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Change in the surface structure and the oxide layer of the Ti6Al4V ELI alloy as a result of mechanical and heat treatment

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Abstract

Surface treatment, both mechanical, chemical and thermal causes a number of changes to the external structure of meterial details. The obtained properties are intended to improve the quality of material details made of a given alloy or pure metal. This paper presents the results of mechanical surface treatment to the thickness of the oxide layer after heat treatment of the TU6Al14V ELI alloy. The experiments were performed for a rod with a diameter of 5 mm cut into semicircular slices. The samples were mechanically activated by mechanical treatment of the surface: sandblasting with glass balls for 5 minutes, sanded with 40, 180, 220 and 800 grit sandpaper for 7.5 and 15 minutes. Using an optical microscope, the microstructure of the samples etched with Kroll's solution was assessed and the surface roughness parameters were measured. The next step was to carry out the heat treatment (at the temperature of 550 ^{o}C, for 5 hours), and then the roughness parameters and the thickness of the oxide layer were measured using a scanning microscope. The conducted research has shown that mechanical treatment of the surface resulting in an increase in surface development causes an increase in the thickness of the oxide layer formed during heat treatment. However, machining to reduce surface development, such as polishing, reduces the thickness of the oxide layer. The test results can be used to obtain the desired thickness of the oxide layer in the production of elements requiring increased resistance to wear or corrosion.

Keywords: titanium alloy, oxide layer, alloy strength.

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 [3, 5, 17, 20]. 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.[13, 15]

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. The thermal oxidation affects wear reduction from 4 to 6 times in comparison to elements not subjected to this process. [1, 6–10, 12]

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^{o}C to temperatures above 850^{o}C. However, it was found out that in the case of treatment at temperatures above 800^{o}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, 16]

2 Materials and methodology

As part of this publication, research on the possibility of producing oxide layer by heating at temperature 550^{o}C in time 5h after prior mechanical treatment were carried out. Research focuses on microstructural changes and the thickness of the produced layers [2, 4, 14, 18, 19].

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The chemical compositions of the titanium Grade23 - Ti6Al4V ELI alloy which was used for the studiesis 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 intohalves resulting in semicircular samples.

Table 1. Chemical composition of titanium Grade23 - Ti6Al4V ELI 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 (2ml 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 ata temperature of550^oC 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 Experimental results

Fig. 1 shows microstructure images for the input sample without mechanical treatment, only after heat treatment, whereas Figs. 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.

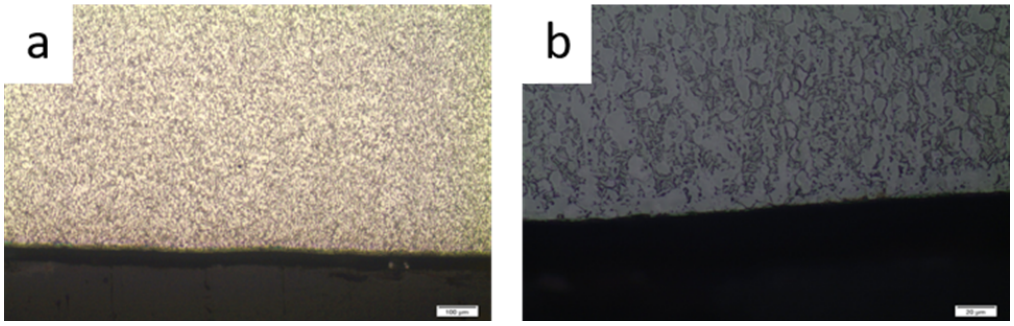

Figure 1. Cross-sections of microstructures of the samples without mechanical treatment
a) magnitude x100, b) magnitude x500

