

VOLUME 61 ISSUE 2 December 2013

Comparative description of structure and properties of Ti-6AI-4V titanium alloy for biomedical applications produced by two methods: conventional (molding) and innovative (injection) ones

J. Klimas, M. Szota, M. Nabiałek, A. Łukaszewicz*, A. Bukowska

Faculty of Production Engineering and Materials Technology, Czestochowa University of Technology, Al. Armii Krajowej 19, 42-200 Częstochowa, Poland * Corresponding e-mail address: alukaszewicz@wip.pcz.pl

Received 23.10.2013; published in revised form 01.12.2013

Materials

ABSTRACT

Purpose: In paper characterized two methods produced titanium alloy: hitherto used in industry – conti-casting and innovative method of obtaining solid amorphous alloy by injection casting. The results of studies comparing the structure and properties of the alloy Ti-6Al-4V produced by both methods.

Design/methodology/approach: Test samples were titanium alloy Ti-6Al-4V produced by two methods: conventional and injection. To achieve the objective pursued performed the following tests: microstructure observation was carried out, the analysis of mechanical properties (microhardness) and corrosion resistance tests were performed in Ringer's liquid, which simulates the human body fluids.

Findings: Microstructural study allowed to observe that titanium alloy T-6Al-4V produced by conventional method has crystalline ordered structure which is characteristic for materials obtained by drawing, with oriented grains and elongated in the direction of drawing. Same alloy produced by injection casting has amorphous structure with occurrences of the single-crystal seeds, that kind of structure has lack of order and regularity. The microhardness study showed, that titanium alloy Ti-6Al-4V produced by drawing has a hardness of less than twice for the same alloy produced by the injection. The corrosion tests conducted in an environment that simulates human body fluids, revealed showed that the materials made by injection have significantly corrosion potential than alloy obtained by drawing.

Originality/value: The paper presents a comparative study of titanium alloy produced by drawing and massive amorphous alloy produced by unconventional method – injection casting. By the results proved that the alloy produced by injection has much better properties than alloy produced by drawing.

Keywords: Titanium alloy Ti-6Al-4V; Massive amorphous alloy; Injection casting; Drawing

Reference to this paper should be given in the following way:

J. Klimas, M. Szota, M. Nabiałek, A. Łukaszewicz, A. Bukowska, Comparative description of structure and properties of Ti-6Al-4V titanium alloy for biomedical applications produced by two methods: conventional (molding) and innovative (injection) ones, Journal of Achievements in Materials and Manufacturing Engineering 61/2 (2013) 195-201.

1. Introduction

Dynamic advances in technology necessitated the search for new materials, suitable to many industries. Since the midtwentieth century began to produce titanium and its alloys, and used on an industrial scale [1]. These materials are used in many fields for example: chemistry industry, aviation technology, energetics as well in automotive industry, architecture, medicine, and other sectors. [2, 3].

An important branch of industry applications of titanium and its alloys is a medicine, which is associated with a number of requirements for materials for biomedical applications. These materials should be of non-toxicity, should not cause allergic reactions, should have high corrosion resistance, mechanical properties suitable and sufficient resistance to wear by friction. In addition, should have a low tendency to form blood clots [4, 5].

In order to obtain elements made of titanium or alloys thereof with complex shape casting method is used [6]. Products obtained by casting are about 20-30% cheaper than achieved by way of plastic processing. For example, this method is used in the aerospace industry for the manufacture of objects of space and missile technology and the manufacture of components with varying degrees of complexity. Casting is also used when the execution of the product is not possible with stamping or welding method. Examples are casings, impellers for pumps, components of complex shape which, during the mass production it is recommended to die-casting. Method of die casting in the mold (technology VDC), has an advantage over conventional technologies [7]:

- in this process is eliminated waxing and shaping of the ceramic form, which is necessary when precision casting process models melted;
- it is possible to obtain a fine-grained structure of the metal, due to fast cooling, and therefore the possibility of obtaining better properties and fatigue endurance compared with elements received by conventional casting technology.

Obtained by casting products can be formed by drawing, consisting of dragging through the tool's hole, the aim of this action is to change the shape and cross-sectional [8].

Injection casting is that when liquid metallic material is injected to copper mold. The technological process of preparation by injection is to placed pre-melted ingot in quartz capillary tube where it is melting induction, after then material is injected to copper mold by gas pressure. Cooling of the melt located in copper mold has a radial course [9].

