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MECHANICAL AND TRIBOLOGICAL PROPERTIES OF OXIDE LAYERS OBTAINED ON Ti-6Al-7Nb ALLOY

WŁAŚCIWOŚCI MECHANICZNE I TRIBOLOGICZNE WARSTW TLENKOWYCH OTRZYMANYCH NA STOPIE Ti-6Al-7Nb

Key words:

titanium alloy, thermal oxidation, oxide layers, hardness, tribological properties

Słowa kluczowe:

stop tytanu, utlenianie termiczne, warstwy tlenkowe, twardość, właściwości tribologiczne

Abstract

The research presented in the paper was focused on determining the effects of thermal oxidation parameters on the mechanical and tribological properties of oxide layers formed on Ti-6Al-7Nb titanium alloy for biomedical applications. Titanium alloy was oxidized at 600 and 700°C for 72h. The study found that, at lower temperatures, the oxidized alloy surface was covered by a nonuniform oxide layer. A 100°C increase in the treatment temperature yielded a uniform layer covering the whole surface of the alloy. Hardness measurements showed

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that thermal oxidation resulted in the formation of oxide layers characterized by a 3-fold higher hardness compared to untreated alloy. Tribological tests showed that thermal oxidation effectively improves the resistance of Ti-6Al-7Nb alloy to sliding wear. Tribological tests demonstrated that an increased oxidation temperature was associated with improved resistance to sliding wear.

INTRODUCTION

A promising trend in the field of materials engineering is aimed at the improvement of application properties of titanium and its alloys by the modification of their surface layer and the formation of coatings with favourable physicochemical and mechanical properties **[L. 1]**. Growing quality requirements for the properties of the surface layer of metallic materials have contributed to the introduction of innovative and more cost-effective technologies for surface treatment. Modern techniques in the field of surface engineering allow for the formation of coatings with different structures, morphology, thicknesses, as well as chemical and phase composition **[L. 2–5]**.

Titanium and its alloys exposed to air oxygen undergo self-passivation **[L. 6–8]**. A passive layer is formed on these materials making them more resistant to the corrosive effects of environmental factors. However, the thickness of the oxide layer formed in this process is insufficient to ensure adequate tribological properties **[L. 9]**. This problem can be solved if an oxide layer of suitable thickness is formed on the surface of titanium and titanium alloys, increasing the resistance of material to corrosion and sliding wear **[L. 3**, **10]**.

The research presented in this paper was focused on determining the mechanical and tribological properties of oxide layers formed during isothermal oxidation on the surface of Ti-6Al-7Nb titanium alloy.

EXPERIMENTAL PROCEDURES

Rods of 40 mm in diameter made of Ti-6Al-7Nb alloy were used in the study. The alloy surface was oxidized in an electric chamber furnace with air circulation. The surface of samples (5 mm-thick discs, 39 mm in diameter) was pretreated by mechanical abrasion on SiC sandpaper, with a grit size from 600 to 2500. Titanium alloy was oxidized at 600 and 700°C for 72 h. After treatment at a given temperature, the samples were removed from the furnace and cooled in air.

The surface morphology of the oxide scale was analysed under a JEOL JSM-6480 scanning electron microscope. Microscopic observations were carried out at 1000x, 2000x, 5000x, and 7000x magnifications.

Microhardness was measured on a Vickers 401 MVD hardness tester. Tests were conducted for the indentation load range of 245-1960 mN (25 to 200 gf). Different loads were applied at which the indenter penetrated the layer and the substrate at different depths. The interval between the applications of loads was 15s. Each sample was tested 7 times and the results were averaged.

Tribological analysis of oxide layers formed on the surface of the Ti-6Al-7Nb alloy was carried out on a T-01 ball-on-disc tester in which the fixed ball of aluminium oxide Al_2O_3 is pressed with a predetermined force F onto the rotating disk made of the tested alloy. The coefficient of friction, linear wear, and volumetric wear were estimated for the tested friction pair. Tribological test parameters were selected according to a VAMAS technical note as follows:

- sliding speed – 0.1 m/s,
- load – 10 N,
- friction path – 1000 m,
- Al_2O_3 ball diameter – 10 mm.

RESULTS AND DISCUSSION

Surface morphology of the oxide scale

Figure 1 presents the surface morphology of the oxide scale formed after the treatment of Ti-6Al-7Nb alloy at 600 and 700°C for 72 h.

