Characterisation of metal/mould interface on investment casting of γ -TiAl

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This paper describes the evaluation of different refractory compounds – SiO₂, ZrO₂ and Y₂O₃ – as face coats of investment casting shells for γ -TiAl.

The effect of the different refractories on the metal–mould interaction is studied in different aspects. Experimental results include characterisation of the constituents present at the metal/mould interface, the segregation profiles of residual elements, namely oxygen, from the interface to the inner part of the samples, the extension and microhardness of the samples external hard case and the samples surface finishing.

Keywords: TiAl, Investment casting, Ceramic mould, Zirconia, Ytria, Silica, Graphite, Ceramic crucible

Introduction

The present application fields of titanium castings involve the production of high geometrical and dimensional precision castings, i.e. near net shape parts, demanding the use of high capability moulding processes and moulding materials with good thermal and chemical stability and refractoriness. During the last decades, ceramics aroused as the best moulding materials to produce near net shape castings, using the investment casting process. However, its use with titanium castings presents some drawbacks, owing to the high reactivity of the alloys, especially with oxygen, mainly at temperatures >500°C. Titanium alloys have low fluidity, so they require a significant amount of superheating, which increases the temperature at the metal/mould interface and consequently the alloy–refractory interaction effect.

Different phenomena can be associated to the metal–mould interaction¹ on investment casting, which effectiveness depends on the nature of the pair metal mould material and the pouring parameters. When casting titanium alloys using the investment casting process the most frequent phenomena is the reaction or/and dissolution of the mould materials by the molten metal. Immediately after pouring, the liquid metal starts to interact with the mould materials and may dissolve or/and react with them, depending on its thermodynamic stability and the kinetics of a possible reaction. On this phase, the interaction products may dissolve in liquid metal, leading to a quite uniform distribution of residual elements. When solidification starts, diffusion mechanisms become very slow and if the metal–mould interaction persists, then a gradient of composition may appear

from the surface to the inside of the casting. When a reaction occurs, the reaction products may appear at the casting surface (if they are not soluble in the liquid metal or their dissolution rate is very small), or they dissolve rapidly and they will not be detected (as it happens with TiO and TiO₂, that dissolve very fast in liquid titanium, increasing its overall oxygen content).

Titanium castings usually reveal a strong oxygen content variation, decreasing from the surface to the inner part of the casting, owing to the oxygen pickup from the mould wall and its diffusion to the inside, even after solidification starts. This variation is usually followed through a hardness variation under a similar profile, as it is well known the direct relationship between both parameters.^{1–4} In practice, titanium castings usually present a hard case at the outer surface, that some call ‘alpha-case’, which must be removed by ‘chemical milling’ before its industrial application.

This paper describes the work developed by the authors concerning the use of several moulding materials on the production of γ -TiAl samples by investment casting, using a multilayer technique, with the purpose to develop a moulding technique that could allow the production of castings without or with reduced extension of ‘alpha-case’.

Experimental technique

Materials selection

The selection of moulding materials was based on the experience formerly acquired by the authors to develop a melting crucible suitable to melt titanium based alloys.^{2,3,5} However, while melting crucibles are usually made of a single refractory material, moulding materials, namely those used on the investment casting process, include both a refractory and a binder, which must be inert against the melting alloy. Although the authors know that the extension of any reaction also depends upon the kinetic behaviour, for a particular system and temperature, the scarcity of data available take these experiments based only on the free energy of formation

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1 Ceramic mould used to pour Ti-48Al

of the simple oxides. According with the standard free energies of formation of metallic oxides, several oxides are thermodynamically more stable than those that might form from Ti and Al (mainly TiO_2 , TiO and Al_2O_3), namely MgO, CaO, ZrO_2 and Y_2O_3 , which may be a good choice to produce ceramic moulds. Although there are some good references to the use of CaO as crucible material for TiAl,^{6,7} it was not selected due to its high hygroscopicity and difficult handling practice. Owing to its low thermal shock resistance, also MgO was not evaluated. Work was carried on using ZrO_2 and Y_2O_3 , and SiO_2 moulds were also evaluated, for the sake of comparison.

Moulds production

TiAl test samples were cylinders 20 mm in diameter and 85 mm long. The production of ZrO_2 , Y_2O_3 and SiO_2 ceramic shells was performed using traditional dip coating/stuccoing techniques. In order to decrease production costs, Y_2O_3 shells were in fact multilayered ZrO_2 based shells, with a Y_2O_3 face coat, produced by using two layers 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 (Table 1). After firing at 1100°C during 4 h, shells were positioned inside a cylindrical metallic box and fixed by filling the remaining space inside the box with sodium silicate/ CO_2 moulding sand (Fig. 1).

