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Effects of the variation in α-phase volume fraction on the thermal stability of TiAl alloys with a lamellar microstructure

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The thermal stability of two-phase ($\alpha + \gamma$) lamellar microstructure in Ti-Al-Mo PST (polysynthetically twinned that has single colony) crystals, containing C or Si, was investigated. In addition, the variation of α-phase volume fraction in Ti-Al-Mo-(C,Si) systems was investigated at several temperatures. Ti-46Al-1.5Mo-0.2C and Ti-46Al-1.5Mo-1.0Si alloys did not recrystallized ('stable' in this paper) during heat treatments at various heating rates and temperatures. Moreover, the α-phase volume fractions of Ti-46Al-1.5Mo-0.2C and Ti-46Al-1.5Mo-1.0Si alloys which were stable compositions, changed less than those of Ti-47Al and Ti-46Al-1.5Mo alloys which were unstable compositions. The instability of the latter alloys was caused by their relatively higher variation of α-phase volume fraction during heating. Therefore, it is suggested that the variation of α-phase volume fraction is an important factor in controlling the thermal stability of lamellar microstructure. © *2004 Kluwer Academic Publishers*

1. Introduction

Alloys based on the intermetallic compound TiAl are candidate materials for high temperature aerospace applications because of their low density, good high-temperature strength and acceptable oxidation resistance [1–3]. If the mechanical properties, particularly the combination of room temperature ductility and strength, are further improved, TiAl-based alloys would be suitable for use in a variety of structural applications.

Previous studies on PST (polysynthetically twinned) TiAl alloys [2, 3] have indicated that a good combination of yield stress and ductility at room temperature can be achieved when the lamellar orientation is aligned parallel to the tensile direction (A direction). There are two ways to control the lamellar orientation in the DS process: using a seed crystal [4–8] and modifying the solidification procedure [9–11]. However, in the latter method, it is difficult to control the lamellar orientation because the probability of obtaining an ingot A lamellar orientation is only one-third due to the relationship between the α phase and the β phase [9–11]. Therefore, methods using the seed alloy are considered more appropriate to control the lamellar orientation.

In DS process using a seed crystal, thermal stability of the lamellar microstructure of the seed alloy is strongly required. If the seed alloy does not have thermal stability until the melting point during heating, recrystallization may occur and change the initial lamellar orientation [7, 8, 12]. Thus, the lamellar orientation of the growing crystal will be different from the A orientation. Moreover, creep resistance can be degraded noticeably by recrystallization, thus, it is very important to investigate the cause of such recrystallization [13]. Furthermore, in order to improve the mechanical properties of TiAl alloys by controlling lamellar spacing, the lamellar orientation must be preserved. Therefore, recrystallization of the lamellar microstructure must be restrained [14].

The thermal stability of lamellar microstructure of TiAl alloys was investigated by partial melting [4–6] using an Optical Floating Zone Furnace (FZ furnace). Ti-46Al-1.5Mo-0.2C (at.%) alloy, which can be used as a seed material, did not recrystallize after partial melting (i.e., on re-solidification, the original crystal orientation is preserved) (Fig. 1b) [4]. However, binary TiAl alloys are not suitable as seed alloys because they produce new grains after partial melting (Fig. 1a) [4].

The purpose of this study is to investigate the factors that affect the thermal stability of TiAl lamellar microstructure. Therefore, we aim to verify the existence of other factors, excluding the decline in thermal stability of lamellar microstructure due to the relationship

