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Forming properties of titanium alloy for biomedical applications

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ABSTRACT

Purpose: A preparation of surface layers on the surface of the titanium alloy Ti-6Al-4V as a result of thermo-chemical treatment and a comparison of obtained layers from baseline. The results of studies comparing the structure and properties of titanium alloy Ti-6Al-4V with produced surface layers.

Design/methodology/approach: Test samples were the titanium alloy Ti-6Al-4V, which was subjected to heat-chemical treatment, consisting in annealing components in air for 1 hour at each temperature: 400°C, 450°C, 500°C, 550°C, 600°C. To achieve pursued objective the following tests: microstructural observation of the cross section, which made it possible to study the thickness of the produced coating study were performed, a study of the geometry of the surface was performed – roughness and hardness were measured. Wear resistance of each layer was shown. Ringer liquid simulates the conditions similar in the human body.

Results: Through the use of thermo-chemical treatment, it is possible to obtain surface layers of different thicknesses on titanium alloy Ti-6Al-4V. A method used for thermo-chemical treatment can produce a layer that affect the increase in hardness, whose value is almost twice higher than that of the without workup alloy. The evaluation of surface topography allowed to state that all samples were subjected to thermal-chemical treatment have a similar level of the development of the surface. The lowest roughness has alloy, annealed at 500°C. The study shows that the corrosion resistance of titanium alloy is dependent on the thickness of the obtained surface layers. The highest corrosion potential has the sample annealed at 500°C.

Originality/value: The paper summarizes comparative studies of titanium alloy and the alloy with produced oxide layers, proving by far superior to the results with produced alloy layers, which one were produced by the injection method, than in the case of alloys produced by the drawing method.

Keywords: Titanium alloy Ti-6Al-4V; Thermo-chemical treatment; Biomaterials

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PROPERTIES

1. Introduction

An important branch of the applications of titanium and its alloys is medicine. The group of materials have high requirements such as: non-toxicity, non-allergenicity, good corrosion resistance [1], mechanical properties, as well as resistance to wear by friction. Furthermore, titanium alloys should have a lack of tendency to form blood clots [2-5].

Titanium and its alloys are use in various fields of medical applications, mainly in cardiac surgery, traumatology, dental implants and bone as, among others, a hip or a knee. The choice of titanium alloys is related to their specific properties, such as: low density, high specific strength and their high biocompatibility [6-8]. However, due to the possibility of overlapping phenomena metalosis – transition alloying elements into the surrounding tissue initiating allergic and inflammatory processes [9-11], or implied by its high thrombogenicity, titanium and its alloys are subjected to various surface treatments limiting these phenomena [12-14]. One method is thermo-chemical treatment aimed at generate passive oxide [15-17] layer on the surface of the implant.

2. Materials and methodology

In this work surface layers of titanium alloy Ti-6Al-4V was modified by thermo-chemical treatment, which chemical composition is shown in Table 1. The modification of the surface layer consisted of annealing the elements in the atmosphere of oxygen for one hour at each temperature: 400ºC, 450ºC, 500ºC, 550ºC, 600ºC.

Table 1.
Chemical composition of titanium alloy Ti-6Al-4V [18]

Chemical composition	Al	V	C	Fe	O	N	H	Ti
Percentage	6.00	4.00	0.03	0.1	0.15	0.01	0.003	rest

A macroscopic image of a titanium alloy samples Ti-6Al-4V is shown in Figure 1. Test samples had been activated by mechanical process using sandpaper before conducted thermo-chemical process is shown in Figure 1.

The samples were subjected to microstructural observations to determine the thickness of the obtained surface layers with a light microscope Axiovert 25, which is equipped with a digital camera to image recording.

The test for wear resistance was carried out using a ball-tester, where in the specified time with the given load and speed, zirconium ball with a diameter 20 mm, acts on the surface of the sample. The degree of resistance to abrasive wear was determined by measuring the area of wear, and weight loss.

