(Ti,Al)N Film on Normalized T8 Carbon Tool Steel Prepared by Pulsed High Energy Density Plasma Technique *

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Under optimized operating parameters, a hard and wear resistant (Ti,Al)N film is prepared on a normalized T8 carbon tool steel substrate by using pulsed high energy density plasma technique. Microstructure and composition of the film are analysed by x-ray diffraction, x-ray photoelectron spectroscopy, Auger electron spectroscopy and scanning electron microscopy. Hardness profile and tribological properties of the film are tested with nano-indenter and ring-on-ring wear tester, respectively. The tested results show that the microstructure of the film is dense and uniform and is mainly composed of (Ti,Al)N and AlN hard phases. A wide transition interface exists between the film and the normalized T8 carbon tool steel substrate. Thickness of the film is about 1000 nm and mean hardness value of the film is about 26 GPa. Under dry sliding wear test conditions, relative wear resistance of the (Ti,Al)N film is approximately 9 times higher than that of the hardened T8 carbon tool steel reference sample. Meanwhile, the (Ti,Al)N film has low and stable friction coefficient compared with the hardened T8 carbon tool steel reference sample.

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Carbon tool steels are widely used to make common cutting tools, dies and measuring tools. With relatively low hardness and poor wear resistance, service life of these tools is relatively short. Adopting an appropriate film deposition technique to prepare high quality protective films on the surface of these tools will be an economic and convenient method to prolong their service life.

With high hardness and excellent tribological properties, the metastable (Ti,Al)N films become a standard protective film for cutting tools.[1,2] The metastable (Ti,Al)N films are usually deposited by PVD, CVD or plasma-enhanced CVD techniques.[1−6] As is well known, the films prepared by PVD, CVD or plasma-enhanced CVD techniques usually have poor adhesive strength to substrate. This disadvantage may limit the applications of the above-mentioned films in situation of severe wear companied with aggressive attack load.

The pulsed high energy density plasma (PHEDP) technique as a relatively new material modification way has many advantages compared with other material modification means. The four most important features of the PHEDP technique are high electron temperature (about 10–100 eV), high plasma density (about 10^{14}–10^{16} cm^{-3}), high axial velocity (about 10–50 km s^{-1}), and high energy density (about 1–10 J cm^{-2}).[7] Based on these features, films prepared by the PHEDP technique are characterized by high adhesive strength to the substrate, high deposition rate, high density.[8] The PHEDP technique is also combined with three effects including deposition, ion implantation and fast self-quenching. During plasma cluster bombardment, temperature of the substrate surface can reach 10^5 K. However, the maximum instantaneous temperature of the substrate is below 500 K.[9,10] The large temperature gradient leads to the fast self-quenching effect. The self-quenching rate can reach about 10^8–10^{10} K s^{-1}. The high self-quenching rate enables nanocrystal or amorphous microstructure formation.[11,12] The operating principle of the PHEDP technique has been introduced in much former literature.[13−18]

In this study, metastable (Ti,Al)N film has been prepared by using PHEDP technique on a normalized T8 carbon tool steel substrate at ambient temperature. Microstructure and composition of the film were characterized by x-ray diffraction (XRD), x-ray photoelectron spectroscopy (XPS), Auger electron spectroscopy (AES) and scanning electron microscopy (SEM). Hardness profile and the dry-sliding wear resistance of the film are evaluated.

The inner electrode of the coaxial plasma gun was made of commercial pure titanium and the outer electrode was made of commercial pure aluminium. Nitrogen gas with a concentration of more than 99.9at.% was used as the working gas. The optimized experimental parameters are as follows: discharge voltage $V_{\rm gun} = 3$ kV, applied voltage $V_{\rm puff} = 1.5$ kV, working nitrogen gas pressure is 1.96×10^5 Pa, gap dis-

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tance between the sample and the coaxial plasma gun is $d = 25\,\mathrm{mm}$, shot number of the pulsed plasma $N_{\mathrm{pulse}} = 5$, base pressure in the chamber is $10^{-3}\,\mathrm{Pa}$, and sample temperature is at ambient temperature.

