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Tribological Evaluation of Diamond Coating on Pure Titanium in Comparison with Plasma Nitrided Titanium and Uncoated Titanium

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Titanium alloys are characterized by poor tribological properties, and the traditional use of titanium alloys has been restricted to nontribological applications. The deposition of a well adherent diamond coating is a promising way to solve this problem. In this study, the tribological properties of diamond-coated titanium were studied using a pin-on-disk tribometer, and the results were compared with those of pure titanium and plasma nitrided titanium. The tribological behavior of pure titanium was characterized by high coefficient of friction and rapid wear of materials. Plasma nitriding improved the wear resistance only under low normal load; however, this hardened layer was not efficient in improving the wear resistance and the friction properties under high normal load. Diamond coating on pure titanium improved the wear resistance of titanium significantly. Surface profilometry measurement indicated that little or no wear of the diamond coating occurred under the test conditions loads. The roughness of the diamond coating was critical because it controlled the amount of abrasive damage on the counterface. Reducing the surface roughness by polishing led to the reductions in both the friction and wear of the counterface.

Keywords diamond, plasma assisted chemical vapor deposition, plasma nitriding, titanium, tribology, wear

1. Introduction

Titanium alloys present weak tribological characteristics, especially poor friction and wear resistance (Ref 1). The deposition of a well adherent diamond coating is a promising way to solve these problems (Ref 2). However, there is still extensive work to be done for the successful industrial application of diamond-coated titanium alloy. The tribological performance of diamond coating should be optimized. The friction and wear characterization of bulk diamond materials are well known, both in air and vacuum condition (Ref 3). However, the tribological characteristics of diamond coatings are quite different from that of bulk diamond because of their polycrystalline nature and the existence of nondiamond carbon at grain boundaries. Preliminary studies showed that the friction and wear properties of polycrystalline diamond coatings depended on many parameters: coating surface topography, crystalline structure of diamond coating, the nature of counterface, and environment (Ref 4, 5).

One of the problems to be solved is the high surface roughness of as-deposited diamond coatings, which causes high coefficient of friction and severe wear of the counterface material (Ref 6). Reducing the surface roughness by polishing the surface of as-deposited coatings leads to reductions in both friction and the wear of the counterface. In this paper, tribological properties of diamond coatings were studied, and the results were compared with that of plasma nitrided titanium and pure titanium.

2. Experimental Procedures

Commercially pure titanium plate with a thickness of 3 mm was mechanically ground with abrasive papers and polished with diamond pastes (6 to 1 μm), then ultrasonically cleaned by acetone before deposition. Deposition of a diamond coating on the titanium substrate was carried out by a MPS4 microwave plasma assisted chemical vapor deposition (CVD) system (Coaxial Power Systems Ltd., UK). The output frequency was 2.45 GHz, and the microwave plasma power was 1 kW. The gas ratio of methane to hydrogen was 196 to 4. The total gas pressure was 30 torr, and the deposition time was 12 h. The diamond coating after deposition was polished using standard metallographic techniques. The coating was mechanically ground with abrasive papers for half an hour and polished with diamond pastes (6 to 1 μm), then ultrasonically cleaned by acetone.

The tribological behavior of diamond coated titanium substrate was evaluated using a ball-on-disk tribometer at room temperature (25 °C). Balls of alumina (with a diameter of 9.5 mm and a surface roughness better than R_a = 0.02 μm) were used as the counterface materials. The normal loads were 2, 5, and 10 N. The sliding distance was 200 m, and the sliding speed was controlled at 0.2 m/s. The coefficient of friction was continuously recorded during each test. The relative humidity during measurement was about 65% ± 3%. The wear track on the worn specimen and the wear scars on alumina balls were examined by scanning electron microscopy (SEM) and electron dispersive x-ray (EDX) analysis.

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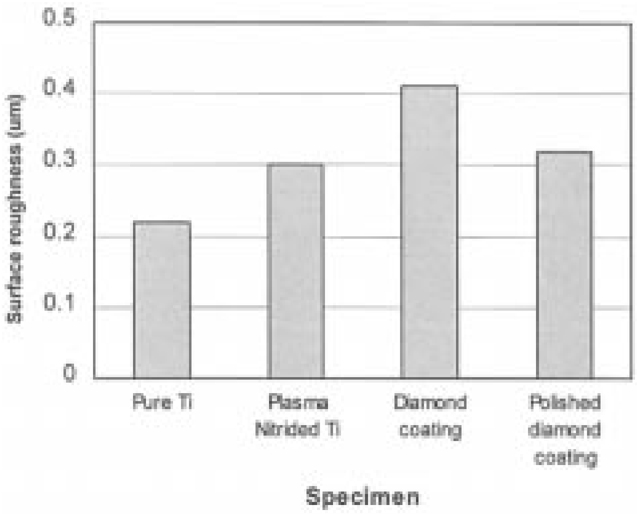


Fig. 1 Comparison of the surface roughness among the untreated pure titanium, plasma nitrided titanium, natural diamond coating, and polished diamond coating. (Obtained from Talysurf 5 laser profilometer)

The tribological performance of diamond coating was compared with plasma nitrided titanium specimen. Plasma nitriding was carried out by same plasma assisted CVD system with the plasma power of 1.5 kW. The total gas flow of nitrogen was 200 sccm, and total gas pressure was 30 torr. The deposition duration was 1 h.

