Fabrication of wavy γ-TiAl based sheet with foil metallurgy

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Abstract: A 0.7 mm-thick wavy γ-TiAl sheet with fully lamellar microstructure was fabricated by hot pressing Ti/Al alternate foils with heat treatment of 640 °C, 15 h+850 °C, 35 h+1350 °C, 2 h. The intermetallic compounds formed during heat treatments were identified by scanning electron microscopy (SEM) and X-ray diffraction (XRD). $TiAl_3$ was the only observed phase at the Ti/Al interface when Al foils were not consumed. After being annealed at 850 °C for 35 h, the microstructure was composed of α-Ti, α_2-Ti_3Al, γ-TiAl and $TiAl_2$. A fully lamellar microstructure formed after annealing at 1350 °C. Most of the angles between the lamellar interface and the sheet plane are below 30°. Using thinner starting foils is favorable to produce sheets with fine microstructure.

Key words: solid state reaction; phase transformation; titanium aluminides; foil metallurgy

1 Introduction

γ-TiAl based materials with fully lamellar microstructure have great potential in aerospace and automotive applications, due to their high specific strength and excellent high temperature properties [1−3]. The lamellar microstructure shows a significant anisotropy in mechanical properties according to the previous studies on polysynthetically twinned (PST) crystals. When the loading axis is parallel to the lamellar boundaries, the materials exhibit an optimal combination of yield strength and ductility [4]. PST crystals with their boundaries parallel or perpendicular to the stress axis show higher yield stress and longer creep life than those with their lamellar boundaries oriented at an intermediate angle to the stress axis [5]. For real fully lamellar polycrystalline γ-TiAl alloy, the high volume fraction of the lamellar colonies inclined at low angles contributes to higher yield strength than the random distribution of lamellar orientation [6, 7]. PST crystals with notch plane perpendicular to lamellar boundaries show a better toughness than that of notch plane parallel to lamellar boundaries [8]. As a result, γ-TiAl sheet materials with lamellar boundaries parallel to the sheet plane are expected to show a good combination of strength,

ductility and toughness. Because of the poor ductility of γ-TiAl alloy, there are no relative simple approaches for producing TiAl-based sheet, especially those with complicated shape, such as honeycomb, corrugation and gator-hide structure[9]. Elemental foils have been used to fabricate intermetallics sheets due to their relatively better ductility.

In the present study, a wavy TiAl sheet with a particular microstructure mentioned above was fabricated by hot pressing the alternate Ti/Al foils, which was a low-cost method. Before the fabrication of the wavy sheet, a flat sheet was produced to investigate the inner mechanism.

2 Experimental

Elemental titanium foils (99.6%, molar fraction, 0.030 mm in thickness) and elemental aluminum foils (99.6%, molar fraction, 0.023 mm in thickness) were employed. The Ti and Al foils were etched in 10% HF and 15% NaOH solution, respectively, water washed, alcohol cleaned and dried. Alternating layers of titanium and aluminum foils were stacked with outer layers of titanium foils. The nominal composition was Ti−46% Al. Two procedures were applied in the present work. In the first procedure, stacked foils were hot pressed at 610 °C

for 3 h, cut into size of 6 mm×6 mm and assembled in a stainless steel sample holder which was sealed in a quartz tube filled with argon, followed by three heating stages: 640 °C, 12 h; 850 °C, 35 h and 1350 °C, 2 h. In the second procedure, to produce wavy TiAl sheet, stacked foils were hot pressed in vacuum better than those stacked under 10^{-2} Pa and 15 MPa. The basic process consisted of successive three heating stages: 650 °C, 15 h; 850 °C, 35 h and 1350 °C, 2 h.

The intermetallic compounds formed during heat treatments were identified by scanning electron microscopy (SEM) and X-ray diffraction (XRD). The distribution of lamellar orientation was estimated based on different parts of specimens by metallographic method.