Melting and Pouring

A Ti-48Al alloy was selected for melting, prepared from commercially pure titanium and aluminium. Melting was carried out by induction melting on a ZrO_2 based ceramic crucible, with an inside layer of Y_2O_3 , and moulds were centrifugally poured $\sim 1600^\circ\text{C}$, according to a technique described elsewhere.^{2,5} The entire processing was performed inside a sealed chamber, where a controlled atmosphere of commercial pure argon was maintained.

Samples characterisation

Samples for characterisation were collected from the middle of the cast cylinders, by sectioning them at 50%

2 Microstructure of Ti-48Al sample cast in ZrO_2 mould, revealing traditional $\alpha_2 + \gamma/\gamma$ microstructure and a few yttrium oxide inclusions

of their height. They were prepared using traditional metallographic techniques: surfaces were mechanically polished and etched with Kroll solution (2%HF, 4% HNO_3 , 94% H_2O). Phase identification and chemical composition were performed by quantitative EDS analysis with standards of pure Ti, Al, Zr and Y using a JEOL JSM 35C scanning electron microscope. A 10 keV potential and a 100 s acquisition time were used. Overall oxygen content was measured by the inert gas fusion (IGF) technique and secondary ion mass spectrometry was used to determine the diffusion profile of oxygen^{9,10} from the surface to the inside of the samples. X-ray diffraction (XRD) was used to identify compounds present on the metal/mould interface. Microhardness was evaluated on a Shimadzu hardness tester, using a 50 g load for 15 s. The surface roughness of the samples was measured on a Perthometer S5P.

Experimental results

The microstructure of every cast sample contains two microconstituents: one with a strong dendritic pattern, with two phases ($\alpha_2 + \gamma$) and a γ interdendritic phase (Fig. 2). The dendritic constituent is present in a higher quantity in every sample and represents an average volume fraction $\sim 90\%$, which is a little lower at the samples surface, following the increase in the cooling rate. Some yttrium oxide inclusions can be found dispersed on the metallic matrix (Fig. 3), which origin it is believed to be the erosion of the Y_2O_3 coating of the melting crucible during melting.

ZrO_2 mould

Concerning the samples chemical composition, no Zr was found in the lamellar constituent, but the γ interdendritic phase reveals a high Zr content at the sample surface (0.51 at.-% at 25 μm from the surface and decreases up to 0.32 at.-% at 50 μm from the same,

Table 1 Composition of ceramic shells used on this work

Face coatings				Backup coatings		
Slurry				Slurry		
	Binder	Refractory	Refractory		Refractory	Refractory
ZrO_2	Colloidal ZrO_2	ZrO_2	ZrO_2	Colloidal ZrO_2	ZrO_2	ZrO_2
Y_2O_3	Colloidal Y_2O_3	Y_2O_3	Y_2O_3	Colloidal ZrO_2	ZrO_2	ZrO_2
SiO_2	Colloidal SiO_2	SiO_2	SiO_2	Colloidal SiO_2	SiO_2	SiO_2

3 Spectrum (EDS) of Y_xO_y inclusion referred in Fig. 2

becoming lower than the equipment detection limit (0.10 at.-% at higher distances). The cast alloy reveals a small contamination with yttrium (0.1 at.-%), owing to the fact that the melt was performed in a Y_2O_3 coated ceramic crucible, which is in accordance with results previously obtained by the authors.^{2,3,5} The overall oxygen content of the alloy, measured by IGF, is 0.27 wt.-%. Considering the oxygen content of the elements of the melting charge, the estimated oxygen content on the original melting charge is ~0.15 wt.-%, and the increase on the oxygen content due to the melting operation is ~0.15 wt.-%.^{2,3} As the oxygen content of the alloy is only 0.27 wt.-%, it can be assumed that the use of ZrO_2 as mould material does not affect the oxygen overall content of the sample.

