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ARTICLE

Effect of Shot Peening Pretreatment on Low Temperature Plasma Nitriding of Ti6Al4V Alloy

Liu Zhen¹, Yu Shouming², Liu Daoxin¹, Shi Hailan¹, He Guangyu³

¹ School of Aeronautics, Northwestern Polytechnical University, Xi'an 710072, China; ² School of Material Science and Engineering, Xi'an University of Technology, Xi'an 710048, China; ³ Aeronautic Engineering Institute, Air Force Engineering University, Xi'an 710038, China

Abstract: In order to improve the wear resistance of titanium alloy, and to prevent the deformation of thin-walled components, taking Ti6Al4V alloy as the object, the effect of shot peening pretreatment on the low-temperature nitriding of titanium alloy was studied. The results show that the pretreatment of shot peening (SP) can effectively promote the plasma nitriding process at low temperature. Under the test conditions of 500 °C, with the increase of shot peening strength, nitriding efficiency of pretreated samples increases gradually, and the surface hardness, load-bearing capacity and the wear resistance of nitrided layer increase gradually. Compared with untreated nitrided samples (Ti6Al4V-PN), when the shot strength increases to 0.25 mmA, the surface hardness of the pretreated nitrided samples (SP(0.25)-PN) increases by 32.7% and the wear rate decreases by 42.3%. The goal of shot peening pretreatment to promote low-temperature plasma nitriding of Ti6Al4V alloy is well achieved.

Key words: Ti6Al4V alloy; shot peening; low temperature nitriding; friction and wear

Titanium alloy has become an attractive candidate for aerospace, automotive and biomedical applications due to its high strength-to-mass ratio, corrosion/oxidation resistance, stability at elevated temperatures, good biocompatibility and fatigue resistance^[1,2]. However, titanium alloys exhibit poor tribological properties, including high and unstable friction coefficients, severe adhesive wear and susceptibility to fretting wear, which restrict its application in engineering^[3]. Surface treatment is one of the important methods to improve the wear resistance of titanium alloy, such as coating technology, surface alloying or lubrication treatment, which can improve the wear resistance of titanium alloy effectively. Among them, plasma nitriding has been proven to improve the surface hardness and tribological properties due to its metallurgically bonded surface alloying modified layer, high bonding strength and environmental friendliness^[4,5]. However, conventional plasma nitriding treatment at 800~1000 °C for up to 80 h tends to cause deformation of thin-walled parts which significantly reduces the mechanical properties of the substrate^[6-8]. Reducing the temperature and duration of plasma nitriding can improve the mechanical properties of titanium alloy, but it cannot provide sufficient nitrogen diffusion depths

owing to slower nitriding kinetics^[9-11]. In recent years, it has been found that surface gradient nanostructures can be formed via methods such as surface shot peening, rolling or grinding, and short-circuit diffusion paths can be formed through grain boundaries, sub-grain boundaries, dislocations, etc, which can effectively promote the nitriding process, and help to achieve low-temperature nitriding^[12,13]. Nevertheless, these methods usually damage the surface integrity due to the surface deformation, which is not conducive to the mechanical properties of the titanium alloy^[14]. Conventional projectile shot peening produces plastic deformation on the metal surface, which increases the dislocation density of the metal surface. Therefore, work hardening on the metal surface, formation of substructures, and introduction of surface residual compressive stress, can significantly improve the fatigue resistance of the metal substrate^[15], and can also promote the surface diffusion process^[16]. Furthermore, the shot peening process is not only simple and efficient, but also low in cost and widely used^[17]. While, few studies have considered whether conventional shot peening can effectively promote the low-temperature nitriding of titanium alloy and how it affects the wear resistance of nitriding layer. Based on the

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Corresponding author: Liu Daoxin, Ph. D., School of Aeronautics, Northwestern Polytechnical University, Xi'an 710072, P. R. China, E-mail: liudaox@nwpu.edu.cn

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above background, in order to improve the surface wear resistance of the titanium alloy, prevent deformation of thin-walled parts, and reduce the adverse effects of nitriding on the inherent mechanical properties of the titanium alloy, in this study, shot peening was employed as a pretreatment step to alter the near-surface microstructure of Ti6Al4V alloy and facilitate kinetics of low-temperature plasma nitriding. Comprehensive characterizations were performed to study the effect of this pretreatment on the wear resistance of the plasma nitrided alloy.

