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Technological capabilities of surface layers formation on implant made of Ti-6Al-4V ELI alloy

MARTA KIEL-JAMROZIK[1]*, JANUSZ SZEWCZENKO[1],
MARCIN BASIAGA[1], KATARZYNA NOWIŃSKA[2]

[1] Silesian University of Technology, Faculty of Biomedical Engineering,
Department of Biomaterials and Medical Devices Engineering, Zabrze, Poland.
[2] Silesian University of Technology, Faculty of Mining and Geology,
Institute of Applied Geology, Gliwice, Poland.

Purpose: The aim of the presented research was to find a combination of surface modification methods of implants made of the Ti-6Al-4V ELI alloy, that lead to formation of effective barrier for metallic ions that may infiltrate into solution. Methods: To this end, the following tests were carried out: roughness measurement, the voltamperometric tests (potentiodynamic and potentiostatic), and the ion infiltration test. Results: The electropolishing process resulted in the lowering of surface roughness in comparison with mechanical treatment of the surface layer. The anodization process and steam sterilization increased corrosion resistance regardless of the mechanical treatment or electropolishing. The crevice corrosion tests revealed that independent of the modification method applied, the Ti-6Al-4V ELI alloy has excellent crevice corrosion resistance. The smallest quantity of ions infiltrated to the solution was observed for surface modification consisting in the mechanical treatment and anodization with the potential of 97 V. Conclusions: Electric parameters determined during studies were the basis for effectiveness estimation of particular surface treatment methods. The research has shown that the anodization process significantly influences the pitting corrosion resistance of the Ti-6Al-4V ELI alloy independent of the previous surface treatment methods (mechanical and electrochemical). The surface layer after such modification is a protective barrier for metallic ions infiltrated to solution and protects titanium alloy against corrosive environment influence.

Key words: *corrosion resistance, ion infiltration, metallic biomaterials, surface modification, titanium alloys*

1. Introduction

Titanium alloys due to their good mechanical properties, low specific weight and good corrosion resistance are used in different fields of medicine [1]–[7]. The Ti-6Al-4V ELI alloy is the most commonly used biomaterial for implants in orthopedy and traumatology, especially in spondylosurgery.

Long-term research and clinical observations of toxicological and allergic influence of ions on human body proved that infiltrated aluminum and vanadium ions have cytotoxic properties. Products of implant degradation in the form of metallic ions or corrosion products could influence intercellular space or penetrate cells, which leads to metallosis. According to that, surface treatment methods are very important in forming physicochemical properties and biocompability of titanium alloys. There is a dependence between the acceptation of implant by a human body and appropriate preparation of the surface layer. The anodization process is used to restrict the infiltration process. Thin oxide layer is growing on implant's surface during this process. Properties of the layer depend on electrolyte, production method, oxidation time and electric parameters of the process [7]–[21].

One technique used to verify the suitability of particular surface treatment method before anodization

* Corresponding author: Marta Kiel-Jamrozik, Silesian University of Technology, Faculty of Biomedical Engineering, Department of Biomaterials and Medical Devices Engineering, ul. Roosevelta 40, 41-800 Zabrze, Poland. Tel/fax: +48 32 277 74 49, e-mail: marta.kiel-jamrozik@polsl.pl
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is voltamperometry (potentiodynamic and potentiostatic) [9]–[21]. The aim of the presented research was to find a combination of surface modification methods of implants made of the Ti-6Al-4V ELI alloy, that lead to formation of effective barrier for metallic ions that may infiltrate into solution.

The work is a continuation of research conducted by the authors on the effect of surface modification on the physicochemical properties of the Ti-6Al-4V ELI alloy [2], [3], [15], [17]–[20].

2. Materials and methods

The Ti-6Al-4V ELI alloy with the chemical composition consistent with the guidelines of ASTM F136–08e1 [23] standard was used in all test procedures performed, Table 1. The titanium alloy was in the form of bars of ∅8 and ∅14 mm.