The samples microhardness has a constant mean value of 289 ± 7 HV on the γ interdendritic phase, all over the sample. However, on the lamellar constituent, it increases from 301 ± 5 HV at distances from the sample surface $>75 \mu m$, up to 362 HV at 25 μm (Fig. 4), resulting on an 'alpha-case' extension of 75 μm for this cast sample. The hardness increase on the $\alpha_2 + \gamma$ constituent at the sample surface is believed to be caused by the increase of the alloy oxygen content in the same region (Fig. 5). In fact, the oxygen concentration and the $\alpha_2 + \gamma$ microhardness variation profiles from the surface of samples, to the inside, are quite similar and match almost perfectly, suggesting their close dependence (the oxygen concentration decreases from the sample surface to the inside of it and becomes almost

5 Oxygen concentration profile from surface to inside of Ti-48Al sample cast in ZrO_2 mould

constant, although revealing some fluctuations, at distances from the surface higher than those on the interval between 75 and 100 μm). This effect is only detected on the lamellar constituent because in γ -TiAl alloys the oxygen fixes essentially on the α_2 phase where its solubility limit is ~21600 ppm, whereas in the γ phase that value is only 230 ppm.^{10,11} Thus, the oxygen content variation curve obtained by secondary ion mass spectrometry (Fig. 5) represents in fact the variation of the oxygen content on the α_2 phase with the distance at the sample surface and it can be qualitatively correlated with the $\alpha_2 + \gamma$ microhardness variation curve for the same region.

Figure 6a shows the microstructure of the sample surface (metal/mould interface) and clearly reveals the presence of an oxide film (continuous on its outside and discontinuous on its inside), ~15 μm thick, that the EDS spectrum identifies as an aluminium oxide and the XRD spectrum of the sample surface region (Fig. 6b) suggests be Al_2O_3 . According to the Ellingham diagrams (Fig. 7), it is not expected that both Ti and Al might reduce the ZrO_2 mould material as, at the pouring temperature, this compound is thermodynamically more stable than any Ti and Al oxide. Although, when the molten metal enters the mould cavity it may dissolve the ZrO_2 ,^{12,13} and the diffusion of Zr and O in the alloy starts. When solidification starts, the ZrO_2 dissolution rate becomes much slower, as well as the diffusion mechanisms, leading to the Zr and O concentration

a $\alpha_2 + \gamma$ constituent; b interdendritic γ phase

4 Microhardness and Zr concentration profiles from surface to inside of Ti-48Al sample cast in ZrO_2 mould

6 a microstructure of surface (metal/mould interface) of Ti-48Al sample obtained in ZrO_2 mould, where Al_2O_3 layer can be seen and b XRD spectrum obtained at surface of sample cast in ZrO_2 mould, revealing presence of Al_2O_3

gradients illustrated in Figs. 4 and 5. Owing to the decrease in the diffusion rate, the O content at the interface is very high during this period and leads to the formation of Al oxides at the casting surface. As the sample temperature drops, the diffusion of Zr and O stops, but as the oxygen atom is much smaller than the Zr atom, the diffusion depth of this element (50 μm , Fig. 4) is significantly smaller than the O diffusion depth (100 μm , Fig. 5). When temperature reaches 750°C and the sample is already solidified the thermodynamic stability of Al_2O_3 and ZrO_2 is inverted and Al_2O_3 becomes more stable than ZrO_2 for lower temperatures (Fig. 7). For this reason, a reaction between the mould material and the aluminium from the TiAl alloy is possible to occur and continue as temperature is dropping, with formation of Al_2O_3 (according to the reaction (1)), leading to the development of a perfectly continuous oxide film, as illustrated in Fig. 6a. On the other hand, such reaction might explain the decrease on the Al content at the casting surface revealed by the EDS analysis (at 15 μm from the casting surface, the Al content of the lamellar constituent is 40.15 wt-%, but the average value on that constituent all over the sample is 46.3 wt-%)

A thin layer of moulding material can be seen adherent to the sample surface, which is easily removed

7 Standard free energy of formation of several oxides, including TiO_2 , showing inversion on relative stability of ZrO_2 and Al_2O_3 at temperature $\sim 750^\circ\text{C}$

with a finger. Average roughness of the sample surface is 2.1 μm (Fig. 8).

Y_2O_3 mould

Samples obtained in Y_2O_3 coated ZrO_2 based moulds reveal a small contamination with Y and O near the surface, as illustrated in Figs. 9 and 10, revealing that the yttria film is not absolutely inert against the molten alloy. According to its thermodynamic stability, it is not predictable that Y_2O_3 might be reduced by the TiAl alloy, what suggests that the presence of Y and O at the casting surface is due to a slight dissolution of the coating by the molten alloy. The oxygen overall content of the sample, measured by the IGF technique, is 0.29 wt-% and the yttrium content is 0.11 at.-%, which result from the melting operation itself, as verified in previous work.^{1,2} This fact means that the moulding material dissolution only affects a very small thickness of metal (25 μm as illustrated in Figs. 9 and 10, where the Y and O contents become a little higher than the average values inside the sample).