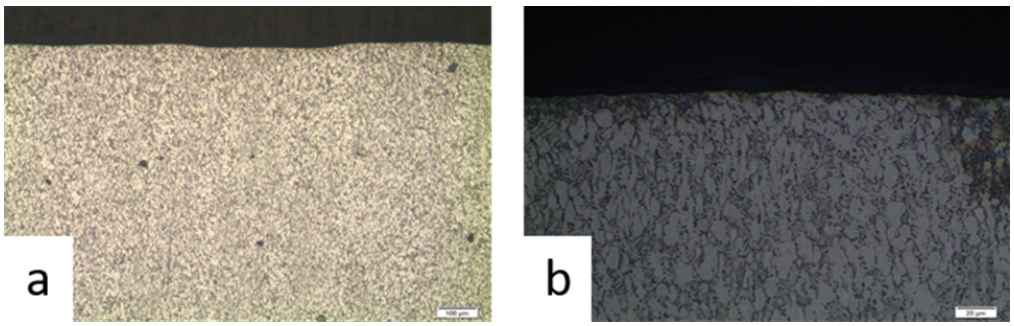

Figure 2. Cross-sections of microstructures of the samples after sandblasting with glass beads
a) magnitude x100, b) magnitude x500

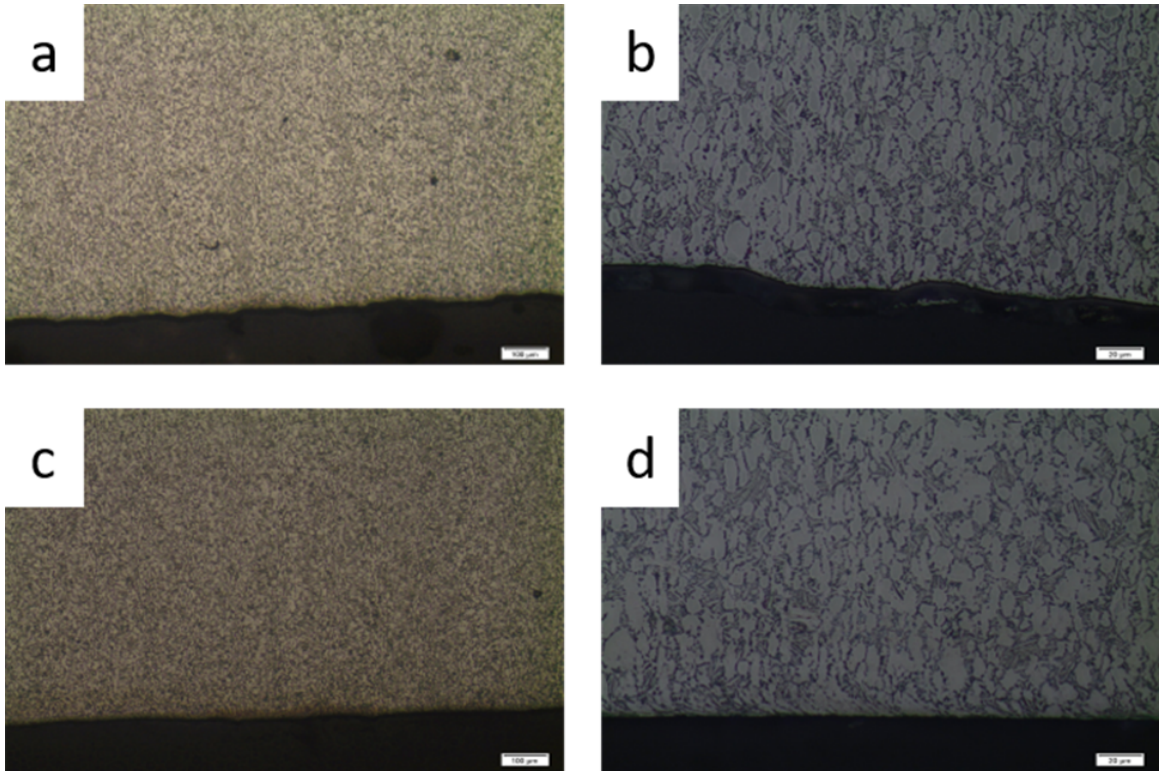

Figure 3. Cross-sections of microstructures of the samples after mechanical activation with sandpaper (grit 40) a, b after 7.5 min., c, d after 15 min., where a and c – magnitude x100, b and d – magnitude x500

