ADHESION OF THE BIOACTIVE LAYER ON TITANIUM ALLOY SUBSTRATE BY TAPE-TEST

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The aim of this work was to check whether the ASTM D 3359 is a suitable method for adhesion measurement of the layers formed on Ti6Al4V surfaces by a) alkali treatment in 10 mol/l NaOH, b) precalcification in SCS (supersaturated calcification solution) and c) in vitro tests in SBF (simulated body fluid). Tape-test is suitable for evaluation of adhesion of coatings after chemical treatment and after short-time precalcification. Adhesion of the coatings is excellent, classified into category 5. The test is not suitable for coatings precalcified for the long time when the big spherical crystals are formed. The cracking of the coatings during drying of samples inhibits obtaining reliable results of adhesion of HA-coatings to substrate after in vitro tests.

INTRODUCTION

Titanium (Ti) and some of its alloys have been used as orthopedic and dental implant materials. Various methods were applied to improve the chemical activity of the surfaces, which is important for direct bonding of implant - living tissue [1,2].

Chemical and thermal treatments seem to be useful for the surface treatment of titanium and its alloys. Chemical activity of the surface of titanium materials is improved by treatment with NaOH solution (5-10 mol/l, 60°C, 24 hours) [2]. The gel-like layer of $TiO_x{\cdot}nH_2O$ with residual Na^+ formed on surface provides good conditions for setting of Ca-P precipitates nucleation and subsequent growing of HA (hydroxyapatite) in solutions. The bioactivity of metal surfaces and the growth of the hydroxyapatite layer are tested in vitro in simulated body fluid (SBF) [3-6]. The surface treated by alkali solution is able to develop a uniform apatite coating in a few days. The process of nucleation is accelerated by precalcification, e.g. by exposition of chemically treated samples in calcification solution with high content of Ca^{2+} and $(PO_4)^{3-}$ ions [3-5].

Implants based on Ti are suitable for mechanically strained positions. Besides of the biocompatibility of the coated implants, the stability and adhesion of the coating at tensile and pressure strain are very important. For this reason, it is necessary to know the behavior of interlayers: substrate–corrosive layer–hydroxyapatite coating. Standard test methods like the tape test, scratch test etc. were developed for evaluating of stability and adhesion of coatings of a thickness from several hundred nanometers to several ten micrometers [7,8]. Only some of the works studying the effect of surface treatments and coatings on Ti and its alloys deal with adhesion of the coatings and the layers and if, then only marginally [9,10].

The present work focuses on evaluation of adhesion of surface layer to ground and sandblasted Ti6Al4V substrates formed after alkali treatment, and layer formed after exposure to supersaturated calcification solution (SCS). Adhesion of the surface layers was tested by ASTM D 3359-02: Standard Test Methods for Measuring Adhesion by Tape; cross-cut tape test (B) [7].

EXPERIMENTAL

The samples of titanium alloy Ti6Al4V (90:6:4 wt. %) with the dimension of 10×10×0.8 mm were ground and sandblasted. One group of Ti6Al4V samples was ground on both sides with SiC paper No. 500, the other group of Ti6Al4V samples was sandblasted by electrocorrundum (∅ 125 μm, pressure = 0.4 MPa) for 3-5 s. The samples were ultrasonically cleaned with acetone and demineralized water for 10 min. Subsequently the samples were etched in concentrated hydrochloric acid at $t = 20°C$ for 90 min and then 3-times washed in demineralized water.

Samples of titanium substrate (Ti - 99.9 wt.%) treated by the same way as a titanium alloy were used as a parallel material in this experiment.

The cleaned samples were chemically treated in concentrated solution of 10 mol/l NaOH at 60°C for 24 hours and then washed in demineralized water. Several Ti6Al4V samples were dried after NaOH treatment and cross-cut tape-test (B) was applied according to ASTM D 3359-02.

The rest of Ti6Al4V samples was precalcified, e.g. after washing in water the samples were exposed to SCS solution of high concentration of Ca^{2+} and PO_4^{3-} ions (Table 1) [2,5]. The samples were soaked in SCS solution in biological thermostat at $t = 37°C$ for 2 hours and for 3 days at $S/V = 0.1$ cm^{-1} (S = sample surface, V = volume of soaking solution). After soaking in SCS, the samples were 3-times washed in demineralized water. Several Ti6Al4V calcified samples were dried and the cross cut tape-test (B) was applied.

After washing in demineralized water, the remaining samples were exposed to simulated body fluid (SBF). The concentration of ions in SBF is shown in Table 1. It is an in vitro examination of bioactivity of the surface, e.g. the ability of the substrate to form a uniform hydroxyapatite layer on its surface. The analysis of Na^+, K^+, Ca^{2+}, Mg^{2+} ions in SBF and SCS solution were performed by atomic absorption spectroscopy (AAS, fy. Elmer Perkin 3100).

The quality of the surfaces of samples and coatings was evaluated after each step of treatment. The changes of relief, morphology of the surface and crystallic products on the surface were evaluated. The chemical composition of the coatings was observed and the adhesion of the coatings to substrates was measured.

The microgeometric surface parameters of Ti6Al4V after mechanical and chemical treatments were measured by HOMMEL TESTER T1000. Following rough surface parameters were evaluated: i) Ra - arithmetic average of absolute value $Z(x)$ within the interval of the measured length l and ii) Rt - sum of height Zp the highest ledge of the profile and depth Zv the lowest cavity of profile within l [11]. The tip of the profilometer moved in a length of 1500 μm.

$$Ra = \frac{1}{l}\int_0^l \left|Z(x)\right| dx$$

where $Z(x)$ - height of the reviewed profile in arbitrary position x.

