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Investigation into the use of CaZrO3 as a facecoat material in the investment casting of TiAl alloys

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HIGHLIGHTS

• To determine the interactions between CaZrO3 filler and stucco materials during shell firing.
• To study the reaction between the CaZrO3 facecoat and TiAl alloy during casting.
• The Al2O3 stucco can react with CaZrO3 filler to form (Zr, Ca)O2 and CaAlxOy at 1650 °C.
• O, Zr and Si ions from the ceramic moulds were observed to penetrate into the TiAl metal.
• The reaction products include (Ti, Zr)5(Al, Si)3 and ZrAl2 phases with high Ti ions solid solution.

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ABSTRACT

Research was carried out to determine the interactions between the filler and stucco materials in CaZrO3 based facecoats during shell firing as well as between the facecoat and a TiAl alloy during the casting process. A ‘flash re-melting’ technique, which gives a similar heating profile to the actual investment casting process, was used to study the phase transformations in the shell moulds. The chemical inertness of the facecoat was then investigated using a sessile drop test using a Ti-45Al-2Nb-2Mo-0.2TiB alloy. In this study, the facecoat compositions and the interaction products between metal and shells were characterized using x-ray diffraction, scanning electron microscopy (SEM) and transmission electron microscopy (TEM). A severe interaction was found between CaZrO3 filler and Al2O3 stucco, which rapidly damaged the shell surface. As well as oxygen, zirconium and silicon ions from the shell moulds were also observed to penetrate into the TiAl metal to form (Ti, Zr)5(Al, Si)3 phases in the metal/shell interfacial areas.

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1. Introduction

Gamma titanium aluminides are a family of low density, high performance alloys with the potential to replace current Ni-base superalloys used in the production of aero-engine components. Investment casting is one of the most economical methods to produce titanium and titanium aluminide alloy products, increasing the components' integrity and mechanical properties, whilst reducing material waste and machining cost [1]. Titanium aluminides are difficult to process mainly due to the low fluidity of the TiAl alloy around its melting temperature [2]. Due to the high affinity of elements such as oxygen, nitrogen etc., titanium and its alloys can easily interact with mould materials during the investment casting process, resulting in an interaction hardened layer being generated at the metal surface [3,4]. This hardened layer contains a large amount of dissolved oxygen, and it is very brittle and susceptible to crack generation and propagation [5].

The research [6–8] shows that facecoat materials such as ZrO2 and Al2O3 have acceptable chemical inertness with interaction layer thicknesses around 200–300 μm. When using Y2O3 as a facecoat material, the interaction layer thickness was greatly reduced to around 10 μm [8–10], therefore, it has been used in the investment casting of these reactive alloys since the 1990s [11]. Although yttria has excellent chemical inertness, it has a number of drawbacks in a production environment; combining high material cost, short slurry life and poor sintering properties. Some
alternative, cheaper facecoat materials based on nitride compounds such as AlN and BN have been studied [12,13] but the poor chemical inerte of these materials made them unsuitable for use in TiAl alloy casting.

From 2001 to 2004, Kim et al. [14,15] demonstrated the use of CaZrO3 based facecoat materials to cast Ti alloys. The results showed that the CaZrO3 facecoat had comparable chemical inertness to ZrO2, with a hardened layer thickness around 200 μm, at a lower cost than yttria based systems. He also reported that the order of chemical inertness of various facecoats as Al2O3 < ZrO2 < CaO < CaZrO3 when casting Ti64. Hence CaZrO3 is a potential facecoat material for casting titanium and its alloys. Later, Li [16] developed a CaZrO3 crucible by sintering a ZrO2 and CaO powders (CaO: ZrO2 = 1:1). In Li’s study, an approximate 350 μm interaction layer thickness was detected at the Ti—6Al—4V (Ti64) alloy surface with some oxides apparent. In 2012, a study was proposed to use CaZrO3 as filler material in the facecoat to cast titanium alloys [17].

However, most of the studies referenced above were based on the analysis of the chemical inertness of CaZrO3 against Ti64 or pure titanium alloys. Due to increased interest in titanium aluminide alloys in the aerospace industry, this study initiated to enhance the understanding of the chemical inertness of CaZrO3 as a facecoat material against molten TiAl alloys. Both Al2O3 and ZrO2 were used as stucco materials, and the interaction between stucco, filler and backup coats were characterized using SEM and EDS. The reactions between alloy and mould were investigated using a sessile drop method [18], and the interaction phases at the metal mould interface areas were identified using transmission electron microscopy (TEM).