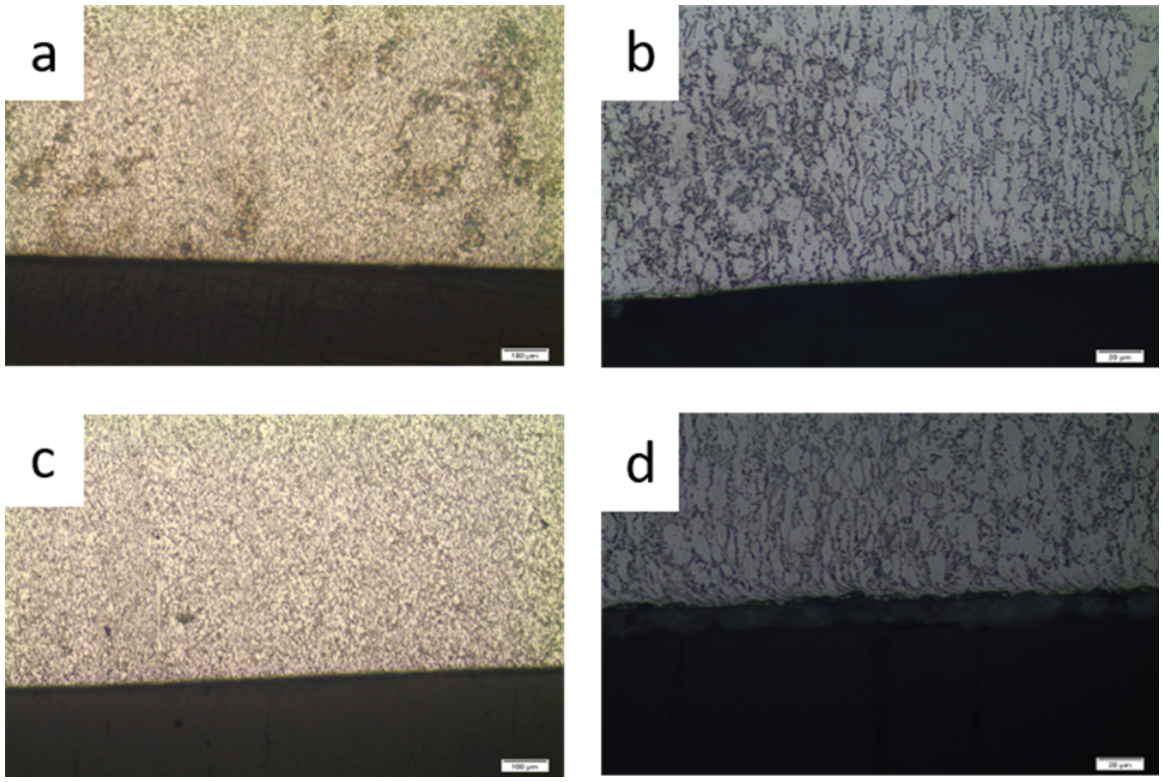

Figure 4. Cross-sections of microstructures of the samples after mechanical activation with sandpaper (grit 180) a, b after 7.5 min., c, d after 15 min., where a and c – magnitude x100, b and d – magnitude x500