The adhesion of the coating to the substrate was measured by ASTM D 3359 cross-cut tape-test (B) [7]. Grid of 6×6 parallel cuts with 1mm gaps between them was made through the coating down to the substrate. The 3M Brand Scotch tape was firmly placed in the area of the grid and after 90 s torn abruptly off at an angle as close to 180° as possible. Damage of the coating was evaluated visually by optical microscope (Jenapol with NIS Elements software) with side illumination. According to the size of the removed area expressed in per cent the samples were divided into the following classification scale: 5 = 0 %; 4 = less than 5 %; 3 = 5-15 %; 2 = 15-35 %; 1 = 35-65 %; 0 = over 65 %. The locality of coating damage was observed in detail by SEM-EDS (JEOL JSM-7000 F).

RESULTS AND DISCUSSION

Surface characterization of Ti6Al4V samples

The grinding and sandblasting was used for mechanical removal of passivating TiO_2 layer and impurities from the surfaces of Ti6Al4V samples. Sandblasting of the samples leads to more considerable surface roughness than grinding as it is shown in figures of Ti6Al4V from optical microscope (Figures 1a, 2a). The figures of mechanically treated samples before and after etching in concentrated hydrochloric acid indicate aggression of acid to Ti6Al4V (Figures 1b, 2b). Several of these samples were etched in solution of sodium fluoride and orthophosphoric acid (0.135 mol/l NaF + 0.5 mol/l H_3PO_4) for 2 min which dissolves the alloy. The results of chemical analyses confirm that the effect of concentrated HCl does not result in selective leach out of single metals from Ti6Al4V. The composition of the sample after etching in concentrated HCl and after cleaning in solution of NaF + H_3PO_4 was identical.

Table 1. The ion concentration and pH of the SCS, SBF solutions and human plasma [2-5].

	ion concentration (mmol/l)									
solution	Na^+	K^+	Ca^{2+}	Mg^{2+}	Cl^-	HCO_3^-	SO_4^{2-}	HPO_4^{2-}	$H_2PO_4^-$	pH
SCS	4	-	5	-	10	1.5	-	-	2.5	6.25
SBF	142	5	2.5	1.5	148	4.2	0	1	-	7.45
Plasma	142	5	2.5	1.5	103	27	0.5	1	-	7.4

The samples treated by HCl were soaked in concentrated NaOH solution to achieve the improvement of surface activity of substrates. A slow corrosion of Ti takes place during soaking in NaOH solution. A uniform hydrated gel-like TiO_2 layer forms on the surface. The effect of aggressive alkali solution on TiO_2 leads to formation of negatively charged hydrates $HTiO_3^-$, which can be saturated by Na^+ or H_3O^+ cation. Figures 1c and 2c show the changes of the Ti6Al4V surface before and after chemical treatment in NaOH solution, the roughening of the surface. At visual observing the surfaces of the samples after exposure to NaOH solution are white, light grey, without metallic luster.

The recorded profiles of surfaces show more considerable changes with sandblasted samples due to chemical treatment (Figure 3a-c). The microgeometric parameters *Ra* and *Rt* are in Table 2.

The records of profiles (Figure 3) show the changes caused by mechanical treatment and aggressive chemical agents. The Ra values of the sandblasted samples are higher than the values of the ground samples. These values grow with the number of subsequent chemical treatments. Sandblasting has a much stronger roughening effect on the sample surface than grinding: makes deeper notches and higher ledges. Etching in HCl leads to deepening of the profiles. The surface is covered by gel-like Na -$TiO_x{\cdot}nH_2O$ layer after soaking in concentrated NaOH solution which is not thicker than a few micrometers. The most considerable changes (deepening and enlargement of ledges) were achieved with the sandblasted sample after treatment in NaOH solution (Figure 3c).

Adhesion of corroded bioactive layers to Ti6Al4V after treatment in NaOH solution

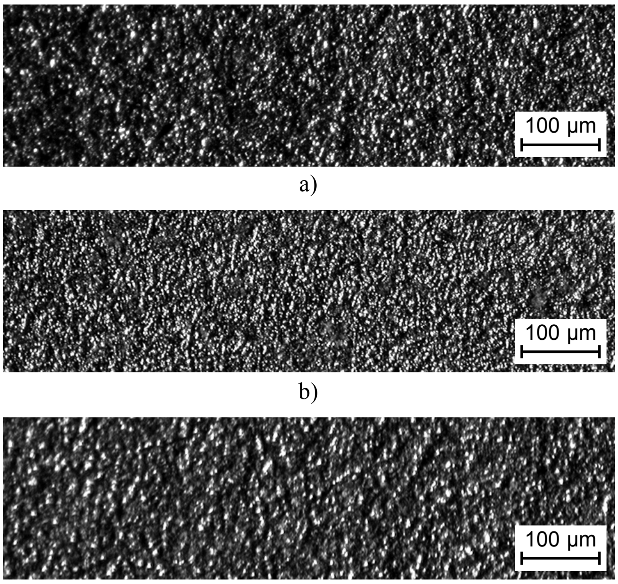
The corrosion of the substrates in concentrated NaOH solution considerably changes their surface (Figure 4, Table 3).

