Improvement in the oxidation resistance of α_2 -Ti₃Al by sputtering Al film and subsequent interdiffusion treatment

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Abstract

Sputtering Al film on α_2 -Ti₃Al alloy and subsequent interdiffusion treatment at 600 °C for 24 h in high vacuum can effectively improve its oxidation resistance, due to a good adhesive TiAl₃ layer formed on the surface. The thicker the Al film, the thicker the TiAl₃ layer that can be formed. Cyclic and isothermal oxidation tests at 800 °C in air show that the α_2 -Ti₃Al with 3–5 μ m Al film can dramatically reduce its oxidation rate, and the parabolic oxidation rate constant K_p of specimen with 5 μ m Al film is only approximately 1/5000 of that of bare α_2 -Ti₃Al. The X-ray diffractometer and scanning electron microscopic results indicate that the TiAl₃ layer has formed a protective and continuous α -Al₂O₃ scale on the outer surface, and inter-reacted with α_2 -Ti₃Al to form γ -TiAl phase at 800 °C after 80 h of oxidation, i.e. there are layers of α -Al₂O₃/ γ -TiAl/ α_2 -Ti₃Al formed on specimens.

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1. Introduction

Titanium aluminides are being considered as promising structural materials for high temperature applications in aircraft and automotive industries, because of their high strength and low density at elevated temperature [1–5]. The major concern for structural applications of Ti₃Al-based alloys is the insufficient oxidation resistance at high temperature and the lack of ductility at room temperature. It was demonstrated for these alloys that, the room temperature ductility could be effectively improved by the addition of β -phase stabilized elements, such as Nb, Mo and V [3,6–8]. Two main approaches, viz., alloying design and surface modification have been used to improve the oxidation resistance at high temperature [6–17]. However, many reported studies indicate that, the improvement in oxidation resistance for Ti₃Al-based alloys favors the methods of surface modification rather than those of alloying design, due to the addition of alloying elements can induce the degradation of ductility [10–17].

It has been reported that the high temperature oxidation resistance limit of α_2 -Ti₃Al alloys is 650 °C [3], because it could not form a protective Al₂O₃ layer on the surface at high temperatures, instead, a mixed TiO₂/Al₂O₃ oxide scale [19–21] is formed. TiAl₃, another intermetallic phase formed in the Ti–Al binary phase diagram, is known as an alumina former with good oxidation resistance at high temperature due to its higher aluminum content [22,23]. Therefore, a layer of TiAl₃ coating deposited on Ti₃Al-based alloys should improve their high temperature oxidation resistance. Many methods such as pack cementation aluminizing [13–16], electro-spark deposition [17] and aluminum cladding [18] have been investigated regarding the formation of TiAl₃ layer on γ -TiAl as well as Ti₃Al-based alloys. However, the poor adhesion of TiAl₃ coating to the substrate resulted from the inherent microcracks and the intrinsic brittleness of the coatings prevents the improvement of oxidation resistance significantly. To overcome these drawbacks, use of the magnetron sputtering deposition technology seems to be a feasible method, which has reported to offer very good mechanical properties of TiAl₃ coatings on γ -TiAl alloy [12,25].

In this study, we aim to modify the surface of α_2 -

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Table 1
Sputtering conditions used in this study

System base pressure (Torr)	Sputtering pressure (Torr)	RF power (W)	Sputtering time (min)	Distance between target and substrate (mm)	Al film thickness (μm)
$<6 \times 10^{-7}$	5×10^{-3}	100	24–240	80	0.5–5

Ti_3Al alloy by sputtering various thicknesses of pure Al films with a subsequent interdiffusion treatment in high vacuum. Thereafter, a TiAl_3 layer is formed on the surface with rather good adhesion to the $\alpha_2\text{-Ti}_3\text{Al}$ substrate. Specimens treated by the aforementioned processes are cyclically oxidized at 800 °C in static air and their weight gains are measured. Based on the measured data and the microstructural observations, the oxidation resistance improvement of $\alpha_2\text{-Ti}_3\text{Al}$ alloy is discussed.

