



of Achievements in Materials and Manufacturing Engineering VOLUME 20 ISSUES 1-2 January-February 2007

# Metamorphosis quality preparing of alloy Ti64 in laboratory conditions

# M. Žitňanský\*, Ľ. Čaplovič, S. Demian

Slovak University of Technology in Bratislava,

Faculty of Materials Science and Technology, J. Bottu 25, Trnava, Slovakia

\* Corresponding author: E-mail address: zitnan@mtf.stuba.sk

Received 30.10.2006; accepted in revised form 15.11.2006

## Industrial management and organisation

# **ABSTRACT**

**Purpose:** The aim of our research was the developing of the method for preparing titanium alloy (Ti64 ELI) by remold in laboratory conditions on our research workplace. As a reason for writing the paper is to inform the technical society.

**Design/methodology/approach:** The objectives were achieved by using differently sources heating of remolded titanium alloy Ti64 ELI, by using of differently conditions by vacuum melting and pouring in to ceramics or copper moulds. As main method used for our research was remolding, purification, casting in the vacuum and than special heat treating by HIP processes. The quality of microstructure was investigated by electron microscopy and tested by Charpy impact test. The mean aim was to get microcastings of very intricate shapes and with very high quality of casting material. Through application four differently conditions of remolding we have found that in our workplace we have good ability to prepare the microcastings with very good quality, which is the main conclusion.

**Findings:** For expectation it is possible by using such a process for production special microcastings from Ti64 ELI alloy. The mean idea of this paper will have practical implications.

**Research limitations/implications:** In this time as a limitation is a little small capacity of plasma burner. **Practical implications:** The result of this paper should be made some changes in practice e.g. as savings of turning and lastly the using of Rapid Prototyping method.

**Originality/value:** The original value of our paper is the testimony above quality of alloy Ti64 ELI as cast. The quality of alloy Ti64 ELI as cast is comparable with certified Ti64 ELI from abroad. The reach a destination of outcome in our laboratory conditions is a perspective method for production of microcastings from alloy Ti64. **Keywords:** Metallic alloys; Preparing quality; Plasma furnace; Microcasting

# **1. Introduction**

The aim of this paper is, to acknowledge the technical public with results, gained in laboratory conditions on the Chair of material engineering. It includes the proposal of a method of manufacturing of veryaccurate micro-castings with a mass lower then 300 g of alloy Ti64 ELI. On our research work-place on the Slovak University of Technology we have – in comparison with the world-wide level certain possibilities of melting and casting of the mentioned alloy. We are speaking only of »certain possibilities«, because the theoretical capacity of the plasma burner is only 45 kW.

We present herewith the achieved results in our laboratory of plasma and vacuum metallurgy. From the aspect of physical metallurgy it is well-known that, in the course of treating of the alloy Ti64 ELI into its final shape of a micro-cast, it is necessary to eliminate series of potential physical-chemical reactions. When taking into consideration that, the chemical composition of the alloy Ti64 ELI consists of elements: Ti, Al, V, Fe, Y, C and in the liquid state there react with the metal also gases as O, N and H, it is clear that, the process of melting is extremely complicated. According to the used melting device, there can appear some, or all of the following physical-chemical eq. (1) to (6).

$$[Ti] + (O) = \langle Ti O_2 \rangle$$
 (1)

$$Ti] + (H) = \langle Ti_x H_2 \rangle$$
 (2)

$$[Ti] + (N) = \langle Ti N \rangle$$
(3)

(4)

$$[Ti] + \langle C \rangle$$
 =  $\langle Ti C \rangle$ 

$$[Ti] + \langle Al_2 O_3 \rangle) = \langle Ti O_2 \rangle + [Al]$$
(5)

$$x [Ti] + y < C > ) + z (N) = < Ti_x C_y N_z >$$
 (6)

Well equipped working places in the advanced part of the world already solved this problem [8 - 13]. From the world-wide known results about the physical-chemical process of melting results that, if we eliminate the shown six equations, we receive a metallic alloy of high quality. There remains only, to assure the required micro-structure by a suitable speed of crystallisation. Such is the most general requirement of good quality of the micro-cast.

# 2. Conditions of the applied experiments

For experiments, there was used imported certified alloy Ti64 ELI with a mass of a batch lower then 800 g. It was melted at various conditions in different melting aggregates as: melting and casting in a vacuum induction furnace DEGUSSA, melting and casting in a vacuum induction furnace with a special, extra high-speed melting of the whole batch, melting and casting in a graphite melting crucible with an induction heating or with an indirect induction heating, using an Mo cylinder, melting in a laboratory plasma- furnace in Cu crucible, in a horizontal or vertical arrangement, casting into a copper, non-cooled ingot mould, or into a ceramic form, using an additional heat processing, in conditions of HIP or in an atmosphere  $H_2$ .