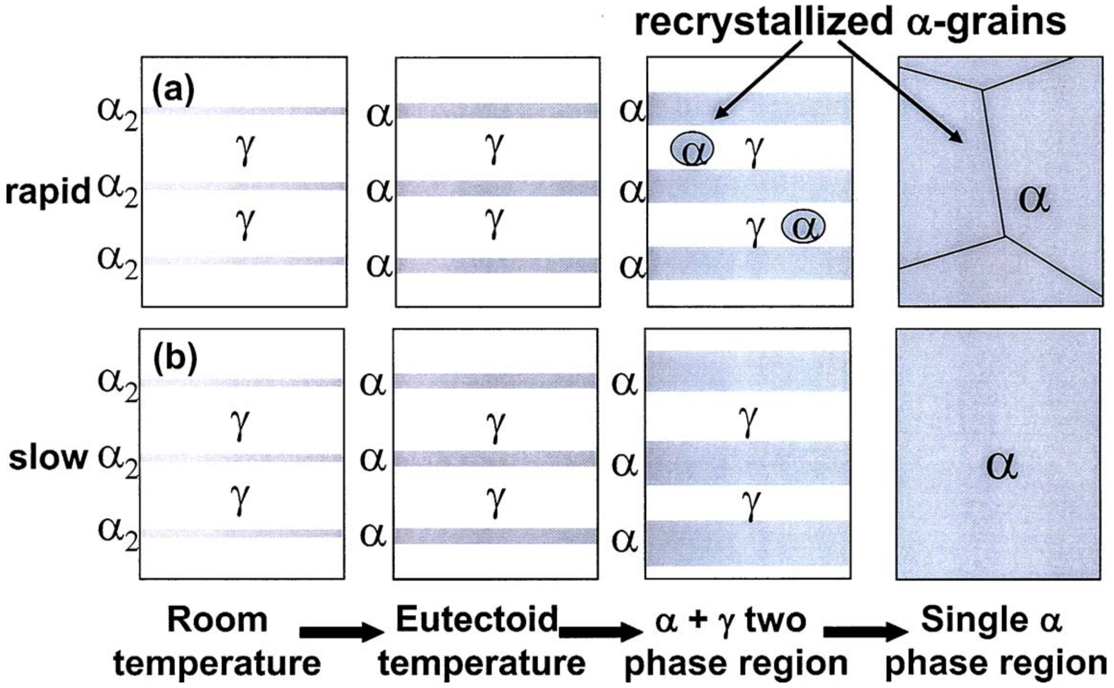

Figure 1 Schematic diagram of the change in volume fraction of constituent phases during: (a) rapid heating and (b) relatively slow heating.

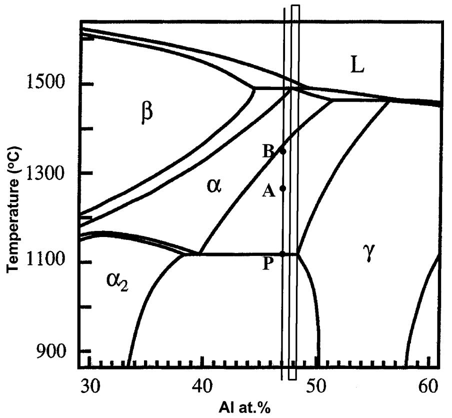

Figure 2 Partial phase diagram of Ti-Al binary system near the stoichiometric TiAl composition.

between the β phase and α phase or the γ phase and α phase [4–8, 12]. According to previous studies [4–8, 12], alloys which do not pass the β single-phase region and $(\beta + \alpha)$ 2-phase region, or γ single-phase, is thermally stable during cooling and heating. Examination of the TiAl binary phase diagram (Fig. 2) shows the existence of a composition region that does not pass β or γ single phase region (Ti-(47.6–48.2)Al). However, TiAl binary alloys are not suitable as seed alloys because recrystallization occurs after the partial melting [4]. Therefore, it can be suggested that there are factors besides the phase relationship.

The factors that affect thermal stability after heat treatment were examined by using unstable (Ti-47Al, Ti-46Al-1.5Mo; unstable composition) [4] and stable (Ti-46Al-1.5Mo-0.2C, Ti-46Al-1.5Mo-1.0Si; stable composition) alloys [4, 5]. In order to confirm the recrystallization, PST crystals of each alloy were used for heat treatment. Thermal stability according to heating rate was investigated, and the α-phase volume fraction in the 2-phase $(\alpha + \gamma)$ region was examined to confirm the effects on the thermal stability of the lamellar microstructure.

2. Experimental procedures

The alloys used in this study were manufactured by quantifying 99.99 wt% Ti, 99.99 wt% Al, 99.9 wt% Mo, 99 wt% TiC powder, and 99.99 wt% Si. Button ingots, approximately 20 g in mass each, were made by vacuum arc melting under an argon atmosphere and remelted more than five times to promote homogeneity. The feeder ingots used during the DS process were manufactured into 14 ϕmm × 100 mm cylinder type by re-melting the arc melting buttons.

PST crystals were manufactured by carrying out the DS process with 5 mm/h growth rate in a FZ furnace. Then, PST crystals were cut to 5 × 5 × 3 mm^3 to manufacture heat treatment specimens. In order to evaluate the thermal stability of the lamellar microstructure, heat treatments were carried out at 1360 and 1420°C for 1 h at a heating rate of 6°C/min, and at 1360°C for 1 h by rapid heating. Heat treatment by rapid heating was performed by quickly placing specimens into a furnace maintained at 1360°C. In addition, to measure the α-phase volume fraction of stable and unstable compositions in the $(\alpha(\alpha_2) + \gamma)$ 2 phase region, heat treatments at 1050, 1150, and 1250°C for 168 h, then water quenching were performed. Furthermore, to measure α-transus temperature, the arc-melted buttons were homogenized at 1200°C for 24 h and heat treatments at temperatures near 1350°C at intervals of 10°C for 6 h were carried out.

