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# Improving Hot Corrosion Resistance of Two Phases Intermetallic Alloy $\alpha_2$ -Ti<sub>3</sub>Al/ $\gamma$ -TiAl with Enamel Coating

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**Abstract.** TiAl intermetallic alloys have attracted great interest among aerospace industry after successful utilization in low pressure turbine blades of aircraft engine which makes dramatic weight saving up to 40% weight saving. However, poor oxidation and corrosion resistance at temperatures above 800°C still become the drawbacks of this alloys, making the development of protective coatings to improve the resistance is important. This study investigates the hot corrosion behavior of two phases intermetallic alloy  $\alpha_2$ -Ti<sub>3</sub>Al/ $\gamma$ -TiAl with and without enamel coating using immersion test method in molten salt of 85%-wt Na<sub>2</sub>SO<sub>4</sub> and 15%-wt NaCl at 850°C. The results show after 50 hours of hot corrosion test, bare alloy showed poor hot corrosion resistance due to the formation of non-protective Al<sub>2</sub>O<sub>3</sub> + TiO<sub>2</sub> mixed scale at the surface of the alloy. Improvement of hot corrosion resistance was obtained in samples protected with enamel coating, indicated by significant decreasing in mass change (mg/cm<sup>2</sup>) by 98.20%. Enamel coating is expected to has the capability in suppressing the diffusion of oxygen and corrosive ions into the substrate layer, and consequently, it improves hot corrosion resistance of the alloy. The study showed that enamel coatings have strong adherent to the substrate and no spallation was observed after hot corrosion test. Nevertheless, the dissolution of oxides components of the enamel coating into the molten salts was observed that lead enamel coating degradation. This degradation is believed involving Cl<sup>-</sup> anion penetration into the substrate through voids in the coating that accelerates the corrosion of the two phases  $\alpha_2$ -Ti<sub>3</sub>Al/ $\gamma$ -TiAl alloy. Even though further observations are needed, it appears that enamel coating could be a promising protective coating to increase hot corrosion resistance of TiAl intermetallic alloys.

**Keywords:**  $\alpha_2$ -Ti<sub>3</sub>Al/ $\gamma$ -TiAl intermetallic, hot corrosion, enamel coating, surface treatment

## INTRODUCTION

Efficient energy conversion systems require lighter materials and higher operating temperatures. In order to achieve that in an aircraft engine, two phases  $\alpha_2$ -Ti<sub>3</sub>Al/ $\gamma$ -TiAl intermetallic alloy has been considered as one of material for replacing nickel base superalloys due to its low density, good structural stability, and high specific strength<sup>1,2</sup>.  $\alpha_2$ -Ti<sub>3</sub>Al phase has good ductility at room temperatures while  $\gamma$ -TiAl phase has high oxidation resistance even though still inadequate for very high temperature applications<sup>2</sup>. Until now, successful utilization of  $\gamma$ -TiAl in low pressure turbine of GENx engines is considered as the greatest breakthrough in the TiAl technology<sup>3,4</sup>. However, to replace more components in aircraft engine, further developments of alloys which can withstand from hot corrosion resistance and oxidation attacks at higher temperature in aircraft engine are needed. Improvement of oxidation and hot corrosion resistance of  $\alpha_2$ -Ti<sub>3</sub>Al/ $\gamma$ -TiAl can be done by applying a protective coating which can produce slow growing oxides such as Al<sub>2</sub>O<sub>3</sub>, Cr<sub>2</sub>O<sub>3</sub>, or SiO<sub>2</sub><sup>5</sup>. Pack aluminizing has long been considered as a method for producing protective coating Al<sub>2</sub>O<sub>3</sub>, but it was reported that this coating can produce crack at TiAl<sub>3</sub> layer which lead to pitting corrosion<sup>6</sup>. Meanwhile, enamel coating with SiO<sub>2</sub> as main composition is attracting researcher to be developed. Xiong et al.<sup>7</sup> have investigated the effect of enamel coating on the hot corrosion behavior of  $\alpha_2$ -Ti<sub>3</sub>Al alloy. The coating was reported to exhibit excellent protection to the alloy. To investigate the effect of enamel coating on hot corrosion resistance of  $\alpha_2$ -

Ti<sub>3</sub>Al/γ-TiAl, we conduct hot corrosion test of bare alloy and enamel coated samples using immersion test method in molten salt of 85%-wt Na<sub>2</sub>SO<sub>4</sub> and 15%-wt NaCl at 850°C up to 50 hours. This paper reports the results of investigation of hot corrosion resistance of α<sub>2</sub>-Ti<sub>3</sub>Al/γ-TiAl alloy with and without enamel coating.