The gel-like titanate layer with residual sodium ions uniformly covers the whole surface area (Figure 4), making a white layer. The porous structure which can be seen under a higher magnification (Figure 4b, in detail). The gel-like layer cracks during drying and it makes a network of cracks. The thickness of the corroded bioactive layer was estimated to be of 1-3 μm.

The quality of the chemically activated surface of Ti6Al4V samples, e.g. gel-like titanate with residual sodium ions is the most important factor for the fast formation of apatite. The results of EDS analyses (Table 3) demonstrate a relatively high concentration of Na^+ ions in the samples´ surface area. The analysis of convex notches confirms increased concentrations of Na^+ and O^{2-} ions. The presence of Na^+ ions in the surface area has a high influence on precipitation of Ca-P compounds at surface of the substrate during the nucleation phase.

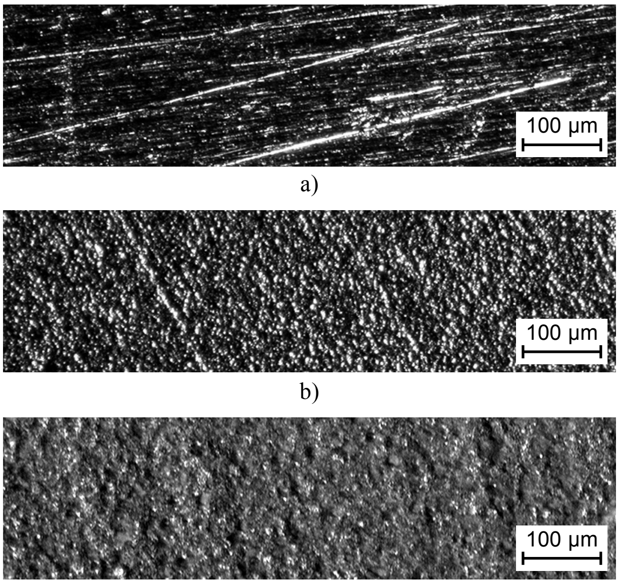
The results of ASTM D 3359 cross-cut tape-test (B): the adhesion of dried gel-like Na-$TiO_x{\cdot}nH_2O$ layers on all type of samples was good and was classified to category 5. The sandblasted and ground titanium alloy surface treated in NaOH solution after application of tape-test is shown in Figure 5. The dark areas in SEM are spots covered by glue, which remains after the test.

The removal of the tape from the ground samples was more difficult than from sandblasted samples.



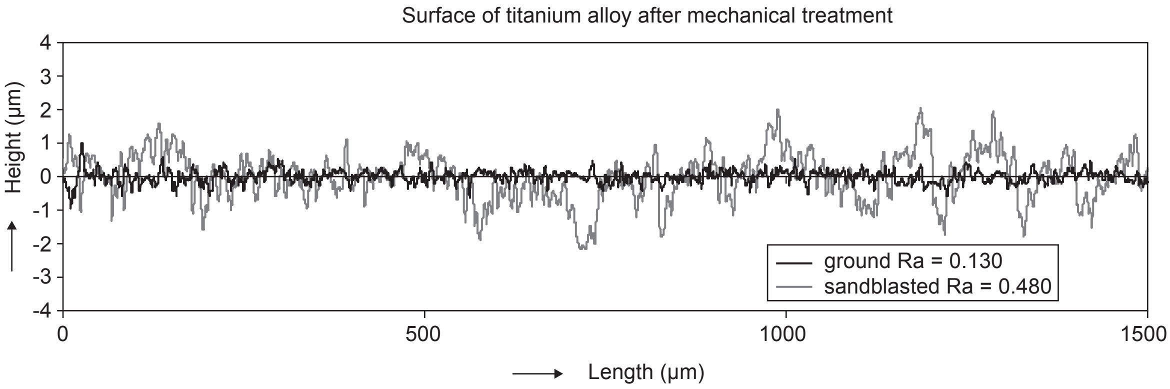
a)
b)
c)

Figure 1. Surface of Ti6Al4V; a) after sandblasting, b) after sandblasting and etching in HCl, c) after sandblasting, leaching in HCl and exposition in NaOH.

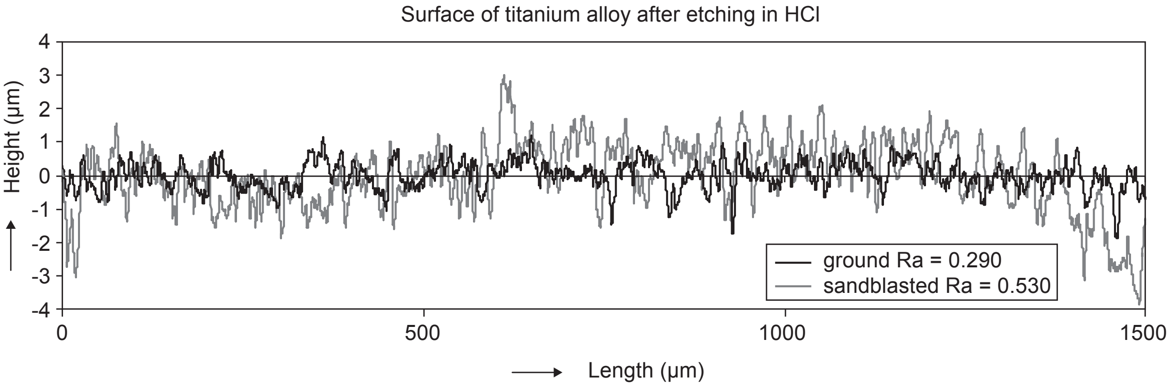


a)
b)
c)

