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Influence of mechanical activation and heat treatment on surface development and oxide layer thickness of Ti6Al4V ELI alloy

M. Szota, A. Łukaszewicz *, A. Bukowska
Faculty of Production Engineering and Materials Technology,
Czestochowa University of Technology,
Al. Armii Krajowej 19, 42-200 Częstochowa, Poland
* Corresponding e-mail address: adrian.luakszewicz@gmail.com

ABSTRACT

Purpose: The paper presents the results of mechanical activation of the surface on oxide layer thickness after heat treatment of TU6Al14V ELI alloy.

Design/methodology/approach: Specimens were made from 5 mm diameter rod cut into semicircular slices. The samples were mechanically activated throughout mechanical treatment of the surface: one sandblasted with glass beads during 5 minutes and other ground with sandpaper grit 40, 180, 220 and 800 during 7.5 and 15 minutes.

Findings: Then microstructure of specimens etched with Kroll solution was observed using an optical microscope and roughness parameters of the surface were measured.

Research limitations/implications: Afterwards heat treatment (550°C, 5 hours) was conducted, then roughness parameters and thickness of the oxide layer were measured by means of a scanning microscope.

Practical implications: The conducted research showed up that mechanical activation of the surface which cause an increase of surface development results in greater thickness of the oxide layer which is formed during heat treatment. Nevertheless, mechanical activation that results in a decrease of surface development, such as polishing, results in a decrease of oxide layer thickness.

Originality/value: The results of the research can be used to obtain the desired thickness of the oxide layer in the production of the elements that require increased wear and corrosion resistance.

Keywords: Surface engineering, Titanium alloys, Heat treatment, Mechanical activation

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BIOMEDICAL AND DENTAL ENGINEERING AND MATERIALS

1. Introduction

The use of titanium and its alloy in many industries, such as chemical, aerospace, automotive or medical, is a result of a combination of very good mechanical properties and corrosion characteristics [1-6]. In order to improve the properties of the final elements from these alloys they are subjected to a surface treatment that improves their performance properties – in particular in the aspect of improving corrosion resistance as well as to improve wear resistance [7-9].

On the basis of literature reports the use of thermal oxidation of the surface layer allows to improve tribological properties of titanium and its alloys [19,20]. The thermal oxidation affects wear reduction from 4 to 6 times in comparison to elements not subjected to this process [10-18].

As part of the work carried out by many authors, it was found out that there is the possibility of oxidation of titanium from the temperature 450°C to temperatures above 850°C. However, it was found out that in the case of treatment at temperatures above 800°C, despite the significant increase in the thickness of the oxide layer, this layer is very brittle and breaks down which is especially observed for titanium Grade 2.

On the other hand, the layers obtained at too low temperatures, after a short oxidation time, are too thin for tribological applications [10,17].

As part of this publication, research on the possibility of producing oxide layer by heating at temperature 550°C in time 5 h after prior mechanical activation were carried out. Research focuses on microstructural changes and the thickness of the produced layers.

2. Materials and methodology

The chemical compositions of the titanium Grade 23 – Ti6Al4V ELI alloy which was used for the studies is presented in Table 1. The material was tested in the form of a rod with a diameter of 5 mm, from which 4 mm thick slices were cut using an angle saw. These slices were transversely cut into halves resulting in semicircular samples.

Table 1.
Chemical composition of titanium alloy

Element	Al	V	C	Fe	O	N	H	Ti
wt.%	6.0	4.0	0.03	0.1	0.1	0.01	<0.003	rest

During studies, surface layers were obtained after mechanical activation and heat treatment. The mechanical treatment consisted of sandblasting with glass beads for 5 minutes, abrasion with sandpapers – grits 40, 180, 220 and 800 during time 7.5 min and 15 min.

The next stage was the preparation of metallographic specimens from the obtained material and etching them with the Kroll's solution (2 ml HF, 2 ml HNO_3, 96 ml H_2O). The microstructure of the surface zone was taken using the Olympus GX41 light microscope.

