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PROPERTIES OF ION IMPLANTED Ti-6Al-4V PROCESSED USING BEAMLINE AND PSII TECHNIQUES

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ABSTRACT

The surface of Ti-6Al-4V (Ti64) alloy has been modified using beamline implantation of boron. In separate experiments, Ti64 has been implanted with nitrogen using a plasma source ion implantation (PSII) technique utilizing either ammonia (NH_3), nitrogen (N_2), or their combinations as the source of nitrogen ions. Beamline experiments have shown the hardness of the N-implanted surface saturates at a dose level of ~$4x10^{17}$ at/cm^2 at ~10 GPa. The present work makes comparisons of hardness and tribological tests of (1) B implantation using beamline techniques, and (2) N implanted samples using ammonia and/or nitrogen gas in a PSII process. The results show that PSII using N_2 or NH_3 gives similar hardness as N implantation using a beamline process. The presence of H in the Ti alloy surface does not affect the hardness of the implanted surface. Boron implantation increased the surface hardness by as much as 2.5x at the highest dose level. Wear testing by a pin-on-disk method indicated that nitrogen implantation reduced the wear rate by as much as 120x, and boron implantation reduced the wear rate by 6.5x. Increased wear resistance was accompanied by a decreased coefficient of friction.

INTRODUCTION

The improvements in surface hardness and wear resistance of N-implanted Ti-6Al-4V (Ti64) have been extensively researched using both beamline [1-3] and plasma source ion implantation (PSII) [4-9] techniques. Boron implantation has also been shown to increase the cavitation erosion resistance [10], the abrasive wear resistance [11], and the fatigue endurance limit [12] of Ti64. The improvements have generally been attributed to the formation of hard compounds such as TiN and TiB_2 with Vickers hardness numbers of 2100 and 3000 kg/mm^2, respectively [13]. This work adds the effects of using NH_3 as the nitrogen gas for PSII processing, and the effects of B implantation on the pin-on-disk wear performance to the knowledge of the positive effects that ion implantation can produce for Ti64.

EXPERIMENTAL DETAILS

Flat samples of the alloy, 4 cm in diameter and 5 mm thick, were mechanically polished to a mirror finish. Ion beam analysis (IBA) indicated the bulk material is 86 at% Ti, 10 at% Al, 3.4 at% V, and 0.7 at% H. PSII processing was done at Los Alamos's facility using 0.3 mtorr of processing gas, and 45 kV, 20 μs pulses repeated at 700 Hz. Either N_2, NH_3, or NH_3 followed by N_2 was used to implant N into Ti64 samples mounted on a cooled stage. A Varian NF3000 implanter was used to implant 75 keV B^+ into Ti64 samples mounted on a cooled stage. In both cases, the sample temperature during implantation was less than 50°C. Ion beam analyses, utilizing the reactions $^{14}N(\alpha,\alpha)^{14}N$ at 8.86 MeV and $^{11}B(\alpha,\alpha)^{11}B$ at 6.63 MeV, were used to measure the retained N and B ion doses, respectively. For the retained dose measurements, the analysis beam was of normal incidence, the scattering angle was 167°, and the collected charge was 20 μC. A 2.0 to 2.3 MeV He^+ beam, and a backscattering angle of 160°, was used to determine the ion distribution in the sample surface. Hydrogen contents of the unimplanted and implanted samples was accomplished using a 2 MeV He^+ beam and a forward scattering angle of 30°. 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® and the measurements were corrected for elastic recovery. Pin-on-disk wear tests were conducted in air at 50% RH 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 Ti64 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 as defined by V/(L•D), where L is the load in Newtons and D is the sliding distance in meters. Each sample was wear tested between three and six times.

RESULTS AND DISCUSSION

A summary of the ion implantation conditions and the retained dose measurements are included in Table 1. Note that H is present in the sample implanted by the PSII process using N_2 gas, even though there is no H included in the molecule. It is believed that desorption of water from the chamber wall and subsequent ion implantation resulted in the observed H concentration and the slight, but measurable, amounts of implanted oxygen. PSII-treated samples contained

Table 1. Retained dose measurements for each ion implantation condition as measured by ion beam analysis. The error in the measurements is ±15%.

