



November 2000

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Pan, L., & Luzzi, D. E. (2000). Interdiffusion and Phase Behavior in Polysynthetically Twinned (PST) TiAl / Ti Diffusion Couples. Retrieved from http://repository.upenn.edu/mse_papers/15

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2000 Fall Meeting Symposium N
High-Temperature Ordered Intermetallic Alloys IX
Publisher URL: http://www.mrs.org/members/proceedings/fall2000/n/N5_53.pdf

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Abstract

Diffusion couples of pure Ti and polysynthetically twinned (PST) TiAl (49.3 at.% Al) were prepared by high vacuum hot-pressing, with the bonding interface perpendicular to the lamellar planes. Diffusion experiments were carried out by annealing the couples in the same furnace at 650, 700 and 850°C for various times. The cross-section of the couple was studied using scanning electron microscopy (SEM) and quantitative wavelength-dispersive x-ray spectroscopy (WDS). A reaction layer whose composition is close to that of the stoichiometric α_2 -Ti₃Al phase formed along the PST TiAl / Ti bonding interface in PST TiAl side. Direct measurements of the thickness of the reaction zone were performed at different phase regions and various boundaries. By assuming the thickness of the reaction zone increases as $(Dt)^{1/2}$, where D is the diffusion coefficient and t is the annealing time, the diffusion coefficients at these temperatures were calculated. Composition profiles in the reaction zone, along the lamellae and at the lamellar interfaces were obtained by WDS analyses.

Comments

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Interdiffusion and Phase Behavior in Polysynthetically Twinned (PST) TiAl / Ti Diffusion Couples

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ABSTRACT

Diffusion couples of pure Ti and polysynthetically twinned (PST) TiAl (49.3 at.% Al) were prepared by high vacuum hot-pressing, with the bonding interface perpendicular to the lamellar planes. Diffusion experiments were carried out by annealing the couples in the same furnace at 650, 700 and 850°C for various times. The cross-section of the couple was studied using scanning electron microscopy (SEM) and quantitative wavelength-dispersive x-ray spectroscopy (WDS). A reaction layer whose composition is close to that of the stoichiometric α_2 -Ti₃Al phase formed along the PST TiAl / Ti bonding interface in PST TiAl side. Direct measurements of the thickness of the reaction zone were performed at different phase regions and various boundaries. By assuming the thickness of the reaction zone increases as $(Dt)^{1/2}$, where D is the diffusion coefficient and t is the annealing time, the diffusion coefficients at these temperatures were calculated. Composition profiles in the reaction zone, along the lamellae and at the lamellar interfaces were obtained by WDS analyses.

INTRODUCTION

Intermetallic compounds made of the light elements Ti and Al are promising candidates for aerospace, automotive and turbine power generation applications[1-5]. In the past decade, special interest has been paid to poly-synthetically twinned (PST) TiAl, composed of alternate lamellae of the γ -TiAl phase and the α_2 -Ti₃Al phase with the orientation relationship $\{111\}_\gamma // (0001)_{\alpha_2}$ and $\langle 1 \bar{1}0 \rangle_\gamma // \langle 11 \bar{2}0 \rangle_{\alpha_2}$ [6]. PST TiAl exhibits low temperature ductility [7, 8] and higher toughness and high-temperature strength than TiAl alloys with other microstructures [9, 10].

At high temperatures the physical and mechanical properties of materials are generally associated with diffusion. In the Ti-Al alloy system, the formation and high-temperature stability of the lamellar structure are controlled by diffusion processes within the two phases and along the γ/γ and γ/α_2 lamellar boundaries. Moreover, diffusion is an important determinant of the creep resistance of the lamellar structure [11, 12]. Therefore, a fundamental understanding of the diffusion mechanisms in PST TiAl alloys is of great importance for the development of titanium aluminide alloys.

