MICROSTRUCTURES OF TiAl BASED ALLOYS PREPARED BY HOT PRESSING ELEMENTAL POWDERS①

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ABSTRACT Elemental Ti, Al powders were hot pressed in Ar atmosphere at various temperatures in order to produce Ti-48Al (%) alloy. The microstructure evolution has been studied in this article through optical microscopy, scanning electron microscopy, EDAX analysis, X-ray diffraction analysis and TEM observation. Results show that reaction between elemental Ti and Al is mainly controlled by diffusion. After hot pressing at 800 ℃, the reaction between Ti and Al powders is incomplete. The hot pressed microstructure mainly consists of Ti_3Al phase and TiAl phase. The higher the hot pressing temperature is, the higher the rate of reactive diffusion and the more complete the crystallization of TiAl and Ti_3Al will be. After hot pressing at 1300 ℃, the Ti_3Al phase is island like. TEM observation shows that the island like Ti_3Al phase and the TiAl matrix are all composed of fine grains. After hot pressing at 1400 ℃, the microstructure consists of large γ grains and coarse lamellar microstructure. Therefore, 1300 ℃ seems to be the premium temperature for reaction synthesis of TiAl based alloy.

Key words TiAl based alloy hot press powder metallurgy

1 INTRODUCTION

TiAl based alloys have been regarded as promising high temperature structure materials, for their high specific strength, rigidity and good high temperature properties[1-3]. However, their applications are hindered by their poor ductility, forgeability and castability. Many methods have been attempted to near net shape processing of TiAl based alloy, elemental powder metallurgy is one of them[4, 5].

The raw materials of elemental powder metallurgy are elemental Ti, Al powders, which are then normally mixed, pressed or extruded and reactively sintered. The reaction between elemental Ti, Al powders is a kind of self-propagation process, and is diffusion control. Owing to diffusivity and solubility difference, Al is far more active than Ti, and is the main diffusion element[6]. During the process, phase $TiAl_3$ and $TiAl_2$ will appear as intermediate product[6, 7]. Phase TiAl and $TiAl_2$ will grow at the expense of $TiAl_3$, after consumption of $TiAl_3$, the growth of TiAl at the expense of $TiAl_2$ and $TiAl_3$ can be described by a classical $\sqrt{t}$-law, where t is annealing time[5]. At equilibrium, Ti_3Al and TiAl will exist, while the ratio between them depends on composition and temperature.

Owing to Kirkendall effect and other reasons[8], pressureless reactively sintered TiAl alloy always have high porosity[8, 9], therefore during these process, pressure is usually necessary. In this article, hot pressing process is adopted to fabricate TiAl based alloy, and the microstructure evolution after different hot pressing condition are discussed in detail, while their porosity distribution and densification behavior had been discussed in other article[10].

2 EXPERIMENTAL

Elemental Ti, Al powders(< 45 μm) were mixed in a mill for 4 h, and the composition of the mixture was Ti-48Al(%), then the mixture

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was die pressed into approx *d* 18 mm × 30 mm cylindrical samples under conventional press at a pressure of 350 MPa. The compacts were then put into graphite die (as shown in Fig. 1) and hot pressed in an Ar atmosphere at temperature ranging from 800 ℃ to 1 400 ℃ for one or two hours. The heating rate was approx 100 ℃/min. After temperature reached 800 ℃, a pressure of 40 MPa was applied. The cooling rate was 40~ 60 ℃/min. The hot pressed samples were then polished and etched using Kroll's solution. Then the samples underwent optical observation, scanning electronic microscope observation, EDAX analysis and X-ray diffraction analysis. The samples of *d* 3 mm × 0.4 mm were spark machined from the hot pressed samples, then mechanically thinned and twin-jetted for TEM observation.