2. Experimental procedures

The ingots of $\alpha_2\text{-Ti}_3\text{Al}$ alloy used in this study were prepared from the raw materials of titanium (99.7%) and aluminum (99.98%) by a vacuum arc remelter under high-purity argon atmosphere. The ingots were re-melted at least six times to ensure the homogeneity of the as-cast structure. After homogenization of the ingots at 1000 °C for 100 h in vacuum, specimens for sputtering were cut by a diamond-saw into the size of $10 \times 10 \times 1 \text{ mm}^3$. Before the sputtering, all surfaces of the specimens were mechanically polished by a standard metallographic procedure to a final polishing of 0.3- μm alumina, then ultrasonically cleaned with acetone and ethanol, and then blown dry. The pure aluminum target for sputtering was also prepared by the same procedure, but the Al ingot was finally sectioned into a disk shape of 50 mm in diameter and 5 mm in thickness.

The Al film was deposited on all the surfaces of the $\alpha_2\text{-Ti}_3\text{Al}$ specimens using an r.f. magnetron sputtering apparatus. The sputtering conditions are given in Table 1. Aluminum films in the thickness range of 0.5–5 μm were deposited, as measured by α -step (Veeco Dektak³ ST). After the sputtering deposition, a subsequent interdiffusion treatment was processed at 600 °C for 24 h in a high vacuum of approximately 3×10^{-7} torr. Isothermal oxidation tests at 800 °C in static air were performed using a thermogravimetric analysis (TGA, TA Module TGA-DTA 1500). Cyclic oxidation tests were conducted to evaluate the oxidation resistance of $\alpha_2\text{-Ti}_3\text{Al}$. The specimens were exposed in a muffle furnace at 800 °C and regularly removed from the furnace at time intervals of 5–10 h, air cooled, weighed and returned to the 800 °C furnace. The weight gains were measured on an analytical balance within an accuracy of $\pm 0.001 \text{ mg}$ (Model: Mettler MT5).

An X-ray diffractometer (XRD, Philips PW1729) with Cu $K\alpha$ radiation at 30 kV, 20 mA and 4° 2 θ /min scanning rate was used to identify the phases of sputtered-films formed by the interdiffusion treatment and the oxidation tests. The surface morphologies and cross-sectional microstructures of specimens were observed by a Leo 1530 scanning electron microscope (SEM) equipped with energy dispersive spectrometry (EDS). Electron probe microanalyzer (EPMA, JEOL JXA-8600 SX) was used for measuring the chemical composition

Fig. 1. The SEM morphologies of $\alpha_2\text{-Ti}_3\text{Al}$ specimen with 3 μm Al film after interdiffusion treatment at 600 °C for 24 h in high vacuum. (a) Surface morphology and (b) cross-sectional morphology.

of phases formed by sputtering, interdiffusion treatment and oxidation tests.

3. Results and discussion

3.1. Interdiffusion of α_2 -Ti₃Al with sputtered Al films

Aluminum films with different thicknesses of 0.5–5 μm were sputtered on the α_2 -Ti₃Al substrate. To control the thickness of Al film accurately, the sputtering rate of Al film should be determined. According to the sputtering conditions of Table 1 and the thickness measurement of α -step, the sputtering rate of Al film is calculated to be 3.5 $\text{\AA}/\text{s}$ [25]. Consequently, the appropriate thickness of Al film can be obtained by controlling the sputtering time. However, the intrinsic stress in the film increases with increasing film thickness, resulting in poor adhesion of coating to the substrate. Once the thickness of Al film is over 5 μm , small parts of the film spall out from the α_2 -Ti₃Al substrate after the interdiffusion treatment. Hence, in this study the thicknesses of the as-sputtered Al films are all less than 5 μm . The as-sputtered Al film is crystalline with a partially amorphous structure in terms of the result of XRD pattern [25].

In order to form a TiAl₃ layer on the α_2 -Ti₃Al surface and to increase the adhesion between the thin film and

α_2 -Ti₃Al substrate, an interdiffusion treatment is conducted for each specimen sputtered with Al film. Fig. 1a and b show the SEM images of top-viewed and cross-sectional morphologies, respectively, of α_2 -Ti₃Al with a 3 μm thick as-sputtered Al film via interdiffusion treatment at 600 $^{\circ}\text{C}$ for 24 h in vacuum. From Fig. 1a, the microstructure was found to be relatively dense, with extremely small grain and no visible crack. The XRD diffraction patterns of the surface indicate that the main product of Fig. 1b is TiAl₃ phase, as shown in Fig. 2. From Fig. 1b, the TiAl₃ layer formed on the outer surface has a uniform thickness with no crack or void introduced at the interface. Thus, the aforementioned interdiffusion procedure is beneficial to the bonding between thin film and substrate. From the results of EPMA and EDS analyses, this layer has a Al:Ti at.% ratio of 3:1, which again confirms that the diffusion layer consists of TiAl₃ phase. The present observation coincides with the XRD patterns shown in Fig. 2.

