# Thermal Stability and Degradation Properties of Aluminide Coated and Uncoated Ti-6AI-4V Alloys Exposed to High Temperature Flame

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#### Abstract

The thermal stability of aluminide coated and uncoated Ti-6Al-4V(Ti-64) titanium alloys in high temperature flame environments has been investigated. The pack cementation method was used to apply the aluminide coatings. The TiAl<sub>3</sub> layer was formed after heat treatment of coated pure Titanium and Ti alloys at 500 °C–700 °C. After being exposed to a high temperature flame, the uncoated titanium alloys formed TiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub>, resulting in a significant weight gain and the loss of the original shape. However, when the aluminide coated titanium alloys were exposed to the flame, a thin layer of Al<sub>2</sub>O<sub>3</sub> was produced as a result of the flame exposure, and the coated specimens maintained their original shape in high temperature flame environments when exposed at ~ 1360 °C for 15 min. The detailed mechanisms for flame resistance of coated specimens, as well as microstructural observations were examined. The temperature distribution of the specimen as well as the stability of the coating layer under flame exposure were discussed systematically.

Keywords Pack cementation · Ti-64 alloy · Coatings · Flame test · Heat treatment

## 1 Introduction

Pure titanium and titanium alloys are widely used in various shapes in the automobile industry, aerospace, and marine industry due to their high specific strength, rigidity, corrosion resistance, and excellent creep properties [1–3]. However, for titanium alloys, oxygen in the atmosphere begins to diffuse into the titanium surface at 400 °C, and as the temperature rises, the TiO<sub>2</sub> oxide layer begins to form [4]. Furthermore, the phenomenon of "alpha case," which is formed on the surface at temperature of 900 °C or higher due to the reaction of the titanium surface layer with oxygen observed with the nature

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of high hardness and brittleness. Because of oxide formation, the mechanical properties of titanium alloy are severely deteriorated, resulting in surface fracture at high temperatures [5]. To enhance the titanium alloy used at higher temperatures, the surface oxidation must be prevented by building protective layer on the surface protective coatings. There are several coating methods such as paint brushing, plasma coating, pack cementation, and chemical vapor deposition had been utilized for alloy coatings [6-10]. Xiong et al. [11] reported the Ti60 alloy which was coated with enamel and vitrified at 900 °C for 30 min by using paint brush method and oxidation experiments were carried out at 700 °C and 800 °C for up to 100 h. Wei et al. [12] observed that by Cr plasma coating to Ti-64 alloy resulted in better cyclic oxidation resistance than NiCrAlY thermal insulation coating after oxidation at 650 °C, 750 °C, and 850 °C. Yang et al. [13] observed the growth kinetics of the coating layer as well as the formation of TiAl<sub>3</sub> phase on the Ti-64 alloy through Al coating by using pack cementation to generate an Al<sub>2</sub>O<sub>3</sub> protective layer on the surface. The diffusion coating method in which the adhesion between the base material and the coating layer is high and the diffusion coating layer is generated inside is essential. Therefore, the diffusion coating methods divided into pack cementation and Chemical Vapor Deposition (CVD) method. It is difficult to fabricate a uniform coating layer on all surfaces of the specimen with



CVD coatings, whereas pack cementation coatings provide every surfaces at the same time as well as coatings on complex surface of substrate. The pack cementation coatings have been studied for improving oxidation resistance in various titanium alloys. There are reports of oxidation resistance evaluated after aluminide coating by performing static oxidation or flame environment tests [14–16]. The studies to predict the coating cycle using computational simulation or aluminide coating in various material systems have been reported [17, 18], and there are several reports on the oxidation resistance of aluminide coatings and coated alloys [19–22]. However, detailed structural evaluations when the alloys are exposed to high-temperature flames have not been well reported.

In this study, the structural behaviors of uncoated and aluminide coated Ti-6Al-4V (Ti-64) alloy were investigated under the Liquefied Petroleum Gas (LPG) flame at the temperature of ~ 1360 C for 15 min and abrasion resistance and oxidation resistance analyzed. The microstructures of the specimens, as well as the coating kinetics, were examined in order to analyze the effect of TiAl<sub>3</sub> formed by aluminide coatings. Further, the Al<sub>2</sub>O<sub>3</sub> layer produced by flame exposure of the TiAl<sub>3</sub> coating layer was examined for the coated Ti-64 alloys. Finally, to identify the heat transfer and dissipation, the effect of flame exposure with specimen condition was discussed.

