



Work on the modification of the structure and properties of Ti6Al4V titanium alloy for biomedical applications

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ABSTRACT

Purpose: The paper attempts to improve the properties of Ti6Al4V titanium alloy by the using of the injection casting method with rapid cooling. The comparison of the structure and properties of the Ti6Al4V titanium alloy formed by two methods: the injection casting under pressure in two variants under air and vacuum with using a rapid cooling and purchased commercially were carried out.

Design/methodology/approach: Samples were produced by two methods: unconventional casting by injection under pressure of gas or vacuum to copper mold with rapid cooling, and a traditional method for the production of titanium alloys in a form of a rod. To achieve the pursued objective the following tests were performed: microstructural observations – light microscope and SEM, corrosion resistance tests, microhardness tests – Vickers method.

Findings: Microstructural observations showed that the Ti6Al4V titanium alloy produced by injection casting method under pressure with rapid cooling is able to produce extremely fine-grained layer and ductile core. That distribution of structures significantly affect on the improvement of a number of properties compared to commercially produced material. Moreover, during process increased corrosion resistance was observed. Those properties have a significant impact on the possibility of using that type of a production method and material in many areas of materials science related with medicine.

Research limitations/implications: In the framework of the studies, tests using living tissues, which would allow to determine whether the produced material is biocompatible and does not cause inflammation, have not been conducted.

Practical implications: The application of injection casting carries some complications, which mainly relate to quartz capillary where ingot is melted. Titanium as a reactive element strongly absorbs silicon out of the capillary causing changes in the chemical composition in the surface layer of the final element. Further studies will be attempted to use a different type of material wherein the alloy will be melted.

Originality/value: The paper presents the improvement of the properties of materials produced under pressure by casting injection, compared to the same material produced by a commercial method.

Keywords: Ti6Al4V titanium alloy; Injection casting; Biomaterials

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PROPERTIES

1. Introduction

In 1968 committees: the Association for the Study of Internal Fixation (ASIF) and Arbeitsgemeinschaft für Osteosynthesefragen (OE) defined properties which should be characteristic for metallic materials for the use in medicine. Those materials must have the appropriate chemical composition to provide good corrosion resistance, established mechanical properties depending on the application, tissue compatibility, a defined quality of surface, wear resistance, adequate electrical properties [1-6].

Attempts to use titanium and its alloys in medicine date back to the 1940s. Long term observations of elements based on titanium and its alloys contributed to the definition of alloys compositions which can be used in medicine [1,7]. Ti6Al4V titanium alloy belongs to that group. Those alloys are used to produce a number of elements: plate osteosynthesis limbs and skull, dental implants, cardiac implants, parts for ENT and reconstruction [3,8,9].

Despite those achievements, work on the modification of the surface layers of produced implants in order to improve the healing process and osseointegration is still underway. Actually there is a possibility to change properties of surface layer by physical, mechanical and chemical methods [10].

The paper presents the improvement of the properties of materials produced by casting by injecting under pressure, compared to the same material produced by a commercial method.

Production process consists of the following steps: preparing ingot, placing ingot in quartz capillary, induction melting, injection to copper mold cooled radial [11-14].

2. Materials and methodology

Three types of samples produced of a titanium alloy Ti6Al4V which chemical composition shown in Table 1

Table 1.
Chemical composition of Ti6Al4V titanium alloy [15]

Chemical Composition	Al	V	C	Fe	O	N	H	Ti
Percentage	6.00	4.00	0.03	0.1	0.15	0.01	0.003	rest

were the subject of the study. Samples were produced by three different production methods. The first type of samples were produced by injection casting with suction under vacuum, the second type samples were produced by the same method as the first one but under pressure of air the third type of samples were produced commercially in a form of a rod.

All kinds of samples were subjected to a series of tests. Microstructural observations were carried out with SEM Jeol 6610LV as well as Axiovert 25 Carl-Zeiss light microscope.

