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## Formation of Alpha Case Mechanism on Titanium Investment Cast Parts

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### 1. Introduction

At present the practical utilization of titanium (Ti) and its alloys ranges from sports equipment to the aerospace, power generation and chemical processing industries, and automotive, marine and medical engineering [1]. Yet, Ti alloy forming processes are much more laborious than one thinks, which is mainly due to the fact that in solid solution Ti combines with carbon, nitrogen and especially oxygen and additionally Ti is constrained to be melted in inert environments or in a vacuum [2]. For the intended applications, Ti and its alloy forming processes should incorporate forging, powder metallurgy and casting. A center bulkhead of the F-22 Raptor fighter aircraft is a good example of the difficulty in Ti alloy forging. Although the final Ti6Al4V alloy component weighs only about 150 kg, it should be forged initially from a single cast ingot of nearly 3,000 kg, which clearly exhibits extremely high machining losses in the Ti alloy forging process [2]. By the way, the powder metallurgy of Ti alloys offers a practicable way of producing complex components with less machining losses than in forging processes. However, the applications of the powder metallurgy have been limited by the freedom of size and shape. In addition, in order to ensure the soundness of the powder metallurgy products without porosity defects, hot isostatic pressing (HIP) is essentially required, which makes the powder metallurgy even more expensive [3].

Casting is a typical net shape forming technique in which molten metals are poured into a mold to produce an object of desired shape. However, the casting of titanium alloys is considered as only a near net shape forming technique. That is because regardless of mold type, titanium alloys casting has a drawback, called alpha-case ( $\alpha$ -case), which makes it difficult to machine them and can lend themselves to crack initiation and propagation, due to their enormous reactivity in molten states [4]. Thus, the depth of  $\alpha$ -case must be taken into consideration in the initial design for casting since the brittle  $\alpha$ -case must be removed by chemical milling. For the reason, the wide use of titanium alloys casting has been limited, although titanium alloys castings are comparable, and quite often superior, to wrought products in all respects [3].

In order to avoid the  $\alpha$ -case problem, the expensive ceramics, such as CaO, ZrO<sub>2</sub> and Y<sub>2</sub>O<sub>3</sub> have been adopted as mold materials since the standard free energy changes of the formation of their oxides are more negative than that of TiO<sub>2</sub>. Regardless of thermodynamic

approaches, the  $\alpha$ -case formation reaction still remains to be eliminated in the chemical milling processes. In order to develop the economic net shape forming technique of titanium alloys by casting process, it is necessary to take a much closer look into the  $\alpha$ -case formation mechanism. Therefore, the exact  $\alpha$ -case formation mechanism must be examined and the development of  $\alpha$ -case controlled mold materials is required for practical applications of Ti alloys.

## 2. Evaluation of the alpha-case formation mechanism

### 2.1 Conventional alpha-case formation mechanism

The investment casting of titanium has a drawback, called ' $\alpha$ -case', which makes it difficult to machine and can lead to crack initiation and propagation, due to their enormous reactivity in molten states [4]. The  $\alpha$ -case is generally known to be developed by the interstitials such as carbon, nitrogen and especially oxygen dissolved from mold materials. In order to avoid this problem, the expensive ceramics, such as  $\text{ZrO}_2$ ,  $\text{ZrSiO}_4$ ,  $\text{CaZrO}_3$ ,  $\text{CaO}$  and  $\text{Y}_2\text{O}_3$  have been adopted for mold materials because their standard free energy changes of the formation of oxides are more negative than that of  $\text{TiO}_2$  as shown in figure 1.

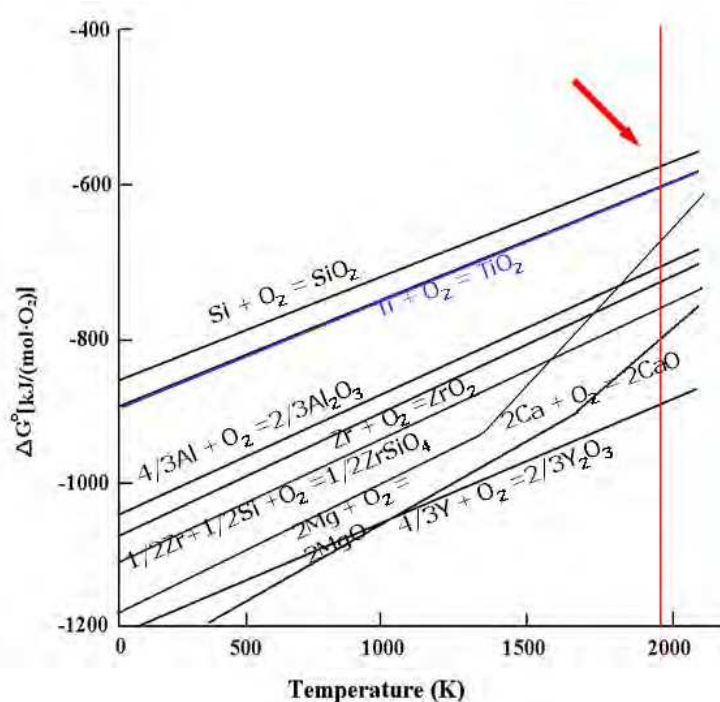


Fig. 1. Standard free energy changes of the formation of various oxides.

Regardless of the thermodynamic approaches, some amount of  $\alpha$ -case still remains to be eliminated with the complex chemical milling processes except for  $\text{Y}_2\text{O}_3$  mold. However, in the case of  $\text{Y}_2\text{O}_3$  mold, there is not enough strength to handling due to the silica-free binder and there occur metal-mold reactions to some extent, too.

### 2.2 Evaluation of alpha-case reaction

The wax patterns for the examination of  $\alpha$ -case reactions were made by pouring molten wax into a simple cylindrical silicon rubber mold ( $\varnothing 15 \times 70$  mm). Subsequently, the patterns

were inspected and dressed to eliminate any imperfection or contamination, and coated with  $\text{Al}_2\text{O}_3$  slurry. The  $\text{Al}_2\text{O}_3$  shell molds were dried at a controlled temperature ( $298 \text{ K} \pm 1 \text{ K}$ ) and a relative humidity ( $40\% \pm 1\%$ ) for 4 hrs. Dipping, stuccoing and drying procedures were repeated three times. After the primary layer coating, the patterns were coated with the back-up layers by the chamotte. To prevent the shell cracks, the dewaxing process of the shell molds were carried out at around  $423 \text{ K}$  and  $0.5 \text{ MPa}$  in a steam autoclave. Finally, the shell molds were fired at  $1,223 \text{ K}$  for 2 hrs.