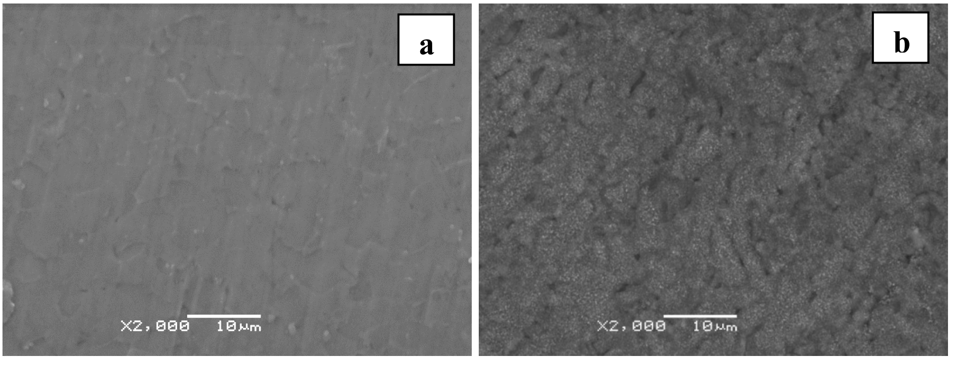

Fig. 1. Surface morphology of the oxide scale formed after treatment at 600°C (a) and 700°C (b)

Rys. 1. Morfologia powierzchni warstwy tlenkowej otrzymanej w temperaturze 600°C (a) i 700°C (b)

The analysis of samples carried out under the scanning electron microscope indicated that treatment at a lower temperature (600°C) led to the formation of an uneven thickness of the oxide layer on the alloy surface. Due to the small thickness of the layer, abrasions caused by the sanding of samples for tribological tests were still visible. An increase in the oxidation temperature to 700°C improved the uniformity of the oxide layer, which now covered the whole surface of the sample. The formed oxide layer was composed of very fine highly dispersed oxide particles. Abrasions caused by sanding were no longer visible, which indicated the greater thickness of the formed oxide layer. The thickness of the formed oxide layers was up to 1µm.

Measurements of surface hardness

Results from the measurement of surface hardness for the Ti-6Al-7Nb alloy before and after oxidation at 600 and 700°C at different loads and oxidation temperatures are presented in **Fig. 2**.

The lowest hardness (345 HV) was found for the nonoxidized surface. The surface exposed to thermal oxidation was characterised by significantly greater hardness. The study demonstrated that treatment at 600°C increased the hardness more than 2-fold to the value of 804 HV. An oxidation temperature increased to 700°C resulted in a 3-fold increase of surface hardness to 1072 HV. The greatest hardness of the Ti-6Al-7Nb alloy was noted at the lowest load of 245 mN.

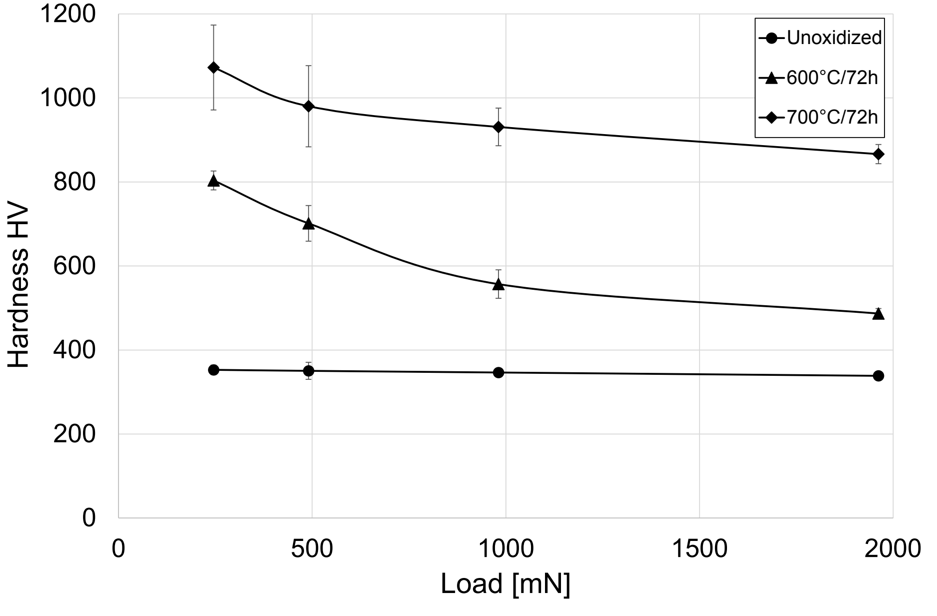

Fig. 2. Surface hardness of the Ti-6Al-7Nb alloy depending on the load and treatment temperature

Rys. 2. Twardość powierzchni stopu Ti-6Al-7Nb w zależności od obciążenia i temperatury utleniania termicznego

The hardness of the surface coated by oxide layers decreased with the increased load. This could be associated with the fact that the indenter penetrated deeper and deeper into the sample and thus perforated the oxide layer at greater loads. In this case, the results from tests were influenced by the lower hardness of the alloy substrate. The study also revealed that the reduction of hardness was greater for the sample oxidized at 600°C. This was due to the lower thickness of the oxide layer formed in this variant of oxidation and a greater effect on the measured substrate hardness, and thus an increased influence of the substrate on the measurement at greater loads. The presented research also revealed that increased oxidation temperature was associated with a greater scatter of measured hardness values. This can be attributed to the increased roughness of oxide layers, which has been reported by other authors **[L. 11]**.

