Influence of the type of ceramic moulding materials on the top layer of titanium precision castings

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Abstract
The article presents the results of the research which was executed to describe the conditions specific of the formation of surface of certain micro-geometry and of the upper layers on precision titanium castings for medical industry. On the ready precision castings some tests were carried out to obtain the surface micro-geometry satisfying the requirements of implants to be used in human organism. The surfaces with specific spherical macro-unevenness were formed as well as plane surfaces of $2 \div 6 \mu m$ roughness. With the help of the light and electron microscopy, the possibility of formation of upper layers directly through an interaction of liquid titanium or Ti6Al4V alloy with the first layer of ceramic mould made from the ceramic mixtures based on Ekosil binder and molochite or zirconia has been confirmed.

Keywords: Innovative casting materials and technologies, endoprosthesis, titanium, investment castings, microstructure.

1. Introduction

Products made from titanium and its alloys are shaped mainly by various processes of plastic working. The data available in this respect indicate that in a very small percent only titanium and its alloys are processed by casting technologies. This situation is mainly due to various problems of the technical nature related with manufacture of castings, e.g. high melting point, high reactivity with refractory lining, susceptibility to formation of casting defects, etc. The need to produce items of very intricate configurations or highly developed surface geometry is, specially in the past few years, a challenge that both the waste-forming and wasteless technologies of fabrication are often incapable to face. This explains why modern casting technologies, investment casting - in particular, are commonly regarded as very useful, indispensable even, tools in manufacture of products not only for the automotive industry or aircraft, but also and mainly for the medical science and engineering.

Titanium castings for medical appliances are most popular in dentistry and orthodontics. Titanium and its alloys in various combinations with ceramics, plastics and/or composites are used for dental prostheses, fillers, fragments of orthodontic devices, and the like ones. There are, however, many other potential medical applications of titanium castings, to name just the orthopaedic surgery and cardiology.

However, castings of, e.g., titanium endoprostheses should meet a number of requirements regarding their physico-chemical properties, mainly engineering, mechanical and tribological, for which the upper level of optimising has already been reached with success. They should also reveal a high degree of resistance to biodegradation in the environment of human organism. Therefore, recently, so strong emphasis has been put on the engineering aspect of shaping the surface layer in products [1, 2, 3]. The data available in literature indicate that the adaptability of an implant in human organism will greatly depend on whether this implant has got on its surface a layer of proper macro- and microgeometry. The challenge of solving this problem is mainly the task of surface engineering, but - as proved in this paper - this is also the task of various casting technologies.
At the first stage of research, four types of implants to be made by casting methods were chosen. Three of them were the baskets of pan of the hip joint, implants of knee joint and the dental prosthesis (fig. 1a,b,c). The choice of items so specific was dictated by their different geometries, special requirements concerning surface topography and top layer.

In the research described in this article attention was focussed on the choice of implants geometry proper for execution of the research and preparation of physical patterns and test castings. At this stage, the surface condition of the wax patterns produced from the experimental implant patterns was analyzed. It has been assumed that the titanium casting top layers made in moulds based on special ceramic materials will be examined under the microscope. The aim of these examinations was to determine the possibility of controlled formation of the top layers in titanium castings through direct interaction with ceramic mould.

2. Methods of investigations

Selection of implants geometry presented in Figure 1 was dictated, first of all, by differences in their surface macro- and micro-geometry. The medical experiences show that the main task of surface geometry in these products should be fulfillment of the requirements presented in Table 1.

<table>
<thead>
<tr>
<th>Requirements for the selected implant surfaces.</th>
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<tr>
<td>basket of hip joint pan</td>
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<tr>
<td>possibly smooth surface of the implant of roughness $R_a &lt; 3 \mu m$ because of contact between “basket” and implant pan and biological pelvis bed</td>
</tr>
</tbody>
</table>

The investigations of surface properties were carried out at the Warsaw University of Technology using physical patterns, ceramic moulds and test castings made by Foundry Research Institute in Kraków. Patterns were prepared from Castaldo red jeweller’s wax, poured to silicone moulds at a temperature of 86$^\circ$C [4]. On the ready patterns, the ceramic moulds were formed in layers; the first layer was made of two types of the ceramic mixture:

a) mixture of ZrO$_2$ flour with Ekosil binder (ZrO$_2$/Ekosil);
b) mixture of molochite (Al$_2$O$_3$ + SiO$_2$) with Ekosil binder (the molochite/Ekosil).