3 Results and discussion

3.1 Hot pressing below Al melting point

Figure 1(a) shows the cross-sectional microstructure of specimen subjected to heat treatment of 610 °C, 3 h in the first procedure. As seen in Fig. 1(a), the interfaces between the bonded Ti and Al foils are fairly straight, indicating a good bonding between Ti and Al foils. At higher magnification, thin reaction layers with an average thickness of 0.5 μm can be observed (Fig. 1(b)), and further heat treatment results show that before Al foils are consumed, there is only one compound layer between Ti and Al foils, which is consistent with the work by YANG et al [10]. XRD analysis (as shown in Fig. 2) shows that this reaction layer is $TiAl_3$. There are several compounds in Ti−Al system, such as $TiAl_3$, $TiAl_2$ TiAl and Ti_3Al. The preferential growth of $TiAl_3$ is the result of interface thermodynamics and growth kinetics. The slight mismatch between the closed planes of Ti, Al and $TiAl_3$ also favors the first formation of $TiAl_3$. TARDY and TU [11] has pointed out that epitaxial relationship of $(112)_{TiAl_3}//(001)_\alpha$ and $\langle 110 \rangle_{TiAl_3}//\langle 110 \rangle_\alpha$ exist between the Ti and $TiAl_3$ layers. Figure 3(a) shows the microstructure of the cross section after the heat treatment of 610 °C, 3 h + 640 °C, 12 h. As seen in Fig. 3(a), Al is consumed and replaced by $TiAl_3$, and there are many pores exist in $TiAl_3$ layer, especially in the center zone where a fissure is formed due to the high concentration of voids caused by Kirkendall effect [12]. When Al diffuses into Ti foils, $TiAl_3$ is formed at the interface and voids are formed in Al foils. When the $TiAl_3$ layer grows towards Al foils, the voids move with the migration of the interface between Al and $TiAl_3$. Hence, the voids exist in the form of rows at the mid-plane sites of the starting Al foils. Al foils are substituted by $TiAl_3$ layers when they are consumed. Thus, rows of voids exist in the mid-plane of $TiAl_3$ layers. When the sample is cooled from high temperature, a
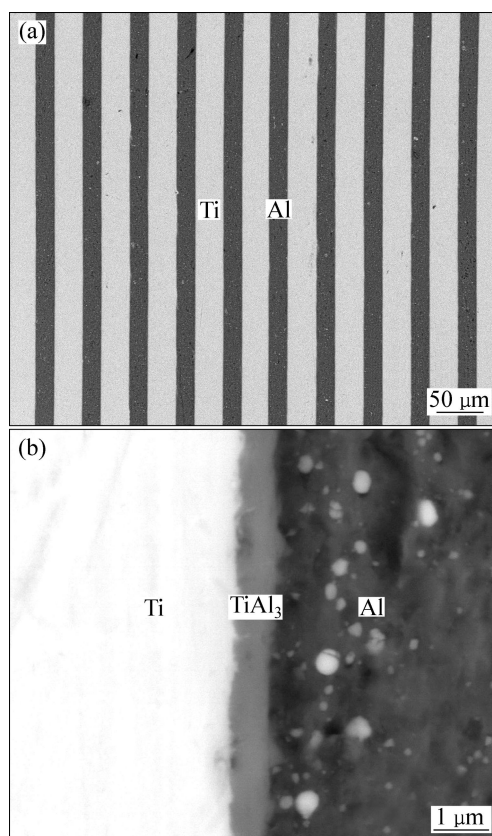

Fig. 1 SEM images of cross section of sample after heat treatment of 610 °C, 3 h (a) and $TiAl_3$ with average thickness of 0.5 μm appeared at Ti/Al interface (b)
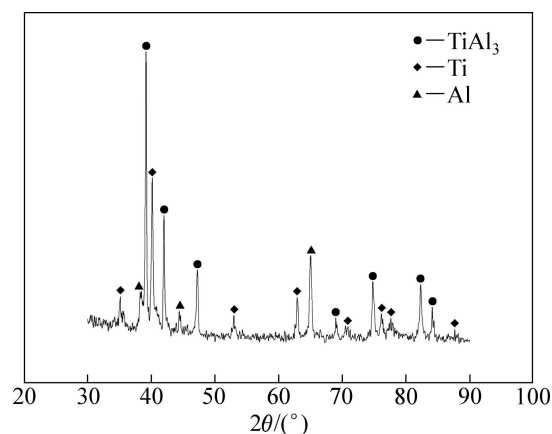

Fig. 2 XRD pattern of cross sections of samples after heat treatment of 610 °C, 3 h

fissure may be formed by cracking along the rows of voids as $TiAl_3$ are extremely brittle. It should be pointed out here that if subjected to a proper pressure, these pores would disappear.

