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The possibility to control the thickness of the oxide layer on the titanium Grade 2 by mechanical activation and heat treatment

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ABSTRACT

Purpose: The paper presents the results of microstructure, surface development and thickness of the oxide layer on the pure titanium Grade 2 after mechanical activation and heat treatment (550°C/5h).

Design/methodology/approach: Studies show that it is possible to control the thickness of the oxide layer by using different materials to change the roughness of surface - mechanical activation before heat treatment. After mechanical activation and heat treatment, the results of the thickness of the oxide layer as well as a level of surface development were obtained, presented and discussed.

Findings: The conducted research have proved that mechanical activation of the surface which cause increase of surface development results in greater thickness of oxide layer which is formed during heat treatment. Nevertheless mechanical activation that results in decrease of surface development, such as polishing, results in decrease of oxide layer thickness.

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

Practical implications: are possible using similar method for passivation titanium alloys for medical application.

Originality/value: The paper presents the possibility of using mechanical preactivation of surface before heat treatment passivation.

Keywords: Titanium alloys, Oxide layer, Titanium passivation, Surface mechanical activation, Heat treatment

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BIOMEDICAL AND DENTAL ENGINEERING AND MATERIALS

1. Introduction

Titanium is one of the most important metals used in modern industry [1]. Titanium and its alloys are used in many branches of the industry thanks to very good mechanical properties, very high corrosion resistance and at the same time low density [2-5]. Some of these properties are due to the ability to naturally self-passivate the titanium and production of non-conductive on its surface. However, the passive layers which appear spontaneously are very thin and therefore do not have adequate properties to be successfully used in the case of work elements – by contact or by friction, because of the poor tribological properties of titanium result mainly from a high and unstable coefficient of friction [6-8].

In order to improve these properties, it is possible to use a high affinity of titanium to oxygen and use it to produce an oxide layer of appropriate thickness, depending on the needs. One of the methods of producing such layers is mechanical activation and heat treatment. As a result of these operations, a relatively thick layer of the crystalline oxide layer appears on the surface of the workpiece [8]. The research of authors focusing on tribological issues shows that the oxide layer produced by heat treatment has ca. 4 to 6 times higher resistance to abrasion than the oxide layer which arises spontaneously on the surface of titanium [9-19].

The paper presents that obtained oxide layer with different thickness is depending on time and used material gradation for mechanical activation.

During the analysis of the current state of knowledge on the acquisition of oxide layers by heat treatment, information was obtained that the layers that are produced by heat treatment at temperatures above 800°C are relatively the thickest, however, they are very fragile and often a subject to spalling – in particular, a material which has been selected for the present studies [20-21].

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 pure titanium Grade 2 were used for the studies is in Table 1. The material was tested in the form of a rod with a diameter of 20 mm from which 3 mm thick slices were cut using band-saw. These slices were cut transversely in half and received "crescents".

Studies were conducted as follow: obtaining samples, mechanical activation, the measurement of surface development, a heat treatment, the measurement of surface development, the preparation of the cross-section of metallographic observations microstructure, the measurement of the thickness of the oxide layer by SEM.

Table 1.
Chemical composition of pure titanium Grade 2

Element	C	Fe	O	N	H	Ti
wt.%	0.005	0.05	0.19	0.04	0.0009	rest

The samples were cut out from rod, after the samples were mechanically activated using glass beads and sandpapers grits – 40, 180, 220, 800. The duration of mechanical activations was: for glass beads 5 min., for sandpaper 7.5 min. and 15 min. All these treatments were aimed at changing the energy state of the surface which stimulate the formation of oxide layers.

After mechanical activation surface topography was examined using profilometer Hommel T-1000. The prepared and studied samples under heat treatment were subjected – 550°C in time 5 h. After heat treatment surface topography was examined again.

The next step was the preparation of metallographic specimens and etching them with lactic hydride reagent – mix 5 mL lactic acid and 5 mL stock solution (3 mL HF and 97 mL HNO_3) The microstructure of the surface zone was taken using the Olympus GX41 light microscope. Magnitudes of images were x100 and x500.