2. Experimental techniques

2.1. Sample preparation

The details of the individual primary and secondary mould layer constituents are listed in Table 1. The moulds were made by first inverting the wax pattern with the primary slurry which consisted of a colloidal ZrO2 binder (Remet, Ti-coat), CaZrO3 filler (ABSCO, -325mesh), liquid polymer (EVO STIK, PVA), wetting agent (Remet, Victawet 12) and anti-foam (Remet, Burst RSD-10). Either Al2O3 (Cermatco, Al2O) or monolithic ZrO2 stucco (UCM 30/60 mesh) was then applied to form the primary facecoat. This coat was dried at a temperature of 21 °C, 50% relative humidity and 0.2 m/s air speed for 24 h. Six backup coats were then applied. An alumino-silicate stucco (IMERYS, Molochite 30/80 mesh) was applied as the secondary stucco in layer 2 and three. A coarser alumino-silicate stucco (IMERYS, Molochite 16/30 mesh) was applied as secondary stucco in layers 4 to 7. Each secondary coat was dried at a temperature of 21 °C, 50% relative humidity and 3 m/s air speed for 90 min. Finally a seal coat of secondary slurry was applied and dried at a temperature of 21 °C, 50% relative humidity and 3 m/s air speed for 24 h. The wax inside the ceramic mould was then removed by steam autoclaving at 8 bar pressure for 5 min, followed by a controlled de-pressurization cycle at 1 bar/minute using a Quicklock Boilerclave® (Leeds and Bradford Boiler Company Ltd., UK).

The shells were firstly isothermally sintered at 1000 °C for one hour and then they were placed into a ‘flash re-melt’ sessile drop test machine [18]. During this test, shells were first pushed into a furnace at 1650 °C for 60 s and pulled back to room temperature. Then, shells with metal placed on the top (facecoat) surface were tested under the same conditions, allowing the molten metal to interact with the ceramic shell. The titanium aluminate alloy used in this experiment was a Ti—45Al—2Mn—2Nb—0.2TiB (at%) alloy.

2.2. Sample characterizations

Ceramic shells were cut after the firing and reaction tests, allowing the cross section of the mould to be examined. Samples were then mounted and polished for microstructural analysis. The cooled metal drops were also cut in half vertically, mounted and polished for microstructural observation using scanning electron microscopy (SEM) (Jeol-7000 series), and the metal/shell interfacial compositions were characterized using energy dispersive x-ray analysis, EDS. A focused ion beam scanning electron microscope (FIB-SEM) was later used to prepare the sample, and the interaction phases were identified using transmission electron microscope (TEM) (Jeol-2100 series).

3. Results

3.1. Isothermal sintering of ceramic shells

The microstructures of the test pieces, after sintering at 1000 °C, are shown in Fig. 1. An inhomogeneous structure was found in the moulds fired at 1000 °C with distinguishable original filler particles. The filler particles in the facecoat have varied shapes and the average filler particle sizes are around 10 μm. There are no ‘necks’ formed between filler particles after firing at 1000 °C. Fig. 1(b)–(d) illustrate the microstructures of mould cross section. The lower layer in the figures is the facecoat and the upper darker layers are the backup coats. The facecoat has an average thickness around 600–700 μm with the average stucco particle size around 200 μm, and no interaction was observed between the filler powders and stuccos. Some cracks appeared in the facecoat layer when using ZrO2 as the stucco, which could be related to the change in the crystal structure of ZrO2 material during shell firing [19].

3.2. Flash sintering of ceramic shells

After being pre-fired at 1000 °C for one hour, moulds were then put into a furnace at 1650 °C for 60 s, which represents the casting conditions in the investment casting process. The microstructure of the mould was examined, as shown as in Fig. 2. It can be seen from Fig. 2(a) that after flash sintering at 1650 °C for only 60 s, a significant sintering process took place in the facecoat, showing the fused spherical CaZrO3 grains within a more homogeneous structure. EDS analysis of the bright inclusions identified the particles to be ZrO2, which is likely to originate from the binder materials. Also, as some ZrO2 particles are similar in size to filler material, the formation of ZrO2 particles may be due to the decomposition of CaZrO3 filler in the facecoat. No significant interaction was found between the ZrO2 stucco and facecoat materials after flash firing at 1650 °C for 60 s, as shown as in Fig. 2(a) and (b). However, a severe interaction was observed between the Al2O3 stucco and the CaZrO3 facecoat, leading to the dissolution of filler and stucco materials.

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Table 1: Details of composition of the mould system.

| Mould code | Slurry type | Binder | Filler | Stucco |
|------------|------------|--------|--------|--------|
| CA         | Primary    | Colloidal ZrO2 sol | CaO3 -325 mesh | Al2O3 -80 mesh |
|            | Secondary  | Colloidal SiO2 sol | Fusion silica -200 mesh | Alumino-silicate -30/80 |
| CZ         | Primary    | Colloidal ZrO2 sol | CaO3 -325 mesh | Zirconia -50/100 mesh |
|            | Secondary  | Colloidal SiO2 sol | Fusion silica -200 mesh | Alumino-silicate -30/80 |

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Fig. 1. The microstructures of different mould systems after firing at 1000 °C for 1 h, (a) & (b) the stucco and facecoat particles interface, (c) the cross section image of CA mould, using alumina as stucco material and (d) the cross section images of the CZ mould, using zirconia as stucco material.