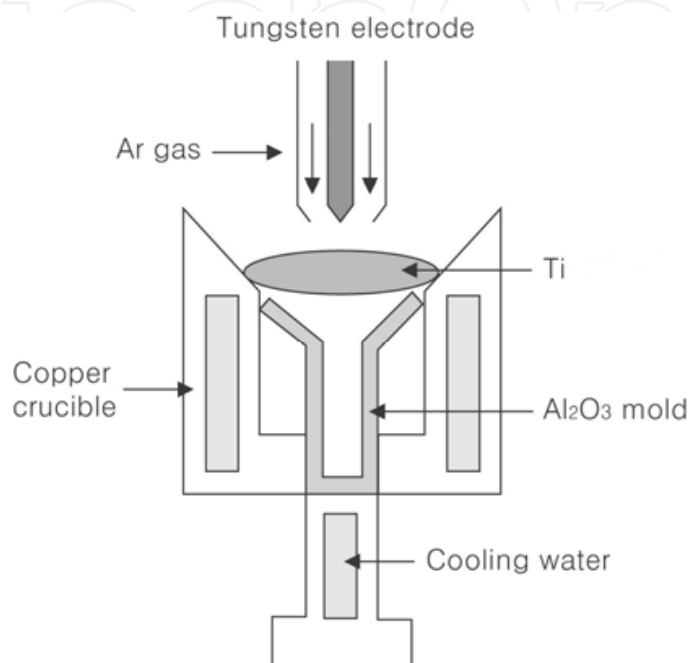


Fig. 2. Schematic diagram of drop casting procedure of titanium with a plasma arc melting furnace.

In order to prevent any contamination from refractory crucibles, the specimens for the  $\alpha$ -case formation examination were prepared in a plasma arc melting (PAM) furnace with drop casting procedure. The pressure in the PAM was controlled at about  $1.33 \times 10^{-1} \text{ Pa}$  by a rotary pump and then in order to minimize the effect of oxygen contamination, high purity argon was backfilled to the pressure of  $4.9 \times 10^3 \text{ Pa}$ .

After the drop casting, Ti castings were taken out of the molds, sectioned, polished and etched using a Keller solution [5]. The microstructure in the reaction region of the castings was observed using Olympus PME3 microscopy, and its hardness was measured using a Mitutoyo MVK-H2 microvickers hardness tester with the condition of  $100 \text{ g}$  load and  $50 \mu\text{m}$  intervals. For a closer examination of the alpha-case formation, the distribution state of the elements analysis at the alpha-case region was performed by SHIMADZU EPMA 1600 and the phase structures of reaction products were identified by JEOL JEM-3011 TEM.

### 3. Thermodynamic calculation of alpha-case formation mechanism

#### 3.1 Evaluation of the alpha-case reaction

In this research,  $\text{Al}_2\text{O}_3$  was selected for the mold material because the standard free energy changes of the formation of its oxides are more negative than that of  $\text{TiO}_2$ . In addition,  $\text{Al}_2\text{O}_3$

features suitable strength, permeability and collapse-ability, which can ensure dimensional accuracy of castings. Fig. 3 shows the microstructure and hardness profile on the surface of pure titanium investment castings poured into Al<sub>2</sub>O<sub>3</sub> mold.

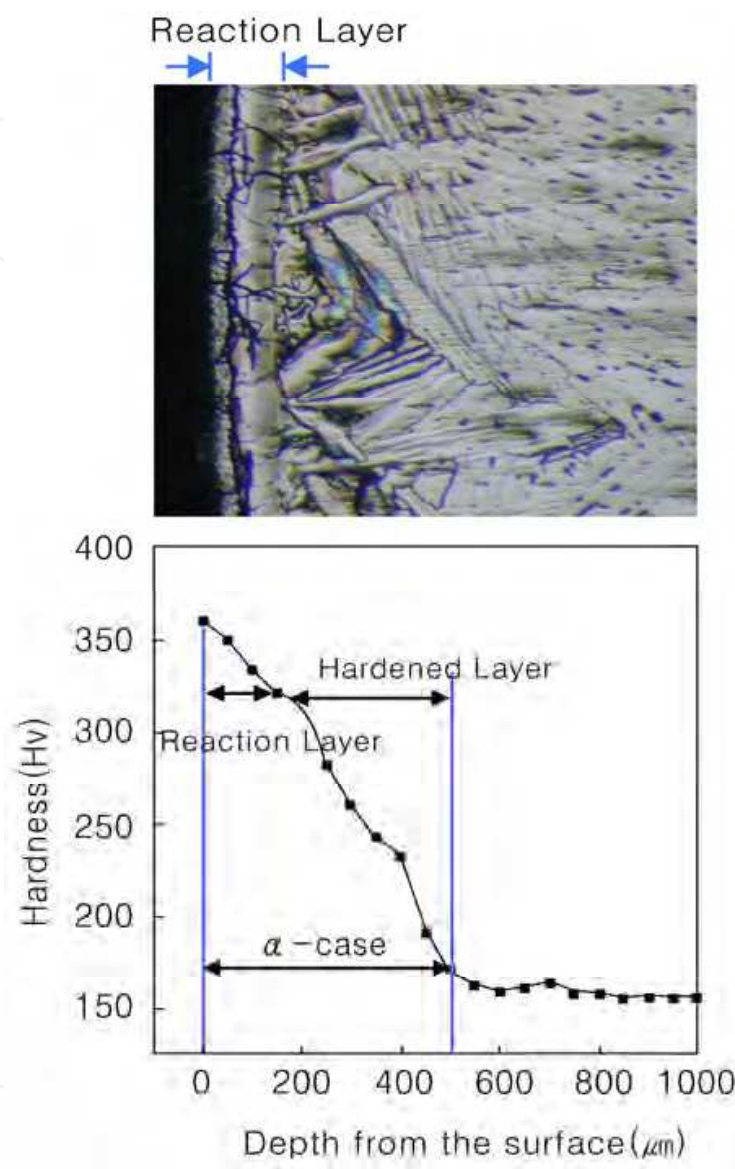
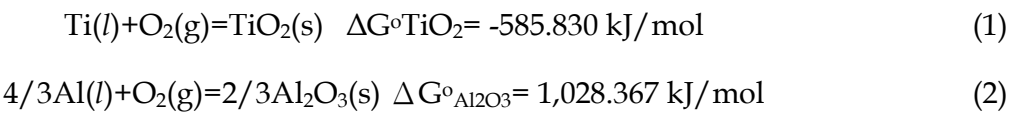


Fig. 3. Microstructure of the interface between Ti and Al<sub>2</sub>O<sub>3</sub> mold, and hardness profile.