The first phase formation in the diffusion system of α_2 -Ti₃Al and Al thin film is the TiAl₃ layer, according to the solid-state diffusion theory [26,27] and an effective heat of formation model [28]. This is due to the formation of TiAl₃ intermetallic, which has more negative formation energy than the other titanium aluminides. The formation of TiAl₃ phase finishes until all the sputtered Al atoms are exhausted during the interdiffu-

Fig. 2. The XRD spectra of α_2 -Ti₃Al specimens with various thicknesses of Al films (from 0.5 to 5 μm) after interdiffusion treatment at 600 $^{\circ}\text{C}$ for 24 h in high vacuum.

Fig. 3. Cyclic oxidation tests in static air at 800 °C for α_2 -Ti₃Al specimens with various thicknesses of Al films which have been interdiffusion treated at 600 °C for 24 h in high vacuum.

sion treatment. This means that the thickness of TiAl₃ layer formed by interdiffusion treatment is highly dependent on the thickness of as-sputtered Al film. It was found that, for a 3- μ m thick Al film, the thickness of TiAl₃ layer can be extended to approximately 4 μ m by the interdiffusion of α_2 -Ti₃Al and Al film, as shown in Fig. 1b.

3.2. Cyclic and isothermal oxidation

The cyclic oxidation tests were evaluated at 800 °C in static air for 80 h. The weight gain Δw of the

oxidation was measured from the weight change of tested specimens, including the remaining and exfoliated scales. Fig. 3 compares the weight gain of sputtered specimens with that of the unsputtered (bare) one. All specimens with sputtered Al film were subjected to the interdiffusion treatment at 600 °C for 24 h in vacuum. Fig. 3 indicates that the thickness of Al film is a major factor for the improvement of oxidation resistance of α_2 -Ti₃Al alloy. For bare α_2 -Ti₃Al alloy which has the highest oxidation rate, severe weight gain and scale spallation can be observed clearly within 80 h of

Fig. 4. Effect of the thickness of Al film on the cyclic oxidation resistance (parabolic rate constant K_p) of α_2 -Ti₃Al specimens oxidized at 800 °C in air. The K_p values are calculated from Fig. 3.

Fig. 5. The TGA isothermal oxidation tests in static air at 800 °C for α_2 -Ti₃Al specimens with various thicknesses of Al films.

exposure in static air at 800 °C. Obviously, α_2 -Ti₃Al specimens with Al films of certain thickness in the 3–5 μm range have excellent oxidation resistance at 800 °C. This comes from the fact that they have a TiAl₃ layer with sufficient thickness, which can form an adhesive and continuous α -Al₂O₃ layer on the surface during the cyclic oxidation tests. At the same time, no

scale spallation occurs during the cyclic oxidation for specimens with 3–5 μm Al film, even at the corners or along the edges of the specimens.

The parabolic oxidation rate constant, K_p , which was calculated from the relation $(\Delta w/A)^2 = K_p t$ can be estimated from the curve fitting of Fig. 3, and this constant can be used as a convenient index for comparing the

Fig. 6. The XRD diffraction spectra of Al-sputtered α_2 -Ti₃Al specimens after interdiffusion treatment and then oxidation at 800 °C for 80 h in air. The thickness of Al film ranges from 0 μm (bare α_2 -Ti₃Al) to 5 μm .

