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CARACTERIZATION OF A HARD SURFACE LAYER (»α CASE«) ON Ti-6Al-4V SURGICAL IMPLANTS

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ABSTRACT

Ti-6Al-4V alloy represents a relatively novel material used for biomedical applications such as surgical implants. Because of the complicated shape of implants the usual processing technique for their production was forging. With the use of a »selfsupporting« ceramic shell mold prototypes of Ti-6Al-4V-based implants were produced by a precision casting process in a centrifugal vacuum furnace, which largely reduces the costs of such products. Optical microscopy, X-ray diffraction technique and hardness measurements, in this study, were focused on properties of the hard surface layer ("α-case").

Keywords: (α + β) Ti-6Al-4V alloy, centrifugal vacuum casting, »α case«, hardness

1. INTRODUCTION

Titanium and its alloys are increasingly used as an implant material because of the combination of high specific strength, high corrosion resistance and good biocompatibility with the human tissue [1]. Among other titanium based alloys Ti-6Al-4V (α + β) alloy is actively used for the biomedical applications, such as hip, shoulder and knee implants, screws, plates, *etc*. [1]. However, processing of this material presents a great problem due to very high reactivity of titanium at high temperatures during melting and solidification. Therefore, production of surgical implants by precision casting process has to be performed in vacuum and argon protective atmosphere, and use of specific shell molds is requered. Nevertheless, examinations showed that inspite of the use of high vacuum, molten metal reacts with oxygen from the shell and creates a thin hard surface layer known as the »α case«. This surface phase is very hard and brittle and therefore presents a place of crack initiation, and is a »weak« point of the structure. For these reasons this phase must be removed before put into service.

In view of these facts, an attempt was made to examine the caracteristics of the »α case« (composition, area, hardness *etc.*), with the idea to find adequate process of its removal.

2. EXPERIMENTAL

Considering very high chemical reactivity of titanium and its alloys the conventional ceramic molds of silica and zirconia are unsuitable for use. Therefore, special attention must be paid to the development of new kind of ceramic shell molds.

In the present work, a conventional »lost wax« procedure was performed to fabricate »selfsupporting« ceramic shell molds which are registered as the original patent [2], (Fig.1a). In the next step an investment casting process was applied to produce a shoulder implant consisting of a stem and a socket. The charged stock of a »master alloy« (a commercial Ti-6%Al-4%V) was remelted in a »Linn« centrifugal furnace in vacuum, the entire system was then filled with argon, and the melt was poured into a previously preheated ceramic mold. Conditions during melting and casting were as follows: pouring temperature – 1700°C, preheated temperature of ceramic shell mold was varied between 500 and 800°C, speed of mold rotation – 200 rpm, vacuum during processing – 1Pa. The castings are shown in Figure 1b.

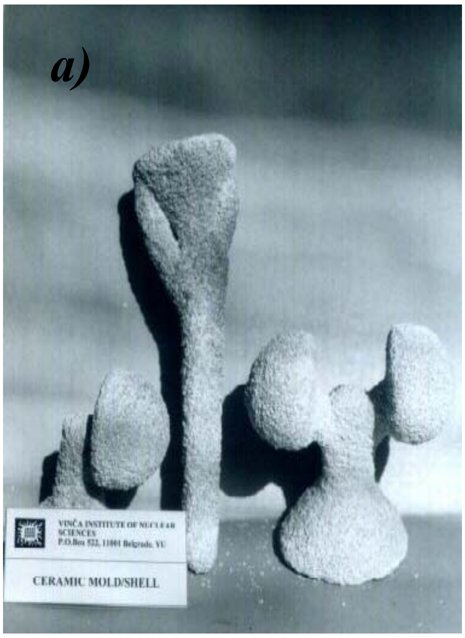

Fig. 1a

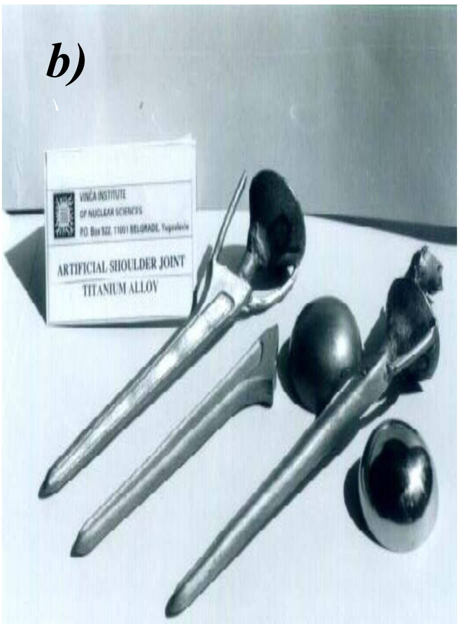

Fig. 1b

Fig. 1 - A shoulder implant consisting of a stem and socket.
a) ceramic shell molds and b) precision castings

X-ray diffraction analysis with Ni-filtered CuK_{α} radiation and light microscopy were used for microstructural characterization. Specimens for these examinations were cut out from the stem and shoulder socket. Kroll's reagent (a mixture of 6 ml nitric acid, 3 ml 40% hydrofluoric acid and 100 ml of distilled water) was used as an etchant for light microscopy. Vickers hardness (HV_{30}) was measured applying load of 30 kg. Microhardness was measured (with 100 g load) on the area starting from the surface to aproximately center of the specimen.

3. RESULTS AND DISCUSSION

Since the casting of shoulder implant is of a rather complicated configuration, the most promising technique was found to be a centrifugal casting. It should be noted that the first castings showed some defects such as micro and macro porosity when the preheating temperature of the shell mold was lower. However, by applying higher preheating temperatures these macro defects were successfully eliminated and a smooth surface was obtained.