1 Experiment

1.1 Materials and specimens

An annealed Ti6Al4V alloy was used as the substrate in this work. Its chemical composition (wt%) was 6.7 Al, 4.2 V, 0.1 Fe, 0.03 C, 0.015 N, 0.03 H, 0.14 O, and balance Ti. The microstructure of this alloy consisted of a primary α phase (the main component) and a transformed β phase, with the following mechanical properties: ultimate tensile strength 1080 MPa, yield strength 1010 MPa, and micro-hardness 3700 MPa.

The specimens for the basic performance test and friction and wear test were processed into discs of $\Phi 30$ mm $\times$ 8 mm. Specimens were ground by a flat wheel machine and then mechanically ground by abrasive papers of 180#, 240#, 400#, 600#, 800#, 1000# and 2000# grits.

1.2 Shot peening and plasma nitriding

Pretreatment of shot peening used a pneumatic blasting machine and the Z-300 ceramic pellet was used as a shot blasting medium. Hardness of Z-300 ceramic pellet was about 7800 MPa and the average diameter was about 0.30 mm. The specimens were blasted in an air powered system at 6.2 MPa for 30 s. The top surface of the coupons was completely covered with shot peening collision dents (200% coverage). The spray angle was 90°.

According to the production experience and the preliminary exploratory test, the shot peening strengths determined were 0.15, 0.20 and 0.25 mmA.

A set of Ti6Al4V samples without shot peening were also prepared and nitrided under the same conditions for comparison (ie, unpretreated nitrided samples, Ti6Al4V-PN). The pretreated and unpretreated samples were cleaned by ultrasonic washer before being placed inside the nitriding chamber. Plasma nitriding was carried out in a pulsed plasma furnace with the maximum working voltage of 800 V, maximum working current of 120 A, frequency of 3~12 kHz and duty cycle of 40%~80%. The plasma nitriding was performed at 500 °C for 24 h in a pulsed plasma furnace. The environment was $\phi(\text{N}):\phi(\text{Ar})=3:1$ and the voltage was 700 V. The duty cycle and frequency were adjusted to about 40% and 8 kHz, respectively for all tests. Subsequently, the nitrided samples were cooled down to room temperature under vacuum conditions.

1.3 Tribological properties measurement

The sliding wear resistance of the nitrided layers was determined with a ball-plate wear tester (HT-1000). During

the sliding wear test, the disk rotated around its center while the ball remained fixed. Testing was performed under the following conditions: wear radius 4 mm, load 5 N, rotation speed 224 r/min, sliding velocity about 0.094 m/s, wear time 30 min, test temperature of room temperature and relative humidity 50%. The GCr15 ball had the following attributes: diameter 5 mm, micro-hardness 62HRC, and roughness 0.05 μm . Changes in the friction coefficients with wear time were recorded by a computer.

The micro-morphology of the wear track was evaluated via SEM, and the section contour of the track was measured with a roughness/contour tester (TR300). The wear volume (V_w) of the track and the wear rates K ($\text{mm}^3\cdot\text{N}^{-1}\cdot\text{m}^{-3}$) of worn sample were determined from Ref. [18]:

$$V_w = 2\pi R A \quad (1)$$

$$K = V_w / P s \quad (2)$$

where R is wear radius (mm); A is wear area (mm^2), which can be obtained by contour curve integration; s is sliding distance (m); P is load (N).

1.4 Microstructure and basic properties of nitrided layers

The toughness of the layers was evaluated via a static indentation tester (WS2005) equipped with a four prismatic diamond indenter. During the static press test, samples were indented for 30 s under a load of 60 and 150 N. A micro-hardness tester (HV-1000) equipped with a Vicker diamond indenter was used to evaluate the hardness and load carrying capability of the nitrided layers, which were subjected to a load of 0.245 N for 15 s. And the depth of nitride layers was determined by glow discharge optical emission spectroscopy (GDOES; GDA 750).

The scanning electron microscope (SEM; VEGA3 XMU) was used to characterize the microscopic morphology of the nitrided layers, and in order to observe the cross-sectional morphology of the nitrided layers more clearly, HNO_3 and HF etching were performed on the nitrided sample after polishing. Furthermore, the chemical composition of the nitrided layer was determined via energy-dispersive X-ray spectroscopy (EDS; INCA Energy 350 EDX analyzer, Oxford Instruments, Oxfordshire, UK). The phase composition of the nitrided layers was determined via X-ray diffraction (XRD; D/Max-RB).