The microstructure of a distinct reaction layer is about 200 μm thick on the interface between Ti and Al<sub>2</sub>O<sub>3</sub> mold. In addition to the reaction layer, there shows a hardened layer about 300 μm thick. The reaction layer and the hardened layer together are called the α-case of titanium castings. However, when the molten titanium (around 2,000 K) is poured into the Al<sub>2</sub>O<sub>3</sub> investment mold, and if the α-case results between Ti and the interstitial oxygen, the reaction could be described as follows:







The above calculations utilized the joint of army-navy-air force (JANAF) thermochemical tables [6]. According to the equation (3), the  $\alpha$ -case formation by interstitial oxygen cannot occur spontaneously. Therefore, the reason why the  $\alpha$ -case reaction is generated cannot be explained by the conventional  $\alpha$ -case formation mechanism.

### 3.2 Alpha-case formation mechanism

For the clear examination of the  $\alpha$ -case formation mechanism, the distribution of the elements of the  $\alpha$ -case and its chemical composition were investigated by EPMA elemental mapping as shown in Fig. 4. On the  $\alpha$ -case region, the oxygen element was uniformly distributed. Also, the Si element originating from the colloidal silica binder was scarcely detected on the surface.

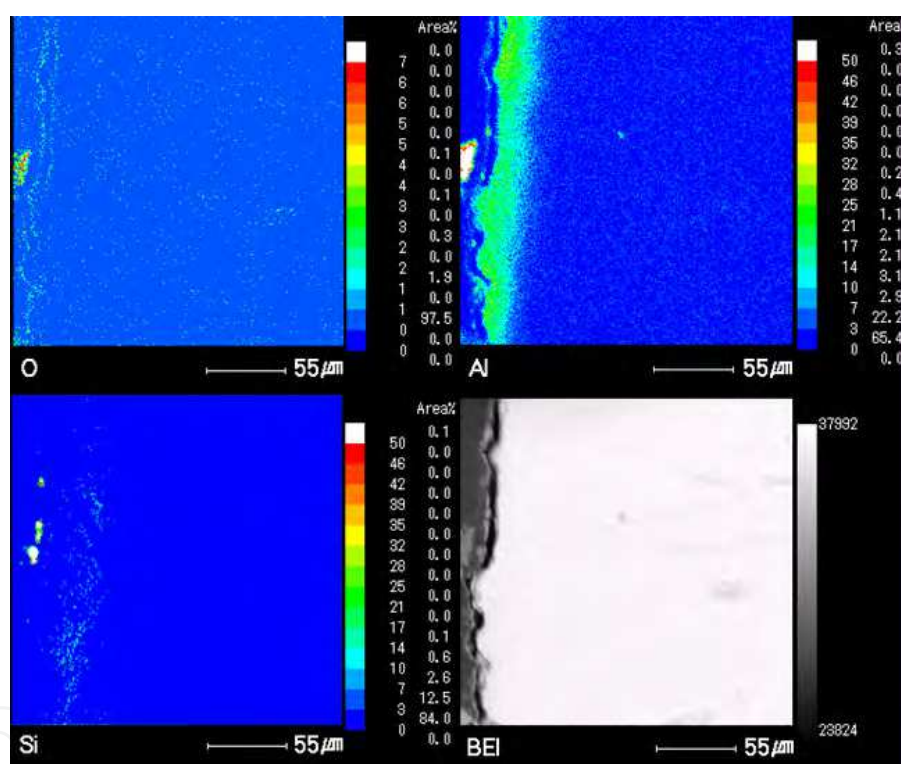


Fig. 4. Comparison of elemental mapping images of O, Al and Si in Ti castings into  $\text{Al}_2\text{O}_3$  mold and BEI image by EPMA.

However, the concentrated Al contamination layer about 30  $\mu\text{m}$  thick was detected on the interface. The EPMA mapping result shows that not only the interstitial oxygen elements but also the substitutional Al elements dissolved from the mold material affect the metal-mold reactions. Until recently, the effect of substitutional metallic element dissolved from mold materials has been ignored as negligibly small [7]. However, Fig. 3 and 4 indicate that the effect of metallic elements cannot be overlooked any more.

The phase identification of the detected Al element will be the very core of  $\alpha$ -case formation mechanism. The phases of the detected Al on the interface were examined by transmission

electron microscopy (TEM). In order to observe  $\alpha$ -case region precisely, the titanium castings specimen was grinded from inside to the surface until about 30  $\mu\text{m}$  and final thinning was carried out using ion milling. Fig. 5 is the cross-sectional bright field TEM image of the  $\alpha$ -case region.

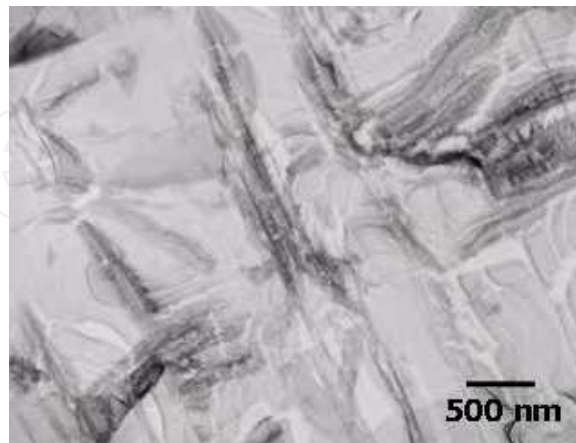


Fig. 5. Bright field TEM image of the  $\alpha$ -case region.

To examine what kinds of phases are presented in the  $\alpha$ -case region, C2 aperture was temporarily removed. Fig. 6 (a) shows ring and spot patterns on the TEM image without C2 aperture. The definite contrast of continuous ring pattern could not be found on the TEM image since the ring pattern was an extremely small size polycrystalline  $\text{TiO}_2$  phase. In the case of spot patterns on Fig. 6 (b), the contrast could be distinguished on the TEM image. And the indexed pattern was a hexagonal close-packed (HCP) phase in the  $[2\bar{1}0]$  beam direction as shown Fig. 6 (b).

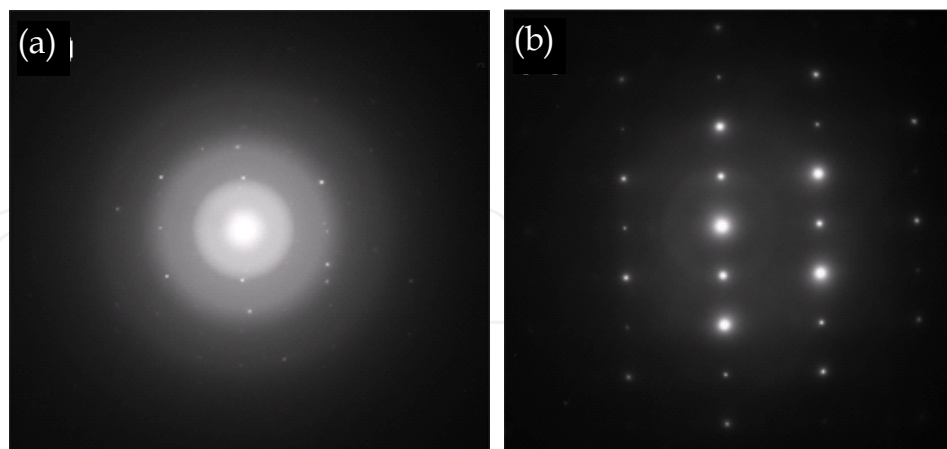


Fig. 6. Results of analytical transmission electron microscopy of (a) ring and spot patterns on the TEM image without C2 aperture on the bright field image, and (b) spot pattern on the TEM image was HCP phase in the  $[2\bar{1}0]$  beam direction.