Current understanding of the diffusion processes in the Ti-Al system is largely based on the studies by Herzig *et al.* [13-16], who performed a series of tracer diffusion experiments, including self-diffusion, in polycrystalline γ -TiAl and α_2 -Ti₃Al. The penetration profiles for diffusion in large grain size (> 1 mm) materials exhibit a $c \propto \exp(-x^2 / 4Dt)$ behavior for instantaneous sources and $c \propto \text{erfc}[x / 2(Dt)^{1/2}]$ behavior for constant sources, as in semi-infinite materials. c is the average layered concentration of the diffusant and x is the penetration depth. In smaller grain size materials (350 - 500 μm), grain boundary diffusion is dominant in regions away from the surface ($> 100 \mu\text{m}$), where the concentration profile follows the numerical rule of $\log c \propto x^{6/5}$.

In the present paper, we present initial results on diffusion couples of Ti and PST TiAl crystal. This is the first experimental attempt to elucidate the diffusion and phase behavior in the two phases of a PST TiAl crystal, and will shed light on the interface diffusion along various lamellar boundaries in PST TiAl.

EXPERIMENTAL PROCEDURES

The composition of the master ingots used in the present study was Ti-49.3 at.% Al. 99.999% Ti and 99.99% Al were arc-melted at least four times to ensure homogeneity of the as-cast ingots. PST TiAl crystals were grown from the master ingots in an optical floating zone furnace under flowing argon gas with a growth rate of 3 mm/h. The back-reflection Laue X-ray diffraction technique was used to align the crystal along certain orientations. Slices parallel to the $\{1\ \bar{1}0\}$ planes, about 0.5 mm thick, which are also perpendicular to the lamellar planes, were cut from the as-grown PST crystals. The directions mentioned here and in the remainder of the paper are with respect to the γ -TiAl phase of the PST crystal. For diffusion-bonding experiments, the slices of PST crystals were mechanically and electrolytically polished in a solution of 6 vol.% perchloric acid (70%), 35 vol.% n-butyl alcohol and 59 vol.% methanol prior to diffusion bonding. Bulk Ti (99.999%) specimens were mechanically polished in parallel using an Allied High Tech MultiPrep polisher using silicon carbide paper to an ultimate finish of 1200-grit. Diffusion couples of PST TiAl and Ti were produced by diffusion bonding in a high vacuum furnace at 600°C for two hours. No extra mechanical stress was applied to the material during diffusion bonding except that from the thermal expansion of the graphite rams. Cross-sections of the as-bonded diffusion couples were cut perpendicular to both the bonding plane and the lamellar interfaces of the PST crystal. This cross-section was the observation surface in the microscope studies.

The as-bonded diffusion couples were subjected to diffusion anneals in the same furnace at three different temperatures under high vacuum conditions. Three diffusion couples were annealed at 650°C for 8 hours, 700°C for 8 hours, and 850°C for 2 hours, respectively. For the convenience of illustration, the diffusion couple annealed at 650°C for 8 hours is denoted as PST-Ti1, the one annealed at 700°C for 8 hours is denoted as PST-Ti2, and the one annealed at 850°C for 2 hours as PST-Ti3. SEM observations and quantitative WDS chemical analyses were carried out with a JEOL6400 scanning electron microscope operated at 15kV.

RESULTS AND DISCUSSIONS

Typical SEM back-scattered electron images of the as-bonded PST-Ti diffusion couple and the annealed PST-Ti1 are shown in figure 1. The upper white part at the very top of each image is the bulk Ti and the lower lamellar structure is PST crystal, with the lighter contrast vertical laths of α_2 -Ti₃Al lamellae and darker γ -TiAl phase visible. A reaction zone with a wavy contour forms on the PST crystal side of the bonding interface and has a clear contrast difference with respect to both phases of the PST crystal. Especially, the image of the annealed diffusion couple (figure 1(b)) shows the reaction zone penetrates into the α_2 lamellae. Quantitative WDS chemical analysis indicated that the composition in the reaction zone is close to the stoichiometric α_2 -Ti₃Al, while in the α_2 lamellae of the PST crystal the Al concentration is around 37 at. %, close to the expected equilibrium concentration of α_2 at 650°C for two-phase γ - α_2 alloys. These compositions are consistent with the contrast in the image.