3.2 Hot pressing at high temperatures

EDS analysis shows that the microstructure of the cross section after the heat treatment of 610 °C, 3 h +
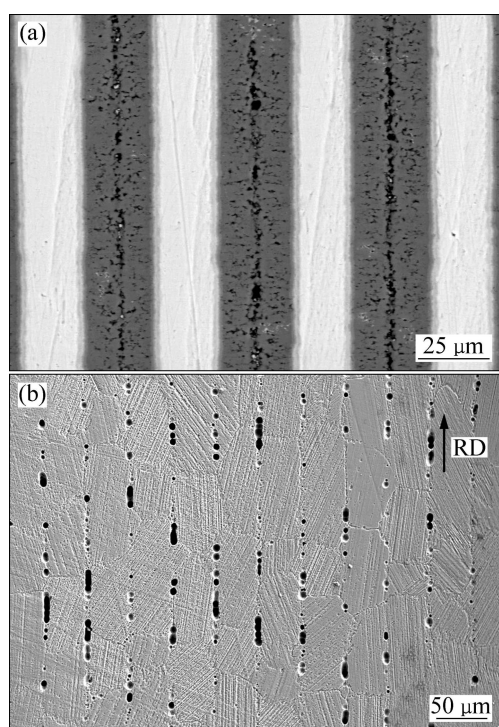
Fig. 3 SEM images of cross section of sample after different heat treatments: (a) 610 °C, 3 h + 640 °C, 12 h; (b) 610 °C, 3 h + 640 °C, 12 h + 850 °C, 35 h + 1300 °C, 2 h

640 °C, 12 h + 850 °C, 35 h is composed of α-Ti, α_2-Ti$_3$Al, γ and TiAl$_2$ layers. This indicates that after all the Al foils have transformed to TiAl$_3$, α_2, γ and TiAl$_2$ form between Ti and TiAl$_3$. Further experimental results show that α_2-Ti$_3$Al, γ and TiAl$_2$ layers widen, and α-Ti and TiAl$_3$ layers become thinner with the increase of annealing time. When TiAl$_3$ is exhausted, α_2-Ti$_3$Al and γ grow at the expense of TiAl$_2$ and α-Ti. XRD patterns shown in Fig. 4 reveal the corresponding microstructure evolution. After heat treatment of 850 °C, 5 h, the microstructure is composed of TiAl$_3$, TiAl$_2$, TiAl, Ti$_3$Al and α-Ti. Judging from the XRD patterns shown in Fig. 4, when subjected to the heat treatment of 850 °C for 20 h, the microstructure consists of TiAl$_2$, TiAl and Ti$_3$Al. Compared with the EDS results, the pattern of α-Ti is absent, which may be caused by the slight quantity of α-Ti. After heat treatment for sufficient time, the microstructure is composed of α_2 and γ [13]. Figure 3(b) shows the cross section microstructure after the heat treatment of 610 °C, 3 h + 640 °C, 12 h + 850 °C, 35 h + 1350 °C, 2 h. It is seen that the lamellar microstructure develops in the whole area of the specimen, i.e., the fully lamellar microstructure develops. The XRD patterns (Fig. 4) show the matrix is composed of α_2-Ti$_3$Al and γ. The distance between two adjacent rows of pores is the total thickness of single Ti foil and single Al foil. The sizes of most colonies in the sheet thickness direction are nearly

the same, equal to the total thickness of one Ti foil and one Al foil, indicating that thinner starting Ti and Al foils are good to yield fine microstructures. Compared with the microstructure shown in Fig. 1(b), the number of pores in the fully microstructure shown in Fig. 3(b) is less. This is the result of the larger force loaded on the specimen when heat treated at higher temperature.