## MATERIALS AND METHODS

To produce samples of α<sub>2</sub>-Ti<sub>3</sub>Al/γ-TiAl alloy, substrate material with composition of Ti-46Al-2Cr-2Mo at% was prepared by melting the pure elements in direct current electric arc furnace followed by homogenization at 1000°C for 10 hours. The alloy samples were obtained by cutting the as homogenized substrate with wire cutting to 8 mm x 8 mm x 2 mm. The substrate samples were then ground in 2000 grit finish and ultrasonically cleaned in alcohol. The weight of each sample was measured using electronic balance with a resolution of 0.01 mg.

Meanwhile, experimental procedure to make enamel coating in this research refers to research of Xiong et al. <sup>7</sup> with a modified spraying technique. Manual air spraying is used instead of controlled air spraying due to equipment limitation. Enamel coating was made from a mixture of raw material with composition as presented in Table 1.

TABLE 1. Chemical composition of enamel coating

SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	ZrO <sub>2</sub>	ZnO	CaO	Na <sub>2</sub> O	TiO <sub>2</sub>	CuO	H <sub>3</sub> BO <sub>3</sub>	Balance
58.26	7.98	5.29	9.00	3.66	5.00	3.54	1.80	5.00	0.47

This enamel mixture was then melted at 1450°C for 10 hours and then quenched in water to obtain enamel frit. The enamel frit was ground for 4 hours to obtain ultrafine enamel frit and then manually air sprayed to the surface of the substrate samples at room temperature and vitrified for 45 minutes at 900°C. After the as enamel coated samples were ultrasonically cleaned in ethanol, they were weighed using an electronic balance and analyzed the surfaces using X-ray diffraction (XRD).

Hot corrosion test was carried out for bare and enamel coated samples in molten salt containing 85% wt. Na<sub>2</sub>SO<sub>4</sub> and 15% wt. NaCl at 850°C for 1, 5, 10, 25, and 50 hours. The samples were immersed completely into the molten salt, moved at regular intervals, air cooled, cleaned in boiling water and blew with hot air. The corroded samples were then weighed for mass change measurement, and analyzed using optical microscope, XRD, and energy dispersive X-ray spectroscopy (EDS) attached on a scanning electron microscopy (SEM).

## RESULTS AND DISCUSSION

Substrate of α<sub>2</sub>-Ti<sub>3</sub>Al/γ-TiAl with composition Ti-46Al-2Cr-2Mo at% was successfully made based on XRD results in surface of bare alloy sample (Fig. 1). In alloy development of titanium aluminide, two phases α<sub>2</sub>-Ti<sub>3</sub>Al/γ-TiAl intermetallic alloy is usually made using general composition of Ti-(45-48)Al-(0.1-10)X at% with X meaning the elements Cr, Nb, V, Ta, Mo, Zr, W, Si, C, and B <sup>8</sup>. This composition resulting up to 20% volume fraction of α<sub>2</sub>-Ti<sub>3</sub>Al phases in the alloy. Element of Cr is added in order to enhance ductility while element of Mo is added to improve creep resistance of the alloy <sup>1</sup>.

