

Processing of γ TiAl, by ceramic crucible induction melting, and pouring in ceramic shells

Joaquim Barbosa¹, C. Silva Ribeiro², Caetano Monteiro¹

¹ Universidade do Minho, Departamento de Engenharia Mecânica, Guimarães, Portugal
Tel: +351 253510220, Fax: +351 253516007, kim@dem.uminho.pt

² FEUP, Departamento de Engenharia Metalúrgica e Materiais, Porto, Portugal
Tel: +351 222041786, Fax: +351 222041792

Keywords: TiAl, induction melting, ceramic crucible, ceramic mould, investment casting

Abstract

The production of high quality titanium and titanium alloys castings is a difficult and expensive task. The main reasons for that are: the high melting point; the extremely high reactivity of titanium alloys against a large amount of elements (solid, liquids and gaseous) at high temperatures, with particular emphasis to oxygen. For this reason, traditional casting techniques and materials cannot be used, both for the melting and the moulding operations, and melting and pouring have to be performed under vacuum or inert gas. However, such demands are extremely important factors to the high cost of titanium castings, and one possible way to decrease it might be the use of traditional casting techniques, with slight changes both on materials and production equipments and procedures.

This paper describes the production technique of γ TiAl castings, using a multi-layered refractory crucible based on stabilized zirconia, and pouring in different refractory investment casting shells. Microhardness and residual elements concentration profiles, from the surface to the inside of castings, characterization of the metal-mould interface, and surface finish of samples will be presented and discussed, and results compared with the available bibliographic references, for this kind of alloy.

1. Introduction

Due to the high reactivity of titanium alloys, there is no material that does not react with the liquid metal, when melting those alloys. Although some ceramics might solve some of the problems, until now no researcher claimed to have produced absolutely clean castings.

There is no precise information on the practical use of ceramics to melt reactive materials. Although there are some insights, protected by patenting, very short public information has been released. It is believed that ceramic materials used in the production of melting crucibles should present some of the following characteristics [1]:

- Higher melting temperature, well above that of the maximum alloy processing temperature;
- It should not react with the melt during processing;

- It should resist thermal shock, because the final superheating of the melt should be done very quickly, due to the high chemical reactivity of the alloys. This procedure leads to very high thermal gradients between the part of the crucible in contact with the melt and the part that is not.

During the last years, the authors have done intensive research work on this field, on the aim of Praxis XXI project ref 3-3.1/CEG/1904/95, dedicated to the development of new processing and characterization techniques of TiAl alloys. Different kinds of crucibles and ceramic moulds have been evaluated on the production of TiAl castings, and results are published elsewhere [1, 4-7].

Among every tested material, and in accordance with some bibliographic references [2,3], some metallic oxides revealed quite good behaviour. Yttrium oxide and calcium oxide are quite stable facing liquid titanium alloys, but they are not 100% inert [1, 4-7]. Besides, their use as crucible materials presents some drawbacks, like the low thermal shock resistance of the crucibles. On the other hand, due to its high higroscopicity, calcium oxide is very difficult to manipulate, and yttrium oxide cost is almost forbidden.

Thermal shock resistance can be improved if fully or partially stabilized zirconium oxide crucible is used, but its chemical reactivity with the melt is higher. In this case, the final alloy usually reveals the presence of important levels of zirconium and oxygen, either in solution, or as oxides [1, 4-7]. The research work that has been developed so far shows that no material accomplishes the crucibles two main demands: chemical compatibility and thermal shock resistance.

Mould material selection faces the same problems as the crucible material selection. In this case, the chemical stability of the mould material plays a very important role, as the contact time between metal and mould at high temperature is very long, due to the poor thermal conductivity of the refractory materials. On the other hand, thermal shock resistance is not a crucial factor, as the high porosity of ceramic materials usually allows significant volume changes of the mould. However, dimensional stability of the moulds is quite important, even crucial, to the dimensional accuracy of the casting, which is a very important demand of near net shape titanium based castings.