The convergent beam electron diffraction (CBED) analysis was carried out for the verification of the phase of spot patterns with the primitive cell volume method [7]. The

primitive cell volume is characteristic material constant. So, the phase of interest can be easily identified by comparing the measured primitive cell volume of an unknown phase using CBED patterns with the known values of the possible phases. This method is accurate (error range of <10%) for the phase identification. In the CBED pattern where zero order Laue zone (ZOLZ) disk and high order Laue zone (HOLZ) ring are present, by measuring the distance of ZOLZ disk from the transmitted beam to the diffracted beam ( $D_1$ ,  $D_2$ ), an internal angle (ANG) between  $D_1$  and  $D_2$ , and HOLZ ring's radius (CRAD), the primitive cell volume of unknown phase can be easily determined using the equation (4). Camera Length (CL) was calibrated with Au standard sample at 200 keV.

$$\text{Cell volume} = \frac{CL^2 \cdot \lambda^3}{D_1 \cdot D_2 \cdot \sin(\text{ANG}) \left[ 1 - \cos \left\{ \tan^{-1} \left( \frac{\text{CRAD}}{CL} \right) \right\} \right]} \quad (4)$$

Fig. 7 (a) shows the CBED pattern of the HCP phase. There are two possible HCP phases of Ti and  $\text{Ti}_3\text{Al}$ . The measured primitive cell volume ( $61.08 \text{ ang}^3$ ,  $CL=521.4 \text{ mm}$ ,  $D_1=D_2=6.5 \text{ mm}$ ,  $\text{ANG}=60^\circ$ ,  $\text{CRAD}=23 \text{ mm}$ ) from CBED pattern corresponded to the theoretic value of  $\text{Ti}_3\text{Al}$  ( $66.60 \text{ ang}^3$ ). This phase identification was in accordance with EDS spot analysis as shown in Fig. 6 (b). The phase of the detected Al from EPMA mapping was  $\text{Ti}_3\text{Al}$  phase. Thus, considering the microstructure, hardness profile, EPMA mapping and TEM, the  $\alpha$ -case is not wholly  $\text{TiO}_2$ , but  $\text{TiO}_2$  and  $\text{Ti}_3\text{Al}$  between Ti and  $\text{Al}_2\text{O}_3$  mold.

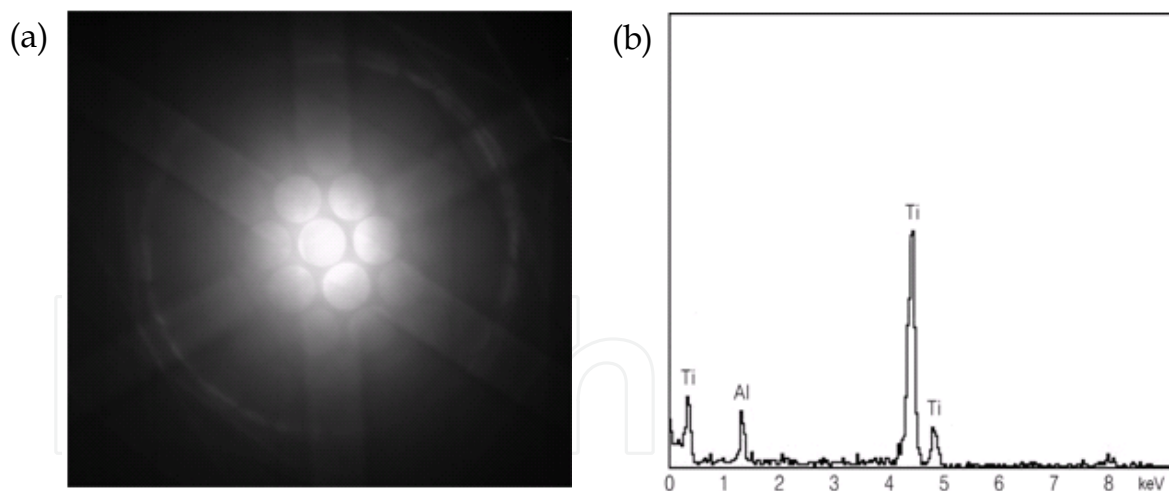


Fig. 7. Convergent beam electron diffraction analysis with the primitive unit cell volume method (a) The measured primitive unit cell volume,  $61.08 \text{ ang}^3$  ( $CL=521.4 \text{ mm}$ ,  $D_1=D_2=6.5 \text{ mm}$ ,  $\text{ANG}=60^\circ$ ,  $\text{CRAD}=23 \text{ mm}$ ,  $\lambda = 0.0251 \text{ \AA}$ ) and (b) EDS spot analysis result.

In this study, from the synthesis of the microstructure, hardness profile, EPMA mapping and TEM, it could be confirmed that the  $\alpha$ -case is formed by not only interstitial oxygen element but also substitutional metallic elements dissolved from mold materials as shown in Fig. 8.



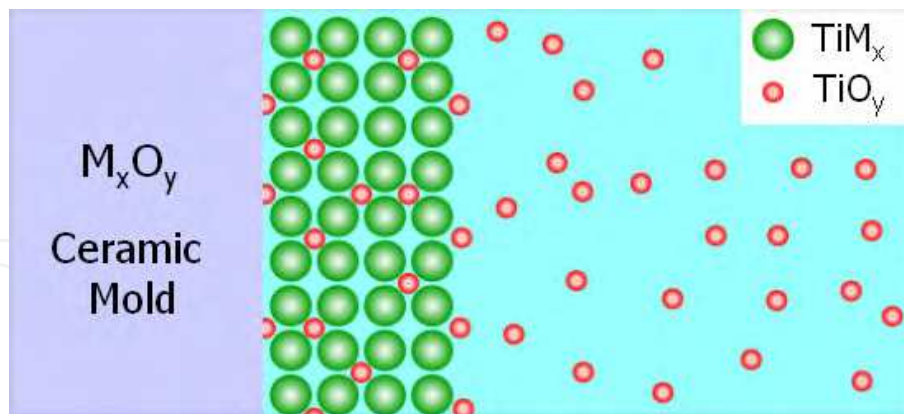


Fig. 8. Schematic diagram of the interstitial and substitutional  $\alpha$ -case formation mechanism of titanium castings.

