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Use of nitrogen ion implantation for modification of the tribological properties of titanium alloy Ti6Al4V

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Abstract. Titanium and its alloys are commonly used in space, aviation, and motorisation industries due to their high strength and corrosion resistance. However, given its poor tribological properties (high friction and wear coefficients), methods for improvement of the tribological properties of titanium are continuously being developed. This study presents the effect of nitrogen ion implantation with energy 60 keV at a fluence of $1 \cdot 10^{17}$ N^+ /cm^2 on changes in the properties of titanium alloy Ti Grade 5 (Ti-6Al-4V). Ion implantation was carried out through a mask with φ = 500 µm diameter holes. Nitrogen implantation through the mask improve the tribological properties of Ti6Al4V, by reducing the mean value of the friction coefficient and reducing tribological wear.

1. Introduction

Titanium and its alloys are common materials used in construction of ships, aircraft, nuclear reactors, space devices, and medical prostheses due to their higher strength, corrosion resistance, and biocompatibility than that of steel and cobalt alloys [1-4]. However, titanium is not used in friction pairs due to the high friction coefficient, high wear, and tendency towards seizure. To extend the scope of the industrial applications of titanium, various techniques for modification of the properties of the surface of titanium components are employed. These include boron coating [5, 6], nitriding [7, 8], thermal oxidation [9, 10], carbonisation [11, 12], laser surface processing [13, 14], and high-temperature low-energy plasma ion implantation [15]. High-temperature surface processing techniques cannot be used for high-precision elements; additionally, the coated layer should not change their sizes. These shortcomings can be avoided with the use of beam ion implantation at room temperature, which does not cause thermal deformation of the implanted elements.

Therefore, we decided to check the impact of nitrogen ion implantation on the tribological properties of titanium. We focused on changes induced by implantation of the sample through a mask with φ = 500 μm diameter holes distributed 500 μm apart. The available literature provides only one general publication suggesting the usefulness of implantation through a mask with φ > 50 μm diameter holes [16]. During implantation, ionic doping is accompanied by disc spraying. The spraying leads to formation of pits and protuberances (surface texturing). Surface texturing facilitates integration of a medical implant with the human or animal body. Besides cobalt alloys, titanium and its alloys are most commonly used in the manufacture of medical implants. We intended to determine the possibility of employing the technique of ion implantation through the mask in the final stage of manufacture of internal combustion engine elements, machine elements and medical implants.

2. Material and methods

A Ti Grade 5 titanium alloy denoted as Ti6Al4V was used in the investigations. Besides titanium, it contains Fe – 0.4%, Al – 5.5-6.75%, V – 3.5-4.5%, N – 0.05%, C – 0.08%, and O – 0.2%. Cylindrical samples with a φ = 25 mm diameter and h=3 mm height were prepared from the material. Prior to the ion implantation, the samples were polished to achieve a roughness value of $R_a \leq 0.1$ μm.

The steel mask used for covering the implanted sample was copper plated. This was applied in order to check whether atoms released by ionic mask spraying would be additionally introduced into the sample during the implantation process.

The samples were implanted with nitrogen ions with energy 60 keV. The fluence of the implanted ions was $1 \cdot 10^{17}$ N^+ /cm². The implantation was carried out in the Institute of Physics, UMCS. During the implantation, the ion beam intensity was reduced to 0.5 mA/cm² to ensure a temperature of the sample below 50°C. Figure 1 shows a microphotograph of the sample. The image was taken with a TESCAN Vega 3LMU scanning electron microscope. The dark circles are areas implanted with a full fluence of nitrogen ions, whereas the dark grey rings around them demonstrate nitrogen ions scattered from the mask.