3. Results and Discussion

3.1 Characterization of Plasma Nitrided Layer and Diamond Coating

Figure 1 shows a comparison of the average surface roughness of an untreated titanium substrate, plasma nitrided titanium, diamond coating deposited on titanium substrate, and polished diamond coating. Compared with the untreated substrate, the surfaces become much rougher after plasma nitriding under 1.5 kW and 1 h or deposition of diamond coating under 1 kW and 30 torr. With the polishing of diamond coating, the surface roughness has been decreased somewhat.

Figure 2 shows the SEM surface morphology of four types of samples, which can support the previous conclusions. Visual observation of the plasma nitrided samples indicates that after

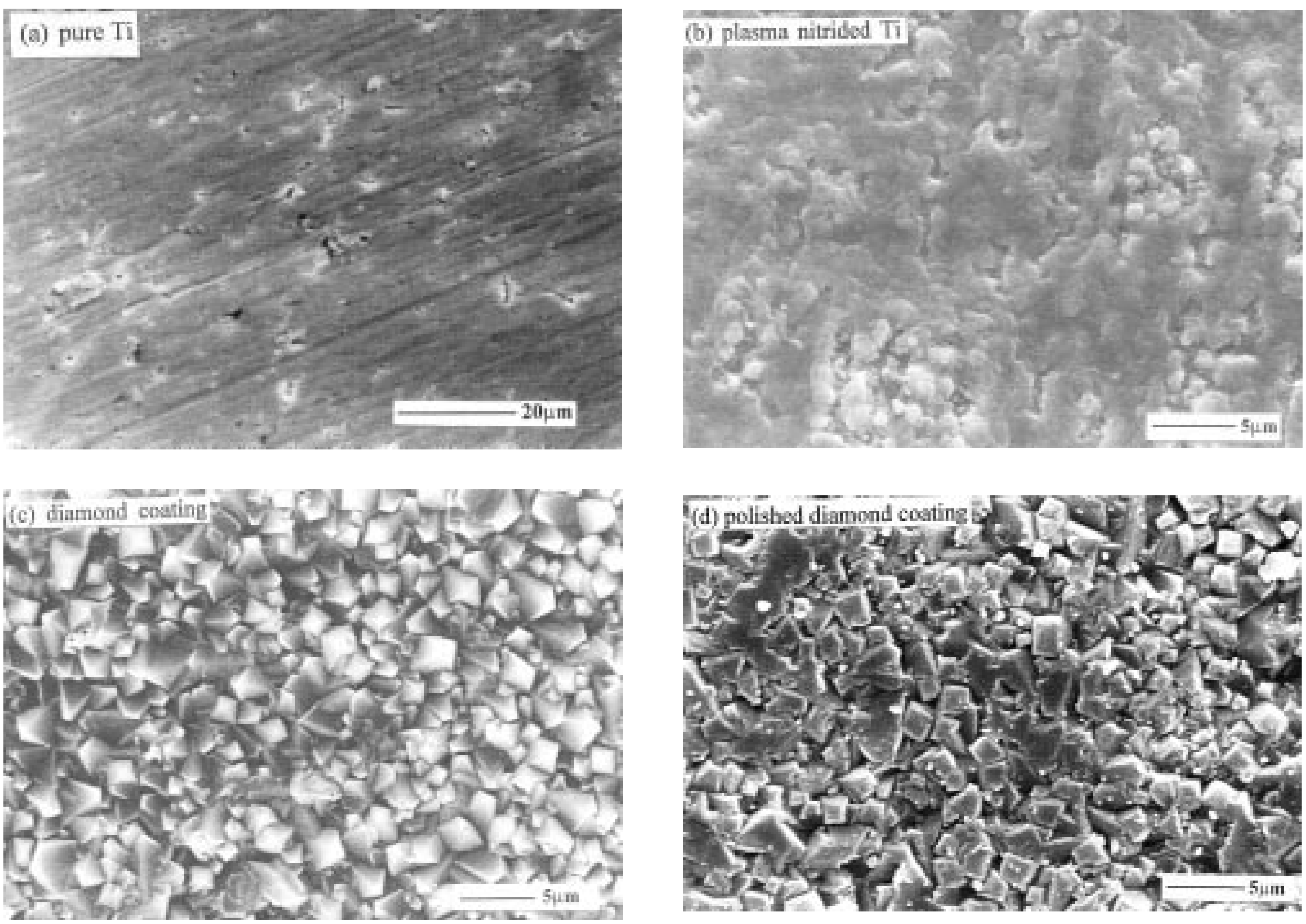


Fig. 2 Surface morphology of four types of specimens. (a) Untreated titanium showing the scratches formed during preparation of specimen. (b) Plasma nitrided titanium indicating the rough nature. (c) Diamond coating showing the sharp tips of asperities. (d) Polished diamond coating showing the relative smoothing surface achieved by damaging the sharp tips of asperities

a short nitriding time, the surface shows a bright yellow color indicating the formation of titanium nitride phase. With an increase in nitriding duration, the sample surface becomes a dark golden color. The deposited diamond coating shows a dense, well-faceted, and polycrystalline morphology, which is predominantly in (111) orientation. For each diamond crystallite, the four (111) planes are inclined relative to the sample surface. The point of intersection of these four (111) faces forms as triangular, sharp asperities, which are clearly shown in Fig. 2(c). The roughness of diamond coating is about 0.42 ± 0.05 μm. After polishing, the diamond coating surface becomes much smoother, indicating the smoothening of the diamond crystals, as shown in Fig. 2(d).