For such samples, surface topography was examined and roughness parameters such as R_a (arithmetic means deviation of profile ordinates from the mean line), R_z (average roughness value by 10 points), R_t (total height of the profile), R_q (the average square deviation of the profile from the mean line along the measurement or elementary section) were determined. This test was carried out using the Hommel T1000 profilometer.

Then a heat treatment was carried out, consisting of heating the samples at a temperature of 550°C during 5 hours. The input sample was not machined. After the completion of the process, the surface geometry analysis, as well as the thickness measurement of the oxide layers, were carried out.

3. Carried out researches

Figure 1 shows microstructure images for the input sample without mechanical treatment, only after heat treatment, whereas Figures 2-6 show the samples after mechanical activation using glass beads, and sandpapers grit – 40, 180, 220 and 800 at times 7.5 and 15 minutes.

Based on microstructural observations, it was found out that the largest development of the surface was reached on the sample after sandblasting using glass beads what was indicated by the largest roughness. Besides, changes were found at the surface of the sample at depth from 4 µm for the samples after mechanical activation with grit 800 paper to 9 µm for the sample after sandblasting with glass beads.

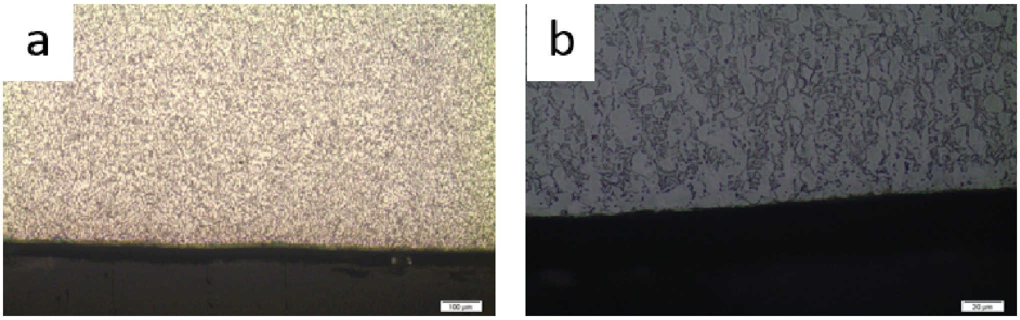

Fig. 1. Cross-sections of microstructures of the samples without mechanical activation of surface a) magnitude x100, b) magnitude x500

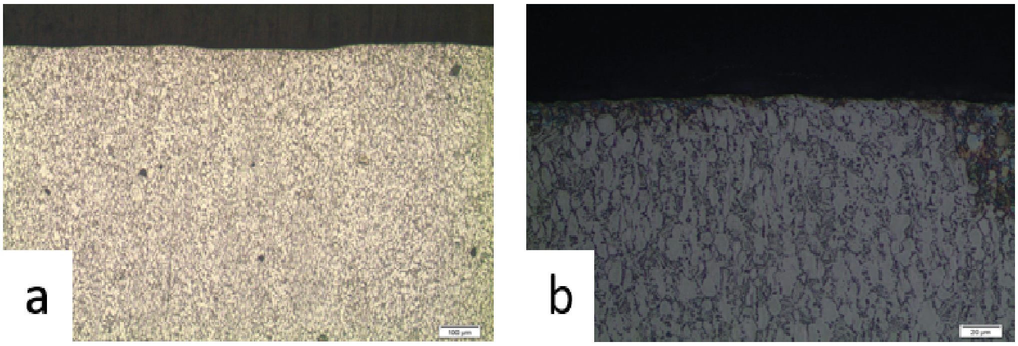

Fig. 2. Cross-sections of microstructures of the samples after sandblasting with glass beads a) magnitude x100, b) magnitude x500