#### **3.** Achieved results

#### 3.1. Preparation of material of casts

When melting in a graphite crucible, the micro-cast was of no good quality, in spite of the fact that, the preparation time of the liquid metal was shortened from usual 30 min to 3 min. It proved that, also in such a short time there was an extremely strong interaction between the wall of the melting crucible and the melted metal. The content of carbon in the titan alloy, due to the physical-chemical reaction according equation (4), increased from original concentration of 0,03 to 0,08 mass % to a value of 1,2 to 1,4 mass %. Such FCHR results in creation of a great amount of carbides of a < Ti C > type and the material becomes extremely brittle and fragile. The value of the notch toughness was lowered by 80 %, in comparison with the original state. This problem we succeeded to eliminate only after preventing completely the contact of the melted metal with the graphite.

We arranged the melting and casting processes in a way shown on Figure 1. Such arrangement of the manufacturing process of the micro-casts can be assured by a very pure material of the cast, without the influence of physical-chemical reactions No. (1) to (4). In our case, there remained limiting the heating capacity of the plasma burner, what causes that, into the Cu form there is poured the metal with a temperature by 100 to  $200^{\circ}$ C higher then the liquid temperature of the given alloy.

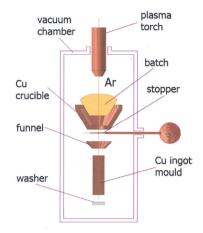


Fig. 1. Arrangement of the melting process in the laboratory plasma furnace, in a vertical Cu melting crucible [1, 2]

#### 3.2. Metallographic analysis of the manufactured material

Metallographic analysis of individual evolution stages of manufacturing a perfect cast from the titan alloy Ti64 ELI, documents the problems described above, when seeking the optimal technologic procedure. Samples of alloys were prepared by a classical metallographic procedure. To make the microstructure visible, we used etching action in a solution 3 ml HNO<sub>3</sub>, 1, 5 ml HF and 10 ml H<sub>2</sub>O.

As can be seen on Figure 2, the major problem of melting and casting from a graphite melting crucible, was the existence of large carbo-nitrides formations, placed in a double-phases lamellar  $\alpha$ + $\beta$  matrix.

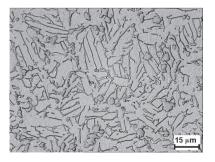


Fig. 2. Microstructure after the casting from a graphite melting crucible (melting 30 min) [1, 2]

Shortening of the interaction time of the melted metal with the graphite wall of the melting crucible from the usual 30 min to 3 min lowered the content of C, however, they continued to influence negatively the resulting mechanical properties of the alloy, Fig. 3. To improve the final properties of the alloy was not possible, even after the following treating in a hot isostatic press (HIP), with an original sequence of heat regime, neither by subsequent rolling. Making slimmer the lamellas  $\alpha+\beta$  in the matrix by the HIP process, admittedly increased the mechanical properties, however the carbo-nitride formations could not be transformed – Fig. 4. The strong plastic deformation during rolling only diminished the size of lamellas, Fig. 5 [14].



Fig. 3. Microstructure after casting from a graphite melting crucible (melting 3 min) [6]

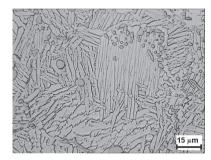


Fig. 4. Microstructure after casting from a graphite melting crucible, with a following HIP process [5]

Using of melting by an indirect heating on a ceramic underlay, in a vacuum furnace, or in a plasma furnace with a horizontal crystallizer eliminated the adverse influence of interactions of metalloids with the melted alloy. However, the high speed of coolingdown the hot-melt created conditions for rising of martenzitic structures with very fine needles  $\alpha' a \alpha''$ , Fig. 6 and 7 [15]. It was proved that this is a solution, which resulted in a metallurgical pure alloy without accessory negative phenomenon's, resulting from physical-chemical reactions. There remained only to solve the process of filling the form by a sufficient amount of melted metal. The adapted plasma furnace with a vertical crystallizer, described above, made possible to achieve the requested parameters.

From a microstructure point of view, the resulting casts were again created by fine needles of martenzitic phases  $\alpha$ ' a  $\alpha$ " and lamellas of the  $\beta$  phase displaced in the volume of original  $\beta$  phase, Fig. 8.

Subsequent application of the HIP process proved that, it is possible to eliminate the martenzitic structure by optimisation of the

heating regime, while maintaining the morphological characteristics of the  $\alpha$ + $\beta$  phases, Fig. 9.