Hardness profile of the metastable (Ti,Al)N film was tested by an H100 Fisher depth-sensing indentation instrument. Tribological property of the (Ti,Al)N film was evaluated with an MXP-2000 ring-on-ring dry sliding wear tester. The upper ring made of hardened GCr15 bearing steel (HRC65) rotated on the surface of the still lower ring. Mating surface of the lower ring was coated with the (Ti,Al)N film prepared by PHEDP technique. Wear test parameters are: load 9.8 N, relative sliding speed 0.5 m/s, sliding distance 250 m. Friction coefficients were recorded automatically. Hardened T8 carbon tool steel (HRC60) was selected as the reference material. Relative wear resistance, i.e. the ratio of wear volume loss of the hardened T8 carbon tool steel specimen to that of the (Ti,Al)N film specimen, was utilized to judge the wear resistance of the (Ti,Al)N film. The larger the ratio, the higher the wear resistance of the film is.

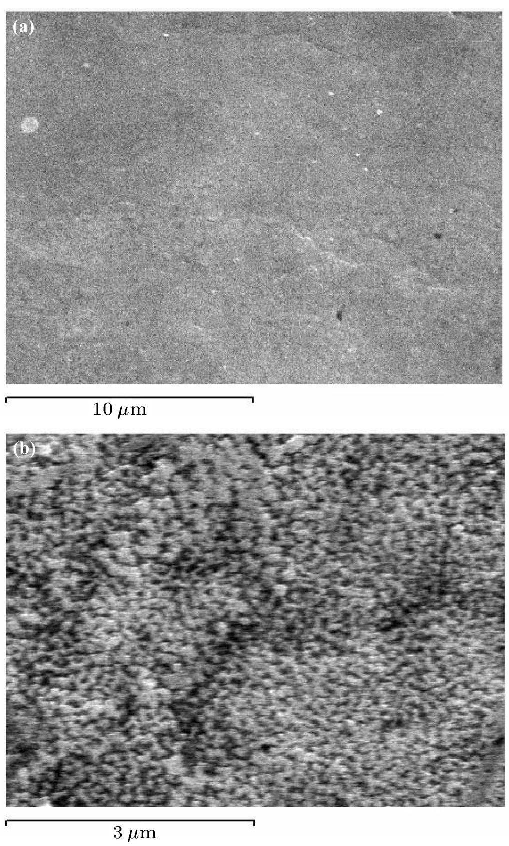

Fig. 1. SEM micrographs of the (Ti,Al)N film taken at different magnifications: (a) 3000 and (b) 10000.

Figure 1 shows surface morphology of the (Ti,Al)N film prepared by using PHEDP technique on normalized T8 carbon tool steel substrate. The (Ti,Al)N film has a cellular surface microstructure consisting of densely packed spherical microcrystalline grains. Mean surface roughness of the film is about $0.06\,\mu\mathrm{m}$ and average size of the grains is about 100 nm. With uniform and fine crystal microstructure, the (Ti,Al)N film can be expected to have good mechanical properties and high wear resistance.

Figure 2 shows the XRD pattern of the film. It is seen that the film mainly is composed of (Ti,Al)N and AlN. The film exhibits a polycrystalline structure with (200) preferential growth. In the synthetic process by the PHEDP technique, crystal growth orientation partly depends on the base temperature. At the early stage, the substrate temperature is closing to the ambient temperature. The tendency of crystal preferential growth is obvious. The film mainly grows along (200) orientation. As the processing time accumulating, temperature of the substrate gradually increases. The tendency of crystal preferential growth is no longer obvious. The film begins to grow along other orientations. According to the XRD analytical result, it can be preliminary concluded that a film mainly composed of (Ti,Al)N and AlN has been prepared on the normalized T8 carbon tool steel substrate by the PHEDP technique.

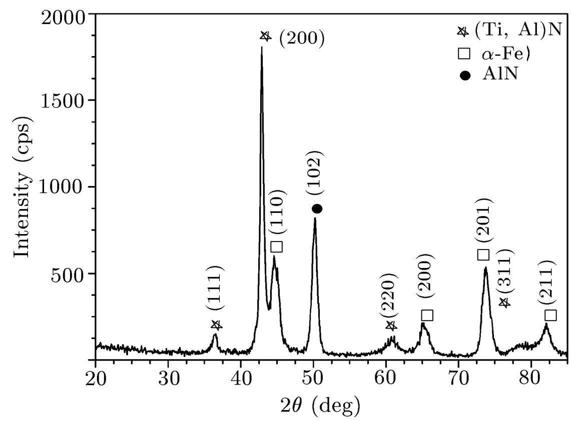

Fig. 2. XRD spectrum of the (Ti,Al)N film prepared by PHEDP technique on normalized T8 plain carbon steel substrate.

