IMPROVED TRIBOLOGICAL BEHAVIOR OF
BORON IMPLANTED Ti-6Al-4V

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ABSTRACT

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Previous research has compared the mechanical properties of Ti6Al4V implanted with nitrogen using the plasma source ion immersion process and Ti6Al4V implanted with boron using the beamline process [1]. Although the nitrogen implanted Ti6Al4V had superior wear resistance it was concluded that the wear resistance of boron implanted Ti6Al4V might be improved to comparable levels if boron were implanted at lower energies to increase the concentration of boron at the surface. Boron implantation of Ti6Al4V has been conducted at combinations of 32 and 40 keV to supplement that done previously at 75 keV. Shallower boron depth profiles with higher B-concentrations in the Ti64 surface have been obtained by tailoring the combinations of ion energy and dose. This work used three different ion energy and dose combinations of 4×10^{17} B-at/cm² at 40 keV plus 2×10^{17} B-at/cm² at 32 keV, 4×10^{17} B-at/cm² at 40 keV, and 4×10^{17} B-at/cm² at 32 keV plus 2×10^{17} B-at/cm² at 40 keV. Comparisons are made between Ti6Al4V with a shallow implanted boron depth profile, Ti6Al4V with a deeper boron depth profile and nitrogen implanted using a plasma source ion implantation process. It has been previously shown that while boron implanted Ti64 has a ~30% higher surface hardness than nitrogen implanted Ti64, the N-implantation reduced the wear coefficient of Ti64 by 25-120x, while B-implantation reduced the wear coefficient by 6.5x or less. The results show that no significant improvement is made in the wear resistance of boron implanted Ti6Al4V by increasing the concentration of boron at the surface from approximately 10% to 43%. Transmission electron microscopy (TEM) and selected area diffraction (SAD) indicated the formation of crystalline TiB in the implanted surface layer. Shallower depth profiles result in reductions of the Ti6Al4V wear coefficient by 6.5x or less which is the same result obtained earlier with the deeper boron depth profile. Surface hardness of Ti6Al4V with shallower boron depth profiles was improved approximately 10% compared to the results previously acquired with deeper boron depth profiles.

INTRODUCTION

This work adds the effects of a more shallow boron depth profile in Ti6Al4V on the pin on disk wear performance and surface hardness to the knowledge of the positive effects that boron implantation can produce for Ti6Al4V [2-4].

EXPERIMENT

Four flat samples of the Ti6Al4V alloy, 4 cm in diameter and 5 mm thick, were mechanically polished to a mirror finish. A Varian NF3000 implanter was used to implant various doses of B⁺ at different combinations of 32 and 40 keV into Ti6Al4V mounted on a stage cooled to approximately -100°C. Ion beam analysis (IBA) utilizing the reaction $^{11}\text{B}(\alpha, \alpha)^{11}\text{B}$ at 6.68 MeV was used to measure the retained boron ion dose. For the retained dose measurements, the analysis beam was of normal incidence, the scattering angle was 167° and the collected charge was 6 μC. A cross-sectional TEM sample was made by mechanical thinning and ion milling. The sample was viewed using a 300 keV electron beam in a CM30 microscope. The properties of the unimplanted and implanted samples were measured using nanoindentation and pin-on-disk wear testing techniques. Nanoindentation tests were performed using a Nanoindenter II® with the continuous

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stiffness option. Pin-on-disk wear tests were conducted in air at 50% relative humidity using a 6 mm diameter ruby ball as the counterface, 11 gm load, a 3 mm track diameter, 50 rpm speed and a test time of 10 minutes. These conditions resulted in a Hertzian contact stress of 333 MPa for the unimplanted Ti6Al4V and a sliding speed of 0.8 cm/s. Coefficient of friction values were electronically monitored and recorded throughout the tests. After wear testing, the cross sectional area of the wear track was measured by surface profilometry at four locations equally spaced around the track. The volume, V in mm^3 , of material removed from the surface was calculated and used to determine the wear coefficient [5] as defined by $V/(L \cdot D)$, where L is the load in Newtons and D is the sliding distance in meters. Each sample was wear tested four times.

RESULTS AND DISCUSSION

The IBA results are shown in the Table. The retained dose measurements show that Ti64 retains most of the implanted B. The difference between the retained doses for the dual-energy implants indicates that having a B concentration at the metal surface reduces the sputter yield and increases the amount of retained B. The peak and surface boron concentrations are measured from the IBA spectra shown in Fig. 1.

Table. Retained dose and boron concentration measurements for each ion implantation condition as measured by ion beam analysis. The error in the measurements is $\pm 15\%$.