Fig. 2. The microstructures of different mould systems after firing at 1650 °C for 60 s (a) CZ mould using zirconia as the facecoat stucco, (b) Higher magnification BEI of CZ mould containing grey fused spherical CaZrO3 grains and bright ZrO2 particles, (c) mould CA using alumina as the facecoat stucco, (d) Higher magnification BEI of CA mould containing bright phase A and grey phase B.
between the facecoat and backup coats, as shown as in Fig. 2(c) and (d). The large dark Al2O3 stucco particles are surrounded by grey and white phases, which are labelled as phase A and B in the higher magnification image 2(d). The EDS analyses of both phases A and B are listed in Table 2, revealing that phase A is a Zr and O rich phase, and phase B, with grey contrast, is a phase rich in Ca, Al and O. These results indicated that, during flash firing, the Al2O3 stucco interacted with CaZrO3 in the facecoat, causing the outwards diffusion of Ca2+ ions and enrichment of Zr4+ ions in the fused CaZrO3 filler particles. According to the CaO–Al2O3–ZrO2 phase diagram [20], at 1873 K (1600 °C) CaZrO3 could interact with Al2O3 to form a ZrO2 phase with CaO solid solution, and CaAl2Oy phases, which is similar to the experimental observations.

3.3. The interaction between TiAl and the shell moulds

After the flash re-melt test, the metal/shell interfacial microstructures are shown in Fig. 3. Severe interactions were observed between TiAl alloy and CA and CZ moulds. With the CA mould, which has alumina as the facecoat stucco, an obvious hexagonal six-fold dendritic structure was observed across the complete sessile drop (3 mm height), and this structure is likely to have resulted from the high amount of oxygen as a solid solute in the metal [21]. New bright phases were also found randomly distributed at the grain boundaries as shown as in Fig. 3(a). Compared to mould CA, using the mould CZ, which has ZrO2 as facecoat stucco, more interaction products were formed at the grain boundaries, as shown as in Fig. 3(b), but no significant six-fold dendritic structure was observed. The higher magnification image of the bright phase in Fig. 3(d) shows that a second phase may also be present within its structure.

TEM techniques were used to identify the interaction products at the grain boundaries and the results are illustrated in Figs. 4 and 5. The TiAl metal matrix consisted of a typical lamellar (α2 + γ) microstructure, and the EDS analysis results revealed that bright phases C, Fig. 4(a), contained The TiAl metal matrix consisted of a typical lamellar (α2 + γ) microstructure, and the EDS analysis results revealed that bright phases C, Fig. 4(a), contained 45.26 at% of Ti, 17.46 at% of Zr, 22.65 at% of Al and 14.63 at% of Si concentrations. The diffraction pattern of this phase shows that it has a simple hexagonal D8 structure, similar to the phase Ti5Si3. The study carried out by Chen [22] indicated that Ti has very high solubility in TiAl alloy and it is able to dissolve around 11 at% in γ phase, and more than 25 at% in α2 phase [23, 24]. Meanwhile, the study carried out by Kotval [25] and Cheng [23] indicated that Al ions can dissolve in Ti5Si3 as Ti5(Al, Si)3 phase. Hence the possible composition of phase C shown is a (Ti, Zr)(Si, Al)3 phase. The formation of (Ti, Zr)(Si, Al)3 phases at the TiAl alloy grain boundaries can be related to the reaction between the TiAl with the ZrO2 present in the facecoat of both types of shell moulds.

Table 2

| Phase | Ca  | Zr  | Al  | Si  | O   |
|-------|-----|-----|-----|-----|-----|
| A     | 6.23| 44.93| 0.82| –   | 48.02|
| B     | 11.95| –   | 27.99| 1.52| 56.54|

Besides phase C, another phase (phase D) was found to be present together with phase C, as shown as in Fig. 5. The EDS analysis shows that the phase D contains 53.53 at% Al, 42.35 at% Ti, and 4.12 at% Zr. The diffraction pattern of phase D has same crystal structures as phase ZrAl2 which was also revealed by Jia [7]. The composition of phase D is likely to be ZrAl2 phase with high Ti ions solid solution.