#### 4. New alpha-case formation mechanism with alpha-case controlled mold

##### 4.1 Alpha-case controlled stable mold fabrication

The  $\alpha$ -case formation mechanism was applied to the development of  $\alpha$ -case controlled stable mold and verified by titanium alloy castings with the  $\alpha$ -case controlled mold materials. To synthesize the  $\alpha$ -case formation products, 10 wt% to 50 wt% of about 45  $\mu\text{m}$  titanium powders were added into  $\text{Al}_2\text{O}_3$  for the purpose of eliminating the  $\alpha$ -case. After blending, the slurry was prepared with the  $\text{Al}_2\text{O}_3$  based powder. The  $\alpha$ -case controlled stable mold manufacturing and titanium casting procedures were carried out at the same condition as mentioned above. The synthesized phases between titanium and  $\text{Al}_2\text{O}_3$  were identified by RIGAKU X-ray diffractometer.

##### 4.2 New alpha-case formation mechanism

Although the above mentioned  $\text{Al}_2\text{O}_3$  has the proper strength, permeability and collapse-ability enough to ensure dimensional accuracy of castings, it could not be applied for mold due to its severe  $\alpha$ -case formation. However, in this study, for the maximization of performance and cost-effectiveness of the investment mold, the  $\alpha$ -case controlled mold was designed on  $\text{Al}_2\text{O}_3$  base.

In consideration of the interstitial and substitutional elements in  $\alpha$ -case formation mechanism, 10 wt% to 50 wt% of titanium powders were blended with  $\text{Al}_2\text{O}_3$  for the previous synthesis of  $\alpha$ -case reaction products into the mold. Then the blended powders were mixed with the colloidal silica and agitated.

Fig. 9 shows the X-ray diffraction result of 50 wt% titanium mixed  $\text{Al}_2\text{O}_3$  powders before curing. The composition of the starting titanium and  $\text{Al}_2\text{O}_3$  remained unchanged after being mixed with colloidal silica. After the mixing and agitation, the mold materials were cured at 1,223 K for 2 hrs. According to XRD analysis,  $\text{TiO}_2$  and  $\text{Ti}_3\text{Al}$  phases were in-situ synthesized on the  $\text{Al}_2\text{O}_3$  base between titanium and  $\text{Al}_2\text{O}_3$  powders as shown in Fig. 10.

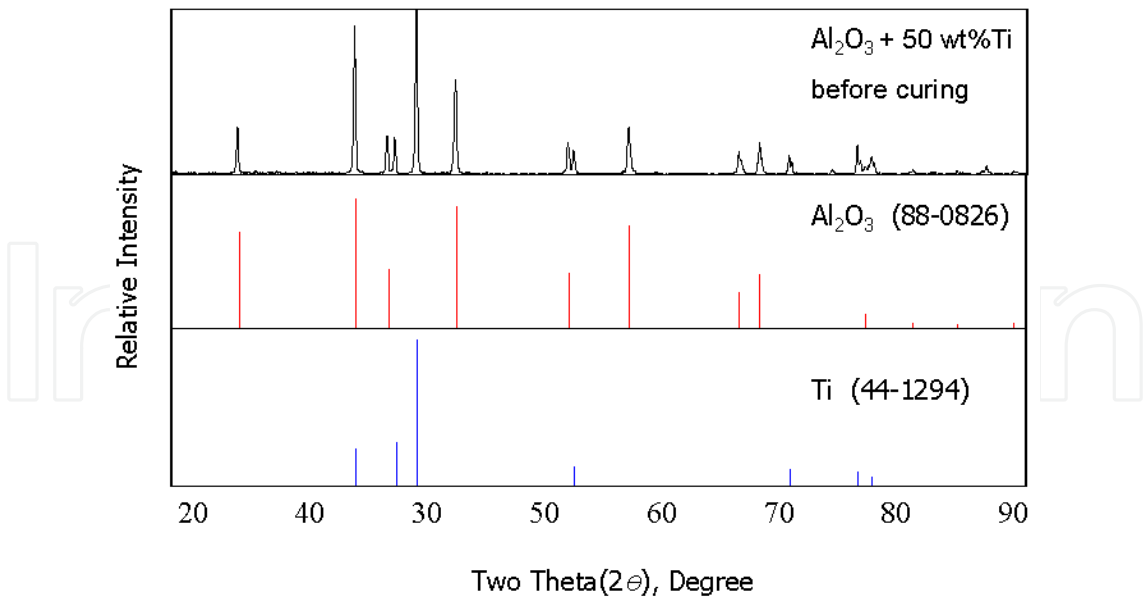


Fig. 9. Illustration of the diffraction patterns and replots for identifying phases in 50 wt% titanium and  $\text{Al}_2\text{O}_3$  mixed with colloidal silica before curing.