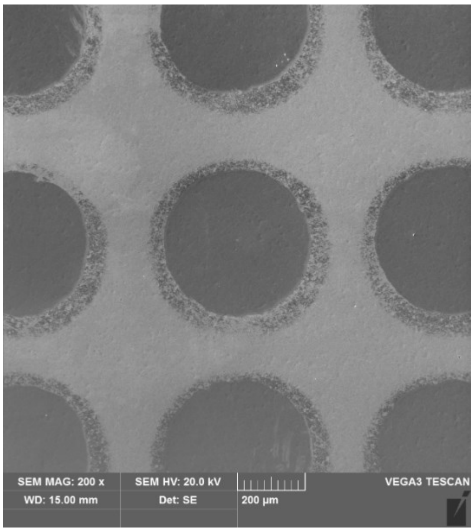

Figure 1. Microphotograph of a Ti Grade 5 sample after nitrogen ion implantation through the mask

Friction and wear coefficients were measured in a tribological test in technically dry friction conditions on a self-made pin/ball-on-disc research stand [17] and on an Anton Paar Nano-Tribometer NTR² (CFM Instrument). A steel ball with a diameter of $\varphi = 2$ mm and pressure force of 500 mN was a counter sample. The wear was measured in a cross-sectional area of the track worn by the ball.

Measurements of the wear track were conducted using a Taylor Hobson Form Talysurf Intra profilometer.

3. Results

The results of the measurements of the friction coefficient of the unimplanted sample and that implanted through the mask are presented in figure 2. During the test, the wear products were aspirated and removed from the friction area by a nozzle under reduced pressure.

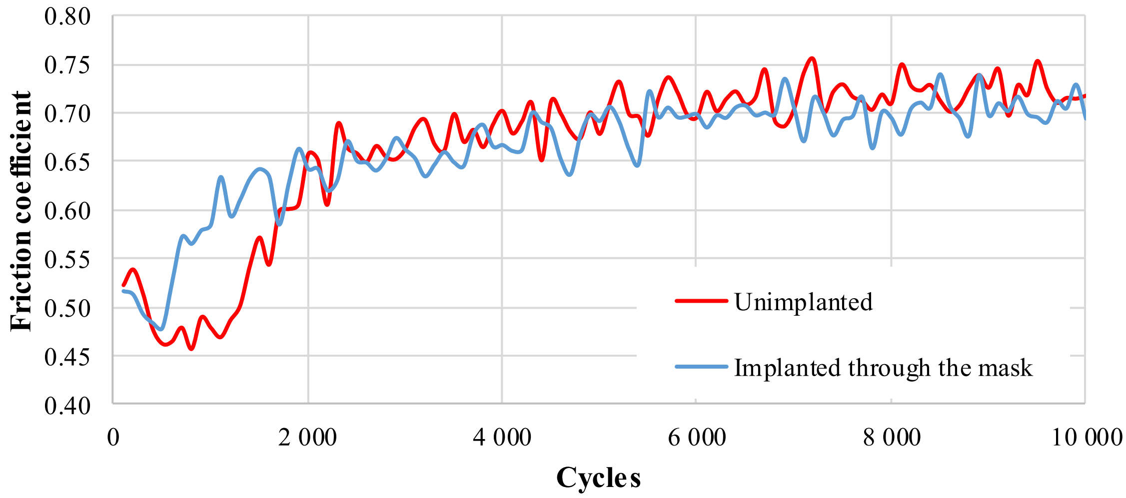

Figure 2. Measurement of the friction coefficient of the unimplanted Ti Grade 5 sample and the sample implanted through the mask with nitrogen ions with energy 60 keV and at a fluence of $1 \cdot 10^{17}$ N^+ /cm². The wear products were removed from the friction area during the test.

The tribological test was applied to the Ti Grade 5 sample implanted with nitrogen ions through a mask with φ = 500 µm diameter holes. Initially, the friction coefficient for the unimplanted sample was approx. 0.55, but later declined to a value of ca. 0.47 during the first 200 rotations. In the successive part of the test, its value gradually increased and reached a value of 0.72. The initial reduction of the value of the friction coefficient was caused by the polishing of the sample surface by the ball. The friction coefficient in the initial stage of the implanted sample testing had the lowest value and then increased to ca. 0.65÷0.75. It was slightly lower than that for the unimplanted sample.