Inside the samples average microhardness values are 299 ± 8 HV on $\alpha_2 + \gamma$ and 291 ± 5 HV on the γ

8 Surface roughness profiles of Ti-48Al samples cast in a ZrO_2 mould, b Y_2O_3 mould and c SiO_2 mould

9 Microhardness and Y concentration profiles from surface to inside of Ti-48Al sample cast in Y_2O_3 coated zirconia mould

interdendritic phase. Microhardness values are a little higher at the samples surface, on the $\alpha_2 + \gamma$ microconstituent (309 HV at 10 μm from the surface), but no significant change was detected on the γ interdendritic phase (Fig. 9). $\alpha_2 + \gamma$ microhardness and oxygen concentration variation profiles from the surface to the inside of samples are similar and match quite well, suggesting once more, its close dependence. The 'alpha-case' extension is 25 μm on these samples and agrees with values referred by Feagin¹⁴ for Ti-6Al-4V samples obtained in Y_2O_3 moulds, although the author refers peak values of 2.5 μm .

Figure 11 shows the sample microstructure at the surface (metal/mould interface), where a thin oxide film ($<1 \mu m$) that the EDS spectrum (Fig. 12) suggests be Y_2O_3 detached from the mould surface can be seen, but no change in the matrix microstructure is evident. The thin Y_2O_3 film is not adherent to the metal and can be easily removed with a finger. The sample reveals a completely clean surface, without adherent mould material. The surface roughness was measured in different areas and the average Ra is 1.6 μm (Fig. 8), which is similar to traditional investment castings.

10 Oxygen concentration profile from surface to inside of Ti-48Al sample cast in Y_2O_3 coated zirconia mould

11 Microstructure of surface (metal/mould interface) of Ti-48Al sample obtained in Y_2O_3 coated zirconia mould, showing thin adherent layer of Y_2O_3

SiO₂ mould

Samples cast in SiO_2 based investment shells reveal as expected a significant contamination with Si at the surface, in both microconstituents (Fig. 13). In $\alpha_2 + \gamma$, the Si content decreases from a value of 0.53 at.-% at 25 μm from the samples surface to values <0.1 at.-% at distances $>100 \mu m$. In the γ interdendritic phase, the Si content is slightly higher, as well as its diffusion depth. Si content decreases from 0.83 at.-% at 25 μm from the surface until values <0.1 at.-%, at distances $>150 \mu m$. Although the alloy contamination is quite important, the Si concentration is lower than referred by other researchers when a Ti alloy is allowed to cool to room temperature in contact with SiO_2 , either as refractory material or as a binder.¹³ As it happens in every other samples, the alloy presents a contamination with Y ~ 0.1 at.-%, which is due to the melting operation, as explained in sections ' ZrO_2 mould' and ' Y_2O_3 mould'. The oxygen concentration profile from the surface to the inner part of the samples shows a similar development to the profile detected in those samples cast in Y_2O_3 and ZrO_2 moulds, although the diffusion depth is much higher in this case – 250 μm (Fig. 14). The difference detected in the oxygen diffusion depth in different mould materials can only be explained by different dissolution rates/kinetics of each mould material in the molten alloy. In fact, cooling time/speed is similar in every cast sample and does not change much between the mould materials that were used.

Figure 15 illustrates one sample microstructure at the surface (metal/mould interface), where an oxide layer with thickness between 20 and 50 μm , can be seen. The

12 Spectrum EDS of Y_xO_y layer present at surface of Ti-48Al sample cast in Y_2O_3 coated zirconia mould

a $\alpha_2 + \gamma$ constituent; b interdendritic γ phase13 Microhardness and Zr concentration profiles from surface to inside of Ti-48Al sample cast in SiO₂ mould 14 Oxygen concentration profile from surface to inside of Ti-48Al sample cast in SiO₂ mould 15 Microstructure of surface (metal/mould interface) of Ti-48Al sample obtained in SiO₂ mould, showing presence of oxide layer 40 μm thick

oxide layer is mainly an aluminium oxide (probably Al₂O₃) (zone 2 in Figs. 16 and 17), although other complex Ti and Al based oxides (zone 3 in Figs. 16 and 17) and other Ti and Si compounds (zone 4 in Figs. 16 and 17) were a small quantity of oxygen may also be present (zone 4 in Fig. 17), are also detected by the SEM/EDS spectrums of different zones of the layer. The poor thermodynamical stability of SiO₂ enables the possibility of several metal-mould reactions to occur, involving either Al or Ti, which usually lead to the formation of oxides and other compounds, according to reactions (2)–(5), for example

