The Influence of Parameters of Heat Treatment on Thickens and Roughness of Oxide Layers on Titanium Alloy Ti6Al4V

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The paper presents the affect of temperature and time of annealing on the thickness of the obtained oxide layer on the surface of titanium alloy Ti6Al4V and also on the geometry of the layer. The studies on seven samples where they were determined from 1 to 7 were carried out. Sample 1 was a reference sample of material that was subjected to the following parameters: for the sample 2 the temperature 350 °C in 5 hours, for the samples 3 and 4 450 °C and in sequence 1 and 10 hours, for the sample 5 and 6 550 °C and in sequence 1 and 10 hours and for the sample 7 temperature 650 °C in 5 hours. Studies have shown that thicker oxide layers with less surface development form on samples occurred after longer heat treatment and higher temperatures. Thanks to these dependencies it is possible to maneuver the time and temperature of the treatment interchangeably, eg. by raising the temperature of annealing time can be shortened to obtain similar parameters of the roughness profile as for the sample processed at a lower temperature in a longer time.

Keywords: titanium alloys, heat treatment, oxide layer

Titanium and its alloys are one of the most commonly used materials in modern industry [1-7]. Thanks to a number of properties - corrosion resistance, low density, high strength, biocompatibility and the ability to osseointegrate, this material is used in industries such as aviation, chemical industry, automotive, cosmonautics and medicine. In addition, titanium and its alloys are characterized by the ability of self-passivation where on the surface of the finished element oxide layers are created possessing interesting properties in particular tribological. However, spontaneously arising oxide layers do not have the appropriate thickness and durability which translates into their quick wear [8-16].

On the basis of these premises, work began to enable improvement of the tribological properties of titanium elements through the production of oxide layers on their surfaces with appropriate thickness and durability. One of the methods of producing these layers is the heat treatment, whereby, depending on the parameters, an appropriate oxide layer is formed. The layers thus produced are characterized by up to six times greater durability in relation to the spontaneously formed layers [17-23].

As part of this publication, the influence of time and temperature of annealing on the formation of oxide layers on Ti6Al4V titanium alloy is presented.

A temperature range of 350 to 650 °C was selected for the tests however these layers also can be formed at higher temperatures. The selection of these temperatures is related to literature reports, where information appears that oxide layers produced at temperatures above 800 °C are very thin and characterized by high brittleness thus they burst during operation which is undesirable in particular in systems working under load [24-41].

Research in the publication allowed to examine the relationship between roughness, thickness of the layer and time and temperature of annealing.

Computational details

Material used in studies was two-phase titanium alloy Ti6Al4V - chemical composition is presented in table 1. The samples were modified by heat treatment in furnace in different temperatures at different times. Times and temperatures used during studies are presented in table 2. The material for the tests was taken from the rod - 4 slices which were cut into 7 *semi-circle* samples. The obtained samples were subjected to heat treatment in air atmosphere in a laboratory furnace. After heat treatment the samples were cooled in the open air.

The next stage of the research was the study of the surface topography and its parameters for this purpose the Hommel T1000 profilometer was used. Determination of surface roughness parameters consisted of contact between measuring needle with differential measuring system and the sample's surface. Three measurements were carried out on the each sample surface. Parameters that have been determined are: R_a (arithmetic mean deviation of profile ordinates from the mean line), R_z (average roughness value by 10 points), R_t (total profile height) and R_q (average square deviation of the profile from the mean line along the elementary section).

After profilometer studies transverse metallographic specimens were made which were subjected to etching with the Kroll's solution (2mL HF, 2mL HNO3, 96 mL H2O).