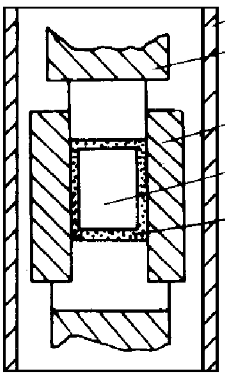

Fig. 1 Hot pressing unit of elemental Ti and Al powders

3 RESULTS

3.1 Outside appearance of hot pressed samples

Fig. 2 shows the outside appearance of hot pressed sample and compact. The height/diameter ratio of the compact is approx. 1.5, however, after large deformation, the hot pressed sample seems regular, no swelling and other apparent defects can be seen.

3.2 Microstructure of TiAl alloy hot pressed at 800 ℃

Fig. 3 shows the optical micrograph and scanning electron micrograph of TiAl alloy hot pressed at 800 ℃ for 2 h. Fig. 3(a) shows that particulate phases are dispersed in the network of porous reaction product of liquid Al and solid Ti. In Fig. 3(b), the microstructure can be more clearly seen, the particulate phase seems to be immerged in the porous matix, where many tiny pores can be seen; some particulate phase show multi-layered structure and precipitates. EDAX semi-quantitative analyses of the precipitate and surrounding area are shown in Table 1.

Fig. 2 Outside appearance of compact and hot pressed sample

Table 1 EDAX results of Fig. 3(b) (%)

Point	Ti	Al	S	Zr	Fe
A	78.86	12.38	6.74	1.57	0.46
B	84.57	15.43	–	–	–
C	73.05	26.95	–	–	–
D	43.55	56.45	–	–	–
E	78.83	21.17	–	–	–

* Every point is the mean value of several similar points.

The EDAX analysis of point *A*, which is on the precipitates, exhibits impurities of S, Zr, Fe, while other points show no such impurities. Point *B* is in the center of the particulate phase, which is a small area between the precipitates; point *C* is at the edge of the particulate phase; point *D* is in the matrix; and point *E* is in the center of another particulate phase. According to Ti-Al phase diagram, point *B* is in α-Ti area; point *C* is in Ti_3Al area; point *E* is at the edge of α-Ti area and point *D* is in TiAl area. Therefore, the matrix mainly consists of TiAl phase,

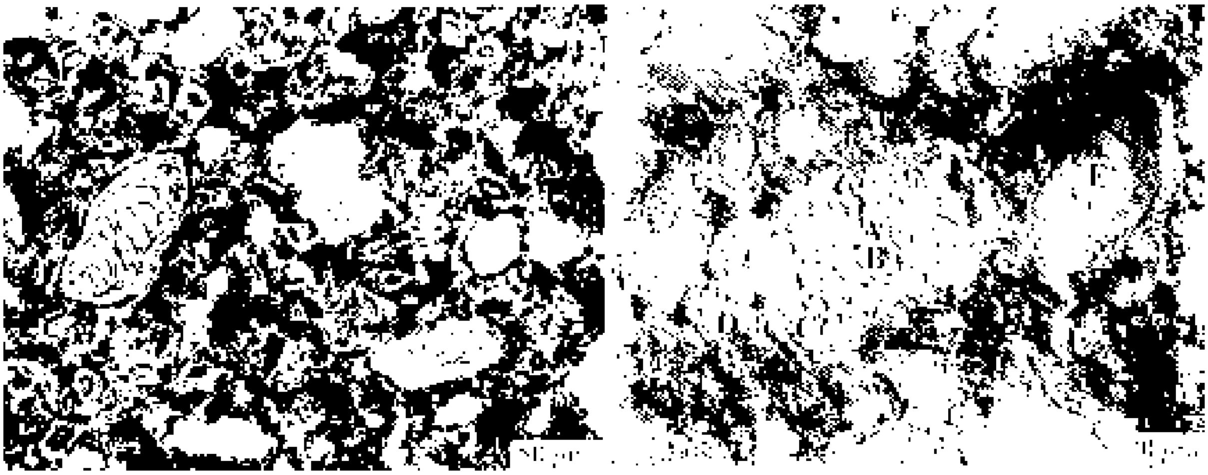

Fig. 3 Microstructure of TiAl alloy after hot pressed at 800 ℃ for 2 h
(a) —Optical micrograph; (b) —SEM(SEI)

while the particulate phase mostly consists of α-Ti and Ti_3Al phases, with the center of them is Al-rich α-Ti. It reveals that at this temperature the reaction between Ti, Al powders is not complete, α-Ti remains and in some of them the impurity elements concentrate to form precipitates.

