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THE TRIBOLOGICAL PROPERTIES OF THE Ti6AI4V ALLOY WITH NITROGEN ION IMPLANTATION

OCENA WŁAŚCIWOŚCI TRIBOLOGICZNYCH STOPU TYTANU Ti6AI4V **IMPLANTOWANEGO JONAMI AZOTU**

Key words:

titanium alloys, ion implantation, friction, wear, SEM/EDS, contact angle.

Abstract

The article presents the analysis of the influence of ion implantation on the properties of titanium alloy used in biotribological systems. The object of the study was the titanium alloy Ti6Al4V implanted with nitrogen ions. Tribological model tests were carried out in combination with a sphere with Al2O3 - a Ti6Al4V alloy disc implanted with N⁺ ions. Experimental friction tests were carried out on pin-on-disc testers in conditions of technically dry conditions and in conditions of lubrication with the Ringer's solution. The tests on the TRB tester were carried out in a swinging motion, while on the T-01 tester in a sliding movement. Friction coefficient and wear were determined for all tests. Surface morphology testing and chemical composition analyses were performed using the Jeol JSM-7100F scanning electron microscope, equipped with an EDS microanalyzer. Surface geometry measurements prior to and after tribological tests were performed on a Taylor Hobson's Talysurf CCI contactless optical profilometer. The optical tensiometer was used to determine the contact angles with demineralized water and Ringer's solution. The tribological tests of the titanium alloy Ti6Al4V lead to the conclusion that implantation of N⁺ ions results in better tribological properties of the alloy. The best tribological characteristics were obtained for a titanium alloy implanted with nitrogen ions under technically dry friction conditions. The influence of the tribological system on Ringer's fluid influenced the reduction of coefficients of friction in the oscillating movement (Tribometer TRB) and sliding motion (Tester T-01M). In the case of a oscillating movement, higher wear of the tested friction pair was observed under friction conditions with the Ringer solution lubrication.

Słowa kluczowe: stopy tytanu, implantacja jonowa, tarcie, zużycie, SEM/EDS, kąt zwilżania.

Streszczenie

W artykule przedstawiono analizę wpływu implantacji jonowej na właściwości stopu tytanu stosowanego

w systemach biotribologicznych. Przedmiotem badań był stop tytanu Ti6Al4V implantowany jonami azotu N⁺. Tribologiczne badania modelowe przeprowadzono w skojarzeniu kula z Al₂O₂-tarcza ze stopu Ti6Al4V implantowana jonami azotu. Eksperymentalne testy tarciowe zrealizowano na testerach typu pin-on-disc w warunkach tarcia technicznie suchego oraz tarcia ze smarowaniem roztworem Ringera. Badania na testerze TRB przeprowadzono w ruchu wahadłowym, natomiast na testerze T-01 w ruchu ślizgowym. Dla wszystkich testów określono współczynnik tarcia oraz zużycie. Przy użyciu skaningowej mikroskopii elektronowej SEM wraz z analizą składu chemicznego EDS oraz profilometrii interferometrycznej obserwowano morfologię oraz topografię powierzchni badanych próbek przed i po testach tribologicznych. Tensjometr optyczny posłużył do wyznaczenia kątów zwilżania wodą demineralizowaną oraz roztworem Ringera. Uzyskane wyniki badań pozwoliły na stwierdzenie, że zastosowanie implantacji jonowej istotnie poprawia właściwości tribologiczne stopu tytanu zarówno w warunkach tarcia technicznie suchego, jak i w warunkach smarowania roztworem Ringera. Najlepsze charakterystyki tribologiczne uzyskano dla stopu tytanu implantowanego jonami azotu w warunkach tarcia technicznie suchego. Oddziaływanie systemu tribologicznego z płynem Ringera wpłynęło na zmniejszenie współczynników tarcia w ruchu wahadłowym (Tribometr TRB) i ślizgowym (Tester T-01M). W przypadku ruchu wahadłowego większe zużycie badanych par trących zaobserwowano w warunkach tarcia ze smarowaniem roztworem Ringera.