The deeper penetration depth of the reaction zone into the α_2 lamellae than into the γ phase leads to an intuitive guess that Ti penetrates or diffuses into the α_2 lamellae at a faster rate. Considering the composition of the material and the morphology of the PST crystal, pure interdiffusion between the α_2 phase of the reaction zone and the α_2 as in a bulk diffusion couple is possible, whereas the γ -TiAl phase in the PST crystal must transform into the α_2 -Ti₃Al phase prior to diffusion as the TiAl composition is at the Ti-rich phase boundary. However, the sharp contrast difference visible deep in the α_2 lamellae marking the boundary of Ti-rich Ti₃Al is not understood. The contrast seen in the SEM image of Figure 1(b) implies a sharp drop in average atomic number of the material at the point within the α_2 lamellae where the boundary between light and dark grey contrast is seen. Compositional analysis by WDS, within the approximately 1 μ m resolution limit of the SEM, confirms that this is associated with a sharp change in Ti concentration. The origin of this behavior will be explored through future TEM analysis.

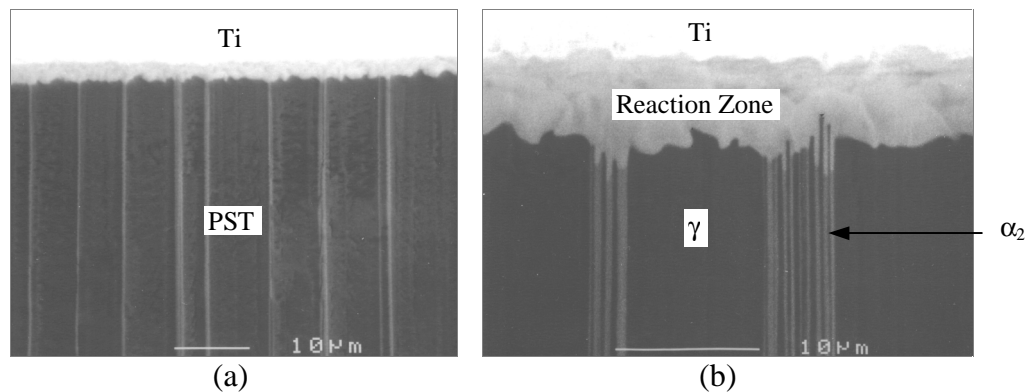


Figure 1. SEM back-scattered electron images of (a) as-bonded PST-Ti diffusion couple and (b) PST-Ti1 annealed at 650 °C for 8 hours after bonding. The upper white part is Ti bulk and the lower lamellar structure is PST crystal, with the light contrast thinner lamellae α_2 -Ti₃Al phase and the darker region γ -TiAl phase. The layer between Ti and PST along the bonding interface is what we call reaction zone.

It was also found through SEM analysis that the penetration depth into the PST crystal of the reaction zone in any one diffusion couple varies. This variation is seen not only from differences in the penetration depth into the γ and α_2 phases, but also when comparing among individual γ lamellae or α_2 lamellae. The clear contrast difference in the SEM back-scattered images between the reaction layer and the two phases of the PST crystal allows a direct measurement of the penetration depth of the reaction zone as a function of position in each specimen. Hundreds of individual measurements of reaction zone thickness as a function of position were made using wide-area maps of each specimen created from many contiguous SEM images of the back-scattered electron signal. In order to create a common reference point, a line across the upper edge of the reaction zone through the images was drawn and regarded as the zero-depth level of the reaction zone, as shown in figure 2.

Measurements were divided into four categories. The penetration depths into α_2 lamellae were treated as one category. For the γ phase, the minima of the penetration depth were measured and labeled “ γ ”. The maxima were labeled “ γ/γ ”, with the assumption that the deepest penetration occurs at the points of the interfaces between the γ lamellae, which are invisible in

the SEM. Finally, the thin γ lamellae sandwiched between α_2 lamellae, were placed in a separate category termed “ γ in α_2 ”. The thickness of the reaction zone, x , was measured vertically from the reference line to the point where the contrast difference marking the boundary of significant Ti penetration occurs, at the marked locations corresponding to each of the four categories as indicated in figure 2. Hundreds of measurements were made and then averaged for each category.