#### 2 Experimental

The pellet with a diameter of 20 mm and thickness of 5 mm of pure titanium and Ti-64 alloys were purchased. The pure titanium and Ti-64 pellets were polished with fine Al<sub>2</sub>O<sub>3</sub> powders and ultrasonically cleaned to investigate the coating kinetics. The composition of the alloy was determined by using Energy Dispersive Spectrum (EDS) analysis on the cleaned specimens. Table 1 shows the chemical compositions of pure titanium and Ti-64 alloy. A milling machine was used to blend a powder mixture of 30 wt. % Al (Objective), 5 wt. % AlCl<sub>3</sub> (Activator), and 65 wt. % Al<sub>2</sub>O<sub>3</sub> (Dummy) for aluminide pack cementation coatings. The pure titanium and Ti-64 pellets with the powder mixture were placed in an alumina crucible and heat treated at 500, 600, and 700 °C in an Ar atmosphere for 6, 12, and 25 h. At high temperatures, Al vapors were deposited on the specimen by volatile halide species in a mixed powder pack. The detailed procedure is available elsewhere [10]. Figure 1 depicts a schematic of the pack cementation process.

Figure 2a shows the setup for the flame test of Ti-64 alloys specimen by using LPG gas. To test oxidation

Table 1 Composition analysis of pure titanium and Ti-64 alloys

Substrate	Ti	Al (wt. %)	V (wt. %)
Pure titanium	bal	0.0092	0.0058
Ti-64 alloy	bal	5.7466	3.8224

resistance, uncoated and coated Ti-64 alloys were exposed to dynamic flame environments from LPG and oxygen gas at ~1360 °C for up to 15 min. Figure 2b shows the flame temperature remains constant after 1 min. The TiO<sub>2</sub> phase formed at 1360 °C, hence this temperature was selected to analyze whether the protective layer formed through coating improves oxidation resistance. In addition, in the case of a gas turbine engine, the operating temperature is 1300 °C and above, the efficiency is expected to increase by more than 50% [23]. The pyrometer was used to measure the temperature of the specimen, and the maximum temperature of the Ti-64 alloy was ~ 1360 °C. The studied specimens were exposed to flame at different time from 1 to 15 min. The mass changes of the uncoated and coated Ti-64 alloys were measured following the high temperature flame test. The X-ray diffraction (XRD, D/Max 2500H, Rigaku) and CuKa radiation with an operating voltage of 40 V were used to determine the phase composition. The surface morphology and cross section were examined with a Scanning Electron Microscope (SEM, JEOL-6300) equipped with an Energy Dispersive Spectrum (Table 2).

#### **3** Result and Discussion

Figure 3 shows the images of uncoated Ti-64 alloy and the coated Ti-64 specimen. The alloy was gray in color before the coating, but the coated specimen was silver due to the aluminide coating. When pack cementation coatings were applied at temperatures ranging from 500–700 °C, the possible formation phases includes Ti<sub>3</sub>Al, TiAl, TiAl<sub>2</sub>, and TiAl<sub>3</sub> as well as other aluminides, previous research confirmed that TiAl<sub>3</sub> is formed at the outermost layer due to concentration gradient [8]. Therefore, the TiAl<sub>3</sub> coating layer formed on the outermost layer is expected to improve the oxidation resistance of the test piece by forming an Al<sub>2</sub>O<sub>3</sub> layer in the high-temperature oxidation tests.

