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MODIFICATION OF THE STRUCTURE AND PROPERTIES OF THE TITANIUM ALLOY TI6AL4V IN BIOMEDICAL APPLICATIONS

MODYFIKACJA STRUKTURY I WŁAŚCIWOŚCI STOPU TYTANU TI6AL4V W ASPEKCIE ZASTOSOWAŃ BIOMEDYCZNYCH

From the time when Per-Ingvar Brånemark discovered oseointegration properties of titanium in 1952 a large-scale studies on the issue of usability this metal in surgery were started. Thanks to the parallel research conducted on independent centers managed to get a number of metal alloys which were implanted into the human body in the form of implants. Among the alloys produced appeared alloy of aluminum and vanadium Ti6Al4V. The mechanical properties, high biocompatibility, low density and for this relatively low-cost, caused that the alloy began to be used as a material for biomedical applications. In the present article analyzed the possibility of modifying the properties of the alloy, by choosing another method of producing – method of injection casting with suction. A comparative analysis of the input material - the alloy commercially produced in the form of a rod of the same material melted by injection under vacuum and in air were carried out. The studies results indicate that using method of injection casting with suction to produce the final item influence on significant improvement in mechanical properties as a result of fragmentation of the grains at the element surface. The mechanical properties play a key role in a tissue-implant-bone system.

Key words: titanium alloy Ti6Al4V, the production of nanocrystalline alloys by injection method, biomaterials

Od 1952roku, kiedy to Per-Ingvar Brånemark odkrył osteointegracyjne właściwości tytanu, rozpoczęto w znacznym stopniu badania w kwestii przydatności tego metalu w chirurgii. Poprzez badania prowadzone przez kilka niezależnych ośrodków naukowo - badawczych w organizm człowiek wszczepiono szereg stopów metali w postaci implantów. Wśród wszczepianych stopów pojawił się Ti6Al4V, którego charakteryzują bardzo dobre właściwości mechaniczne, wysoka zgodność biologiczna, niska gęstość przy stosunkowo niskiej cenie. Spowodowało to, że stop ten zaczął być używany jako materiał do zastosowań biomedycznych.

W niniejszym artykule przeanalizowano możliwość modyfikowania właściwości stopu, poprzez zmianę metody wytwarzania, wtłaczania w atmosferze próżni lub w powietrzu. Przeprowadzono analizę porównawczą materiału wyjściowego - stopu, jaki wytwarza się komercyjnie w przemyśle w postaci prętów, a materiału uzyskanego metodą wtłaczania z zastosowaniem próżni oraz w powietrzu. Wyniki badań wykazują, że zastosowanie metody wtłaczania wpływa na znaczącą poprawę właściwości mechanicznych w wyniku rozdrobnienia ziaren na powierzchni elementu. Właściwości mechaniczne odgrywają istotną rolę w połączeniach: tkanka - implant - kość.

1. Introduction

The initial often unsuccessful, repeatedly associated with adverse effects on trial implanting titanium into the human body dates back to the forties of the last century [1 - 3]. Intensive development work on the application of titanium alloys for orthopedic and dental surgery took place since the discovery by Bannemark titanium ability to osseointegration. [4 - 6]. During these studies mechanical properties, corrosion and biocompatibility were evaluated. With the development of implant design and the collection of clinical experience with their use, as well as on the basis of studies in the field of biomechanics and the biotolerance implanted metallic materials was found that the most suitable characteristics for biomedical applications has titanium alloy Ti6Al4V ELI. This alloy successfully used for many fields of medicine were begun [7]. Properties such as satisfactory mechanical properties, modulus of elasticity similar to the module bone and excellent biotolerance are not synonymous with the fact that this material is ideal for medical applications. Repeatedly the problem of fretting corrosion were observed, in particular in joint arthroplasty where as a result of frictional wear of the cooperating parts (implant - implant, the implant - bone) particles / metal ions penetrated into the tissue which

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contributed to postoperative complications. According to the literature, negative symptoms of patients after implantation of hip prostheses made of alloy Ti6Al4V even reached 8.5% [8] and 7.3% [9] all of the treatments and their consequence was the need of reoperation [7].