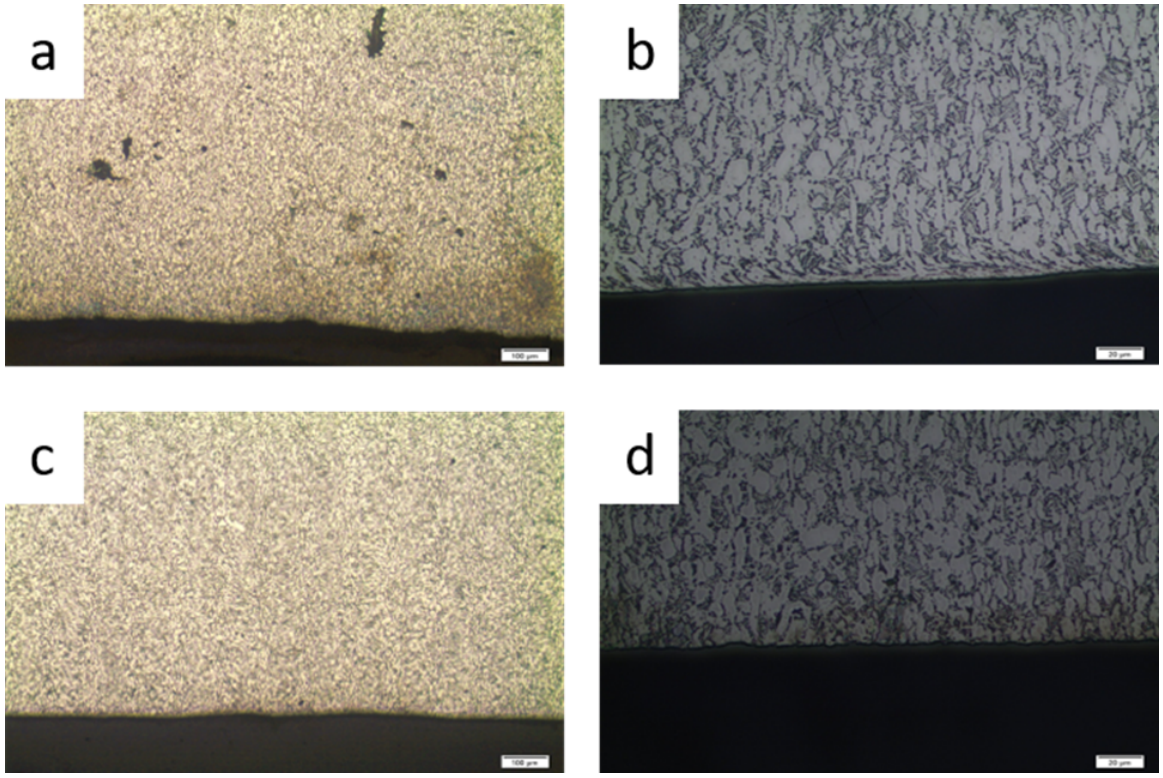

Figure 5. Cross-sections of microstructures of the samples after mechanical activation with sandpaper (grit 220) a, b after 7.5 min., c, d after 15 min., where a and c – magnitude x100, and b and d – magnitude x500