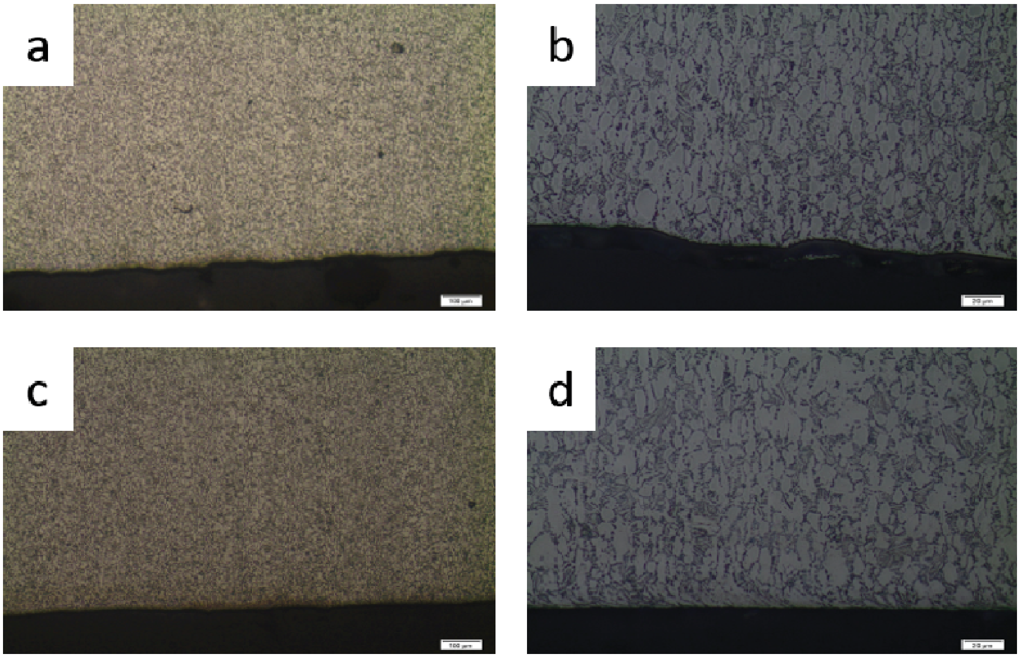

Fig. 3. Cross-sections of microstructures of the samples after mechanical activation with sandpaper (grit 40) – a, b after 7.5 min., c, d after 15 min., where a and c – magnitude x100, and b and d – magnitude x500