However, in the case of the sample implanted through the mask, the friction coefficient reached a value of approx. 0.6 already after 1000 rotations (in the case of wear product aspiration), whereas this value was reached by the unimplanted sample after ca. 1800 cycles.

The presence of wear products in the friction area has a visible effect on the value of the friction coefficient, which is visible in figure 3. The presence of wear products in the friction area affects the increase of friction resistance, which increases the friction coefficient. Extraction of wear products also affects the reduction of tribological wear, which was presented in paper [18]

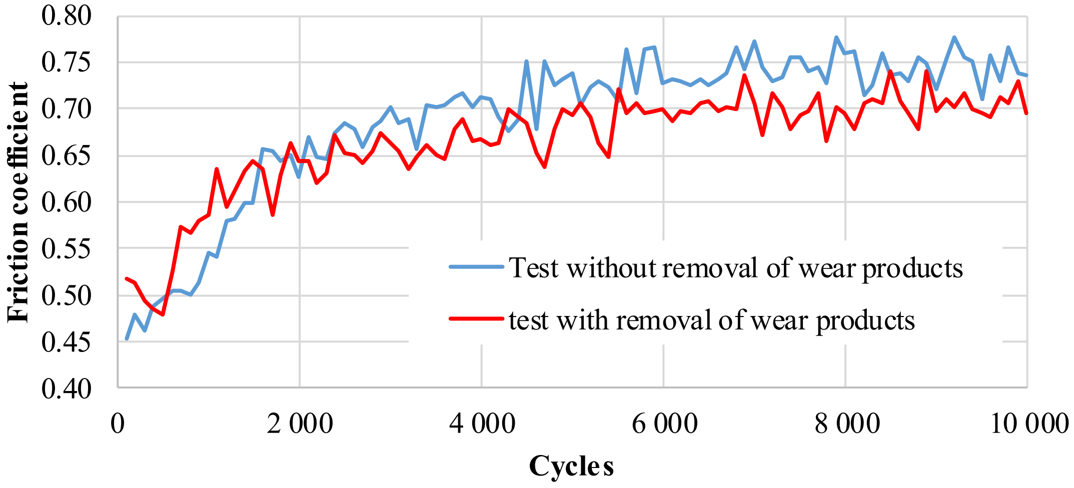

Figure 3. Friction coefficient for the Ti Grade 5 sample implanted through the mask with nitrogen ions with energy 60 keV and at a fluence of $1{\cdot}10^{17}$ N^+ /cm²

Changes in the relative wear values during the tribological test are presented in figure 4. The wear of the unimplanted sample was higher than that in the sample implanted through the mask. Similar results for sample implanted with homogeneously fluence of nitrogen ions are presented in the paper [19]. Minimum wear was shown by a sample implanted with energy 60 keV at a fluence of $5{\cdot}10^{16}$ N^+ /cm². In this test wear values of sample implanted through the mask and unimplanted sample are only comparable in the region of 1500 cycles. After 10 000 cycles, the sample implanted through the mask exhibited lower wear than the unimplanted sample. Implantation of nitrogen ions causes changes in the friction coefficient and tribological wear of the Ti Grade 5 alloy because due to doping of nitrogen ions hard titanium compounds such as TiN are formed, which increase hardness and wear resistance.