2. Experimental technique

2.1 Melting operation

An approach to overcome the problem was evaluated by the authors, by using a ceramic crucible made of a high thermal shock resistant material, inside coated with a different compound, chemically stable, or slightly reactive with the titanium alloy, in order to decrease the probability of a metal-crucible interaction to occur. A Ti-48Al (at.%) alloy was selected for melting, prepared from commercially pure titanium and aluminium.

Melting was performed on a multi-layer crucible, ZrO_2 based, with an inside thin layer of Y_2O_3 that was applied using a technique similar to investment casting (figure 1). A commercially available yttrium oxide emulsion was used to produce the Y_2O_3 inside film. The multi-layer crucible was afterwards dried at 250°C.

The melting procedure was the same as used in previous work and was published elsewhere [1,4-6].

2.2 Mould production

Different moulding materials were evaluated (ZrO_2 , SiO_2 , Y_2O_3), by pouring centrifugally Ti-48Al in moulds based on those materials. Production of ZrO_2 , SiO_2 and Y_2O_3 ceramic shells was performed using traditional dip coating / stuccoing techniques. In order to decrease production costs, Y_2O_3 shells were in fact multi-layered ZrO_2 based shells, with an Y_2O_3 face coat, produced by using 2 face coatings of a Y_2O_3 slurry (consisting of colloidal Y_2O_3 + Y_2O_3 powder refractory) and external coatings made of ZrO_2 slurries and ZrO_2 refractory.

Shells were positioned inside a cylindrical metallic box, and sustained inside by filling the remaining space inside the box with sodium silicate/ CO_2 moulding sand (figure 2).

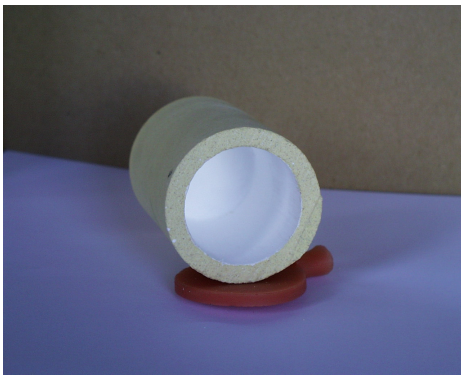


Fig.1 - ZrO_2 crucible inside coated with Y_2O_3

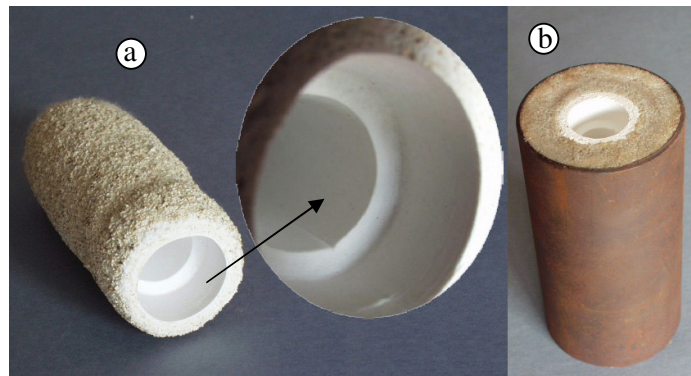


Fig.2 - Ceramic shell (a), and mould ready for pouring (b)

The entire processing (melting, pouring and cooling) was performed inside a sealed chamber, where a controlled atmosphere of commercial pure argon was maintained. The chamber was only opened after the sample reached room temperature.

2.3 Samples characterization

Cast specimens were cylinders 20 mm in diameter and 85 mm long, and were poured around 1600°C. Samples for characterization were collected from the middle section of them, by cutting the cylinders at half their height, and prepared using traditional metallographic techniques.

Experimental results include microstructure identification, chemical composition and microhardness values of each microconstituent, “alpha-case” characterization and surface roughness measurement. Phase and chemical composition identification were performed by quantitative EDS analysis with standards of pure Ti, Al and Zr, using a JEOL JSM 35C scanning electron microscope. Oxygen measurement was performed by Secondary Ion Mass Spectroscopy (SIMS) and IGF technique, and X-Ray diffraction was used to identify compounds present on the metal-mould interface. Microhardness was evaluated on a Shimadzu hardness tester, using a 50g load, for 15 seconds. The surface roughness of the samples was measured on a Perthometer S5P.