Figure 3 shows the cross-section morphology of plasma nitrided titanium. The top layer (or the compound layer) consists of TiN and Ti_2N (up to about 7 μm thick) (Ref 7). The boundary between the compound layer and the substrate material contains a nitrogen-rich, solid-solution hardened zone.

Figure 4 shows cross-section morphology of diamond coating system that clearly reveals existence of three different layers: the diamond coating, TiC layer, and heat-affected and carbon/hydrogen diffused titanium layer (Ref 8). The diamond coating is about 4 μm thick and is dense and homogeneous. The TiC layer is porous and rough, which is easily removed during the grinding and polishing processes. Microhardness measurements reveal that beneath the TiC layer, there is a hardened zone in the titanium substrate with a thickness of about 20 μm, which is generated by the diffusion of carbon and hydrogen into the titanium substrate.

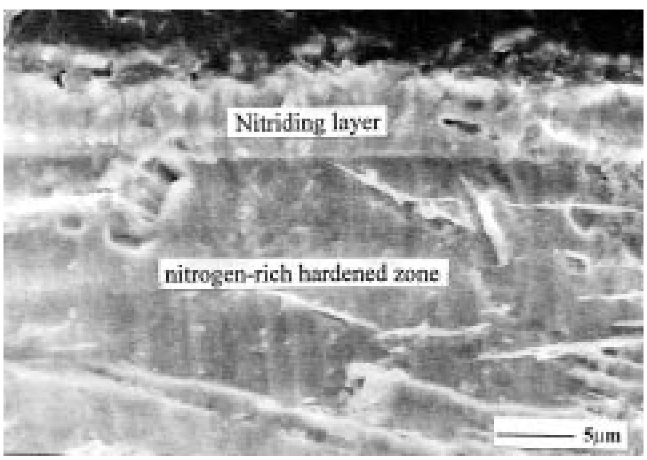


Fig. 3 Cross section of plasma nitrided layer on pure titanium

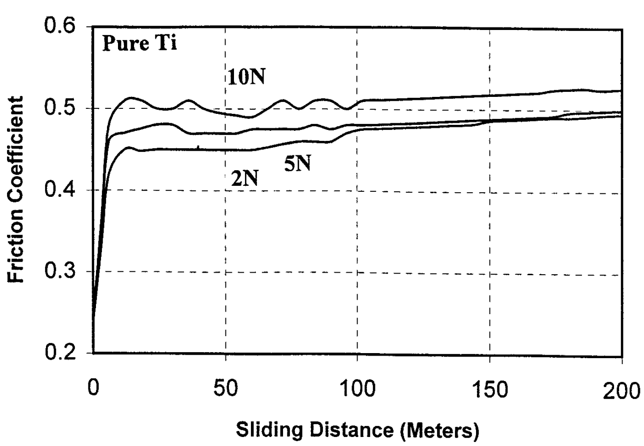


Fig. 5 The friction coefficient curves of pure titanium under different normal loads sliding with Al_2O_3 ball

3.2 *Tribological Behavior*

Pure Titanium Substrate. Figure 5 shows the coefficient of friction curves for the untreated titanium samples under different normal loads. For untreated pure titanium, the long-term coefficient of friction remains a constant at about 0.5 and is almost independent of the applied normal load. Figure 6 shows the cross section profiles of the wear track obtained from the Talysurf profilometer (Taylor Hobson, Inc., Rolling Meadows, IL) under different normal loads. With an increase in normal load, both wear depth and wear width increase significantly. Under the test conditions applied, the dominant wear mechanisms for untreated titanium are oxidative wear and abrasive wear. Figure 7(a) and (b) shows the worn surface morphology of wear track, indicating the extensive scratching or ploughing of the substrate. There is usually a large quantity of wear debris, and EDX analysis indicates that the debris are actually oxide particles. The delamination wear mechanism can also be observed, as shown in Fig. 7(c), but this is not prominent.