Another negative factor which limits the use of titanium alloys in medicine is insufficient abrasion resistance of these materials. Following problems were observed: the accumulation of wear product from the abrasion process of the implant in contact with the backbone, or tissue, which leads to changes in shape of the prosthesis as well as the formation of allergenic factors and inflammation, and hence to shorten the life of the implant [1, 10, 11].

Previously mentioned aspects have contributed to carrying out further work for the search the methods of hardening and improving the physicochemical properties of surface of titanium alloys, in order to improve their tribological properties and reduce the amount of corrosion products [7].

Preparation of massive amorphous alloys by injection, consists of injecting a liquid alloy into a mold under gas pressure. The production process runs in following steps: prepared ingot is placed in a quartz crucible where is melted inductively. Then, the liquid alloy is injected under pressure gas to the copper mold, which is cooled radially [12 - 14].

The technology used for melting metal charge in a quartz crucible can cause enters the silicon to the produced material. In the case of materials for medicine appendix of this element is not adversely affected – on the contrary is a necessary and highly desirable building component of the human body. According to literature [15] this element is present in all healthy tissues.

Silicon plays a special role in creating and the functioning of connective tissue. Participates in the bone growth, formation of collagen and bone matrix mineralization. Silicon also affects the hormonal balance in mammals and supports the coronary system. [15, 16].

2. Material and methodology

The study was conducted on three types of samples produced from titanium alloy Ti6Al4V, which chemical composition is shown in Table 1. The first type of sample was produced from the titanium alloy by the injection with rapid cooling with suction in vacuum atmosphere, the second was also prepared by injection, using a suction in air atmosphere, The third type of sample is a rod of tested alloy.

TABLE 1

Chemical composition of titanium alloy Ti6Al4V [17]

Chemical composition	Al	V	C	Fe	O	N	H	Ti
Percentage, wt%	6,00	4.00	0,03	0,1	0,15	0,01	0,003	Rest

Thus obtained sample were subjected to observation of the microstructure using scanning electron microscope Joel 6610LV and light microscope Axiovert 25 Carl - Zeiss. In order to determine chemical composition a scanning electron microscope with an attachment EDS made by Oxford was used

In order to determine the surface topography, and its parameters studies by using T 1000 Hommel profilometer were performed. Determination of surface roughness in contact with the test surface by the engagement of a needle with a differential measurement system were made.

The mechanical properties of the produced materials on the basis of abrasion resistance were determined. Abrasion resistance tests by using ball-tester wherein the specified time with the given load and speed, the ball zirconium, having a diameter of 20 mm, influenced on the surface of the sample were carried out. The degree of resistance to abrasion was determined by comparison on the basis of microstructural observation areas of abrasion.

On the samples microhardness tests were carried out. Vickers studies with a load of 980.7 mN - HV 0.1 using semi-automatic microhardness tester FM - 7 from FUTURE – TECH were performer.

3. Results

The following figures are shown the titanium alloy Ti6Al4V. Subsequently Figure 1 are shown the structure by means of electron microscopy in Figure 2 and 3 using light microscopy. Analysis of the chemical composition given in Table 2 while the EDS spectrum in Figure 4.