2 Results and Discussion

2.1 Basic characteristics of nitrided layers

Fig. 1a shows the cross-sectional morphology of Ti6Al4V alloy nitrided at a low temperature of 500 °C for 24 h. It can be seen that no obvious nitrogen diffusion zone (DZ) and compound layer formation can be observed. The cross-sectional morphologies of the nitrided samples pretreated with shot strength of 0.15, 0.20, 0.25 mmA (samples are denoted as SP(0.15)-PN, SP(0.20)-PN, SP(0.25)-PN, respectively) are shown in Fig. 1b~1d. Similar cross-sectional morphologies were developed on the surfaces of both the pretreated and unpretreated nitrided samples. No obvious bright white layer (compound layer) is observed. There is no significant

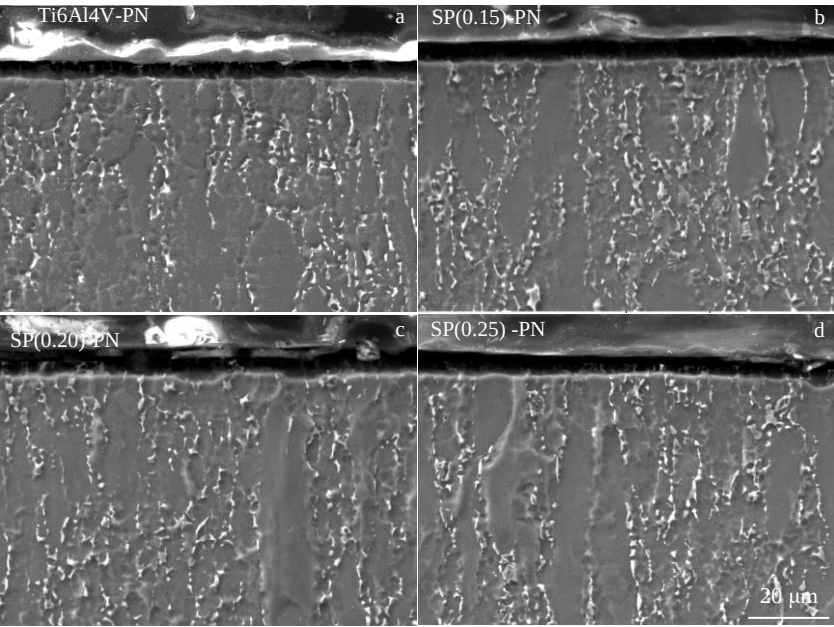


Fig.1 Cross-sectional morphologies of untreated (a) and pretreated (b~d) nitrided samples: (b) SP(0.15)-PN, (c) SP(0.20)-PN, and (d) SP (0.25)-PN

difference in the metallographic morphology of all nitrided samples.

Plasma nitriding studies on steel^[19] show that the formation of surface nitrided layer is affected by the equilibrium process between the amount of nitrogen absorbed by the nitrided surface from the nitriding atmosphere and the amount of nitrogen diffused into the interior of the workpiece. In Fig. 1, the high-magnification metallographic morphology fails to observe the distinct nitride phase. This may be due to the thinner layer of the film, which needs further analysis by XRD and GDOES.

2.2 XRD results

Fig. 2 shows the XRD patterns of Ti6Al4V alloy, unpretreated nitrided samples (Ti6Al4V-PN) and pretreated nitrided samples (SP(0.15)-PN, SP(0.20)-PN, SP(0.25)-PN). The Ti6Al4V alloy is mainly composed of α -Ti phase and a small amount of β -Ti phase. In addition to the α -Ti phase, the unpretreated nitrided sample (Ti6Al4V-PN) also has a small amount of TiN and $\text{TiN}_{0.3}$ nitride phases, but the diffraction peak of β -Ti disappears. Furthermore, the position of the α -Ti peak shifts slightly to a small angle. This may be due to the interstitial solid solution of N atoms in the Ti6Al4V alloy lattice during nitriding process, resulting in an increase in the interplanar spacing d and a decrease in the diffraction angle θ . The pretreated nitrided samples are similar to the unpretreated nitrided samples in phase composition, containing α -Ti, TiN and $\text{TiN}_{0.3}$ nitride phases. It indicates that the shot peening pretreatment cannot change the phase composition of the samples, but the content of the TiN and $\text{TiN}_{0.3}$ phases increases. In addition, the position of the α -Ti peak of the pretreated nitrided sample is slightly shifted to a large angle, because the surface stress is formed by shot peening, and the