3. Experimental results

The influence of moulding materials on the contamination of Ti-48Al samples is very significant. Samples obtained in ZrO_2 and SiO_2 moulds reveal the presence of high levels of Zr and Si, especially on the surface region, and their concentration decrease to the inside of samples. The presence of Zr and Si is followed by the presence of oxygen, which concentration also decreases from the outside to the inside of the castings. The presence of such residual elements leads to an important increase of microhardness on the samples surface, with oxygen playing the most relevant role on that increase. In fact, oxygen concentration and microhardness variation profiles from the surface of samples, to the inside, are quite similar, and match almost perfectly, suggesting their close dependence. The extension/thickness of such “hard case”, usually called *alpha-case*, depends on the moulding material, and for the materials tested, it is maximal on castings obtained in SiO_2 shells (230 μm). At the metal-mould interface, different oxides were identified, with special relevance to Al_2O_3 (Figures 3 and 4). Detailed characterization and results discussion of samples obtained on ZrO_2 and SiO_2 moulds were published elsewhere (1, 7).

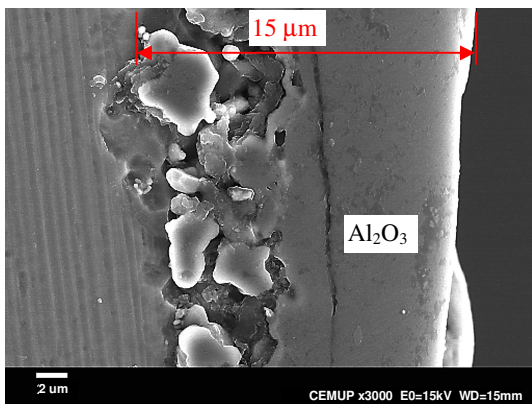


Fig.3 - Metal- ZrO_2 mould interface

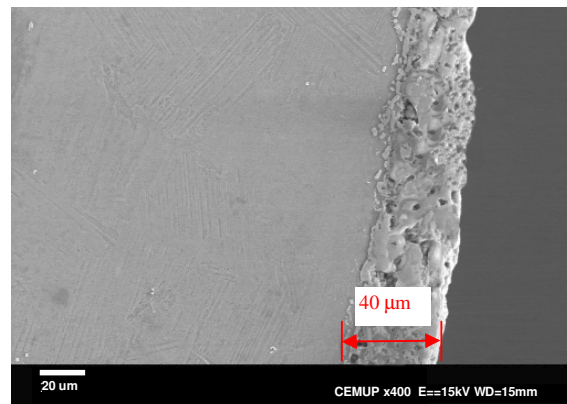


Fig.4 - Metal- SiO_2 mould interface

Regarding samples poured in multi-layered $ZrO_2 + Y_2O_3$ ceramic shells, the as cast microstructure contains two microconstituents: one with a strong dendritic pattern, with two phases ($\alpha_2 + \gamma$), and an interdendritic γ phase (figure 5), that is similar to the microstructure of samples obtained in ZrO_2 and SiO_2 moulds, and expected to Ti-48Al alloy [8,9].

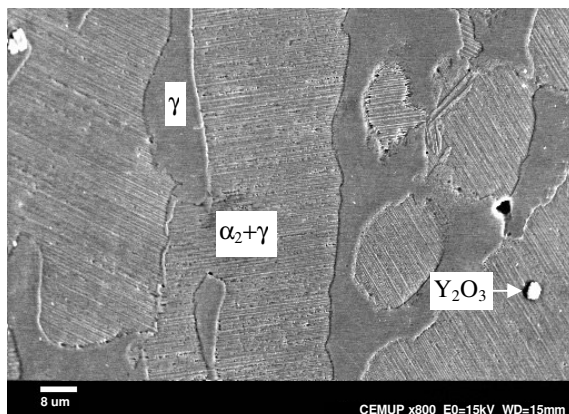


Fig.5 - Microstructure of a Ti-48Al sample poured in a multi-layered $ZrO_2 + Y_2O_3$ ceramic shell

The same samples (obtained in ZrO_2 shells with Y_2O_3 contact layer), reveal a small increase of Y and O contents near the casting surface (figures 6 a) and b)). This indicates that Y_2O_3 is not absolutely inert facing TiAl, and that some sort of metal-mould interaction does occur. Considering the free energy of formation of Y_2O_3 and Ti and Al oxides, it is not predictable any kind of reaction, as Y_2O_3 is much more stable than those oxides. Thus, the increase on the O and Y contents at the surface of the castings is thought to be due to the dissolution of the Y_2O_3 by molten Ti-48Al.

