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A study on diffusion couples of Ti and polysynthetically twinned (PST) Ti–Al: II. Interdiffusion results

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Abstract

Interdiffusion in multi-phase diffusion couples of polycrystalline Ti and polysynthetically twinned (PST) Ti–49.3 at.% Al, with the diffusion direction parallel to the lamellar planes, is investigated in the temperature range 973–1173 K. A reaction zone (RZ) of the α_2-Ti$_3$Al phase forms between the end materials and exhibits deeper penetration in the α_2 lamellae than in the primary γ lamellae. Direct measurements of the RZ thickness reveal a parabolic growth of the RZ, indicating a diffusion-controlled growth macroscopically. Concentration profiles from the Ti, through the RZ, into the PST γ and α_2 lamellae are measured by X-ray spectroscopy in a transmission electron microscope. Deviations from a diffusion-controlled composition profile indicate some extent of interface-controlled growth. The interdiffusion coefficients are found to be largely independent of composition with a temperature dependence that obeys the Arrhenius relationship with a pre-exponential factor of $D_0 = (7.56 \pm 7.14) \times 10^{-5}$ m^2/s and an activation enthalpy of $Q = (255.6^{+8.9}_{-8.3})$ kJ/mol.
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1. Introduction

Titanium aluminide-based intermetallic alloys have great potential in high-temperature structural applications in aerospace and automotive industries [1,2]. Mechanically superior alloys of this system usually have a two-phase lamellar structure of the γ-TiAl phase and the α_2-Ti$_3$Al phase. Diffusion plays an important role in the formation and high-temperature stability of the lamellar structure. Furthermore, diffusion is an essential determinant of some mechanical properties, such as creep resistance. Therefore, knowledge of the diffusion process in polysynthetically twinned (PST) Ti–Al 'single' crystal, composed of alternate layers of γ-TiAl and α_2-Ti$_3$Al, is of great importance for the understanding and development of titanium aluminide alloys.

There is a large amount of diffusion data available on the Ti–Al system, including tracer self-diffusion coefficients in polycrystalline γ-TiAl [3], α_2-Ti$_3$Al [4] and PST Ti–Al [5] alloys, interdiffusion coefficients in single-phase diffusion couples made up of polycrystalline TiAl alloys [6], Ti$_3$Al alloys [4] as well as in multi-phase diffusion couples [7]. Recently, grain boundary (GB) tracer diffusion experiments were also carried out by Herzig et al. [8] in the grain boundaries of TiAl, Ti$_3$Al and the PST materials. However, tracer diffusion experiments measure the average layered concentration profile. The technique cannot distinguish the effects of each individual phase and of the various types of interfaces in a PST crystal on diffusion. Little has been known about the fundamental correlation between the microstructure and diffusion properties, especially in reactive interdiffusion cases. In the present paper, we present interdiffusion results in multi-phase diffusion couples of Ti and PST Ti–Al crystal. The microstructural evolution of the system has been reported in the previous paper.

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2. Experimental procedures

Diffusion-anneals of the as-bonded diffusion couples of Ti and PST Ti–Al were carried out in a high vacuum furnace

Table 1
The bonding and annealing conditions of PST-Ti diffusion couples

	Temperature (K)	Time (h)
Bonding	873	2
Annealing	973	8+6+6
	1023	4+6+6
	1073	2+4+4+4
	1123	2+2+2+2
	1173	2+2+2+2

at five different temperatures for various times, as listed in Table 1. The addition signs in the table indicate a series of consecutive diffusion-anneals on the same diffusion couple at the same temperature, with the numbers following the addition signs representing the additional annealing times. The bonding interfaces of PST Ti–Al were the $\{1\bar{1}0\}$ planes of γ-TiAl phase, assuming the lamellar planes in the γ phase were (111). Thus, the lamellar interfaces were perpendicular to the bonding interface.

