Temperature elevated N ion implantation of Ti6Al4V alloys using the plasma source

ZHAO Qing, ZHENG Yong-Zhen, Mo Zhi-Tao, TONG Hong-Hui, GENG Man

(*Southwestern Institute of Physics, Chengdu 610041*)

Abstract Specimens of Ti6Al4V alloy were implanted with nitrogen ions of $4\times10^{18}\,cm^{-2}$ at temperatures from 100 to 600°C. Auger Electron Spectroscopy (AES), microhardness measurements and pin-on-disk wear testing, Scanning Electron Microscopy (SEM), and Glancing angle X-ray Diffraction (XRD) were utilized to evaluate the surface property improvements. The thickness of implanted layers increased by about an order of magnitude when the temperature was elevated from 100 to 600°C. Higher surface hardness and wear resistance were also obtained in the high temperature implantation. The XRD image showed the presence of nitrides of titanium at the implanted surface.

Keywords Ti6Al4V alloy, Anti-wearing, Plasma source ion implantation of Nitrogen

CLC numbers TG146.2+3, TN305.3, TG155.2+2, TG115.5 A

1 INTRODUCTION

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Ti6Al4V alloys widely applied [illegible] cal processing facilities, to med[illegible]ical implants, and so on bec[illegible] ical and chemical properties such as high strength-to-weig[illegible]e[1]. However, the wear behavior of titanium alloys in a tr[illegible] is [illegible] satisfactory[2,3]. There are reports on the effects of nitrogen ion implantation on hardness, wear resistance, composition and structure of Ti6Al4V[4,5]. However, so far the process is not economical as to industrial application on complicate workpieces in metallurgical sector. One of its major limitations is very shallow depth of penetration achieved. Ions having typical energies in the range of 50~90 keV, produce a hardened region which is only 60~100 nanometers deep. For most engineering applications experience wears, this level of hardened zone thickness is insufficient.

Treatment at elevated temperatures by plasma source ions implantation(PSII) is a suitable and effective means of nitriding steels[6]. It gives rise to increased surface hardness, and a nitrogen-strengthened diffusion zone is produced that extends beyond the implantation range and improves the load-bearing capacity of implanted lay[7,8].

In this paper, we concentrate on the diffusion related phenomena that occur during PSII at elevated temperature and describes the results of different temperature nitrogen plasma source ion implantation (N-PSII) of Ti6Al4V alloy.

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2 EXPERIMENTAL PROCEDURES

Annealed $\alpha+\beta$ Ti6Al4V alloy with dimensions of 60mm×60mm×60mm were used as base material, and cut out into pieces. The 30mm×20mm×5mm pieces were used in wear and microhardness test and the 15mm×10mm×5mm pieces were used mainly for AES (Auger Electron Spectroscopy) and XRD (X- ray diffraction) analysis. All samples were mirror polished to a final roughness of 0.02 μm.

Fig.1 shows a schematic diagram of the PSII-IBED (the plasma source ion implantation-ion beam enhanced deposition) system. More details about this device are given elsewhere[9]. Prior to treatment, the system is pumped to a base pressure of at least 1.5×10^{-3} Pa to minimize oxygen contamination. The chamber is then filled with nitrogen to the working pressure of 5.3×10^{-3}Pa. The plasma will be generated by a hot-cathode discharge, whilst the ions are accelerated from the plasma by high voltage pulses (40 kV) applied directly to the workpiece. Pulse frequencies in the range of 100-600Hz, with a pulse duration of 20μs, are employed in order to achieve the desired temperature of 100, 200,400 and 600°C. The treatment was carried out for 833, 417, 208 and 130 min, respectively, and then the samples were cooled in vacuum. Total applied doses were of 4×10^{18}ions·cm^{-2}. The temperature of the backside of the specimens was monitored by a K-type thermocouple, more details about measurement system have been reported in Ref.[10].