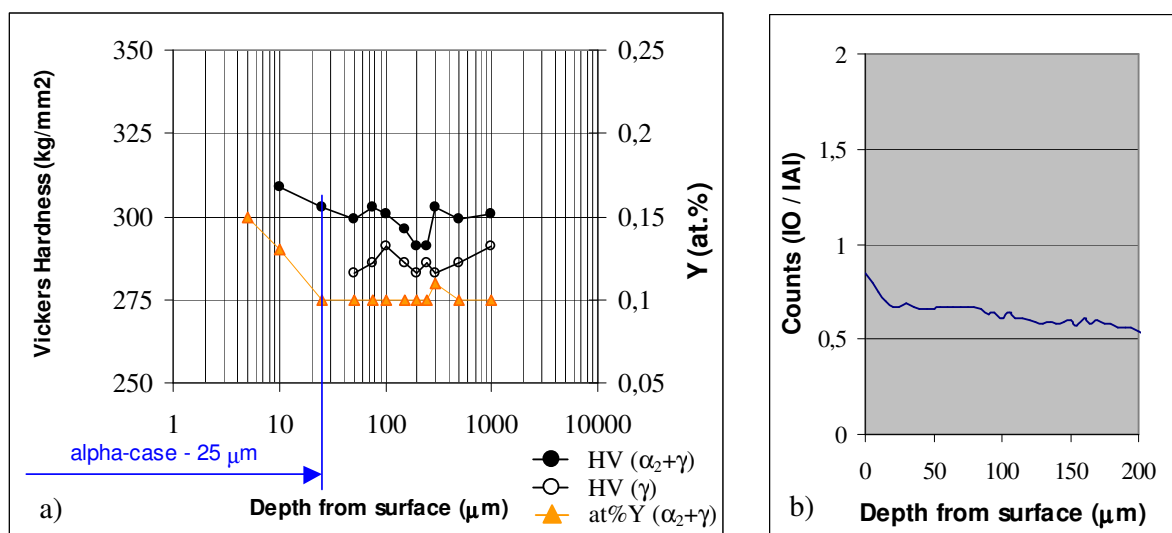


Fig.6 - a) Microhardness and Y concentration profiles and b) Oxygen concentration profiles, from the surface to the inside of a Ti-48Al sample poured in a multi-layered $ZrO_2 + Y_2O_3$ ceramic shell.

This interaction was formerly verified, by melting Ti-48Al in similar crucibles, and let the alloy solidify inside the crucible [1,7]. On those experiments, overall content of Y and O on the alloy was about 0,1 at.% and 0,25 wt.% respectively, and no sign of metal-mould reaction, with formation of different compounds at the metal-mould interface, was found.

On the samples poured in multi-layered $ZrO_2 + Y_2O_3$ ceramic shells, the average oxygen content of the alloy, measured by IGF (Inert Gas Fusion) technique is 0,29 wt%, and the Y average content is 0,11 at.%. These values are very close to those found on samples that cooled inside the crucible. This means that the moulding material dissolution only affects a very small thickness of metal (about 25 μm), leading to a slight increase in those elements content in the $\alpha_2+\gamma$ constituent (the Y content on the interdendritic γ phase remains constant all over the sample, and the solubility limit of oxygen on the same constituent is known to be about only 230 ppm [10]), as well as to a small increase in the $\alpha_2+\gamma$ constituent hardness (309 HV at 10μm from surface and 299 HV on bulk, in the $\alpha_2+\gamma$ constituent).

In what concerns metal-mould interface, a very thin film of Y_2O_3 (< 1 μm) adherent to the sample surface is present and no other oxides or compounds are detected (figure 7). However, the mechanical resistance of the Y_2O_3 crucible layer is not totally satisfactory, as some small particles of Y_2O_3 are present on the microstructure (see figure 5). Firing the crucibles at higher temperatures

than those used on this work might solve this problem, and it will be one of the main tasks for the future research work of authors on this area.