Close to the oxide layer, the Al concentration is lower than the average value of the sample (41 at.-% against 46.8 at.-% in $\alpha_2 + \gamma$ and 49.8 at.-% against 53.9 at.-% in the interdendritic γ phase). In some areas, the Al content is even lower, as in zone 1 of Fig. 16, where a Ti rich phase is present (68.3Ti, 31Al, 0.6Si, 0.1Y (at.-%)). The low Al content of those areas and the nature of the oxide layer suggest that the metal-mould interaction at the samples surface involves mainly reactions between Al and the mould materials, with Ti playing a secondary role on the interaction. This might be explained by the higher stability of Al₂O₃ when compared with TiO₂, the most stable Ti oxide, that avoids, or makes more difficult, the reduction of the mould material by the titanium present on the metallic alloy.

Microhardness decreases from the surface to the interior of the samples, assuming constant values at different distances from the surface, in each microconstituent (Fig. 13). In $\alpha_2 + \gamma$ microhardness decreases from 412 HV at 25 μm from the sample surface until values around 300 HV at distances from the surface

16 Microstructure of surface (metal/mould interface) of Ti-48Al sample obtained in SiO₂ mould, showing presence of thick complex oxide layer

17 Spectrums (EDS) of zones 2, 3 and 4 referred in Fig. 16

>250 μm . In the interdendritic γ phase, microhardness decreases from 316 HV at 25 μm from the surface to 290 HV at distances >50 μm . Owing to such variation, the 'alpha-case' extension in these samples is $\sim$ 250 μm , although it is much lower than that referred by Wictorin¹⁵ for commercially pure titanium cast in SiO_2 based moulds (500 μm). Although the presence of oxygen seems to be the main factor to contribute to the hardness increase in $\alpha_2 + \gamma$, a possible contribution of the Si presence to such increase can not be excluded. However, its effect can not be confirmed or evaluated, as available data is not enough. In the interdendritic γ phase, where oxygen has no influence in microhardness (as explained in section 'ZrO₂ mould'), Si segregation and microhardness profiles do not match, although their behaviour is similar – Si concentration becomes constant at distances from surface >150 μm , but microhardness values become constant at distances >75 μm . The Si effect in microhardness is not known, however, such results suggest that for Si contents <0.5 at.-% (concentration at 75 μm from the sample surface) its effect in microhardness can be neglected.

A layer of moulding material can be seen adherent to the sample surface, which removal is only possible by using a mechanical action. In some areas where roughness was possible to be measured, Ra was found to be 4 μm (Fig. 8), although this value is not representative of the overall surface condition.

Conclusions

1. The influence of the mould material in the contamination of Ti–48Al castings obtained in ceramic moulds is important. However, such contamination occurs only at the casting surface, until a distance from the surface that depends on the mould material and solidification time.

2. The main contaminants are oxygen and the metallic element present at the oxide used as mould material – Si, Zr or Y.

3. The chemical contamination leads to an increase in the casting microhardness, according to a profile that matches the segregation profile of oxygen in the same region.

4. The best results are achieved in moulds made of zirconia, with an inside (contact) coating of Y_2O_3 . On samples obtained by using such moulds, the 'alpha-case' extension is 25 μm and the microhardness at the surface

is 306 HV (maximum value), which agrees with the available references for the Ti–48Al alloys. Samples do not show evidence of metal–mould reaction and only very some small residues of mould material are detected adherent to the samples surface.

5. Casting in ZrO_2 moulds leads to the formation of an Al_2O_3 layer at the samples surface, $\sim$ 15 μm thick. Also a significant increase on microhardness in areas close to the surface is detected, which reaches a peak value of 362 HV, leading to an 'alpha-case' extension of 75 μm .

6. Using SiO_2 moulds leads to the formation of a complex oxide layer between 20 and 50 μm thick, at the casting surface. A significant increase in microhardness on the same region leads to an 'alpha-case' extension of 250 μm , where microhardness values of 412 HV can be found.

7. The surface finishing of the cast samples also depends on the mould material, although its variation is not very significant. The best results correspond to those samples cast in Y_2O_3 coated zirconia moulds, which surface roughness is $\sim$ 1.6 μm , followed by samples obtained in ZrO_2 ($R_a = 2.1 \mu\text{m}$) and SiO_2 moulds ($R_a = 4 \mu\text{m}$).

8. The results obtained in this work suggest that it is possible to cast titanium aluminides with very low contamination and good surface finishing if Y_2O_3 is used as face coat of the moulds. The use of ZrO_2 and SiO_2 as mould material for the same purpose is not possible, because metal–mould reaction lead to high hardness values at the castings surface, chemical contamination of the alloy and surface oxidation.

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