Light microscopy examinations of the specimen, revealed a surface layer (»α case«) with the very coarse grains (Fig. 2). This structure largely differs from the structure of the inner part of the specimen consisting mainly of the α phase plates showing a characteristic Widmanstätten structure and a small fraction of the β phase. The »α case« layer spreads approximately 150 μm into the inner part of the specimen.

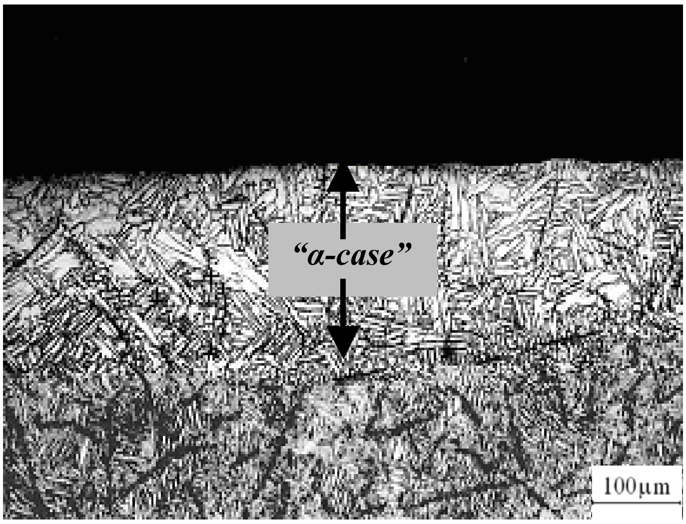

Fig. 2 - Light microscope. Microstructures of Ti-6Al-4V alloy in the as-cast condition.

X-ray diffraction analysis (Fig. 3a) of as-cast specimens from which the surface layer was removed proved the existence of the α phase with c.p.h. lattice (a = 0.29230 nm and c = 0.4672 nm, with c/a ratio of 1.59), β phase with b.c.c lattice (a = 0.3221 nm) and retained f.c.c. phase. However, in examining the surface layer significant changes in the X-ray diffraction patterns appeared. Beside the presence of the α phase, few peaks of f.c.c. phase were detected. According to the values of the lattice parameter of this phase (a = 0.427 nm) it could present TiC_x or TiOC. A new phase that was not still identified appears in the same pattern. Curiously, the examination of the surface of the specimen did not reveal the presence of the β phase, which is the one of the constituents of this alloy. This result leads to the conclusion that the »α case« is a stabilized α phase in which oxygen, and, possibly, a few other elements such as nitrogen and hydrogen, are dissolved. It should be mentioned that this assumption needs a further clarification.

Macrohardness measurements showed that the hardness of the »core« of the specimen was 320 $HV_{30,}$ but it was not possible to determine hardness of the »α case« due to its low depth. However, applying micro hardness measurements it becomes clear that hardness of the »α case« is aproximately 1,8 times greater than the core structure. This difference in hardness clearly explains why the removal of the layer is neccesary.

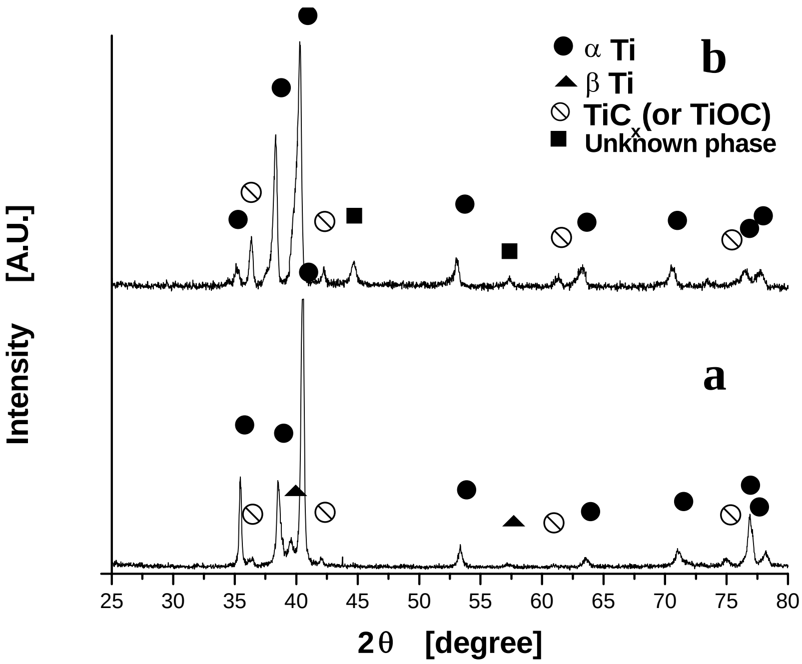

Fig. 3 - X-ray diffraction pattern of Ti-6Al-4V alloy specimen;
a) inner part of the specimen without the surface layer,
b) The surface of the specimen with the »α case« layer.

4. CONCLUSIONS

The processing technology of a »selfsupporting« ceramic shell mold has been successfuly verified during casting of surgical implants of Ti-6Al-4V alloy.

It was demonstrated that the prototypes of implants of Ti-6Al-4V alloy may be processed *via* investment casting using a centrifugal vacuum furnace.

Light microscopy examinations of the specimen, revealed a surface layer »α case« which largely differs from the remainder of the structure that consists of transformed β phase containing plates of α phase with a characteristic Widmanstätten structure.

The depth of the thickness of the surface layer was aproximately 150 µm.

X-ray diffraction analysis showed the great difference between the surface and the core structure, indicating that the »α case« is most probably oxygen sabilized α phase.

Microhardness measurements of the surface layer is aproximately 1,8 times greater then the core structure. Therefore, removal of the » α case« is neccesary.

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REFERENCES

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[2] Number of the patent application: 449/03.