improvement of oxidation resistance. Here, A is the surface area of tested specimens and t is the time. Fig. 4 shows the relation between calculated K_p value and the thickness of Al films. The K_p value for the cyclic oxidation of 3- μm thick Al film at 800 °C is $1.15\text{E}-3 \text{ mg}^2/\text{cm}^4 \text{ h}$, in comparison with $2.294 \text{ mg}^2/\text{cm}^4 \text{ h}$ for the bare $\alpha_2\text{-Ti}_3\text{Al}$. The K_p value for the former is approximately 1/2000 of the latter. Furthermore, the K_p value for the cyclic oxidation of 5- μm thick Al film at 800 °C can be further reduced to $4.28\text{E}-4 \text{ mg}^2/\text{cm}^4 \text{ h}$, which is approximately 1/5000 of the K_p value for the bare $\alpha_2\text{-Ti}_3\text{Al}$. In other words, the K_p value is found to decrease upon increasing the TiAl_3 layer thickness, indicating that the oxidation resistance of $\alpha_2\text{-Ti}_3\text{Al}$ alloy with 3–5 μm thick Al films can be improved dramatically, to the extend of above three orders of magnitude, as compared to the bare $\alpha_2\text{-Ti}_3\text{Al}$ alloy. Figs. 3 and 4 indicate that, in the thickness range of 3–5 μm Al film, the improvement in the oxidation resistance of $\alpha_2\text{-Ti}_3\text{Al}$ is mainly dependent on the TiAl_3 thickness. The thicker the TiAl_3 layer, the higher the oxidation resistance obtained, since the thicker TiAl_3 layer can form a thicker protective and continuous $\alpha\text{-Al}_2\text{O}_3$ layer on the substrate for long-term oxidation at 800 °C.

Isothermal oxidation tests were carried out by TGA at 800 °C up to 70 h to compare the oxidation properties of $\alpha_2\text{-Ti}_3\text{Al}$ with and without Al film, as shown in Fig. 5. From Fig. 5, the sputtered Al film and subsequent interdiffusion treatment can substantially reduce the oxidation rate of $\alpha_2\text{-Ti}_3\text{Al}$. This result is in agreement with the cyclic oxidation results as shown in Fig. 3.

3.3. Surface and cross-sectional morphologies of specimens after 800 °C cyclic oxidation

Fig. 6 shows the XRD spectra of $\alpha_2\text{-Ti}_3\text{Al}$ specimens with and without Al film, which have been interdiffusion-treated at 600 °C for 24 h and then cyclically oxidized at 800 °C in air. The XRD pattern of bare Ti_3Al specimen shown in Fig. 6 has strong peaks corresponding to TiO_2 (rutile) with a few weak $\alpha\text{-Al}_2\text{O}_3$ peaks, indicating that the oxides formed consist of a mixture of Al_2O_3 and TiO_2 . Also from Fig. 6, for specimens with 3–5 μm Al film, the $\alpha\text{-Al}_2\text{O}_3$ layer is formed exclusively on the outer surface. At the same time, TiAl_3 has reacted with $\alpha_2\text{-Ti}_3\text{Al}$ to form the $\gamma\text{-TiAl}$ phase after oxidation at 800 °C for 80-h oxidation [24], as the XRD spectra of $\gamma\text{-TiAl}$ shown in Fig. 6. The results of Figs. 3 and 6 indicate that, when the thickness of Al films is over 3 μm , a protective $\alpha\text{-Al}_2\text{O}_3$ scale, instead of a fragile TiO_2 one, is formed on the outer surface of the specimen.

Fig. 7 shows the SEM surface morphologies (top-view) of the oxidation scale after oxidation at 800 °C for 80 h for: (a) bare $\alpha_2\text{-Ti}_3\text{Al}$, (b) 3 μm Al film and (c) 5 μm Al film. The sputtered specimens in Fig. 7b

Fig. 7. The SEM surface morphologies of the oxidation scale after oxidation at 800 °C for 80 h for (a) $\alpha_2\text{-Ti}_3\text{Al}$; (b) 3 μm Al film and (c) 5- μm Al film. The specimens were interdiffusion treated in high vacuum before the oxidation.

and c were interdiffusion-treated at 600 °C for 24 h in high vacuum before the cyclic oxidation. Fig. 7a reveals that, for the bare $\alpha_2\text{-Ti}_3\text{Al}$ specimen, only large quanti-

Fig. 8. The SEM cross-sectional images of the specimens of Fig. 7 for (a) α_2 -Ti₃Al; (b) 3 μ m Al film; (c) 5 μ m Al film; and (d) EDS composition analyses of points 1–5 shown in (c).

ties of crystalline TiO₂ oxide were formed on the surface, which is consistent with the reported investigations [19–21,29]. In contrast, for the 3- μ m Al film specimen, a dense and stable α -Al₂O₃ layer without brittle rutile has formed on the oxide scale, as shown in Fig. 7b. Additionally, a very small amount of needle-like α -Al₂O₃ is also found at some areas. In general, the needle-like α -Al₂O₃ is formed at the early oxidation stage and/or at relatively low temperatures. After a long exposure at higher temperatures, a dense α -Al₂O₃ transited from the cluster of needle-like α -Al₂O₃ could be observed [17]. This α -Al₂O₃ layer exhibits a good barrier against inward oxygen diffusion, resulting in an improved oxidation resistance of α_2 -Ti₃Al at 800 °C in air. In the case of 5 μ m Al film specimen, the needle-like α -Al₂O₃ increases in number on the surface, as shown in Fig. 7c.

