination.

Sliding tests were carried out using a pin-on-disk tribometer. The experiments were conducted in air at room temperature. The riders were spheres of polycrystalline alumina or 100C6 steel with a diameter of 5 mm. The motion was reciprocal and the length of the wear track was 15 mm. Test conditions are shown in Table 1.

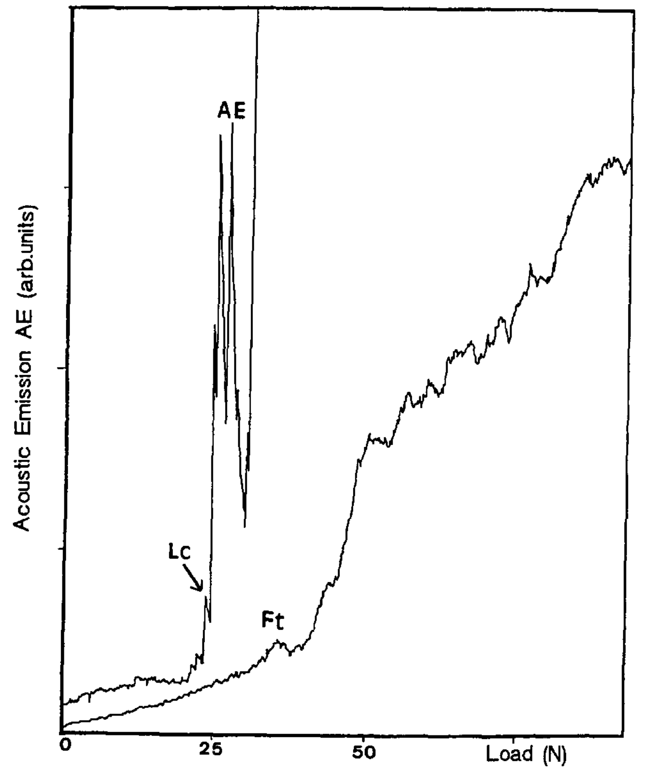

Fig. 1. Acoustic emission signal intensity (AE) and tangential force (Ft) as a function of normal load. Lc is the critical load.

The substrate was an HSS steel (Z85WDCV6542) with a hardness of 880 Hv. It was first polished with SiC paper. Smooth surfaces with a roughness of $Ra = 0{\cdot}02\,\mu m$ were obtained after polishing the samples with diamond pastes down to $1\,\mu m$.

The friction coefficient was measured continuously during the sliding tests. Worn surfaces were investigated by scanning electron microscopy and wear volumes were measured by three-dimensional profilometry.

TiN and TiCN were obtained by reactive ion plating.[6,7] Before deposition, the substrate to be coated was ion-cleaned with argon for 15 min. Titanium vapour was created by evaporation of titanium by means of an electron beam. Before the reactive gas (nitrogen) was admitted into the evaporation chamber, a titanium layer of about 200 nm thick was deposited on the substrate in order to improve the adhesion of TiN film. The latter was deposited using a substrate bias of −200 V at a temperature of 500°C. The deposition rate was $3\,\mu m\,h^{-1}$. TiCN coating was produced by adding acetylene gas to N_2 during deposition.

3 Results and Discussion

3.1 Characterization of the coatings

Vickers micro-hardness measurements were performed using a light load (15 g) in order to make negligible the influence of substrate on hardness measurements. Five indentations were made and diagonals were measured using a scanning electron microscope. The hardness values were averaged to obtain the following results: TiN (2100 ± 50 Hv), TiCN (2600 ± 65 Hv).

X-ray diffraction using Cu K_α radiation was conducted. The results show that TiN and TiCN have an f.c.c. structure with a strong (111) preferred orientation (Fig. 2). The composition of TiN and TiCN was analysed by energy dispersive X-ray spectroscopy (EDS). An atomic ratio Ti/N of 0·9 was found for TiN while the average stoichiometry of TiCN was $Ti_{44{\cdot}2}C_{33{\cdot}3}N_{22{\cdot}5}$.

Figure 3 shows depth profiles obtained by glow discharge optical spectrometry (GDOS) on $3\,\mu m$-thick TiN and TiCN coatings. The interface shows an inter-diffusion layer of less than $1\,\mu m$. Adhesion of the coating to the subtrate was investigated using the scratch test technique as described in Section 2. The critical scratch-test loads obtained for TiN and TiCN were, respectively, 25 and 39 N indicating that the adhesion strength of titanium carbonitride is higher than that of titanium nitride.

