



## An investigation of a TiAlO based refractory slurry face coat system for the investment casting of Ti–Al alloys

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### ABSTRACT

The investment casting process offers great freedom of design with the economic advantage of near net shape production and has been widely used to process TiAl alloys. An investigation was undertaken to develop stable and cheap refractory slurry systems, suitable for use as a face coat in the investment casting of titanium aluminides. A TiO<sub>2</sub>–Al<sub>2</sub>O<sub>3</sub> (TiAlO) based face coat slurry was investigated and compared with a traditional yttria based face coat slurry. After sintering, the chemical inertness of the manufactured shells in contact with molten TiAl alloys was tested by simulating the casting process using a flash re-melting test, in which sample shells were in contact with titanium aluminides at given temperatures for varying time durations and subsequently re-solidified. Compared with yttria, the TiAlO face coat had relatively lower chemical inertness against molten alloys, forming a thick hardened layer at the metal/shell interface with massive interaction products along grain boundaries. The reaction also changed the wetting behaviour of the TiAl on the TiAlO face coat with the contact angle decreasing as a function of interaction time.

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### 1. Introduction

Titanium aluminides have been used in engineering applications such as car engine valves and turbochargers, and are now being used for gas turbine aero-engines to replace nickel base superalloys in low-pressure turbine blades. This is made possible due to their superior properties, especially the specific strength and creep resistance up to temperatures around 650 °C [1].

The major efforts made to introduce titanium aluminides into the aerospace market in recent years have resulted in limited success. The principal factors, which held back the manufacture of 'mass market' TiAl-based components, are microstructural and chemical heterogeneity, brittleness, low room temperature ductility and poor workability [2]. Due to these problems wrought methods such as forging and rolling, which are very prone to chemical and microstructure inhomogeneity, have their intrinsic limitations, whilst powder metallurgy is considered very expensive. Investment casting which can produce near net shape components with a good surface finish and high metal yield, at the lowest projected cost, is a subject of growing interest [3].

Due to the very high reactivity of molten TiAl [4], the main problem encountered in investment casting has been the

interaction between the molten alloy and mould. This reaction is usually manifested as an elevated near-surface oxygen content with high hardness, decreasing with the distance from the surface [5,6]. Reaction products were also found to form along the grain boundaries in the vicinity of the interfacial region. The reaction also causes the wetting properties of the TiAl on the ceramic shell to change with different reaction temperatures and times in different ceramic systems [7–9].

Research in refractory materials for investment casting started last century. Saha et al. [10,11], used SiO<sub>2</sub> based face coat moulds and have an interaction layer larger than 500 μm, subsequently, oxides such as CaO, ZrO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> were developed for face coat use but still serious interaction between the mould and molten metal occurred with some unwanted face coat particles found at the interface areas. Until around 2000, new face coats made of the rare earth oxide Y<sub>2</sub>O<sub>3</sub> have been used to cast TiAl with detected interaction layers of around 4 μm [12]. However, the difficulties with using this kind of face coat are the pre-gelation of the face coat slurry, which limited the slurry life to around few hours, and the high production cost of the yttria mould. Therefore, developing a long life, stable face coat slurry with lowest cost whilst having a reasonable chemical inertness, is a new objective for investment cast TiAl alloys.

In this paper, the chemical inertness of the new face coat containing TiO<sub>2</sub>–Al<sub>2</sub>O<sub>3</sub> (TiAlO) is investigated by reaction with

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a Ti–45Al–2Mn–2Nb–0.2B (at %) alloy and compared to a face coat manufactured from yttria. The interaction between the shell and metal was investigated using energy dispersive spectrometry (EDS), scanning electron microscopy (SEM), transmission electron microscopy (TEM), micro-hardness testing, optical microscopy and X-ray diffraction (XRD). The contact angle and radius of the sessile droplets were measured using a 3D laser scanner.

## 2. Materials and methods

### 2.1. Mould preparation

Ceramic moulds with yttria and TiAlO based face coats were produced, where the primary slurry consisted of a ceramic filler, colloidal binder, wetting agent, and anti-foam agent. Details of the slurry composition are shown in Table 1. The moulds were made by first investing a wax pattern of dimensions of  $100 \times 100 \times 50$  mm into the primary slurry and then coating with stucco (for the yttria face coat, the stucco is yttria, and for the TiAlO face coat, the stucco is  $\text{Al}_2\text{O}_3$ ). The coat was dried in an environment maintained at 21 °C, 50% relative humidity and 0.4 m/s air speed for 24 h. For both coat systems, the same backup slurry system was then applied (the details of the backup slurry composition are also listed in Table 1). Several backup coats were made using alumino-silicate based materials and each coat was dried at the same conditions as those for the face coat for around 90 min. The wax inside the ceramic mould was then removed by steam autoclave set at 8 bar pressure for 4 min, followed by a controlled de-pressurisation cycle at 1 bar/min using a Quicklock Boilerclave™ (Leeds and Bradford Boiler Company Ltd, UK). Moulds were then cut into small rectangular pieces of dimensions  $20 \times 40 \times 5$  mm and fired at a temperature of 1200 °C for 1 h in air.