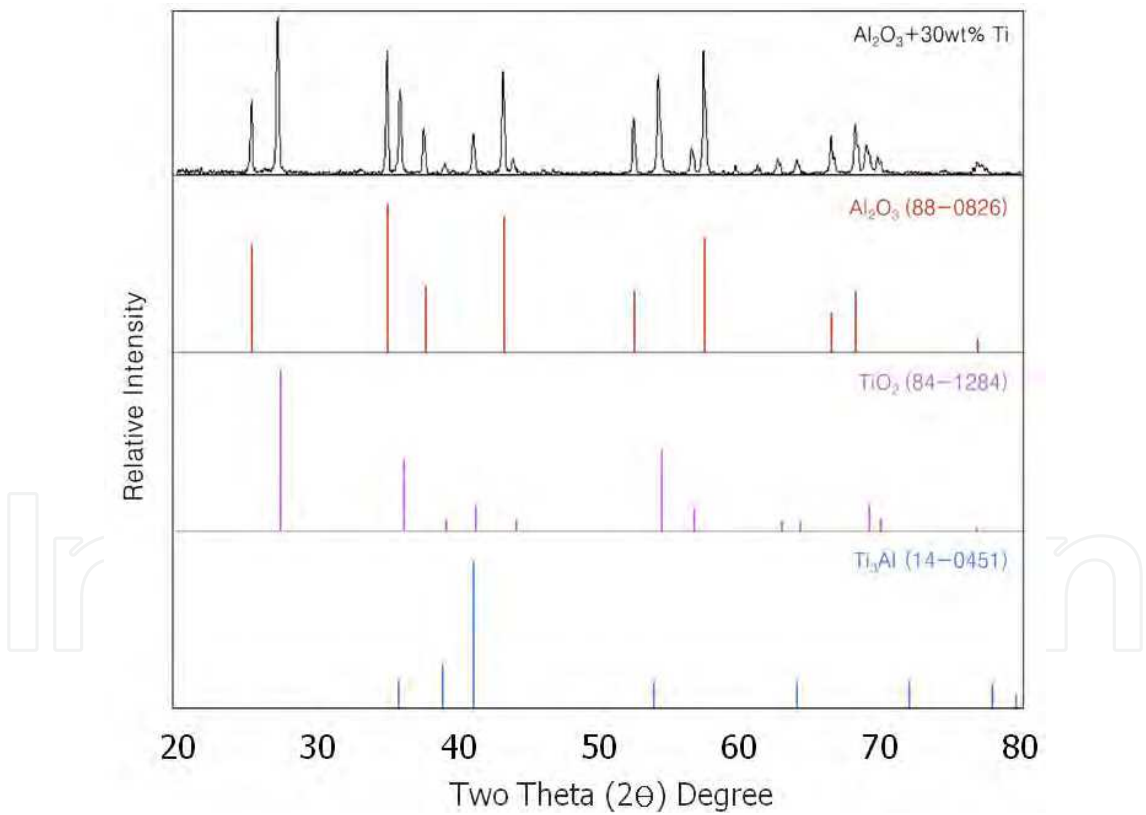


Fig. 10. X-ray diffraction pattern of synthesized  $\alpha$ -case controlled stable mold materials.

The scan angles were from 20° to 80° and the pattern was verified with JCPDS card numbers 88-0826 ( $\text{Al}_2\text{O}_3$ ), 84-1284 ( $\text{TiO}_2$ ) and 14-0451 ( $\text{Ti}_3\text{Al}$ ) [8]. No trace of the starting titanium reagent was detected by XRD analysis. Fig. 11 shows the variation of XRD patterns with the amount of titanium powders. The component ratio of  $\text{TiO}_2$  and  $\text{Ti}_3\text{Al}$  phases increased

gradually with the addition of titanium. Consequently, the synthesis of the  $\alpha$ -case formation products  $\text{TiO}_2$  and  $\text{Ti}_3\text{Al}$  phases into  $\text{Al}_2\text{O}_3$  based mold could be possible by the addition of titanium powders.

The effect of  $\alpha$ -case controlled stable mold which contained the interstitial metal-mold reaction product  $\text{TiO}_2$  and the substitutional metal-mold reaction product  $\text{Ti}_3\text{Al}$  was verified with titanium casting.

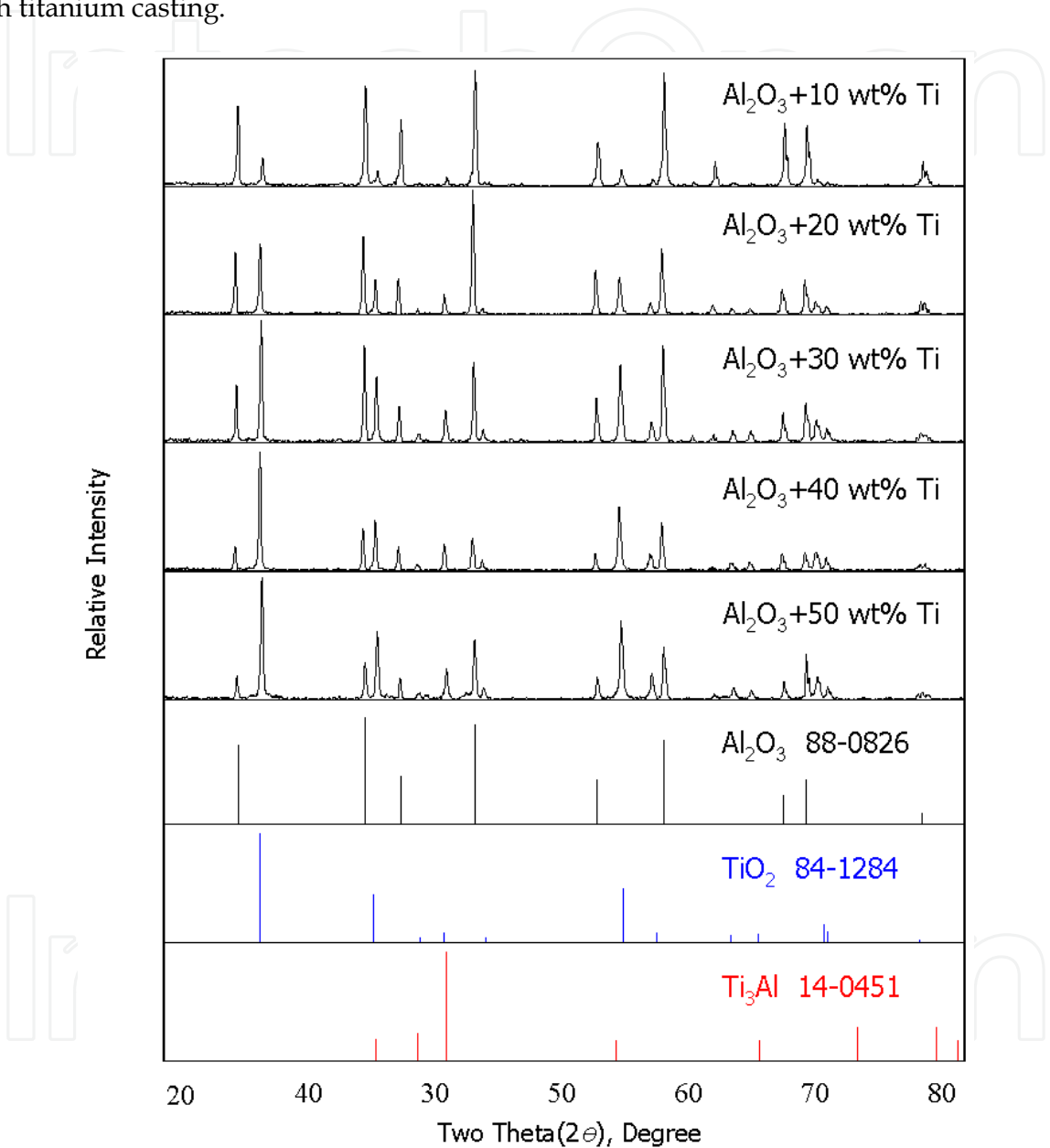


Fig. 11. Illustration of the diffraction patterns and replots for identifying phases from 10 wt% to 50 wt% titanium and  $\text{Al}_2\text{O}_3$  after curing at 1,223 K for 2 hrs.

Fig. 12 (a) indicates that in the case of  $\text{CaO}$  stabilized  $\text{ZrO}_2$ , the expensive and thermally stable mold, the externals of titanium castings lost metallic luster as a result of the  $\alpha$ -case formation reactions. However, in the  $\alpha$ -case controlled stable mold, its characteristic luster of titanium was well preserved as shown in Fig. 12 (b).

