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A novel approach to accelerate the reaction between Ti and Al

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ABSTRACT

Pure Ti foils and SiCp/Al composite foils were employed to investigate the parabolic growth kinetics of $TiAl_3$ at 660 °C. Compared with pure Al foils, the introduction of SiC particles significantly refined $TiAl_3$ grain size by the solid solution of silicon. Corresponding refinement mechanisms were concluded from the perspective of the nucleation of $TiAl_3$. Micromechanics analysis shows that the fine $TiAl_3$ grains own a small viscous resistance, and subsequently an improvement in the reaction rate could be achieved. This meaningful law also applies extensively to Ni/Al and Fe/Al systems.

1. Introduction

Gamma titanium aluminide (γ-TiAl) sheets have attracted extensive attention in recent years as promising candidates for a variety of structural applications in the aerospace, automotive and turbine power generation markets (Rester et al., 2011; Zghal et al., 2005). So far, most feasible production methods are commonly based on the hot rolling of TiAl ingots, but its limited ductility and poor hot workability lead to unacceptable production costs (Cui et al., 2012a,b). In order to avoid the direct deformation of brittle TiAl, an innovative method was proposed by employing Ti and Al foils to synthesize TiAl sheets via reaction annealing (Xu et al., 2006; Yang et al., 2010; Zhang, 2010). This method is particularly useful in the preparation of brittle materials. However, it poses the following two challenges: (i) numerous void formation due to the Kirkendall effect, which could be duly eliminated by modifying the annealing parameters (Cui et al., 2012a,b; He et al., 2007); (ii) unable to meet stringent demand of production due to the long reaction time required during annealing at ~660 °C to convert all of the elemental Al into $TiAl_3$ (Cui et al., 2012a,b; Xu et al., 2006). In order to solve this problem, we draw on the lessons from experience in aluminum and magnesium alloys that adding alloying elements was considered to be effective in refining grain size and increasing the nucleation rate (Kashyap and Chandrashekar, 2001; Marioara et al., 2006; Pelleg et al., 2000).

In the present work, pure Ti foils and SiCp/Al composite foils were used to study the growth kinetics of $TiAl_3$. SiC particles were selected because (i) it serves as a silicon carrier for refining $TiAl_3$ grain size; (ii) fabricating TiAl matrix composites with a perfect reinforcement-matrix interface can be attained by an in situ process (Wu et al., 2013). Refinement mechanism of $TiAl_3$ grains, from the perspective of nucleation, was also studied. For a given temperature at 660 °C (the melting point of pure Al), both growth kinetics of the $TiAl_3$ layers and micromechanics of the $TiAl_3$ grain have been investigated.

2. Experimental procedures

Elemental Ti foils (99.6% purity, 100 μm thick) and 3 vol.% SiCp/Al composite foils (99.6% purity, 92 μm thick, 40 nm SiC as reinforcement) were employed. The Ti and SiCp/Al foils were etched in 10 vol.% HF and 10 wt.% NaOH solutions, respectively, water flushed, alcohol cleaned, dried and cut into samples with size of 50 mm × 50 mm. Alternating layers of Ti and SiCp/Al foils were stacked with outer layers of Ti foils. Hot pressing was conducted at 515 °C for 1.5 h under 40 MPa in vacuum of $\sim 10^{-3}$ Pa. The hot-pressed laminates were cut into smaller samples with size of 10 mm × 10 mm for subsequent reaction annealing. Annealing experiments were carried out at 660 °C with diffusion time varying from 5 min to 3 h.

The samples after annealing were examined by a transmission electron microscope (TEM, FEI Tecnai F30) and a scanning electron microscope (SEM, FEI Quanta 200F) equipped with an

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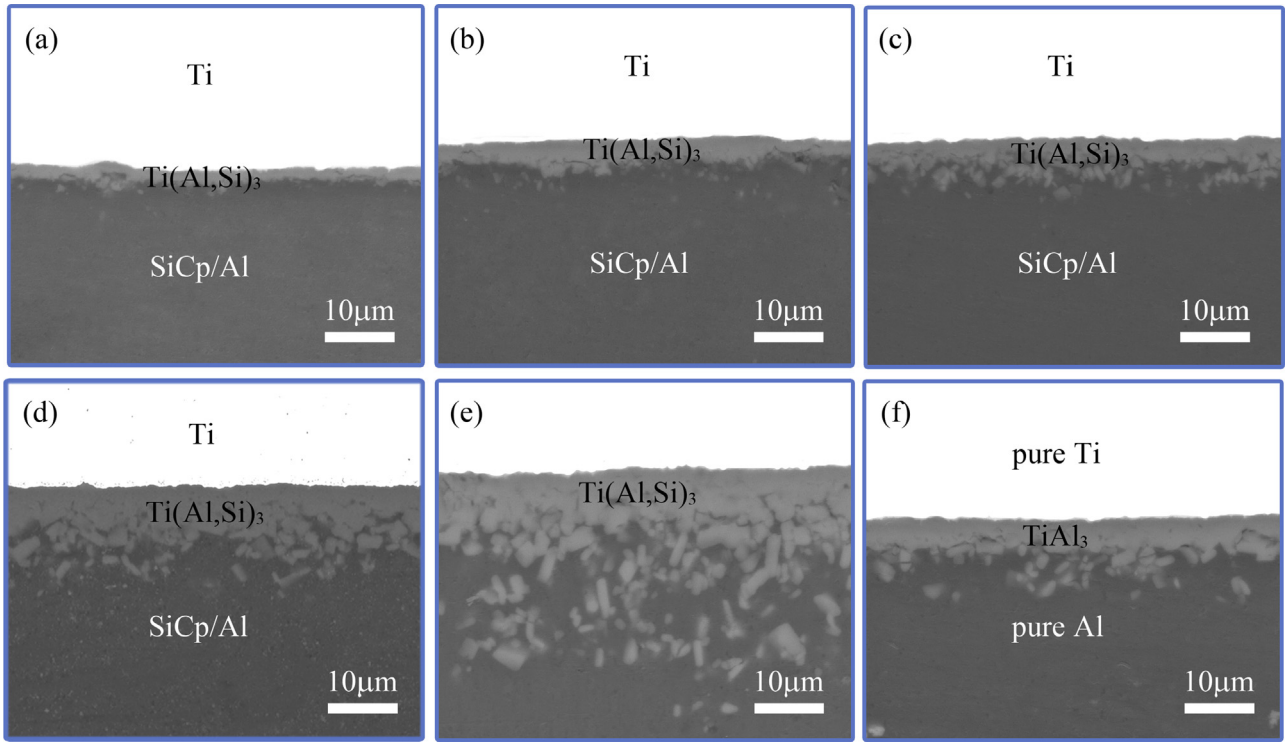