The distribution of the reaction zone thickness verified the deeper penetration into the α_2 lamellae than for the other three categories. For γ or α_2 lamellae with about the same thickness, the penetration depths usually are not the same. Hence, the thickness of the α_2 lamellae is not the dominant factor in the deeper penetration. This implies a faster penetration of Ti into the α_2 phase than into the γ phase.

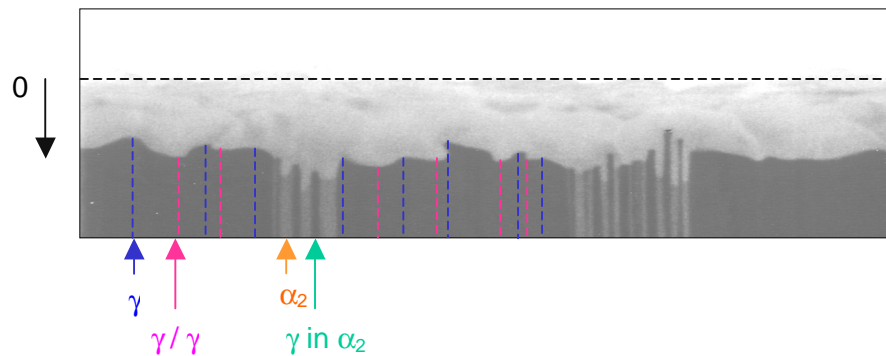


Figure 2. Illustration of the direct measurement method of the thickness of the reaction zone.

Under the approximation that the penetration depth, i.e. the thickness of the reaction zone, is approximately equal to $(Dt)^{1/2}$, where D is the diffusion coefficient and t is the annealing time, the diffusion coefficients for the four categories were calculated. The results are presented in table I. The values for the diffusion coefficients determined from the present measurements are 3-4 orders higher than the Ti self-diffusion coefficients in single phase γ -TiAl or α_2 -Ti₃Al materials obtained from tracer diffusion experiments for diffusion through the bulk[14, 15]. It should be noted that the tracer diffusion experiments were on large grain, random polycrystals with a low density of interfaces.

Table I. Calculated results of diffusion coefficients for PST-Ti1, PST-Ti2 and PST-Ti3.

	t (hr)	T (K)	γ	γ/γ	γ in α_2	α_2
			D (m ² /s)	D (m ² /s)	D (m ² /s)	D (m ² /s)
PST-Ti1	8	923	4.15E-16	5.80E-16	5.46E-16	9.33E-16
PST-Ti2	8	973	1.81E-15	2.42E-15	2.33E-15	3.94E-15
PST-Ti3	2	1123	2.39E-15	3.21E-15	3.06E-15	6.00E-15

In order to study the details of the concentration gradient across the reaction zone and into the PST crystal, quantitative WDS analyses with ZAF corrections were carried out on PST-Ti2 using pure Ti and Al as standards. For these initial measurements and to examine the unexpected

sharp contrast difference seen in the SEM images, the profile across the reaction zone and into the α_2 lamellae was studied. Two models were applied to analyze the composition profiles. One assumes that the concentration profile is determined by volume diffusion in the α_2 phase, i.e., the diffusion couple acts just like a bulk Ti/Ti₃Al diffusion couple. By applying the constant source condition and letting $c|_{x \rightarrow \infty}$ be the Ti concentration in the α_2 phase in PST TiAl, which is about 63 at.%, i.e.

$$c_1 = c|_{x < 0} = 1, \quad c_2 = c|_{x \rightarrow \infty} = 0.63, \quad (1)$$

an analytical solution to Fick's diffusion equation for the case of two semi-infinite bulk diffusion couples is obtained:

$$\frac{c - c_2}{c_1 - c_2} = \frac{1}{2} \operatorname{erfc}\left(\frac{x}{2\sqrt{Dt}}\right) \quad (2)$$