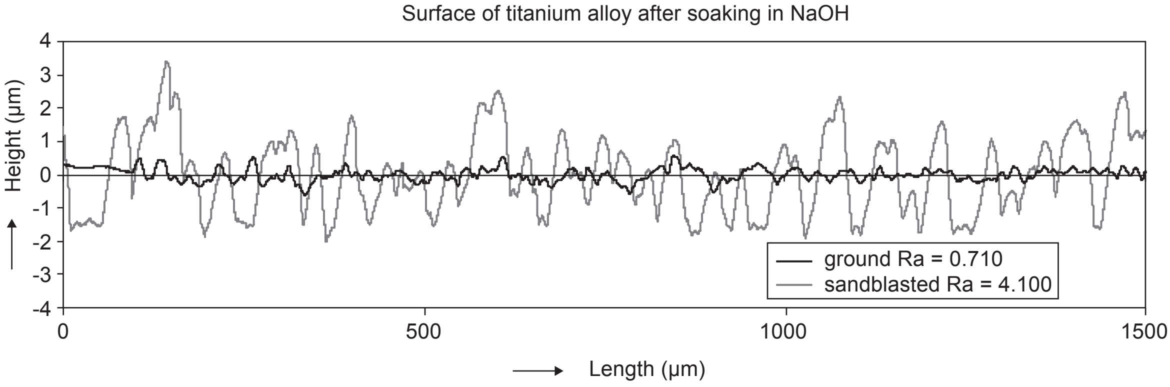
Figure 2. Surface of Ti6Al4V; a) after grinding, b) after grinding and etching in HCl, c) after grinding, leaching in HCl and exposition in NaOH.



a)



b)



c)

Figure 3. Profile comparison of the ground and sandblasted Ti6Al4V samples: a) surface after mechanical treatment; b) surface after etching in HCl; c) surface after soaking in NaOH.

Table 2. Parameters of the surface roughness *Ra* and *Rt* of ground and sandblasted Ti6Al4V samples after chemical treatment in HCl and NaOH solutions.

reagent	*Ra*		*Ra*	*Rt*	
	ground	sandblasted	*Ra*-sandblasted/*Ra*-ground	ground	sandblasted
surface after mech. treatment	0.130	0.480	3.7	1.100	3.720
surface after HCl	0.290	0.530	1.8	2.670	4.340
surface after NaOH	0.710	4.100	5.8	5.670	22.32

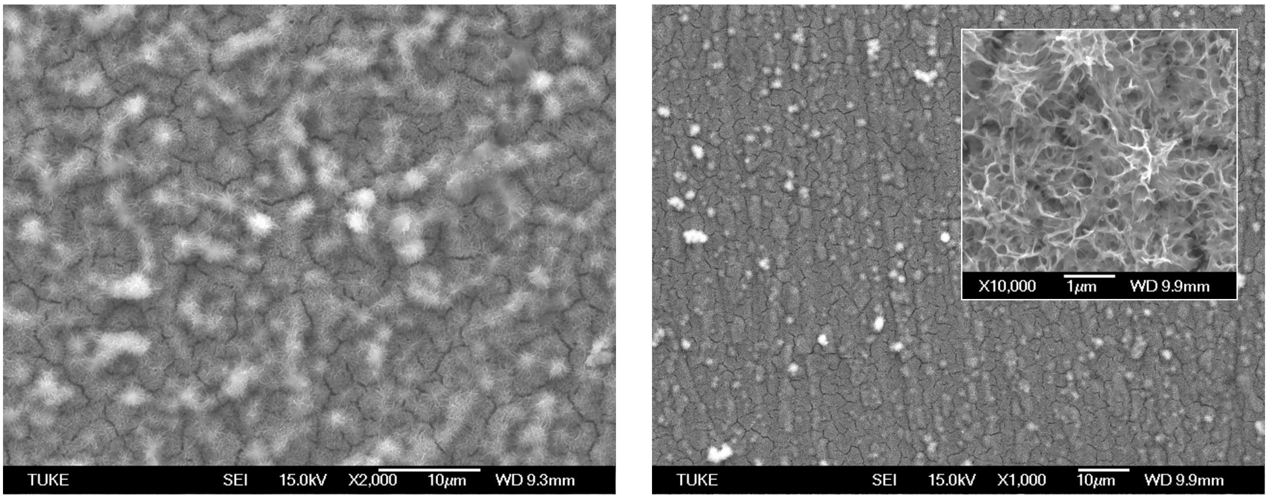
The glue from the tape remained on numerous areas of the coating. In case of sandblasted samples, the width of cut was bigger after removing of the tape. It can be caused by the marked roughness of the surface. The roughness of the sandblasted samples probably causes unequal pressure during the tape-test. The tape-test results applied on ground and sandblasted samples with corrosive bioactive Na-$TiO_x \cdot nH_2O$ layer are comparable, classified to category 5.