Samples were prepared by injection casting have the shape of rods - these samples were produced using the apparatus which is in Fig. 1. The device is designed and build on Facultyof Manufacturing Engineering and Materials Technology -Częstochowa University of Technology. Apparatus for the preparation of solid amorphous alloys is composed of: vacuum work chamber, vacuum transformer coil position controller, induction furnace, gauge, pumping system, control panel injection process and cooling, control rotary and diffusion pump, vacuum gauges and cooling medium. Scheme shown in Fig. 1.

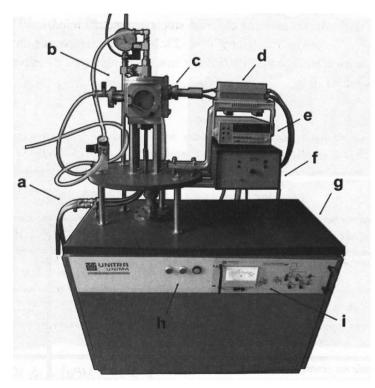


Fig. 1. Apparatus for the preparation of solid amorphous alloys: a) cooling medium connection, b) working chamber, c) vacuum transformer coil position controller, d) transformer, e) gauge, f) induction furnace g) pumping system, h) control panel injection process and cooling i) vacuum gauges and control rotary and diffusion pump

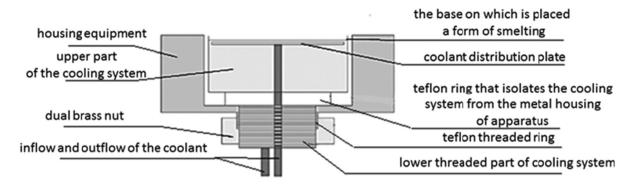


Fig. 2. Scheme of cooling system

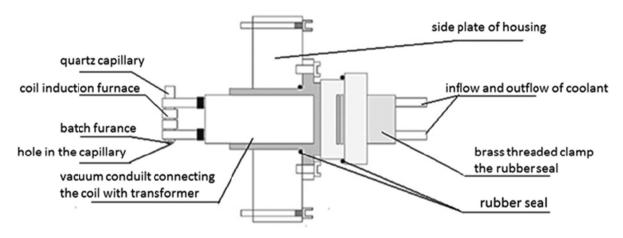


Fig. 3. Scheme of vacuum connection of the inductor coil

In this device uses some interesting solutions, their application streamlines the production process These solutions are: the small volume of the working chamber, so that the pump down time is short - which allow for the full production cycle repeated in a short time; despite the use of a plurality of flexible connections, it is possible to obtain a high vacuum from 5 to 10^{-5} MPa. By placing the handle for attaching the quartz capillary outside the working chamber, and through the use of the seal by the rubber seals system - there is a possibility of reducing the working chamber (increase in the number of castings made in a given unit of time). Cooling copper mold, which is placed on the top plate of the cooling system allows a constant flow of cooling medium - water or liquid nitrogen steam. Scheme shown in Fig. 2.

This design allows for quick and uniform cooling of the mold. The cooling system is isolated from the other metal by rings made of PTFE. By obtaining a high vacuum, it is possible to manufacture the materials at a temperature close to the temperature of liquid nitrogen (77K) [9]. The copper mold is composed of five components: a core mold, which consists of two elements, two further serve as terminal of internal mold core and a copper plate with a hollow bore concentrically. At the bottom of the mold, there is a vent channel, which has the affects on increasing fill factor in a part of the hollow core. Scheme of vacuum connection of the inductor coil, shown in Fig.3.

The use of such a connection allows complete control over the position of the inductor in a horizontal position, What is happening thanks to its retractable design of the side plate device additionally there is a possibility to set the angle of rotation of the coil inductor [9]. Advantages of injection casting are: possibility of performing a full production cycle in short time, obtaining a high vacuum, fast cooling process of copper mold, producing solid amorphous samples, nonstop copper mold temperature control in the range from liquid nitrogen temperature to room temperature [9-11].

Tal	bl	le	1.
I U	0.		•••

Chemical compo	osition of titanium	alloy Ti -	6Al - 4V	12]
----------------	---------------------	------------	----------	-----

Chemical composition	Al	V	C	Fe	О	Ν	Н	Ti
Percentages	6.00	4.00	0.03	0.1	0.15	0.01	0.003	rest

2. Material and methodology

Test samples were titanium alloy Ti-6Al-4V, whose chemical composition is given in Table 1.

Test samples were produced by two methods. First method was commercial continuous casting, and then formed into a rod by drawing. The rod was cut into pellets with dimensions:

- diameter d = 25mm
- height h = 7 mm.