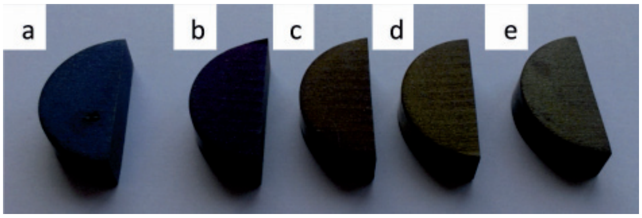


Fig. 1. Macroscopic image of samples with a modified surface layer as a result of thermo-chemical treatment in different temperatures: a) 600°C, b) 550°C, c) 500°C, d) 450°C, e) 400°C

The samples were tested microhardness. Vickers hardness tests were performed at a load 980.7 mN – HV 0.1 using a semi-automatic microhardness FM-7 company FUTURE-TECH.

In order to determine the surface topography, and its parameters studies were conducted using a profilometer Hommel T 1000. The determination of surface roughness parameters were performed in touch with the test surface by the engagement of the needle with a differential measurement system.

The next step was to study the electrochemical properties performed in Ringer solution at a temperature of 37°C. The composition of the solution was as follows: 0.39 g of potassium chloride; 8.6 g of sodium chloride; 0.48 g of calcium chloride to a solution of 1 dm^3. Electrochemical studies were performed using a potentiostat model 7050 by AMEL company digitally controlled via a computer PC equipped with Juniorassist software in a three-electrode system. The reference electrode was a calomel electrode (NEK), while the reference electrode was a platinum wire, test samples were a working electrode. A given potential during the tests varied from the cathode towards the anode in the range from E_{start} = (-2) V to E_{finish} = 8 V in relation to a saturated calomel electrode (NEK). Each sample was tested five times, a representative sample w/o treatment and all samples with thermo-chemical treatment.

3. Results

Microstructure of the produced titanium alloy surface layers is shown in Figure 2. Table 2 shows results of the thickness of the obtained layers.