4. Discussion

4.1. The interaction between filler particles and stucco in the facecoat

The stability and the inertness of the CaZrO3 facecoats were investigated during the mould firing and casting process, using both Al2O3 and ZrO2 as facecoat stuccos. No interaction was observed between the CaZrO3 filler and stuccos when moulds were sintered at 1000 °C for both mould systems. When moulds were heated to 1650 °C for 60 s, a severe interaction was observed between the CaZrO3 filler and Al2O3 stucco. The interaction causes the degradation and dissolution of the Al2O3 stucco which penetrated from facecoat into the backup coats. In the surrounding areas of the Al2O3 stucco, the composition of facecoat were changed from CaZrO3 and Al2O3 to a mix of (Zr,Ca)O2 and CaAl2Oy. The reaction can be seen represented as:

\[
\text{CaZrO}_3 + \text{Al}_2\text{O}_3 \rightarrow (\text{Zr,Ca})\text{O}_2 + \text{CaAl}_{2}\text{O}_y
\] (1)

No reaction was observed in the facecoat system using ZrO2 as the stucco material, although the cycling of mould temperatures caused crystal structure changes in the ZrO2 stucco, resulting in cracks generated in facecoat associated with the volume change in this stucco [19]. This work shows that, during the high temperature casting process, the inertness of facecoat materials is not only dependent on the composition of facecoat, but also related to interaction products between the fillers and stuccos.

4.2. The interaction between the facecoat and TiAl alloy

As previously known, the reactivity of the facecoat and TiAl alloy was dependent on the Gibbs free energy changes during the interaction. According to the free energy calculations [17], the CaZrO3 facecoat thermodynamic inertness lies between CaO and ZrO2 materials. However, due to the reaction between the filler stucco materials, the actual interactions between the facecoat and molten TiAl were more complicated than previously understood.

The use of Al2O3 as the stucco material can cause an interaction to take place between the filler and stucco, resulting in the formation of phases (Zr, Ca)O2 and CaAl2Oy at the mould surface, directly in contact with the metal. During flash re-melting, molten TiAl interacted with (Zr, Ca)O2 and CaAl2Oy which formed a solid-solution with Si penetrating through from the backup coats [26]. Zr ions from the facecoat and Si from backup coats penetrated into the metal and reacted with TiAl to form a Ti5Si3 with a Zr solid solute [24, 25]. The reaction between Ti and Si is presented in equation (2):

\[
5\text{Ti} + 3\text{Si} \rightarrow \text{Ti}_5\text{Si}_3
\] (2)

Besides forming a silicide phase, Zr ions also form possible combined (Ti, Zr)(Si, Al)3 phase and ZrAl2 phase with a high Ti ions solid solution, which were also revealed in this work. Meanwhile, the obvious six-fold dendritic microstructure in the sessile drop microstructures illustrated that the oxygen from the facecoat also penetrated into metal during testing using mould applied with Al2O3 as the stucco. The inertness of CaZrO3 was reduced by the reaction with Al2O3, forming phases (Zr,Ca)O2 and CaAl2Oy. Using ZrO2 as the stucco did not cause the filler/stucco interaction. But the cracks in facecoat due to the volume change of ZrO2 stucco did accelerate the penetration of Si from backup coat to the surface, producing more Si bearing interaction phases during metal/shell interaction. No Ca ions were detected at the metal interfacial areas,
Fig. 3. The microstructures of the sessile drop metals tested at 1650 °C for 60 s (a) with mould using Al₂O₃ as stucco in the facecoat, (b) with mould using ZrO₂ as stucco in the facecoat (c) interaction products in metal (d) the higher magnification image of the interaction product.

Fig. 4. Showing (a) the TEM image of the interaction product, (b) the diffraction pattern of phase C and (c) the EDS analysis of phase C.
and the Ca ions could only be detected in the facecoat, similar to the results reported by Gomes [27].

5. Conclusions

In this experiment, a CaZrO3 based material was used as the facecoat material to investment cast a TiAl alloy. According to these results some conclusions can be summarized as follow:

The Al2O3 stucco can react with CaZrO3 filler to form (Zr, Ca)O2 and CaAl2O4 at temperatures around 1650 °C. No interaction was observed when using ZrO2 as the stucco material except for some cracks appearing in the facecoat due to the volume change of ZrO2 material during thermal processing. When evaluating the inertness of the facecoat material, the interaction products in the TiAl alloy surface need to be taken into account at casting temperatures. The reaction between CaZrO3 filler and Al2O3 stucco led to six-fold dendritic microstructures appearing in the TiAl alloy due to oxygen penetration from the facecoat. Besides oxygen, Si and Zr ions from the facecoat also penetrated into the TiAl alloy to form (Ti, Zr)5(Si, Al)3 and ZrAl2 phase with high Ti ions solid solution. No Ca ions were detected in metal/shell interfacial area in this experiment.

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