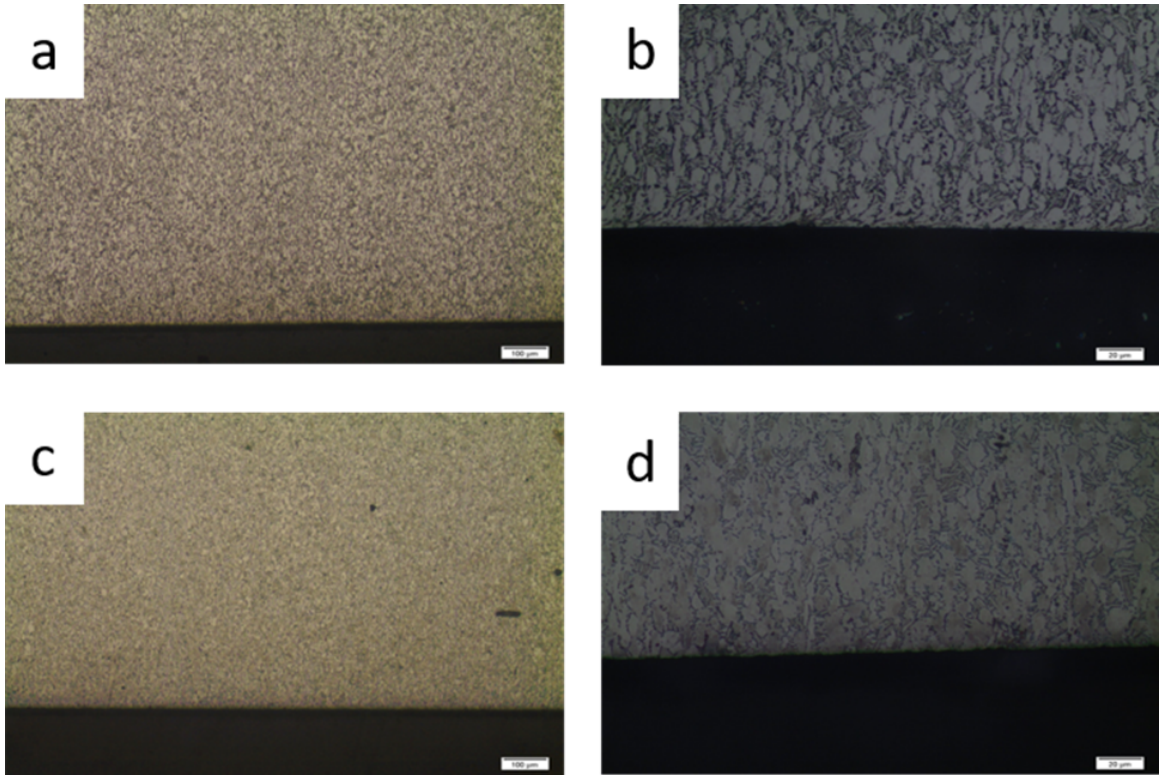

Figure 6. Cross-sections of microstructures of the samples after mechanical activation with sandpaper (grit 800) a, b after 7.5 min., c, d after 15 min., where a and c – magnitude x100, b and d – magnitude x500

Based on microstructural observations, it was found out that the largest development of the surface was reached onthe 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.

Table 2. Roughness values for the samples after 7.5 minutes of sanding and 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 grit 40	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**
Sandpaper grit 180	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**
Sandpaper grit 220	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**
Sandpaper grit 800	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**

In order to analyze the results and the differences between the samples depending on the duration of the machining, surface development studies were carried out. The values of the roughness parameters in the samples mechanically activated with sandpaper during 7.5 and 15 minutes, after sandblasting with glass balls, and the samples without mechanical activation before heat treatment are presented in Tables 2 and 3. However, the values of the roughness parameters after heat treatment are presented in Tables 4 and 5 For each parameter, three measurements were made for each sample. Then the arithmetic means (values in bold) were calculated.

Table 3. Roughness values for the samples after 15 minutes of sanding 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 grit 40	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**
Sandpaper grit 180	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**
Sandpaper grit 220	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**
Sandpaper grit 800	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 4. Roughness values for the samples after 7.5 minutes of sanding and 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 grit 40	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**
Sandpaper grit 180	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**
Sandpaper grit 220	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**
Sandpaper grit 800	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**

Table 5. Roughness values for the samples after 15 minutes of sanding and 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 grit 40	1.55	8.51	11.58	2.10
	0.93	5.75	7.95	1.33
	0.85	5.09	6.65	1.11
	1.11	**6.39**	**8.73**	**1.51**
Sandpaper grit 180	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**
Sandpaper grit 220	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**
Sandpaper grit 800	0.09	0.91	1.29	0.12
	0.10	0.81	1.13	0.13
	0.12	1.18	1.67	0.17
	0.10	**0.97**	**1.36**	**0.14**