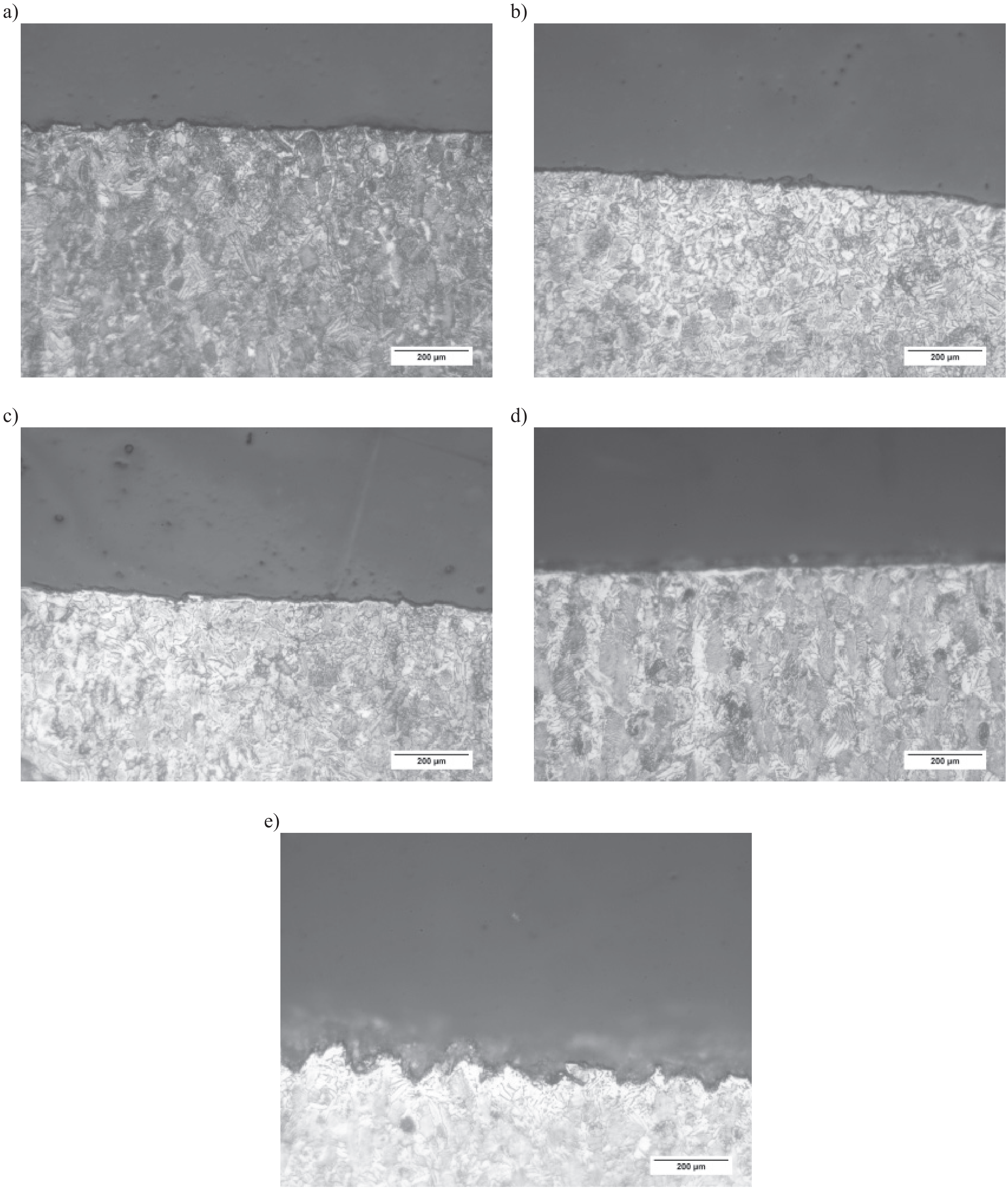


Fig. 2. Microstructure of titanium alloy Ti-6Al-4V after thermo-chemical treatment in temperatures: a) 400°C, b) 450°C, c) 500°C, d) 550°C, e) 600°C from arising the surface layers

Table 2.
Summary of the thickness of the surface layer obtained

Sample	Surface thickness, μm
600°C	130-220
550°C	104
500°C	33
450°C	24
400°C	20
w/o treatment	null

Table. 3.
Values of the abrasion areas occurred during testing abrasion resistance

Sample	Surface area of abrassion, μm^2
600°C	64772.5
500°C	80604.3
550°C	88264.9
400°C	136571.6
450°C	152880.4
w/o treatment	161938.8

Microstructural observations of the cross section of a titanium alloy with a surface layer formed have enabled to determine the thickness of the resulting layer. A thicker surface layer of oxides is allowed to obtain with the higher temperature process. Abrasion areas formed on the individual samples as a result abrasive wear are shown in Figure 3. Values of the abrasion areas occurred during testing abrasion resistance is shown in Table 3.

The smallest abrasion area after abrasion resistance test was recorded for a sample after thermo-chemical process in 600ºC temperature, in sequence at 500ºC temperature, the lowest wear resistance was characterized by the sample, which has not undergone any thermo-chemical treatment.

Microhardness results are summarized in Figure 4 using a column chart, and shows that microhardness depends on the treatment temperature of the sample.