As seen in Fig. 3(b), only one kind of alignment exists in one lamellar colony. The alignments of most colonies are nearly parallel to RD. The distribution of lamellar interfaces is shown in Fig. 5(a). 80% of the angles between the lamellar interface and the rolling direction are smaller than 30°. On the contrary, the frequency around 40°−90° is quite low. As a result, most of the lamellaes are parallel to the sheet plane.
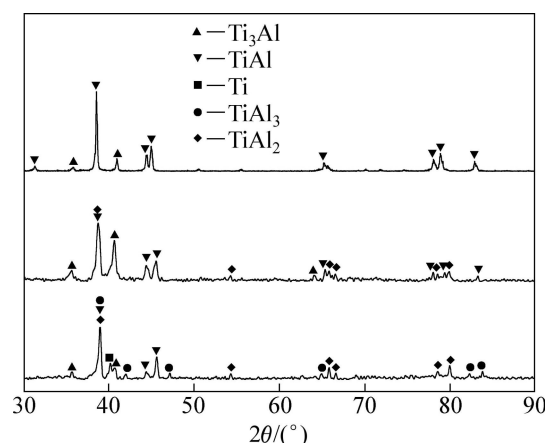

Fig. 4 XRD patterns of samples after different heat treatments: (a) 640 °C, 12 h + 850 °C, 35 h + 1350 °C, 2 h; (b) 640 °C, 12 h + 850 °C, 20 h; (c) 640 °C, 12 h + 850 °C, 5 h

3.3 Formation of oriented lamellar microstructure

When Ti−(35−48)%Al (molar fraction) alloy is cooled from the α single phase region to the (α_2 + γ) two-phase region, (111)$_\gamma$ lamellar planes are parallel to the (0001)$_\alpha$ planes [14]. So if the rolled α-Ti foils and sheets can be held in the α-Ti single phase region at high temperature (>1200 °C) by enriching Al, a final fully lamellar microstructure where the lamellaes are parallel to the sheet surface is obtained by cooling to the (α_2 + γ) two phase region. In Ti−Al alloy, when α phase forms from β phase, the (0001)$_\alpha$ may lie in any six possible (110)$_\beta$ planes according to the orientation relationship of (0001)$_\alpha$ //(110)$_\beta$ and $\langle 11\bar{2}0 \rangle_\alpha$ //$\langle 111 \rangle_\beta$ [15]. That is, if the transformation of $\alpha \rightarrow \beta \rightarrow \alpha$ takes place, α-Ti foils subjected to such a phase transformation have a weaker (0001)$_\alpha$ texture than the starting ones. As a result, the transformation of $\alpha \rightarrow \beta$ should be avoided. In present work, enriching Al is performed at 850 °C, which is a relatively high temperature below the transformation

temperature of $\alpha \rightarrow \beta$. Figure 6 shows a (002) pole figure of the starting titanium. The pole densities are projected onto the surface of the foils and the mean pole density is used as a unit. The area of the high pole density is
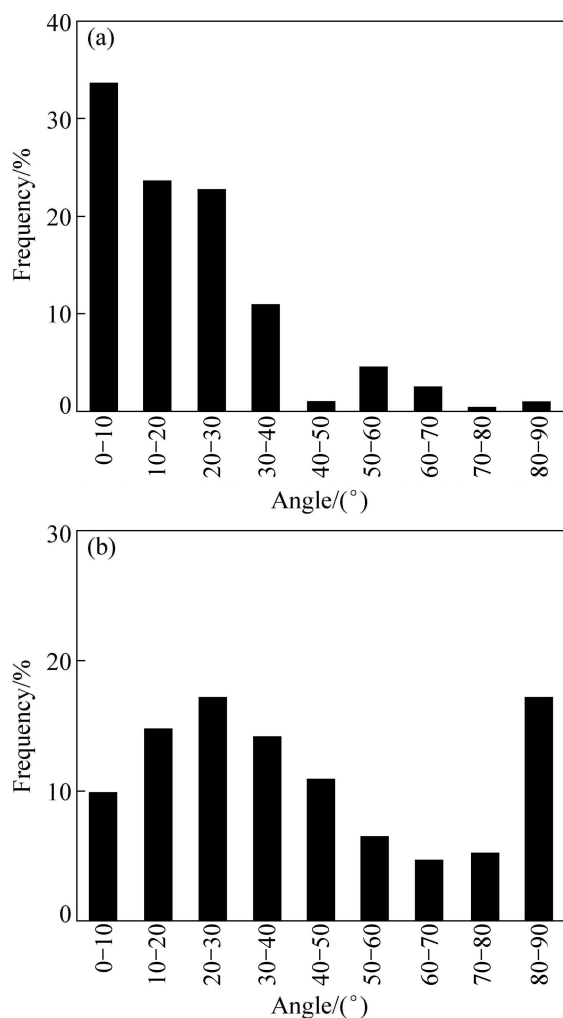