Table 1. Chemical composition of the Ti-6Al-4V ELI alloy [23]

	Chemical analysis, %							
	Element							
	Al	V	Fe	O	C	N	H	Ti
ASTM F746 standard	5.5–6.5	3.5–4.5	0.25	0.130	0.080	0.050	0.012	balance
Certificate for ∅8 mm	5.97	3.88	0.13	0.110	0.009	0.005	–	balance
Certificate for ∅14 mm	5.95	3.96	0.15	0.114	0.003	0.004	0.0037	balance

The modification of surface layer included: mechanical treatment (shot peening, mechanical polishing, sandblasting) and electrochemical treatment (electropolishing, anodization). Steam sterilization was also considered in studies pertaining formation of protective properties of the surface layer of the implants. The shot peening process was carried out with the use of ceramic beads and wetting medium. The mechanical polishing was performed with the use of sisal brushes and polishing paste. To get a high gloss mirror surface implants were polished using wheels made of canvas and polishing paste. The surface of the sample was sandblasted after shot peening. Sandblasting was performed in a blast cabinet with glass beads. The electropolishing process was carried out in the chromic acid bath (E–395 POLIGRAT GmbH) with current density i = 10–30 A/dm^2. The anodization was made in the electrolyte based on phosphoric and sulfuric acid (Titan Color POLIGRAT GmbH). The anodization process was realized at different potentials: 57 V, 77 V, 87 V, 97 V. The set of potentials was composed taking into account values of voltage that are commonly applied by producers of medical devices for anodization. Hence the influence of anodization process conditions on the physiochemical properties of the modified surface layer of implants made out of the Ti-6Al-4V ELI alloy was investigated in a way that simulates real life use. The steam autoclave MOCOM Basic Plus was used for the sterilization process at a temperature T = 134 °C, under pressure p = 2.10 bar in time t = 12 min. The steam sterilization process is important because every implant must be sterilized before implantation into human body.

Surface roughness was checked with the SURTRONIC 3+ (Taylor Hobson) profiler. The length of sections measured was Lc = 1.25 mm, and the accuracy of roughness measurement was ±0.02 μm. From the measurement, the value of surface roughness parameter specifying the arithmetic average of ordinates profile Ra (according to the standard PN-EN ISO 4287:1999 [24]) was obtained. Five measurements were performed for five specimens of each surface type considered.

To estimate the effectiveness of particular surface treatment methods, the study of pitting and crevice corrosion was carried out [25]. Tests were realized in Ringer's solution (B. Braun Melsungen AG) for the following parameters: temperature T = 37 ± 1 °C, pH = 6.9 ± 0.2. The voltamperometric tests (pitting corrosion resistance – potentiodynamic method, crevice corrosion resistance – potentiostatic method) were carried out with the use of potentiostat VoltaLab® PGP201 (Radiometer). The saturated calomel electrode was applied as the reference electrode, the platinum electrode was the auxiliary electrode. Bars of 14 mm in diameter made from the Ti-6Al-4V ELI alloy were used for pitting corrosion tests [25]. The applied scan rate was 3 mV/s until maximum measuring range

+4000 mV or anodic current density i = 1 mA/cm^2 were reached. Bars of 8 mm in diameter were used for crevice corrosion tests. In this procedure artificial crevice was used to facilitate breakdown, which is inducted by polarization to +800 mV. If the corrosion had not been initiated in the first 20 s of the test (anodic current drops rapidly or is very small), then stimulation was made for 15 min. After this time the test was interrupted and material of the test specimen was regarded as resistant to crevice corrosion in the test environment, when at potential of +800 mV there was no increase of anodic current. Otherwise, when anodic current increased by potentials lower than +800 mV, the test result was negative and the material of the specimen did not fulfil normative guidelines about crevice corrosion resistance [25].

The ion infiltration tests were made for the Ti-6Al-4V ELI alloy after the sterilization process. On the basis of the results obtained concentrations of Ti, Al, V ions infiltrated to Ringer's solution were determined. The samples were in Ringer's solution for 28 days at a temperature T = 37 ± 1 °C. The concentration of metallic ions was calculated using the JY 2000 spectrometer, which used atomic spectroscopy method with induction plasma (ICP AES). The source of induction was plasma torch (argon created plasma) connected to the 40.68 MHz frequency generator. To create analytical curve dilute analytical materials (Merck) were used.

3. Results

In the first step, roughness of the surface of the samples made of Ti-6Al-4V ELI alloy after different modifications was checked. It was found that application of mechanical treatment proposed in the work (shot peening, mechanical polishing and sandblasting) makes it possible to treat a surface layer with roughness Ra = 0.50 μm. The electropolishing process affected the surface texture. Significant reduction of the surface roughness (Ra = 0.28 μm) was observed after applying this type of treatment. On the other hand, anodization at different potentials had no influence on the surface topography, regardless of the preceded surface treatments.

Next, voltamperometric tests were carried out. Registered anodic polarization curves were the basis for determination of characteristic corrosion values of the Ti-6Al-4V ELI alloy, Table 2, Fig. 1.