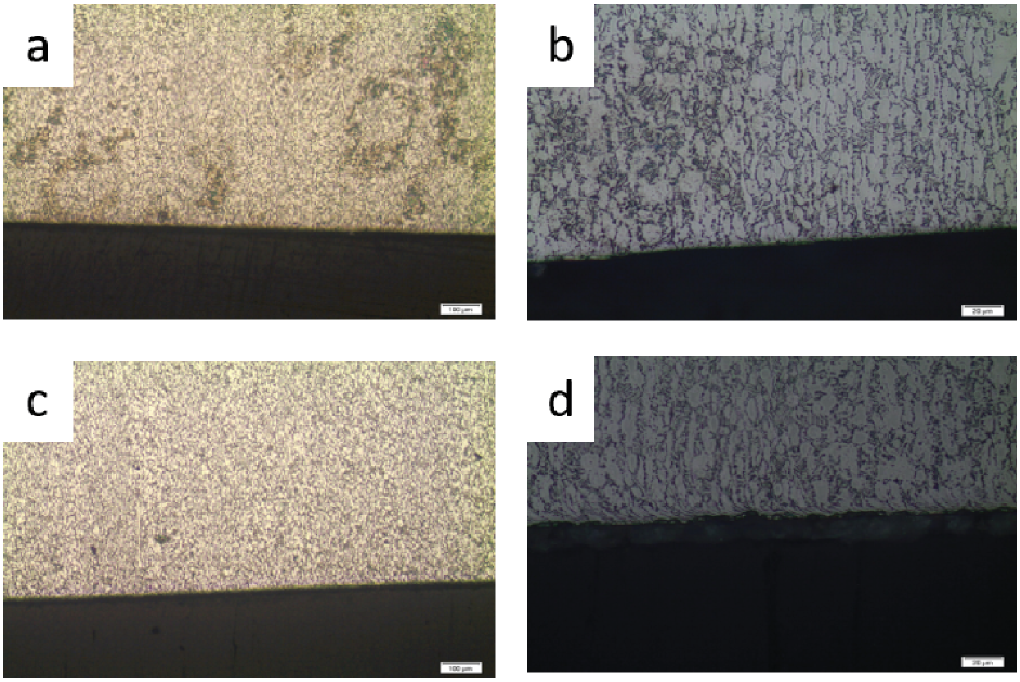

Fig. 4. Cross-sections of microstructures of the samples after mechanical activation with sandpaper (grit 180) – a, b after 7.5 min., c, d after 15 min., where a and c – magnitude x100, and b and d – magnitude x500

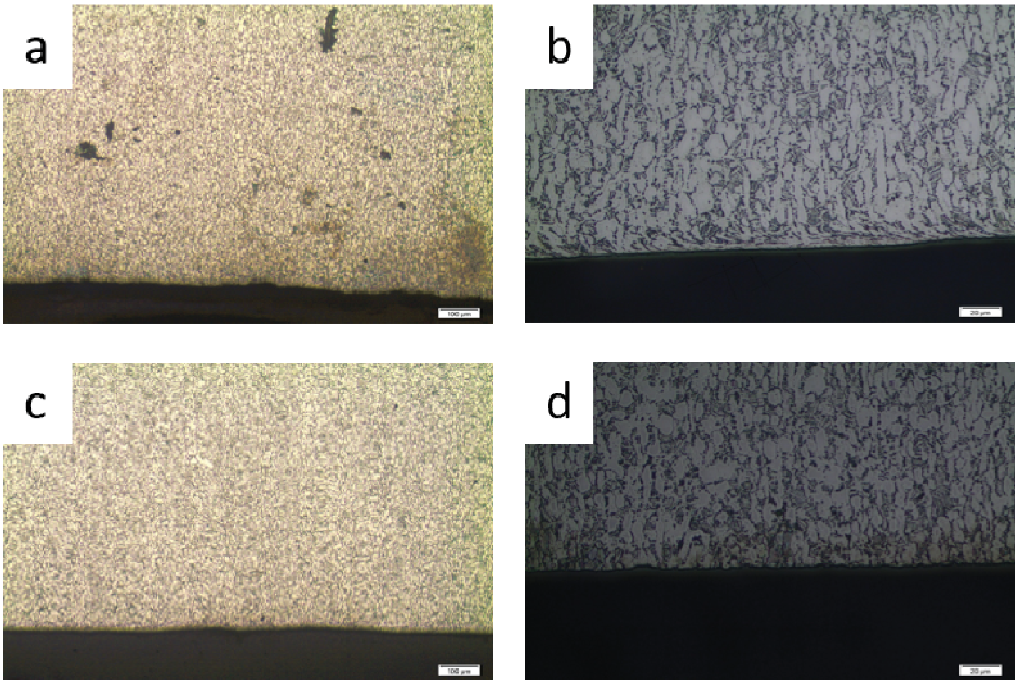

Fig. 5. Cross-sections of microstructures of the samples after mechanical activation with sandpaper (grit 220) – a, b after 7.5 min., c, d after 15 min., where a and c – magnitude x100, and b and d – magnitude x500