Microhardness tests were carried out with microhardness-tester FUTURE-TECH by Vickers method under load 980.7 mN. Studies were conducted on a surface layer as well as in a core.

The produced samples were subjected to X-ray qualitative analysis to determine the phase composition. X-ray diffraction was performed on the X-ray apparatus Seifert 3003 T – T with using cobalt lamp filtered radiation with wavelength of X-ray $\lambda = 0.179$ nm, glow current 30 mA and voltage 40 kV.

The next step was electrochemical studies performed in Ringer solution at the temperature of 37°C. The composition of the solution contains: 0.39 g potassium chloride: 8.6 g sodium chloride: 0.48 g calcium chloride to 1 dm³ of solution. Studies were conducted on the device AMEL model 7050 in a three-electrode system (NEK, platinum wire, sample). The potential during the measurement was in the values of -1.5V to 3.5V. Each sample was tested five times, the result was representative by the curve lying in the middle of the repeated curves. The values were read using the Junior Assist software.

3. Results

Figure 1 shown microstructures of Ti6Al4V titanium alloy produced by different methods recorded with SEM microscope as well as a light microscopy in Figure 2.

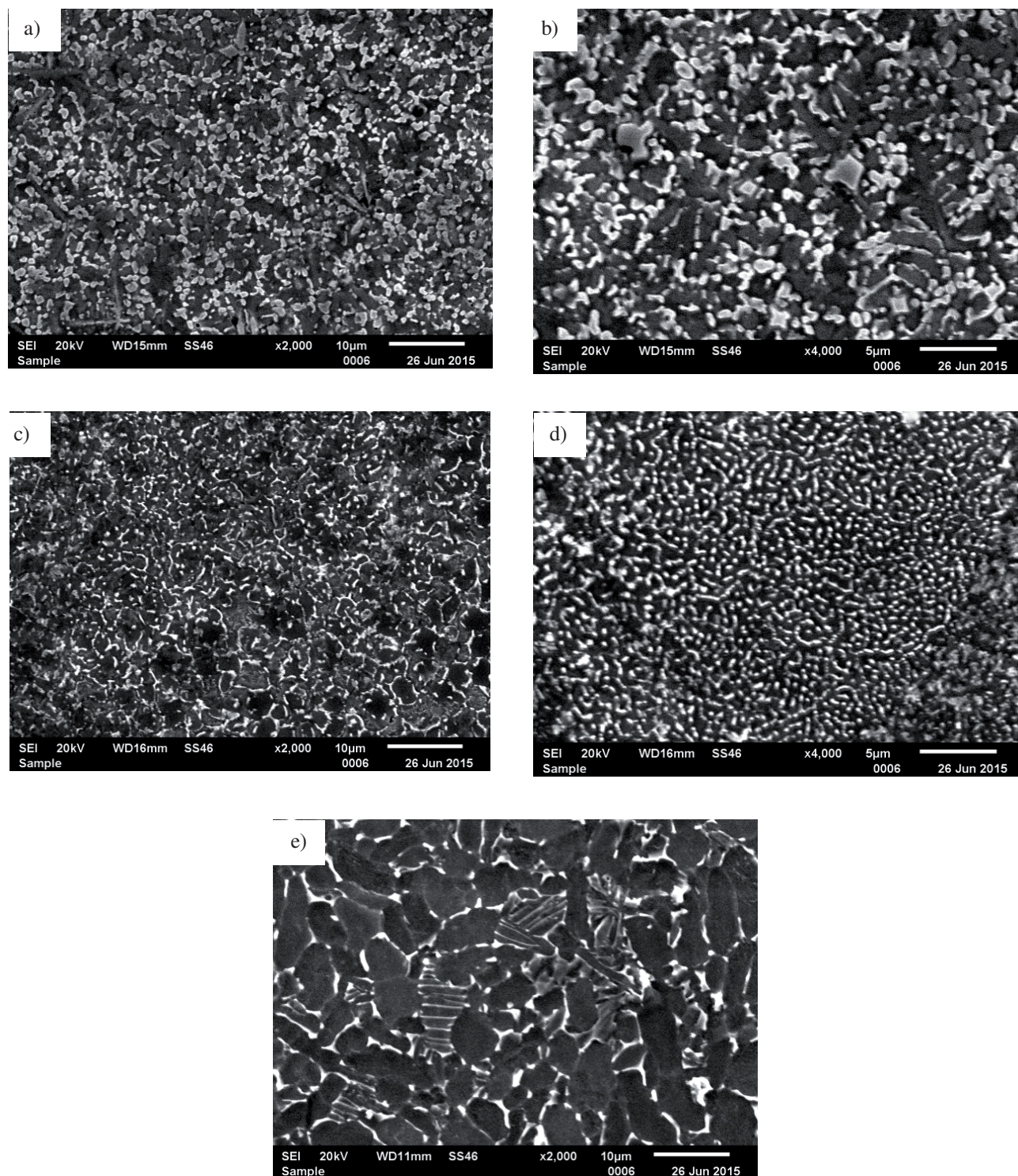


Fig. 1. Microstructures of Ti6Al4V titanium alloy a) and b) produced by injection method with vacuum, c) and d) produced by injection method under air pressure, e) commercial - rod, SEM

