

## Interfacial reactions between Ti–1100 alloy and ceramic mould during investment casting

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Received 10 May 2011; accepted 25 July 2011

**Abstract:** The characteristics of the interfacial reaction between Ti–1100 melt and different primary coating materials was investigated. The effect of refractory materials and mould temperature on the reaction was analyzed by comparing the thickness, distribution of elements and microhardness of  $\alpha$ -case layer. The  $\alpha$ -case layer thickness of Ti–1100 casting with ZrO<sub>2</sub> primary coating is approximately 38  $\mu\text{m}$ , which is higher than that with Y<sub>2</sub>O<sub>3</sub> primary coating (18  $\mu\text{m}$ ). Ti–1100 casting using ZrO<sub>2</sub> primary coating shows higher surface microhardness than that using Y<sub>2</sub>O<sub>3</sub> primary coating. The higher mold temperature results in more severe interfacial reaction. With the same primary coating material and mould temperature, Ti6Al4V alloy presents better stability than Ti–1100 alloy.

**Key words:** Ti–1100 alloy; interfacial reaction; investment casting; primary coating material

### 1 Introduction

Ti–1100 alloy is a successful near-alpha alloy working at 600 °C designed for compressor disks and blades in the aerospace industry [1–2]. For most 600 °C high temperature titanium alloys, forging is the primary processing method [2–5]. But for complex components, investment casting gives more advantages, such as lower cost, higher flexibility in design, and reduction in the machining of the titanium components [6–8]. However, titanium alloys have an inherent reactivity with the mold primary materials at high temperatures during investment casting [9]. The reactions between the titanium melt and mold materials result in the formation of the  $\alpha$ -case layer [10], which will deteriorate the surface and change the mechanical properties of titanium castings [11–12], though the  $\alpha$ -case layer can be eliminated in the chemical milling processes.

Although some researchers investigated the interfacial reactions between cp-Ti, Ti6Al4V or TiAl melt and different refractory materials [13–16], and discussed the  $\alpha$ -case formation mechanism [11–12, 17], there was no literature on the characteristics of interfacial

reactions between high temperature titanium alloys and mold materials. As a multi-element alloy, the interfacial reactions between melt and primary coating materials may be different from other alloys. Some papers defined the depth of  $\alpha$ -case layer with the surface microhardness test [13, 18]. However, it is inaccurate to define the thickness of reaction layer, especially for thin reaction layer. Generally, the interface reaction between the mold and molten titanium alloy leads to the changes of elements content in the surface of the titanium casting. Therefore, the distributions of elements on the surfaces of titanium alloys castings will assist in defining the thickness of  $\alpha$ -case layer.

In this work, the influence of primary coating material and mold preheating temperature on the  $\alpha$ -case layer of Ti–1100 casting was evaluated by line scanning and microhardness test. The purpose of this work is to investigate the microstructure, composition of the reaction layer, and the surface microhardness change of high temperature titanium Ti–1100 casting.

### 2 Experimental

In this study, the ZrO<sub>2</sub> (CaO stabilized) and Y<sub>2</sub>O<sub>3</sub>