Tribological properties

Tribological tests were carried out on a T-01 ball-on-disc tribometer to determine linear wear, volumetric wear, and the coefficient of friction. **Figure 3** presents the intensity of linear wear for the tested tribological pair without treatment, and with oxide layers formed after treatment at temperatures of 600 and 700°C.

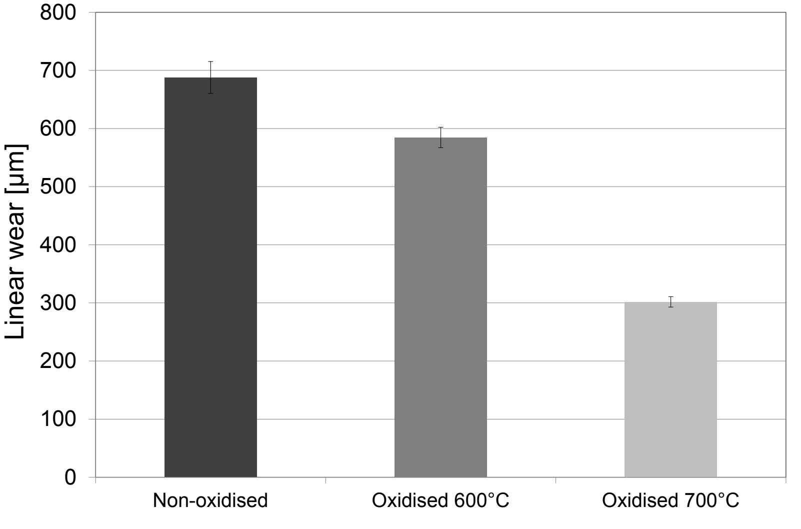

Fig. 3. Intensity of wear for the friction pair
Rys. 3. Wykres intensywności zużycia pary trącej

Tests demonstrated that the sliding wear for the tribological pair decreased with the increasing temperature of isothermal annealing. The lowest linear wear was found for a sample of the Ti-6Al-7Nb alloy oxidized at 700°C. The value of linear wear was more than 2-fold lower than that for the untreated alloy.

Results from the measurement of the initial and stabilised coefficient of friction are compared in **Fig. 4**. The coefficient of friction for the untreated alloy was 0.49. A comparable value of this coefficient for untreated Ti-6Al-7Nb alloy was reported by Fellah et al. **[L. 12]**. For the tribological pair comprising the sample with an oxide layer formed at 600°C and an Al_2O_3 ball, the initial coefficient of friction was more than 2-fold lower compared to the specimen without the oxide layer. With the progressing wear of the oxide layer, the coefficient of friction increased and stabilised at a level comparable to that for the untreated specimen. The coefficient of friction for the oxide layer formed at 700°C was significantly higher than that for the nonoxidized substrate. This could be due to the greater roughness of the oxide scale. After the complete wear of this layer, the coefficient of friction decreased to the value obtained for the nonoxidized material.

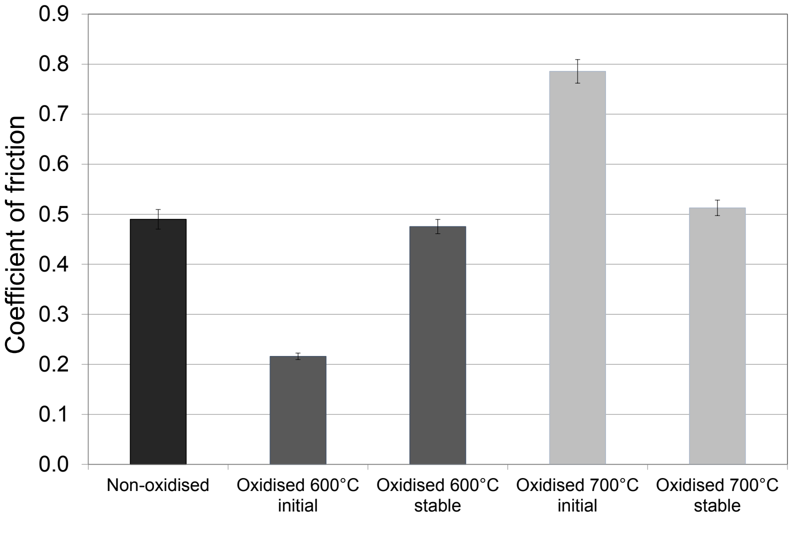

Fig. 4. Coefficient of friction for untreated Ti-6Al-7Nb alloy and alloy oxidized at 600 and 700°C

Rys. 4. Współczynnik tarcia dla nieutlenionego oraz utlenionego stopu Ti-6Al-7Nb w temperaturze 600 i 700°C

Figure 5 presents the results of measurements of the volumetric wear for discs made of nonoxidized and oxidized Ti-6Al-7Nb alloy. The presence of the oxide layer reduced the volumetric wear (**Fig. 5**). The greatest reduction of the volumetric wear was noted for a disc oxidized at 700°C. The obtained result does not coincide with the results of hardness measurements. This is due to the fact that the studies were performed using a load of 10N, which meant that the layer was relatively quickly worn down and further tribological interaction was with the substrate.