Fig. 1. Interfacial features: (a)–(e) multi-layered Ti–(SiCp/Al) composite sheets annealed at 660 °C for 5 min, 15 min, 30 min, 1 h and 3 h, respectively; (f) multi-layered Ti–Al sheets annealed at 660 °C for 1 h.

energy dispersive X-ray spectrometer (EDX). All examinations were performed on the cross-sections parallel to the direction of hot pressing.

3. Results and discussions

3.1. Microstructure characterization

Typical backscattered electron (BSE) micrographs of multi-layered Ti-(SiCp/Al) composite sheets annealed at 660 °C for different times are shown in Fig. 1a–e. The resulting microstructure consists of three phases, namely, Ti (gray white region), the reaction $TiAl_3$ intermetallic compound (dark gray region) and SiCp/Al (dark region). A comparison experiment in Fig. 1f was also performed by employing pure Ti and Al foils. The thickness of the $TiAl_3$ layers increased from 11 μm (Fig. 1f) to 15 μm (Fig. 1d) by adding SiC. EDX results in Fig. 2b show that there existed diffusion of Si element into $TiAl_3$ layers, and rod-shaped Al_4C_3 phase was also detected in the Al matrix (Fig. 2a), which indicated the decomposition of SiC particles. This could be attributed to the exothermic reaction ($Ti + Al \rightarrow TiAl_3$) during the annealing. Therefore, the reactions that took place are as follows:

$$Ti + Al \rightarrow TiAl_3 \quad (1)$$

$$SiC + Al \rightarrow Al_4C_3 + [Si] \quad (2)$$

$$TiAl_3 + [Si] \rightarrow Ti(Al, Si)_3 + [Al] \quad (3)$$

3.2. Growth kinetics of the $TiAl_3$ layer

The average thickness values of the $TiAl_3$ layers after annealing at different times are collected in Fig. 3b. As for a given temperature (at 660 °C), the thickness of an interdiffusion layer on diffusion time can be described by the following empirical relationship (Wagner, 1969):

$$\Delta x = kt^n \quad (4)$$

$$\ln \Delta x = n \ln t + \ln k \quad (5)$$

where Δx is the thickness of the reaction layer (m), k is the rate constant ($m\,s^{-1/n}$), t is the diffusion time (s), and n is the kinetic exponent. Linear regression analysis in Fig. 3a gives a best-fit straight line, with the kinetic exponent value of 0.54 for 660 °C ($n = 0.5$ for growth controlled by parabolic growth kinetics; $n = 1$ for growth dominated by linear growth). Thus the growth of the $TiAl_3$ layers displays a standard diffusion-controlled parabolic growth behavior. The relation between the thickness of $TiAl_3$ layer and annealing time can be characterized by the following equation:

$$\Delta x = 2.3 \times 10^{-7} t^{1/2} \quad (6)$$

The predicted thickness of the $TiAl_3$ layer in Fig. 3b is consistent with the experimental values, which indicates that Eq. (6) is adaptable and reasonable for describing the growth behavior of the $TiAl_3$ layer at 660 °C.

A summary of the reported investigations for interdiffusion in the Ti/Al couples is presented in Table 1. The kinetic exponent value, n, of $Ti(Al,Si)_3$ is 10% higher than that of $TiAl_3$. As is generally acknowledged (Cui et al., 2012a,b; Xu et al., 2006), the reaction rate decreases progressively with increasing annealing time, due to hindrance for further diffusion caused by the newly-formed $TiAl_3$ layer. Therefore, this slight improvement in the kinetic exponent, caused by the solid solution of silicon, is also meaningful.

3.3. Morphology and formation mechanism of $TiAl_3$

The morphology of $TiAl_3$ in Fig. 1 was categorized into two distinct classes: (i) $TiAl_3$-rich layers at the interface, and (ii) $TiAl_3$ spherules pushed into the SiCp/Al layer. The latter could enhance the reaction rate significantly depending on a shorter diffusion