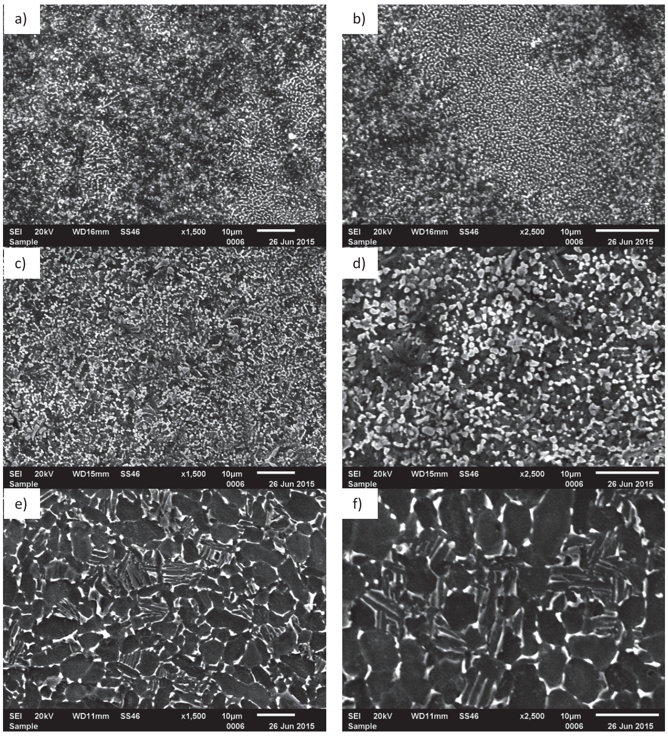

Fig.1. Microstructures of titanium alloy Ti6Al4V a), b) produced by injection method with suction under vacuum atmosphere c), d) produced by injection method with suction in air atmosphere, e), f) in the form of rod, SEM, respectively

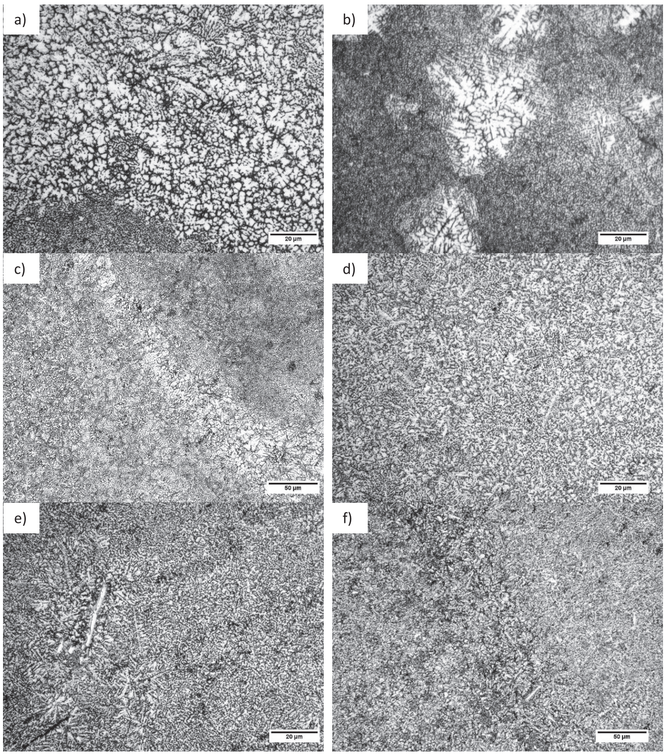

Fig. 2. Microstructures of titanium alloy TI6Al4V a), b) and c) produced by injection method with suction under vacuum atmosphere d), e) and f) produced by injection method with suction in air atmosphere, respectively

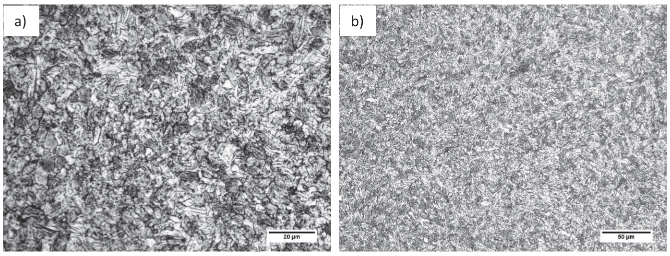

Fig. 3. Microstructures of titanium alloy Ti6Al4V in the form of rod

TABLE 2

The results of EDS analysis

Alloy, wt %	Ti	Al	V	Si
Titanium alloy produced by injection method with suction under vacuum atmosphere	83.19	5.79	5.35	5.67
Titanium alloy produced by injection method with suction in air atmosphere	86.79	5.93	3.95	3.33
Titanium alloy in the form of rod	88.98	6.55	4.47	-