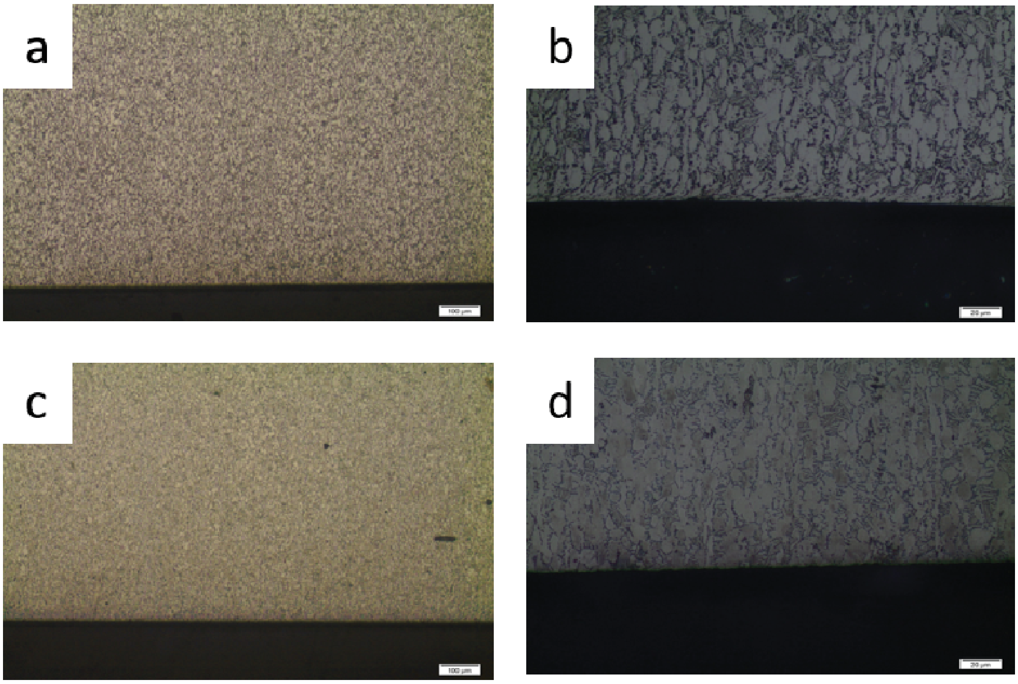

Fig. 6. Cross-sections of microstructures of the samples after mechanical activation with sandpaper (grit 800) – a, b after 7.5 min., c, d after 15 min., where a and c – magnitude x100, and b and d – magnitude x500

In order to analyse the differences, depending on the time of mechanical activation, studies of the surface development were carried out. Values of roughness parameters on samples mechanically activated with abrasive paper during 7.5 and 15 minutes, after sandblasting with glass beads as well as samples without mechanical activation before heat treatment are presented in Table 2. Values of roughness parameters after heat treatment are presented in Table 3. For each parameter, three measurements for each sample were carried out. Afterwards, arithmetic means were calculated (bold values).

The obtained results confirm microscopic observations, where it was rightly observed that the largest surface development has the sample after sandblasting with glass beads. The results indicate a specific relationship where the surface roughness value for the samples after grinding with sandpapers grit – 800, 220 and 180 is decreased – there is a decrease in R_a value after heat treatment, relative to these values for the samples before processing. Interestingly, for papers with these grits, there is an apparent decrease in the surface development for longer mechanical activation times, whereas the opposite occurs in the case of sandpaper grit – 40. In the case of the sample after sandblasting with glass beads and heat treatment reduction of the surface development were observed. These dependencies can be explained by the appearance of a thin film of oxides on the surface of the studied samples, appearing of oxides in irregularities on the surface of the samples fill them and reduce the surface development.

In order to analyse the thickness of these layers and the influence of the time of mechanical activation, tests were carried out using a scanning microscope. An exemplary measurement is shown in Figure 7, whereas a comparison of the results of oxide layer thickness measurements is presented in Table 4.