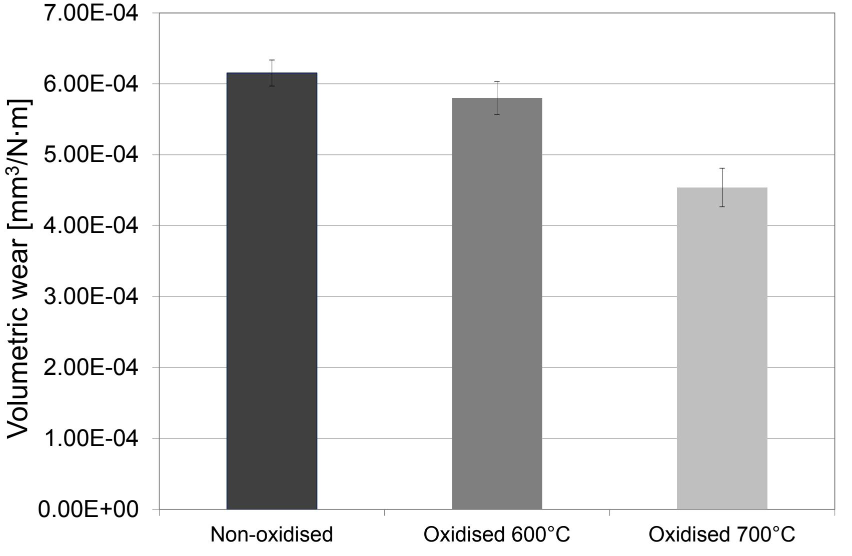

Fig. 5. Volumetric wear of the disc after tribological tests
Rys. 5. Zużycie objętościowe tarczy po badaniach tribologicznych

CONCLUSIONS

Analysis of the research data has led to the following conclusions:

1. The analysis of oxide morphology indicated that the surface of the Ti-6Al-7Nb alloy oxidized at a lower temperature (600°C) was covered by a nonuniform layer of oxide scale. The oxide layer formed at 700°C consisted of very fine highly dispersed oxide particles, which was more uniform, and covered the whole surface of the tested substrate.
2. Thermal oxidation resulted in the formation of oxide layers characterized by significantly greater hardness compared to the untreated alloy. The greatest hardness (1072 HV) was found for the surface oxidized at 700°C.
3. Surface modification of the Ti-6Al-7Nb alloy by isothermal oxidation effectively improves the resistance of material to sliding wear. Tribological tests demonstrated that thermal oxidation could reduce the TiO_2 friction coefficient and decrease the linear wear, the area of the wear tracks, and volumetric wear. The oxide layer formed at 700°C reduced wear by 26% compared to untreated samples.
4. The oxide layer formed at 600°C allowed for a more than 2-fold reduction in the value of the coefficient of friction for the tribological friction pair. The coefficient of friction for the oxide scale formed at 700°C was significantly higher than that for the nonoxidized substrate, which could be due to the greater roughness of the oxide layer.

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Streszczenie

Przedstawione w pracy badania koncentrują się nad określeniem wpływu parametrów utleniania termicznego na właściwości mechaniczne i tribologiczne warstw tlenkowych otrzymanych na biomedycznym stopie tytanu Ti-6Al-7Nb. Proces utleniania termicznego przeprowadzono w temperaturze 600 i 700°C w czasie 72 h. Stwierdzono, że w niższej temperaturze utleniania powierzchnia stopu była pokryta nierównomiernie warstwą tlenko-

wą. Podwyższenie temperatury utleniania termicznego o 100°C pozwoliło uzyskać jednolitą warstwę, w całości pokrywającą powierzchnię stopu. Pomiary twardości wykazały, że proces utleniania termicznego umożliwił otrzymanie warstw tlenkowych charakteryzujących się 3-krotnie wyższą twardością w porównaniu z twardością stopu niepoddanego utlenianiu. Badania tribologiczne wykazały, że utlenianie termiczne stanowi skuteczną metodę poprawy odporności na zużycie ścierne stopu Ti-6Al-7Nb. W testach tribologicznych wykazano, że wraz ze wzrostem temperatury utleniania odporność na zużycie ścierne ulegała zwiększeniu.