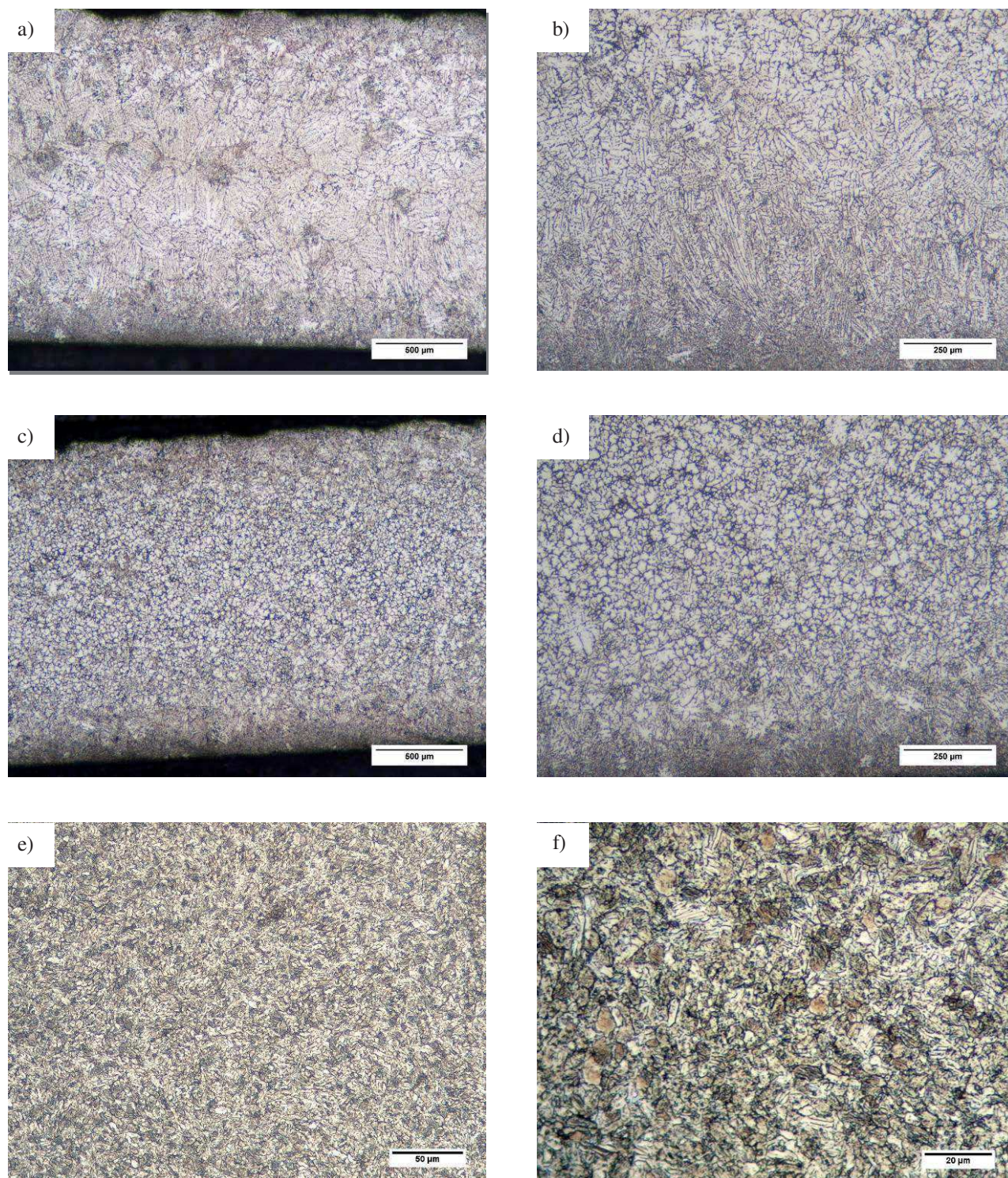


Fig. 2. Microstructures of Ti6Al4V titanium alloy produced by injection casting a) and b) suction with vacuum c) and d) suction under air pressure, e) and f) commercial - rod. Magnitude from 50 to 500

Microstructural observations allowed to notice structural differences between the titanium alloy produced by the injection method with vacuum and under air pressure and commercial titanium alloy in the form of the rod. The structure of titanium alloy produced by the injection casting is not uniform throughout its volume. In figures of microstructural cross-section samples produced by the injection method with vacuum and under air pressure (Figs. 2a and 2c), zonation is visible. In areas where the rate of heat removal was the biggest by contact with the copper form – surface and near surface areas both have very fine structure. Moving deeper into the core there is an area with long thin ordered columns in the direction of heat removal. In the core of samples produced by injection the grains are equiaxed. Those samples compared to samples produced commercially have smaller grains in the entire volume.

Structure of commercial Ti6Al4V titanium alloy in the form of the rod is homogeneous in the entire volume.

The comparison of microhardness tests of all samples (surface) was shown in Table 2. Table 3 summarizes the microhardness of the core and subsurface areas.

Studies of microhardness showed that the highest hardness has a sample formed by casting with air atmosphere. The hardness of this sample is over 900 HV0.1 higher than commercial and almost 500 HV0.1 higher than sample produced with vacuum.

Microhardness distribution on the cross-sectional of samples confirmed the microstructures observations - finer grain, greater hardness. In both cases samples produced by injection casting has the greater hardness in the area of the surface layer than in the core.

Results of X-ray studies are shown on X-ray diffraction patterns. Diffraction pattern of Ti6Al4V titanium alloy produced by the commercial method is shown in Figure 3a and produced by injection casting with vacuum and air pressure in Figures 3b and 3c.

Quality X-ray analysis revealed peaks derived from the phase $Ti\alpha$ and $Ti\beta$ for all three samples. Diffraction patterns of samples produced by injection casting have peaks characteristic of crystalline materials – wide and blurred background which is characteristic for nanocrystalline and amorphous materials.

Results of the electrochemical studies is shown on polarization curves in Figure 4.

Corrosion tests carried out in environments simulating human body fluids showed that the materials produced by injection casting in both variants have higher corrosion potentials than a commercial alloy. The highest corrosion resistance has sample produced by the injection method with air pressure. The lower current density while higher potential corrosion, the higher corrosion resistance. Components produced by injection casting, are more resistant to corrosion in the environment of body fluids than components made from the alloy commercially produced.

