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Tribological properties of 2024 aluminum alloy plasma-based ion implanted with nitrogen then titanium①

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Abstract: 2024 aluminum alloy was implanted with nitrogen then titanium at different titanium target sputtering currents by plasma-based ion implantation(PBII). The appearances were observed by atomic force microscope, and the surface hardness was measured with Knoop hardness tester and the mechanical property microprobe. Ball-on-disc dry wear experiments were performed under ambient air conditions, to study the tribological properties of the modified layers against GCr15 steel ball, employing various loads and a constant sliding speed. After dual modifications, surface hardness at 100 nm depth could reach to 9 GPa, increasing by about 5 times; tribological properties at lower load(e.g. 1 N) were obviously improved, with the friction coefficient(below 0.2) decreasing by over 60%, and the wear life(800 times) increasing by about 5 times. Meanwhile, with the increase of the sputtering current, the appearance is smooth, the surface hardness tends to a slow and even variation, the wear life presents a parabola-like change, and the friction coefficient and the adhesive wear degree decrease. However, tribological properties are reduced with the increase of the load due to the modified layer rapidly getting thin.

Key words: aluminum alloy; plasma-based ion implantation; sputtering current; tribological properties

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1 INTRODUCTION

Owing to low density and high specific strength, aluminum and its alloys are extensively used in many fields, especially in aviation and space industry. But low hardness and low wear resistance often limit their engineering applications. Surface modification for aluminum and its alloys by ion implantation offers the possibility of widening their applications where high wear resistance and low density are required[1-5]. Since nitrogen ion is convenient to obtain and easy to control, and AlN has excellent mechanical properties, nitrogen ion implantation has been intensively investigated to make AlN on aluminum and its alloys in the last few years, resulting in higher surface hardness, lower friction and higher scratch resistance without altering the good bulk properties of the alloys. Particularly, PBII has unique advantages, for example, it has eliminated the line-of-sight restrictions of beam-line implantation, implantation and deposition can be performed alternately, and it is an economical and effective approach for the surface modification, thus it is of an increasing importance to modify materials[5].

However, nitrogen-implanted layer, where nitrogen or AlN often presents Gaussian-like distribution, is generally less than 0.3 μm thick, thereby the surface modification only by nitrogen implantation is also limited. It must be further studied to increase the thickness, improve hard phase distribution and reinforce the alloying effect as to improve the hardness and wear resistance of aluminum alloys. The results have proved that it is able to reach to this aim by proper PBII processes performed on the nitrogen-implanted layer[6].

2024 aluminum alloy plasma-based ion implanted with nitrogen then titanium at various sputtering currents which influence on the structure of the produced modified layers has been investigated[7]. The aim of this paper is to study the effect of the sputtering current on the surface hardness and the tribological properties of the modified samples.

2 EXPERIMENTAL

PBII experiments were performed on the DLZ-01 facility whose details were provided elsewhere[6]. 2024 aluminum alloy substrate samples have a size of d 40 mm × 5 mm for wear test and d 10 mm × 5 mm

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for the others. Prior to implantation, the samples were treated by solution strengthening, mechanically polished to a surface roughness about 25 nm, ultrasonically cleaned with absolute alcohol and acetone for 5−10 min, and dried in air. Before implanted with nitrogen, the samples were sputtered with Ar^+ at a bias voltage of 2 kV for 30 min, to remove the residual surface contaminations, and then the samples were implanted in term of the implanting parameters listed in Table 1. Ar^+ ions were used to sputter titanium target during implantation with titanium.

Table 1 Implantation parameters

Samples	Modified process	Titanium target sputtering current/ mA
S0	Unmodified	
S1	Implanted with N(75 kV, 40 μs, 80 Hz, 180 min) and then with Ti(75 kV, 30 μs, 80 Hz, 60 min)	40
S2	Implanted with N(75 kV, 40 μs, 80 Hz, 180 min) and then with Ti(75 kV, 30 μs, 80 Hz, 60 min)	400
Others	Implanted with N(75 kV, 40 μs, 80 Hz, 180 min) and then with Ti(75 kV, 30 μs, 80 Hz, 60 min)	40 − 400

XPS measured experiments have introduced in elsewhere[7].

The surface hardness measurement was performed with HX-1000 Knoop hardness tester with loading time of 15 s, load of 0.02, 0.03, 0.05, 0.10 and 0.25 N, respectively. The nanohardness at different depths was measured using Nano-Indenter™ II tester with the mechanical property microprobe, indenting depth to surface of 20, 50, 100, 200, 300, 400 and 500 nm, respectively.