The last stage of the research were microscopic observations to determine the effect of heat treatment on the surface layer and the formation of oxides. Observations

Table 1
CHEMICAL COMPOSITION OF TITANIUM ALLOY Ti6Al4V USED DURING STUDIES

Element	Al	V	C	Fe	O	N	H	Ti
wt. [%]	6.00	4.00	0.03	0.1	0.15	0.01	0.003	rest

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Table 2
TIMES AND TEMPERATURES USED DURING STUDIES

Sample no.	Temperature of heat treatment	Time of heat treatment
1	Sample without heat treatment	
2	350°C	5h
3	450°C	1h
4	450°C	10h
5	550°C	1h
6	550°C	10h
7	650°C	5h

were carried out on two microscopes - light microscope Olympus GX41 with magnitudes x100 and x500 and SEM Jeol JSM 6610LC with EDS analyser.

Results and discussions

Based on microscopic observations it was found that the largest development of the surface has the initial sample because of . These observations are confirmed by literature reports regarding the times and temperature of annealing [42]. Observations with light microscope allowed to reveal within the surface the deformation of grains - it is result of method of obtaining samples - cutting with a circural saw (fig. 3b, 5b, 6b, 7b). These changes were observed at the depth 3-10µm from the surface.

In order to confirm the microscopic observations regarding the surface development of the studied materials the geometric profiles of all samples were analyzed. Three measurements were taken for each of the sample determining the R_a and R_z parameters the results were averaged and summarized in the table (Table 3). The results confirmed microscopic observations indicating that longer heat treatment contributes to the decrease in surface development which may result from the accumulation of oxides in unevenness on the surface of the material. The results are confirmed by literature reports which indicate that oxides allow to reduce the unstable coefficient of friction for titanium what means lower roughness [43].

The microscope observations were confirmed by the surface profile research where it was found that with the increase of the oxidation time the surface roughness decreases which is particularly visible for the samples 3, 4, 5 and 6. Moreover higher temperature of heat treatment reduces roughness even for shorter time of annealing. This relationship can be seen by comparing the samples 6 and 7 where both samples have similar surface development level (R_a parameter) but time treatment of the sample 7 was shorter by half in comparison to the sample 6.

As it was mentioned above thicker oxide layer reduce its roughness. In order to confirm this supposition, measurements of the oxide layer thickness were made using scanning electron microscopy. An exemplary measurement is shown in figure 8. Layer thickness results are summarized in table 4.

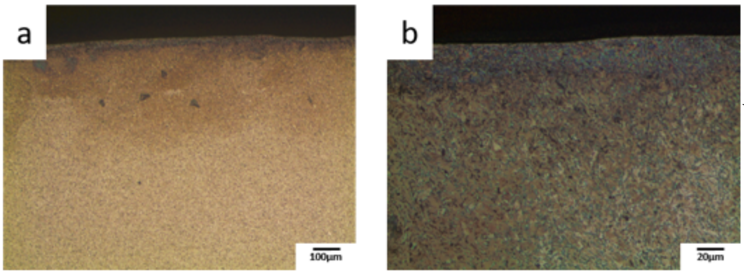

Fig. 1. Cross-sections of microstructures of the sample without mechanical activation of surface a) magnitude x100, b) magnitude x500 (sample 1).

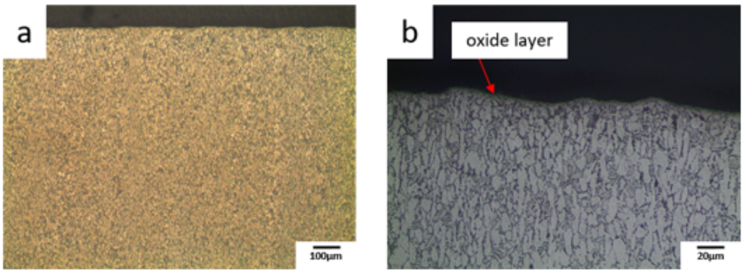

Fig. 2. Cross-sections of microstructures of the sample after heat treatment 5h 350°C a) magnitude x100, b) magnitude x500 (sample 2).