The results obtained in the experiment are confirmed by microscopic observations, where it was rightly observed that the sample has the greatest surface development after sandblasting with glass beads. The results show a specific relationship: the surface roughness value for samples after grinding with 800, 220 and 180 grit abrasives is reduced,

there is a decrease in the Ra value after heat treatment compared to the values for the samples before treatment. Interestingly, in the case of sandpaper with the above-mentioned gradation, there is a marked reduction in the development of the surface with longer machining times, while in the case of sandpaper with the gradation of 40 the opposite is true. In the case of the tested sample, a reduction in surface development was observed after sandblasting with glass beads and heat treatment. These dependencies can be explained by the appearance of a thin layer of oxides on the surface of the tested samples, the appearance of oxides in irregularities on the surface of the samples fill them and limit the surface development.

In order to analyze the thickness of the oxide layers and the effect of mechanical treatment time, tests were carried out using a scanning microscope. The measurement results are shown in Fig. 7, while the comparison of the measurement results of the oxide layer thickness is shown in Table 6.

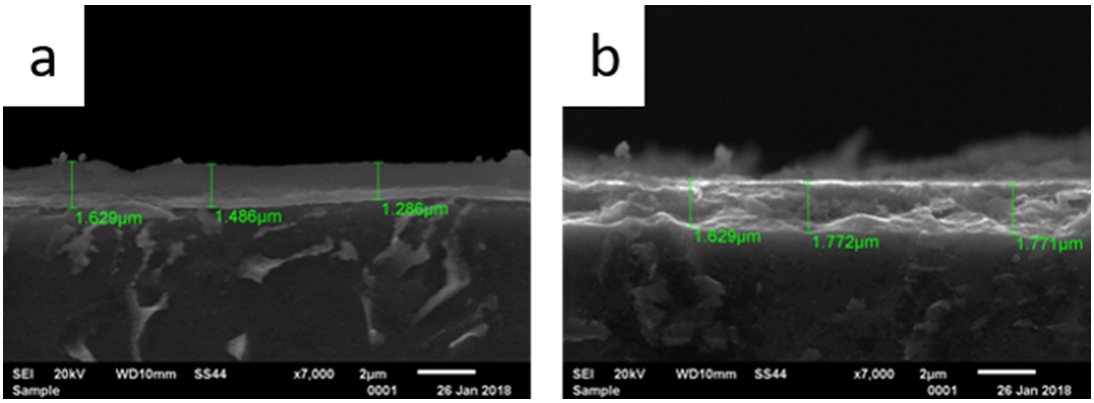

Figure 7. Example of observed values of oxide layer thickness on the sample's surfaces after mechanical activation with sandpaper (grit 40) and heat treatment (550^{o}C/5h) a) mechanical activation 7.5 min., b) mechanical activation 15 min.

Table 6. Values of oxide layer thickness after heat treatment

Mechanical treatment	Layer thickness [μm]			
	1	2	3	arithmetic mean value
Without mechanical activation	1.157	1.200	1.007	1.121
Glass beads sandblasting	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

On the basis of literature reports, it was confirmed that the results of oxide layer thickness measurements were carried out correctly.[11]. 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 sandpapergrits - 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.

4 Conclusions

The conducted experimental tests showing the effect of mechanical treatment and heat treatment of the Ti6Al4V titanium alloy showed the possibility of controlling the thickness of the oxide layer on the alloy surface. The analysis of the results shows that the thickness of the oxide layer on the alloy surface increases with increasing surface roughness. This fact can be used in the production of elements requiring increased resistance to wear or corrosion. The appearance of the ceramic TiO_2 layer slows down corrosive processes due to the lack of electrical conductivity. The tribological properties also improve due to the mechanical properties of TiO_2. However, pitting corrosion is a threat, in order to avoid it, the appropriate degree of surface development should be selected as a result of properly conducted thermal and mechanical treatment.

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