Fig. 5 Distribution of angles between rolling direction and lamellar interfaces after different heat treatments: (a) 640 °C, 12 h + 850 °C, 35 h + 1300 °C, 2 h; (b) 640 °C, 12 h + 1000 °C, 10 h + 1350 °C, 2 h
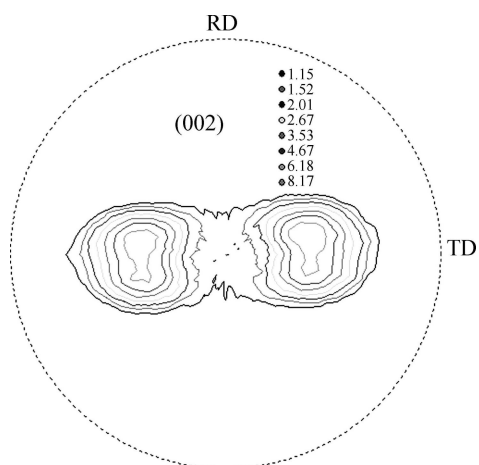

Fig. 6 (002) pole figure of starting titanium foil

situated at about 30° to 40° from the center of the figure in the transverse direction. The maximum pole density is about 8.

As discussed above, when heat treated above 850 °C, not all Ti atoms in the starting Ti foils exist in the form of α-Ti, and some of them transform into γ-TiAl and Ti_3Al. Fortunately, these transformations do not weaken the $(0001)_\alpha$ texture obviously. When the starting Al foils transform into $TiAl_3$, the diffusion couple becomes $Ti/TiAl_3$. Although it is failed to determine the first formation phase in $Ti/TiAl_3$ diffusion couple, previous studies have shown that γ-TiAl forms first at the $Ti/TiAl_3$ interface [16]. Because the misfit between the close packed plane $(111)_\gamma$ and $(112)_{TiAl_3}$ is only about 1%, if γ-TiAl precipitates from $TiAl_3$, it should obey the orientation relationship of $(111)_\gamma // (112)_{TiAl_3}$. This relationship needs experimental evidence, but the rationality of this deduction can be proved in the transformation of $\gamma \rightarrow \alpha$. γ phase transforms into α phase according to the orientation relationship of $(0001)_\alpha // (111)_\gamma$ and $\langle 11\bar{2}0 \rangle_\alpha // \langle 110 \rangle_\gamma$ [17]. The misfit between the close packed plane $(111)_\gamma$ and $(0001)_\alpha$ is also only about 1%. Ref. [11] pointed out that the formation of $TiAl_3$ in Ti/Al diffusion couple obeys the relationship of $(112)_{TiAl_3} // (0001)_\alpha$. As a result, γ phase layer inherits the $(0001)_\alpha$ texture of the starting Ti foils. When the specimen is annealed at 850 °C, the α phase becomes unstable if the Al concentration in α-Ti exceeds the equilibrium concentration, and orders into α_2 phase with the same basal plane [18]. Obviously, α_2 phase layer holds the $(0001)_\alpha$ texture of the starting Ti foils. When heat treated at 1350 °C, α_2 and γ transform into α-Ti according to the relationship mentioned above, and the $(0001)_\alpha$ texture still exists at 1350 °C. A fully lamellar microstructure with a proper alignment of lamellae can be obtained when the sheet is cooled from 1350 °C. To reveal the importance of keeping α-Ti during the heat treatment, another experiment was performed. Alternate Ti/Al foils were hot pressed by the procedure of 640 °C, 12 h + 1000 °C, 10 h + 1350 °C, 2 h. A fully lamellar microstructure develops. The distribution of the angles between rolling direction and the lamellar interfaces is shown in Fig. 5(b). As seen in Fig. 5(b), the distribution is more random compared with Fig. 5(a). This is the result of the transformation of $\alpha \rightarrow \beta$ at 1000 °C, which weakens the $(0001)_\alpha$ texture.