Beamline Incident Boron Dose and Energy	Retained Dose (10^{17} B-at/ cm^2)	Peak Boron Concentration (at% B)	Surface Boron Concentration (at% B)
4×10^{17} at/ cm^2 @ 40keV	3.9	44.4	25.9
4×10^{17} at/ cm^2 @ 40keV plus 2×10^{17} at/ cm^2 @ 32keV	5.0	52.4	37.5
4×10^{17} at/ cm^2 @ 32keV plus 2×10^{17} at/ cm^2 @ 40keV	6.0	56.5	42.8

The boron distributions for the unimplanted and implanted Ti6Al4V samples are shown in Fig. 1. The spectra show the boron distribution to be approximately Gaussian as expected from monoenergetic ions used in beamline processes. Fig. 1 provides evidence that the boron distribution extends to the surface of the Ti6Al4V through the use of

Fig. 1. IBA spectra of boron implanted Ti6Al4V. The retained dose for the implanted samples are indicated in units of 10^{17} B-at/ cm^2 .

combinations of lower ion energies. The profile with the 6×10^{17} at/cm² retained dose is not positioned on the Ti edge because of carbon contamination of the sample that occurred during implantation. TRIM predicts the range of boron ions implanted at 32 keV to be 75 ± 40 nm and the projected range for boron ions implanted at 40 keV to be 94 ± 47 nm and this is in close agreement with the depths corresponding to the spectra in Fig. 1.

The hardness and Young's modulus results, as measured by nanoindentation are shown in Figs. 2-3. Fig. 2 indicates that the implanted samples have similar hardness profiles. These results show a 10% improvement in hardness over the results from previous work involving implantation of Ti6Al4V implanted with boron ions at higher energy with a deeper depth profile. There was no significant change in elastic modulus (Fig. 3)

Fig. 2. Hardness profiles for the unimplanted and ion implanted Ti6Al4V samples. The extent of the error bars is indicated by 2σ . The retained dose for the implanted samples are indicated in units of 10^{17} B-at/cm².

Fig. 3. Modulus profiles for the unimplanted and ion implanted Ti6Al4V samples. The difference between samples is $\leq 2\sigma$. The retained dose for the implanted samples are indicated in units of 10^{17} B-at/cm².

Pin-on-disk wear testing reveals a maximum reduction of 6.5x in the wear coefficient (Fig. 4) which is equivalent to that achieved during previous work where Ti6Al4V was implanted with higher energy boron ions with a resultant deeper boron depth profile. It was the main objective of this research to determine if a more shallow boron depth profile would result in any significant improvement in the wear coefficient and comparison of this recent data with previous results indicates that there is no significant tribological advantage to lower energy ion implantation.

Fig. 4. Average wear coefficients and coefficient of friction for unimplanted and ion implanted Ti6Al4V. The retained ion doses in units of 10^{17} at/cm^2 are shown along the bottom x-axis. The reduction in the wear coefficient for each implantation condition is shown above each column.

Scanning electron micrographs (SEM) of the wear tracks on the unimplanted and implanted samples are shown in Fig 5. Note the predominance of an abrasive wear mechanism in both cases, but especially on the unimplanted sample. The implanted samples exhibited far less wear debris and the wear scar is barely visible as a few parallel scratches in the surface.

The SAD (Fig. 6) of the unimplanted region shows the expected α -Ti hexagonal structure (JCPDS 5-682). The implanted region shows three rings from the orthorhombic TiB structure (JCPDS 5-700) and α -Ti. The micrograph does not indicate a clear distinction in morphology between the B-implanted and unimplanted region. The thin amorphous layer that appears at the sample edge is believed to be due to damage during ion milling. XTEM clearly shows the formation of TiB phase.

CONCLUSIONS

It has been shown that using lower energy for the boron implantation process can result in a higher retained boron dose closer to the surface of Ti6Al4V. The shallower boron depth profile in the surface of Ti6Al4V does not significantly improve the wear resistance over that with a deeper boron depth profile. Surface hardness of the Ti6Al4V

with the shallow boron depth profile is approximately 10% better than that with the deeper boron depth profile. Surface hardness of boron implanted Ti6Al4V processed using beamline implantation is approximately 25-33% higher than nitrogen implanted Ti6Al4V processed using plasma source ion implantation. The nitrogen implanted Ti6Al4V has significantly greater wear resistance than boron implanted Ti6Al4V by more than an order of magnitude [1].

Fig. 5. SEM micrographs of the wear tracks on the unimplanted (left) Ti6Al4V sample and the implanted sample (right) with the greatest improvement in wear resistance (3.9 B-at/cm² retained dose) indicating a predominantly abrasive wear mechanism. Both micrographs have the same magnification.

Fig. 6. XTEM micrograph and SAD of the B-implanted and unimplanted regions.

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REFERENCES

1. K.C. Walter, J.M. Williams, J.S. Woodring, M. Nastasi, D.B. Poker, and C.P. Munson, *Mat. Res. Soc. Symp. Proc.* 438(1997)627-632.
2. K.S. Zhou and H. Herman, *Surf. Technol.* 18, 51 (1983).
3. R.N. Bolster, I.L. Singer and R.G. Vardiman, *Surf. Coat. Technol.* 33, 469 (1987).
4. D.W. Zhang, X.P. Zhang, W.C. Yu. and Z.G. Wang, *Surf. Coat. Technol.* 58, 119 (1993).
5. K. Holmberg, A. Matthews, Coatings Tribology: Properties, Techniques and Applications in Surface Engineering, Elsevier, 1994, p. 53.

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