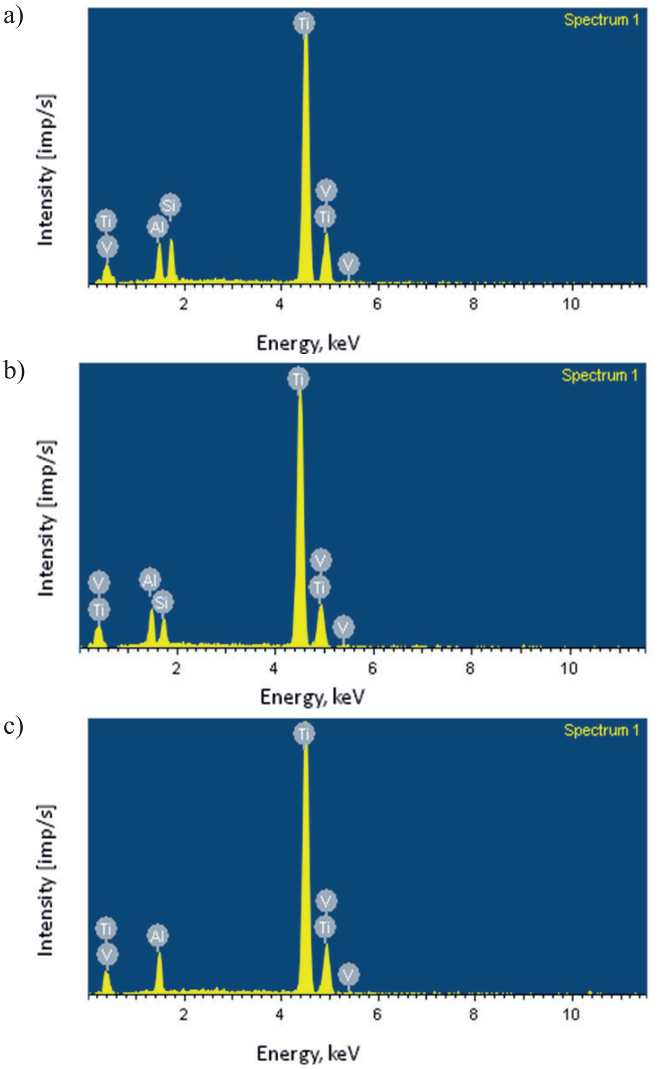

Fig.4. EDS spectrum of titanium alloy Ti6Al4V produced by: a) injection method with suction under vacuum atmosphere b) injection method with suction in air atmosphere, c) in form of rod, respectively

On the basis of the microstructural observations can be seen the structural differences between the titanium alloy prepared by injection method with suction under vacuum and in air atmosphere, and in form of rod obtained by conventional method. The main difference is the degree of grain refinement, materials obtained by the injection, have a fine grain structure, and the conventional material is characterized by higher grain size, what is clearly visible in Figure 1. Can be observed nanocrystalline structure in the case of samples produced by injection method. The structures of titanium alloys produced by injection is not homogeneous over the entire surface, characterized by lack of order and regularity. Can be observed (Figure 2b, e), that occur here dendritic expanding of crystalline nucleating. The structure of titanium alloy produced by conventional method is homogeneous throughout its volume.

Analysis of chemical composition was performed to determine whether the method of producing of titanium alloys prepared by placing the ingot, the quartz crucible has an impact on the chemical composition of the final material. Based on the analysis of EDS, it can be seen that the chemical composition of produced samples has changed. In the alloy produced conventionally disclosed significant elements: titanium, aluminum and vanadium, while the alloys after the pressing, also appeared silicon. This element appeared in significant numbers between 3.33% to 5.67% the total mass fraction. This is due to the diffusion of silicon from the quartz crucible, in which samples are subjected to melt.