After the heat treatment, a scanning electron microscope (SEM) equipped with an energy-dispersive spectrometer (EDS) and an optical microscope (OM) was used to characterize the microstructures. A solution of

5% HF – 5% HNO_3 – 90% H_2O by volume was used to perform etching. An image analyzer was used to calculate the volume fraction of each phase that was observed in the back-scattered (BS) images using SEM.

3. Results and discussion

3.1. Recrystallization behavior of stable and unstable compositions

In our previous studies [4–6], the thermal stability of the lamellar microstructure of TiAl-Mo-(C, Si) alloys was confirmed by partial melting in a FZ furnace. In this study, the important factors determining the thermal stability of lamellar microstructure of TiAl-Mo-(C, Si) alloys were investigated. The thermal stability of the lamellar microstructure of each specimen was examined by carrying out heat treatments of stable and unstable compositions. Heat treatments were not carried out at high temperatures near melting point, but at temperatures within the range of 1250 to 1420°C. Moreover, in order to clearly determine the recrystallization during heat treatment, PST crystals were used for heat treatment. Ti-47Al, Ti-46Al-1.5Mo, Ti-46Al-1.5Mo-0.2C, and Ti-46Al-1.5Mo-1.0Si alloys were used for heat treatments of (1360°C, 1420°C)_(1 h)_(F. C.)_(6°C/min) (that is: temperature_holding time_cooling rate_heating rate). In Table I, the four alloys are marked with X if recrystallization occurred after the heat treatment or with O if recrystallization did not occur. In addition, the results of the partial melting of the alloys from the previous study are included in Table I [4, 6].

TABLE I The result of partial melting and heat treatment of Ti-47Al, Ti-46Al-1.5Mo, Ti-46Al-1.5Mo-0.2C and Ti-46Al-1.5Mo-1.0Si alloys. (temperature_holding time_heating rate, All alloys were furnace cooled after heat treatments)

	Partial melting [4, 6]	1360°C _1 h_6°C/min	1420°C _1 h_6°C/min
Ti-47Al	X	O	X
Ti-46Al-1.5Mo	X	O	O
Ti-46Al-1.5Mo-0.2C	O	O	O
Ti-46Al-1.5Mo-1.0Si	O	O	O

(O: not recrystallized, X: recrystallized).

For Ti-46Al-1.5Mo-0.2C and Ti-46Al-1.5Mo-1.0Si alloys which are stable compositions, recrystallization did not occur after the partial melting and heat treatment at 1360 and 1420°C. However, Ti-47Al, which is an unstable composition [4], was stable at 1360°C, but was unstable at 1420°C. Fig. 3 shows lamellar microstructures of Ti-47Al and Ti-46Al-1.5Mo by OM after heat treatment at (1420°C)_(1 h)_(F. C.)_(6°C/min). As shown in Fig. 3, when heat treated at 1420°C, in the case of Ti-46Al-1.5Mo (Fig. 3b), recrystallization did not occur. However, when Ti-47Al alloy was heat treated at 1420°C, surface grains formed and grew into specimens (Fig. 3a). Results from Table I and Fig. 3 do not clearly show the difference between the stable and unstable compositions. Although the unstable compositions were recrystallized during partial melting, their lamellar orientations were almost preserved when heat treated at a slow heating rate. Therefore, in order to investigate factors affecting the thermal stability, a fast heating rate was used for heat treatment to create an environment similar to that of the partial melting process to compare the thermal stability of stable and unstable compositions.

Fig. 4 shows the result of the heat treatment of (1360°C)_(1 h)_(F. C.)_(rapid heating). For Ti-47Al (Fig. 4a) alloy, the original lamellar orientation completely disappeared after heat treatment. For Ti-46Al-1.5Mo (Fig. 4b) alloy, about 30% of the original lamellar microstructure remained after heat treatment. In Fig. 4b, the grain on the right side shows the preserved lamellar orientation after heat treatment and the left side shows the changed lamellar orientation. Thus, among the unstable compositions that cannot be used as seed alloys, the thermal stability of the lamellar microstructure is different. Moreover, the lamellar microstructure of Ti-46Al-1.5Mo alloy was considered to be more stable than that of the Ti-47Al alloy. In addition, the lamellar microstructures of Ti-46Al-1.5Mo-0.2C (Fig. 4c) and Ti-46Al-1.5Mo-1.0Si (Fig. 4d) alloys before the heat treatment were preserved. Fig. 4c and d show bending of the lamellar boundary direction. However, the bending was not caused by the heat treatment, but rather it was induced during the DS process [4, 6, 8] because the lamellar orientations before and after the heat treatment did not show any changes. When this result is compared with Table I and Fig. 3, it was found that as