Fig. 5. Microstructure after casting from a graphite melting crucible, with a subsequent rolling [3]



Fig. 6. Microstructure after melting and casting from a ceramic underlay (Degussa) [1,2]

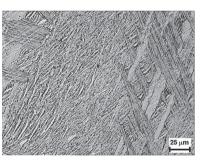


Fig. 7. Microstructure after casting from a plasma furnace with a horizontal crystallizer [4]

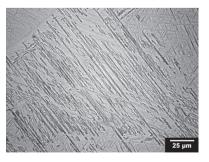


Fig. 8. Microstructure after casting from a plasma furnace with a vertical crystallizer [1, 2]



Fig. 9. Microstructure after casting from a plasma furnace with a vertical crystallizer, after HIP [1, 2]

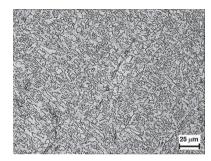


Fig.10. Microstructure after a special heating procedure in H<sub>2</sub> [7]

From the point of view of the resulting morphology, as well as from the final mechanic – technologic characteristics of the titan alloy Ti64 ELI it was proved that, for achieving of optimal properties, it is necessary the alloy after casting to process by a special sequence of heating in H<sub>2</sub>. The resulting microstructure has a suitable and favourable composition in size and morphology of lamellas of  $\alpha$ + $\beta$  phases, what leads to a final result of extraordinary favourable improvement of exploitation properties of this alloy, Fig.10.

## 4.Conclusions

In conclusion can be stated that, the planned goal of the research was achieved. The technology, proposed and verified on the research working place renders micro-casts of very high quality, which can be processed into the final shape, e.g. into small implants into the human skeleton. The result in the shown extent is original, applicable to requirements of practice and the process is also economically very advantageous. For broader exploitation of reached results in practice, it is necessary, to increase significantly the capacity of the plasma burner, or to elaborate further alternative of heating the batch by the plasma jet.

# Acknowledgements

This result was based on results of the grant No. APVT- 20-050 702 and VEGA SR 1/1081/04, where Prof. Dr. Ing. Marcel Žitňanský, Dr.Sc. is the responsible researcher, Doc. PhD. Ing. Ľuboš Čaplovič is the co-researcher and Ing. Svetozár Demian is reasearcher.

## **References**

- M. Zitnansky, L. Caplovic, Research and development of titanium alloy for implantats, Ministry of Education SR, VEGA, 1/1081/04-06.
- [2] M. Zitnansky, L. Caplovic, Development of method for preparation human body anatomical implantate, Grant APVT-20-050702, 04-06.
- [3] M. Zitnansky, M. Marek, I. Schindler and L. Caplovic, The influence of rolling parameter of Ti 6Al 4V alloy on notch toughness. Forming (2002), International Scientific Conference, Plasticity of materials Luhacovice, 2002, 339-344.
- [4] M. Zitnansky, M. Kursa, L. Caplovic, Influence of melting by plasma treatment on microstructure of Ti6Al4V, Tytan and its alloys, Czestochowa (2002) 211-220.
- [5] M. Zitnansky, L. Caplovic, Effect of the thermomechanical treatment on the structure of titanium alloy Ti6A14V, Journal of Materials Processing Technology 157-158 (2004) 643-649.
- [6] M. Zitnansky, L. Caplovic, cooperation with U\_R\_M in Chemnitz, BRD (2002).
- [7] M. Zitnansky, L. Caplovic, cooperation with A.A. Iljin, IMC – MATI-Medtech, Moskva (2002).
- [8] J. Matthew and Jr. Donachi, Titanium a Technical Guide, 2<sup>nd</sup> Edition, ASM International, The Materials Information Society 225.
- [9] M. Zitnansky, M. Kursa, L. Caplovic, Influence of melting by plasma treatment on microstructure of Ti6Al4V, Tytan and its alloys Czestochowa (2002) 211-220, ISBN 83-7193-183-2.
- [10] D.M. Brunette, P. Tengvall, M. Textor, P. Thomse, Titanium in Medicine. Springer-Verlag, Berlin, Heidelberg, (2001).
- [11] B.D. Ratner, A.S. Hoffman, F.J. Schoen, I.E. Lemons, Biomaterials Science 2<sup>nd</sup> Edition, Elsevier Academia Press, California – Lomdon, (2004).
- [12] M. Long, H. I. Rack, Titanium alloys in total joint replacement-a materials science perspective, Biomaterials 19 (1998) 1621 – 1639.
- [13] Material and Manufacturing Engineering and Technology COMMENT '2005. Journal of Materials Processing Technology 162 – 163 (2005) 209 – 214.
- [14] M. Zitnansky, L. Caplovic, M. Greger, The influence of rolling on the structure of Ti 6Al 4V. Proceedings of the 10th Jubilee international Scientific Conference, Achievements in Mechanical and Materials Engineering, 2001, 631-636.
- [15] M. Zitnansky, L. Caplovic, Effect of the thermomechanical – heat treatment on the structure of model titanium alloy Ti6Al4V, Proceedings of the 9th Jubilee international Scientific Conference, Achievements in Mechanical and Materials Engineering, 2000, 589-592.

570