The results of measurement of surface roughness are shown in Table 3. Examples of the surface roughness profile of studied materials are shown in Figure 5.

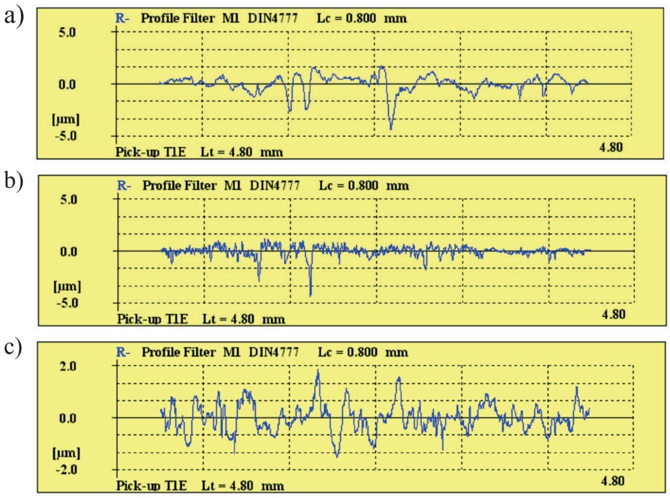

Fig.5. Surface roughness profile of the titanium alloy samples Ti6Al4V produced by: a) injection method with suction under vacuum atmosphere b) injection method with suction in air atmosphere, c) and in form of rod, respectively

Profiles analysis allowed for the identification of indicators roughness. The highest indicators has a sample produced by injection with suction under vacuum. Sample obtained in this way has almost twice the value of the roughness (Ra), than in the case of the sample produced conventionally. Value Ra of sample produced by injection method with suction in air atmosphere is lower than for the conventional sample. Parameter Rq - root mean square elevation profile for both samples produced by injection method is higher than produced by conventional method.

Microscopic images obtained wipe during testing of abrasion resistance on the surface of the test samples over time 0.5 h are shown in Figure 6.

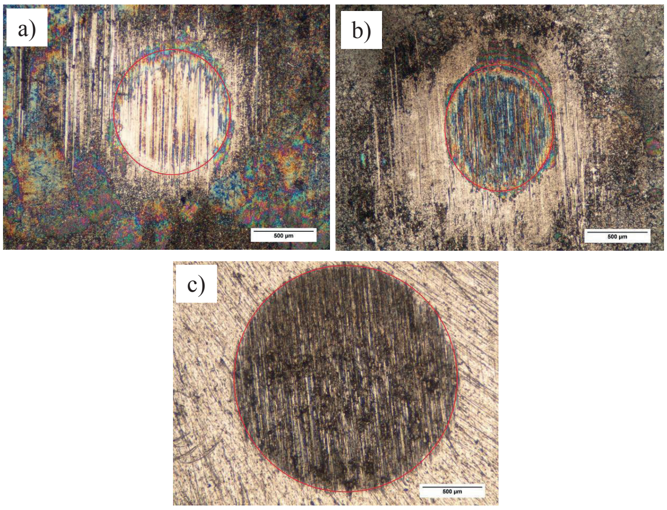

Fig. 6. Pictures of wipe formed on the surface of individual samples of titanium alloy Ti6Al4V produced by: a) injection method with suction under vacuum atmosphere b) injection method with suction in air atmosphere, c) and in form of rod, respectively

Microstructural observations of surface samples submitted to impact zirconia balls allowed to conclude, that the samples produced by injection method under vacuum