The last step was SEM observation to determine the thickness of oxide layers.

3. Results

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

Microstructural observations allowed to state that the highest surface roughness is characterized by the sample subjected to sandblasting using glass beads. With this sample, the impact of structural changes can be observed up to 12 μm. The smallest development of the surface was characterized by the sample subjected to mechanical activation using sandpaper with grit 800 in 15 minutes – the impact of structural changes can be observed up to 6 μm.

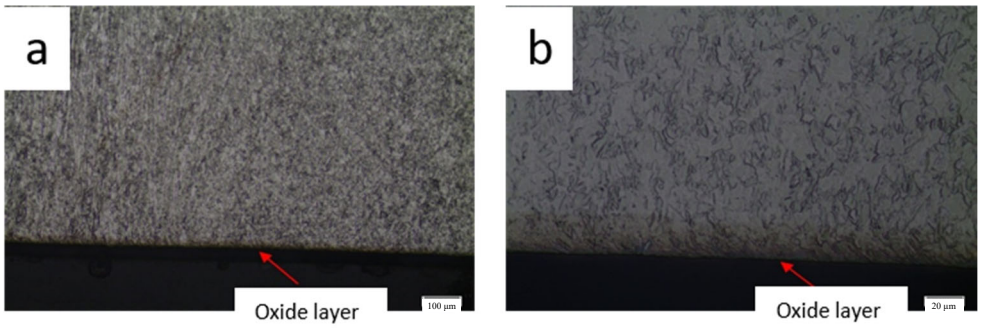

Fig. 1. Cross-sections of microstructures of the samples without mechanical activation of surface: a) magnitude x100, b) magnitude x500

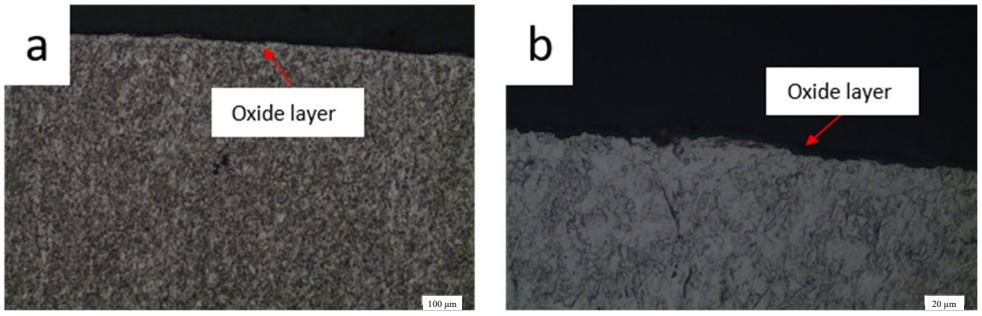

Fig. 2. Cross-sections of microstructures of the samples after sandblasting with glass beads: a) magnitude x100, b) magnitude x500