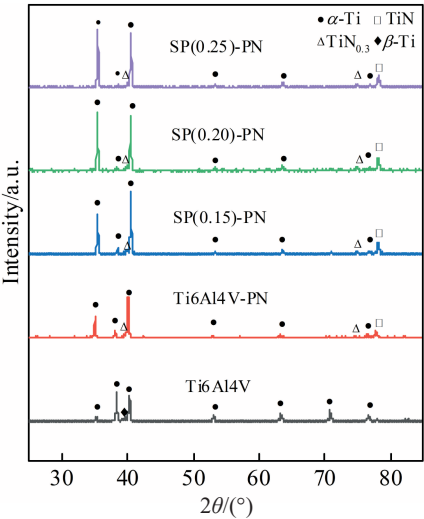


Fig.2 XRD patterns of unpretreated and pretreated nitrided samples and Ti6Al4V alloy

interplanar spacing d decreases. Therefore, the diffraction angle θ increases, resulting in a shift of the peak position to the right. It can be seen from the XRD analysis that TiN and $\text{TiN}_{0.3}$ phases exist on the surface of pretreated and unpretreated nitrided samples. Due to their low content or shallow depth, it is difficult to distinguish in metallographic analysis (Fig.1).

2.3 Hardness and depth of nitrided layers

The surface hardness of pretreated and unpretreated nitrided samples is shown in Table 1. The surface hardness of Ti6Al4V alloy is 3684 MPa. And the surface hardness of the

Table 1 Surface hardness of pretreated and untreated nitride samples (MPa)

Ti6Al4V-PN	SP(0.15)-PN	SP(0.20)-PN	SP(0.25)-PN
5035	5393	6075	6748

untreated nitrided sample is 5035 MPa, which is 37.1% higher than that of the Ti6Al4V substrate. The surface hardness of nitrided samples pretreated with 0.15, 0.20 and 0.25 mmA shot strength is 5393, 6075 and 6748 MPa which are 6.9%, 20.8% and 32.7% higher than that of untreated nitrided samples, respectively, and 46.6%, 65.6% and 81.8% higher than that of Ti6Al4V alloy when compared with Ti6Al4V substrate.

It is worth noting that pure shot peening also increases the surface hardness of the titanium alloy^[20,21]. This research shows that the above shot peening pretreatment also causes the surface hardness of Ti6Al4V alloy to increase, which is attributed to the work hardening phenomenon caused by the increase of surface dislocation density, but the hardness increase caused by this reason is small, all below 20%. In addition, the subsequent nitriding process at 500 °C also cause the dislocation to recombine and recovery, which reduce the work hardening phenomenon^[22]. Therefore, the increase in hardness caused by the above-mentioned shot peening pretreatment and plasma nitriding is mainly attributed to the increase of the dislocation density, the refinement of the structure to promote the diffusion of nitrogen atoms, the increase of the concentration of nitrogen atoms on the surface of the titanium alloy, and the improvement of the surface

activity. This promotes the formation of a relatively hard TiN, TiN_{0.3} compound phases^[23-25], which in turn lead to an increase in surface hardness.

Fig. 3 shows the depth of the nitrided layers of pretreated and untreated samples. It can be seen that the depth of the nitrided layers is very thin regardless of pretreatment. However, the depth of the nitrided layer pretreated with 0.25 mmA shot strength is slightly higher than that of other samples. It is further verified that 500 °C low-temperature nitriding introduces a nitriding layer on the surface of the samples, but due to the shallow depth of the layer, it is difficult to distinguish by metallographic analysis (Fig.1).

2.4 Toughness and bonding strength

In order to characterize the bonding strength between the nitrided layers and the Ti6Al4V alloy substrate, and the apparent toughness of the nitrided layers, the static indentation method was used for evaluation. Fig.4 shows the indentation morphology of untreated and pretreated nitrided samples. There is no obvious delamination phenomenon for all the samples, which indicates that the metallurgically bonded nitriding layer can be obtained regardless of pretreatment, and the bonding strength is well. Under external load of 60 N (Fig. 4a₁~4d₁), except for the nitrided sample pretreated with 0.25 mmA shot strength, cracks occur in other samples. Under the external pressure of 150 N (Fig. 4a₂~4d₂), all nitrided samples are cracked, but the number and length of cracks decrease with the increase of shot strength. It is indicated that the pretreatment of shot peening improves the apparent toughness of the nitrided layers. The apparent toughness of the nitrided samples pretreated with 0.25 mmA shot strength is