3.3 Microstructures of TiAl alloy hot pressed at other temperatures

Fig. 4 shows the optical micrographes of TiAl alloy hot pressed at 900, 1 000 ℃ for 2 h and 1 200, 1 300, 1 400 ℃ for 1 h respectively.

At 900 ℃ and 1 000 ℃ (Fig. 4(a), 4(b)), the size and amount of precipitates seems decreased. At 1 200 ℃ and 1 300 ℃, the Ti_3Al phase agglomerates to island like. The TiAl alloy underwent much larger deformation at 1 200 ℃ and 1 300 ℃ during hot pressing, most deformation would be realized through the flow of TiAl matrix and deformation of Ti particles, some of which would agglomerate during this process and transform to Ti_3Al phase after reactive diffusion. The island like Ti_3Al phase had been reported by other researchers[11]. In the meantime, large amount of grains and twins appear. At 1400 ℃, the microstructure of TiAl alloy consists of coarse γ grains (20~ 50 μm) and lamellar microstructure α_2+ γ(100~ 200 μm). It indicates that the reactive diffusion has approached equilibrium at that temperature, but the coarsening effect of microstructure is serious.

EDAX semi-quantitative analyses show the composition of island like Ti_3Al particulates and TiAl matrix. However, the Ti content of Ti_3Al phase drops and the Al content rises, it seems that the composition of the alloy becomes more homogeneous as hot pressing temperature rises, as shown in Table 2.

Table 2 Composition of Ti_3Al phase and TiAl matrix in various hot pressed samples(%)

Samples		Ti	Al
1 000 ℃, 2 h	Ti_3Al	71.69	28.31
	TiAl	50.22	49.78
1 200 ℃, 1 h	Ti_3Al	60.73	39.27
	TiAl	48.83	51.17
1 300 ℃, 1 h	Ti_3Al	55.47	44.53
	TiAl	45.22	54.78

* Every point is the mean value of several similar points.

3.4 X-ray diffraction analyses of hot pressed samples

Fig. 5 shows the XRD patterns of TiAl based alloys hot pressed at 800 ℃ (TA1) and 1 300 ℃(TA2). The diffraction peaks of TA1 show broadening effect, while that of TA2 are sharp and clear. TA1 mainly consists of Ti_3Al and TiAl phases, and the peaks of α-Ti and other intermediate phases are mostly covered by that of above two main phases and the background. Although the main phases of TA2 are similar to that of TA1, There is a small unknown peak at about 41°, which needs further investigation.

3.5 TEM observation of hot pressed samples

Owing to difficulty in preparing of P/M samples, only limited area and diffraction patterns in hot pressed samples can be seen, but some of the results are still useful.

Fig. 6(a) shows the island like Ti_3Al phase in sample hot pressed at 1200 ℃, it can be seen that the island like Ti_3Al are composed of equiaxed Ti_3Al grains (diffraction pattern shown in Fig. 6(d)) with straight grain boundaries. Fig. 6(b) shows fine, equiaxed TiAl grains with

Fig. 4 Optical micrographs of TiAl alloy hot pressed at various temperatures
(a) —900 ℃, 2 h; (b) —1000 ℃, 2 h; (c) —1200 ℃, 1 h; (d) —1300 ℃, 1 h; (e) —1400 ℃, 1 h