PSII Gas & Energy	Retained Dose (10^{17} N-at/cm^2)	Retained Dose (10^{17} H-at/cm^2)	Beamline Ion & Energy	Retained Dose (10^{17} B-at/cm^2)
45 kV N_2	2.2	0.6	75 keV B^+	2.0
45 kV NH_3	3.1	0.5	75 keV B^+	4.6
45 kV NH_3+N_2	3.2	0.7	75 keV B^+	7.7

between 0.6 and 0.9x10^{17} O-at/cm^2 in their surfaces. Within error, all PSII treated samples have the same H content near the surface. The distribution of N and H in the PSII treated samples can be seen in Figs. 1-2. Note that the N distribution is inferred only from the reduction in the Ti in the spectra (Fig. 1). Examination of the N distribution profiles indicates the N_2 implanted sample has the shallowest implantation depth and supports the speculation that a significant fraction of the implanted ions are N_2^+. The NH_3 implanted samples have a slightly deeper implantation depth

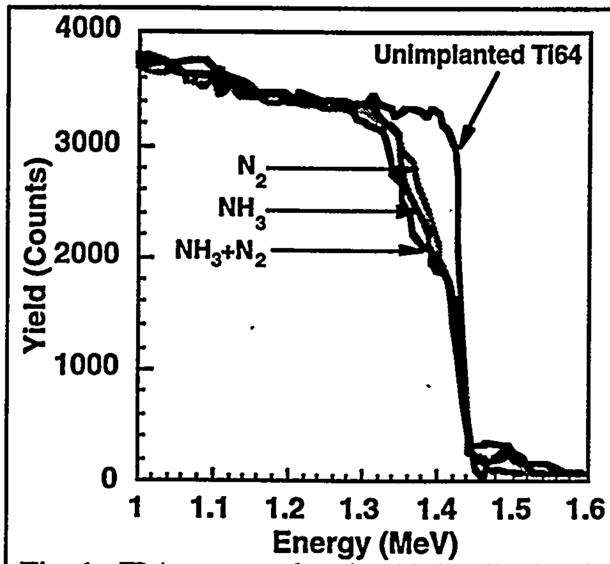

Fig. 1. IBA spectra showing N distribution in PSII treated Ti64.

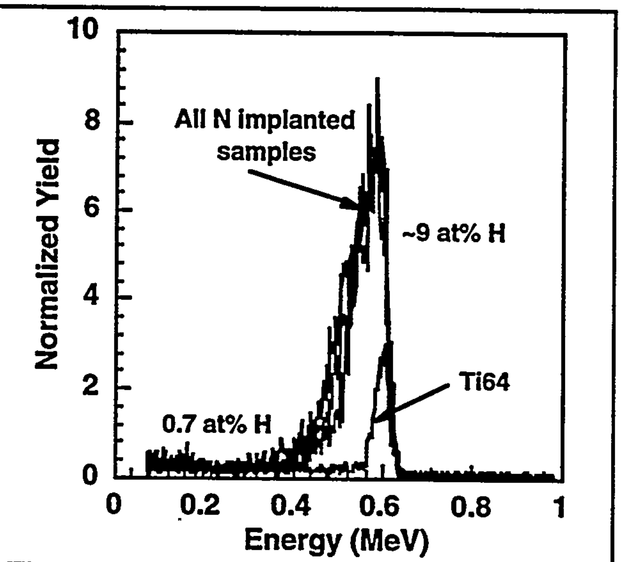

Fig. 2. IBA spectra showing H distribution in PSII treated Ti64.

and a correspondingly greater retained dose. The range of a 45 keV N^+ ion in Ti64 is estimated by TRIM to be 75±39 nm and is in reasonable agreement with the depth corresponding to the IBA spectra in Fig 1. The typical Gaussian profile is not observed for PSII treated samples because of the distribution of ion energies inherent to the process. Quantitative analysis indicates a peak N concentration of 35-40 at% for each PSII treated sample. The H distribution extends to the same depth as the nitrogen distribution, but the H concentration peaks at ~9at% at the sample surface.