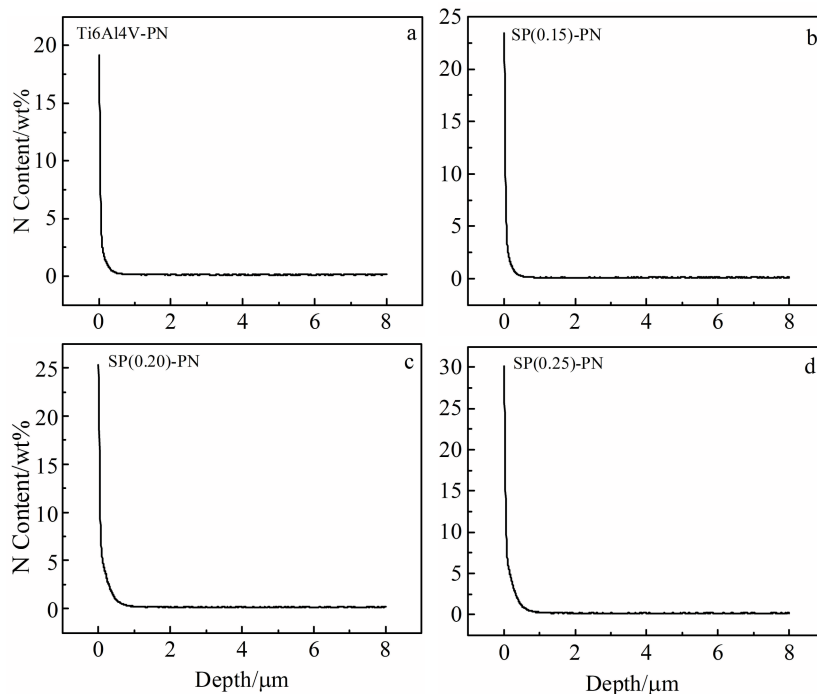


Fig.3 Change of N content with depth of nitrided layers of untreated (a) and pretreated (b~d) nitrided samples: (b) SP(0.15)-PN, (c) SP(0.20)-PN, and (d) SP(0.25)-PN

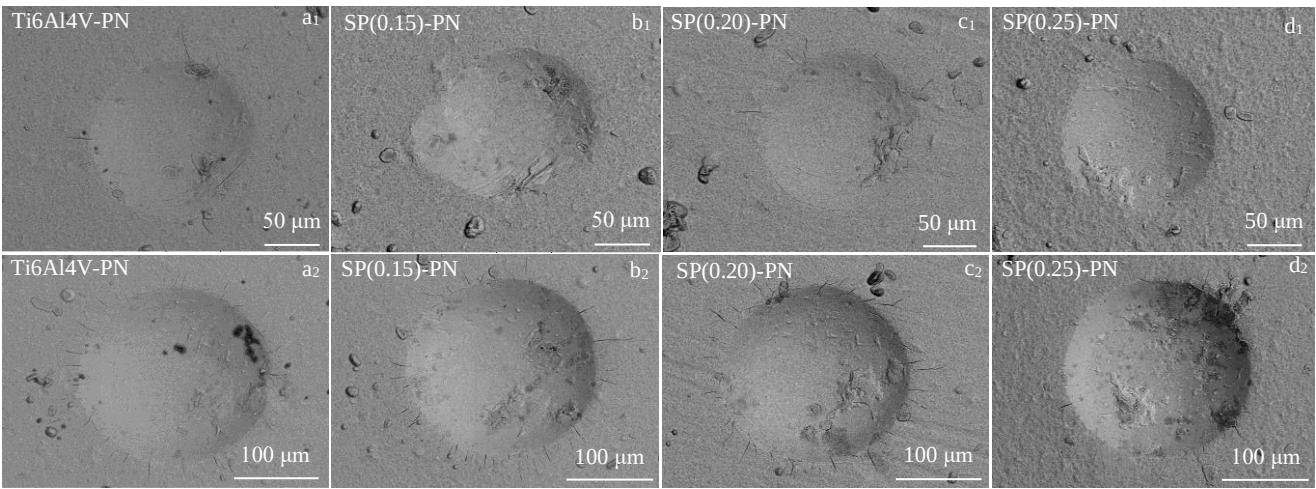


Fig.4 Indentation morphologies of untreated and pretreated nitrided samples under the load of 60 N (a₁~d₁) and 150 N (a₂~d₂)

the highest, which is related to the highest surface hardness and the best bearing capacity.