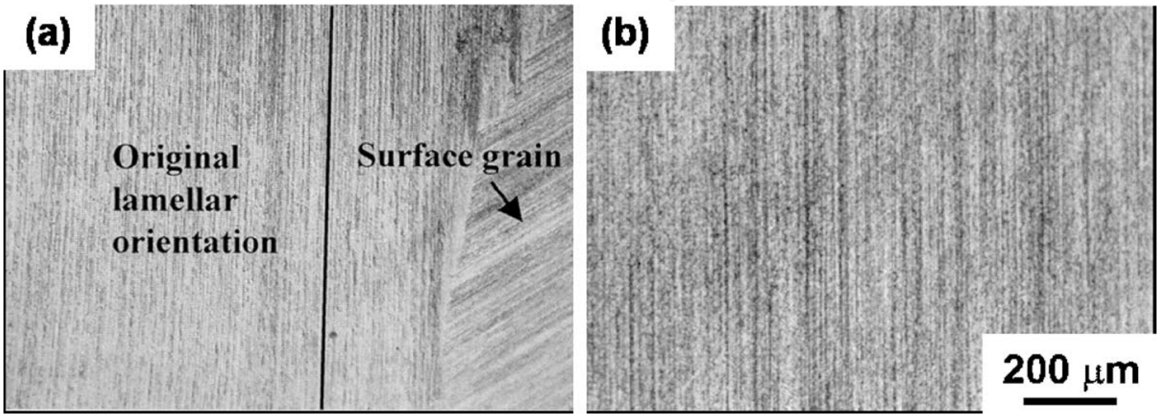

Figure 3 Optical microstructures of (a) Ti-47Al and (b) Ti-46Al-1.5Mo alloys after heat treatment at 1420 for 1 h at a heating rate of 6°C/min.

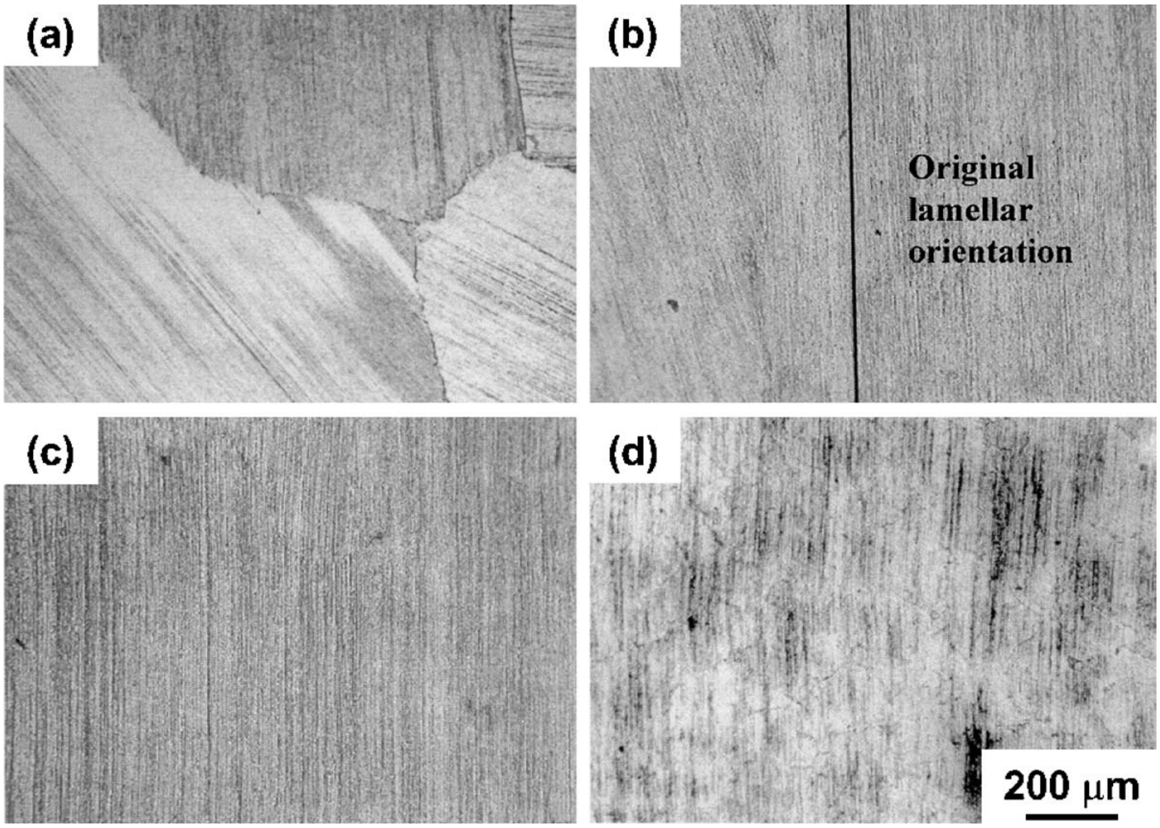

Figure 4 Optical microstructures after heat treatment at 1360°C for 1 h by rapid heating: (a) Ti-47Al, (b) Ti-46Al-1.5Mo, (c) Ti-46Al-1.5Mo-0.2C, and (d) Ti-46Al-1.5Mo-1.0Si, respectively.

the heating rate increased, the tendency of recrystallization of unstable alloys also increased.

