

A Study of Nitrogen Ion-Implanted Ti-6Al-4V ELI by Plasma Source Ion Implantation at High Temperature

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Plasma source ion implantation target temperatures are estimated by measuring the diffusion coefficient of nitrogen in the target and subsequently deducing the temperature. The diffusion coefficient is measured by comparing measured nitrogen concentration profiles to Monte Carlo simulations. Auger results indicated the affected zone of implantation is about 0.5 μm thick. Surface Knoop hardness improved from 400 to 900 (for 1-g applied load). Wear behavior was studied using a pin-on-disk wear tester. The wear data show a factor of 30 increase in wear lifetime (1 μm wear depth was chosen as the failure criterion). Comparison shows that the wear behavior of Ti-6Al-4V ELI (extra low interstitial) is as good as that of cobalt-chromium alloy, another candidate for surgical applications.

1. Introduction

ION implantation has been shown to be an effective process for improving the wear, corrosion, and fatigue properties of metals, and a new approach called plasma source ion implantation (PSII)^[1-5] shows promise for wide industrial application. One application of this surface-modification technique is the implantation of Ti-6Al-4V with nitrogen to increase the wear resistance of artificial hip and knee joints. Previous work has concentrated on room-temperature PSII, and encouraging results have been achieved.^[6,7] However, considering that the implantation-affected zone of room-temperature PSII is very shallow (less than 2000 Å), its utility for components requiring long lifetimes is a concern. In response to this concern, high-temperature PSII was developed to obtain a thicker implanted zone so that the wear properties may be improved.

2. Experimental Procedure

Ti-6Al-4V ELI flat specimens were cut to 25-mm square, 3 mm in thickness from 150-mm bar stock and prepared for implantation by mechanically grinding and polishing to 0.06 μm (from peak to valley). The original microstructure was α + intergranular β resulting from simple mill annealing.

The implantation was done at 50 keV to a retained dose of 6×10^{17} atoms/cm². The base pressure was 2×10^{-6} torr, and the operating neutral pressure was 3.5×10^{-4} torr. The average power density was 3 W/cm² including secondary electron emission (the true power density is lower than this value), which is about ten times higher than that of room-temperature PSII.

Wear tests of cobalt-chromium alloy specimens and the unimplanted and implanted Ti-6Al-4V ELI specimens were conducted under identical conditions using a pin-on-disk wear tester with a 3-mm diameter ruby ball and using Hanks' solution (a human body electrolyte salt simulation) as lubricant.

The rotational speed of the disc was 40 rev/min, giving a linear speed (ball relative to the disc) of 25.1 mm/s for a wear track with a diameter of 12 mm. The applied load is 10 g, giving a Hertzian stress of $\sigma_{\text{max}} = 458$ MPa, which is about 37 times higher than the contact stress $\sigma = 12.4$ MPa, recommended by ASTM^[8] and less than half of the yield stress for Ti-6Al-4V ELI. The wear test was stopped occasionally to measure the

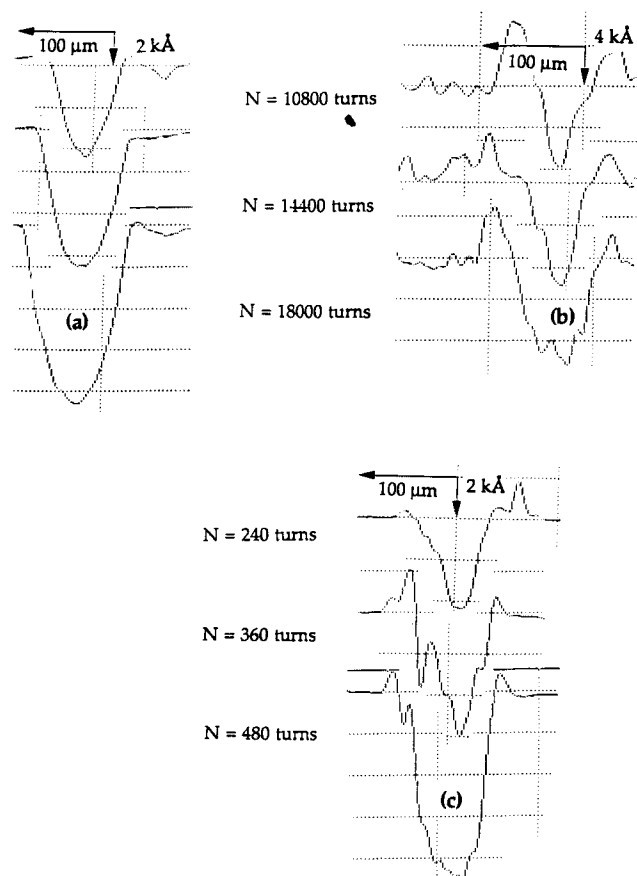


Fig. 1 Typical wear track profiles. (a) Cobalt-chromium alloy. (b) High-temperature PSII-treated Ti-6Al-4V ELI. (c) Untreated Ti-6Al-4V ELI.