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INTRODUCTION

In the recent years, an increasing market demand for biomaterials has been observed. This is mostly due to the increased quality of life and standard of living and the more and more common diseases of affluence, for example, degenerative joint disease. Moreover, due to the stringent requirements that must be met by implant materials, they are among the most often tested and the most expensive materials produced by men. Every year, the number of implant procedures and operations increases worldwide. Medical implants are used in order to replace damaged or ill body parts and must meet strictly defined requirements concerning their medical composition, cleanliness, corrosion resistance, endurance, and biocompatibility with the human body [L. 1]. Due to the requirements that must be met by biomaterials, the number of metals used in implants is very limited. As a result of their beneficial properties, titanium and its alloys are used in a wide range of medical applications, including in endoprostheses of hip and knee joints and in endoosseous dental implants. Titanium alloys are characterized good mechanical properties, very high resistance to corrosion, and biocompatibility. Compared to austenitic steels and cobalt alloys, their specific weights and Young modulus are lower. The very good biotolerance of titanium in living organisms results in the occurrence of the process of osteointegration - adhesion of bone tissue to the titanium surface of the implant. Titanium is characterized by a high affinity to oxygen and, as a result, its surface is easily covered by a passive layer of TiO₂ which prevents corrosion. A limitation on the use of titanium alloys in medical implants is its fairly low resistance to abrasion and the risk of the occurrence of metalosis [L. 2]. Researchers continue to work on new technologies that can reduce the unnecessary risk and complications. More and more often, surface engineering techniques, such as ion implantation, are used [L. 3-5].

According to W. Rosiński [L. 6, 7], ion implantation is a process that consists in the introduction of atoms of any kind to the surface layer of a solid thanks to the energy acquired by the atoms, which have been ionized in an accelerating electric field. During implantation, atoms of the base material and the implanted ions become "mixed." The energy of the implanted ions is usually in the range of several keV to several MeV, while the thickness of the implanted layers is in the range of a fraction of a micrometre to several micrometres. Due to the kinetic nature of the process, the material can be admixed with practically any element, regardless of the thermodynamic conditions and limitations.

An important advantage of the process of implantation is that there are no problems of adhesion between the modified layer and the core of the material. In the case of such a layer, adhesion is not very important because there is no clear boundary between the modified layer and the core of the material. What needs to be emphasized is that ion implantation does not result in a change of the dimensions of the workpiece. It does not involve the application of an additional layer and, consequently, it practically does not change the dimensions of the workpiece and is often used as the final treatment of the surface. Ion implantation is better than other methods of modification, because it is a non-equilibrium process, which means that it makes it possible to achieve a modified layer with a chemical composition that cannot be produced using other surface engineering techniques. Clearly the biggest advantages of this technology are the following:

- The possibility to implant any element from the periodic table in a thin surface layer, regardless of the thermodynamic conditions and limitations;
- The implantation is performed at temperatures $\leq 200^{\circ}$ C, which makes it possible to avoid changes in dimensions and shapes of the tested samples; and
- The purity of processing in vacuum conditions is guaranteed [L. 8].

Ion implantation is an effective method used to modify the performance characteristics of titanium and its alloys used in biomedical engineering **[L. 9]**.

Implantation with nitrogen, carbon, and oxygen ions are intended to improve the mechanical and tribological properties, the corrosion resistance, and the biocompatibility of titanium alloys. Moreover, the bioactivity of titanium alloys is enhanced by way of the implantation of sodium, magnesium, and silver ions. Implantation with silver and fluorine ions results in strong anti-bacterial characteristics. In conclusion, ion implantation broadens the possibilities for the use of titanium alloys in biomedical applications [L. 10].

The objective of this paper is to demonstrate that the process of ion implantation leads to the improvement of the tribological properties of the titanium alloy Ti6Al4 both in technically dry friction conditions and in conditions of friction with lubrication with Ringer's solution.

MATERIAL AND METHODOLOGY

The object of the study was the titanium alloy Ti6Al4V, whose composition is shown in **Table 1** and whose mechanical properties are shown in **Table 2**. The studied alloy is characterized by high biocompatibility, high resistance to corrosion, and high biotolerance, compared to other metallic materials.