3.2. The difference in the recrystallization behavior according to the heating rate

When the Ti-47Al, Ti-46Al-1.5Mo alloys were heat treated by rapid heating even with equal temperature and holding time, the recrystallization tendency increased (Table I, Fig. 4a and b). In order to ascertain this tendency more clearly, Ti-47Al alloy which was considered to be the most unstable composition, was heat treated by varying the heating rate. Fig. 5 shows the microstructure after heat treatment by the following steps: the put-in-times to the furnace were varied among 5, 30, and 60 min, and then held for 1 h, and furnace cooled. It was found that as the heating rate decreased, the grain size grew and the recrystallization tendency decreased. In particular, in Fig. 5c, where the heating rate was the slowest, only surface grains formed. Moreover, smaller grains and larger number of grains could be observed when the specimen was immediately put in (Fig. 4a), thus, it can be considered that as the heating rate increased, the recrystallization tendency also increased.

In order to investigate the effect of the heating rate on the recrystallization, it is necessary to determine the phase region of heat treatment temperatures. Thus, the α-transus temperature of each alloy was determined. In order to determine the α-transus temperature of each composition, arc-melted buttons were homogenized at 1200°C for 24 h, and temperatures in the vicinity of 1350°C with intervals of 10°C were selected and held for 6 h followed by furnace cooling. In Table II, the α-transus temperature of each alloy is classified. Table III shows the phase regions of each alloy composition at 1360 and 1420°C, based on the α-transus temperature, TiAl binary phase diagram, and the TiAlMo ternary phase diagram [15]. The heat treatment temperatures (1360 and 1420°C) are in the $(\alpha + \gamma)$ 2-phase region or the α single-phase region.

When the PST crystals are heated from room temperature to the α single-phase region or the $(\alpha+\gamma)$ 2-phase region, the α/γ volume fraction should be controlled by the change of width and number of the plates in the lamellar microstructure as shown in Fig. 1. Under the

TABLE II The variation of α-phase volume fraction at 1050, 1150, 1250, 1350°C and α-transus temperature of Ti-47Al, Ti-46Al-1.5Mo, Ti-46Al-1.5Mo-0.2C and Ti-46Al-1.5Mo-1.0Si alloys

	1050°C	1150°C	1250°C	1350°C	α-trans. temp.
Ti-47Al	12.0%	10.9%	42.9%	96.8%	1370°C
Ti-46Al-1.5Mo	32.3%	37.9%	60.1%	100%	1320°C
Ti-46Al-1.5Mo-0.2C	22.4%	31.0%	69.3%	100%	1340°C
Ti-46Al-1.5Mo-1.0Si	26.4%	36.9%	75.0%	95%	1400°C

TABLE III Phase region of Ti-47Al, Ti-46Al-1.5Mo [15], Ti-46Al-1.5Mo-0.2C and Ti-46Al-1.5Mo-1.0Si alloys at 1360 and 1420°C

	1360°C	1420°C
Ti-47Al	$(\alpha + \gamma)$ region	α region
Ti-46Al-1.5Mo [15]	α region	α region
Ti-46Al-1.5Mo-0.2C	α region	α region
Ti-46Al-1.5Mo-1.0Si	$(\alpha + \gamma)$ region	α region