Cross-sections of the as-bonded diffusion couples were cut perpendicular to both the bonding interface and the lamellar interfaces of the PST crystal, i.e. $\{11\bar{2}\}$ of the γ phase, and were observed with a JEOL 6400 scanning electron microscope (SEM) operated at 15 kV. Transmission electron microscope (TEM) specimens were cut from the annealed diffusion couples using a Strata DB 235 dual beam focused ion beam (FIB) system at Lehigh University. Most of the thin foils prepared have foil normals close to $\langle 11\bar{2} \rangle$, in about the same surface orientation as the SEM specimens. TEM specimens were examined in a JEOL 2010F TEM operated at 200 kV. Energy dispersive X-ray spectroscopy (EDS) in the scanning transmission electron microscopy (STEM) mode of the 2010F was used to measure the chemical composition profiles of the diffusion couples. A TEM specimen was prepared from a bulk polycrystalline Ti–34 at.% Al (α_2-Ti$_3$Al single phase) sample as a standard. The bulk Ti–34 at.% Al sample was prepared by arc-melting, followed by 10 h annealing at 1273 K in a high vacuum furnace to ensure homogeneity. It was then sectioned and mechanically polished. In addition, wavelength dispersive spectroscopy (WDS) in a JEOL 6400 SEM was applied to confirm its composition using pure Ti and Al as standards. The average of 10 readings was 33.45 at.% Al. A TEM specimen was then prepared from the bulk specimen by conventional thinning, dimpling and ion milling. The quantitative chemical analysis utilizes the Cliff-Lorimer ratio technique [9].

3. Results and discussions

As reported in the previous paper, a reaction layer or reaction zone (RZ) with wavy contour in the PST Ti–Al side emerges at the bonding interface after annealing and its thickness increases with annealing time. Electron diffraction patterns in TEM have verified that it is the α_2-Ti$_3$Al phase,

which follows from the equilibrium phase diagram. To elucidate the growth kinetics of the reaction layer, SEM observations and measurements on back-scattered electron images were carried out on the bulk diffusion couples after each anneal. The clear contrast difference in the SEM back-scattered electron images between the RZ and the PST crystal allows a direct measurement of the thickness of the RZ, as shown in Fig. 1. The upper white part is the polycrystalline Ti and the lower lamellar structure is the PST crystal, with the gray vertical thin laths the α_2-Ti$_3$Al lamellae and the majority dark lamellae the γ-TiAl phase. The light gray layer between the Ti and the PST lamellae is the so-called RZ.

A series of SEM back-scattered electron images were recorded over a continuous region to form a map of each diffusion couple at each annealing condition. Since the RZ/Ti interface was mostly straight, a line across the interface of the RZ and Ti through the images was drawn and regarded as the zero-thickness level of the RZ, as illustrated in Fig. 1. The thickness of the RZ, x, was measured at each point where the RZ exhibited local minima or maxima in thickness adjacent to the γ-TiAl lamellae, categorized as γ-min and γ-max, respectively. In order to obtain the net increase of x corresponding to each individual annealing period, the average RZ thickness of the as-bonded diffusion couples was subtracted from the measurements in the annealed diffusion couples. Hundreds of measurements were made and averaged for both categories. The growth of the reaction layer was then illustrated by plotting x^2 versus t at each temperature, as shown in Fig. 2, with the solid and dotted lines representing the results at γ-min and γ-max, respectively. It is revealed that x increases parabolically with annealing time t, which indicates that the growth of the α_2-Ti$_3$Al phase in the RZ is mainly diffusion-controlled at a macroscopic level. It is also seen that the difference between the measured thickness of the RZ at the γ-min and γ-max locations increases with time indicating different effective local diffusion coefficients. This effect is believed to be due to the presence of γ/γ interfaces in the material, which do not produce contrast in SEM back-scattered electron images. Therefore, γ-max
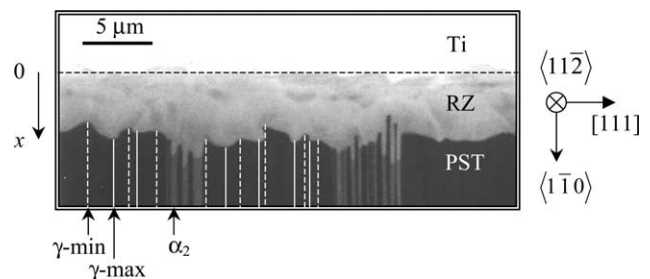

Fig. 1. Illustration of the measurements of the RZ thickness on SEM back scattered electron images. The upper white part is the polycrystalline Ti and the lower lamellar structure is PST crystal, with the gray vertical laths the α_2-Ti$_3$Al lamellae and the dark thicker laths the γ-TiAl phase. The light gray layer between the Ti and PST is the so-called RZ.