3.2 Friction against 100C6 steel ball

Figure 4 shows the evolution of friction coefficient (μ) as a function of sliding distance. The starting values of the friction coefficient are, respectively, 0·5 and 0·3 for TiN and TiCN. They increase gradually during sliding until they reach a plateau. The latter is 0·63 for TiN and 0·53 for TiCN. SEM observations of the wear track show large metal transfer from steel onto TiN (Fig. 5). The metal transfer was lower for TiCN. Figure 6 shows the volume wear values of steel after sliding against the coatings.

Table 1. Test conditions

Test conditions	*System*	
	TiN (or TiCN) / steel ball	*TiN (or TiCN) / alumina ball*
Normal load (N)	10	25
Sliding velocity ($mm\,s^{-1}$)	2	2
Total sliding distance (m)	8	8
Relative humidity (± 5%)	50	50
Temperature (± 3°C)	19	19

The higher wear volume was found after sliding against TiN (the softer coating) which presents the higher friction coefficient (Fig. 4). This result shows that the wear of the steel ball does not increase with the hardness of the coating but depends on the friction coefficient between steel and the film. On the other hand, the coatings did not suffer any wear.

3.3 Friction against alumina ball

The friction coefficients are 0·20 for TiN and 0·15 for TiCN (Fig. 7). In addition, no wear of the

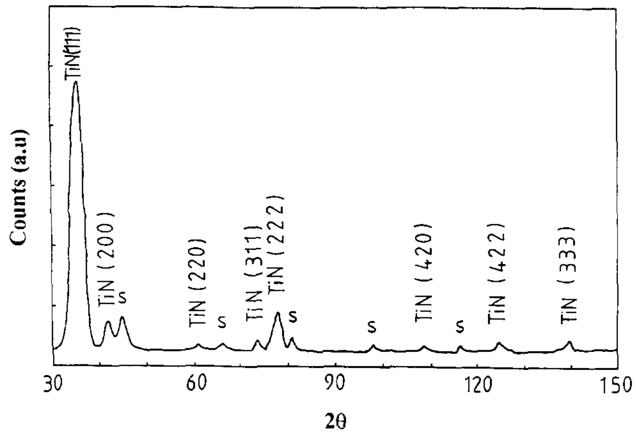

Fig. 2. X-ray diffraction spectra of TiN coating. The peaks corresponding to the steel substrate are marked with S. Identical spectra were obtained for TiCN.

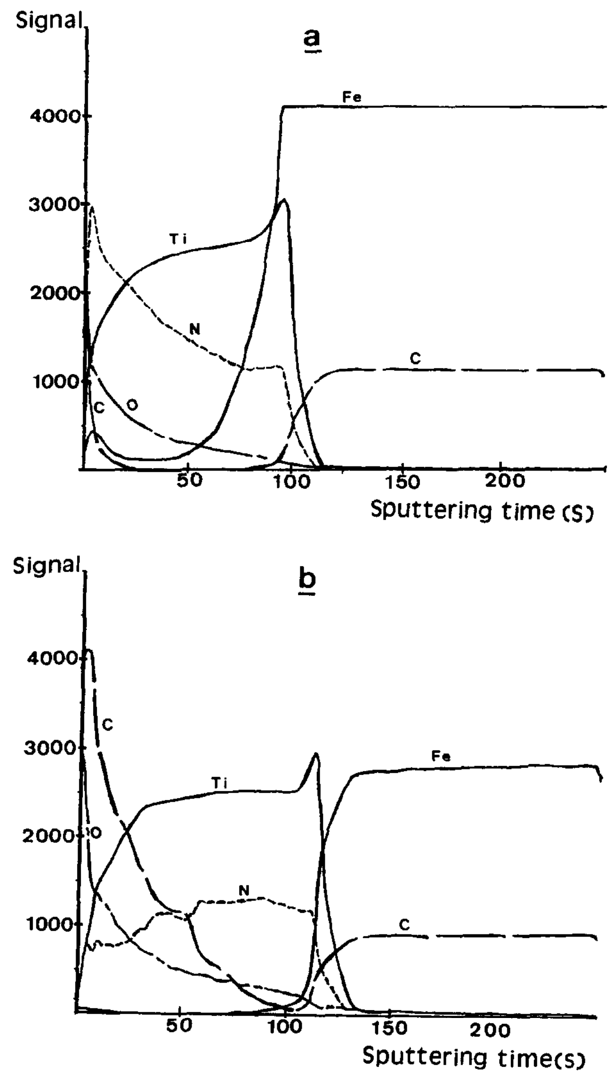

Fig. 3. Depth profiles obtained by GDOS from (a) TiN and (b) TiCN coatings deposited on steel substrate.