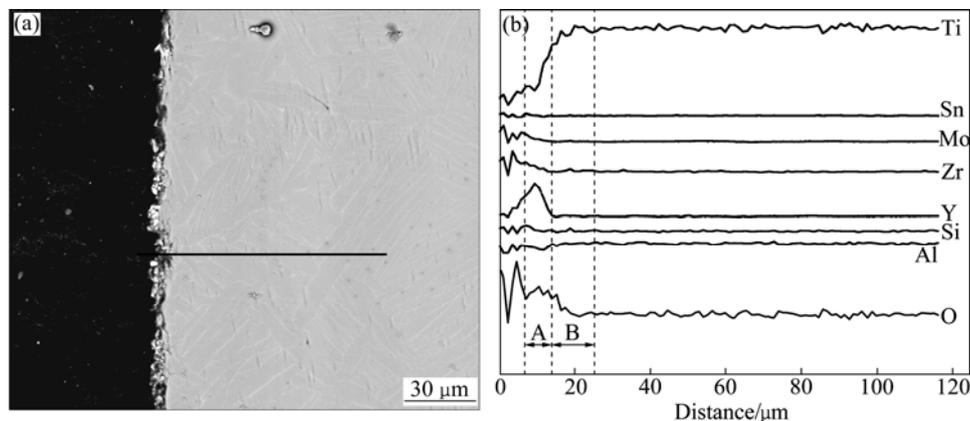
powders (45  $\mu\text{m}$ ) were chosen to prepare the primary coating slurry respectively, and zirconia sol was used as binder. A wax column with a diameter of 12 mm and a length of 40 mm was coated with  $\text{ZrO}_2$  (CaO stabilized) and  $\text{Y}_2\text{O}_3$  powder (45  $\mu\text{m}$ ) based slurry, and sprinkled with  $\text{ZrO}_2$  and  $\text{Y}_2\text{O}_3$  sand (125  $\mu\text{m}$ ), respectively. Alumina and silicon sol were chosen as back-coat materials for the two types of mold shell. After the fabricating of mold shell, all molds were heated in an electric furnace at a temperature of 950  $^\circ\text{C}$  for 1 h in order to achieve enough strength. 40 g Ti-1100 alloy was filled into the molds with  $\text{ZrO}_2$  primary coating at room temperature and 400  $^\circ\text{C}$  respectively, and 40 g Ti6Al4V alloy was also cast with the same mold for comparison. A centrifugal vacuum pressure machine (LZ5, Luoyang Common Machine Factory, China) was used to cast alloys samples. The microstructure and quantitative elements distribution of the reaction layer of titanium castings were examined by scanning electron microscopy (SEM, FEI, QUANTA 200F). The surface microhardness test was performed with load of 0.98 N for 15 s.

### 3 Results and discussion

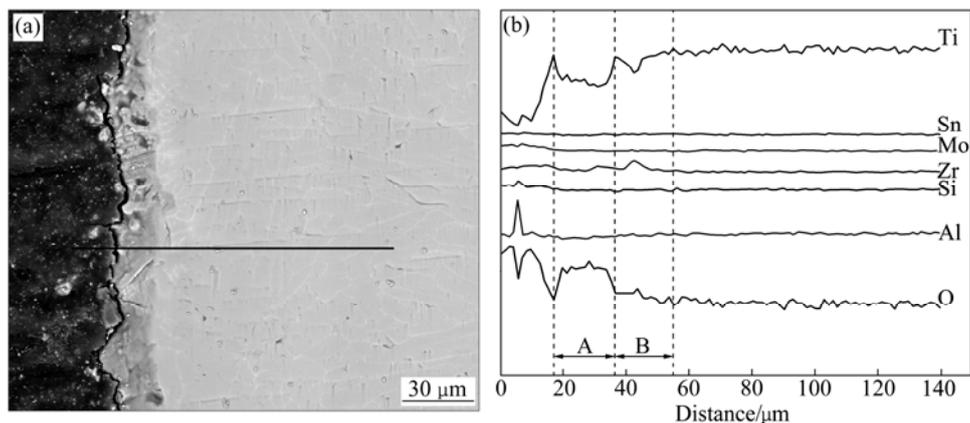
It can be seen that there is a distinct reaction layer

between Ti-1100 casting and mold shell for two primary coating from Figs. 1 and 2. However, the thickness of reaction layer for  $\text{ZrO}_2$  primary coating is obviously thicker than that of those with  $\text{Y}_2\text{O}_3$  primary coating. The  $\alpha$ -case layer is composed of a reaction layer (A) and a harder layer (B). Layer A can be readily found from BEI images because the phases on the Ti casting surface are different from those in the matrix, which are also distinct from layer B. By line scanning, the reactive diffusion characters of the element between titanium alloys and mold shell can be drawn easily. It is found that the thickness of  $\alpha$ -case layer for Ti-1100 casting fabricated with the  $\text{Y}_2\text{O}_3$  primary coating is approximately 18  $\mu\text{m}$ . It is composed of 2 layers: the outer layer (A) is the reaction layer with a thickness of about 7  $\mu\text{m}$ ; the inner layer (B) is the harder layer with an approximate thickness of 11  $\mu\text{m}$ .