On the basis of the studies conducted it has been stated that the average value of the corrosion potential for samples in the initial state was equal to E_{corr} = –95 mV. The sterilization process increased it to equal E_{corr} = +85 mV. The anodic polarization curves assigned showed the presence of the average transpasivation potential E_{tr} = +1830 mV, for the samples before and after the sterilization process, respectively. Values of polarization resistance R_p for the sample in

Table 2. Corrosion resistance of the Ti-6Al-4V ELI alloy after different surface treatment

Sample	Average value of potentials, mV				Average value of polarization resistance, kΩcm^2 R_p		Average value of corrosion current density, nA/cm^2 i_{corr}	
	E_{corr}		E_{tr}					
		S		S		S		S
1	–95 ± 5	+85 ± 8	+1830 ± 12	+1830 ± 14	557 ± 8	345 ± 5	50 ± 3	80 ± 5
1/2/3/4	–168 ± 7	–140 ± 7	+1480 ± 15	+1497 ± 13	278 ± 5	341 ± 6	90 ± 4	80 ± 4
1/2/3/4/5	–450 ± 3	–355 ± 9	–	–	720 ± 9	674 ± 9	40 ± 2	40 ± 1
1/2/3/4/57V	+10 ± 8	+70 ± 9	–	–	3100 ± 12	3000 ± 9	3.2 ± 1	2.6 ± 1
1/2/3/4/77V	+110 ± 4	+120 ± 8	–	–	3300 ± 14	2100 ± 10	2.3 ± 1	1.7 ± 0.5
1/2/3/4/87V	+130 ± 5	+185 ± 5	–	–	2460 ± 10	2700 ± 10	1.1 ± 0.5	3.6 ± 0.6
1/2/3/4/97V	+120 ± 9	+195 ± 3	–	–	2000 ± 11	1260 ± 6	1.9 ± 0.5	1.5 ± 0.3
1/2/3/4/5/57V	–30 ± 10	+260 ± 6	–	–	7960 ± 9	9950 ± 9	8.0 ± 1	9.0 ± 1
1/2/3/4/5/77V	–180 ± 6	+175 ± 4	–	–	11150 ± 19	15200 ± 14	8.0 ± 1	12 ± 2
1/2/3/4/5/87V	–220 ± 7	+240 ± 2	–	–	24400 ± 12	7130 ± 10	10 ± 2	10 ± 1
1/2/3/4/5/97V	–35 ± 8	+410 ± 7	–	–	13260 ± 14	17300 ± 14	13 ± 2	20 ± 2

1 – grinding, 2 – shot peening, 3 – mechanical polishing, 4 – sandblasting, 5 – electropolishing, XV – anodization (X represents the applied potential), S – sterilization.

the initial state and after the sterilization process were calculated with the use of Stern's method and were as follows: R_p = 557 kΩcm^2 and R_p = 345 kΩcm^2. In the next step, anodic polarization curves for the surface after mechanical treatment were determined. For the surface modified in that way the value of the corrosion potential E_{corr} was on the average level E_{corr} = –168 mV.

The steam sterilization process causes insignificant increase of the average corrosion potential to the level of E_{corr} = –140 mV. In the case of surface after the anodization process the average value of the corrosion potential was higher E_{corr} = +92 mV and E_{corr} = +142 mV, without and after the sterilization process, respectively. Anodic polarization curves for that group (after mechanical treatment, after mechanical treatment and anodization) before and after the sterilization process indicated the existence of passive range up to E = +4000 mV. No rapid jump of the anodic current density was observed in the whole measuring range, which could indicate the initiation of the pitting corrosion. The average values of the polarization resistance were as follows:

- for the surface after mechanical treatment: before the sterilization process – R_p = 278 kΩcm^2; after the sterilization process – R_p = 341 kΩcm^2,
- for the surface after mechanical treatment and anodization: before the sterilization process – R_p = 2715 kΩcm^2; after the sterilization process – R_p = 2265 kΩcm^2.