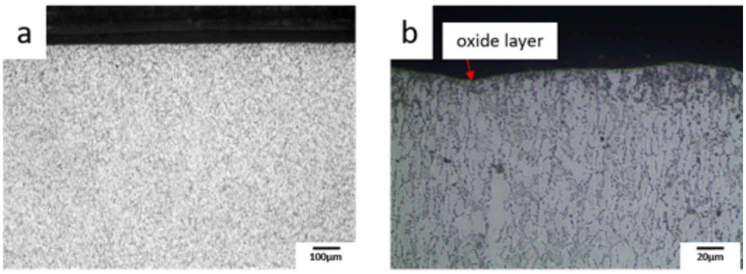

Fig. 3. Cross-sections of microstructures of the sample after heat treatment 1h 450°C a) magnitude x100, b) magnitude x500 (sample 3).

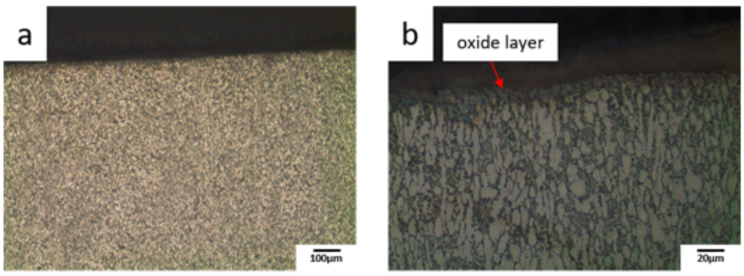

Fig. 4. Cross-sections of microstructures of the sample after heat treatment 10h 450°C a) magnitude x100, b) magnitude x500 (sample 4).

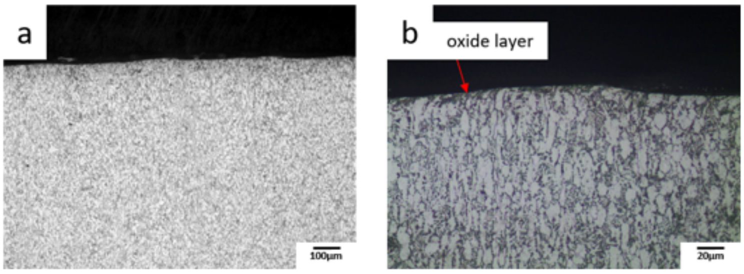

Fig. 5. Cross-sections of microstructures of the sample after heat treatment 1h 550°C a) magnitude x100, b) magnitude x500 (sample 5).

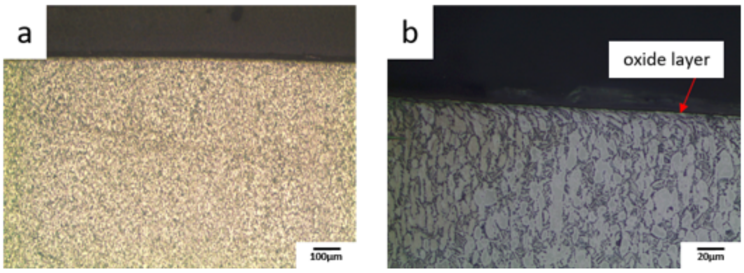

Fig. 6. Cross-sections of microstructures of the sample after heat treatment 10h 550°C a) magnitude x100, b) magnitude x500 (sample 6).

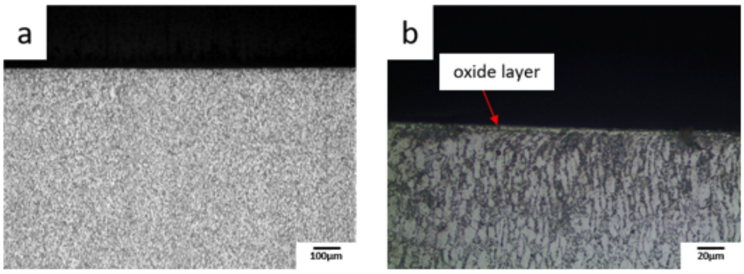

Fig. 7. Cross-sections of microstructures of the sample after heat treatment 5h 650°C a) magnitude x100, b) magnitude x500 (sample 7).