The B distributions for the beamline implanted samples are shown in Fig. 3. The spectra show the boron distribution to be approximately Gaussian as expected from monoenergetic ions used in beamline processes. TRIM predicts the range of a 75 keV B^+ ion to be 165±67 nm in Ti64 and this is in close agreement with the depth corresponding to the spectra in Fig. 3. The peak B concentration for the highest retained dose is ~55 at%. Note that the B distribution does not begin to extend to the Ti64 surface except for the sample with the highest retained dose.

The hardness and elastic modulus results, as measured by nanoindentation are shown in Figs. 4-5. Fig. 4 indicates that all the PSII treated samples have essentially the same hardness profiles and a hardness of 9 GPa at a depth of 50 nm. This hardness value is in good agreement with that reported by Williams et al. [1]. The hardness of the Ti64 surface is increased by 1.5x for B implantation at the lowest dose, with an approximately 2.5 fold increase in hardness (at 50 nm) is produced by B implantation to a retained dose of 7.7×10^{17} B-at/cm^2. The elastic modulus (Fig. 5) is not significantly changed by either N or B ion implantation and differs from unimplanted Ti64 by ≤ 25%.

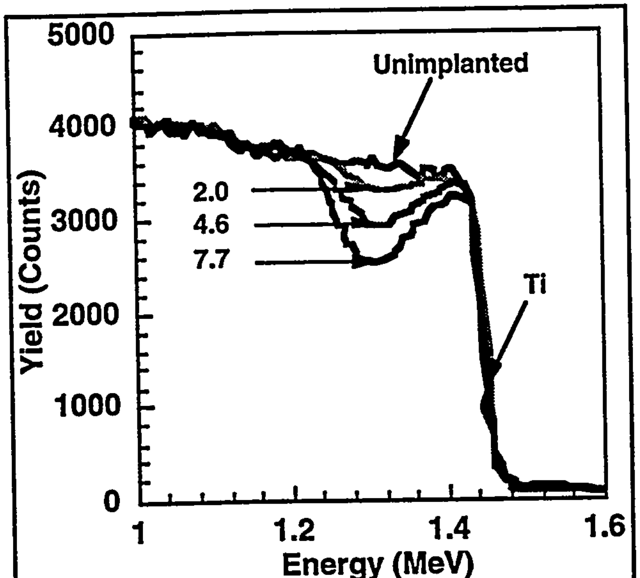

Fig. 3. IBA spectra of B implanted Ti64. The retained dose is indicated in units of 10^{17} B-at/cm^2.

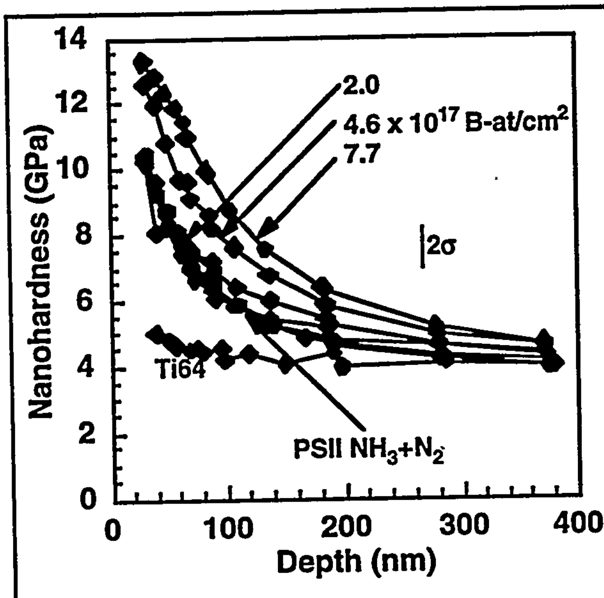

Fig. 4. Hardness profiles for all ion implanted samples. The extent of the error bars is indicated by 2σ.