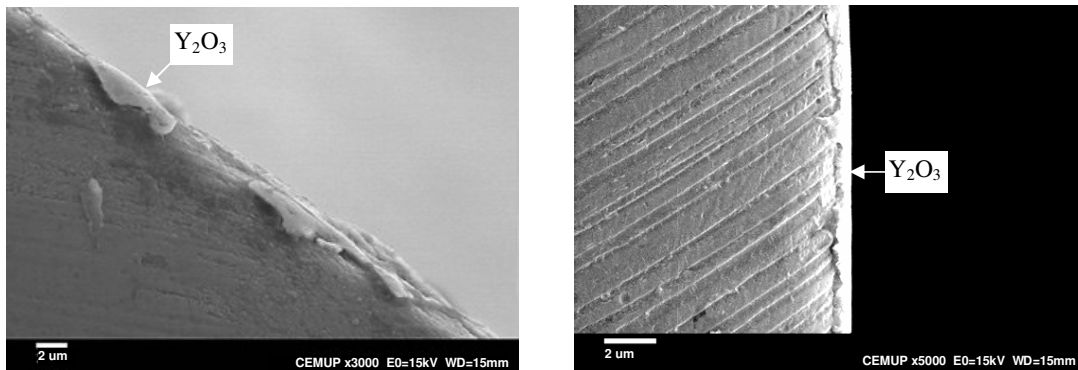


Fig.7 - Y_2O_3 film at the surface of a Ti-48Al sample obtained in multi-layered $ZrO_2 + Y_2O_3$ shell

Surface roughness was measured in different areas of the sample surface, and Ra has an average value of $1,6 \mu m$, which is similar to traditional investment castings. A segment of the roughness profile is presented on figure 8.

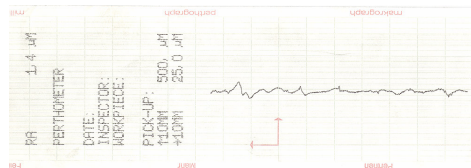


Fig.8 - Roughness profile of a Ti-48Al sample obtained in multi-layered $ZrO_2 + Y_2O_3$ shell

Conclusions

1. A new technique to produce TiAl castings was developed, by using a multi-layered $ZrO_2 + Y_2O_3$ melting crucible and pouring in a ZrO_2 investment shell, with an inside Y_2O_3 layer.
2. Using this technique, Ti-48Al castings with no more than 0,11 at.% Y and 0,25 wt.% O can be produced, with a surface finishing similar to steel investment castings.
3. Ti-48Al castings produced using the developed technique have a microhardness around 300 HV. However, microhardness might achieve slightly higher values at distances from the casting surface less than $25 \mu m$.
4. Castings produced using this process might present some non-metallic inclusions of Y_2O_3 , smaller than $3 \mu m$, on a volume fraction less than 1%.

References

- [1] J. Barbosa, *PhD thesis*, University of Minho, Portugal, 2001.
- [2] C. Frueh, *International Journal of Cast Metals Research*, Vol.9, n°4, 1996, p.233-240.
- [3] T. Sato, Y. Yoneda, N. Matsumoto, *58th World Foundry Congress*, Cracow, Poland, 1991.
- [4] J. Barbosa, *Key Engineering Materials*, Vol.188, 2000, p.45-54.
- [5] J. Barbosa, *International Journal of Cast Metals Research*, n°12, 2000, p.293-301.
- [6] J. Barbosa, *Key Engineering Materials*, Vol. 230-232 (2002), p. 106-109.
- [7] J. Barbosa, *Materials Week 2001 Congress*, Munich, Germany, October 2001.
- [8] M. Takeyama, *Structural Intermetallics*. (1993), p. 167-176.
- [9] A. Duarte, *Materials Research*. Vol. 2, n°3 (1999), p. 191-195.
- [10] A. Menand, *Acta Materialica*. Vol. 44, n° 12 (1996), p. 4729-4737.