As shown in Fig. 2, the thickness of  $\alpha$ -case layer for Ti-1100 casting fabricated with the  $\text{ZrO}_2$  primary coating is approximately 38  $\mu\text{m}$  and is also divided into two layers: a reaction layer (19  $\mu\text{m}$ ) and a harder layer (19  $\mu\text{m}$ ). Therefore, the reaction between Ti-1100 melt and  $\text{ZrO}_2$  primary coating is more seriously than that with  $\text{Y}_2\text{O}_3$  primary coating. Compared Fig. 1 with Fig. 2, it can be seen that Ti element and O element presented



**Fig. 1** Microstructure (a) and elemental distribution curves (b) on interface between Ti-1100 casting and  $\text{Y}_2\text{O}_3$  primary coating with mold temperature of 400  $^\circ\text{C}$



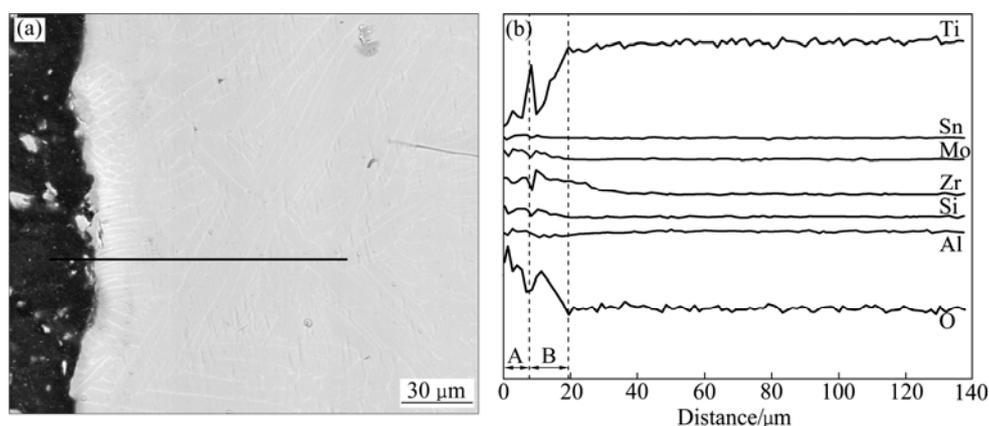
**Fig. 2** Microstructure (a) and elemental distribution curves (b) on interface between Ti-1100 casting and  $\text{ZrO}_2$  primary coating at mold temperature of 400  $^\circ\text{C}$

the same diffusion regular in the  $\alpha$ -case layer, respectively. From outer layer (A) to inner layer (B), the content of Ti element increases with depth. While, the content of O element decreases. This indicates that the interaction between the shell mold and melting titanium is a bidirectional diffusion process. Ti atoms diffuse from the matrix into the surface of the primary coating, and O atoms diffuse from the primary coating into the matrix. Thus, the  $Ti_xO_y$  compounds form. In Fig. 1, the content of Y element in the reaction layer is much higher than that in harder layer, but it is the same for the content of Y element in reaction layer and matrix. It can be explained that there are  $Y_2O_3$  materials in the reaction layer, but the Y atoms don't diffuse from the reaction layer to the harder layer. On the other hand, there is higher content of Zr element in the reaction layer because the zirconia sol was used as the binder in this study. The content of Al element in the reaction layer is lower than that in the harder layer and matrix, indicating that the Al atoms diffused from the matrix into the primary coating, and involved in the reactions. The detailed formation of this reaction should be explored later. As shown in Fig. 2, there is no obvious variation of content for all elements from the reaction layer to the matrix, except Ti, O and Zr