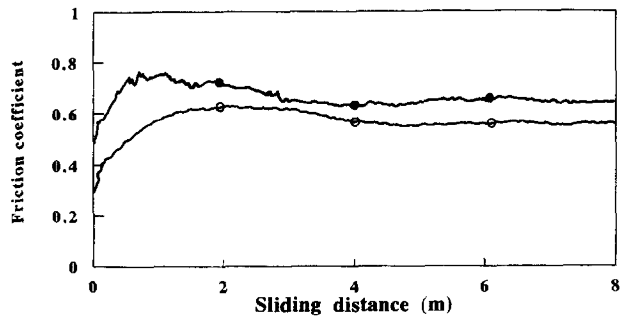

Fig. 4. Evolution of the friction coefficient as a function of sliding distance for TiN (●) and TiCN (○) in sliding contact against a steel ball. Test conditions are shown in Table 1.

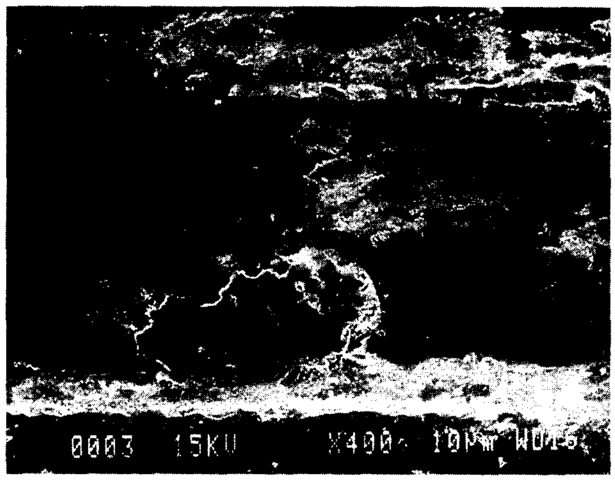

Fig. 5. Metal transfer from the steel ball onto the TiN coating after the wear test.

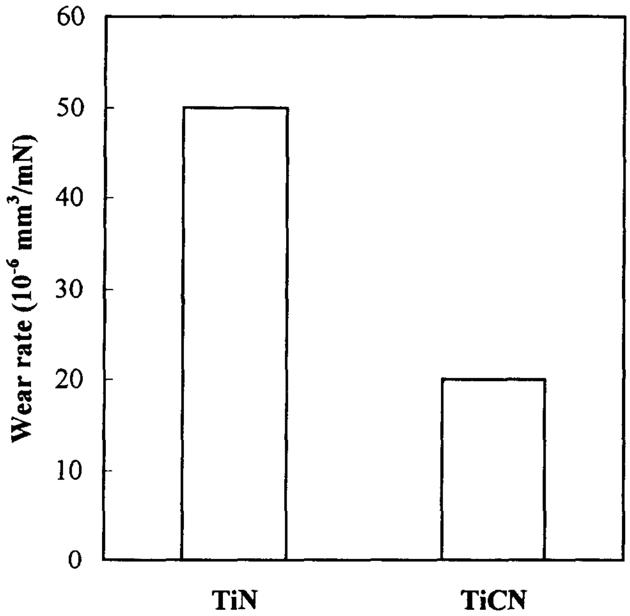

Fig. 6. Wear volume of the 100C6 steel ball after sliding against TiN and TiCN coatings.

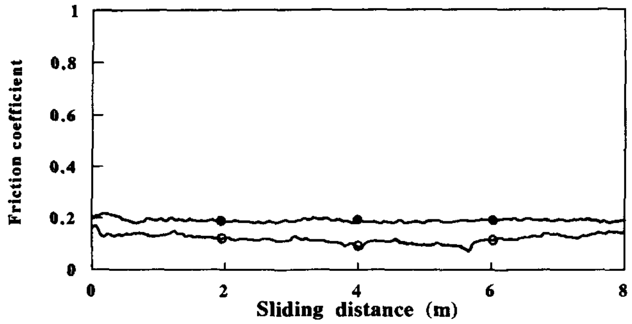

Fig. 7. Evolution of the friction coefficient as a function of sliding distance for TiN (●) and TiCN (○) in sliding contact against an alumina ball. Test conditions are shown in Table 1.