The appearances were measured using Nanoscope IIIa-D3000 atomic force microscope (AFM), with long-wide pin, NP-S pin tip, the height of the tip of 180 μm, and the elastic constant of the tip of 0.12 N/m.

The ball-on-disk dry sliding wear experiments against GCr15 steel ball with diameter of 5 mm, hardness of HRC 61 and R_a of 25 nm were performed in an environment with temperature of 20 − 25 ℃ and humidity of 30% − 50%. The applied loads 1, 2 and 5 N were selected, respectively, the sliding speed maintained at 0.06 m/s, and the diameter of the wear track circle maintained at 30 mm.

Wear data were automatically recorded by a computer. Wear life for each modified layer is defined by the number of sliding cycles(n) when the layer was just worn out, i e, a steady fraction coefficient sharply increased to the max, then fluctuated to another steady value. The wear track was observed by scanning electron microscope(SEM) and its depth and width were measured with a profilometer, and then the wear volume was obtained. The wear volume divided with the product of the number of sliding cycle and sliding load is defined as the wear rate whose reciprocal is called the wear resistance.

3 RESULTS AND DISCUSSION

3.1 Composition depth profiles

Fig. 1 shows XPS depth profiles of the modified samples S1 and S2. For S1, as shown in Fig. 1(a), its modified layer is composed of an approximately 90 nm-thick titanium-implanted layer where titanium is decreased along the implanting direction and an about 300 nm-thick nitrogen-implanted layer where nitrogen presents a Gaussian-like distribution. Of course, the two layers are naturally overlapped since nitrogen diffuses out. It is obvious that the modified layer at low sputtering current(40 mA), where titanium is added, is thicker than the single nitrogen implanted layer, with evener nitrogen distribution[7]. For sample S2, there is a titanium-deposited layer besides the similar modified layer of S1, and its thickness is about 480 nm, as shown in Fig. 1(b).

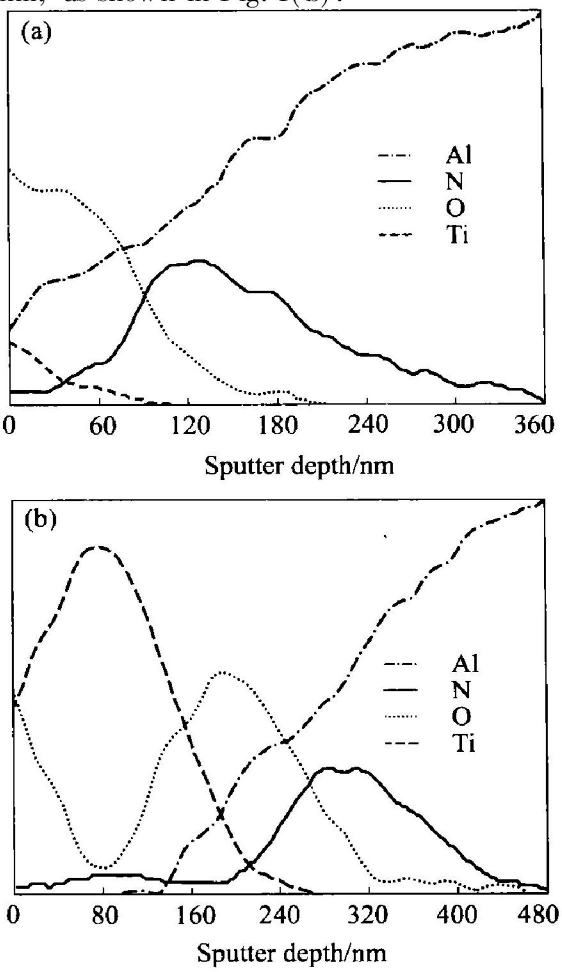

Fig. 1 XPS depth profiles of modified layers of samples
(a) —S1; (b) —S2

3.2 Surface hardness

Fig. 2 shows the Knoop microhardness of samples as a function of load. The hardness decreases with the increase of load, and it has a sharp change in lower range of load, while a slow change in higher range because the hardness combines the effect of the modified layer and the substrate, and the lower load corresponds to the thinner penetrating depth, resulting in more effect of the modified layer; and vice versa[8]. But the hardness of S2, a little lower than that of S1 while the load being 0.02-0.03 N, has a slower and evener change while the load being 0.02-0.25 N, consistent with its depth profiles. The hardness of S0 is nearly that of the substrate (1.41 GPa) since its surface oxide layer is too thin to produce much effect on hardness, while those of S1 and S2 are much higher at each corresponding load, with maximum values of 2.32 and 2.25 GPa, respectively. In addition, their hardness is some higher than that of the single nitrogen-implanted sample[6]. Fig. 3 shows the effect of the sputtering current on Knoop hardness while the load being 0.02 N. It is obvious that the hardness has a parabola-like change with the sputtering current, implying there is an appropriate sputtering current corresponding to an optimal hardness.