Adhesion of the coatings on Ti6Al4V after calcification

Samples of Ti6Al4V treated by NaOH solution were exposed in SCS for a time from 0.5 hours up to 3 days. Higher concentration of Ca^{2+} and PO_4^{3-} ions in SCS in comparison with SBF (Table 1) creates good conditions for a faster heterogeneous nucleation of Ca-P compounds and provides a uniform covering of the exposed surface by numerous nuclei. The relatively

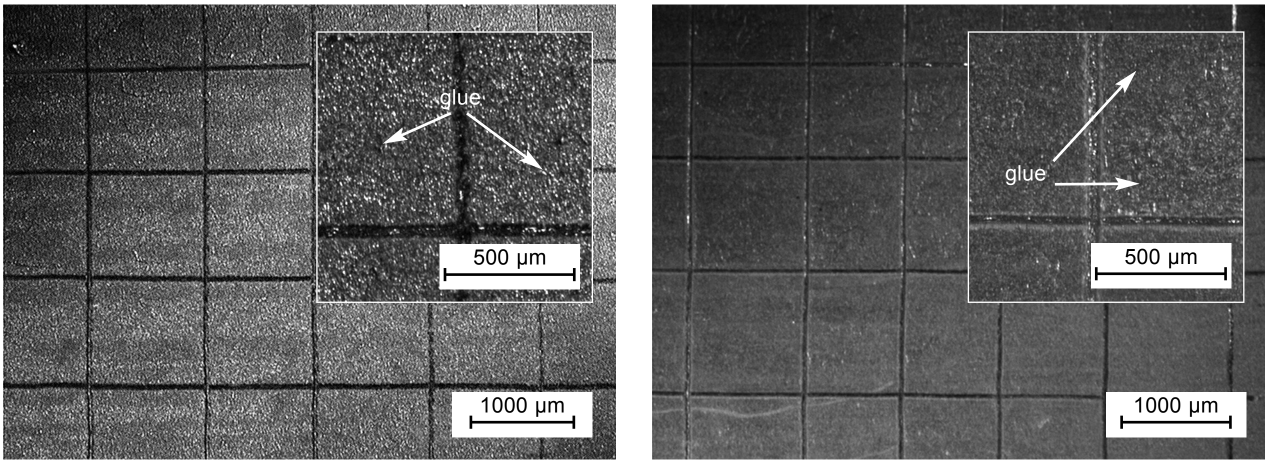
Table 3. EDS analyses of the surface of Ti6Al4V after exposure to NaOH solution.

sample	element (at %)					
	Ti	Al	V	O	Na	Ca
Ti6Al4V - ground	32.6	2.0	0.8	58.1	5.7	0.6
Ti6Al4V - sandblasted	30.5	1.2	0.6	59.1	7.7	0.9



a) b)

Figure 4. (SEM) Surface of Ti6Al4V after exposure to NaOH solution: a) sandblasted, b) ground.



a) b)

Figure 5. (OM) Surface of Ti6Al4V sample treated in NaOH solution after adhesive measurement of gel-like titanate layer: a) sandblasted, b) ground.

high concentration of PO_4^{3-} and the increase of *pH* in the solution locally accelerate the process of precipitation. The nuclei are preferentially formed in the energetically most favorable places, i.e. in the convex notches, where the sodium ion concentration is high.

The results of EDS analyses positively demonstrate that the exchange of Na^+ to Ca^{2+} takes place immediately. Forming of the small crystals, densely covering the surface can already be seen after 2 hours of exposition. The surfaces of ground and sandblasted Ti6Al4V samples treated by NaOH solution got completely covered by needle-like crystals of Ca-P compounds in the course of 3 days of exposure to SCS. A uniform coating of small crystals densely forming the spherulites was created on the surface of ground Ti6Al4V samples. The Ca/P molar ratio in the crystal spherulites is 1.3-1.1 (EDS analysis of spherulite is 16/13.5 at %). The thickness of the coating is in the range of 20-50 µm.

The established good adhesion of $Na\text{-}TiO_x{\cdot}nH_2O$ layer of alkali treated Ti6Al4V samples to the surface gives assumption that the adhesion of $Ca\text{-}TiO_x{\cdot}nH_2O$ layer formed during exposure to SCS would not get worse. The results of adhesivity measurement with both substrates were identical. The adhesion of the coatings formed in the course of 2 hours in SCS measured by tape-test was equal to the adhesion of the corrosive layers formed in alkali solution. The adhesion was very good and was classified into category 4 with all samples. In some cases the glue remained on samples surfaces. The damage of 2-hour and 3-day SCS coatings formed after the tape-test are shown in Figure 6. The surface of the samples where the duration of exposure was short was almost without damage. The big spherulites of Ca-P crystals formed during the 3-day exposure were widely damaged after the tape-test. The upper layer of the big crystals remained on the tape and the lower layer remained on the substrate. The sandblasted samples were covered by bigger spherulites therefore the damage to these samples was greater.

If the adhesion of the basic layer is higher than the strength of the formed crystals, then it is very difficult to evaluate the adhesion of the coating. The test is not suitable for the coatings calcified for more than one day. The adhesion test of the corroded bioactive layer on the surfaces of Ti6Al4V samples mentioned earlier is a significantly better means to collect information on adhesion of coating.

Adhesion of HA- coatings on Ti6Al4V after *in vitro* test

The bioactivity of ground and sandblasted chemically treated Ti6Al4V samples calcified by SCS was investigated in SBF during 7 days.