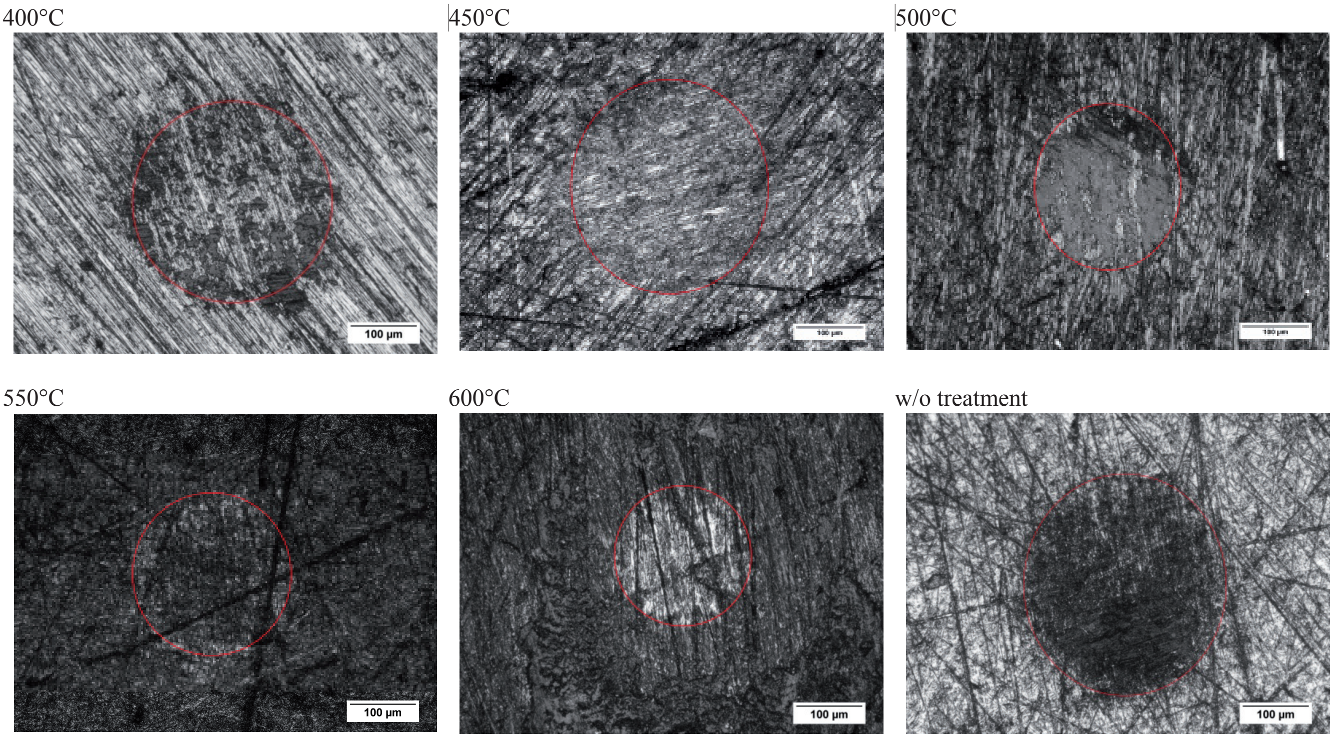


Fig. 3. Abrasive areas formed on the surface of each sample as a result of abrasive wear

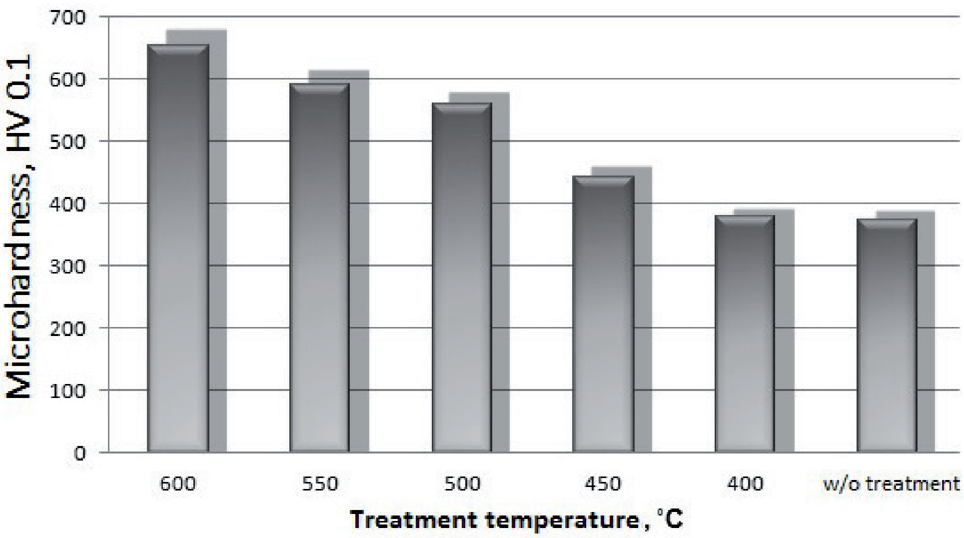


Fig. 4. Summarized microhardness results

Table. 5.
Summary of surface roughness parameters

Sample	Roughness parameters, µm					
	R_a	R_z	R_{max}	R_t	R_p	R_q
w/o treatment	0.29	2.71	4.24	5.23	1.7	0.41
400°C	0.21	1.93	2.48	2.64	1.08	0.28
450°C	0.20	1.98	2.51	2.87	1.16	0.27
500°C	0.19	1.94	2.63	2.73	1.39	0.26
550°C	0.20	1.89	2.46	2.65	1.10	0.27
600°C	0.23	2.02	2.70	3.08	1.35	0.31

Microhardness analysis shown dependence on temperature increase of the thermochemical process and microhardness increase of a surface layer. Alloy annealed at the highest temperature exhibits the greatest hardness, almost twice higher in comparison with same alloy without thermo-chemical treatment.