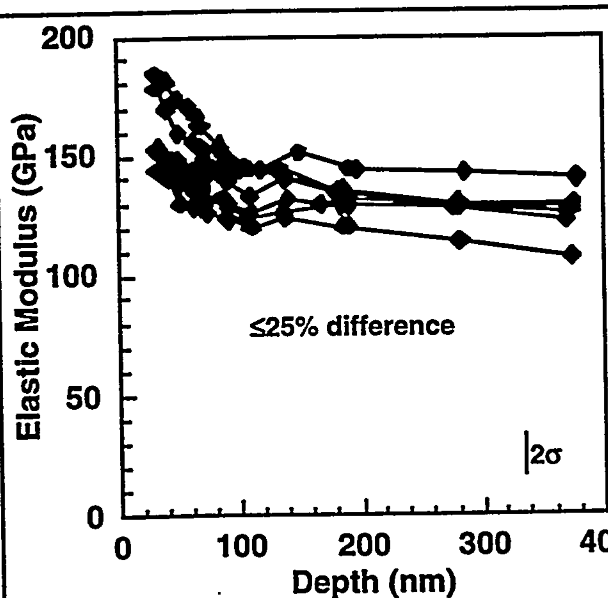

Fig. 5. Modulus profiles for all ion implanted samples. The extent of the error bars is indicated by 2σ.

Pin-on-disk wear testing indicated a wide range of wear coefficients, as shown in Fig. 6. The PSII sample treated with NH_3+N_2 performed the best under these test conditions and provided a 120x reduction in the wear coefficient of Ti64. The Ti64 sample retaining the highest B dose exhibited a reduction of 6.5x in the wear coefficient, but the improvement is an order of magnitude less than that for the nitrogen implanted samples. The coefficient of friction values for each sample are also shown in Fig. 6. Note that any reduction in the wear coefficient corresponds to a reduction in the coefficient of friction. There are two values for μ shown for the B implanted sample with the retained dose of 7.7×10^{17} at/cm^2. The lower value of 0.13 corresponded to early times in the wear test and the higher value of 0.27 occurred after 250 cycles. It should be noted that for the B implanted samples, the lowest coefficient of friction and the highest reduction in wear coefficient corresponds to the highest B concentration at the Ti64 surface (Fig. 3).

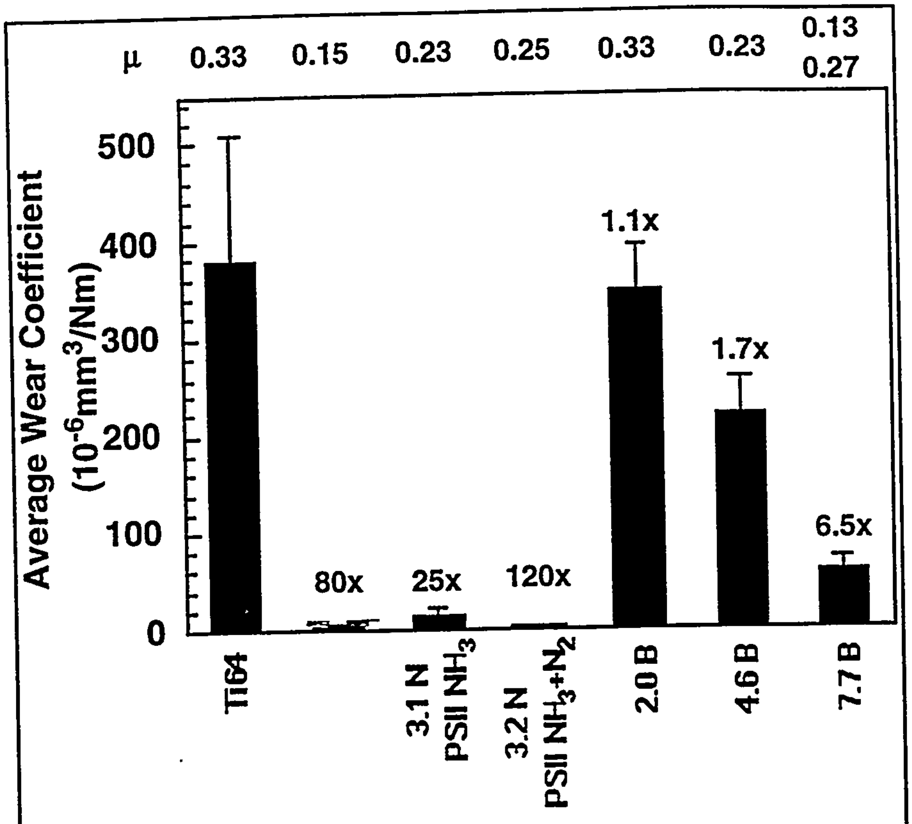

Fig. 6. Average wear coefficients and coefficient of friction for unimplanted and ion implanted Ti64. The retained ion doses in units of 10^{17} at/cm^2 are shown along the bottom x-axis. The reduction in the wear coefficient for each implantation condition is shown above each column.