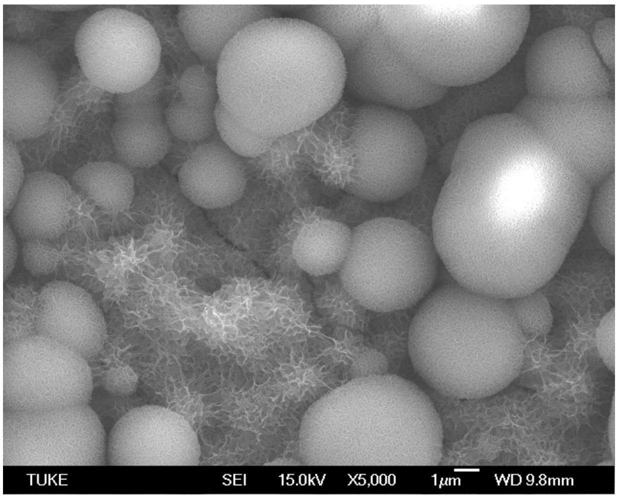
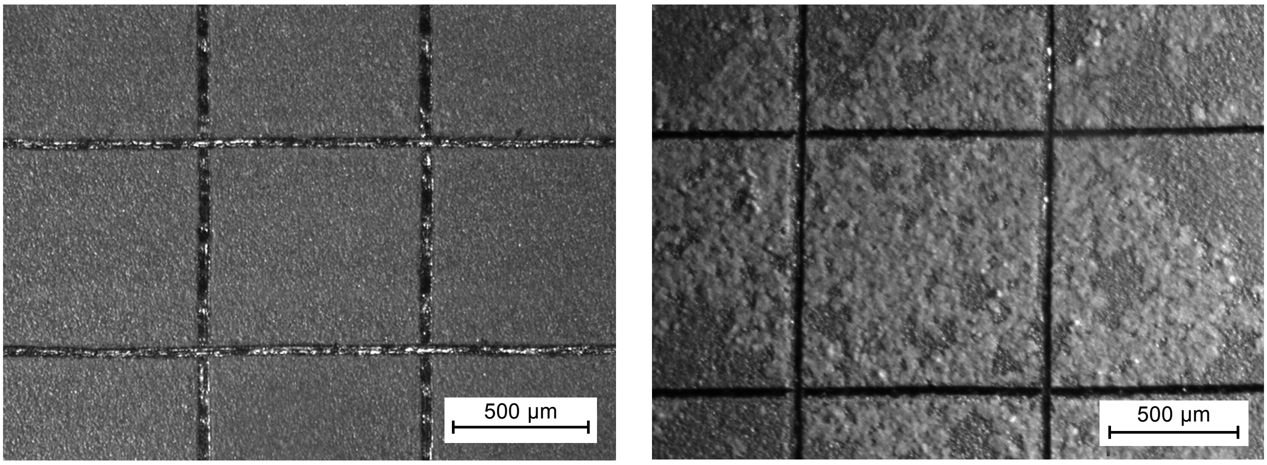


Figure 7. (SEM) Growth of HA-coating on surface of Ti6Al4V during exposure to SBF, (surface pretreated in NaOH + 1 day in SCS).



a)

b)

Figure 6. (OM) Surface of Ti6Al4V samples after tape-test: a) 2 hours in SCS b) 3 days in SCS (white area - defective coating on the surface).

The HA-coating did not form on the surfaces without previous treatment in NaOH solution. The treatment in NaOH solution is necessary because it allows a faster covering of Ti6Al4V substrates by HA-coating. The forming of HA-coating on the sample calcified for 1 day is shown in Figure 7. The rosette-like intergrown apatite nanocrystals precipitated from SBF have a globular habitus. A significantly thicker and broken-up HA-coating was formed on samples previously calcified for 2 hours in comparison with the non-calcified sample. The spherulites of needle-like crystals formed in SCS were transformed into fine rosette-like microcrystals during the 7 days of exposure to SBF. The coating shrinks during drying. Tension is generated in the coating which is compensated by cracking of the coating. In case of the thicker coatings the cracks were more considerable. It results in decreasing of adhesion of the HA-coating to the substrate, as suggested by the tape-test.

The surfaces of Ti6Al4V samples after tape-test are shown in Figures 8a-c (SEM and OM).

Classification results of adhesion of the coatings evaluated by tape-test are summarized in Table 4.

Samples without precalcification showed a wider damage along the cuts after tape-test. The range of damage of the coating with ground and sandblasted Ti6Al4V was samples comparable. Samples without calcification were classified to category 2, those with short-time calcification (2 hours) to category 3 and the samples with long-time calcification (3 days) were classified to category 2. The coating on the samples calcified for 2 hrs cracked less than the coatings of others samples. During the tape-test the damage occurred only along cuts of coatings calcified for 2 hours and the glue from the tape remained on the coating. In the case of 3 days-calcified samples the coating showed damages of the surface as well. After "*in vitro*" test the coating of samples calcified for 3-days is thicker and for that reason the damage of the coating is larger.

The results of tape-test indicate that the adhesion became worse with increasing of thickness of the coatings. The cracking is attributable to development of tensile strength in the shrinking coating during its dehydratation. The thickness of coating influences the extent of the cracking. The tension on the interface of substrate - HA-coating is higher. It is possible that the microcracks are present at the interface before the tape-test already which results in the local detachment of the coating. Therefore the tape-test is not a suitable method for the measurement of the HA-coatings adhesion, which form on metal substrates from SBF.

The details of areas with removed apatite coatings are shown in Figure 9 a,b (SEM). Figures and EDS analyses of HA-coatings and damaged areas confirm that the coating detached almost right above the substrate.