The material that constituted a countersample in the tested friction nodes were balls made of aluminium oxide (III) $-Al_2O_3$, with diameters equal to 6 and 10 mm, burdened with a normal force equal to 10 N. The most important mechanical properties of Al_2O_2 are shown in **Table 3**.

Table 1. Chemical composition of titanium alloy Ti6Al4V

Tabela 1. Skład chemiczny stopu tytanu Ti6Al4V

Element							
Ti	Al	V	Fe	0	С	Ν	Н
total	5.50-6.75	3.5–4.5	max. 0.4	max. 0.2	max. 0.08	max. 0.05	max. 0.015

Table 2. Mechanical properties of Ti6Al4V alloy

Tabela 2. Właściwości mechaniczne stopu tytanu Ti6Al4V

Material	Young's modulus	Tensile strength	Compressive	Hardness	Density
	E [GPa]	R _m [MPa]	strength [MPa]	[Vickers]	[g/cm3]
Ti6Al4V	110–114	960–970	970	349	4.42

Table 3. Mechanical properties of aluminium(III) oxide

Tabela 3. Właściwości mechaniczne tlenku glinu (III)

Material	Young's modulus E [GPa]	Tensile strength R _m [MPa]	Compressive strength [MPa]	Hardness [Vickers]	Density [g/cm3]
Al ₂ O ₃	393	206–300	2070–2620	1365	3.987

Tribological tests were performed using disks with the diameter of 20 mm and the height of 10 mm. The key process before implantation of the samples was the preparation of their surfaces. The samples were ground and polished. Then ion implantation was performed on the face surfaces of the titanium alloy Ti6Al4V. The parameters of the surfaces prepared in this manner are shown in **Table 4**. Ion implantation was performed using the Idonus ion implantator, with an ion dose of $5x10^{17}$ N⁺/cm², with energy equal to 35 kV.

The roughness of the surface plays a significant role, because the development of the surface of titanium implants increases the possible biomechanical contact at the implant-bone boundary and affects the speed of adsorption of proteins **[L. 11]**. Continuous testing and modification of the surface of implants are intended to achieve quicker osteointegration and enable instantaneous or early burdening of the prosthesis **[L. 12]**.

The disks were tested using a Jeol JSM 7100F scanning electron microscope equipped with an EDS analyser. The tests included the observation of the morphology of the surface and the analysis of the chemical composition in micro-areas (**Fig. 3**).

The Taylor Hobson coherent Talsurf CCI Lite co-op interferometer analysed the geometric structure of the specimen surfaces before and after tribological tests. The most important parameters of the geometric structure of the surface and depth and field of abrasion

		Va		
Parameter	Specification	Ti6Al4V	Ti6Al4V implanted with N ⁺ ions	Unit
Ra	arithmetical mean height	0.058	0.099	μm
Rp	maximum profile peak height	0.100	0.255	μm
Rv	maximum depth of profile	0.204	0.271	μm
Rz	maximum height of profile	0.304	0.526	μm
Rsk	skewness of profile	-1.403	-0.402	-
Rku	kurtosis of profile	5.538	4.019	-

 Table 4. Parameters of the surface geometry

Tabela 4. Parametry struktury geometrycznej powierzchni

after tribological tests were determined during the research (Figs. 5–10).

In order to determine the effects of implantation with nitrogen ions, tribological tests were performed. Tests of the friction wear of the tested friction couples were performed in the TRB Tribometer and in the T-01M tester in technically dry conditions and in conditions of lubrication with the Ringer's solution. Both testers work on a ball-disk set. The tests were performed with the parameters specified in **Table 5**. The tribological tests were performed using the Ringer solution. The presence of the solution was intended to create conditions similar to the natural tribological conditions present in a human body. During the tests, 1.5 ml of the Ringer's solution was applied on the sample with a pipette. The chemical composition of the Ringer's solution is shown in **Table 6**.