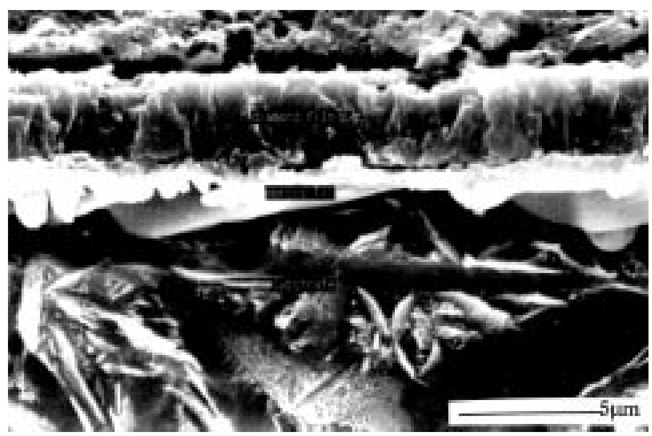


Fig. 4 Cross section of diamond coating on pure titanium

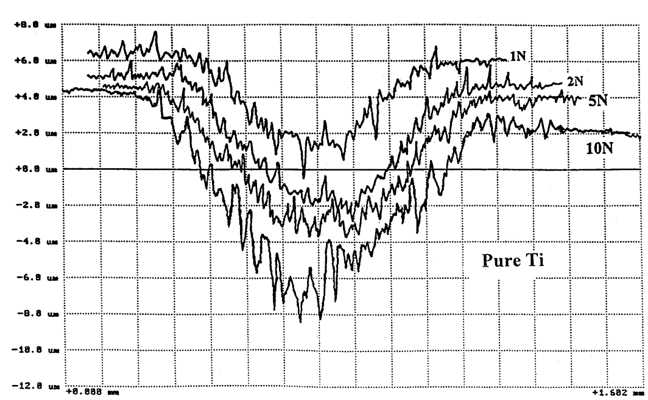


Fig. 6 Cross section profiles of the wear track of pure titanium under different normal loads sliding with Al_2O_3 ball

Examination of the worn Al_2O_3 ball using EDX indicates the transfer of titanium substrate on the balls. In conclusion, high coefficient of friction and rapid wear of materials characterize the tribological behaviors of untreated titanium (Ref 1).

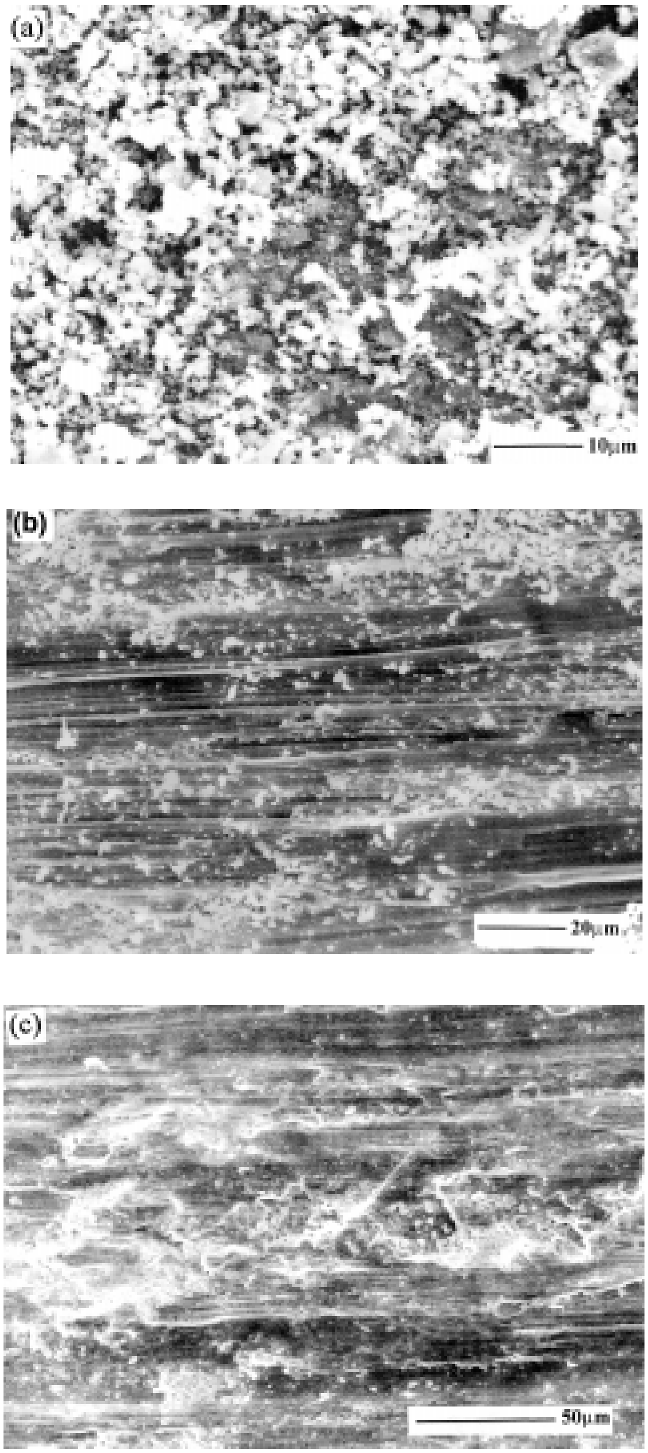


Fig. 7 Worn surface morphology of untreated pure titanium substrate. (a) A large quantity of wear debris indicating the oxidative and abrasive wear. (b) Extensive ploughing on the worn pure titanium substrate. (c) The delamination wear observed on the wear track

Plasma Nitrided Titanium. Figure 8 shows the coefficient of friction of plasma nitrided samples under different normal loads. Figure 9 shows the depth and width of wear tracks of the worn plasma nitrided samples. Wear and friction results of plasma nitrided specimens show that there is a critical load at which the wear mechanism is probably changed. Under a normal load of 2 N, the coefficient of friction remains very low (about 0.2) during the long-term sliding process. The wear of plasma nitrided sample under normal loads of 2 N is difficult to detect from the cross-section profiles, as shown in Fig. 10. Figure 10(a) shows the worn surface morphology of plasma nitrided sample under a normal load of 2 N and a sliding distance of 200 m, indicating only a mild wear of the nitrided layer.