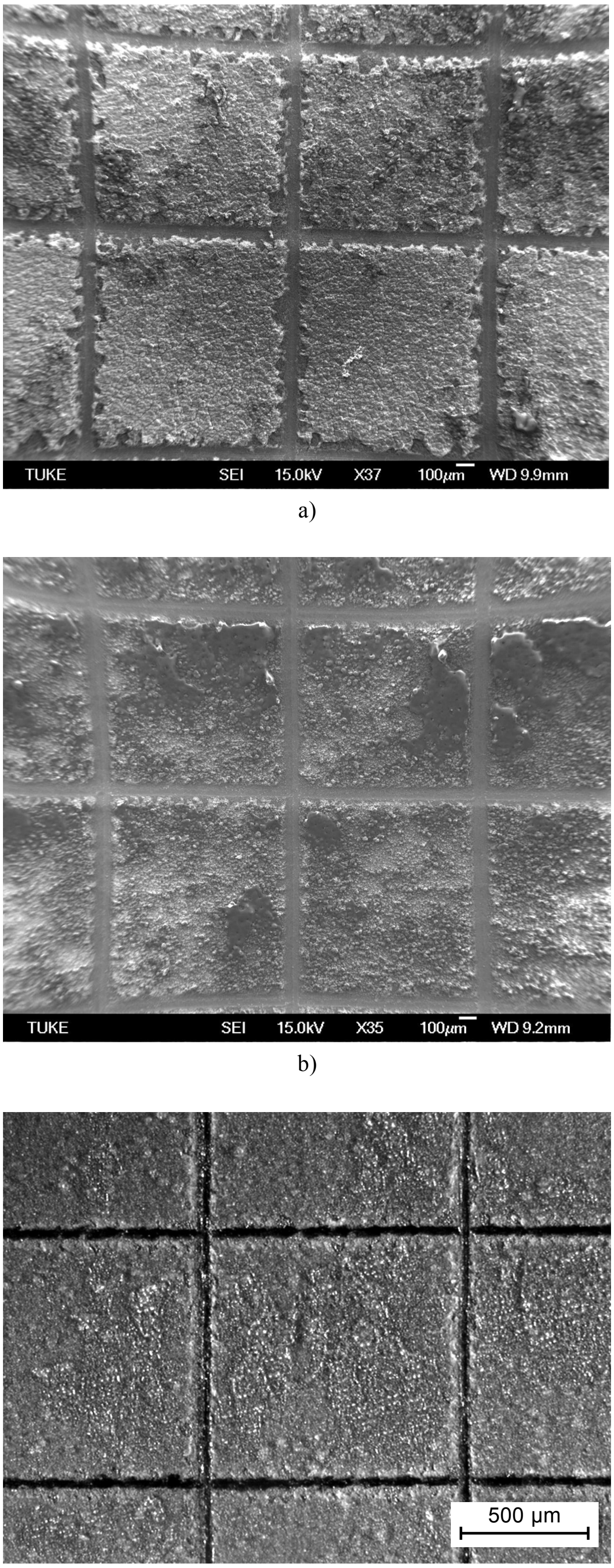


Figure 8. Coating on the surface of sandblasted Ti6Al4V sample after 7 days in SBF: a) without calcification, pretreated in NaOH, b) calcified in SCS for 2 hours and pretreated in NaOH solution, c) calcified in SCS for 3 days and pretreated in NaOH solution; the dark areas in SEM are the glue from tape.

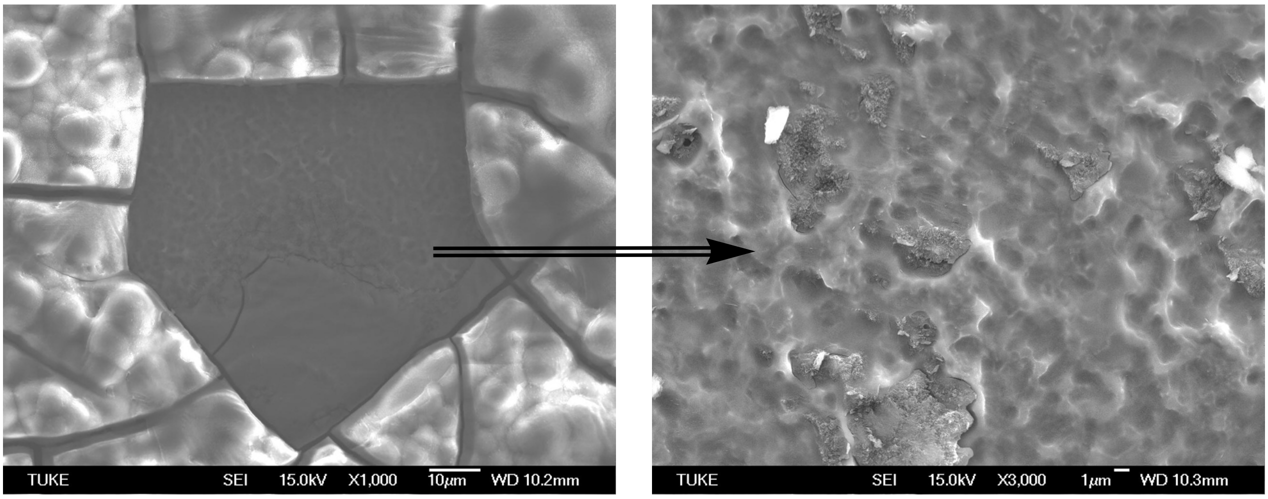
A thin non-compact HA-layer remains in the area from where the coating detached after the tape-test. In case of thick coatings, the undetached HA-layer is thicker in the damaged area. The tape-test removes only the upper layer due to cohesion of the coating. Due to a higher concentration of Ca and P ions in the fractured areas of sandblasted samples in comparison with ground samples it can be supposed that the cavities formed by sandblasting are filled by apatite and the coating is removed almost right at the basic level of the substrate.