Table 2.
Microhardness comparison of samples surfaces

Microhardness, HV0.1	Titanium alloy produced by injection casting with vacuum	Titanium alloy produced by injection casting with air pressure	Titanium alloy produced commercially - rod
	795.1	1225.9	387.2
	793.2	1230.3	360.5
	787.2	1218.2	324.1
	797.8	1232.7	338.8
	789.2	1245.9	340.1
Average HV0.1	792.5	1230.6	350.14

Table 3.
Microhardness comparison of samples in cross-sections

Cross-section	Microhardness HV0.1	
	Titanium alloy produced by injection casting with vacuum	Titanium alloy produced by injection casting with air pressure
Subsurface area	802.7	1243.9
	774.1	1060.3
Core	764.2	865.8
	770.4	996.7
Subsurface area	798.8	1248.5

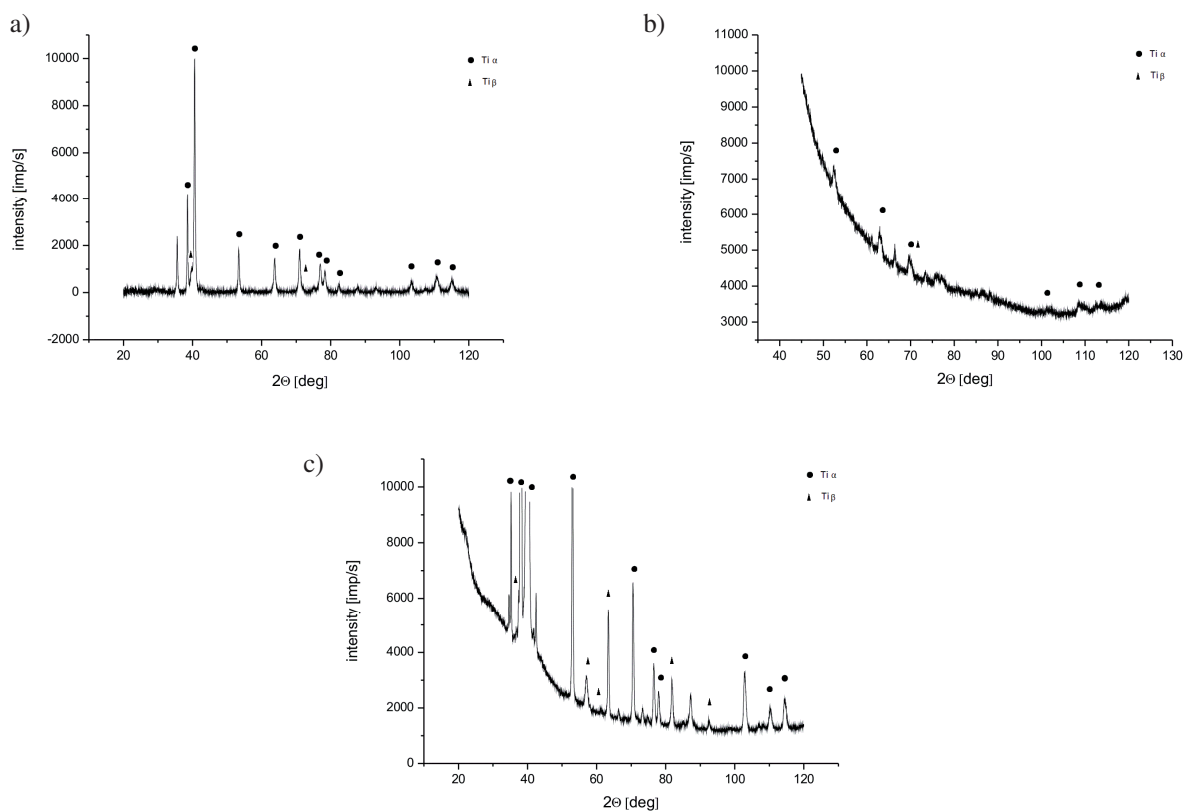


Fig. 3. Diffractions patterns of Ti6Al4V titanium alloys: a) commercial – rod, b) produced by injection method with vacuum, c) produced by injection method with air pressure