The surfaces of all samples, both treated and untreated, were characterized by XRD. Auger Electron Spectroscopy(AES) with Ar+ ion gun sputtering was used to determine the nitrogen concentration depth profiles. The microhardness testing was conducted on an Hα-1 microhardness indenter system with loads of 25 g. Wear measurements were performed using a pin-on-disk wear tester. Samples are mounted on a rotating disk and a SiC ball of 5 mm in diameter is impressed on the samples with a 25 g load without lubrication. The disk is rotated at 150 r/min. Each piece was tested for 2000 revolutions. The wear tracks and the SiC ball wear scars were examined using SEM.

3 RESULTS AND DISCUSSION

The microhardness of the samples implanted at various temperatures is compared with the value of the untreated sample as shown in Fig.2. It's seen from Fig.2 that the microhardnesses have been significantly improved comparing with the untreated sample, although there is a slight improvement in the surface hardness of the sample treated at 100°. The maximum hardening is observed at 600°C. From these results, it can be seen that by selecting suitable processing temperature, the hardness increases in the range 150~200% are possible. These can be associated with the formation of hard and stable nitride phase in the near surface region of the sample at different temperatures, which is confirmed by the XRD results. Our observations are in agreement with those reported by Johns[11].

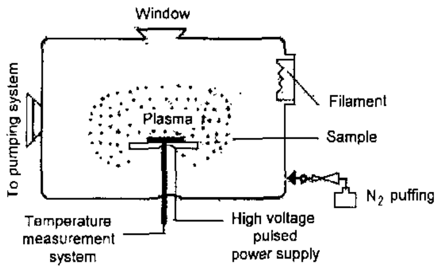

Fig.1 Schematic diagram of PSII-IBED system

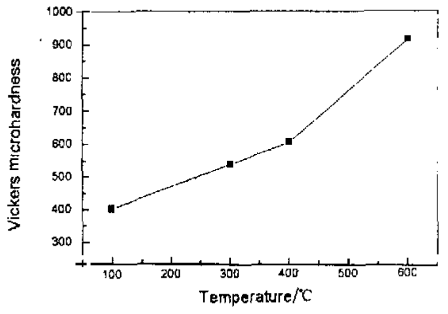

Fig.2 Microhardness as a function of temperature at load of 25 g

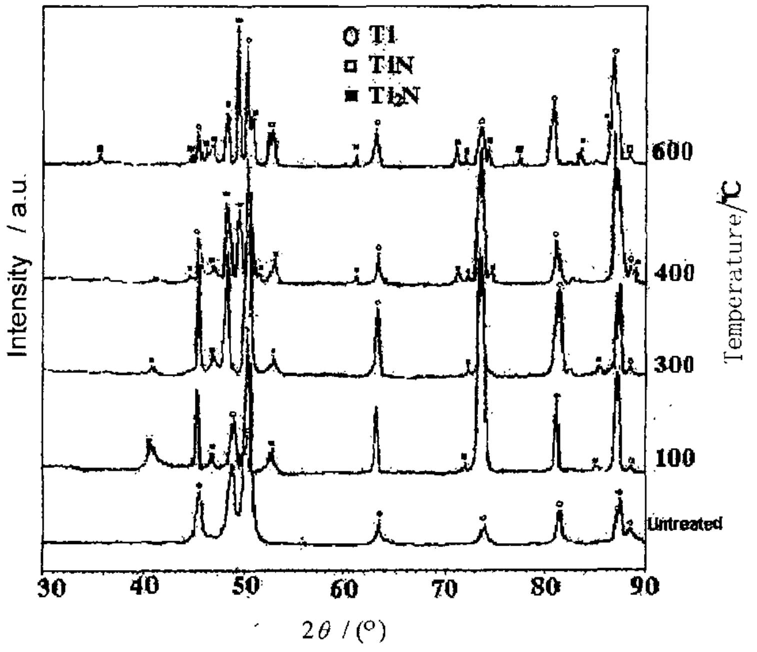

Fig.3 XRD patterns from the nitrogen-PSII-treated samples at different temperatures