The tribological tests in the TRB tribometer were performed in an oscillating movement, in the range of α from 0 to 30°. A general view of the tester and a schematic drawing of the operation of the friction couple are shown in **Fig. 1**. An analysis of the results of the preliminary tests indicated that the reference sample was characterized by numerous signs of wear. Therefore, in further tests, the number of cycles was limited to 2,000.

The tribological tests were also performed in a sliding motion. The T-01M tester was used to determine the friction coefficient and to determine the wear of the tested matched materials. A general view of the tester and a schematic drawing of the operation of the friction couple are shown in **Fig. 2**.

Table 5.	The parameters of tests
Tabela 5.	Parametry testu

	Unit	Tribometer TRB	T-01M	
Friction pair		Al ₂ O ₃ ball – Ti6Al4V disc or Ti6Al4V disc implanted with N ⁺ ions	Al ₂ O ₃ ball – Ti6Al4V disc or Ti6Al4V disc implanted with N ⁺ ions	
Angle	٥	30	-	
Load	N	10	10	
Sliding velocity	m/s	0.0143	0.1	
Friction path	m	5	500	
Humidity	%	40 ± 5	50 ± 5	
Temperature	°C	23±1	22±1	
Lubricating liquid	uid Ringer's solution		solution	

Table 6. Chemical composition of Ringer's solutions (1000 ml)

Tabela 6. Skład chemiczny płynu Ringera

Substance	NaCl	KCl	CaCl
Quantity	8.6 g	0.30 g	0.48 g

b)

a)



Fig. 1. View of a) TRB tribotester. b) pin on disc Rys. 1. Tribotester TRB: a) widok ogólny, b) węzła tarcia





Fig. 2. View of: a) T-01M, b) pin on disc Rys. 2. Tester T-01M: a) widok ogólny, b) węzła tarcia

Measurements of the wetting angle are very useful in the evaluation of the degree of changes in the degradation of biomaterials in the event of a tissue response. The wetting angle θ is formed between the surface of a solid and a tangent to the surface of a drop routed from the point of contact of the three phases: solid-liquid-air [L. 14]. The wetting angle of materials is an indicator of the wetting properties [L. 13]. High wettability (hydrophilicity) is present at small wetting angles (below 90°) and low wettability, where hydrophobicity is present at wetting angles above 90°. According to [L. 15], the wettability of the surface of a material is an important parameter used to evaluate the biological interactions between the material and a living organism. In designs of materials to be used in implants, those interactions pertain to the adsorption of proteins, which results in adhesion and the proliferation of cells. Research [L. 16, 17] on the impact of wettability of surfaces and the associated hydrophobicity or hydrophilicity on biomaterialbiological environment interactions demonstrates that hydrophilic surfaces are conducive to adhesion and the activity of osteoblasts and many other cell types [L. 18]. The wetting angle was measured using an Attension Theta tensiometer made by Biolin Scientific. The test in which the static wetting angle was determined consisted in precise setting of a drop of the measurement liquid on the tested surface and in the immediate performance of the measurement. Using a disposable pipette, drops of liquid with the volume of approximately 4 μ l were placed on the surface of the sample, maintaining the same minimum height of the needle above the tested surface. A photo of the drop was taken using a Firewire digital camera 2 seconds after the drop was placed on the surface of the sample. At least six drops were placed on each sample, each in a different location. The photos of the drops were analysed using the OneAttension software. Then the results of the test were averaged and presented in the form of diagrams (Fig. 13).

RESULTS AND DISCUSSION

Figures 3a and **3b** show the morphology of the surfaces, with the measurement points indicated, and the characteristic radiation spectrums, together with an EDS analysis of the chemical composition of the Ti6Al4V alloy and the Ti6Al4V alloy implanted with N⁺ ions.

In the course of the EDS analysis, all elements in the Ti6Al4V titanium alloy and their percentage weight share were identified. In the case of the ion-implanted disk, the analysis demonstrated the presence of nitrogen on the surface of the sample. The morphology of the surface of the sample implanted with N⁺ ions indicated non-homogeneity of the top surface. The analysis of the chemical composition that was performed demonstrated that both the dark areas and the light areas have the same chemical composition. On the other hand, in the face surface of the reference sample, there were small scratches that were most often caused during the surface preparation process (grinding, polishing).