2.5 Friction and wear properties

2.5.1 Friction coefficient

The variation of the friction coefficient with the sliding time is shown in Fig. 5. It can be seen that the friction coefficient of the Ti6Al4V alloy increases rapidly to around 0.5 in the initial running phase, the running-in phase is completed at about 10 min, and the friction factor is basically stable at around 0.53. The variation of friction coefficient of nitrided samples pretreated with different shot strengths is similar to that of untreated nitrided samples, except that the running-in period is shorter, and the increase range of friction coefficient is not much different after shot peening pretreatment. The friction coefficient comparison of different surface state samples shows that the nitriding treatment cannot reduce the friction factor of the Ti6Al4V alloy.

2.5.2 Section contour and specific wear rate

The section contour of each surface state sample is shown in Fig.6. It can be seen that the wear of the Ti6Al4V alloy is

the most serious, and the deepest point of the wear scar is 30 μm. The maximum depth of wear track of nitrided samples pretreated with 0.15, 0.20 and 0.25 mmA shot strength is about 18, 15 and 8 μm, respectively. That is, as the shot strength increases, the wear resistance of the nitrided sample is gradually increased, which is closely related to the gradual increase of the surface hardness.

After calculation, the wear rate of Ti6Al4V alloy, untreated and pretreated nitrided samples is shown in Fig. 7. It can be seen that the nitriding treatment reduces the wear rate of the Ti6Al4V alloy by 48.9%. That is, nitriding treatment can significantly improve the wear resistance of the Ti6Al4V alloy. Pretreatment with shot peening further improves the wear resistance of the nitrided samples, and the higher the shot strength, the lower the wear rate of the nitrided samples. The nitrided sample pretreated with the highest shot strength (SP(0.25)-PN) has the best wear resistance, whose wear rate is 70.5% lower than that of Ti6Al4V alloy, and 42.3% lower than that of the untreated nitrided sample

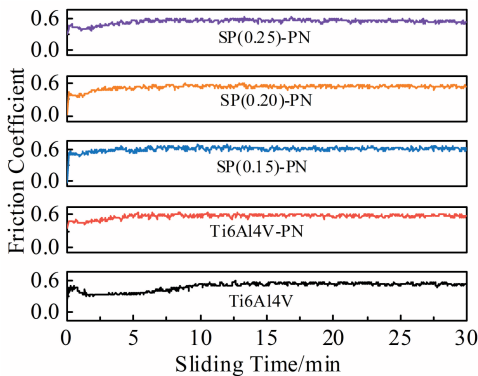


Fig.5 Variation of the friction coefficient of nitrided samples and Ti6Al4V alloy

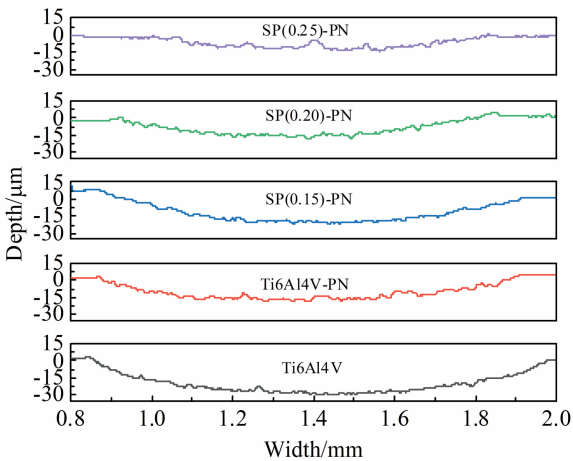


Fig.6 Section contour of pretreated and untreated nitrided sample and Ti6Al4V alloy

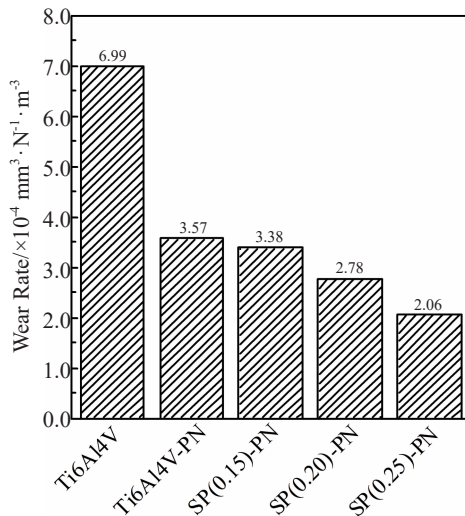


Fig.7 Wear rate of untreated and pretreated nitrided samples and Ti6Al4V alloy

(Ti6Al4V-PN).