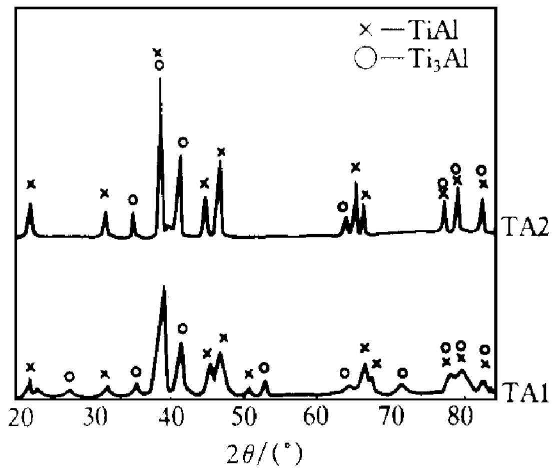

Fig. 5 XRD patterns of TiAl-based alloys

size of several micrometers in the matrix of the same sample (diffraction pattern shown in Fig. 6 (d)).

Fig. 6(e) indicates the lamellar structure in sample hot pressed at 1400 ℃. Large amount of dislocations can be seen in the α_2/γ boundary and a thin γ lamellae with high dislocation density was nucleating in α_2 lamellea.

4 DISCUSSION

The reaction between Ti, Al elements is diffusion controlled, including the formation of such intermediate products as $TiAl_3$ and $TiAl_2$, although they have not been confirmed in this work. According to EDAX analysis of the material hot pressed at 800 ℃ for 2 h, elemental Ti and Al are not fully reacted. The center of the former Ti particle is α-Ti solid solution with high content of Al, the edge of the particle is Ti_3Al phase, and the outside is porous Al-rich TiAl phase. At reaction temperature above the melting point of Al, a porous layer of $TiAl_3$ formed after intensive reaction between liquid Al and solid Ti[11]. The $TiAl_3$ layer was disintegrated due to the penetration of transient liquid Al[8, 9] and outside pressure, resulting in a structure of remaining Ti particle (α-Ti+ Ti_3Al) and network of porous $TiAl_3$. In subsequent annealing, $TiAl_3$ phase was consumed and finally transformed to TiAl phase, however, the porous network structure was inherited because of insufficient densification behavior. Although intermediate phases can not be seen clearly in XRD patterns (Fig. 5), it can be deduced that they exist after hot pressing at this temperature because their peaks are covered by the broad background and broad TiAl, Ti_3Al peaks. Therefore, at this temperature and annealing time, the reaction between Ti, Al elements is incomplete.

At higher reaction temperature, the diffusion of Ti, Al elements becomes faster. On the other hand, as temperature rises, the material becomes more ductile and densification process proceeds more completely with higher degree of deformation and elimination of pores, hence the diffusion distance between Ti_3Al phase and TiAl phase is shortened. Hot pressing at 1 200 ℃, 1 300 ℃ for only 1h will lead to a more fully reacted microstructure than that at 1 000 ℃ for 2 h. Furthermore, the crystallization of Ti_3Al and TiAl phases becomes more complete, i. e. their diffraction peaks appear sharper. Therefore 1 300 ℃ will be an appropriate temperature for reaction synthesis of TiAl based alloy, because at a higher temperature of 1 400 ℃, microstructure coarsening happens.

5 CONCLUSIONS

(1) After hot pressing at 800 ℃, the reaction between Ti, Al powders is incomplete, multi-layered structure and precipitates may appear in the former Ti particles, moreover, the crystallization of Ti_3Al and TiAl grains is incomplete.

(2) After hot pressing at 1 300 ℃ for 1h, the microstructure of the sample shows island like Ti_3Al phase and TiAl matrix, both of which consist of fine grains under TEM observation. As hot pressing temperature continues rising to 1 400 ℃, microstructure coarsening of TiAl based alloy happens, therefore 1 300 ℃ seems to be the premium temperature for reaction synthesis of TiAl based alloy.

