Microstructural refinement mechanism by controlling heating process in multiphase materials with particular reference to γ-TiAl

Hanliang Zhu, a) K. Maruyama, and J. Matsuda

Graduate School of Environmental Studies, Tohoku University, 02 Aobayama, Sendai 980-8579, Japan

(Received 9 October 2005; accepted 9 February 2006; published online 28 March 2006)

The microstructural refinement mechanism by controlling heating rate was investigated in TiAl alloys as an example of multiphase materials. During heating in the α+γ dual phase field of TiAl alloys, the phase transformation of γ to α takes place. The increase in heating rate raises the extent of superheating, leading to increases in driving force and the nucleation rate of the α phase. At higher heating rate, numerous α nuclei form and lengthen, resulting in a fine α2/γ lamellar microstructure after subsequent cooling. The theoretical prediction was demonstrated by the experimental results obtained on a fully lamellar Ti-48Al alloy. © 2006 American Institute of Physics. [DOI: 10.1063/1.2190076]

It is well established that the microstructure of multiphase materials can be refined by fast cooling after annealing heat treatment. For example, fast cooling has always been applied to decrease the lamellar spacing of fully lamellar microstructure of TiAl alloys to improve the creep resistance.1–4 However, fast cooling results in structural degradation has been found to deteriorate the creep strength greatly.3,5–9 Thus, enhancement of creep resistance in multiphase materials with particular reference to γ-TiAl

The microstructural refinement mechanism by controlling heating rate may have some effects on the prior α/γ lamellar morphology before cooling. Different transformation pathways have been utilized in cycle heat treatment processes to refine the grain size of TiAl alloys.13,14 However, less attention has been paid to the heating process for refining the lamellar microstructure of TiAl alloys. During heating to high temperature, the heating process parameter such as heating rate may have some effects on the prior α/γ lamellar morphology as well as the final α2/γ lamellar microstructure.

During heating in the α+γ field, the phase transformation of γ to α proceeds by either growth of preexisting α plates transformed from α2 plates or precipitation on the four {111} habit planes of γ plates when the driving force is sufficient.11 A previous study has shown that the α phase precipitates as laths with rapid nucleation, rapid lengthening, and slower thickening.15 Consequently, the distribution of α precipitates depends on the number of nuclei governed by the driving force of the phase transformation. The driving force for the α precipitates can be expressed as

\[ \Delta G_v = \Delta H \frac{\Delta T}{T_e} \]

where \( \Delta H \) is the difference in enthalpy between the final temperature of \( T_f \) and the eutectoid temperature \( T_e \), and \( \Delta T \) is superheating. If the heating rate is changed in the temperature range from \( T_0 \) to \( T_f \), the superheating is expressed as

\[ \Delta T = T_f - T_e = T_0 + \nu_h t - T_e, \]

where \( \nu_h \) is the heating rate and \( t \) is time. It is clear that the increase in heating rate raises the extent of superheating, leading to an increase of the driving force for the α precipitation.

From previous studies,17,18 it is found that the formation of γ phase within the α phase during cooling was a diffusion-displacive transformation, which exhibits crystallographic features consistent with martensitic process. Exchange fluxes of Ti and Al atoms between the two phases arise associated with interfacial defect motion to achieve the compositional change required. It is speculated that during heating, the difference in compositions between the α nucleus and the γ matrix is much smaller when compared with the reversed transformation of γ to α. Furthermore, the α2/γ interfacial energy is low even at room temperature.19,20 Therefore, the coherent interfacial energy contributed from the difference in composition is very low in the nucleation stage. If it is supposed that a disclike lamellar nucleus with a

---

FIG. 1. Relevant portion of the Ti-Al phase diagram.

---

a)Electronic mail: hlzhu@material.tohoku.ac.jp
radius of $r$ and height of $h$ (Fig. 2) is formed while ignoring the coherent interfacial energy, the free energy change associated with the nucleation process involves the reduction of a volume free energy $-V\Delta G_{v}$, the increase of an incoherent interfacial free energy $A\gamma_{i}$, and a misfit strain energy $V\Delta G_{s}$ (Ref. 16)

$$\Delta G = -V(\Delta G_{v} - \Delta G_{s}) + A\gamma_{i}$$

and

$$\Delta G = -\pi r^{2} h(\Delta G_{v} - \Delta G_{s}) + 2\pi rh\gamma_{i}.$$  

The critical radius $r^{*}$ is given by $d(\Delta G)/dr = 0$,

$$r^{*} = \gamma/(\Delta G_{v} - \Delta G_{s}).$$

and the activation energy barrier for forming the critical nucleus is $\Delta G^{*}$,

$$\Delta G^{*} = \pi h\gamma^{2}/(\Delta G_{v} - \Delta G_{s}).$$

If the concentration of heterogeneous nucleation sites is $C_{i}$ per unit volume, the heterogeneous nucleation rate of the $\alpha$ precipitation during heating can be expressed by the same equation as used for the cooling$^{16}$

$$N = aC_{i} \exp\left(-\frac{\Delta G_{m}}{kT}\right) \exp\left(-\frac{\Delta G^{*}}{kT}\right),$$

where $\Delta G_{m}$ is the activation energy per atom for atomic migration, $a$ is a factor that includes the vibration frequency of the atoms and the area of the critical nucleus, and $k$ and $T$ have their usual meanings. $a$ and $\Delta G_{m}$ are assumed to be constant to evaluate the effect of temperature on nucleation rate. The total strain energy contributing to $\Delta G^{*}$ can be reduced effectively by nucleation on the stacking faults$^{16}$.

