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Structure and tribological performance of helium-implanted layer on Ti6Al4V alloy by plasma-based ion implantation

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ABSTRACT

The present paper concentrates on tribological performance of Ti6Al4V alloy treated by helium plasma-based ion implantation with a voltage of −30 kV and a dose range of 1, 3, 6 and 9 × 10^{17} He/cm^2. X-ray photoelectron spectroscopy (XPS), Transmission electron microscopy (TEM) and Atomic force microscopy (AFM) were used to characterize composition, structure and surface morphology, respectively. The variation of hardness with indenting depth was measured and tribological performance was evaluated. The uniform cavities with a diameter of several nanometers are formed in the helium-implanted layer on Ti6Al4V alloy. Helium implantation enhances the ingress of O, C and N and produces TiO_2, Al_2O_3, TiC, TiN in the near surface layer on their removal from the vacuum and exposure to normal atmospheric condition. In the near surface layer, the hardness of implanted samples increases remarkably comparing with the untreated sample, and the maximum peak increasing factor is up to 2.9 for the sample implanted with 3 × 10^{17} He/cm^2. A decrease in surface roughness, resulting from the leveling effect of sputtering and re-deposition during implantation, has also been observed. Comparing with the untreated sample, implanted samples have a good wear resistance property. And the maximum increase in wear resistance reaches over seven times that of the untreated one for the sample implanted with 3 × 10^{17} He/cm^2. The wear mechanism of implanted samples is abrasive-dominated.

1. Introduction

Helium ion implantation into titanium and titanium alloys has attracted more and more attention due to applications such as fusion reactor technology, sophisticated catalysis, biocompatible implants, selective solar absorbers and radiation protection materials [1–4]. Ion-implanted helium is insoluble in metal and easily trapped by defects and precipitated out to form gas bubbles under appropriate conditions. When gas escapes from bubble, cavity structures can be produced in the implanted layer on metal substrate [5,6]. Comparing with beam-line ion implantation, the interest in plasma-based ion implantation (PBII) comes from recognition that surface with large areas and complicated shapes may need to be implanted. Also the lower energies involved in PBII can form cavities closer to the surface and cavities near surface are beneficial to catalytic application and so on [4,7]. Although, a few studies have reported the formation of cavities on titanium alloy [8,9], there has been lack of information on hardness and tribological performance. Kennedy et al. [10] found that helium implantation enhances the ingress of O and N into the implanted layer on exposure to atmosphere conditions. Beyond doubt, the ingress of O and N into the implanted layer on titanium alloy surface assuredly influences their surface hardness and tribological performance. This paper focuses on the structure and tribological performance of the helium-implanted layer on Ti6Al4V alloy.

2. Experimental details

Commercial Ti6Al4V alloy with a microstructure of α-phase plus strip β-phase was used as the substrate materials. Before treatment the samples with dimensions of 20 × 20 × 5mm^3 were mechanically ground and polished to a mirror-finish. The PBII treatment was carried out in a type DLHZ-01 installation of Harbin Institute of Technology at a base pressure of 5 × 10^{-3} Pa and a working pressure of 0.1 Pa with helium gas of 99.999% in purity. The samples were sputter-cleaned with 1 kV argon ion for 20 min before implantation. The helium plasma was generated by radio frequency (RF). The target temperature was kept at approximately 350 °C. Implantation energy of −30 kV was conducted with doses of 1, 3, 6 and 9 × 10^{17} He/cm^2.

X-ray photoelectron spectroscopy (XPS) analysis was performed using a PHI 5700 ESCA spectrometer employing an Al Kα

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(1486.6 eV) X-ray source operated at 13 kV and 300 W. Depth profiles were obtained by sputtering with a 3 kV argon ion beam. Analysis and sputtering were performed in an alternating mode with a sputtering rate of about 1.3 nm/min. The cavity structure was characterized by a JEM-2010 transmission electron microscopy (TEM). TEM specimens were prepared through grinding the unimplanted side to less than 50 μm with emery paper, and thinning by chemical polishing on a single side (the implanted side was protected). Nano-hardness tests were performed by continuous stiffness measurement (CSM) method in a type Nano Indenter XP of American MTS Corporation. The hardness is defined by the ratio of the load to the projected contact area, $H = P/A$. Six indentations were made in each sample and final hardness was the average value of the six indentations. The tribological experiments were conducted on a ball-on-disk tester of a type CJS111A. Conditions of the tribo-experiments are listed in Table 1. The worn surface morphology was observed by scanning electron microscopy (SEM).