Optical microscopy of all the wear tracks indicated a predominantly abrasive wear mechanism for all the samples (Fig. 7). It is clear from Fig. 7c that the PSII treated surface never

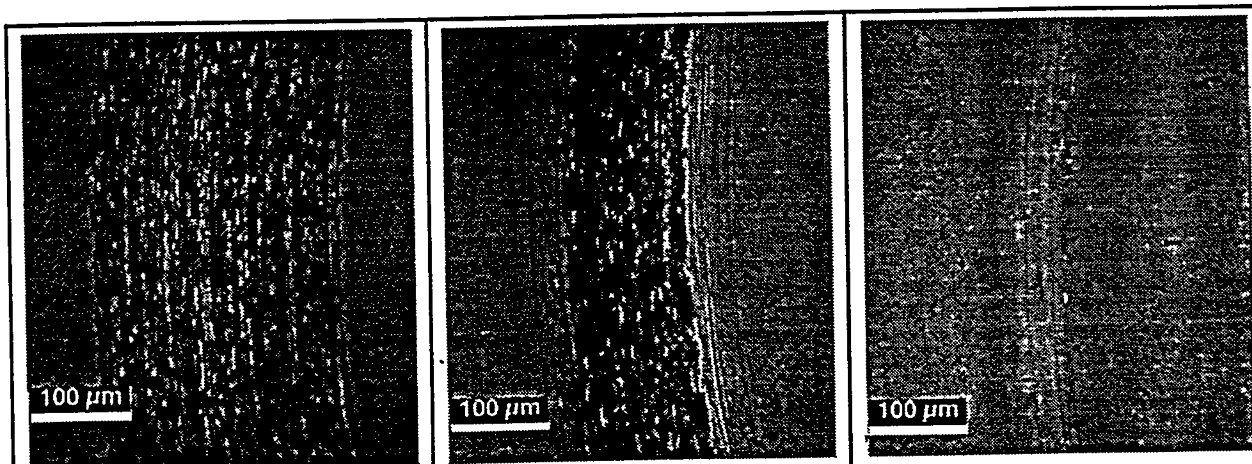

Fig. 7a. Optical micrograph of the wear track on unimplanted Ti64.

Fig. 7b. Optical micrograph of the wear track on 7.7×10^{17} B-at/cm^2 implanted Ti64.

Fig. 7c. Optical micrograph of the wear track on Ti64 treated by PSII with NH_3+N_2.

wore through the implanted zone. This is probably due to the suppression of wear debris formation and severe abrasive wear present for the unimplanted sample (Fig. 7a). The B implanted sample with the highest dose exhibits intermediate behavior between the unimplanted and PSII treated surfaces. The high energy of the B implantation process produced a buried layer (Fig. 3) and did not result in a significant B concentration at the Ti64 surface. The absence of a hard titanium-boride surface allowed the generation of wear debris and the onset of severe abrasive wear as shown in Fig. 7b.

CONCLUSIONS

It has been shown that a PSII process, using either N_2, NH_3 or their combination, produces a surface with equivalent hardness as for N implanted Ti64 using a beamline approach. In addition, the presence of H in the implanted layer does not appear to affect the surface hardness. Thus, PSII processes using NH_3 could take advantage of the reduced ion mass present the plasma and achieve a greater implantation depth relative to PSII processes using N_2. Implantation of B, with retained doses greater than 2×10^{17} at/cm^2, gives a harder Ti64 surface but does not produce equivalent reductions in the wear coefficient. Comparing the N and B distributions in Figs. 1 & 3, it is speculated that the implanted B is implanted too deeply and insufficient B is present at the Ti64 surface to retard the initiation of severe abrasive wear. Additional research using a lower energy B implantation process, which would increase the B concentration at the Ti64 surface, should be conducted to determine whether B implantation can provide superior wear resistance over N implantation.

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