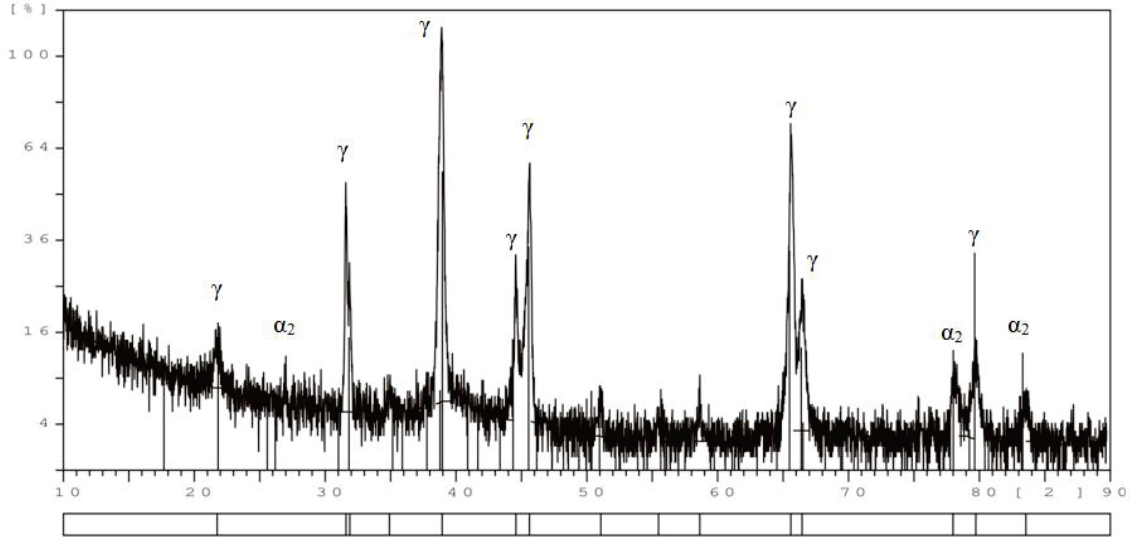


FIGURE 1. XRD spectra of substrate

Oxidation and hot corrosion resistance become important factors for the use of two phases  $\alpha_2$ -Ti<sub>3</sub>Al/ $\gamma$ -TiAl intermetallic alloy at elevated temperatures. This is due to formation of non-protective TiO<sub>2</sub> + Al<sub>2</sub>O<sub>3</sub> porous scale rather than compact of protective Al<sub>2</sub>O<sub>3</sub> layer<sup>6</sup>. Inability to form this protective oxide causes the alloy suffer degradation due to hot corrosion. Figure 3 shows that the alloy already has pitting corrosion after 1 hour of hot corrosion test. Due to non-protective TiO<sub>2</sub> + Al<sub>2</sub>O<sub>3</sub> porous scale on the surface of the alloy, migration of Cl<sup>-</sup> ions into the substrate occurs and lead to pitting corrosion. It can be seen that after 50 hours of hot corrosion test (Fig. 2 (b)) uncoated substrates suffered from hot corrosion and showed significant damage of pitting corrosion with 718.71  $\mu$ m depth of corrosion.

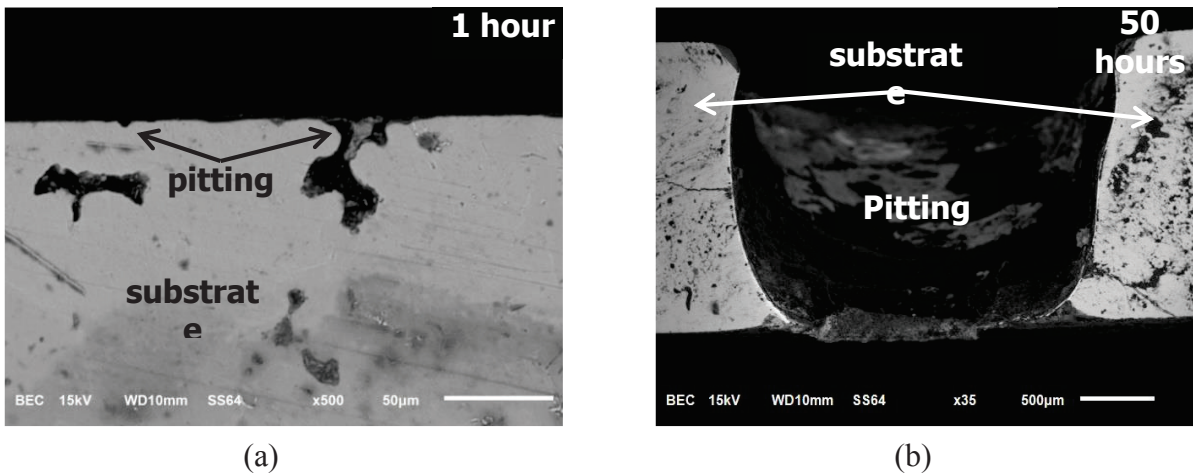
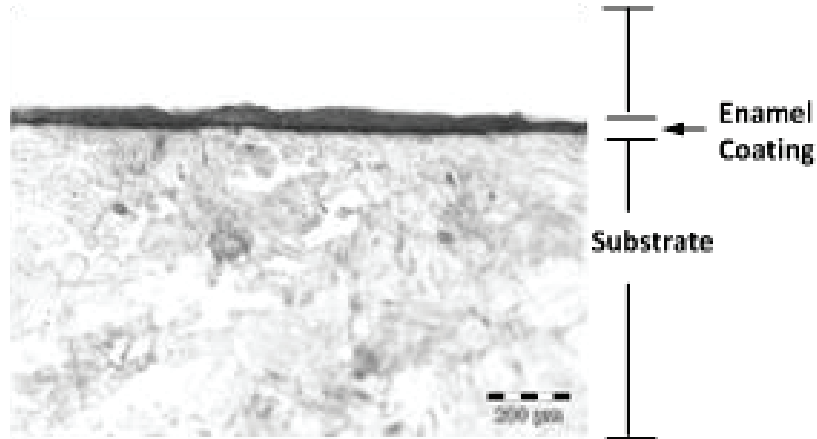