The measurement results of surface roughness parameters are summarized in Table 5 and surface roughness profiles are shown in Figure 5.

Evaluation of surface topography allowed to state in that all samples which were subjected to thermal-chemical treatment have a similar level of development of the surface. The smallest value of roughness, has an alloy annealed at temperature 500ºC.

Results of electrochemical study shows polarization curves in Figure 6.

Other parameters determined from the curve of corrosion are given in Table 6.

A summary of the corrosion curves – the chart showing sample with the best resistance to corrosion, as compared to baseline is presented in Figure 7.

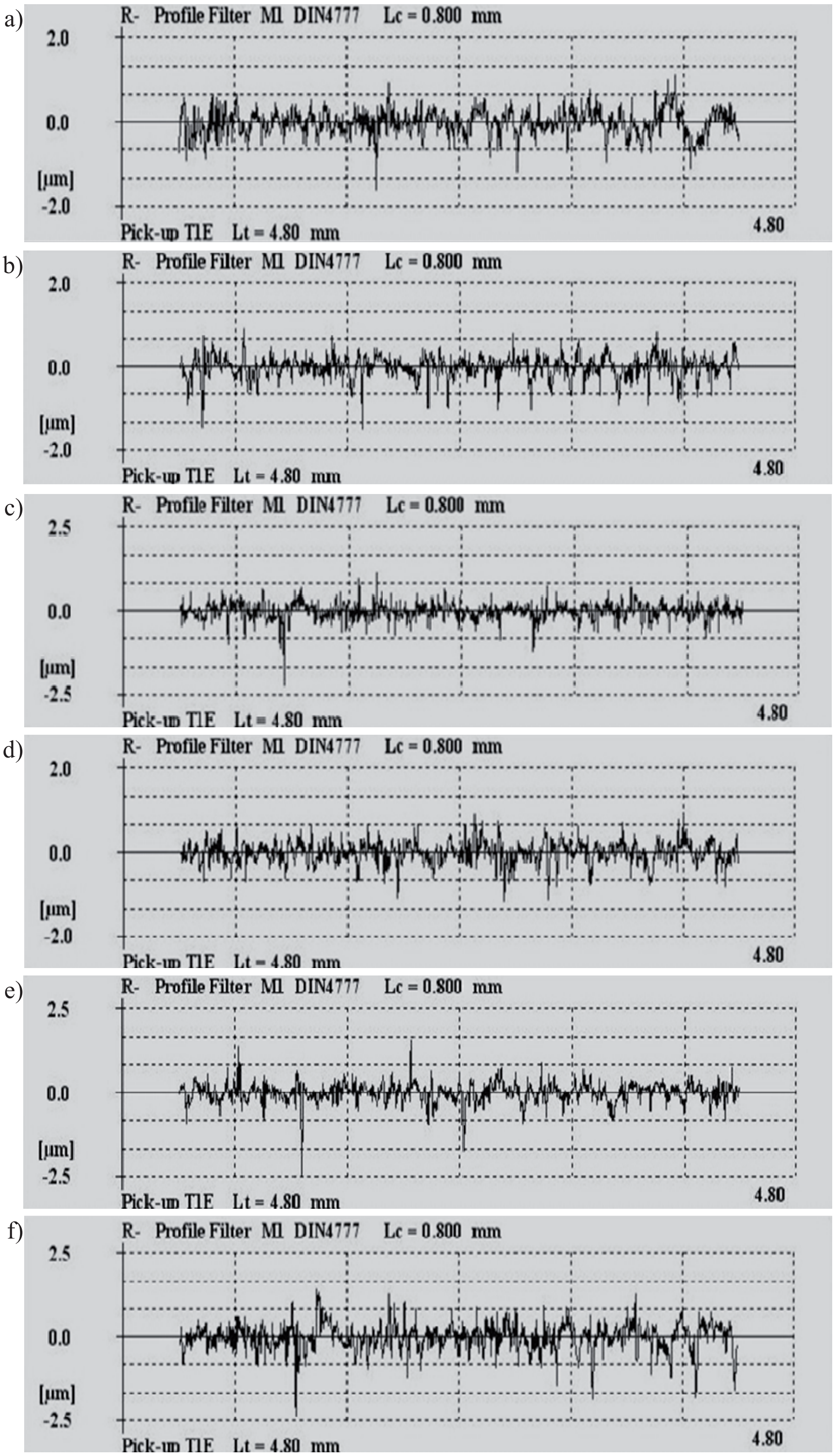
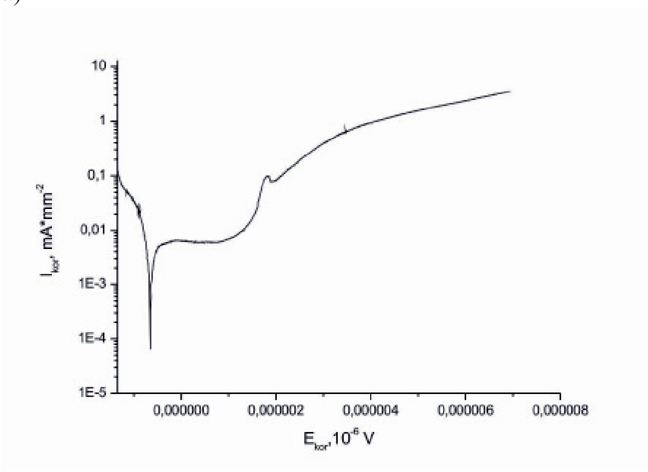