Macroscopic picture of a titanium alloy sample Ti-6Al-4V produced by continuous casting are presented in Fig. 4. Second sample was produced by injection casting for used to massive amorphous material. Macroscopic picture of that sample are presented in Fig. 5.



Fig. 4. Macroscopic picture of a sample of titanium alloys Ti - 6Al - 4V produced by continuous casting and drawing

These samples were submitted for microstructural by light microscope - Axiovert 25 Carl Zeiss, which has camera.

Samples were subjected to microhardness testing. The studies were conducted by Vickers method at a load 980,7 mN - HV 0,1The device on which the measurement was performed is FM - 7 by Future Tech.

The next step was to study the electrochemical that were performer in Ringer's Liquid in temperature 37° C. Solution composition was as follows: 0.39 g potassium chloride; 8.6 g sodium chloride; 0.48 g calcium chloride per 1 dm³ of solution. Electrochemical studies were performed using a potentiostat

AMEL model 7050 digitally controlled using a PC equipped with software Juniorassist in three-electrode system. The reference electrode was a calomel electrode (NEK), while the comparative electrode was a platinum wire, test samples were consisted of a working electrode. The potential point during the measurements varied from in the direction of the anode, in the range of $E_{pocz} = -1,5V$ to $E_{kofc} = 5,0V$ in reference to the saturated calomel electrode (NEK). Each sample was tested five times, considered to be representative curve is that one which is in the middle of repeated curves.



Fig. 5. Macroscopic picture of that sample titanium alloy Ti - 6Al - 4V produced by injection casting

3. Results

Sample images of microstructure titanium alloys produced by continuous casting is shown in Fig. 6a, and instead produced by injection casting in Fig. 6b.

Microstructural observations titanium alloy produced by two methods, allowed the identification of the resulting structure. In the case of a sample obtained conventionally occurs crystalline structure with regular homogenous grain shape with characteristic of this method directional arrangement of grains. (Fig. 3a) parallel to the direction of drawing. Structure of titanium alloy produced by injection casting is structure partially crystalline, and in most predominates amorphous structure which is characterized by a lack of order and regularity. Results of microhardness test were presented in Table 2. These results are presented in the form of column graph microhardness dependence on the state of the sample in Fig. 7.

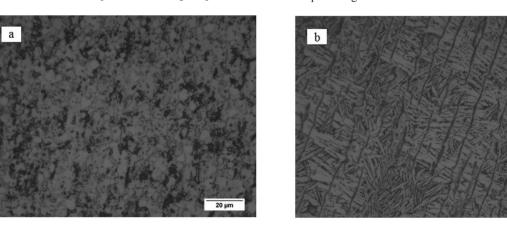


Fig. 6. Microstructure of titanium alloy Ti-6Al-4V produced by a) conventional casting, b) produced by injection casting

Table 2.

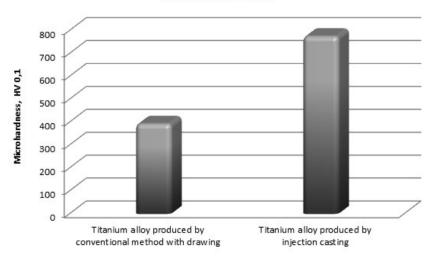
Summary of results of microhardness

Type of sample	Microhardness. HV0.1	Average. HV0.1
	371.9	
-	385.1	
Titanium alloy produced by conventional method with drawing	398.8	393.1
	410.9	
	398.8	
	754.7	
Titanium alloy produced by injection casting	784.5	
	794.2	775.1
	749.0	
	793.2	

Table 3.

The results of electrochemical measurements titanium alloy, fabricated by different methods. Research conducted in the Ringer's liquid.

Type of sample	E _{kor} , V	I kor, mA*mm ⁻²
Titanium alloy produced by conventional method with drawing	-0.18	0.144
Titanium alloy produced by injection casting	-0.78	0.006



Microhardness

Fig. 7. The averaged results of microhardness presented on column graph

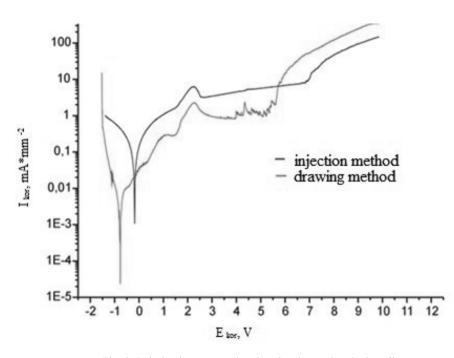
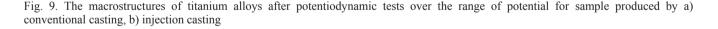


Fig. 8. Polarization curves showing the electrochemical studies

b)

a)



The study demonstrated microhardness for the sample of titanium alloy Ti - 6Al - 4V produced by drawing, that its value is almost twice less than sample produced by injection casting. Arithmetic average micorhardness of the sample produced by injection casting is equal 775,1HV0,1 and for sample produced by conventional method is equal only 393,1HV0,1.