TABLE 3

Summary of parameters of surface roughness sample

Sample	Number of measurement	Parameter, µm						
		Rq	**Rt**	**RSm**	**Rz**	**Ra**	**Rp**	**Rmax**
Titanium alloy produced by injection method with suction under vacuum atmosphere	measurement I	0.96	8.60	0.121	5.20	0.72	2.20	7.45
	measurement II	0.81	6.13	0.148	3.44	0.58	1.77	6.13
	measurement III	1.77	9.27	0.129	6.61	0.99	2.77	10.27
	Average	**1.18**	**8.00**	**0.133**	**5.08**	**0.76**	**2.25**	**7.95**
Titanium alloy produced by injection method with suction in air atmosphere	measurement I	0.34	4.10	0.045	1.94	0.24	1.39	4.10
	measurement II	0.50	5.63	0.041	2.88	0.34	1.23	5.63
	measurement III	0.62	6.11	0.087	2.90	0.41	3.03	6.11
	Average	**0.49**	**5.28**	**0.058**	**2.57**	**0.33**	**1.88**	**5.28**
Titanium alloy in the form of rod	measurement I	0.54	3.65	0.082	2.34	0.41	1.61	3.13
	measurement II	0.54	3.41	0.091	2.54	0.41	1.81	3.10
	measurement III	0.46	2.39	0.080	1.96	0.37	1.35	2.39
	Average	**0.51**	**3.15**	**0.084**	**2.28**	**0.40**	**1.59**	**2.87**

Where:

Rq - root mean square elevation profile,
Rt – total height profile,
RS_m - the average width of the grooves of profile elements,
Rz – the greatest height profile,
Ra – arithmetic average of ordinates profile,
Rp – the height of the highest peak profile,
Rmax – the maximum deviation.

TABLE 4

Summary of microhardness tests results

	Titanium alloy produced by injection method with suction under vacuum atmosphere	Titanium alloy produced by injection method with suction in air atmosphere	Titanium alloy in the form of rod
Microhardness, HV0,1	774.9	1245.9	338.8
	793.2	1280.3	341.3
	733.5	1229.7	313.4
	792.8	1232.7	340.1
	788.8	1245.9	330.8
Average, HV0,1	**776.6**	**1246.9**	**332.9**

and in air atmosphere have smaller areas of wiping (in the range of 800-900μm) than in the case of the sample produced by the conventional method (about 1800μm). Greater resistance to abrasion have produced titanium alloys by the injection with suction than the same alloy produced by the commercial method. Results of microhardness tests are shown in Table 4.

The study microhardness were showed that in the case of titanium alloy sample Ti6Al4V produced by injection casting with suction under vacuum, that its microhardness is higher more than double than sample produced by conventional method. In contrast, the highest hardness has sample produced by injection method with suction in air atmosphere. Microhardness of that sample is higher over 900 HV0,1 than material produced by conventional method, and almost 500 HV0,1 higher than material produced by injection method with suction under vacuum.

4. Conclusions

Injection method allows to modify the structure of a titanium alloy impact on its properties. The applied method of injection resulted in fragmentation of the structure to order of nanometric magnitude. Grain refinement improves mechanical properties. Technology of production alloy by injection method is connected, as shown by EDS analysis, with the diffusion of silicon. According to literature sources it is not harmful element to human body, does not cause allergic reactions, but it contributes to increase the hardness and abrasion resistance.

Evaluation of surface topography allowed to conclude that the greater development of the area are characterized by injection casting materials compared to conventional materials. Greater surface development is desirable in terms of application on implants which is associated with the faster osseointegration process.

Studies of abrasion resistance shown the fact that samples produced by injection method with suction have higher abrasion resistance than samples produced by conventional method. Increasing of tribological properties is important because the elements often work in friction systems.

Microhardness analysis shown that alloys produced by injection method with suction in air atmosphere has higher microhardness value in comparison with alloy produced by conventional method and produced by injection method with suction under vacuum. The significant increase in hardness can be associated with the occurrence of dendritic structure.

The applied method of production of titanium alloy Ti6Al4V by injection casting with suction to copper mold shown fact that samples produced by this method have better properties than samples produced by conventional method or same method but without suction. This is due to faster cooling of the sample by the mold. Samples produced by injection method with suction have better mechanical properties what is associated with nanocrystalline structure. This kind of structure increase hardness and abrasion resistance.

Summary materials produced by this method can be used in many branches of engineering where high microhardness, great abrasion resistance and smooth surface is required.

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