Figure 4 shows the average friction coefficients recorded in both testers during the tests in the technically dry friction conditions and in the conditions of friction with lubrication using the Ringer's solution.

By comparing the values of the friction coefficient, it was found that the process of implantation with N⁺ ions significantly contributed to the improvement of the tribological properties of the titanium alloy Ti6Al4V. The process reduced the value of the friction coefficient both in the technically dry friction conditions and in the conditions of friction with lubrication using the Ringer's solution during the tests performed on both devices. As a result of the tests performed on the TRB, the lowest value of the friction coefficient, $\mu_{avg} = 0.36$, was obtained for the friction couple consisting of a disk made of the titanium alloy Ti6Al4V implanted with N⁺ ions and a ball made of Al₂O₃ in conditions of friction with lubrication using the Ringer's solution. In the case of tests performed in the sliding motion, the recorded friction coefficient for a disk implanted with nitrogen ions were approx. 45% lower in the case of technically dry friction and



Fig. 3. SEM images and analyses of the chemical composition: a) Ti6Al4V, b) Ti6Al4V nitrogen ions Rys. 3. Obrazy SEM oraz analizy składu chemicznego: a) Ti6Al4V, b) Ti6Al4V implantowany jonami N⁺



Fig. 4. Coefficient of friction Rys. 4. Współczynnik tarcia

approx. 50% lower in the case of conditions of friction with lubrication using the Ringer's solution, compared to the reference disk.

After the tribological tests were completed, the traces of wear were measured on the samples. The measure that was adopted was the maximum depth and the surface area of the wear on the cross-section. **Figures 5–7** show the isometric images and the wear profiles of the tested disks.

Measurements performed using an optical profilometers demonstrated that, in the technically dry friction conditions, the top surface was worn evenly in both tested materials. An analysis of the wear profiles of the samples where lubrication with the Ringer's solution was applied has led to the conclusion that the lubricant increased the wear. In both cases, there was



Fig. 5. An isometric image of the trace of wear and the wear profile in a cross-section for technically dry friction: a) Ti6Al4V, b) Ti6Al4V nitrogen ions

Rys. 5. Obraz izometryczny śladu wytarcia oraz profil wytarcia na przekroju poprzecznym podczas tarcia technicznie suchego: a) Ti6Al4V, b) Ti6Al4V implantowany jonami N⁺



Fig. 6. An isometric image of the trace of wear and the wear profile in a cross-section in the conditions lubrication the Ringer's solution: a) Ti6Al4V, b) Ti6Al4V nitrogen ions

Rys. 6. Obraz izometryczny śladu wytarcia oraz profil wytarcia na przekroju poprzecznym po tarciu ze smarowaniem roztworem Ringera: a) Ti6Al4V, b) Ti6Al4V implantowany jonami N⁺



Fig. 7. The relationship between the depth (a) and the surface area of the wear (b) and the type of material and lubricant used

Rys. 7. Zależność głębokości (a) i pola wytarcia (b) od rodzaju materiału i zastosowanego środka smarowego

a characteristic groove that reached the maximum depth of 52.8 μm in the reference sample and 37.9 μm in the sample implanted with nitrogen ions. The tests performed using the Ringer's solution indicated that the disks implanted with N⁺ ions were more resistant to friction wear. The wear of the sample implanted with N⁺ ions was approx. 25% smaller compared to the reference sample.

The results of the measurements of wear of the tested friction couples after the tribological tests in sliding motion are shown in **Figs. 8–10**. The tests demonstrated that the titanium alloy implanted with nitrogen ions was characterized by higher resistance to friction wear.