The inverse complementary error function of $2(c - c_2)/(c_1 - c_2)$ versus x from the experimental data is plotted in Figure 3a. If the model of volume diffusion in a semi-infinite bulk is applied, the data should fall on a straight line with the diffusion coefficient determining the slope of the line. The data falls remarkably well on a straight line through the reaction zone and into the α_2 lamellae, but has a discontinuity around the depth 11-12 μm corresponding to the contrast difference in the SEM image, whose origin will rely on the future TEM study to clarify. Using the data points at depths less than that corresponding to the discontinuity to calculate the diffusion coefficient, a value of $D = 3.15 \times 10^{-15} \text{ m}^2/\text{s}$ was obtained, which is comparable with the value obtained by the measurement of reaction zone thickness, $3.94 \times 10^{-15} \text{ m}^2/\text{s}$.

The second model applied to analyze the data is the grain boundary diffusion model. For type B and C grain boundary diffusion kinetics, there is a numerical rule [17] that $\ln c$ is proportional to $x^{6/5}$. We plotted the same Ti concentration profile as $\ln c$ versus $x^{6/5}$ (see figure 3b). In this case, the data does not display an obvious linear aspect and the discontinuity is seen at a value of 18-19 $\mu\text{m}^{6/5}$, which suggests that the diffusion under these conditions in the PST system follows either type A or a mixture of type A and B kinetics, where the bulk diffusion effect cannot be ignored.

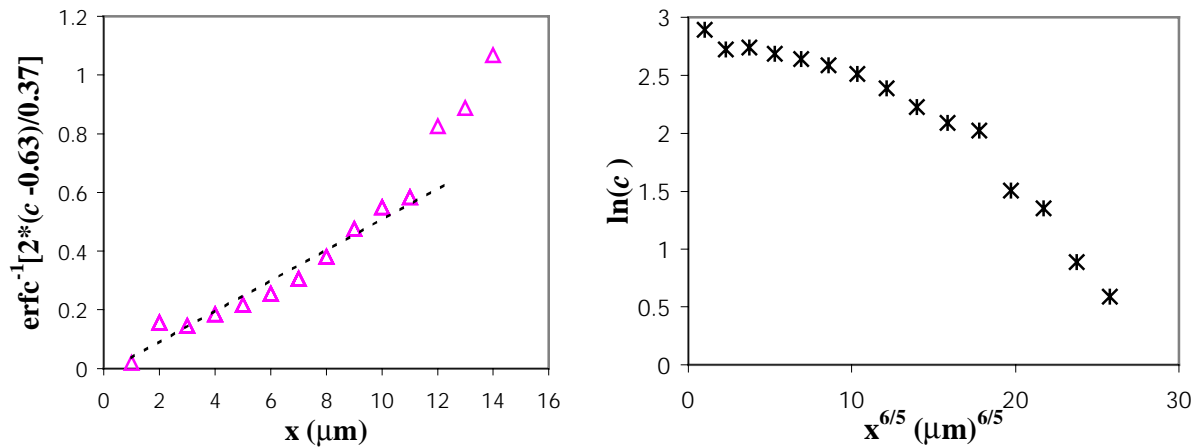


Figure 3. (a) Composition profile in α_2 lamella fitted by complementary error function, (b) Composition profile in α_2 lamella fitted by $\ln(c)$ vs. $x^{6/5}$.

CONCLUSIONS

In diffusion couples of Ti and PST TiAl, a reaction layer forms with composition close to the stoichiometric α_2 -Ti₃Al phase. Ti diffuses into the PST α_2 phase while the PST γ phase transforms into the α_2 phase. Diffusion coefficients obtained by directly measuring the thickness of the reaction layer are 3-4 orders higher than the self-diffusion coefficients from the tracer diffusion experiments in single phase polycrystalline material. The composition profiles in α_2 lamellae showed surprising features that rely on future TEM studies to solve.

ACKNOWLEDGEMENT

This research was supported by National Science Foundation grant no. DMR96-15228.

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