3. Results and discussion

3.1. Composition and structure

Fig. 1 shows the TEM images of the near surface layer and helium-implanted layer for untreated and implanted samples, respectively. Compared with untreated sample (Fig. 1(a)), implanted samples have a cavity layer, in which the cavity size is several nanometers, and moreover, the cavity size increases with the increase of dose (Fig. 1(c)). After 10 min of argon ion sputtering, XPS spectra of the implanted sample are shown in Fig. 2. Apart from the expected Ti, the survey spectrum (Fig. 2(a)) clearly indicates the presence of O, C and N, and their contents are 28.9, 59.26, 10.8 and 1.04 at.% for Ti, O, C and N, respectively. In the near surface layer, the ingress of O, C and N can be attributed to the improvement of the surface activity and the generation of defect channels by implantation. Therefore, O, C, N from the atmosphere are easy to adsorb and enter into the substrate. The oxygen content in the near surface layer is the highest due to its higher activity. In order to identify the state of the ingress elements, O1s, C1s and N1s core spectra were deconvoluted. And element reference energies, which were employed for XPS spectra deconvolution, are listed in Table 2. In Fig. 3, the deconvolutions reveal that the ingress of oxygen is combined with Ti and Al to form TiO_2 and Al_2O_3. Carbon presents as TiC from contamination, and nitrogen presents as TiN. AFM images of the sample surface before and after implantation are shown in Fig. 3. Implantation induces a significant decrease in surface roughness Rq from 4.823 nm to 2.977 nm due to the leveling effect of sputtering and re-deposition during implantation.

3.2. Nano-hardness

The variation of hardness with indentation depth was shown in Fig. 4. At the depth of about 100 nm from surface, all implanted samples have a noticeable increase in hardness compared with the untreated one, and the increasing factor of peak hardness reaches 1.2–2.9. A maximum peak hardness of 15.4 GPa was achieved by the 3×10^{17} He/cm^2 dose implantation. The above XPS results demonstrate that TiO_2, Al_2O_3, TiC and TiN were formed in the near surface layer. It is believed that these compounds are the main reason for the improved hardness. In addition, the hardness increase might also be partially attributed to the effect of the lattice distortion, the dislocation hardening, and the residual compressive stress formed by implantation.

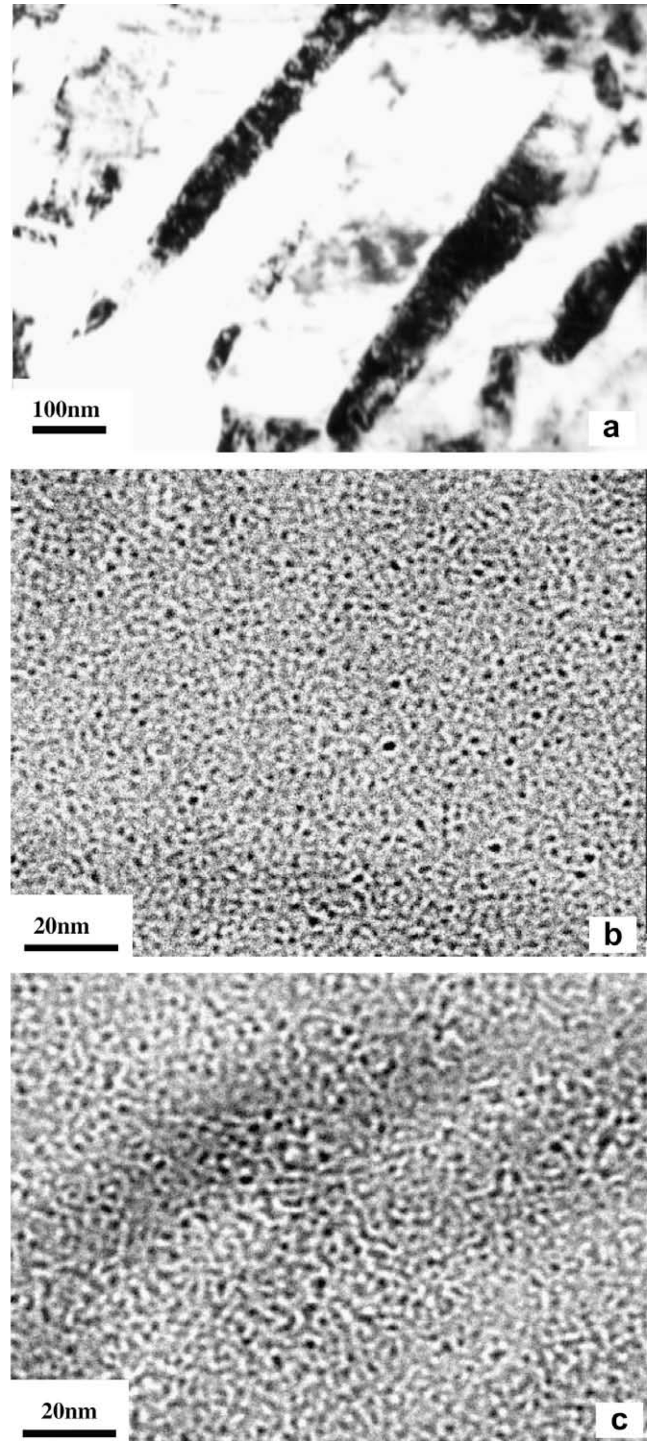

Fig. 1. TEM images of near surface layer and helium-implanted layer of (a) untreated sample, (b) −30 kV, 3×10^{17} He/cm^2, (c) −30 kV, 9×10^{17} He/cm^2, respectively.

Table 1
Conditions of tribo-experiments.

Disk	Ti6Al4V implanted and untreated samples
Ball (φ6 mm)	GCr15 steel
Load (N)	0.98
Diameter of wear circle (mm)	7
Sliding speed (mm/s)	2.93
Environment	Dry air
Temperature	Room temperature