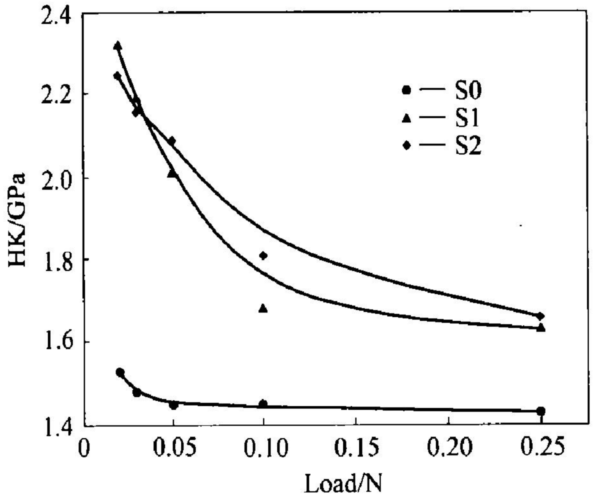

Fig. 2 Knoop hardness as a function of load for S0, S1 and S2

Since the modified layer is so thin, it is more reasonable to determine its absolute hardness quantitatively with mechanical property microprobe[9,10], as shown in Fig. 4. For S0, its hardness decreases from 4.5 GPa in 20 nm rapidly to 1.9 GPa in 100 nm and finally to 1.8 GPa. The hardness of modified samples S1 and S2 is much higher than that of S0 in corresponding penetrating depth, with peak hardness over 9 GPa at 100 nm and 8.4 GPa in 20 nm, respectively. As to S2, its hardness also has a slow and even change from 8.4 GPa in 20 nm to 6.8 GPa in 300 nm and to 2.2 GPa

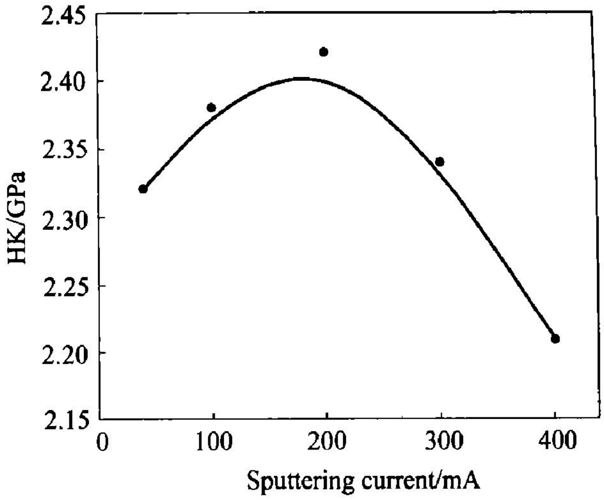

Fig. 3 Knoop hardness (load 0.02 N) as a function of sputtering current

in 500 nm. This is mainly related to the thicker titanium-deposited layer reducing the effect of the substrate, concretely, those from 20 to 200 nm are related to much α-Ti and a little TiO_2 and TiN in the titanium-deposited layer, while those from 200 to 500 nm are associated with the increase of thickness of the modified layer[7]. Thus the sputtering current has effect on hardness by affecting the structure and the thickness of modified layer.