### 2.2. Metal sample preparation

The alloy used in this experiment was a Ti–45Al–2Mn–2Nb–0.2B (at%) alloy with a melting temperature of around 1650 °C. The cast Ti–Al alloy was cut into cylindrical pieces of 6 mm height and 10 mm diameter and placed on the face coat side of ceramic plates, prior to the sessile drop ‘flash re-melt’ test.

### 2.3. Flash re-melt testing

The schematic of the furnace arrangement used for the flash re-melt test is shown in Fig. 1. Initially, a metal and ceramic disc sample pair was located in the cold chamber 1 under a vacuum of  $\sim 0.005$  mbar, while chamber 2 was heated to 1650 °C. A hydraulic ram then quickly raised the sample pair into chamber 2. The contact time was measured as the elapsed time from the ram reaching the full limit of its stroke. The ram was retracted into chamber 1, after a prescribed reaction time (30 s, 40 s, 50 s, and 60 s), to allow the specimen to cool down.

### 2.4. Characterization

The face coat and the reaction product between ceramic shells and flash re-melted alloy were characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM), micro-hardness testing and transmission electron microscopy (TEM). The sessile drop radius and contact angle were measured using a laser profile scanning head attached to a coordinate measuring machine (CMM) (Model: Impact 1000 CMM, Manufacturer: International Metrology Systems). The laser scanner was a Zephyr KZ25, Kreon Technologies and the computer software used for data analysis was Virtual DMIS and Polygoia.

## 3. Results

### 3.1. Mould surface XRD analysis

Fig. 2 shows the XRD spectra obtained from the yttria and TiAlO face coats sintered at different temperatures. No peak change was detected for the yttria face coat when the sintering temperature increased from 1000 °C to 1550 °C. However, for the TiAlO face coat, the phase transformation took place through the sintering temperature range as decomposition and reformation of  $\text{TiAl}_2\text{O}_5$  phase from  $\text{TiO}_2$  and  $\text{Al}_2\text{O}_3$  took place. High sintering temperatures such as 1400 °C seem to be more favourable for the formation of  $\text{TiAl}_2\text{O}_5$ . Therefore the shell surface composition of the yttria face coat was found to be  $\text{Y}_2\text{O}_3$  at all temperatures, whilst for the TiAlO shell it varied from  $\text{TiO}_2$ ,  $\text{Al}_2\text{O}_3$ , to  $\text{TiAl}_2\text{O}_5$ .

### 3.2. Sessile drop contact angle change

The change of sessile drop contact angle and radius with increasing contact time between the metal and ceramic shell at a reaction temperature of 1650 °C are illustrated in Fig. 3. In both face coat systems, it can be seen that the contact angle decreased with increasing contact time, while the radius of the metal drops increased with contact time. The largest change of the metal drop contact angle and radius in both systems took place with the contact time between 30 s and 40 s, and it then slowed down. Compared to the yttria face coat, metal on the TiAlO system shows a much higher of change rate of the contact angle in the first 10 s from around 142° to around 30° and the drop radius increased from 8 mm to 18 mm, more than 5 times the rate of that with the yttria face coat. For the yttria face coat, the metal wetting on the shell varied at a very slow rate which remained nearly constant.

### 3.3. Micro-hardness at the metal/shell interface

Due to the interaction between the ceramic shell and molten TiAl, the metal/ceramic interface shows a higher hardness than metal matrix (Fig. 4). For the yttria face coat, the highest hardness was found at the interface at around 570 Hv and it then gradually dropped to around 450 Hv at a distance of 50  $\mu\text{m}$  away from the interface. No clear trend was observed linking the increase in the hardened layer thickness to the contact time between molten metal

**Table 1**  
Slurry formulations.

Slurry description	Filler	Binder	Wetting agent	Anti-foam
TiAlO	$\text{TiO}_2$ $\text{TiAl}_2\text{O}_5$ (10–30 $\mu\text{m}$ ) $\text{TiAl}_2\text{O}_5$ (60–120 $\mu\text{m}$ )	Alumina binder	Victawet 12 (REMET)	Burst RSD-10 (REMET)
Yttria	–200 mesh yttria powder	Yttria colloidal sol polymer (PVA)	Victawet 12 (REMET)	Burst RSD-10 (REMET)
Back up coat	Molochite (–200 mesh) (ECC)	LP-BV silica sol Adbond BV Polymer		

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