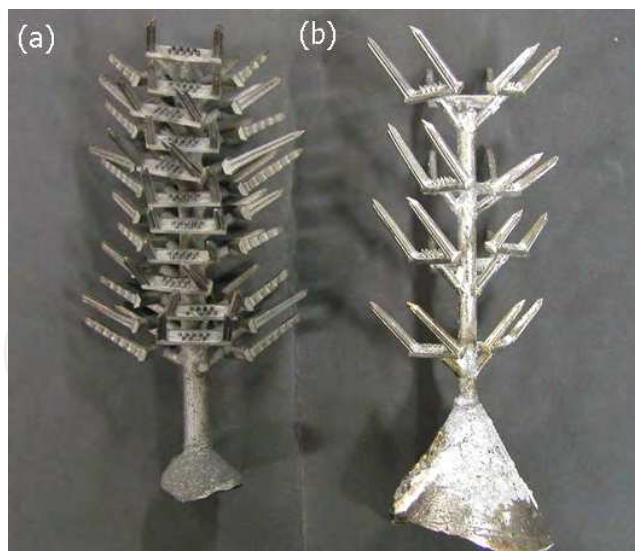


Fig. 12. Comparison of externals of titanium castings (a) CaO stabilized  $\text{ZrO}_2$  mold and (b) the  $\alpha$ -case controlled stable mold.

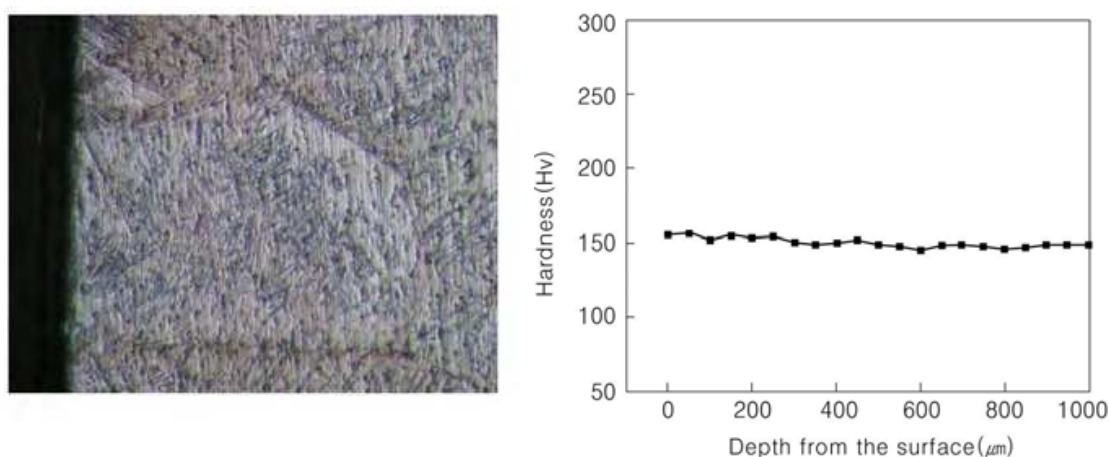


Fig. 13. Microstructure and hardness profile between pure titanium and  $\alpha$ -case controlled stable mold.

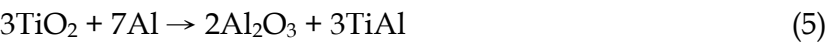
This external examination result of the  $\alpha$ -case controlled stable mold is in good accordance with the microstructure and hardness profile as shown in Fig. 13. The effect of prevention of  $\alpha$ -case formation can be obtained after addition of more than 2 wt% titanium.

#### 4.3 Noble synthesis of the alpha-case controlled stable mold

The  $\alpha$ -case controlled stable mold is much less expensive than the conventional mold materials for titanium such as  $\text{ZrO}_2$ , CaO stabilized  $\text{ZrO}_2$  and  $\text{Y}_2\text{O}_3$ , and the complete control of  $\alpha$ -case is possible. However, in this study, the noble synthesis route was conceived, which is more cost-effective than the above route by the inexpensive  $\text{TiO}_2$  and aluminum raw materials.

The homogeneous powders containing 30 mol%  $\text{TiO}_2$  powders (anatase, 45  $\mu\text{m}$ , 99% pure) and 70 mol% aluminum powders were prepared by blending. After blending, the powders were mixed with colloidal silica and agitated. The  $\alpha$ -case controlled stable mold

manufacturing and titanium casting procedures were carried out at the same condition as mentioned above. The 3:7 molar ratio of  $\text{TiO}_2$  and aluminum was chosen to yield the  $\text{Al}_2\text{O}_3$  and  $\text{TiAl}$  final product after in-situ synthesis according the following reaction.



In order to synthesize the  $\text{Al}_2\text{O}_3$  and  $\text{TiAl}$  phases, the mixed  $\text{TiO}_2$  and aluminum powders were cured at the 1,323 K for 2 hrs. However, the equation (5) is an ideal reaction, and thus it is very difficult to synthesize the ideal product by the simple blending and after agitation.

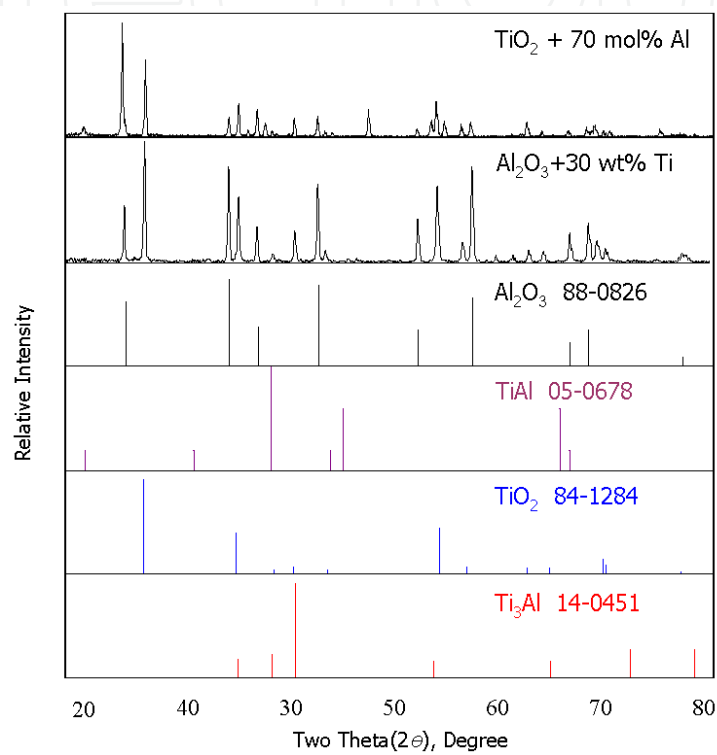


Fig. 14. Illustration of the diffraction patterns and replots for identifying the synthesized phases between  $\text{TiO}_2$  and aluminum powders.