X-ray diffraction patterns for all specimens are shown in Fig.3. No nitride is detected in samples treated with N-PSII at 100°C. The presence of TiN is observed in the surface microstructure of samples treated at 300°C. The peaks for this phase are small and difficult to be distinguished. The TiN peaks are clearly visible at 400°C and relatively large at 600°C. Ti_2N peaks are clearly observed simultaneously. It suggests that a greater volume fraction of TiN and Ti_2N has been evolved in the substrate region of the Ti6Al4V sample. This may be a result of the higher doses imparted by N-PSII at this temperature, as well as the enhanced mobility of nitrogen at higher temperatures.

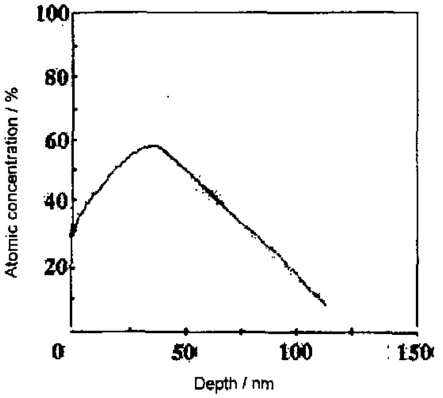

Fig.4 Nitrogen concentration profile of the sample implanted at temperature 100°

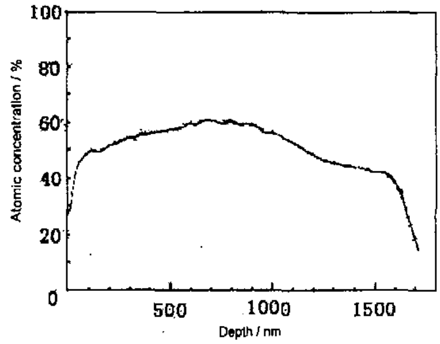

Fig.5 Nitrogen concentration profile of the sample implanted at temperature 300°C

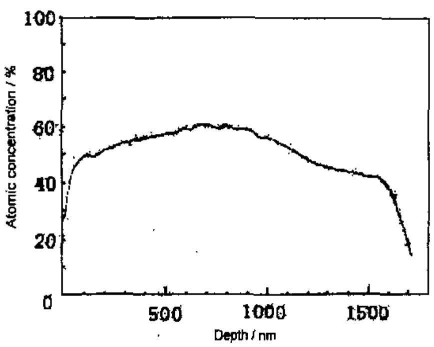

Fig.6 Nitrogen concentration profile of the sample implanted at temperature 400°C

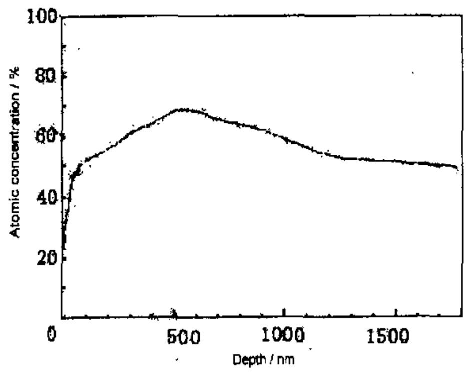

Fig.7 Nitrogen concentration profile of the sample implanted at temperature 600°C

AES profiles for nitrogen on the surface of the samples treated at 100°C, 300°C, 400°C and 600°C are presented in Figs.4~7, respectively. At the lowest temperatures (100°C), we obtained a surface peak characteristic of implantation alone (see Fig.4). At 300°C, we observed a wide peak which includes implantation and diffusion (see Fig.5), the peak concentration of nitrogen reaches about 62at.%. while the temperature increases to 400°C (seeing Fig.6), the profile shows a higher peak concentration of nitrogen about 72at.%. For a higher implantation temperature of 600°C (see Fig.7), the nitrogen peak concentration drops to about 65at.%, and the nitrogen distribution becomes a broadened plateau indicating a thicker nitride layer formed at the higher temperature. These results show enhanced nitrogen retention and diffusion to depths greater than 1 μm at the elevated temperature implantation in Ti6Al4V alloy.