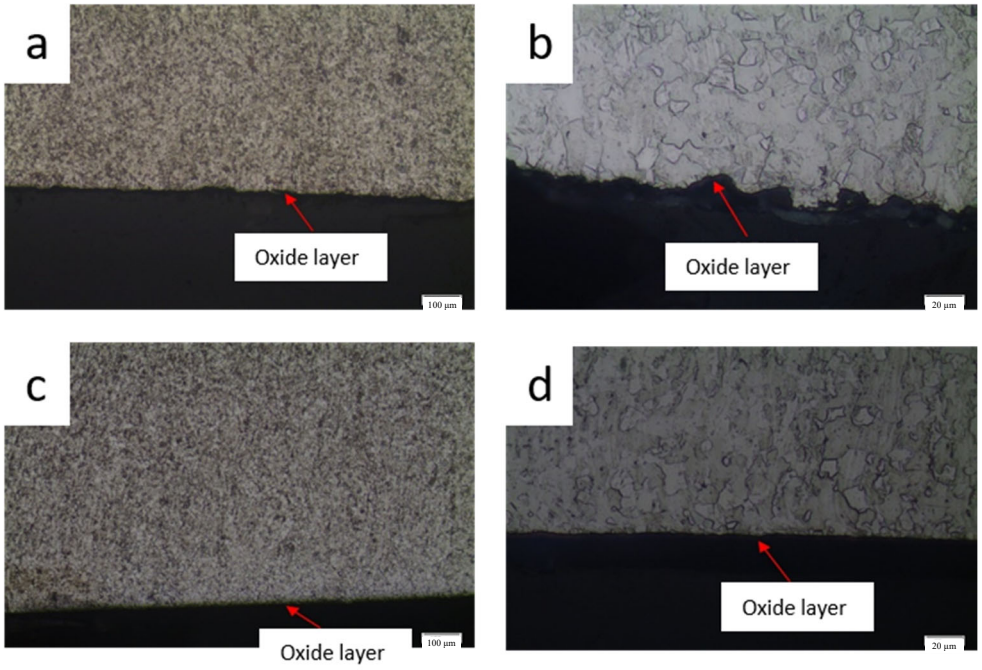

Fig. 3. Cross-sections of microstructures of the samples after mechanical activation with sandpaper (grit 40): a), b) after 7.5 min., c), d) after 15 min., where a) and c) – magnitude x100, and b) and d) – magnitude x500

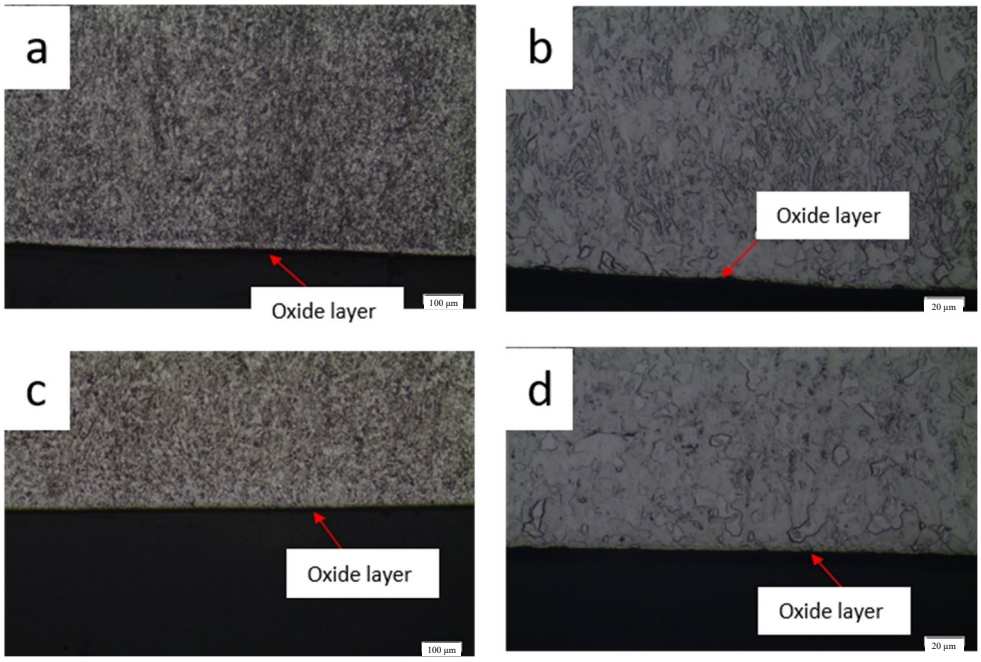

Fig. 4. Cross-sections of microstructures of the samples after mechanical activation with sandpaper (grit 180): a), b) after 7.5 min., c), d) after 15 min., where a) and c) – magnitude x100, and b) and d) – magnitude x500