CONCLUSION

The treatment in the concentrated alkali solution increases the surface reactivity of grinded and sandblasted Ti6Al4V substrates. The corroded layer immediately interacts with the Ca^{2+} and PO_4^{3-} ions in calcium-phosphate solutions. As a parallel experiment the titanium sandblasted and ground samples were treated and coated by the same way as a Ti6Al4V samples.

Table 4. Classification of adhesion of the different coatings to the Ti6Al4V and Ti surface.

	Ti6Al4V				Ti			
	ground		sandblasted		ground		sandblasted	
sample/treatment	removed area	category	removed area	category	removed area	category	removed area	category
After chemical treatment in NaOH (corrosion coating)	0 %	5	0 %	5	0 %	5	0 %	5
After chemical treatment in NaOH and 2 hrs. in SCS (calcified coating)	0-4 %	4	0-4 %	4	0-4 %	4	0-4 %	4
After chemical treatment in NaOH, 3 days in SCS (crystalline coating)	0-4 %	4	28 %	2	0 %	5	0 - 4 %	4
After chemical treatment in NaOH and 7 days in SBF (HA-coating)	27-30 %	2	27 %	2	30-40 %	1	10-15 %	3
After chemical treatment in NaOH, 2 hrs in SCS and 7 days in SBF (HA-coating)	10-15 %	3	10-15 %	3	10-15 %	3	10-15 %	3
After chemical treatment in NaOH, 3 days in SCS and 7 days in SBF (HA-coating)	30 %	2	30 %	2	55.7 %	1	20 %	2



a) b)

Figure 9. (SEM): a) Area with removed HA-coating, which formed in the course of 7 days in SBF on the surface of ground Ti6Al4V chemically treated in NaOH; without calcification; b) fracture area appearance of substrate versus HA-coating after tape-test.

The tape-test ASTM D 3359 B allows to compare and classify the adhesion of several- microns-thick coatings. The test results showed:

- The tape-test is suitable for evaluating of adhesion of the corrosive layers on Ti and Ti6Al4V, i.e. the alkali treated layers and of these corrosion layers after a short-time calcification (exposure to SCS for 0.5-2 hours). In spite of cracking, the coatings showed a very good adhesion to all types of substrates and were classified into category 5.
- The adhesion of the crystalline coatings formed in the course of a long-time calcification cannot be measured by tape-test. The test is not a suitable to give real information of the adhesion of basal coating. The damaging of the coating and breaking of the crystals are very significant in case the surface is coated with non-uniform crystal spherulites with different size.
- The hydrated HA-coatings which formed on Ti and Ti6Al4V surfaces in SBF are compact but crack during drying on air. The formation of the microcracks negatively influences the results of the tape-test. The relief of globular coatings which is considerable for sandblasted samples influences the results of tape-test as well. The cracking of the HA-coatings makes this test unsuitable.

The scratch test could give more objective results as to the adhesion of the coatings formed in each step of treatment. But the question of how to prepare crackless samples for measurement of adhesion remains still open. The coating of the samples does not crack only if the samples are kept wet, immerged to solutions.

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HODNOTENIE SÚDRŽNOSTI BIOAKTÍVNEJ VRSTVY NA Ti6Al4V SUBSTRÁTE TAPE-TESTOM

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Práca je zameraná na hodnotenie súdržnosti povrchovej vrstvy k substrátu, ktorá sa vytvorí na brúsených a tryskaných vzorkách Ti6Al4V: 1) v procese alkalického lúhovania, 2) v prekalcifikačných roztokoch (SCS) a c) počas *in vitro* testov (SBF). Zvolené chemické úpravy tryskaním a brúsením opracovaných vzoriek, t.j. očistenie povrchu v koncentrovanom roztoku HCl a následná aktivácia povrchu v roztoku NaOH, zväčšujú drsnosť povrchov. Paralelne boli testované aj titánové substráty, upravené rovnakým spôsobom ako titánová zliatina. Adhézia povrchových vrstiev sa testovala ASTM D 3359-02: Standard Test Methods for Measuring Adhesion by Tape - cross cut tape-test (B). Tape-test B dovoľuje porovnávať a klasifikovať adhéziu niekoľko mikrometrov hrubých povlakov k substrátu. Výsledky skúšok ukázali: Tape-test je vhodný pre hodnotenie adhézie povlaku po chemickej úprave a po krátkodobej prekalcifikácii. Adhézia povlakov je dobrá, prevažne odpovedá triede 5. Test nie je vhodný pre prekalcifikované povlaky pokryté vrstvou kryštalických sférolitov, ktoré sa získajú pri viacdennej expozícii vzoriek v roztokoch SCS. Hydroxyapatitový (HA)-povlak, ktorý sa tvorí na Ti a Ti6Al4V v SBF (*in vitro* test) je v roztoku súdržný, ale pri vysušení praská. Efekt praskania, vznik mikrotrhlín negatívne ovplyvňuje výsledky tape-test. Praskanie povlaku nedovoľuje získať vierohodné výsledky, informáciu o adhézii HA-povlakov k substrátu. Objektívnejšie výsledky o adhézii povlakov v jednotlivých štádiách úprav by možno mohol dať scratch test. Aj tu ostáva otvorená otázka, ako pripraviť vzorky, aby nedošlo k praskaniu povlakov. K praskaniu nedochádza len, ak sú vzorky mokré, uchované v roztokoch.