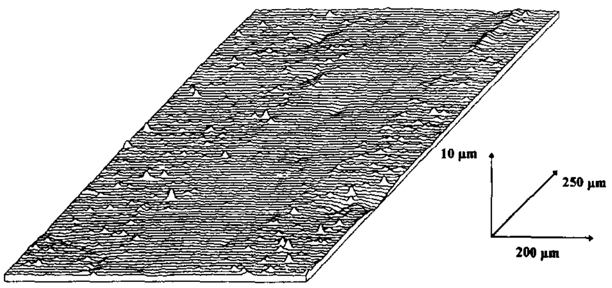

Fig. 8. Wear scar of TiN and TiCN (the same result was obtained for both) at the end of the tests presented in Fig. 7.

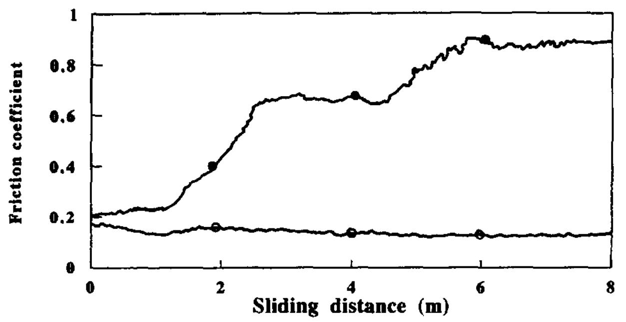

Fig. 9. Same as Fig. 7 but the applied load was 40 N instead of 25 N.

coatings was detected (Fig. 8). In order to test wear resistance of the coatings under more severe conditions, we conducted additional tests under a load of 40 N (instead of 25 N).

For TiN, the friction coefficient increases from 0·2 to 0·88 (Fig. 9) and an important material removal was detected (Fig. 10(a)). In the case of TiCN, the increase of applied load did not have any effect on friction (Figs 7 and 9) nor on wear (Figs 8 and 10(b)).

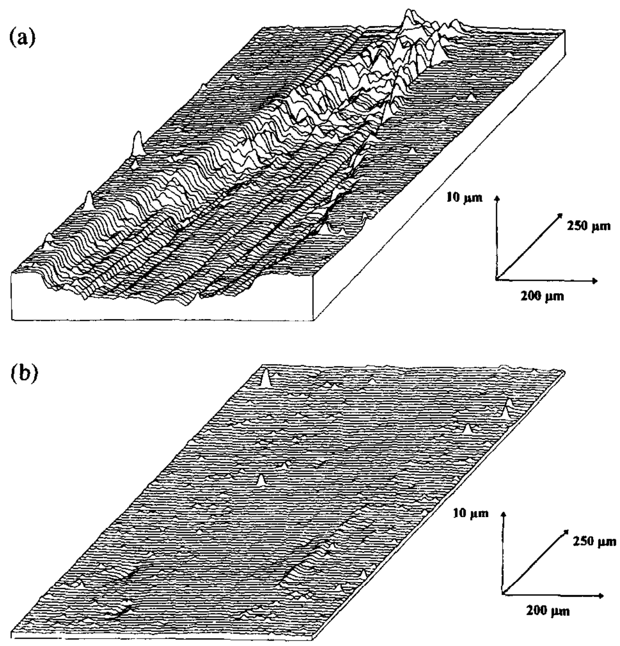

Fig. 10. Wear scar of (a) TiN and (b) TiCN at the end of the friction tests presented in Fig. 9.

4 Conclusion

X-ray diffraction, hardness tests, EDS and GDOS analysis were conducted to characterize TiN and TiCN films prepared by PVD. Scratch-tests were conducted and showed that the critical load Lc is higher for TiCN (39 N for TiCN instead of 25 N for TiN), indicating that TiCN adheres better to the substrate than the titanium nitride.

Friction tests against 100C6 steel and alumina were conducted using a ball on disk tribometer. TiCN showed a lower friction coefficient and higher resistance to wear than TiN.

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