The results of the electrochemical polarization study were presented by curves of polarization which are shown in Fig. 8.

Other values of the parameters determined from the potentiodynamic curves are shown in Table 3.

The macrostructures of titanium alloy Ti-6Al-4V after potentiodynamic tests over the range of potential for sample produced by conventional and injection casting were presented on Fig. 9a and 9b.

The corrosion tests conducted in an environment that simulates human fluid showed that the material produced by injection casting has a higher corrosion potential than the alloy produced by drawing. The lower density of electric current with higher potential corrosive, the higher corrosion resistance. recorded polarization curve for a sample produced by the injection is shifted in the direction of the positive values of potential w compared to sample molded and shaped by drawing. Components produced by injection are characterized by higher biotolerance than components produced by conventional casting.

Materials

4. Conclusions

The method used for preparing the titanium allov Ti-6Al-4V. affects the type of structure, and by this material property. Method of forming a melt by drawing provides a conventional melt with crystalline structure. Fast cooling during molding the sample produced by injection allows obtained amorphous structure which is characterized by a lack of order and regularity. Microhardness analysis showed that the alloy obtained by this method shows almost twice the hardness, as compared with the same alloy produced by conventional method. Higher hardness is equal lower abrasiveness of these components. That property is very important when alloy is used to produce the implant. The study was sowed that corrosion resistance of titanium alloy Ti-6Al-4V is dependent is on the chosen technology manufacturing. This statement is confirmed by the observations of macroscopic - better corrosion resistance is when was used alloy produced by injection casting confirmed by polarization curves. From a medical point of view, titanium alloy Ti-6Al-4V produced by injection casting, has a much better performance than the same alloy produced by conventional casting.

References

- A. Modrzyski, K. Grzeskowiak, Novel methods of casting titanium alloys, Titan and its alloys and processing techniques used In technology and medicine, Publishing House Czestochowa University of Technology, Czestochowa, 2002.
- [2] J. Kaczynski, Titan, Publishing House WNT, Warszawa 1961.

- [3] R. Melechow, K. Tubielewicz, W. Blaszczuk, Titan and its alloys, Publishing House Czestochowa University of Technology, Czestochowa, Czestochowa 2004.
- [4] M. Jurczyk, J. Jakubowicz, Biomaterials, Publishing House Poznan University of Technology, Poznan, 2008.
- [5] J. Klimas, A. Dudek, M. Klimas, Surface Refinement of Titanium Alloy Ti6Al4V ELI, Engineering of Biomaterials R.15 (2012) 116 -117.
- [6] M. Kullick, Contribution to the introduction of casting factor 1.0 for titanium castings. Proceedings of the 10th Word Conference on Titanium, Congress Center Hamburg, Germany, 2003.
- [7] G. Wegman, J. Albreht, G. Luetjering, Microstructure and mechanical properties of titanium casting. Zeitschrift f
 ür Metallkunde 88 (1997) 764 - 773.
- [8] J. Luksza, A. Skolyszewski, F. Witek, W. Zachariasz, Wires made of steel and special alloys, Publishing House WNT, Warsaw, 2006.
- [9] M. Nabialek, Preparation and properties of amorphous and nanocrystalline alloys of iron, Publishing House Czestochowa University of Technology, Czestochowa, 2012.
- [10] M. Nabialek, M. Szota, Method for producing massive amorphous alloys based on group of 3d transition metals with high microhardness, Material Engineering 32/4 182 (2011) 620-623.
- [11] M. Nabialek, L. Jeziorski, M. Szota, J. Jedryka, The use of molten metal suction method for the preparation of a partly crystalline melt Ti-Zr-Be-Cr, Material Engineering 33/4 188 (2012) 264-266.
- [12] ISO 5832-3.Im-plants for surgery-Wrought titanium-6aluminium-4 vanadium alloy. ASTM F-136-Specification for Titanium-6Al-4VELI Alloy for Surgical Implant Applications.