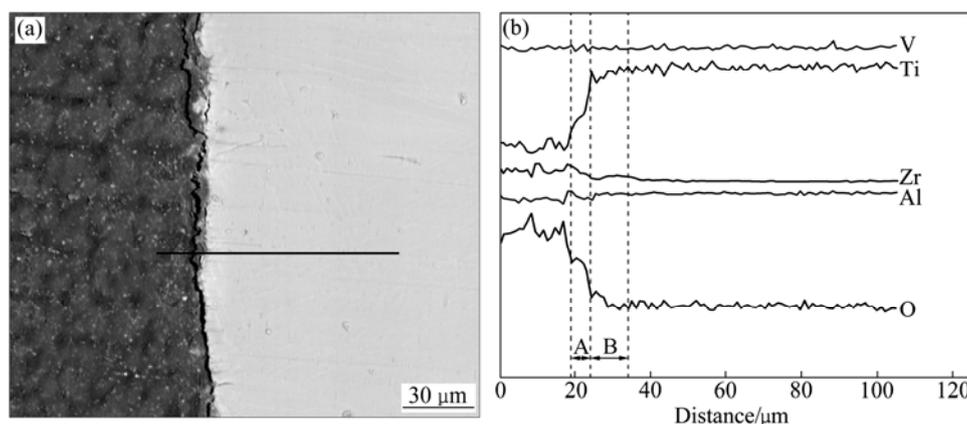
elements. However, the content of Zr element in the harder layer is higher than that in the reaction layer and matrix. The results indicate that the Zr atoms from  $ZrO_2$  refractory materials and zirconia sol involve in the reactions and diffuse into the harder layer at high temperature.  $Y_2O_3$  is more stable than  $ZrO_2$  because of its lower standard free energy [19]. Our experiment results are consistent with this conclusion. Based on the results,  $Y_2O_3$  primary coating is the better choice for Ti-1100 alloy investment casting.

From Figs. 1 and 3, it is found that the thickness of the harder layer increases from 12  $\mu m$  to 19  $\mu m$  with the mold temperature increasing. On one hand, the high mold temperature can retard the solidification of the melting alloy; on the other hand, more serious interfacial reaction may occur during solidification because of higher mold temperature.

In order to investigate the characteristics of interfacial reactions between high temperature titanium Ti-1100 alloy and mold materials, Ti6Al4V alloy was also filled into the same mold at the same mold temperature for comparison. As shown in Fig. 4, Ti6Al4V alloy produces the thinner reaction layer with the same experimental conditions. The elements Ti, O



**Fig. 3** Microstructure (a) and elemental distribution curves (b) on interface between Ti-1100 casting and  $ZrO_2$  primary coating at mold temperature of 20 °C



**Fig. 4** Microstructure (a) and elemental distribution curves (b) on interface between Ti6Al4V casting and  $ZrO_2$  primary coating at mold temperature of 400 °C

and Zr have the same distribution character for both of the alloys, but Al and V elements exhibit unobvious changes in the reaction layer.

ZHU et al [20] predicted the interfacial reaction tendency of SiC and intermetallics composites by calculating the activity coefficients, and the calculated results agreed with the experimental values. In this work, Ti-1100 (Ti-6Al-2.75Sn-4Zr-0.4Mo-0.45Si) alloy is divided into two ternary systems, which neglects the influence of microcontent elements of Mo and Si. Based on the Troop's ternary solution model and Miedema's model [21], the activity coefficients of titanium in different ternary systems at different temperatures were calculated. It can be drawn from Fig. 5 that the activity coefficient of titanium increases with the rise of temperature. Titanium exhibits similar activity coefficients in both Ti-Al-V and Ti-Al-Sn systems. However, higher activity coefficient presents in the Ti-Al-Zr system. The activity of titanium in the alloy system increases with the addition of Zr and Sn elements, indicating that more severe interfacial reaction may occur for Ti-1100 alloy than for Ti6Al4V alloy.