The SEM images of the surface of uncoated and coated Ti-64 alloy after heat treated at 600 °C for 6 h are shown in Fig. 4a, b. Both specimens have a similar morphology and the coated layer is made up of agglomerated spherical



Fig. 1 Schematic of pack cementation process



Fig. 2 a The image of flame test b temperature variation of Ti-64 alloy with respect to exposure time

 Table 2
 Kinetic
 parameter, k
 values at different temperatures for coated pure titanium and Ti-64 alloy

Temperature	Pure titanium	Ti-64 alloy	
500 °C	$1.63 \times 10^{-15} \text{ m}^2/\text{s}$	$8.6 \times 10^{-17} \text{ m}^2/\text{s}$	
600 °C	$2.75 \times 10^{-14} \text{ m}^2/\text{s}$	$5.7 \times 10^{-14} \text{ m}^2/\text{s}$	
700 °C	$5.8 \times 10^{-14} \text{ m}^2/\text{s}$	$4.2 \times 10^{-13} \text{ m}^2/\text{s}$	



Fig. 3 The image of specimen **a** uncoated Ti-64 alloy **b** coated Ti-64 alloy

small particles. Figure 5 shows the typical XRD results of uncoated Ti-64 alloy and coated Ti-64 alloy at 600 °C and 700 °C for 6 h. The  $\alpha$ -Ti phase was confirmed in the uncoated Ti-64 alloy, and the TiAl<sub>3</sub> phase formed confirmed on the surface of coated specimen. Similar studies have been published, in which the outmost TiAl<sub>3</sub> layer was observed for coated Ti alloys [24–26]. Even though different pack cementation compositions and coating conditions, the formation of the TiAl<sub>3</sub> outer layer was reported when titanium base alloys were coated with Al pack cementation coating [11, 27–30].

Figure 6a, b shows a cross-sectional SEM images of coated pure titanium and Ti-64 alloy at 600 °C for 6 h. The pure titanium and Ti-64 alloys were coated at the same condition to investigate the effect of specimen composition on the coating layer. The TiAl<sub>3</sub> layer was uniformly appeared on the surface of specimens. As seen in the cross-sectional images, the thickness of TiAl<sub>3</sub> layer on pure titanium and Ti-64 alloy are approximately 35 µm and 45 µm, respectively. The thickness of the coated layer on Ti-64 alloy is larger than that of pure titanium under the same conditions. possibly due to the presence of Al in the Ti-64 alloy. On the other hand, some cracks appeared on the coating layers because of internal stress caused by a mismatch in the coefficient of thermal expansion (CTE) between the TiAl<sub>2</sub> layer and the specimen during the cooling time after the pack cementation process. The CTE of pure titanium, Ti-64 alloy, and TiAl<sub>2</sub> have been reported to be  $8.6 \times 10^{-6}$  K<sup>-1</sup>,  $9.0 \times 10^{-6}$  K<sup>-1</sup>, and  $15 \times 10^{-6}$  K<sup>-1</sup>, respectively. According to Koo et al. [24] the ductility of TiAl<sub>3</sub> was reduced while the hardness increased compared to the substrate. However, it has been reported that micro cracking does not cause significant microstructural and/or compositional changes due to the coatings excellent adherence to the underlying substrate [27].

Figure 7 shows the coating layer thickness as a function of coating time for the specimen. When the growth kinetics were plotted against time, the relationship could be written as  $x = \sqrt{kt}$  where x is the thickness of the coating layer, k is the kinetic parameter, and t is the time. The estimated k values for pure titanium specimen are  $1.63 \times 10^{-15}$ ,  $2.75 \times 10^{-14}$ , and  $5.8 \times 10^{-14}$  m<sup>2</sup>/s, while for Ti-64 alloy are  $8.6 \times 10^{-17}$ ,  $5.7 \times 10^{-14}$ , and  $4.2 \times 10^{-13}$  m<sup>2</sup>/s at temperatures of 500 °C, 600 °C, and 700 °C, respectively. The kinetic parameters increased with increasing temperature and heat treatment



Fig. 4 SEM image of the surface a uncoated Ti-64 alloy b coated Ti-64 alloy at 600 °C for 6 h



**Fig. 5** XRD of the **a** uncoated Ti-64 alloy **b** coated Ti-64 alloy after the pack cementation coatings at 600 °C and **c** 700 °C, for 6 h

time, and the coating thickness increased with increasing coating time, as shown in Fig. 7a, b.