However, with the normal load increased to 5 and 10 N, the predominant wear mechanism changes to that of delamination or spallation. On the worn surface, the crushing or spallation of compound layer can be observed, as shown in Fig. 10(b). After the spallation of the compound layer, the worn surface shows the typical extensive abrasive wear. The coefficient of friction of nitrided sample under 5 N increases abruptly after the initial phase of sliding and continues to rise with the further sliding (see Fig. 8). For the test under a normal load of 10 N, the coefficient of friction also increases abruptly to a high value of 0.6 during the beginning of the wear process; then it varies significantly. Cross section of wear track under normal loads of 5 and 10 N can detect the wear of the nitrided layer as shown in Fig. 9. Energy dispersive x-ray analysis on worn Al_2O_3 ball indicates the transfer of materials from the plasma nitrided layer.

Plasma nitriding can improve the friction property and wear resistance of pure titanium by increasing the surface hardness and forming a compressive residual stress in the nitrided layer. However, the formation of a thin and brittle compound layer is not efficient to improve the friction property and wear resistance under high normal loads as shown by the previous results.

Diamond Coating. Figure 11 shows the coefficient of friction curves of (111) texture diamond coatings under different sliding conditions. Usually there is a significant fluctuation of the dynamic coefficient of friction at the beginning of the test, and the mean value is quite high at about 0.45 to 0.55. At a steady stage, the coefficient of friction is rather unstable and fluctuates between 0.45 and 0.5. The triangular (111) diamond

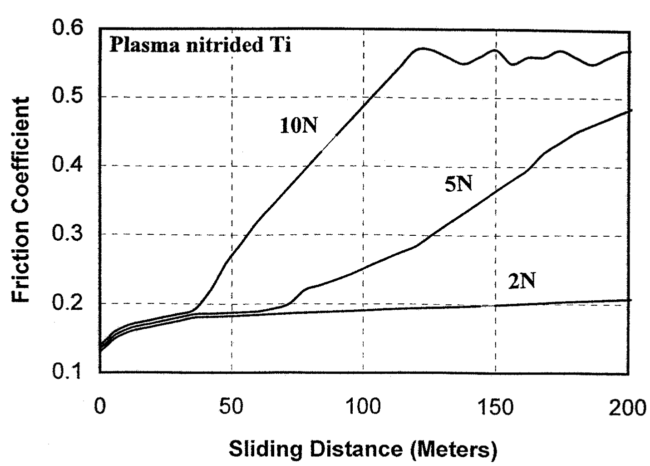


Fig. 8 The friction coefficient curves of plasma nitrided titanium under different normal loads sliding with Al_2O_3 ball

coatings have many sharp asperities. These sharp asperities may induce high tangential forces on the counterbody and can dig in and cut the surface of the alumina ball surface. The high coefficient of friction is mainly caused by a severe abrasion and ploughing action by asperities of diamond coating (Ref 9).

A lot of debris is observed on the diamond coating surface, but no evidence of severe damage of diamond coatings could be found. The EDX analysis indicates that the debris layer consists predominantly of Al_2O_3. Debris piles up at the leading edge of the diamond crystals, and much wear debris were loosely accumulated in the valleys between the asperities (see Fig. 12). The agglomeration of much smaller wear debris forms a thick transfer layer. It is the interaction of these transfer debris layers with the counterface that controls the friction behavior during wear tests (Ref 10, 11). No delamination of the coating can be observed. The wear of the diamond film was too low to be measured in all tests performed. Raman analysis shows that there is no change in the Raman peak on the worn diamond coating surface as compared with the as-deposited coating, indicating that no phase transformation has occurred during sliding.

Scanning electron microscopy observation shows the extensive ploughing and removing of materials on the alumina ball (see Fig. 13). Figure 14 shows the wear volumes of alumina counterface under different normal loads. The higher the normal load, the larger the wear volume of alumina ball. The steady coefficient of friction during long-term sliding may be due to the presence of the transferred debris layer and the enlarged real contact of the counterface (Ref 12).

After polishing the diamond coatings using standard metallurgraphic techniques, the surface roughness of diamond coating is reduced to 0.32 ± 0.05 μm. The coefficient of friction of diamond coatings is reduced significantly and stabilized at a much lower value, as shown in Fig. 11. The larger amount of rounded asperities result in a larger real contact area, and hence, a smaller tangential force is achieved leading to a lower coefficient of friction at the initial stage. There is again no visible wear scar on the polished coating surface, and the transferred debris of the counterface has also been reduced significantly as shown in Fig. 15 (Ref 13).