2.5.3 Wear scar morphology

Fig.8 shows the wear micro-morphology of Ti6Al4V alloy. Abrasive wear is the main mechanism, accompanied by the adhesion wear. This is attributed to the low hardness of Ti6Al4V alloy and the high hardness of the friction-matched GCr15 steel, which easily cause abrasive wear and adhesive wear of Ti6Al4V alloy^[26]. EDS analysis of the wear scar area of Ti6Al4V alloy shows that the surface contains more O element in addition to Ti element (Table 2), indicating that there is also oxidative wear on the surface of the Ti6Al4V alloy. It is because due to the surface temperature rise caused by frictional wear, the surface activity is increased, and it is easy to react with oxygen in the air to cause oxidative wear. A small amount of Fe element is derived from the adhesion transfer of the GCr15 steel.

Fig. 9 shows the wear morphologies of untreated and pretreated nitrided samples. The results show that the main failure mechanisms of all nitrided samples are abrasive wear, adhesion wear and oxidative wear. That is to say, nitriding treatment does not completely change the wear mechanism of the Ti6Al4V alloy. The nitrided sample pretreated with 0.25

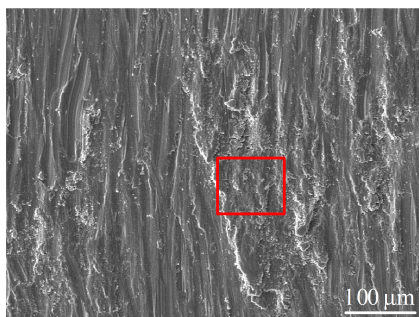


Fig.8 Wear micro-morphology of Ti6Al4V alloy

Table 2 EDS analysis of wear scar surface of Ti6Al4V alloy in Fig.8

Element	wt%	at%
O	21.68	44.35
Al	4.58	5.56
Ti	69.09	47.22
V	2.49	1.60
Fe	2.16	1.27

mmA shot strength has the best wear resistance, and its abrasive wear and adhesive wear are less than those of Ti6Al4V substrate, which is attributed to the high hardness and good apparent toughness of nitrided layers.

2.6 Discussion

Ti6Al4V alloy exhibits poor wear resistance when GCr15 steel with high hardness is used as the counter face for ball disk wear test, which is attributed to its low hardness, poor thermal conductivity and high friction coefficient^[3]. Low-temperature nitriding treatment improves the surface hardness of Ti6Al4V alloy and forms TiN, TiN_{0.3} compound phases on the surface, which can effectively improve abrasive wear resistance, adhesive wear and oxidation wear resistance. Thus, the wear resistance of the Ti6Al4V alloy is improved. Shot peening pretreatment improves the surface dislocation density of Ti6Al4V alloy and forms refined substructure^[27,28]. Further, the short-circuit diffusion route of the nitrogen element to the Ti6Al4V alloy is increased^[29], thereby promoting the low temperature nitriding process of the Ti6Al4V alloy. And as the shot strength is increased, the dislocation proliferation becomes more serious on the surface of the alloy, the microstructure becomes finer, more short-circuit diffusion paths occur, and the chemical activity of the surface becomes stronger. Thereby, the diffusion rate of the N element into the Ti6Al4V alloy is increased, and the formation rate of the TiN and TiN_{0.3} compound phases is higher (Fig. 2). Therefore, the higher the surface hardness of the Ti6Al4V alloy (Table 2), the greater the depth of the strengthening layer, the better the bearing capacity and apparent toughness of the surface layer (Fig. 4). It is worth noting that the nitriding temperature of 500 °C selected in this study is low, which provides insufficient diffusion driving force for N element to Ti6Al4V alloy^[16]. Even if the pretreatment of shot peening is used to increase the short-circuit diffusion path, the diffusion promoting effect of the N element on the Ti6Al4V alloy is not very significant, so that the promotion effect of the shot peening on low-temperature plasma nitriding of Ti6Al4V alloy is not very significant. Therefore, if it is desired to increase the nitriding efficiency and the depth of the nitrided layer during production, the nitriding temperature should be appropriately increased.