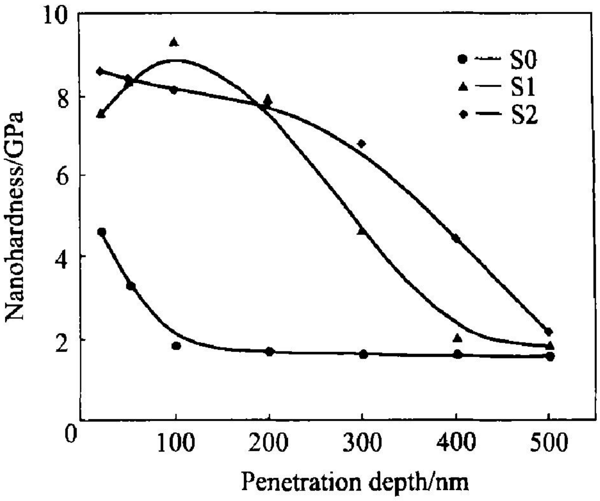

Fig. 4 Nano-hardness as a function of penetrating depth for samples S0, S1 and S2

3.3 Surface appearances

Fig. 5 shows the appearances and roughness of S1 and S2 by AFM. As shown in Fig. 5(a), the appearance of S1 consists of a large number of little heaves and concaves, with R_{max} of 438 nm and R_a of about 20 nm. But the size of heaves and concaves of S1 is some larger than those of the single nitrogen-implanted sample, possibly owes to more defects caused by irradiation damages of Ti^+ implanting and sputtering[11,12]. However, for S2, its appearance is smoother, with smaller and more regular heaves and concaves, R_{max} decreases to about 150 nm and R_a decreases to 15 nm. This closely due to the covering and polishing effects of the continuously deposited titanium atoms[6], as shown in Fig. 5(b). Obviously, the sputtering current also brings influence on the appearances.

3.4 Tribological properties

Fig. 6 shows tribological properties of S0, S1 and S2 as a function of the load. For each sample, with increasing the load, the friction coefficient increases, the wear life shortens and the wear rate increases, respectively. And for S1 and S2, the friction coefficient decreases, wear life prolongs and wear rate decreases at a certain load in comparison with those of S0 or the single nitrogen-implanted sample. Moreover, those of S2 have some improvement compared with those of S1. Specially, those of S1 and S2 with load of 5 N are highly improved than those of S0 with load of 1 N, indicating their load carrying capacities are also improved.

Fig. 7 shows the influence of the sputtering current on tribological properties under various loads. At a certain sputtering current, with increasing the load, the friction coefficient increases, the wear life shortens and the wear rate increases owing to the modified layer rapid getting thin at higher contacting stress; while under a certain load, the friction coefficient decreases, the wear life shows a parabola-like change and the wear rate shows an opposite parabola-like change, implying there is an optimal sputtering current corresponding to appropriate tribological properties.

Fig. 8 shows the SEM morphologies of wear tracks worn out under 1 N for S 1 and S 2 . As shown in Fig. 8(a), on the wear tracks of S1, there are clear grooves and transferred substance with the same compositions as those of S0 using the energy spectrum . The wear track presents adhesive wear. And those of S2 are more evidently