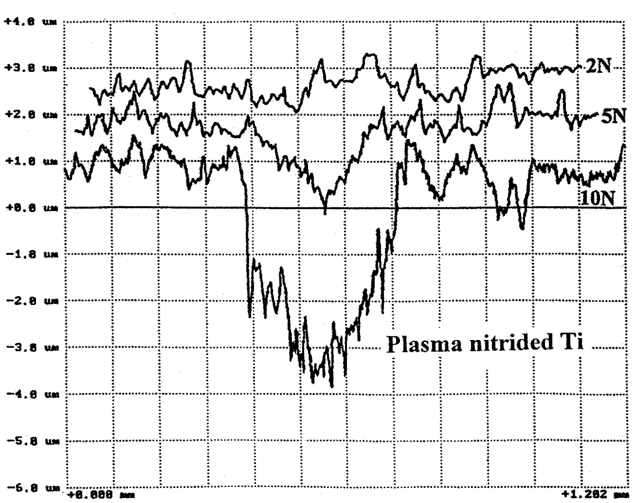
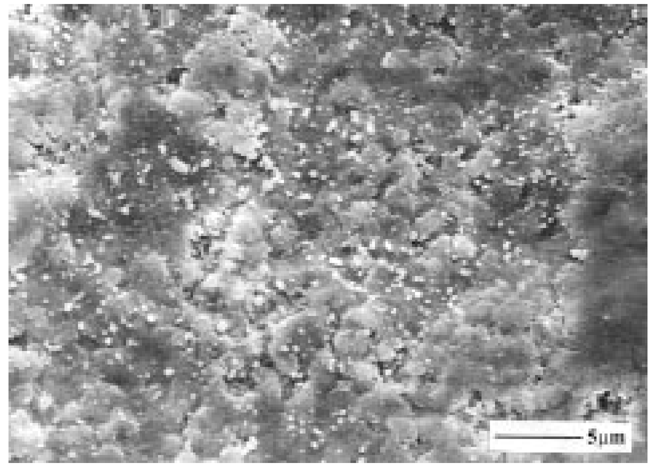
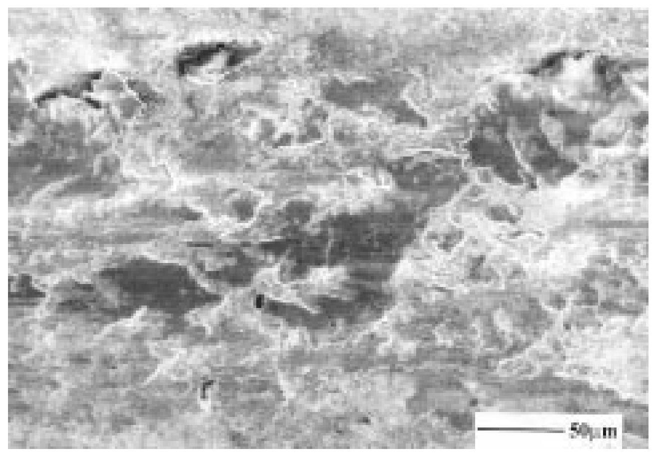


Fig. 9 Cross-section profiles of the wear track of plasma nitrided titanium under different normal loads sliding with Al_2O_3 ball



(a)



(b)

Fig. 10 Worn surface morphology of plasma nitrided layer. (a) Worn surface morphology showing not much wear observed under a normal load of 2 N. (b) Crushing of the plasma nitrided layer under a normal load of 10 N

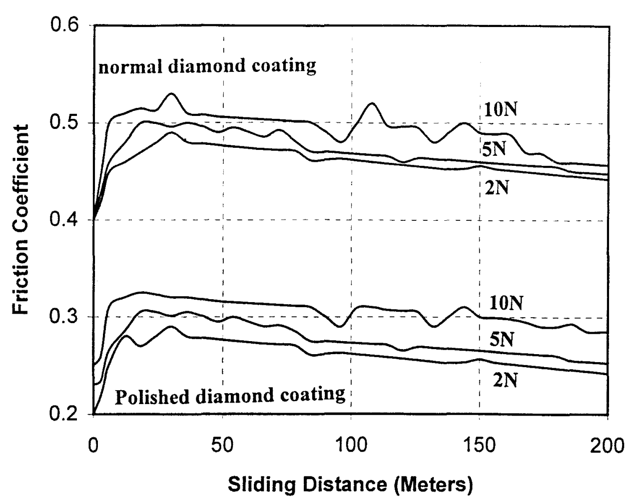


Fig. 11 The friction coefficient curves of normal diamond coating and polished diamond coating under different normal loads sliding with Al_2O_3 ball