The activation energy of coated pure titanium and Ti-64 alloy was calculated by using log (k) values and temperatures. When the log (k) was plotted against temperature, the relationship could be written as, log  $(k) = \log (ko) - \frac{Q}{2.3R} \left[\frac{1}{T}\right]$ , where Q is the activation energy, R is the gas constant, and T is the temperature. The activation energy and temperature range for coated pure titanium and Ti-64 alloy are compared with literature and given in Table 3.

According to Fig. 8, the calculated activation energy for pure titanium and Ti-64 alloy are 57.6 and 96.5 kJ/mol, respectively. The activation energy values for the formation of aluminides, including diffusion coatings, are shown in Table 3. The activation energies varied between 33 and



Fig. 6 Cross-sectional SEM image of the coated specimens of a pure titanium b Ti-64 alloy heat treated at 600 °C for 6 h



Fig. 7 Variation of thickness with coating time of a pure titanium b the Ti-64 alloy at 500 °C, 600 °C, and 700 °C

Table 3 Comparison of activation energy for the formation of  ${\rm TiAl}_3$  during Ti and Al reaction

Composition	Range of T (°C)	Q (kJ/mol)	References
TiAl/Al or Ti-(5, 10, 25 wt.%)	516-640	95	[20]
Al/Al(purity as above)			
OT41alloy (Ti-1.0-25Al- 1.0Mn (wt. %))/Al	530–630	105.1	[33]
99.99% Ti/99.99% Al	540-650	34	[21]
99.5% Ti/99.5% Al	525-575	33.2	[32]
TiI30,TiI50 alloy	700–920	97	[ <mark>19</mark> ]
Pure titanium/Al	500-800	57.6	This work
Ti-64 alloy/Al	500-800	96.5	This work

105 kJ/mol. The calculated activation energy in this investigation were within the previously reported range.

The pictures of uncoated and coated Ti-64 alloys exposed in a high temperature flame at 1360 °C is shown in Fig. 9. When an uncoated Ti-64 alloy is exposed to high temperatures, the titanium in the alloy reacts with oxygen, resulting in the formation of TiO<sub>2</sub>, and the surface turns brown. Furthermore, the Ti-64 alloy lost its original shape, and the oxidized pieces peeled off from the substrate, indicating that the alloy was severely damaged by the flame. However, when the aluminide coated Ti-64 alloys were exposed to flame, the situation changed noticeably. The original shapes of the Ti-64 alloys were perfectly preserved even after 15 min of flame exposure.

Figure 10 depicts XRD patterns of uncoated and coated Ti-64 alloy after 3 min of flame exposure. When the uncoated alloy was exposed in the flame environment



Fig. 8 Variation of log k with respect to reciprocal of temperature of coated pure titanium and Ti-64 alloy

without coatings, the  $TiO_2$  phase was identified, as shown in Fig. 10a, and the  $TiAl_3$ ,  $TiO_2$ , and  $Al_2O_3$  phases were detected in the coated specimen, as shown in Fig. 10b. The  $TiAl_3$  coating layer remained on the coated specimen, and  $Al_2O_3$  served as the primary protective layer. However, XRD analysis of the uncoated specimen reveals that  $TiO_2$  was a main phase that was attributed to causing serious damage to the Ti-64 specimen.

Figure 11 shows the surface morphology of the coated Ti-64 alloy after 3 min of flame exposure. Many particles on the surface appear to be mixed and then unevenly stacked following the flame. To maintain mass balance, Al should







Fig. 10 XRD pattern of a uncoated b coated Ti-64 alloy after flame tests for 3 min

diffuse out of  $TiAl_3$  when  $Al_2O_3$  is generated by the reaction  $TiAl_3$  and oxygen [3]. The EDS analysis show the compositional values of the oxide surface in Fig. 11b. The existence

of  $Al_2O_3$  and  $TiO_2$  phases in the material is confirmed by EDS measurements.

Figure 12 shows the cross-sectional image of Ti-64 alloy after 3 min flame test of aluminide coated specimen under the condition of 600 °C 6 h. The residual TiAl<sub>3</sub> layer is about 6.08 µm and EDS measurements identified a thin Al<sub>2</sub>O<sub>3</sub> layer with a thickness of ~ 1 µm. However, due to the brittle nature of Al<sub>2</sub>O<sub>3</sub>, several parts of the scale detached from the surface during the polishing process. The thickness of the remaining TiAl<sub>3</sub> layer is thin for Ti-64 specimen exposed 3 min flame test, flame tests were carried out for up to 15 min to check whether the specimen could be protected during the flame test for a longer period of time.