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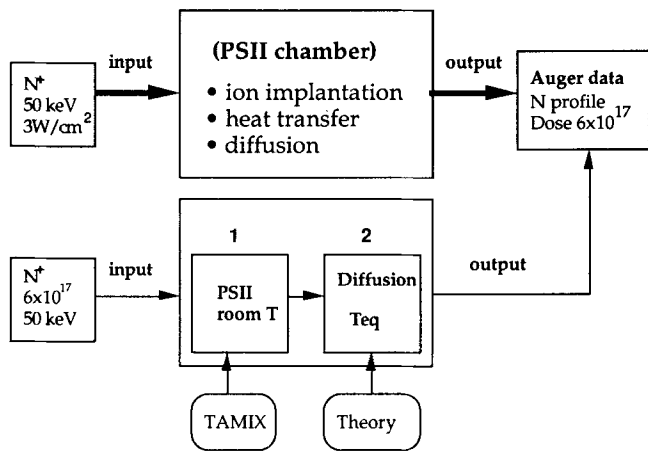


Fig. 2 Estimation of equivalent temperature.

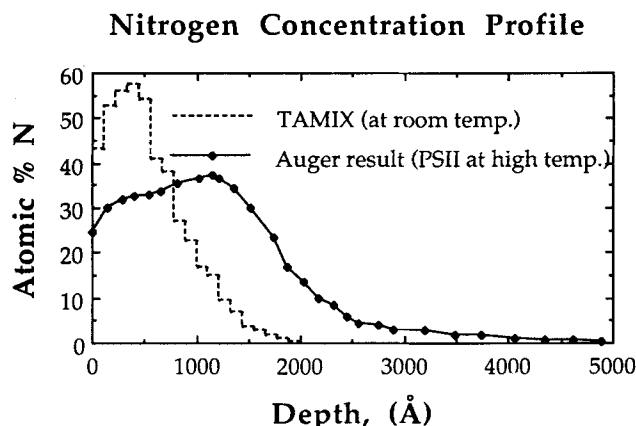


Fig. 3 Nitrogen concentration profiles.

wear track, using an Alpha-step profilometer. A typical series of wear track profiles is shown in Fig. 1.

3. Theory

Direct measurement of a PSII target temperature is quite difficult, due to the presence of high voltage bias on the target. Therefore, specimen temperatures are estimated in this study by comparing measured implantation profiles to computer simulations of the profile, taking advantage of diffusional effects to predict the specimen temperature. Because there is a temperature gradient in the specimen, the concept of equivalent temperature was used for simplification. At this equivalent temperature, diffusion alters the implantation profile from the classical gaussian. Thus, one can compare simulations of implantation without diffusion to measured profiles with diffusion to estimate the diffusion coefficient. Knowing the temperature dependence of the diffusion coefficient, the equivalent temperature can be deduced from the value for the diffusion coefficient. The concept is shown schematically in Fig. 2, and the calculation process is as follows:

Microhardness Variation with Applied Load

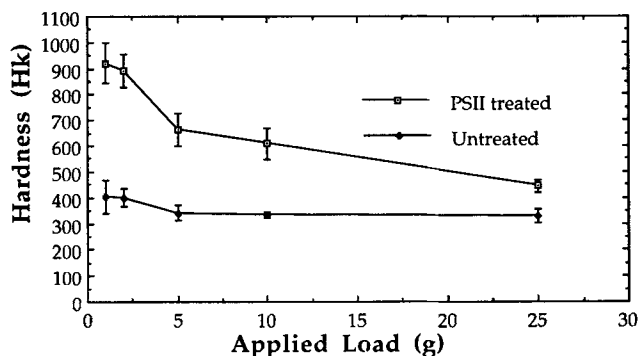


Fig. 4 Microhardness variation with applied load.

1. Run a simulation (the Monte Carlo Code TAMIX)^[9] at room temperature (without diffusion) to obtain the nitrogen concentration profile
2. Use the basic diffusion theory to estimate the true diffusion coefficient from the measured implantation profile (solid curve in Fig. 3) and simulation profile (dotted curve in Fig. 3), according to the following relation:

$$D = (\sigma_1^2 - \sigma_0^2) / 2t \quad [1]$$

where D is the diffusion coefficient, σ_0 represents the half-width-at-half-maximum of the simulated profile without diffusion, σ_1 represents the half-width-at-half-maximum of the measured profile with diffusion, and t represents the diffusion time.