Chemical composition of the film was also tested by XPS. Before recording the spectra, Ar^{+} ion bombardment at 4 kV for 10 min was carried out to remove the surface contaminants. Figures 3(a), 3(b), and 3(c) show the Ti $2p_{3/2}$, Al $2p_{3/2}$ and N $1s$ XPS spectra of the film, respectively. The corresponding peak of Ti $2p_{3/2}$ at 455.9 eV corresponds to the standard binding energy of (Ti,Al)N, the corresponding peak of Al $2p_{3/2}$ at 74.3 eV corresponds to the standard binding energy of (Ti,Al)N and AlN, and the corresponding peak of N $1s$ at 397.9 eV corresponds to the standard binding energy of (Ti,Al)N and AlN. The XPS analytical results also testify the presence of (Ti,Al)N and AlN in the film.

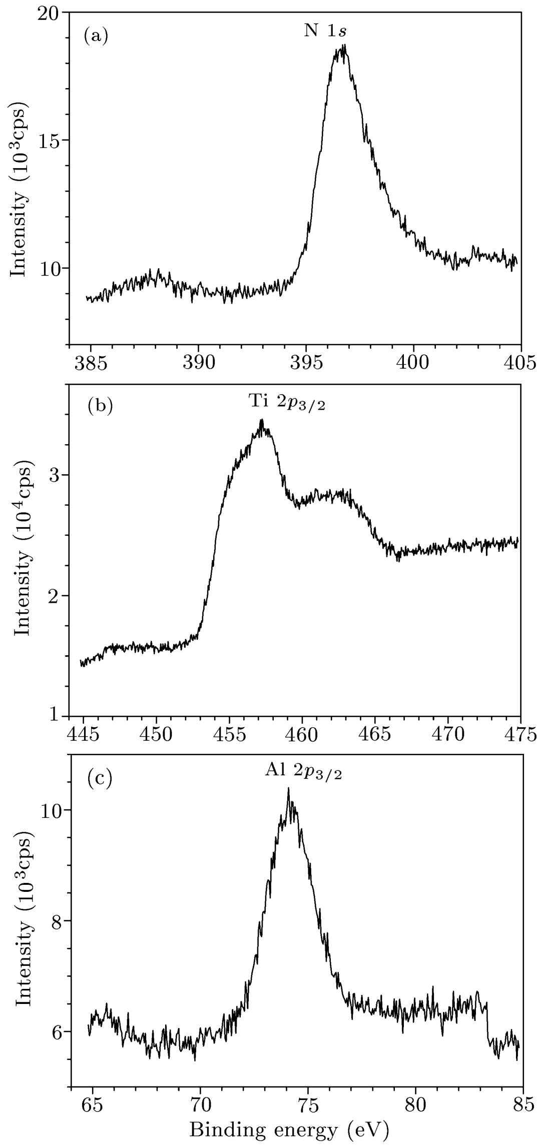

Fig. 3. XPS spectra of the (Ti,Al)N film prepared by the PHEDP technique on the normalized T8 carbon tool steel substrate: (a) N 1*s*, (b) Ti 2*p*, and (c) Al 2*p*.

Figure 4 shows the AES profile of the (Ti,Al)N film. The Ar^+ etching rate is approximately 30 nm/min. According to Fig. 4, it can be calculated that the thickness of the film is about 900 nm, and the transition interface between the film and the substrate is about 150 nm. The presence of broad transition interface reveals that the (Ti,Al)N film is sufficiently mixed with the T8 carbon tool steel substrate, which is very helpful for improving the film/substrate adhesive strength.

From the analytical results of XRD, XPS and AES, it can be concluded that the film mainly consisting of (Ti,Al)N and AlN has been prepared by using the PHEDP technique on a normalized T8 carbon tool steel substrate, and there exists a wide transition interface between the (Ti,Al)N film and the normalized T8 carbon tool steel substrate.

Figure 5 shows the hardness distribution curve of the (Ti, Al) N film. The mean hardness value of the (Ti,Al)N film is approximately 26 GPa, which is about 5 times higher than that of the normalized T8 carbon tool steel substrate. Hardness distribution is uniform except in the transition interface area of the film. Because near the interface area, thickness of (Ti,Al)N and AlN hard phases is relatively low. With the increasing indentation depth, influence extent of the substrate becomes more significant. Thus, the hardness value of the film decreases gradually till to the level of the substrate. The hardness test result is also consistent with the AES analytical result.