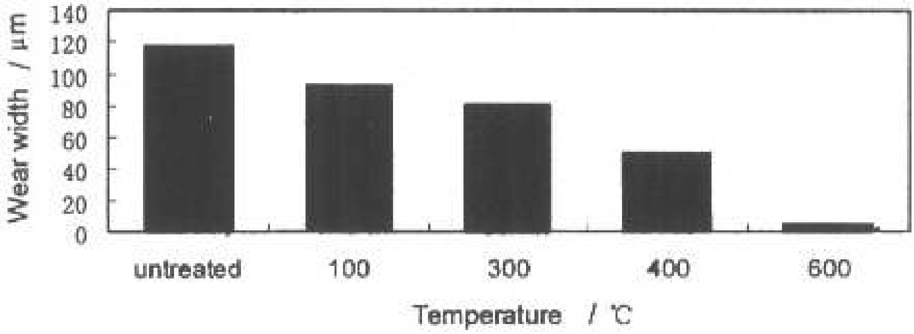

Fig.8 Width of the wear scar after 2000 revolutions of pin-on-disc wear test (25g load SiC pin)

Fig.9 SEM micrograph of the wear scar treated at 100°C

Fig.10 SEM micrograph of the wear scar treated at 600°C

The effect of surface modification was observed in the wear testing on the wear width of the treated sample compared to the untreated one. Fig.8 depicts the wear width with a load of 25 g after 2000 turns. For the untreated piece, wear tracks were wide and deep. Similar results were also exhibited by SEM micrographs of wear tracks (Fig.9) of Ti6Al4V sample treated by N-PSII at temperatures of 100°C, indicating no clear improvement of the wear resistance. However, significant improvement in wear rates is observed for the sample treated at 600°C, very shallow and narrow wear tracks can be seen in Fig.10. This large decrease is possibly due to the nitrogen precipitates near the surface area and a thick modified layer formed after the high temperature implantation. Analogues effects on the surface of alloy 909 were reported by Li *et al*[8].

4 CONCLUSIONS

The treatment of Ti6Al4V alloy at temperatures of 300~600°C gives higher surface hardness and deeper nitrogen-strengthened diffusion zones, resulting an increase in the load-bearing capacity of the implanted layer. N-PSII process of Ti6Al4V alloy at a temperature of 600°C resulted in higher hardness and wear resistance due to significantly deeper penetration of nitrogen and stable Ti_2N and TiN precipitate. Thus diffusion has been shown to play an important role in the N-PSII process.

References

1 Donarchie M J ed. Titanium and titanium alloys. American Society of Metals. Metals Park. Ohio. 1982.
2 Elder J E, Thamburaj R, Patnaik P C. Surf Eng, 1989. **5**:55
3 Mucha A. Braun M. Surf Coat Technol, 1992, **50**:135
4 Han S. Kim H. Lee Y. Surf Coat Technol, 1996, **82**:270
5 Loinaz A, Rinner M, Alonso F *et al.* Surf Coat Technol, 1998, **103-104**:62
6 Blawert C, Mordike B, Collins G A *et al.* Surf Coat Technol, 1998. **103-104**:240
7 Collins G A. Hutchings R, Tendys J. Mater Sci Eng, 1991, **A139**:171
8 Li X, Samandi M, Dunne D *et al.* Surf Coat Technol, 1996. **85**:28
9 Shang Z K, Chen K Q, Zeng X C *et al.* Surf Coat Technol, 1996, **85**:105
10 Zhao Q, Tong H H, Chen Q Q *et al.* Nuclear Fusion and Plasma Physics (in Chinese), 2000. **23**:48
11 Johns S M, Bell T, Samandi M *et al.* Surf Coat Technol, 1996. **85**:7