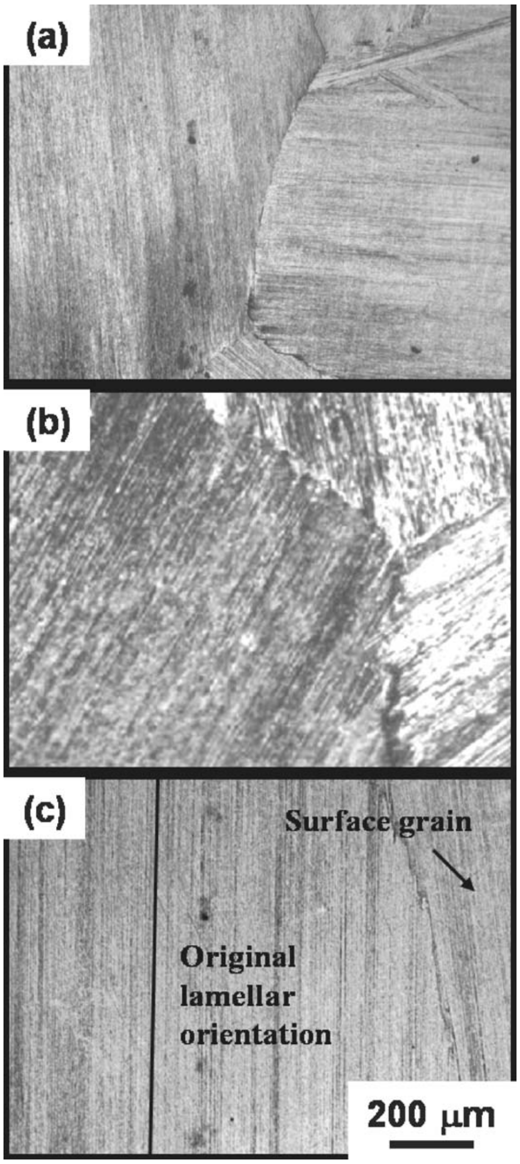

Figure 5 Optical microstructures of Ti-47Al alloy after heat treatment at 1360 for 1 h according to the duration input time: (a) 5 min, (b) 30 min, and (c) 1 h, respectively.

slow heating rate (Fig. 1b), the original lamellar microstructure was preserved since the α/γ volume fraction change could be controlled by the width and number of lamellar plates. However, under the fast heating rate (Fig. 1a), new α grains could be easily created inside the γ lamellae because there was insufficient time to fit the α/γ volume fraction to the equilibrium volume fraction [16].

3.3. Effects of α-phase volume fraction on the thermal stability of lamellar microstructure

From the results in Table I and Figs 3 and 4, the lamellar microstructure of the Ti-47Al alloy was found to be thermally unstable. This effect was explained by the decrease in thermal stability because of phase relationships [4–8, 12] as already discussed in our introduction. However, because the aluminum content at the end-point of the γ single phase region at the eutectoid temperature is higher than that of the $L + \beta \rightarrow \alpha$ peritectic reaction, alloys of composition Ti-(47.6–48.2)Al can be considered to be stable (Fig. 2). Therefore, it was concluded that there are other factors excluding the decrease in thermal stability by phase relationship that could affect the thermal stability of the lamellar microstructure. When the heating rate was increased, rapid recrystallization occurred in unstable compositions (Fig. 4a and b). Because the α-phase volume fraction change was very large at the heat treatment temperature of 1360°C, the lamellar microstructure could not be maintained. Moreover, the amount of recrystallization increased when compared to the stable composition. When heated to high temperatures, as illustrated in Fig. 1, in order to maintain the lamellar microstructure, the α-phase volume fraction change must be controlled by the number and width of the lamellar plates by using the diffusion of Ti and Al atoms. Therefore, it seems preferable to have a high α_2-phase volume fraction at room temperature and little change in α-phase volume fraction.

Fig. 6 shows the microstructures after heat treatment of Ti-47Al alloy. Heat treatment was carried out at

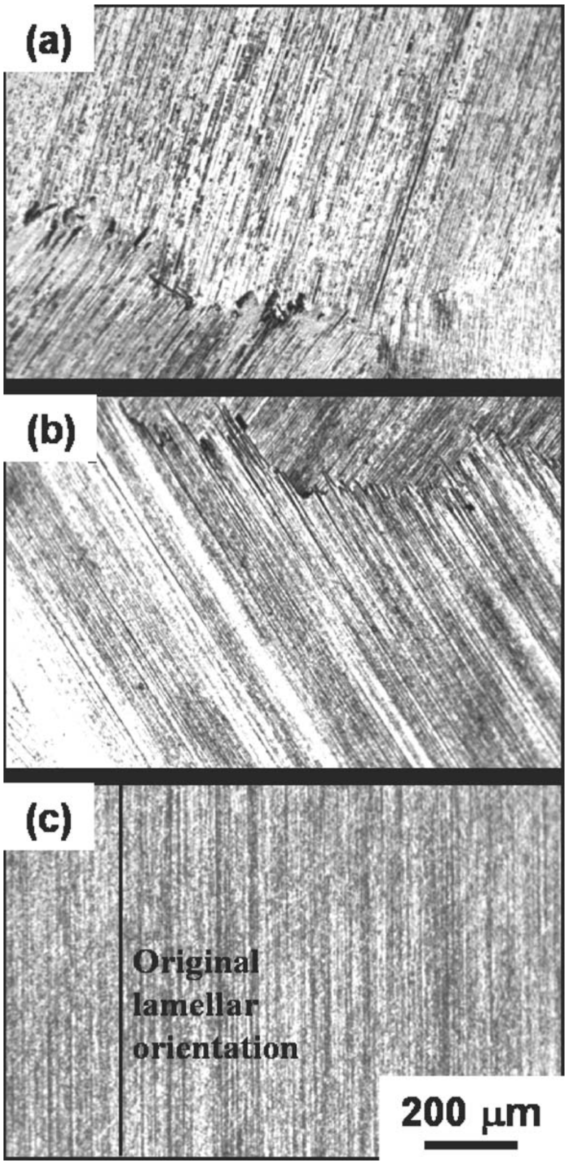

Figure 6 Optical microstructures of Ti-47Al alloy after heat treatment for 1 h by rapid heating at (a) 20°C higher, (b) 20°C lower, and (c) 100°C lower than α-transus temperature (1370°C).