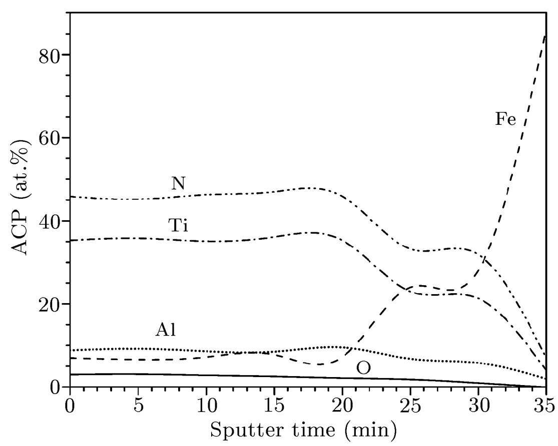

Fig. 4. AES profile of the film prepared by the PHEDP technique on the normalized T8 carbon tool steel substrate.

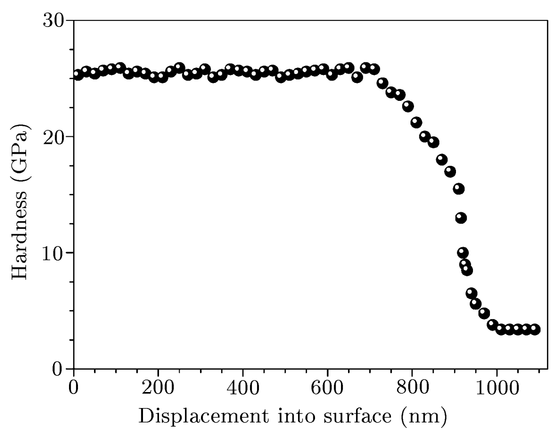

Fig. 5. Hardness of the (Ti,Al)N film as a function of the indentation depth.

Wear test result shows that the relative wear resistance of the (Ti,Al)N film is about 9 times higher than that of the hardened T8 carbon tool steel sample, which indicates that the (Ti,Al)N film prepared by using PHEDP technique as a protective film can largely improve the wear resistance of the T8 carbon tool steel.

Figure 6 shows the variations of friction coefficient with test time under dry sliding wear test condition. The friction coefficient of the (Ti,Al)N film oscillates around 0.37, which is substantially smaller than that of the hardened T8 carbon tool steel specimen (oscillated around 0.62).

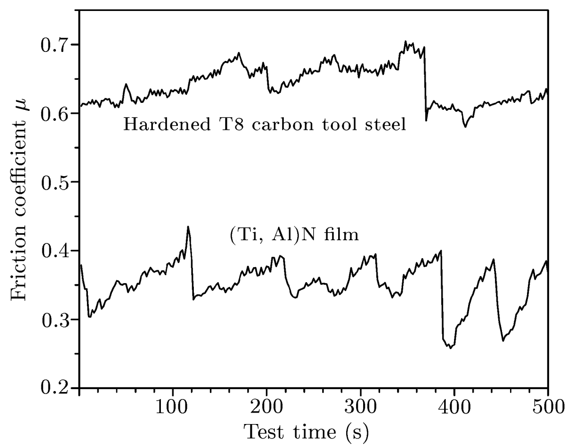

Fig. 6. Variations of friction coefficients versus test time for the (Ti,Al)N film and the hardened T8 carbon tool steel.

From the wear test result, it can be concluded that the (Ti,Al)N film has excellent wear resistance, low and stable friction coefficient. The (Ti,Al)N film can greatly improve the wear and friction properties of the normalized T8 carbon tool steel.

In conclusion, a novel (Ti,Al)N film has been prepared on a normalized T8 carbon tool steel substrate by using pulsed high energy density plasma technique. The (Ti,Al)N film has a homogeneous and fine microstructure consisting of (Ti,Al)N and AlN spherical microcrystalline grains. There exists a wide transition interface between the (Ti,Al)N film and the normalized T8 carbon tool steel substrate. The (Ti,Al)N film has high hardness, high wear resistance, low and stable friction coefficient under dry sliding wear test condition.

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