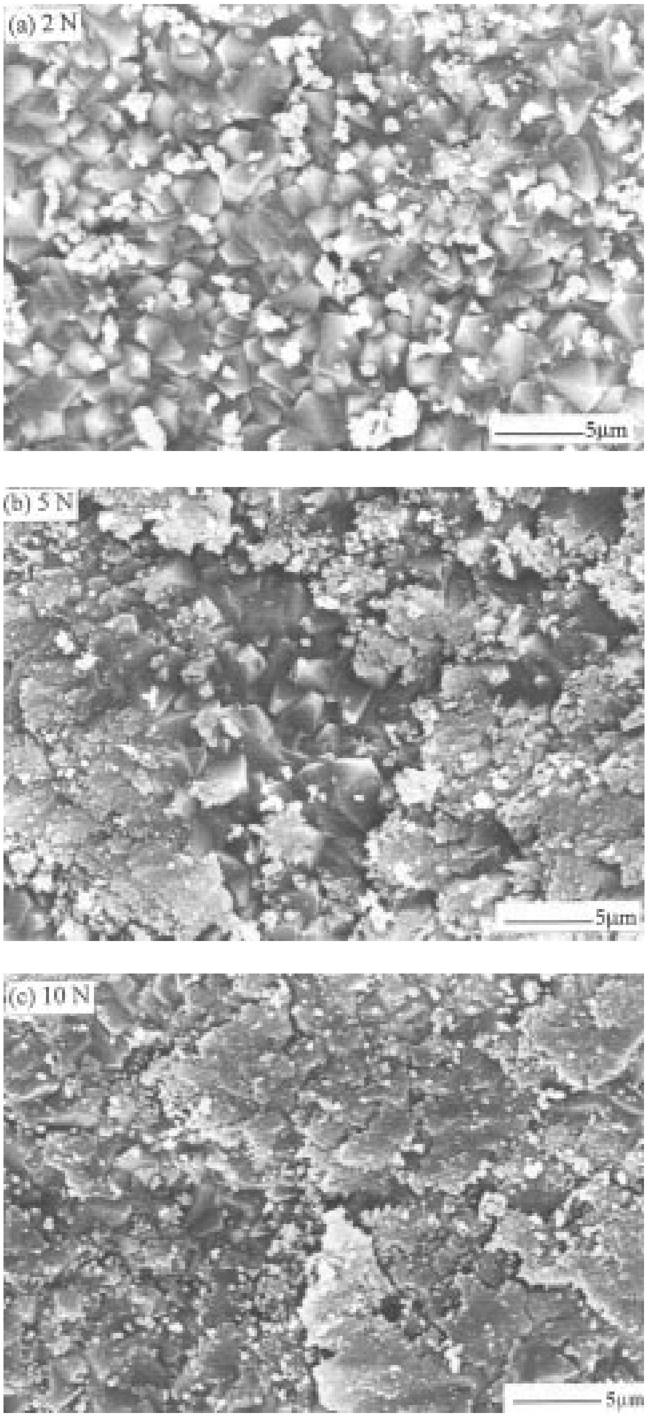


Fig. 12 Worn surface morphology of diamond coating under different normal loads. (a) 2 N. (b) 5 N. (c) 10 N

Figure 16 shows a comparison of the wear scar of the three types of specimen sliding with Al_2O_3 ball under different normal loads. The wear resistance of titanium can be improved significantly by diamond coating.

The high hardness and roughness of the diamond coating leads to extensive abrasive wear. This leads to a transfer of wear debris from counterface to diamond coating. Consequently, the observed coefficient of friction is not typical of the diamond/counterface couple but is typical of the transferred wear debris layer against the counterface. The measured coefficient of friction, therefore, does not illustrate the real friction between the diamond coating and the counterface (Ref 14). Due to the influence of the initial surface roughness and the large amount of the transfer material from the counterface, it is difficult to assess the true wear behavior of the diamond coating. In future work, a scratch test will be used to study the wear and friction properties of the diamond coated specimen.

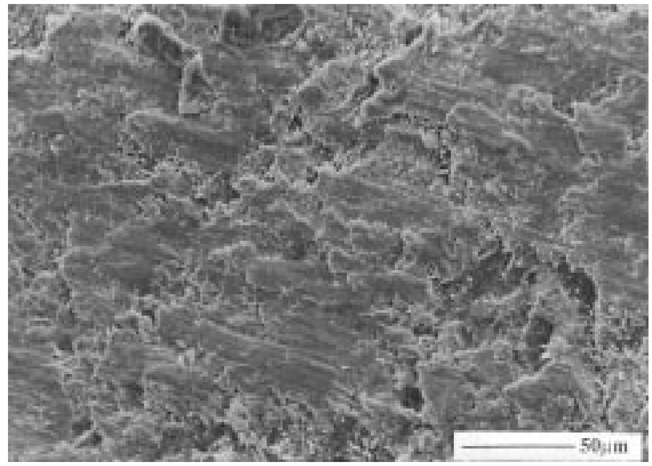


Fig. 13 The extensive ploughing and removing of materials on the Al_2O_3 ball after wear test on diamond coating

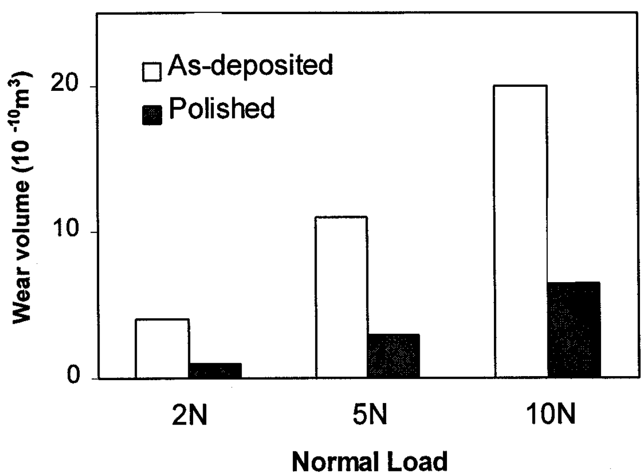


Fig. 14 The wear volumes of Al_2O_3 ball under different normal loads

4. Conclusions

The following conclusions can be drawn:

- High coefficient of friction and rapid wear of materials characterized tribological behaviors of untreated titanium.
- Plasma nitriding improved wear resistance only under low normal load. However, this hardened layer was not effective in improving the wear resistance and the friction properties under high normal load.