On the basis of literature reports, it was confirmed that the results of oxide layer thickness measurements were carried out correctly [16]. The results show a dependence that more extended mechanical activation contributes to the increase of surface development, and thus to the increase of the oxide layer thickness – as can be seen for glass beads and sandpaper grits – 40 and 180. In contrast, the decrease in the oxide layer thickness, after a longer mechanical activation, is observed for grit 220 and 800, which is the result of a decrease in surface roughness due to polishing.

Table 2.
Roughness values for the samples before heat treatment

Sample			Roughness parameters, µm			
			R_a	R_z	R_q	R_t
Without mechanical activation			0.64	3.39	8.81	1.04
			0.49	2.35	3.39	0.62
			0.43	2.86	3.55	0.55
			0.52	**2.87**	**5.25**	**0.74**
After sandblasting with glass beads			2.32	10.61	13.20	2.63
			2.94	11.56	15.32	2.76
			1.78	12.92	17.43	2.82
			2.35	**11.70**	**15.32**	**2.74**
Sandpaper	40	7.5 min	0.49	4.05	7.84	0.66
			0.49	3.71	6.23	0.67
			0.75	5.41	9.51	1.07
			0.58	**4.39**	**7.86**	**0.80**
		15 min	0.64	4.20	6.48	0.86
			0.69	4.08	5.50	0.94
			1.02	5.85	9.27	1.46
			0.78	**4.71**	**7.08**	**1.08**
	180	7.5 min	0.66	4.29	7.39	0.91
			0.44	2.94	6.08	0.65
			0.51	3.36	6.89	0.77
			0.54	**3.53**	**6.79**	**0.78**
		15 min	0.65	4.27	6.82	0.87
			0.38	3.08	3.98	0.53
			0.61	4.82	6.57	0.84
			0.55	**4.06**	**5.79**	**0.75**
	220	7.5 min	0.54	4.04	5.67	0.79
			0.55	4.69	5.87	0.76
			0.32	2.70	3.10	0.42
			0.47	**3.81**	**4.88**	**0.66**
		15 min	0.36	2.60	3.48	0.46
			0.32	2.34	3.17	0.41
			0.26	2.31	3.60	0.35
			0.31	**2.42**	**3.42**	**0.41**
	800	7.5 min	0.13	1.08	1.42	0.16
			0.13	1.20	1.63	0.17
			0.14	1.22	1.51	0.18
			0.13	**1.67**	**1.52**	**0.17**
		15 min	0.12	1.08	1.61	0.15
			0.11	0.99	1.36	0.14
			0.12	1.05	1.25	0.15
			0.12	**1.04**	**1.41**	**0.15**

Table 3.
Roughness values for the samples after heat treatment

Sample			Roughness parameters, µm			
			R_a	R_z	R_q	R_t
Without mechanical activation			0.73	4.69	10.63	1.10
			0.45	2.59	3.40	0.58
			0.39	2.81	5.25	0.62
			0.52	**3.36**	**6.43**	**0.77**
After sandblasting with glass beads			2.20	10.20	13.01	2.76
			2.30	12.22	14.25	3.36
			2.33	10.39	13.81	2.37
			2.28	**10.94**	**13.69**	**2.83**
Sandpaper	40	7.5 min	0.72	4.88	8.23	1.10
			0.68	4.33	6.22	0.90
			0.89	5.49	7.50	1.14
			0.76	**4.90**	**7.32**	**1.05**
		15 min	1.55	8.51	11.58	2.10
			0.93	5.57	7.95	1.33
			0.85	5.09	6.65	1.11
			1.11	**6.39**	**8.73**	**1.51**
	180	7.5 min	0.58	2.98	5.12	0.78
			0.55	3.66	5.88	0.75
			0.48	3.35	4.76	0.69
			0.54	**3.33**	**5.25**	**0.74**
		15 min	0.53	3.39	4.49	0.68
			0.42	3.75	5.30	0.57
			0.55	3.97	4.58	0.73
			0.50	**3.70**	**4.79**	**0.66**
	220	7.5 min	0.27	2.33	2.73	0.36
			0.32	2.47	3.26	0.41
			0.34	3.15	4.48	0.46
			0.31	**2.65**	**3.49**	**0.41**
		15 min	0.32	2.68	4.30	0.43
			0.32	2.28	2.99	0.42
			0.24	2.39	3.05	0.32
			0.29	**2.45**	**3.45**	**0.39**
	800	7.5 min	0.16	1.43	2.83	0.23
			0.21	1.58	3.92	0.35
			0.17	1.42	1.91	0.22
			0.18	**1.48**	**2.89**	**0.27**
		15 min	0.09	0.91	1.29	0.12
			0.10	0.81	1.13	0.13
			0.12	1.18	1.67	0.17
			0.1 0	**0.97**	**1.36**	**0.14**