temperatures 20°C higher, 20°C lower, and 100°C lower than the α-transus temperature (1370°C). Then, the alloys were held for 1 h, and finally furnace cooled. Recrystallization occurred only by heat treatment at temperatures 20°C higher (Fig. 6a) and 20°C lower (Fig. 6b) than the α-transus temperature. This indicates that the thermal stability of the lamellar microstructure rapidly diminished at temperatures near the α-transus temperature because the α-phase volume fraction within this temperature range changed rapidly, as demonstrated by computing the α-phase volume fraction of the Ti-47Al alloy on the TiAl binary phase diagram (Fig. 2). The α-phase volume fractions at the eutectoid temperature P (1107°C), heat treatment temperature A (1270°C), and B (1350°C) are 15.7, 60.7, and 92.1 vol% respectively. Considering the difference in temperature between points P and A, A and B, the α-phase volume fraction change between points A and B was greater. This is considered to be the reason for rapid recrystallization during heat treatment.

In order to experimentally examine the effect of the change in α-phase volume fraction on the thermal stability of lamellar microstructure, the volume fraction of the α and γ phases of each alloy at 1050, 1150, and 1250°C were determined. The Ti-47Al, Ti-46Al-1.5Mo, Ti-46Al-1.5Mo-0.2C, and Ti-46Al-1.5Mo-1.0Si alloys were used for heat treatments of (1050, 1150 and 1250°C)_(168 h)_(W. Q.)_(6°C/min). Calculation of the α-phase volume fraction was performed by computing the ratio of the bright grey region in SEM image to the total region using an image analyzer. When B2 phase, carbides, or silicides were present, those regions were excluded and the remaining region was taken as 100%. Table II shows the calculated α-phase volume fraction values for each composition. The values of α-phase volume fraction at 1350°C are calculated approximately using the α-transus temperature of each alloy. From the table, the results for the Ti-47Al alloy were very close to those calculated using the lever rule from the Okamoto [17] phase diagram. The results of Table II are illustrated in Fig. 7. Considering these results, the α-phase volume fraction of unstable Ti-47Al alloy at 1050°C was lower than that of stable Ti-46Al-1.5Mo-0.2C and Ti-46Al-1.5Mo-1.0Si alloys, and the α-phase volume fraction changed rapidly between 1150 and 1350°C. Therefore, this change in volume fraction can initiate recrystallization of the lamellar microstructure (Fig. 4a). For the Ti-46Al-1.5Mo alloy, the α-phase volume fraction at 1050°C was relatively high compared to stable alloys. However, at temperatures below 1360°C (1250–1350°C) at which recrystallization was considered to occur (Fig. 4b), the α-phase volume fraction rapidly increased as in the case of the Ti-47Al alloy. Because of this rapid change in the α-phase volume fraction, recrystallization occurred during heat treatments by rapid heating. The α-phase volume fraction of stable alloys at 1050°C was relatively high, and even at high temperatures there was no region where the α-phase volume fraction changed rapidly. Therefore, the lamellar