Table 3
RESULTS OF ROUGHNESS PROFILES

Sample	Parameters [µm]					
	R_a	R_a (avg.)	R_z	R_z (avg.)	R_t	R_q
1	3.58	**3.84**	15.43	**15.53**	18.06	4.29
	3.44		13.70		16.74	4.04
	4.49		17.45		19.26	5.09
2	2.36	**2.16**	13.45	**11.98**	16.54	2.87
	2.16		11.77		15.89	2.66
	1.98		10.73		13.98	2.43
3	2.07	**2.15**	9.58	**9.91**	12.53	2.51
	2.08		9.69		11.40	2.50
	2.29		10.47		13.29	2.83
4	1.67	**1.49**	7.43	**6.61**	9.77	2.06
	1.69		6.86		8.02	2.01
	1.12		5.54		7.92	1.44
5	0.48	**0.76**	2.23	**3.62**	3.08	0.60
	1.05		4.75		8.76	1.32
	0.76		3.87		5.68	1.00
6	0.45	**0.53**	1.80	**2.65**	2.60	0.55
	0.44		2.48		4.04	0.58
	0.71		3.67		4.70	0.89
7	0.56	**0.58**	3.37	**3.75**	5.79	0.77
	0.63		3.93		8.47	0.88
	0.54		3.96		6.49	0.75

On the basis of literature reports, it was confirmed that the results of oxide layer thickness measurements were carried out correctly [43].

On the basis of observations with scanning electron microscope the predicted dependence was confirmed that along with a higher temperature and longer heating the oxide layer increases in thickness. At the same time on the basis of the surface geometry studies it can be seen the dependence that the thicker oxide layer has lower roughness profile. The deviation from this observation is

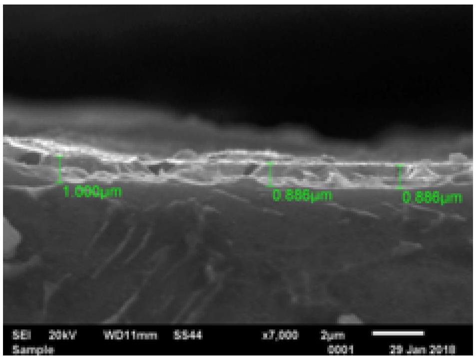

Fig. 8. Way of measuring thickness of the oxide layer - example for the sample after heat treatment 5h 350°C.

the sample 5 which has thinner oxide layer in comparison to sample 4 but has lower profile development. Which may lead to the conclusion that temperature plays a more important role in the formation of oxide films than time.

Table 4

VALUES OF OXIDE LAYERS THICKNESS AFTER HEAT TREATMENT

Sample	Measurement [µm]			
	1	2	3	arithmetic mean
1	0.120	0.190	0.100	0.137
2	1.000	0.888	0.888	0.925
3	0.980	1.100	1.090	1.056
4	1.530	1.610	1.590	1.577
5	1.180	1.210	1.190	1.933
6	1.920	1.880	1.960	1.920
7	1.810	1.790	1.840	1.813

Conclusions

On the basis of the conducted research regarding the possibilities of oxide layers formation by heat treatment including the change of their thickness and roughness profile depending on the chosen parameters - time and temperature some dependences between time and thickness of the oxide layer were observed. Interestingly in the case of the tested samples the temperature played a greater role in the formation of a less rough layer than time as seen in the samples 5 and 4, where comparing layer thickness and surface development clearly shows that sample 5 despite much shorter time and due to higher temperature has a lower roughness.

Thanks to these observations it can be concluded that it is possible to predict how a thick oxide layer will be obtained after heat treatment for a given material. By summarizing the results it is possible to select the annealing parameters - temperature and time to obtain layers with the interested surface geometry and thickness which can be very important in particular for process design for elements requiring very high dimensional accuracy after oxidation. In other hand the lower time and temperature allow to obtain oxide layer with higher surface development what is important for implants due to better osseointegration.

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Manuscript received: 21.01.2018