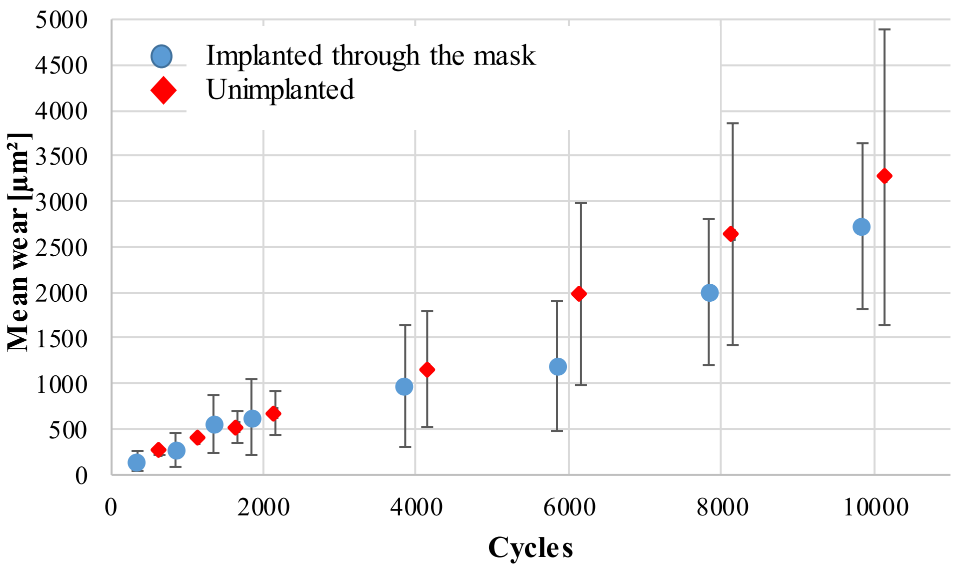

Figure 4. Mean wear of Ti Grade 5 samples with a standard deviation of the population

To elucidate the changes occurring during the friction process of the implanted and unimplanted Ti Grade 5 samples, microphotographs of the wear track were taken – figure 5a and 5b. Below, figure 5c and 5d show the chemical composition of the sample along the track. The track on the unimplanted sample (figure 5a) is irregular with longitudinal recesses and several overlapping thin layers of the sample, indicating wear off and micro-welding of the ball rubbing the sample surface with the sample material. After the implantation (figure 5b), the appearance of the track changed radically. A relatively smooth bottom of the track covered with a titanium oxide layer with minimal carbon content was visible. The increased carbon content on the surface originated from the residues of the oil vapours from the ion implanter. The carbon present on the sample surface of the unimplanted sample may have originated from the ball. The micro-X-ray analysis did not reveal the presence of copper ions on the surface of the sample implanted through the sample.