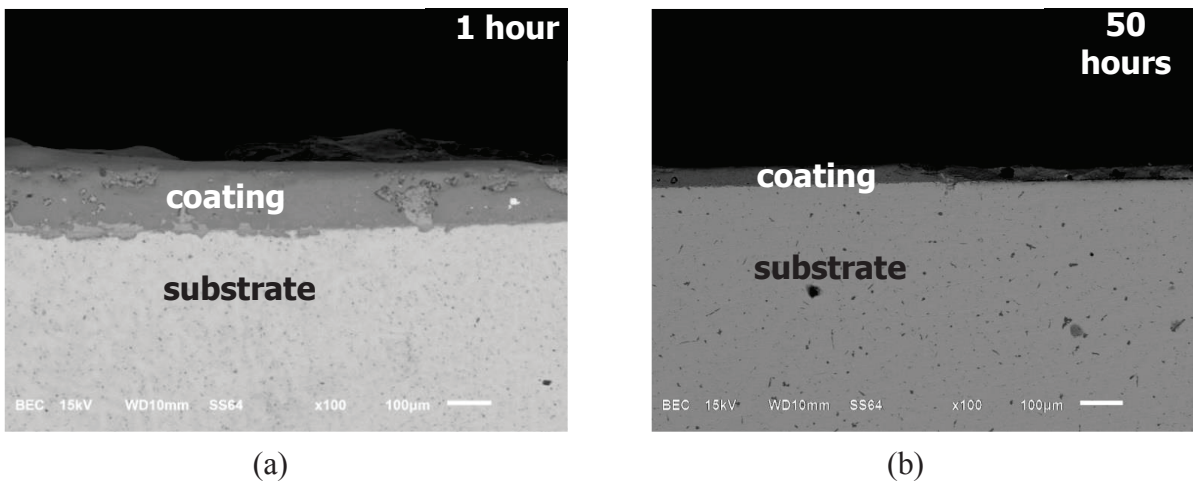
FIGURE 2. The cross-section microstructure of bare alloy (a) after 1 hour of hot corrosion test, and (b) after 50 hours of hot corrosion test

In order to improve hot corrosion resistance of this alloy, enamel coating is promising candidate with SiO<sub>2</sub> as the main constituent of coating which has low solubility in molten salt. It is also reported that enamel coating has good compatibility with titanium aluminide substrate<sup>7</sup>. In this experiment, enamel coating adheres well to  $\alpha_2$ -Ti<sub>3</sub>Al/ $\gamma$ -TiAl substrate which indicates good compatibility between coating and substrate as shown in cross-sectional sample of enamel coated sample in Fig. 3. However, thickness of coating is not uniform due to manual air spraying.



**FIGURE 3.** The cross-section microstructure of the samples coated with enamel coating

After 50 hours of hot corrosion test, enamel coated samples were relatively undamaged and still adheres well to the substrate as shown in Fig. 4. In addition, there were no spallation observed and no corrosion penetration up to the substrate was observed, indicated that enamel coating have a good ability to withstand the hot corrosion attack.



**FIGURE 4.** The cross-section microstructure of sample with enamel coating (a) after 1 hour of hot corrosion test, and (b) after 50 hours of hot corrosion test

Furthermore, enamel coating has excellent stability in molten salt which provide good protection to the substrate from hot corrosion. This is supported by result of an experiment in Fig. 5 which shows the corrosion rate of specimens after hot corrosion test. After 50 hours of corrosion test, it is clearly seen that significant mass change occurred in uncoated samples with  $-69.05 \text{ mg/cm}^2$  comparing with  $0.75 \text{ mg/cm}^2$  in enamel coated sample. Addition of enamel coating gave reduction of mass change at 98.20%.

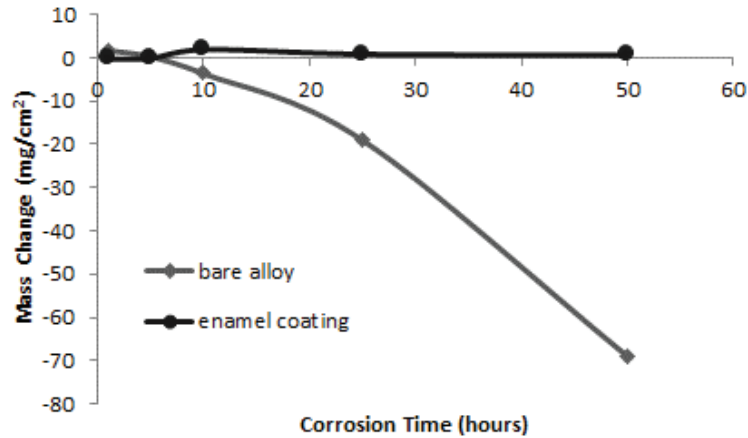


FIGURE 5. Corrosion kinetics of samples in Na<sub>2</sub>SO<sub>4</sub>+NaCl melts at 850°C