3 Conclusions

1) TiN and TiN_{0.3} compound phases form on the surface of untreated nitrided samples (Ti6Al4V-PN). Compared with

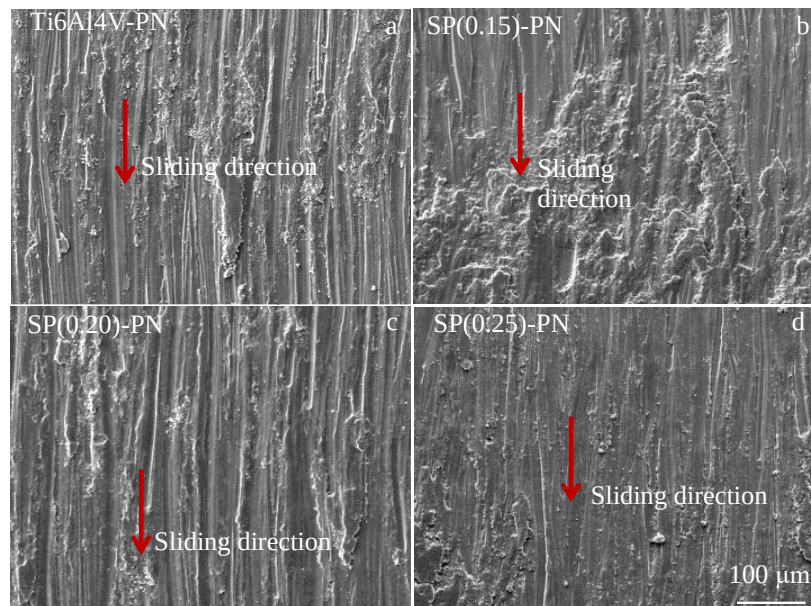


Fig.9 Wear morphologies of untreated (a) and pretreated (b~d) nitrided samples: (b) SP(0.15)-PN, (c) SP(0.20)-PN, and (d) SP(0.25)-PN

Ti6Al4V substrate, the surface hardness and wear rate of nitrided Ti6Al4V alloys are increased by 37.1% and reduced by 48.9%, respectively.

2) Pretreatment of shot peening promotes the low-temperature plasma nitriding process of the Ti6Al4V alloy. As the shot strength increases, the degree of promotion of nitriding process increases. Compared with untreated nitrided samples (Ti6Al4V-PN), the surface hardness of the sample pretreated with 0.25 mmA shot strength is increased by 32.7% and the wear rate is reduced by 42.3%.

3) Pretreatment of shot peening promotes the plasma nitriding process of Ti6Al4V alloy. Due to the increase of dislocation density on the surface and formation of refined microstructures, the diffusion of nitrogen into the Ti6Al4V alloy substrate is promoted. Furthermore, surface activity of the Ti6Al4V alloy is increased to promote the formation of nitride phases.

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喷丸预处理对 Ti6Al4V 合金低温等离子体氮化的影响

刘 珍¹, 于首明², 刘道新¹, 史海兰¹, 何光宇³

(1. 西北工业大学 航空学院, 陕西 西安 710072)

(2. 西安理工大学 材料科学与工程学院, 陕西 西安 710048)

(3. 中国人民解放军空军工程大学 航空航天工程学院, 陕西 西安 710038)

摘 要: 为改善钛合金表面耐磨性能, 同时达到防止薄壁零部件变形和节约能源的目的, 以 Ti6Al4V 钛合金为对象, 研究了喷丸强化预处理对钛合金低温渗氮层及耐磨性的改善作用。结果表明, 喷丸强化预处理能够有效促进钛合金表面低温离子渗氮过程, 在 500 ℃低温渗氮试验条件下, 随着喷丸预处理强度的增大, 钛合金渗氮效率逐步提高, 渗氮层的表面硬度、承载能力和表观韧性逐步增加, 使得渗氮层的耐磨性能逐步提高。当喷丸预处理强度增加到 0.25 mmA 时, Ti6Al4V 钛合金渗氮层的表面硬度比单纯渗氮处理试样提高 32.7%, 磨损率降低 42.3%, 使钛合金基体的磨损率降低 70.5%, 较好地实现了喷丸预处理促进钛合金低温离子渗氮的目标。

关键词: Ti6Al4V 钛合金; 喷丸强化; 低温氮化; 摩擦磨损

作者简介: 刘 珍, 女, 1995 年生, 硕士, 西北工业大学航空学院, 陕西 西安 710072, E-mail: liuzhen_@mail.nwpu.edu.cn