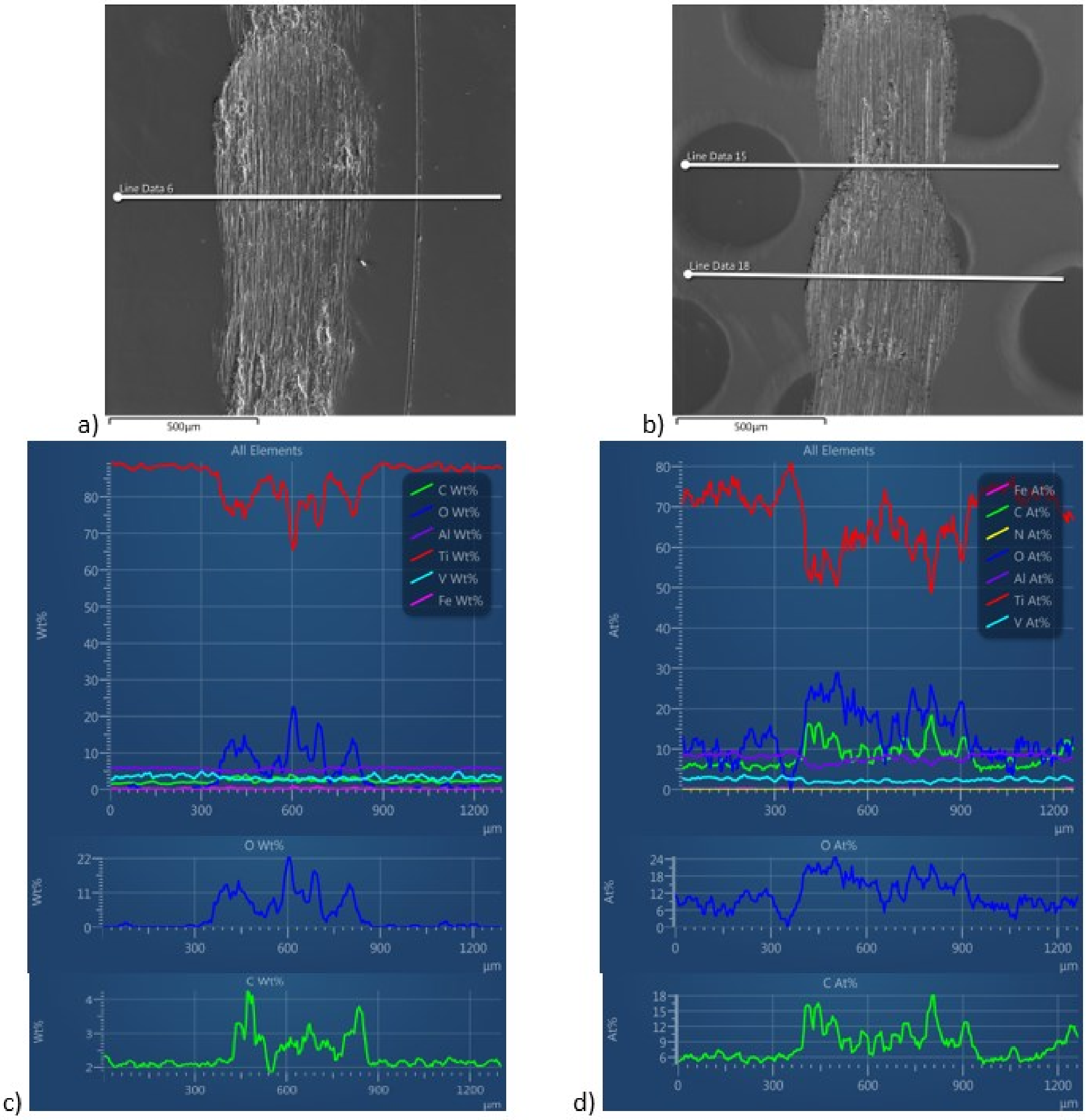

Figure 5. Microphotograph of the track worn on the Ti Grade 5 sample before (a) and after (b) implantation through the mask (line data 18) and the relative content of oxygen and carbon in the unimplanted (c) and implanted(d) sample

4. Summary

Implantation of nitrogen ions in titanium Ti Grade 5 alloy samples (Ti-6Al-4V) induces changes in the tribological properties, i.e. it significantly reduces sample wear and lowers the friction coefficient to a lesser extent. Removal of wear products from the sample surface during the tribological tests ensures similar operating conditions in the friction pair in the individual measurements. Elimination of the random distribution of fragments of worn material exerts an effect on the friction coefficient.

A change in the course of the friction coefficient depending on the type of the implantation process is evident. The use of the mask causing non-homogenous distribution of ions in the analysed sample results in a more rapid increase in the friction coefficient, compared with that of the unimplanted sample. However, the mean value of the friction coefficient calculated for the entire tribological test reaches a lower value than for the unimplanted sample. The mean value of wear the implanted sample is maintained at a similar level to unimplanted to about 2000 revolutions. However, in the further part of the test, the mean value of tribological wear is lower for the sample implanted by the mask.

The results of the micro-X-ray analysis suggest that the improvement of the tribological properties is caused by the presence of carbon atoms; therefore, further investigations will involve carbon ion implantation. The samples have already been prepared. Carbon ions have been implanted with energy 120 keV at fluences of $5 \cdot 10^{16}$ ions/cm² and $1 \cdot 10^{17}$ N^+ /cm².

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