Fig. 5. Summary of surface roughness parameters in each of temperature which were used in thermo-chemical treatment a) 400°C, b) 450°C, c) 500°C, d) 550°C, e) 600°C, f) without treatment

a)



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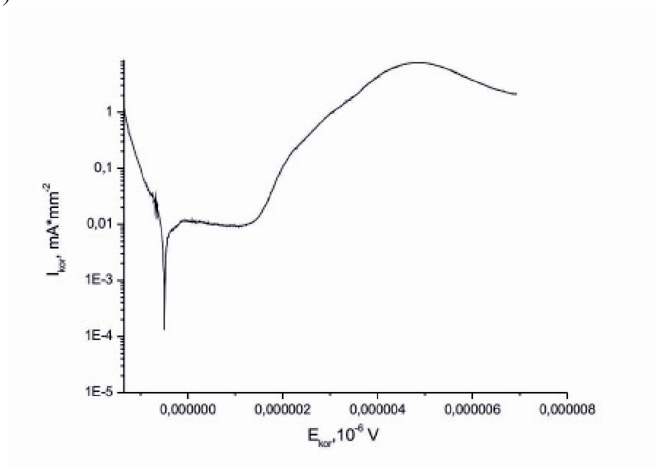

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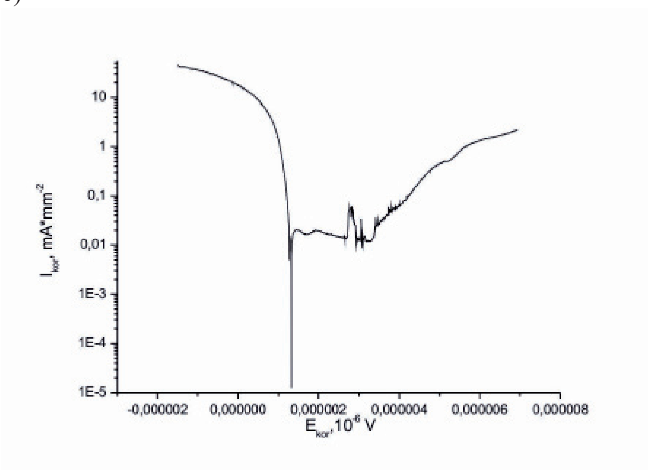
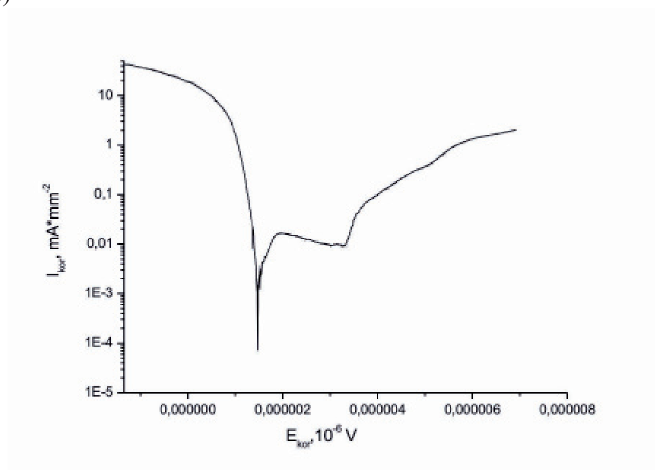
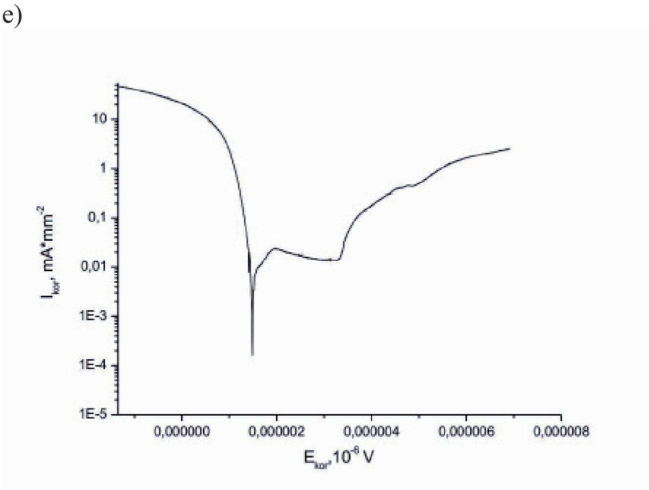


d)



e)



f)

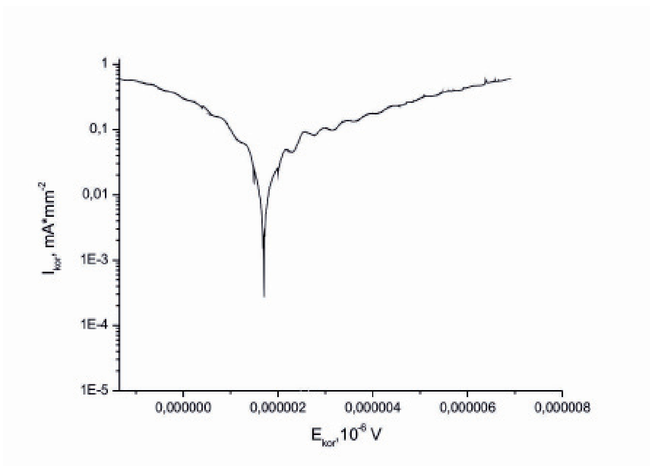


Fig. 6. Polarization curves showing performed electrochemical studies after thermo-chemical treatment in temperatures a) 600°C, b) 550°C, c), without treatment d) 400°C, e) 450°C, f) 500°C

Table. 6.
Results of electrochemical studies for titanium alloy after thermal-chemical treatment in each temperature

Sample	$E_{cor,}$ V *10^{-6}	$I_{cor,}$ mA*mm^{-2}
600°C	-6.11*10^{-7}	0.00196
550°C	-5.29 *10^{-7}	0.00499
w/o treatment	1.33 *10^{-6}	0.01167
400°C	1.58 *10^{-6}	0.00375
450°C	1.56 *10^{-6}	0.00764
500°C	1.66 *10^{-6}	0.00087