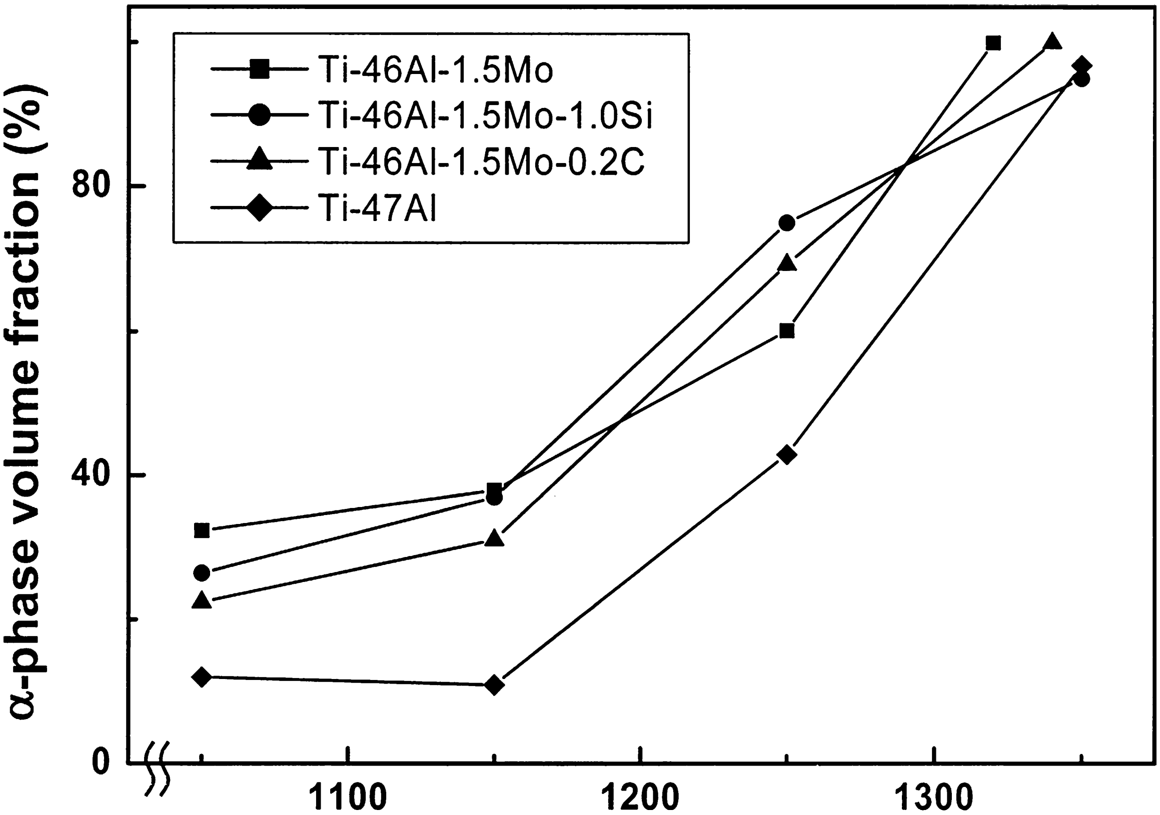

Figure 7 Variation of the α-phase volume fraction with various heat treatment temperatures in Ti-47Al, Ti-46Al-1.5Mo, Ti-46Al-1.5Mo-0.2C, and Ti-46Al-1.5Mo-1.0Si alloys, respectively.

microstructure could easily be maintained. For the Ti-46Al-1.5Mo alloy, when partially melted at high temperature, recrystallization could occur because of the phase relationship when α-phase was formed from β-phase. This can also be considered to be a factor that decreases the thermal stability of the Ti-46Al-1.5Mo alloy as indicated in Table I. It can be concluded that in order to have a thermally stable lamellar microstructure, the β single-phase or $(\beta+\alpha)$ 2-phase regions must not be passed during cooling, and the change in α-phase volume fraction must be very small during heating or cooling.

The criterion for determining the thermal stability of the lamellar microstructure in this study was whether the lamellar orientation was maintained during heating and cooling. However, as mentioned above, when carrying out creep tests or when improving the mechanical properties by adjusting the lamellar spacing, thermal stability of the microstructure is required. Recently, it has been found that during creep tests, precipitates such as carbide [14, 19] or silicide [20, 21] impede the movement of dislocations and improve the thermal stability of the lamellar microstructure. Therefore, precipitates such as carbide or silicide can improve the thermal stability of the lamellar microstructure.

In this study, factors influencing the thermal stability of the lamellar microstructure were investigated. It was found that in order to have thermally stable lamellar microstructures, the β single-phase or $(\beta + \alpha)$ 2-phase region must not be passed during cooling, and the change in α-phase volume fraction must be very small during heating or cooling. In future work, we will investigate the occurrence of recrystallization when alloys pass through the β-single phase or γ-single phase regions because of their phase relationships.

4. Conclusions

The important factors that can influence the thermal stability of TiAl with a lamellar microstructure were investigated. The results of this research are summarized as follows:

1. It has been verified that Ti-46Al-1.5Mo-0.2C and Ti-46Al-1.5Mo-1.0Si alloys that are stable compositions, did not recrystallize at all heat treatments.
2. In the case of unstable alloys, recrystallization occurred rapidly when heat treated by rapid heating. This was because there was insufficient diffusion time for the volume fraction to reach equilibrium since the $\alpha \leftrightarrow \gamma$ transformation was impeded. This implied that the change in α-phase volume fraction was controlled by the formation and growth of new α grains, not the change of number and thickness of the lamellae.
3. In the $(\alpha + \gamma)$ 2-phase region, when the α-phase volume fraction changed rapidly, the thermal stability of the lamellar microstructure can be decreased. This was confirmed as the important controlling factor of thermal stability of lamellar microstructures.

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