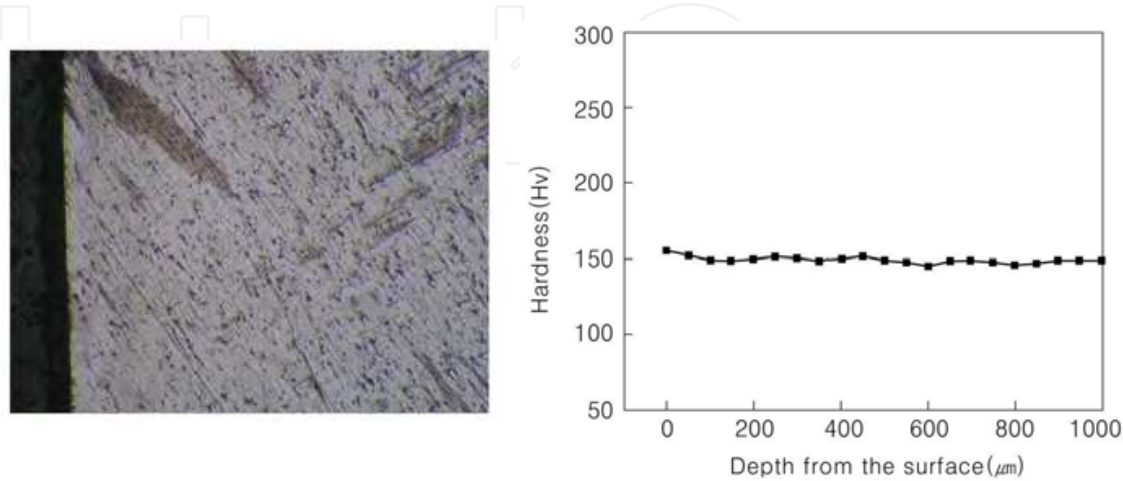


Fig. 15. Microstructure and hardness profile between pure titanium and noble route  $\alpha$ -case controlled stable mold.



According to the XRD analysis as shown in Fig. 14, the major phases are anatase  $\text{TiO}_2$  and rutile  $\text{TiO}_2$  as well as the in-situ synthesized  $\text{Al}_2\text{O}_3$ ,  $\text{Ti}_3\text{Al}$  and  $\text{TiAl}$  phases since the partially non-reacted anatase  $\text{TiO}_2$  remained and the  $\text{TiAl}$ ,  $\text{Ti}_3\text{Al}$  intermetallic compounds and rutile  $\text{TiO}_2$  were synthesized between anatase  $\text{TiO}_2$  and aluminum powders. As a result, the synthesis which is similar to the titanium powders added  $\alpha$ -case controlled stable mold can be obtained by the economical route. The  $\alpha$ -case free titanium casting can be possible by the noble route synthesized  $\alpha$ -case controlled stable mold which contained the  $\alpha$ -case formation products such as  $\text{Al}_2\text{O}_3$ ,  $\text{TiO}_2$ ,  $\text{TiAl}$  and  $\text{Ti}_3\text{Al}$  phases as shown in Fig. 15.

## 5. Conclusion

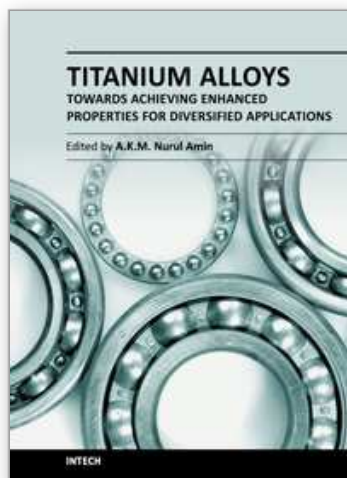
By the conventional  $\alpha$ -case formation mechanism regardless of thermodynamic approaches, the alpha-case generation cannot be explained. In order to ascertain the reason,  $\alpha$ -case formation mechanism was closely examined and from the mechanism, two kinds of  $\alpha$ -case controlled stable molds were developed.

1. Regardless of thermodynamic approaches, about 500  $\mu\text{m}$  thick  $\alpha$ -case was generated between titanium and  $\text{Al}_2\text{O}_3$  mold. The reason why the  $\alpha$ -case generated cannot be explained by the conventional  $\alpha$ -case formation mechanism, which is known to be formed by the interstitials, especially oxygen dissolved from mold materials.
2. In spite of having used pure titanium, the concentrated aluminum contamination layer about 30  $\mu\text{m}$  thick was detected on the interface by the EPMA elemental mapping. From the results of the TEM phase identification, the phase of the detected aluminum from EPMA mapping was identified as  $\text{Ti}_3\text{Al}$  phase.
3. Considering the microstructure, hardness profile, EPMA mapping and TEM analysis, it could be confirmed that the  $\alpha$ -case is formed not only by interstitial oxygen element but also by substitutional metallic elements dissolved from mold materials.
4. The synthesis of the  $\text{TiO}_2$  and  $\text{Ti}_3\text{Al}$  phases which are the  $\alpha$ -case reaction products between titanium and  $\text{Al}_2\text{O}_3$  mold can be obtained by the simple curing of  $\text{Al}_2\text{O}_3$  mold added titanium powders. The complete control of  $\alpha$ -case formation can be possible with the  $\alpha$ -case controlled stable mold.
5. The  $\alpha$ -case free titanium casting can be possible by the noble route synthesized  $\alpha$ -case controlled stable mold which is in-situ synthesized between  $\text{TiO}_2$  and aluminum powders to obtain the  $\alpha$ -case formation product such as  $\text{Al}_2\text{O}_3$ ,  $\text{TiO}_2$ ,  $\text{TiAl}$  and  $\text{Ti}_3\text{Al}$  phases.
6. Consequently, the economical net-shape forming of titanium alloys without  $\alpha$ -case formation and the verification of a newly established  $\alpha$ -case formation mechanism, can be possible using the  $\alpha$ -case controlled stable molds.

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## **Titanium Alloys - Towards Achieving Enhanced Properties for Diversified Applications**

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The first section of the book includes the following topics: fusion-based additive manufacturing (AM) processes of titanium alloys and their numerical modelling, mechanism of  $\alpha$ -case formation mechanism during investment casting of titanium, genesis of gas-containing defects in cast titanium products. Second section includes topics on behavior of the  $(\alpha + \beta)$  titanium alloys under extreme pressure and temperature conditions, hot and super plasticity of titanium  $(\alpha + \beta)$  alloys and some machinability aspects of titanium alloys in drilling. Finally, the third section includes topics on different surface treatment methods including nanotube-anodic layer formation on two phase titanium alloys in phosphoric acid for biomedical applications, chemico-thermal treatment of titanium alloys applying nitriding process for improving corrosion resistance of titanium alloys.

### **How to reference**

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