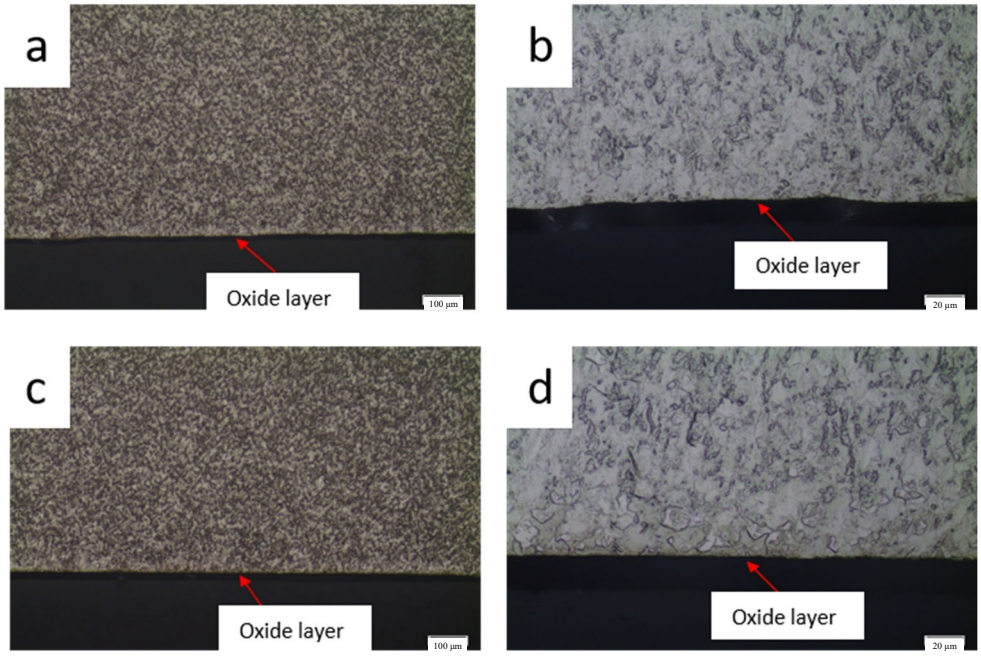

Fig. 5. Cross-sections of microstructures of the samples after mechanical activation with sandpaper (grit 220): a), b) after 7.5 min., c), d) after 15 min., where a) and c) – magnitude x100, and b) and d) – magnitude x500