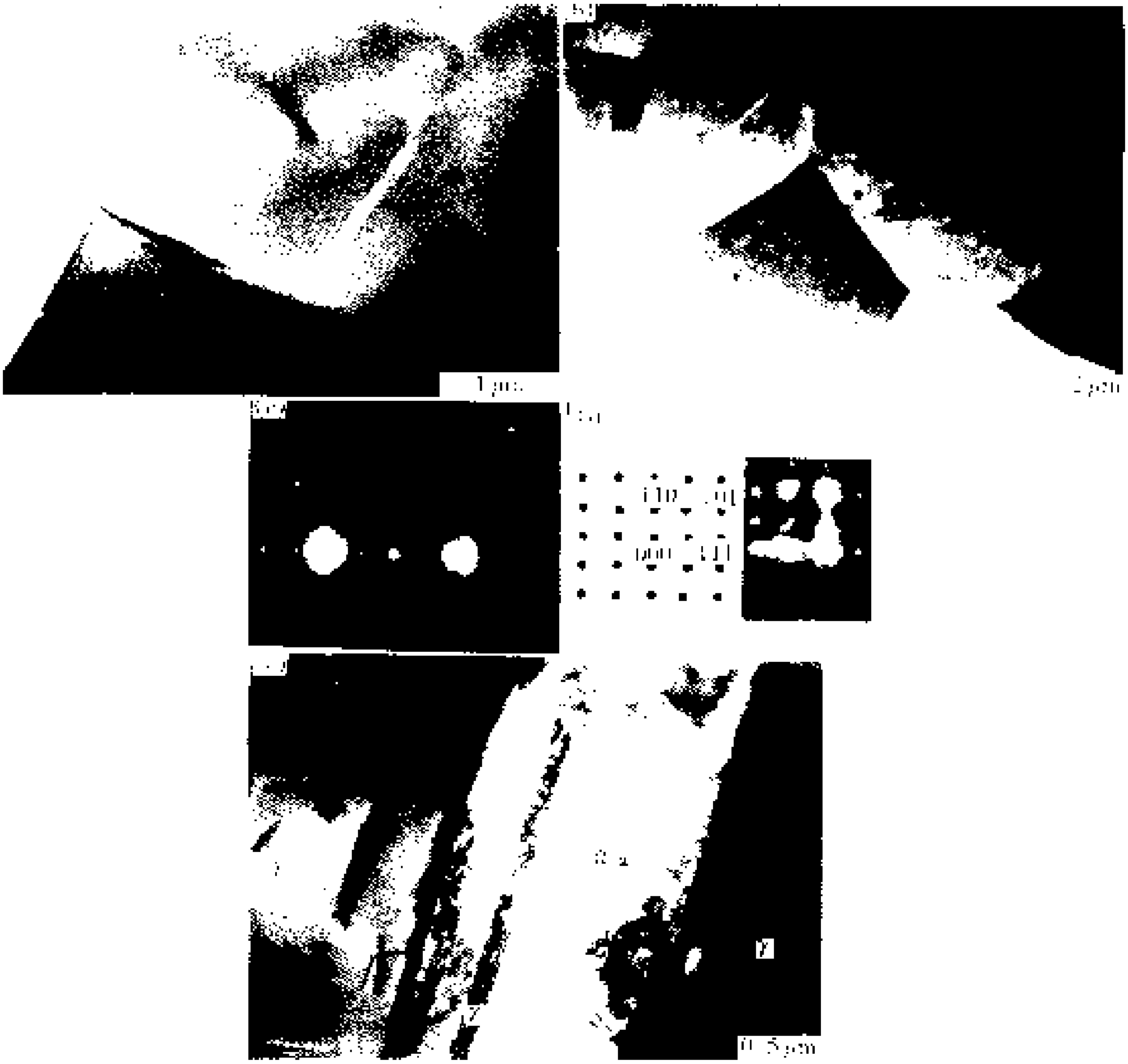

Fig. 6 TEM observation of typical microstructure in hot pressed samples
(a), (c) —Ti_3Al; (b), (d) —TiAl grains; (e) —lamellar structure

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