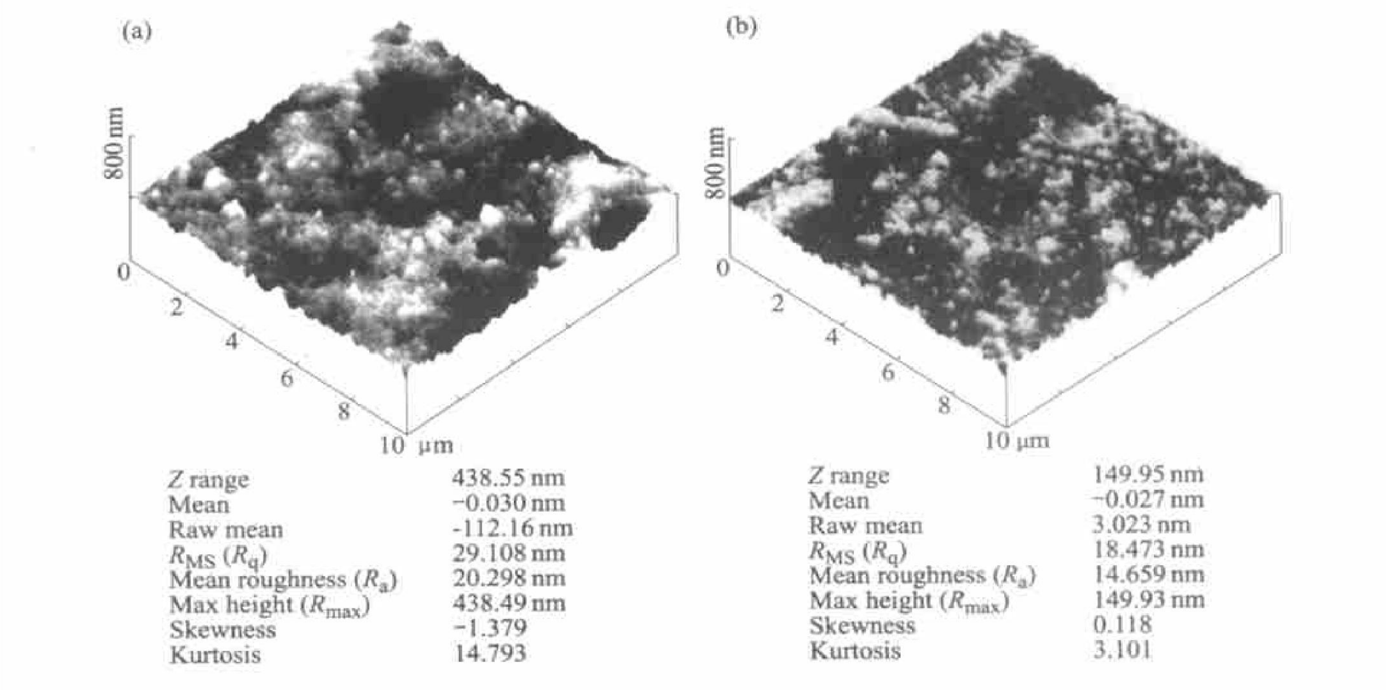

Fig. 5 AFM images and roughness of surfaces for samples S1(a) and S2(b)