The ability of the enamel coating to increase hot corrosion resistance to the substrate is basically due to relatively high content of SiO<sub>2</sub> as the main constituent of enamel coating. Ishitsuka and Nose<sup>9</sup> (cited in Xie and Wang<sup>10</sup>) states that SiO<sub>2</sub> has low solubility in molten salt. They investigated the solubility of SiO<sub>2</sub> in molten salt and found that the dissolution mechanism of SiO<sub>2</sub> is incompatible with acid or alkaline leaching mechanism, but only through physical dissolution. Nevertheless, the solubility of SiO<sub>2</sub> in the enamel coating could increase as the microstructure of the enamel coating is difference with that of pure SiO<sub>2</sub> due to the influence of Ca<sup>2+</sup> and Na<sup>+</sup><sup>7</sup>.

With low solubility in molten salt, SiO<sub>2</sub> which is the main constituent of enamel coating can act as a barrier between substrate and molten salt. The main microstructure of the enamel coating is a glassy structure which resist from penetration of molten salt ions into the coating<sup>10</sup>. However, as the enamel coating consists of a mixture of several oxides having different solubility in molten salt, the dissolution of coating constituents into molten salt is predicted to occur at the end of hot corrosion test. The EDS results in Table 2 confirmed the decreasing of ZrO<sub>2</sub>, ZnO, CaO, TiO<sub>2</sub>, CuO, H<sub>3</sub>BO<sub>3</sub> compounds in the enamel coating with ZnO as the most decreasing compound from enamel coating constituents. This indicates dissolution of several enamel coating oxides in molten salt during hot corrosion and lead to the faster migration of Cl<sup>-</sup> ions into the coating through voids and reacts with oxides in the enamel coating<sup>10</sup>. Xie and Wang<sup>10</sup> reported that Cl<sup>-</sup> ions can react with oxide coating constituent to form chloride on the outer surface of coating and dissolved in molten salt. This phenomenon is supported with EDS result that confirms the present of Cl<sup>-</sup> ions in enamel coating. The Cl<sup>-</sup> ions can diffuse into the coating and reach the substrate-coating interface that makes the substrate corroded. The corrosion products essentially consist of Ti or Al ions that diffuse further to the substrate surface.

TABLE 2. EDS result of enamel coating after 50 hours of hot corrosion test

Enamel Composition	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	ZrO <sub>2</sub>	ZnO	CaO	Na <sub>2</sub> O	TiO <sub>2</sub>	CuO	H <sub>3</sub> BO <sub>3</sub>	Cl <sup>-</sup>	Bal.
Before Test (%)	58.26	7.98	5.29	9.00	3.66	5.00	3.54	1.80	5.00	0.00	0.47
After Test (%)	64.16	11.90	4.26	1.72	0.11	16.12	1.43	0.00	0.00	0.22	0.08
Difference (%)	5.90	3.92	-1.03	-7.28	-3.55	11.12	-2.11	-1.80	-5.00	0.22	-0.39

Further study needs to be conducted to find out the mechanism of the reaction at the interface between substrate of α<sub>2</sub>-Ti<sub>3</sub>Al/γ-TiAl alloy with enamel coating. Tang et al.<sup>5</sup> reported that, based on the thermodynamic aspects, Al<sub>2</sub>O<sub>3</sub> can be formed on the substrate-coating interface layer due to the reaction of Al with ZnO. The formation of a protective oxide Al<sub>2</sub>O<sub>3</sub> requires further study on the effect of adhesion properties of the enamel coating and substrate. In addition, the alloy surface roughness of samples affects the mechanical interlocking between substrates and the enamel coatings.

## CONCLUSION

Enamel coating on  $\alpha_2$ -Ti<sub>3</sub>Al/ $\gamma$ -TiAl alloy can be used for improving hot corrosion resistance of alloy. This is because relatively high content of SiO<sub>2</sub> as the main constituent of enamel coating which has low solubility in molten salt. At the end of hot corrosion test, there are no corrosion depth and spallation observed on the substrate with enamel coating. However, decreasing content of several oxides will lead to enamel coating degradation.

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