The application of electropolishing effectively improves corrosion properties of the Ti-6Al-4V ELI alloy. An increase of the average value of the polarization resistance with reference to mechanically treated samples was observed: before the sterilization process R_p = 720 kΩcm^2, after the sterilization proc-

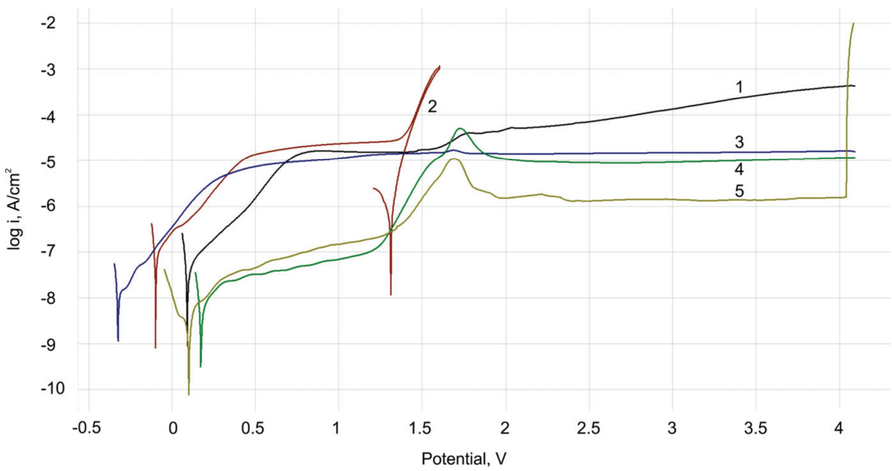

Fig. 1. Anodic polarization curves for the samples after the sterilization process: 1 – 1, 2 – 1/2/3/4, 3 – 1/2/3/4/5, 4 – 1/2/3/4/87 V, 5 – 1/2/3/4/5/87V

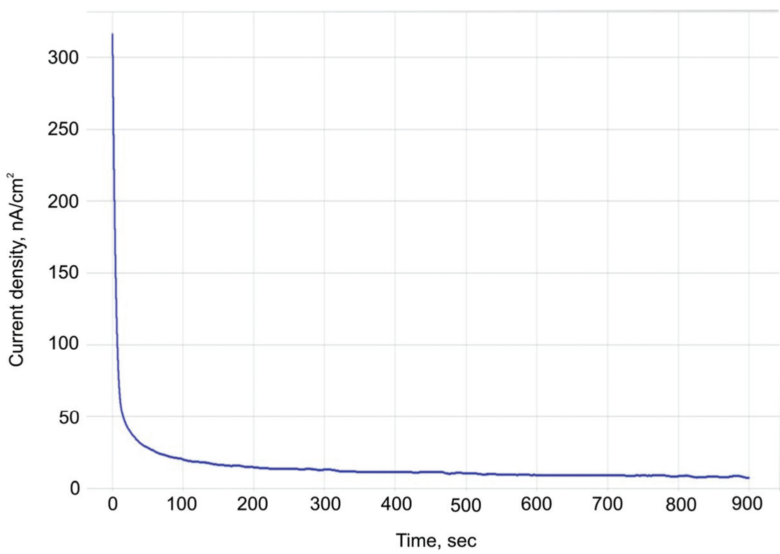

Fig. 2. Current density curve for the sample 1/2/3/4/77V after the sterilization process

ess: R_p = 674 kΩcm^2. The passive area was noticed in the whole anodic range (E = +4000 mV), which is a beneficial effect. The anodization process after mechanical treatment and electropolishing caused large increase of the average value of the polarization resistance to R_p = 14192 kΩcm^2 – before the sterilization process and R_p = 12395 kΩcm^2 after the sterilization process. The sterilization process for the Ti-6Al-4V ELI alloy after electropolishing and anodization led to the average value of the corrosion potential on the level E_{corr} = +271 mV.

The crevice corrosion tests revealed that independent of the modification method applied, the Ti-6Al-4V ELI alloy is fully resistant to this type of corrosion, Fig. 2.

Chemical composition of Ringer's solution in which samples of different surface treatments were immersed for 28 days (Table 3) revealed that the maximum average concentration of Ti ions (1.79 ppm) was registered for the sample with electropolished surface – 1/2/3/4/5. For samples with anodized surface the maximum average concentration of Ti ions (1.70 ppm) was observed for those with mechanical treatment and anodization applied – 1/2/3/4/87 V, while the minimum concentration was found for samples that had undergone electropolishing and anodization – 1/2/3/4/77 V.

For Al ions the minimum average concentration (0.161 ppm) was registered for samples with surface after mechanical treatment and anodization – 1/2/3/4/97 V, while the maximum (0.707 ppm) for those after mechanical treatment only – 1/2/3/4.