Therefore, $\Delta G^{*}$ is mainly controlled by the driving force for precipitation $\Delta G_{p}$, and larger driving force will cause a larger decrease in the activation energy barrier. The variations of the exponential terms in the above equation $\Delta G^{*}$, and nucleation rate with temperature, are shown schematically in Fig. 3. $\Delta G^{*}$ decreases while increasing the superheating as in the case of cooling in which $\Delta G^{*}$ decreases while increasing the undercooling. However, the reverse changes of the superheating and the undercooling with the temperature determine the difference in their effects on the nucleation rate. The nucleation rate is negligible at very high undercooling because diffusion is too slow at low temperature, whereas the nucleation rate increases with increasing the superheating until a maximum value at the $\alpha$ transus temperature. Therefore, the nucleation rate can increase greatly with the increases in the superheating and the driving force, which can be achieved by the increase in heating rate.

Figure 4 shows microstructure evolutions during heating at different heating rates in the $\alpha + \gamma$ field and cooling from this field schematically. In the case of slow heating, the pre-existing $\alpha$ laths transformed from $\alpha_{2}$ laths have more time to grow by thickening to form coarse $\alpha$ laths [Fig. 4(b)]. After subsequent cooling, a relatively coarse $\alpha_{2}/\gamma$ lamellar microstructure is formed. In the case of fast heating [Fig. 4(c)], the slower thickening process of $\alpha$ laths cannot consume all the increased free energy by heating. Therefore, nucleation of the $\alpha$ phase might occur in local places such as $\gamma'/\gamma$ interfaces where the driving force increases for nucleation are sufficient$^{11}$. With continuous heating, numerous $\alpha$ nuclei form and lengthen. As a result, the precipitated $\alpha$ lamellae become fine at the final temperature. During subsequent cooling, the fine $\alpha$ lamellae transform to fine $\alpha_{2}$ and $\gamma$ lamellae. Thus, fast heating results in refined $\alpha_{2}/\gamma$ lamellar microstructure.

To verify the microstructural refinement potential of fast heating, experiments were carried out on a polycrystalline Ti-48 mol % Al alloy. Samples were first solution treated at 1700 K in the single $\alpha$ phase field and then furnace cooled at a cooling rate of about 0.5 K s$^{-1}$. A fully lamellar microstructure was produced in the Ti-48Al alloy after the solution treatment [Fig. 5(a)], which is designated as as-grown hereafter. Then the samples were subjected to secondary annealing treatments with controlled heating rate ranging from $1.7 \times 10^{-3}$ to 0.067 K s$^{-1}$ in the temperature range from 1500 to 1550 K. They were held at 1550 K until the total duration from 1500 K reaches six hours, and thereafter furnace cooled to room temperature at a cooling rate of about 0.5 K s$^{-1}$. At the heating rate of $1.7 \times 10^{-3}$ K s$^{-1}$, the cooling was started after 10 h.

The microstructures were still fully lamellar after the secondary annealing treatments, but the lamellar morphologies within lamellar colonies were significantly refined$^{21}$ (Fig. 5). The density of $\alpha_{2}$ lamellae in the annealed microstructures [Figs. 5(b)–(d)] increases significantly compared to that in the as-grown microstructure [Fig. 5(a)]. For example,
ample, $\alpha_2$ lamellar spacing decreases from an average value of 3.5 $\mu$m in the as-grown microstructure to 2.2 $\mu$m in the annealed microstructure for the heating rate of 0.002 K s$^{-1}$ (Fig. 6). Furthermore, the $\alpha_2$ lamellar spacing in the annealed microstructure decreases with increasing heating rate (Fig. 6). However, some $\alpha_2$ lamellae are terminated within lamellar colonies [Figs. 5(c) and 5(d)] and the average length of $\alpha_2$ lamellae decreases with the increase of the heating rate (Fig. 6). In addition, most of the $\alpha_2$ lamellae become thinner with an increasing heating rate (Fig. 5). A reduction of creep rate by one order of magnitude from the as-grown microstructure was achieved at the optimum heating rate.$^{21}$

In summary, during heating in the $\alpha+\gamma$ dual phase field of TiAl alloys, the phase transformation of $\gamma$ to $\alpha$ takes place. Fast heating raises the extent of superheating, increasing the driving force and nucleation rate of $\alpha$ precipitation greatly. Numerous fine $\alpha$ lamellae are formed in the microstructure at high temperature. During subsequent cooling, $\alpha$ lamellae transform to $\gamma$ and $\alpha_2$, resulting in a fine $\alpha_2/\gamma$ lamellar microstructure. Similarly, it is expected that the microstructure could also be refined in other multiphase materials by controlling the heating process.

Financial support from the 21st century COE program in Tohoku University Materials Research Center and the Ministry of Education, Science, Sports and Culture, Japan (Grant No. 17360309) are gratefully acknowledged.

19C. L. Fu and M. H. Yoo, Scr. Mater. 37, 1453 (1997).