The percentages of the remaining mass to the initial mass of the uncoated and coated Ti-64 alloys were plotted about the exposure period in order to detect oxidation kinetics, as shown in Fig. 13. After 3 min of exposure to the high temperature flame, the remaining mass of the uncoated Ti-64 alloys increases considerably while the mass of the coated alloy remained nearly constant in the coated specimen. In other words, due to the creation of Al<sub>2</sub>O<sub>3</sub> during flame exposure durations, coated Ti-64 alloys displayed exceptional oxidation resistance. Because Al<sub>2</sub>O<sub>3</sub> is formed, it is assumed that the oxidation mechanism of titanium aluminides during



Fig. 11 SEM image of coated Ti-64 alloy after flame exposure at 1360 °C for 3 min



Fig.12 Cross-sectional SEM image of the coated Ti-64 alloy after flame test at 1360  $^\circ C$  for 3 min



Fig. 13 Remaining mass percent of the uncoated and coated Ti-64 alloy after flame tests with respect to time

flame exposure is comparable to that of static oxidation [3]. Because of its extremely low permeabilities for hydrogen, nitrogen, oxygen, and metal ions, the production of  $Al_2O_3$  scale is known to provide an oxidation barrier on many alloys [27]. Furthermore,  $Al_2O_3$ , which has a thermal conductivity coefficient of less than 30 W/mK, serves as a thermal barrier on the Ti-64 alloy [3, 31].

The schematic diagram of flame test is shown in Fig. 14 for coated and uncoated Ti-64 alloy. When a high-temperature flame test was performed on Ti-64 specimens at 1360° C. for up to 15 min, the weight of the uncoated specimens increased significantly due to formation of TiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> oxides as the time increased. The pieces of oxides peeled off in case uncoated specimen as flame expose time increased. The coated specimen weight change before and after flame exposure almost negligible and which is attributed to the



Fig. 14 Schematic of flame test for coated and uncoated Ti-64 alloy

 $Al_2O_3$  layer formation, which is acts as anti-oxidation layer which was formed in a high-temperature environment in TiAl<sub>3</sub> layer. The observed thickness of  $Al_2O_3$  was about ~ 1 µm, when the flame test was performed at 1360° C for 3 min. Therefore, it shows the effect of Al pack cementation coating in the high temperature oxidation test on Ti-64 alloy. However, further study is needed in order to identify the degradation upon various temperature, the current study clearly supports the efficiency of aluminide coatings and expands the use of Ti alloys in high-temperature flame environments.

### 4 Conclusions

The oxidation stability of coated and uncoated Ti-64 alloys was examined in this work by using the flame test. Under the argon environment, pure titanium and Ti-64 alloys were pack cementation coated with a powder mixture of Al, AlCl<sub>3</sub>, and Al<sub>2</sub>O<sub>3</sub>, resulting in the formation of titanium aluminide  $(TiAl_3)$ . At the same condition, the activation energy of pure titanium and Ti-64 alloy was 51.4 and 96.5 kJ/mol, respectively. In order to judge the feasibility of the Ti-64 alloys for use as high temperature application, the TiAl<sub>3</sub> coated and uncoated Ti-64 alloy was exposed to high temperature flame over 1300 °C up to 15 min. With the increasing exposure time, the TiO<sub>2</sub> layer formed on the bare Ti-64 alloys, and TiO<sub>2</sub> layer was peeled off from the specimen surface. However, when the aluminide-coated Ti-64 alloy was exposed to a high temperature flame, the residual mass percent remained nearly constant, owing to the creation of a protective Al<sub>2</sub>O<sub>3</sub> layer. It suggest that the TiAl<sub>3</sub> coated Ti-64 alloy is suitable for high temperature applications due to the formation of Al<sub>2</sub>O<sub>3</sub>.

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#### Declarations

**Conflict of interest** The authors declared that they have no conflict of interest to this work.

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