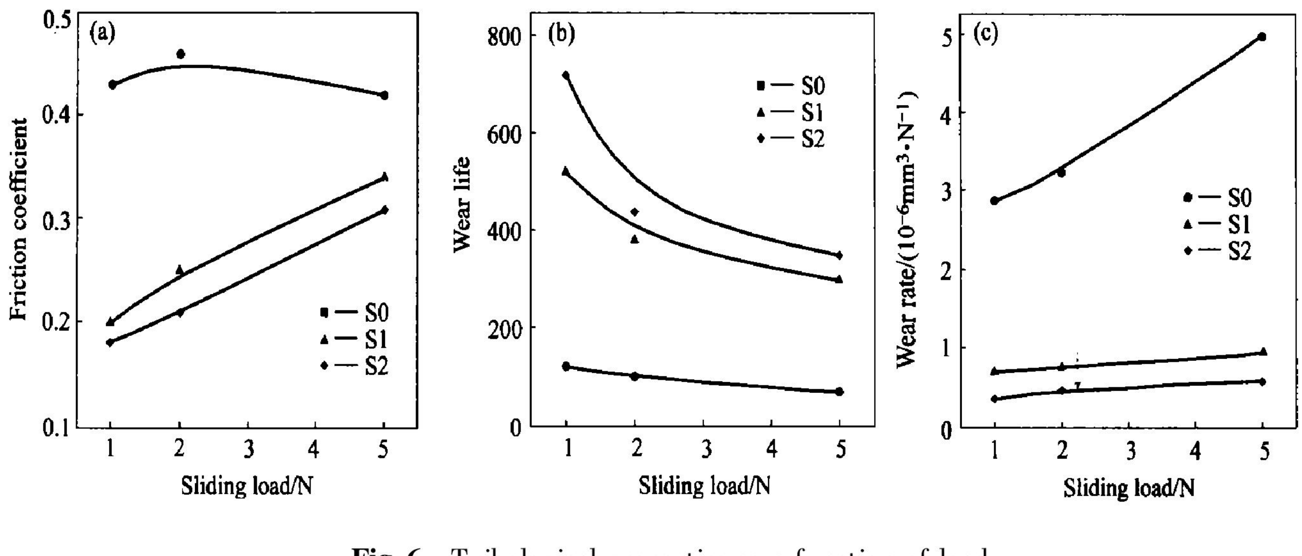

Fig. 6 Tribological properties as a function of load

(a) —Friction coefficient; (b) —Wear life(times); (c) —Wear rate

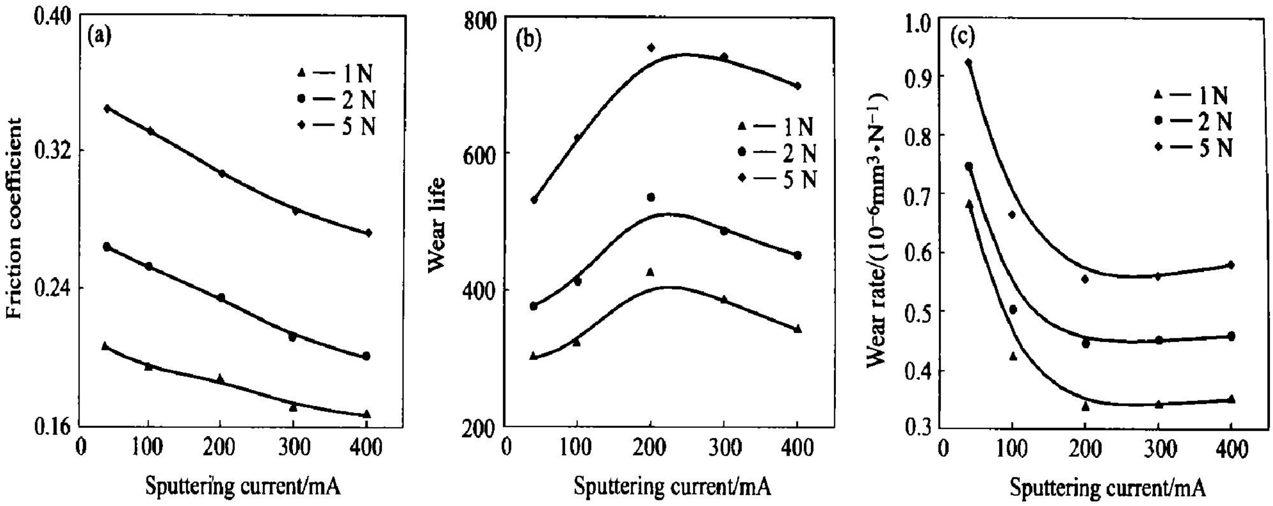

Fig. 7 Tribological properties as a function of sputtering current
(a) —Friction coefficient; (b) —Wear life(times); (c) —Wear rate

Fig. 8 SEM morphologies of wear tracks worn out under 1 N for S1(a) and S2(b)

improved than those of S1, as revealed in Fig. 8(b). The wear tracks are smooth with little transferred substance, where more shallow grooves form only by plastic deformation, and with few worn out, so its adhesive degree distinctly decreases mainly due to the increase of the thickness of the modified layer able to reduce contacting stress[9, 13, 14].

The structure of the modified layers has obvious improvement. As indicated in Ref. [7], the structure of modified layer strongly depended on the titanium target sputtering current being a threshold value. When the sputtering current is lower than that value, the modified layer is improved in the composition distribution, as shown in Fig. 1(a), i e, it shows more evident strengthening effects such as alloying and fine dispersion strengthening than the single nitrogen-implanted layer[1, 6], thus it presents more improvement in hardness and tribological properties. When the sputtering current is higher than that value, the modified layer is more considerably improved in thickness and appearance than that of S1, as displayed in Fig. 1(b) and Fig. 5. Although the titanium-deposited layer containing much α-Ti can not make marked contributions to surface hardness value, it is helpful to making the surface hardness change slowly and evenly, dispersing or reducing the friction contacting stress and preventing the cracks from propagating[9, 13, 14], and it is also beneficial to reducing the adhesive degree, as exhibited in Fig. 8.

However, when the sputtering current is too low, the thickness of the modified layer is difficult to be increased, thus the appearance, the surface hardness and tribological properties are difficult to be improved. Contrarily, when the sputtering current is too high, the titanium-deposited layer is too thick and contains too much α-Ti, resulting in markedly negative effects on the properties. Therefore, there is an optimal sputtering current corresponding to appropriate surface hardness and tribological properties, as shown in Fig. 3 and Fig. 7.

As mentioned above, implanting titanium can improve the surface hardness and tribological properties of aluminum alloy to some extent. However, it is not suitable for the final modification because the titanium-deposited layer contains much α-Ti. If a large amount of nitrogen is introduced into the titanium-deposited layer, or DLC films are formed on it, or other proper steps are taken on it, the surface hardness and tribological properties will be improved remarkably.

4 CONCLUSIONS

1) 2024 aluminum alloy is improved in appearances, surface hardness, tribological properties by plasma-based ion implanted with nitrogen then titanium.

2) The sputtering current has brought evident effect on appearances and properties. With increasing the sputtering current, the appearances are smooth, the surface hardness tends to a slow and even variation and the adhesive wear degree is reduced; and there exists an appropriate sputtering current value corresponding to optimal surface hardness and tribological properties.

3) Tribological properties reduce with increasing the load since the modified layer rapidly gets thin.

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