3. Calculate the equivalent temperature using the temperature dependence of the diffusion coefficient, according to:

$$D = D_o \exp(-Q/kT_{eq}) \quad [2]$$

where D_o represents the frequency factor, Q is the activation energy for diffusion, and T_{eq} is the equivalent temperature. For this study the constants were chosen to be:^[10]

$$D_o = 0.21 \text{ cm}^2/\text{s} \quad [3]$$

$$Q = 2.32 \text{ eV} \quad [4]$$

and the specimen temperature was estimated to be about 600 °C. In this study, the Monte Carlo Code TAMIX was used for the room-temperature simulation.

4. Results and Discussion

Using the scanning auger microscope (SAM), the nitrogen concentration profile of the implanted specimen is shown in Fig. 3. The implantation-affected zone is approximately 5000 Å, with a peak nitrogen concentration of about 40 at%. The surface Knoop hardness was measured for both implanted and unimplanted cases using a microhardness indenter and is summarized in Fig. 4. As the applied load increases, the hardness decreases, and after PSII treatment, the hardness increases more than two times for a 1-g load. No difference was found in

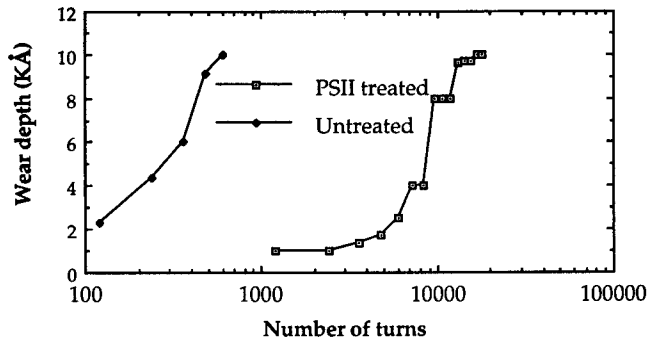


Fig. 5 Pin-on-disk wear depth versus number of turns for Ti-6Al-4V ELI.

bulk hardness between implanted and unimplanted cases (H_R is about 31).

Wear data are shown in Fig. 5 for both untreated and PSII-treated Ti-6Al-4V ELI specimens. It is clear that, even after the wear track depth exceeds the implantation-affected depth (about $1\ \mu\text{m}$), wear resistance is still improved compared to the untreated case. If $1\ \mu\text{m}$ of wear track depth is chosen as a failure criterion, the wear lifetime is increased by a factor of 30 after PSII treatment. Because the release of wear debris is a major concern to clinical applications,^[11-14] the cross-sectional area of a wear track, which is related to the wear mass loss, is compared in Fig. 6 to results for another candidate cobalt-chromium alloy, which is considered superior for wear. It can be seen clearly from this figure that high-temperature PSII-treated Ti-6Al-4V ELI exhibits wear resistance comparable to the cobalt-chromium alloy.

5. Summary

The target temperature during nitrogen plasma source ion implantation on Ti-6Al-4V ELI is estimated by comparing measured nitrogen concentration profiles to Monte Carlo simulations. The high-temperature nitrogen PSII treatment can increase the thickness of the implantation-affected zone from $2000\ \text{\AA}$ to approximately $5000\ \text{\AA}$ on Ti-6Al-4V ELI. After high-temperature nitrogen PSII treatment, the wear behavior of Ti-6Al-4V ELI is as good as the cobalt-chromium alloy.

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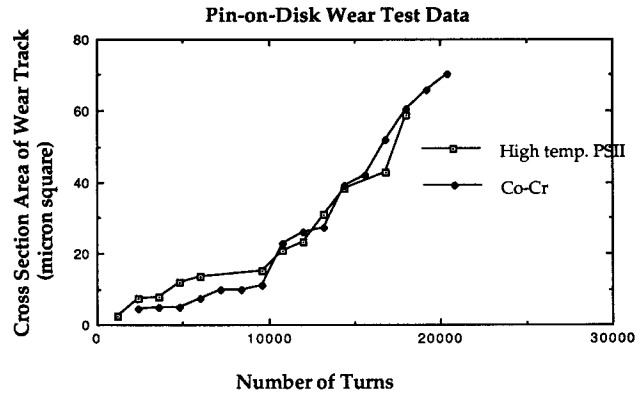


Fig. 6 Comparison of wear mass loss between Ti-6Al-4V ELI and cobalt-chromium alloy.

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