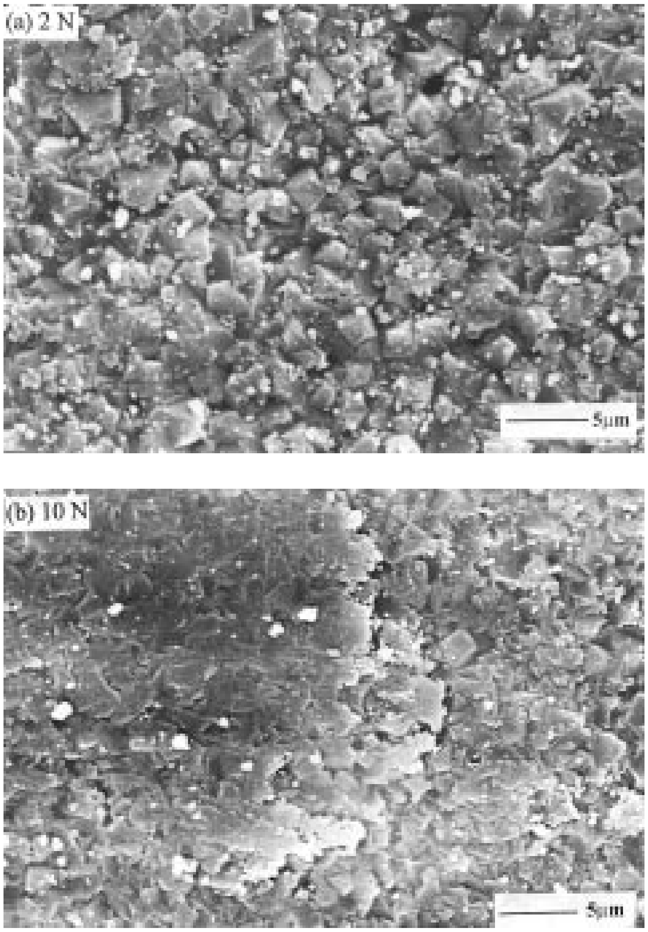


Fig. 15 Worn surface morphology of polished diamond coating under different normal loads. (a) 2 N. (b) 10 N

- Diamond coating on pure titanium improved the wear resistance of titanium significantly. Surface profilometry measurement indicated little or no wear of the diamond coating occurred under different normal load.
- The roughness of the diamond coating was critical because it controlled the amount of abrasive damage to the counterface. Reducing the surface roughness by polishing led to reductions in both friction and wear of the counterface.

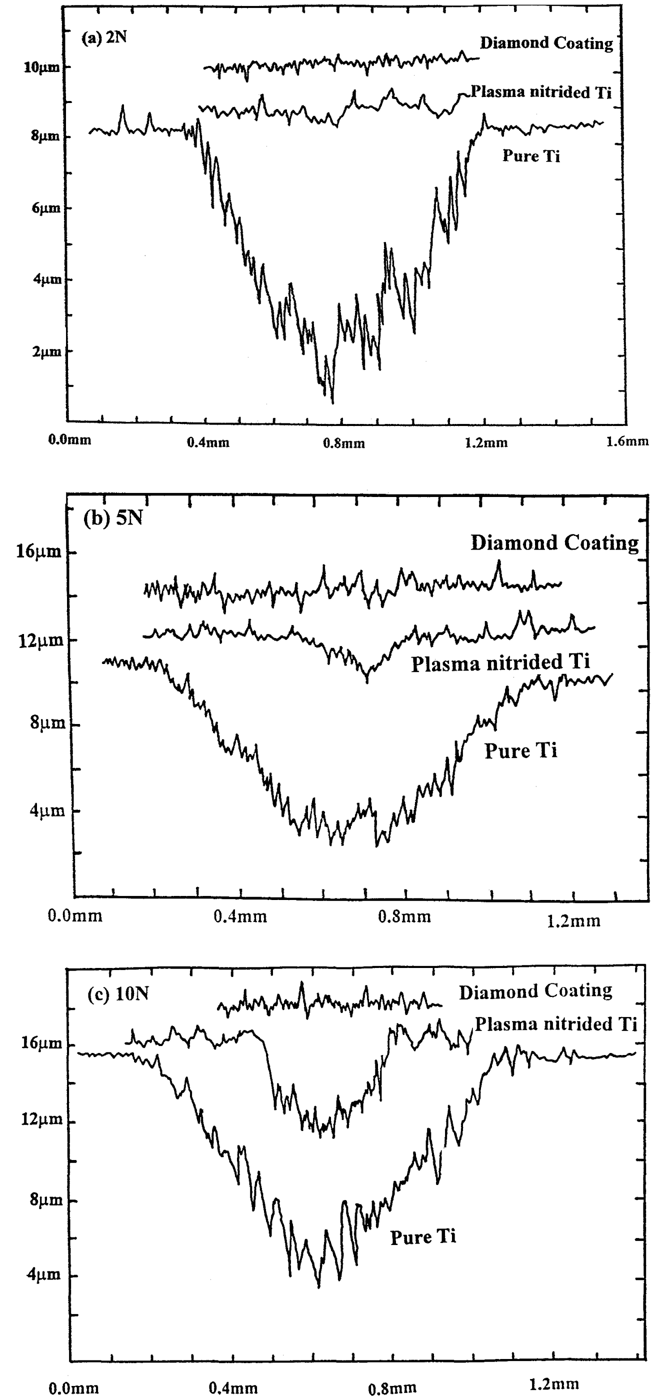


Fig. 16 Comparison of the wear scars of three types of specimen sliding with Al_2O_3 ball under different normal loads

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