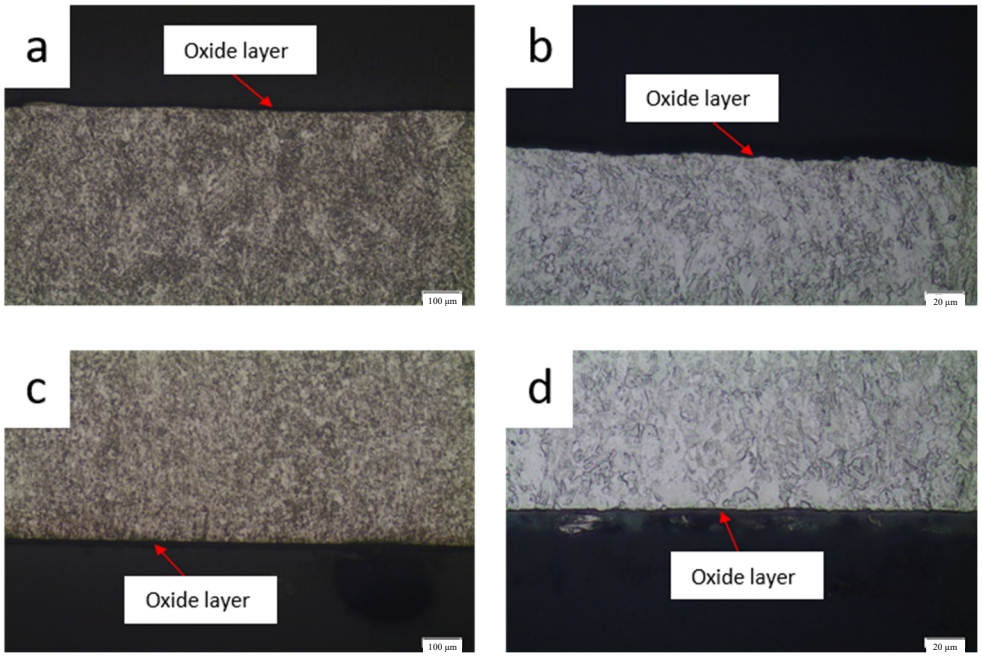

Fig. 6. Cross-sections of microstructures of the samples after mechanical activation with sandpaper (grit 800): a), b) after 7.5 min., c), d) after 15 min., where a) and c) – magnitude x100, and b) and d) – magnitude x500

In order to analyse the difference in the roughness profile – depending on the time of mechanical activation and the use of the heat treatment, surface geometry measurements were made – three measurements for each sample. The values with means are summarized in Tables 2 and 3.

Results confirm microscopic observations. The largest surface development has the sample after sandblasting with glass beads. For the samples activated by papers with grit 40, 180 the relationship where the longer time of activation has a proportional effect on the surface development can be observed. But for the samples activated by papers with grit 220 and 800 longer time of activation affects inversely proportional to surface roughness.

After heat treatment in comparison to same samples before heat treatment there is the decrease of surface development for all samples except for the sample without mechanical activation which may be the result of a lower energy state of the surface, and thus less uniform formation of oxides on the surface of the material.

Lower surface development for rest samples after heat treatment is indicated by appearing of oxides in irregularities on the surface of the samples. Oxides fill the irregularities and reduce the surface roughness.

The results are confirmed by literature reports, which indicate that oxides allow reducing the unstable coefficient of friction for titanium [6-8].

Table 2.
Roughness values for the samples before heat treatment

Sample	Roughness parameters, μm			
	R_a	R_a mean	R_z	R_z mean
Without mechanical activation	0.65	0.75	5.48	5.34
	0.65		5.16	
	0.97		5.40	
After sandblasting with glass beads	3.14	3.76	17.41	19.25
	3.64		18.19	
	4.50		22.15	
Sandpaper grit 40/7.5 min.	0.92	0.94	6.46	5.97
	0.86		5.14	
	1.04		6.32	
Sandpaper grit 40/15 min.	1.10	1.08	7.21	6.24
	1.12		5.82	
	1.02		5.71	
Sandpaper grit 180/7.5 min.	0.47	0.45	3.20	3.00
	0.60		3.94	
	0.30		1.88	
Sandpaper grit 180/15 min.	0.43	0.60	2.98	4.05
	0.70		4.55	
	0.68		4.64	
Sandpaper grit 220/7.5 min.	0.31	0.31	2.56	2.90
	0.33		2.97	
	0.31		3.18	

Sample	Roughness parameters, µm			
	R_a	R_a mean	R_z	R_z mean
Sandpaper grit 220/15 min.	0.27	0.28	2.47	2.47
	0.30		2.60	
	0.29		2.36	
Sandpaper grit 800/7.5 min.	0.18	0.19	1.31	1.48
	0.21		1.58	
	0.20		1.57	
Sandpaper grit 800/15 min.	0.15	0.15	1.12	1.21
	0.16		1.23	
	0.16		1.30	