Table 3. Results of ion infiltration from the surface of the Ti-6Al-4V ELI to Ringer's solution after 28 days

Sample	Ion penetration, ppm		
	Ti	Al	V
1	1.68	0.456	0.055
1/2/3/4	1.63	0.437	0.055
1/2/3/4/5	1.79	0.810	0.080
1/2/3/4/57V	1.38	0.698	0.062
1/2/3/4/77V	1.34	0.664	0.060
1/2/3/4/87V	1.70	0.267	0.044
1/2/3/4/97V	1.44	0.161	0.033
1/2/3/4/5/57V	1.54	0.688	0.060
1/2/3/4/5/77V	1.51	0.699	0.060
1/2/3/4/5/87V	1.56	0.684	0.041
1/2/3/4/5/97V	1.42	0.603	0.044

The maximum average concentration of V ions (0.080 ppm) was noticed for specimens with electropolished surface – 1/2/3/4/5, while the minimum (0.033 ppm) when the sample surface had undergone mechanical treatment and anodization – 1/2/3/4/97 V.

Between specimens with the surface in the initial state (grinding) and after mechanical treatment the difference in the quantity of ion concentration was insignificant. Less metallic ions infiltrated into Ringer's solution when the surface was mechanically treated and anodized (1/2/3/4/XV – Ti = 1.47 ppm, Al = 0.448 ppm, V = 0.049 ppm) in comparison with the surface after electropolishing and anodization (1/2/3/4/5/XV – Ti = 1.51 ppm, Al = 0.669 ppm, V = 0.053 ppm).

4. Discussion and conclusions

The Ti-6Al-4V ELI alloy is the most commonly used biomaterial for implants in orthopedy and traumatology, especially in spondylosurgery. Methods of forming physicochemical properties of alloy surface, more and more often are based on anodic oxidation. Various authors [2], [3], [7]–[10], [13], [15]–[22] investigated the influence of anodization of the Ti-6Al-4V ELI alloy on corrosion resistance in tissue environment. The steam sterilization process is a commonly used treatment of metallic implants before implantation to the human body. So, it is very important to take into consideration the sterilization process in preliminary investigation for metallic biomaterials.

The aim of anodic oxidation is to create a surface layer which will be a barrier for metallic ion infiltration and protection against effects of corrosive environment. Clinical observation of the titanium alloy biocompability showed that the vanadium could initiate cytotoxic reactions and, in consequence, neurological disorders, furthermore aluminum could influence the bone softening, damage to neurons and disadvantageously affect the activity and function of enzymes and neurotransmitters, which could cause brain and blood vessel disease. Titanium can cause allergies or peri-implant reaction in the implant–bone tissue interface. Depending on the oxidation degree of titanium there are oxides with different stoichiometry composition: Ti_3O, Ti_3O_2, TiO, Ti_3O_5, Ti_2O, Ti_2O_3, TiO_2. The most stable of the oxides is TiO_2. Of all alloying elements of the Ti-6Al-4V ELI alloy, major attention must be paid to Al and V ions. Infiltration of metallic ions and corrosion products into the human body can result in metallosis, thus very important are some aspects of creating and forming the surface layer on metallic implants [1]–[5], [7]–[11].

In the paper, the usefulness of particular variants of the surface treatment methods used in order to restrain the infiltration of metallic ions into the solution was evaluated. The process of steam sterilization was also taken into consideration. The results obtained were the basis for selecting the most suitable surface treatment method of the Ti-6Al-4V ELI alloy, which produces the best barrier limiting penetration of metallic ions and corrosion products into the human body. According to the studies carried out it can be concluded that the anodization process and steam sterilization increase the corrosion resistance regardless of mechanical treatment or electropolishing. This is confirmed by remarkable differences in the level of polarization resistance R_p in comparison with the initial state (ground surface) resistance and the number of metallic ions infiltrated into the solution from the alloy surface (Ti = 1.42 ppm, Al = 0.603 ppm, V = 0.044 ppm). For the samples in the initial state and after mechanical treatment the transpassivation potential E_{tr} was observed. The transpassivation phenomenon occurs during the growth of the corrosion current density, which proves initiation of the pitting corrosion. Simultaneously, the development of pitting is suppressed by re-creation of the oxide layer on the material surface. It can also be noticed that the value of the anodization potential plays a crucial role in modeling the influence of the alloy surface resistance to the action of corrosive environment. Steam sterilization process does not decrease the corrosion resistance. Corrosion potential E_{corr} for sterilized surfaces was higher in the positive direction in comparison with those not sterilized. The most suitable surface modification method for the Ti-6Al-4V ELI alloy was anodic oxidation at 97 V potential carried out after the mechanical treatment (shot peening, mechanical polishing, sandblasting). For this combination, the lowest concentration of ions infiltrated into the solution was observed. The average concentration values were observed for Al (0.161 ppm) and V (0.33 ppm), which can be caused by proper hardening of the alloy during sandblasting.

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