The remaining layers of the ceramic moulds were based on Ekosil binder and silica sand of the grain size from 0,1 to 1mm. The wax was removed from moulds in an autoclave, and moulds were next baked for 2 hours at a temperature of 900$^\circ$C. Castings from pure titanium and Ti6Al4V alloy were made in Titancast 700 vacuum furnace, and after cooling in air were cleaned by sand blasting.

For examinations under the microscope were chosen fragments of precision cast endoprostheses (fig.1). The investigations focused on analysis of the structure of the casting top layer produced in ceramic moulds with two different mixtures used for the first, potentially reactive, layer of the mould. Those were the mixtures of Ekosil binder with ZrO$_2$ or with molochite. Castings were poured from two types of metal: pure titanium and Ti6Al4V alloy. The samples for examinations carried out by the light and electron microscopy, chosen from castings made in a suitable combination of moulding and casting materials, were marked by “x” in Table 2.

The examinations were made on OLYMPUS IX70 and OLYMPUS SZX9 light microscopes and on HITACHI 3500N
electron scanning microscope with EDS; the latter microscope was also used for analysis of the chemical composition of the investigated materials. The specimens for tests were embedded in phenolic resin with graphite filler, and then they were ground with abrasive paper of gradation #200 ÷ #2400 and polished with 3µm diamond suspension. The specimens were etched in Keller’s reagent.

Table 2.
Scheme of materials combination used in examinations by the light and electron microscopy.

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<tr>
<th>Materials used for the first layer of ceramic mould</th>
<th>Metal used for castings</th>
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<tr>
<td>ZrO₂/Ekosil</td>
<td>x</td>
</tr>
<tr>
<td>molochite/Ekosil</td>
<td>x</td>
</tr>
</tbody>
</table>

3. Results of examinations

Figure 2 shows exact reproduction of a fragment of the knee joint implant pin surface with characteristic, spherical unevenness. On the casting are visible, superfluous in this case, fine particles (smaller than the dimensions of the macro-unevenness), which prove the possibility of reproducing also these sizes of the unevenness from pattern surface. Casting of the plane pattern surfaces is not inconvenient, either. They are quite well reproduced in the casting and characterized by suitable roughness of 2÷6µm (fig.3).

It has also been proved that the casting has smaller roughness than the ceramic mould as a result of the previously documented [5] metal properties, which restrain the metal from wetting fully all the capillaries in a ceramic mould and thus reduce the casting surface roughness.

Figure 4 shows the chosen photographs of microstructures in the top layers of castings made from pure titanium and Ti6Al4V alloy. They indicate that the surface of castings based on ZrO₂ does not show the presence of distinct, structural products of reaction. Photographs b and c in Figure 4 illustrate the influence of molochite on the top layer of titanium casting microstructure. The top layer of different structures is visible in both titanium and Ti6Al4V castings.
Electron scanning microscope analyses show (figs.5 and 6) in these layers a variable content of different chemical elements diffusing from the ceramic mould inside the casting. For pure Ti in the top layer, a particularly raised content of aluminum and oxygen is observed (fig.5).

These results indicate a strong reaction of liquid Ti alloy with mould material based on Al₂O₃, ZrO₂, CaO. It is possible that these reactions, controlled in a suitable way, may contribute to conscious formation of a top layer on titanium castings.

4. Conclusions

From the conducted researches and the obtained results the following conclusions are drawn:

1. It is possible to make precision castings from pure titanium or Ti6Al4V alloy for items of complex shapes and complicated surface macro- and micro-geometry.
2. As a result of the conducted investigations the formation of a top layer in castings from pure Ti and Ti6Al4V alloy directly in the mould has been proved. The next, planned investigations should enable establishing the possibility of controlled formation of the top layer by a method of direct influence on the surface of casting through the first layer of a ceramic mould.

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References