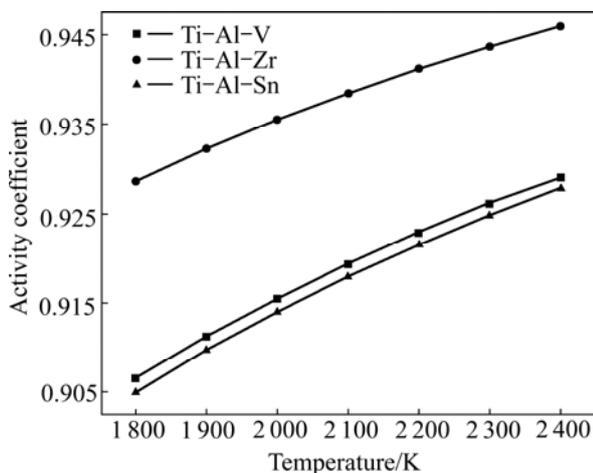


Fig. 5 Activity coefficient of titanium in different ternary systems at different temperatures

It can be concluded from Fig. 6 that the sections of Ti-1100 casting fabricated with  $ZrO_2$  coating presented the thickest reaction layer and the highest surface microhardness. Ti6Al4V casting produced the lowest surface microhardness, but had the similar depth of reaction layer with Ti-1100 casting using  $Y_2O_3$  primary coating.

After a depth of harder layer, the microhardness reduced slowly with depth. This may be due to the secondary layer formed near the harder layer. The formation of this intermediate layer did not result from the reaction between molten titanium and investment. SUNG and KIM [12] suggested that it should be the

hardening layer. Fabrication and characterization of this layer should be carried out as part of the continuing work.

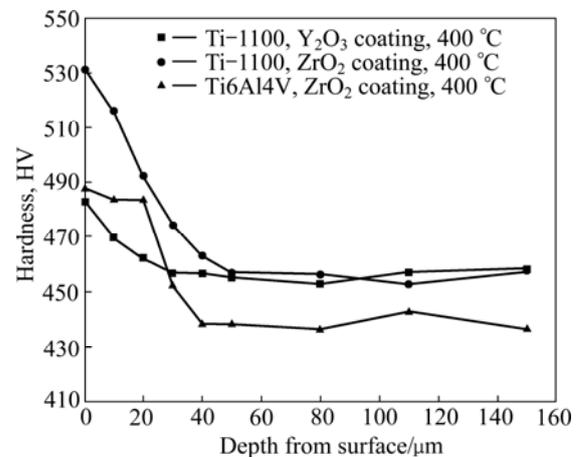


Fig. 6 Changes of microhardness from outermost surface to inner aspect of specimens

## 4 Conclusions

- 1) The interfacial reactions layer ( $\alpha$ -case layer) consists of a reaction layer and a harder layer. There is an inferior hard layer between the harder layer and the matrix. The reaction layer can be defined from BEI, but the harder layer needs to be defined with distributions of elements and microhardness on the surfaces of titanium alloys.
- 2) The  $Y_2O_3$  primary coating presents better stability for the Ti-1100 melting. Higher mold temperature results in more serious interfacial reaction.
- 3) Different types of primary coating materials produce different distribution of elements in  $\alpha$ -case layer. The detailed mechanism of interfacial reaction will be explored later.

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## 熔模铸造 Ti-1100 高温钛合金的界面反应

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**摘要:** 研究不同面层材料的 Ti-1100 高温钛合金界面反应特性。通过对比反应层的厚度, 元素分布以及微观硬度研究了面层型壳材料以及型壳预热温度对界面反应的影响。结果表明: 采用 ZrO<sub>2</sub> 面层材料型壳浇注的 Ti-1100 铸件反应层的厚度和硬度明显高于采用 Y<sub>2</sub>O<sub>3</sub> 面层材料型壳浇注的合金; 型壳预热温度越高, 界面反应越剧烈; 在相同的型壳面层材料和型壳预热温度条件下, Ti6Al4V 合金铸件的界面反应  $\alpha$  层比 Ti-1100 合金铸件界面反应层薄, 显微硬度低, 表现出较好的稳定性。

**关键词:** Ti-1100 合金; 界面反应; 熔模铸造; 面层材料

(Edited by LONG Huai-zhong)