Because Ti and Al foils have good ductility, TiAl sheet with complex shape can be fabricated by this method in the second procedure. Ti and Al foils with wavy shape are preformed, assembled alternately and hot pressed. Figure 7(a) shows a wavy sheet during a

thickness of 0.7 mm fabricated with the second procedure. Figure 7(b) shows the fully lamellar microstructure of the cross section of the sheet edge, there are few pores in the matrix. As can be seen in Fig. 7(b), the colony size is larger than that in Fig. 3(b), and this should be the result of the merger of adjacent colony with the same orientation.
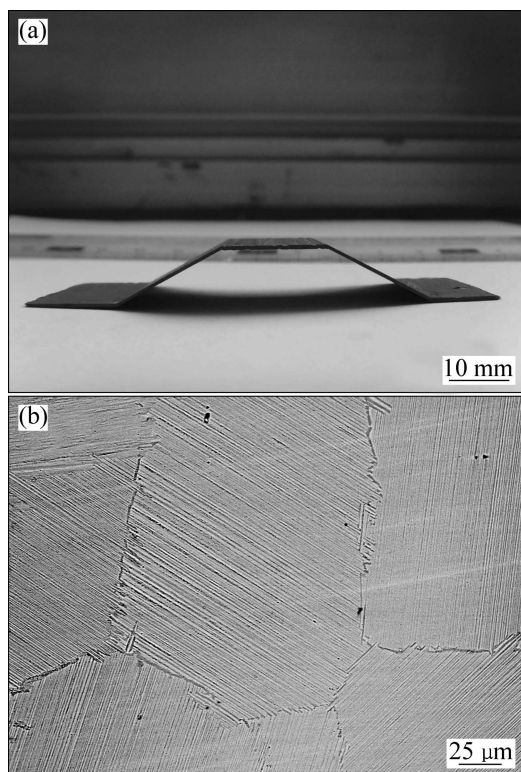

Fig. 7 Photo of wavy γ-TiAl sheet (a) and SEM image of cross section of wavy sheet edge (b)

4 Conclusions

1) A 0.7 mm-thick wavy γ-TiAl sheet with fully lamellar microstructure can be fabricated by hot pressing alternate Ti/Al foils. Most of the angles between the lamellar interface and the rolling direction are below 30°.

2) The thinner starting Ti and Al foils are good for yielding fine microstructures. This may contribute to the fabrication of complicated shaped γ-TiAl alloy based sheet with a particular lamellae orientation.

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箔冶金法制备 *γ*-**TiAl** 基波纹板

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摘 要：用热压逐层叠置的 Ti/Al 箔的方法制备具有全片层结构的 *γ*-TiAl 波纹板。热处理工艺为 640 °C，15 h + 850 °C，35 h + 1350 °C，2 h。用扫描电镜（SEM）和 X 射线衍射(XRD)分析热处理过程中产生的金属间化合物相。在 Al 箔完全消耗之前，Ti/Al 界面仅有 $TiAl_3$ 相生成。在 850 °C 热压 35 h 后，基体由 α-Ti, α_2-Ti_3Al, *γ*-TiAl 和 $TiAl_2$ 相组成。冷却得到全片层结构，大部分片层与板面的夹角小于 30°。采用较薄的原料箔可以获得相对细小的微观组织。

关键词：固态反应；相变；钛铝；箔冶金

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