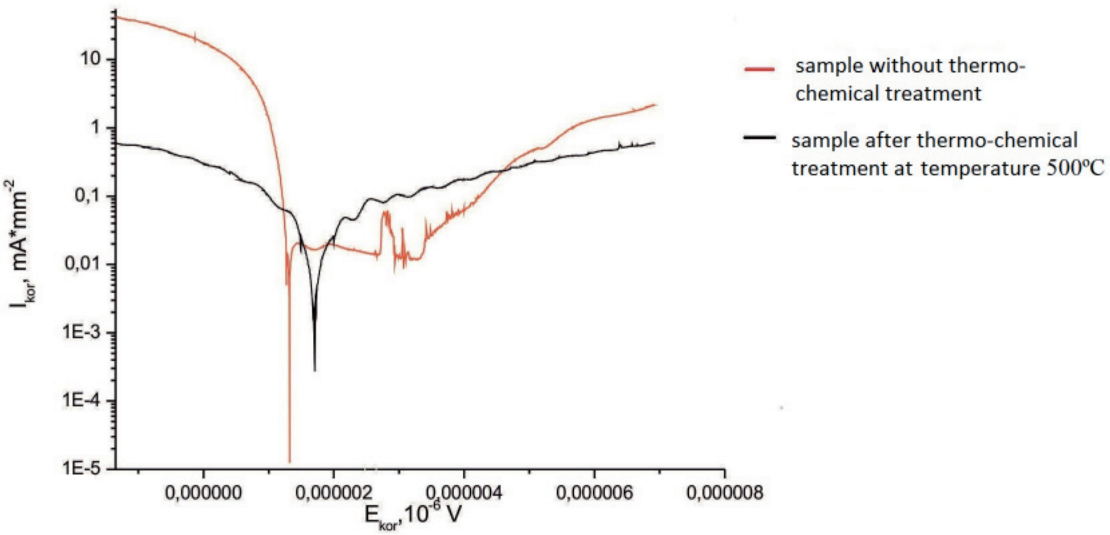


Fig. 7. Polarization curves shown comparison of electrochemical state for sample without thermal-chemical treatment and for sample with thermal-chemical treatment in temperature 500°C

Corrosion tests carried out in environments simulating human body fluids showed that a sample treated at temperature 500°C has the highest corrosion potential. The lower current density, while higher potential corrosion is the higher corrosion resistance. The chart of the sample treated at a temperature at 500°C is shifted in the direction of the most positive potentials in comparison with other samples.

4. Conclusions

The applied method of thermo-chemical treatment The applied method of thermo-chemical treatment allows to obtain different thickness of the surface layers on titanium alloy Ti-6Al-4V. The higher treatment temperature is used, the thicker oxide layer may be. The thickest oxide surface layer was obtained on the sample surface at annealing temperature equaled 600°C. The obtained layer is characterized by irregular thickness over the entire surface and waviness.

The test for resistance to wear enabled to determine how thermochemical treatment has influence on tribological resistance. Microstructural observation allowed to state that the best abrasion resistance has a sample after thermal-chemical treatment at temperature 600°C. Relatively to inferior wear resistance was characterized by a baseline sample.

Microhardness analysis showed that the higher treatment temperature increased the hardness. Alloy annealed at the highest temperature exhibits the greatest hardness, twice bigger in comparison with a baseline sample without thermal-chemical treatment. Higher hardness provides less abrasion of rubbing elements for the connection implants.

The evaluation of surface topography allowed to state that all samples, after process of thermal-chemical treatment have a similar level of development of the area. The lowest surface roughness is characterized by alloy at temperature annealed 500ºC, where the parameter of the arithmetic mean ordinate profile was 0.19 µm, and the value of the parameter for the not heat-treated sample was 0.29 µm.

The study shows that the corrosion resistance of titanium alloy Ti-6Al-4V depends on the thickness of the resulting oxide layers on material surface. The best resistance to corrosion processes is alloy treated at temperature 500ºC. It has the highest corrosion potential with simultaneously low current density.

From the medical point of view the best properties of the sample has been treated at temperature 500ºC, it has the best resistance to corrosion processes, what in biomedical aspects is one of the main requirement, moreover, has the highest level of surface development and one of the better hardness (as compared to the other samples) and mostly the best abrasion resistance.

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