The SEM cross-sectional microstructures of the same specimens of Fig. 7 are shown in Fig. 8. Fig. 8a reveals that the microstructure of the oxide layer of α_2 -Ti₃Al is similar to that of γ -TiAl, but there is no continuous Al₂O₃ barrier, and a discontinuous Al₂O₃-rich sublayer exists above the TiO₂/Al₂O₃ mixture [19]. From Fig. 8b and c, the microstructure of specimens with 3 or 5 μ m Al film is not complicated: only two layers coexist, as indicated by XRD results of Fig. 6 and EDS com-

position analyses of points 1–5 shown in Fig. 8d. The XRD and EDS results show that the outer thin layer is due to α -Al₂O₃, the inner thick layer is due to γ -TiAl and the substrate is due to α_2 -Ti₃Al. An adhered α -Al₂O₃ scale formed on the surface of γ -TiAl layer can provide excellent protection against the oxidation attack. From Fig. 8b and c, one can find that the thicker the Al film, the thicker the α -Al₂O₃ layer on the outer surface. As to the γ -TiAl formation, it appears to be reacted from TiAl₃ and α_2 -Ti₃Al, as confirmed by the XRD spectra shown in Fig. 6. This phenomenon can be understood from the phase formation sequence of Ti₃Al–Al thin film system, as further discussed in another paper [24]. The thickness of γ -TiAl layer increases upon increasing the Al film thickness, as shown in Fig. 8b and c. In this study, we find that the alumina scale can maintain its integrity with no microcrack and spallation after 80-h cyclic oxidation.

4. Conclusions

The oxidation resistance of α_2 -Ti₃Al alloy can be improved by sputtering Al film followed by the inter-diffusion treatment at 600 °C for 24 h in high vacuum. The Al film has its thickness accurately controlled by magnetron sputtering process, but this thickness cannot

exceed 5 μm because the intrinsic stress in the film can spall it out from the substrate via the interdiffusion treatment. After the interdiffusion treatment, the Al-sputtered $\alpha_2\text{-Ti}_3\text{Al}$ alloy forms a TiAl_3 layer on the surface due to the first phase formation of the interaction between $\alpha_2\text{-Ti}_3\text{Al}$ and Al thin film. This TiAl_3 layer exhibits good adhesion with the substrate and plays an important role in the long-term oxidation resistance. Experimental results show that the thickness of TiAl_3 layer is strongly dependent on Al film and the thicker the Al film, the thicker the TiAl_3 layer that can be formed. Cyclic and isothermal oxidation tests at 800 °C in air indicate that the $\alpha_2\text{-Ti}_3\text{Al}$ alloy with TiAl_3 layer on the surface can dramatically reduce the oxidation rate if the sputtering thickness of Al film can reach 3–5 μm . The parabolic rate constant K_p of cyclic-oxidized specimens with 3 and 5- μm Al film thickness can be reduced to approximately 1/2000 and 1/5000, respectively, as compared to that of specimens without sputtering Al film. From the XRD and SEM+EDS results of cyclic-oxidized specimens at 800 °C, the TiAl_3 layer not only result in the formation of a continuous $\alpha\text{-Al}_2\text{O}_3$ scale on the outer surface, but also inter-react with $\alpha_2\text{-Ti}_3\text{Al}$ substrate to form $\gamma\text{-TiAl}$ layer during the oxidation. After the cyclic oxidation at 800 °C for 80 h in air, the cross-sectional microstructures of specimens with Al film show that there are layers of $\alpha\text{-Al}_2\text{O}_3/\gamma\text{-TiAl}/\alpha_2\text{-Ti}_3\text{Al}$, in which the $\alpha\text{-Al}_2\text{O}_3$ layer maintains its integrity with no microcracks and spallation.

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