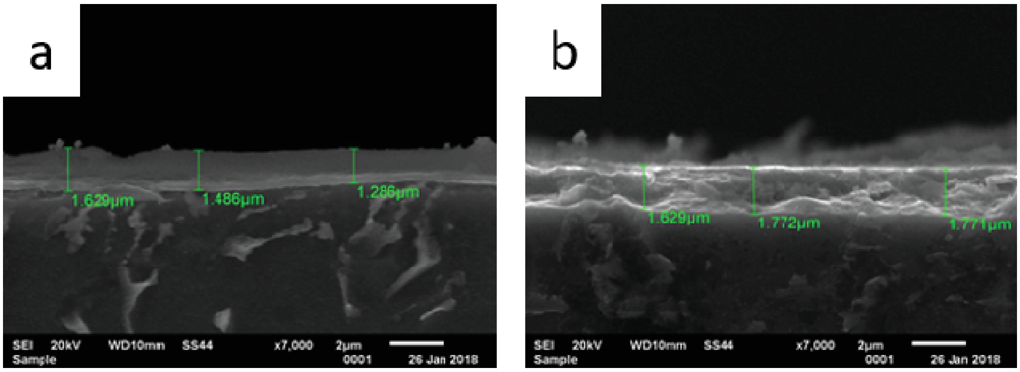

Fig. 7. Example of observed values of oxide layer thickness on the sample's surfaces after mechanical activation with sandpaper (grit 40) and heat treatment (550°C/5h), a) mechanical activation 7.5 min., b) mechanical activation 15 min

Table 4.
Values of oxide layer thickness after heat treatment

Sample	Measurement, µm			
	1	2	3	arithmetic mean
Without mechanical activation	1.157	1.200	1.007	1.121
After sandblasting with glass beads	4.115	3.343	2.657	3.372
Sandpaper grit 40/7.5min	1.257	1.114	1.143	1.171
Sandpaper grit 40/15min	1.629	1.772	1.771	1.724
Sandpaper grit 180/7.5min	1.223	1.286	1.294	1.194
Sandpaper grit 180/15min	1.643	1.429	1.600	1.557
Sandpaper grit 220/7.5min	1.130	1.171	1.182	1.161
Sandpaper grit 220/15min	1.127	1.136	1.112	1.125
Sandpaper grit 800/7.5min	0.692	1.002	0.998	0.897
Sandpaper grit 800/15min	0.789	0.857	0.943	0.863

4. Conclusions

The tests carried out related to the outflow of mechanical activation and heat treatment of Ti6Al4V titanium alloy showed the possibility of controlling the thickness of the oxide layer on the surface of the alloy.

The analysis of the results allowed to state that with the increase of surface roughness, the thickness of the oxide layer on the surface of the alloy increases.

This dependence can be used in the production of elements requiring increased wear and corrosion resistance because the appearance of a ceramic layer TiO_2 slows down corrosion processes due to the lack of electrical conductivity of this layer and improve tribological properties because of mechanical properties of TiO_2.

However, to avoid pitting corrosion, an appropriate level of surface development should be selected.

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