The results of the measurements shown in the diagrams demonstrate that, for the sample made of the Ti6Al4V alloy, the depth and surface area of the



Fig. 8. An isometric image of the trace of wear and the wear profile in a cross-section for technically dry friction: a) Ti6Al4V, b) Ti6Al4V nitrogen ions

Rys. 8. Obraz izometryczny śladu wytarcia oraz profil wytarcia na przekroju poprzecznym podczas tarcia technicznie suchego: a) Ti6Al4V, b) Ti6Al4V implantowana jonami N⁺



Fig. 9. An isometric image of the trace of wear and the wear profile in a cross-section in the conditions lubrication the Ringer's solution: a) Ti6Al4V, b) Ti6Al4V nitrogen ions

Rys. 9. Obraz izometryczny śladu wytarcia oraz profil wytarcia na przekroju poprzecznym po tarciu ze smarowaniem roztworem Ringera: a) Ti6Al4V, b) Ti6Al4V implantowana jonami N⁺



Fig. 10. The relationship between the depth (a) and the surface area of the wear (b) and the type of material and lubricant used Rys. 10. Zależność głębokości wytarcia (a) i pola wytarcia (b) od rodzaju materiału i zastosowanego środka smarowego

wear were bigger compared to the sample with surface implanted with ions. The depth of the wear in the crosssection was approx. 5% smaller in technically dry friction conditions and 4% smaller in conditions of friction with lubrication using the Ringer's solution, compared to the disk made of Ti6Al4V. Macroscopic observations demonstrated that the countersample working with a Ti6Al4V disk was characterized by a significantly greater wear compared to the countersample working with the disk implanted with ions. Based on the results of the tests, it was concluded that implantation with N^+ ions results in a significant increase of resistance of the titanium alloy Ti6Al4V to friction wear.

The next stage of the study involved determination of the value of the wetting angles. **Figures 11–12** show examples of wetting angles of surfaces of disks made of the Ti6Al4V alloy before and after ion implantation for distilled water and for the Ringer's solution. The average values of the recorded wetting angles are shown in **Fig. 13**.



Fig. 11. A photo of the distilled water: a) Ti6Al4V, b) Ti6Al4V nitrogen ions Rys. 11. Przykładowe obrazy kropli wody destylowanej na powierzchni: a) Ti6Al4V, b) Ti6Al4V implantowany jonami N⁺



Fig. 12. A photo of the Ringer's solutions: a) Ti6Al4V, b) Ti6Al4V nitrogen ions Rys. 12. Przykładowe obrazy kropli roztworu Ringera na powierzchni: a) Ti6Al4V, b) Ti6Al4V implantowany jonami N⁺



Rys. 13. Średnie kąty zwilżania powierzchni stopu Ti6Al4V

Smaller wetting angles, for both distilled water and the Ringer's solution, were observed for the sample implanted with N^+ ions. Consequently, the implanted sample was characterized by better surface wetting.

In the case of distilled water, wettability improved by 30%, and, in the case of the Ringer's solution, it improved by 20%. This demonstrates that both tested materials were hydrophilic, which is conducive to adhesion of cells **[L. 19]**.

CONCLUSIONS

As a result of the tests and analyses that were performed, the following final conclusions were formulated. The observations of the structure of the morphology of the surface and the analyses of the chemical composition demonstrated that, at the assumed parameters of ion implantation of the titanium alloy Ti6Al4V, a top layer was produced that was enriched with nitrogen and did not contain any inclusions. The measurements of the amplitude parameters of the geometric structure of the surface indicate a 40% increase in the Ra parameter after the ion implantation, compared to the reference sample. As a result of the friction tests, different tribological characteristics were obtained, depending on the type of movement performed during the tests (different testing equipment). In the case of tests performed in the sliding motion, the recorded friction coefficient for a disk

implanted with nitrogen ions were approx. 45% lower in the case of technically dry friction and approx. 50% lower in the case of conditions of friction with lubrication using the Ringer's solution, compared to the reference disk. The tribological tests of the titanium alloy Ti6Al4V lead to the conclusion that the implantation of nitrogen ions results in better tribological properties of the alloy. Implantation with nitrogen ions significantly improved the wettability of the surface. In the case of distilled water, wettability improved by 30%, and, in the case of the Ringer's solution, it improved by 20%. In order to demonstrate an increase in the adhesion of cells, the scope of the tests must be expanded to include microbiological tests. The results of the tests demonstrated that ion implantation improves the characteristics of the titanium alloy Ti6Al4V.

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