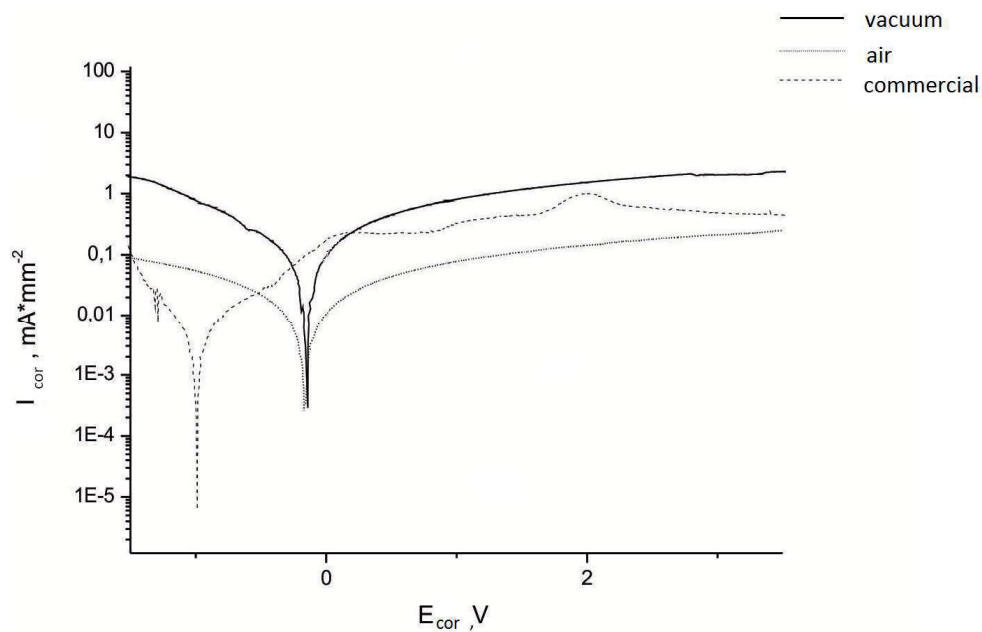


Fig. 4. Comparison of polarization curves for all samples

4. Summary

The method used for obtaining Ti6Al4V titanium alloy effects on the type of obtained structure and therefore the material properties. The injection casting method allows to obtain better mechanical and electrochemical properties. The method discussed in the article and resulted in the fragmentation of structures, which contributed to the mechanical properties – microhardness. The injection casting method with rapid cooling caused zonal structure in entire volume of produced materials. It is possible to identify a zone with fine grains at the interface between surface layer and copper mold is zone with pillar dendrites and equiaxed crystals. The zonal structure can provide hard surface and more ductile core what is important for elements that carry loads.

Micorhardness of the obtained materials was radically increased. The analysis showed that microhardness of the alloy cast by injection with air pressure has the greater microhardness. The lowest hardness is characterized by the alloy purchased commercially. Greater microhardness of the produced sample is achieved at the surface and decreases towards the core.

Qualitative X-ray analysis confirmed that after applying injection casting there have been structural changes but not phase. Diffraction pattern of all samples revealed peaks of the phases: Ti α and Ti β .

The electrochemical studies showed that the material produced by injection casting under gas pressure, has the highest corrosion potential. It allows to say that the method has a direct effect on the functional properties of produced components for medicine.

The casting method by injecting (under pressure of gas and vacuum) allows for the modification of the starting alloy structure. The resulting material has a much better mechanical and electrochemical properties than the material purchased commercially in the form of a rod. That method of production can compete for the currently used in particular for materials for medicine. In addition, it can be used in other industries.

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