Table 3.
Roughness values for the samples after heat treatment

Sample	Roughness parameters, µm			
	R_a	R_a mean	R_z	R_z mean
Without mechanical activation	1.27	1.24	7.02	6.91
	1.40		7.84	
	1.05		5.84	
After sandblasting with glass beads	2.58	2.07	14.23	12.41
	1.29		8.63	
	2.36		14.37	
Sandpaper grit 40/7.5 min.	0.85	0.98	6.30	7.15
	1.13		7.72	
	0.96		7.44	
Sandpaper grit 40/15 min.	1.89	1.90	10.05	10.53
	1.79		10.98	
	2.03		10.55	
Sandpaper grit 180/7.5 min.	0.55	0.66	3.60	3.92
	0.44		2.73	
	1.00		5.43	
Sandpaper grit 180/15 min.	0.72	0.62	4.89	4.51
	0.49		3.96	
	0.66		4.70	
Sandpaper grit 220/7.5 min.	0.26	0.39	2.03	3.42
	0.29		2.48	
	0.64		5.76	
Sandpaper grit 220/15 min.	0.35	0.32	3.13	2.60
	0.31		2.17	
	0.31		2.51	
Sandpaper grit 800/7.5 min.	0.16	0.17	1.44	1.45
	0.22		1.60	
	0.14		1.30	
Sandpaper grit 800/15 min.	0.11	0.09	0.81	0.84
	0.08		0.79	
	0.09		0.91	

In order to analyse the thickness of these layers and the influence of the time of mechanical activation studies were carried out using a scanning microscope. Way of measuring the thickness of the oxide layer is presented in Figure 7. Comparison of the results of oxide layer thickness measurements is presented in Table 4.

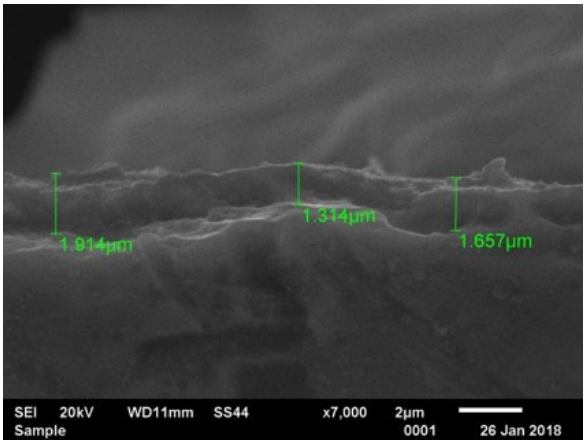

Fig. 7. Way of measuring the thickness of the oxide layer – example for the sample after mechanical activation during 15 min. with sandpaper grit 40

Table 4.
Values of oxide layer thickness after heat treatment

Sample	Measurement, µm			
	1	2	3	arithmetic mean
Without mechanical activation	1.257	1.212	1.109	1.193
After sandblasting with glass beads	3.265	3.481	3.373	3.373
Sandpaper grit 40/7.5min	1.262	1.214	1.243	1.239
Sandpaper grit 40/15min	1.914	1.314	1.657	1.628
Sandpaper grit 180/7.5min	1.293	1.296	1.312	1.300
Sandpaper grit 180/15min	1.520	1.497	1.630	1.549
Sandpaper grit 220/7.5min	1.198	1.186	1.194	1.193
Sandpaper grit 220/15min	1.133	1.128	1.099	1.120
Sandpaper grit 800/7.5min	0.890	1.002	1.024	0.972
Sandpaper grit 800/15min	0.839	0.957	1.016	0.937

The results show a dependence, for glass beads and sandpapers grit 40, 180, where the longer process of mechanical activation leads to a thicker oxide layer. On the other hand for samples after mechanical activation with sandpapers grit 220 and 800 have a thinner oxide layer after the longer process of activation in comparison to the shorter one.

On the basis of literature reports, it was confirmed that the results of oxide layer thickness measurements were carried out correctly [16].

4. Conclusions

Conducted research on the possibility of controlling the thickness of the oxide layer for pure titanium grade 2 allowed to determine the relationship between the time of mechanical activation and the surface development thus obtained, followed by heat treatment, to the thickness of the oxide layer. The results allow stating that it is possible to produce (control) the oxide layer with interesting thickness through appropriate mechanical activation, even for the same heat treatment parameters.

These dependencies can be used when machining titanium elements that must meet the appropriate tribological and corrosive requirements. Taking into account the selection of the appropriate roughness of the oxide layer, in order to avoid its chipping or the appearance of pitting corrosion in its depressions.

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