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Formation of fine fully-lamellar microstructure of TiAl-based alloy in rapid heating cyclic heat treatment process①

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[Abstract] The optical observation results of neocrystallization nucleation and growth of fine fully-lamellar (FFL) α_2/γ microstructure of a TiAl-based alloy in rapid heating cyclic heat treatment process were reported. The characteristics of $\alpha+\gamma\rightarrow\alpha$ transformation under rapid heating conditions were analysed. A model for explaining the nucleation and growth mechanism of FFL α_2/γ microstructure was proposed.

[Key words] rapid heating; cyclic heat treatment; nucleation and growth; model; mechanism

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1 INTRODUCTION

Due to its advantages in density, specific rigidity, high-temperature specific strength and burning resistance and so on, the TiAl-based alloys are considered a new generation of structural materials of the greatest application potentiality[1~3]. Like most other intermetallics, the room-temperature brittleness is the largest barrier which limits their practical applications as high-temperature structural materials, and how to improve their room-temperature ductility is a challenging task. In the coming years, the emphases will be put on how to further determine the relationships among processing, microstructure and mechanical properties[4].

Because of its advantages in near net shaping and cost, the foundry methods will be the first way for the TiAl-based alloys to obtain industrial applications[2]. But the as-cast TiAl-based alloys are generally composed of coarse lamellar colonies, and their room-temperature ductility is almost zero. Only through refining the microstructures by alloying, thermomechanical treatment or special heat treatment, can the cast alloys be used as high-temperature structural materials. By means of the above methods, several kinds of microstructures were developed[5~17]. It is proved that it is more probable for FFL microstructure to succeed in applications.

ZHANG et al[13~15], WANG[16] and XIE et al[17] separately studied the effects of cyclic heat treatments on microstructures and mechanical properties. The common points of the technologies adopted by them lie in: relatively low heating rate, relatively long holding time, relatively high cooling rate, and intermediate phase transformations by which the fully-lamellar (FL) microstructures form.

By means of a Gleeble-1500 thermal simulator and a GP-30(A) type induction heating machine, the authors studied the cyclic heat treatment technology of a TiAl-based alloy under the conditions of high heating rate, short holding time and relatively slow cooling rate, and found that the FFL α_2/γ microstructure can directly form without intermediate transformations, and the nucleations mainly occur at the grain boundaries, but at the phase interfaces along certain special directions as well.

In this paper, the authors reported the optical observation results of nucleation and growth of the FFL α_2/γ microstructure, analysed the characteristics of the $\alpha+\gamma\rightarrow\alpha$ transformation, and finally proposed a nucleation and growth mechanism of the microstructure in rapid heating cyclic heat treatment process.

2 EXPERIMENTAL

The material used is as-cast Ti-33Al-3Cr (mass fraction, %) alloy. The dimensions of the samples are diameter d 10 mm × 15 mm or d 12 mm × 60 mm. The heat treatment technology used is schematically shown in Fig. 1. All the cyclic heat treatment tests were performed on a Gleeble-1500 thermal simulator or a GP-30(A) induction heating machine, and the main technological parameters are as follows: heating rate 25~ 3 200 ℃/s, holding temperature 1 310~ 1 340 ℃, holding time 2 s~ 5 min, cycling number 1 ~ 9 time(s), and cooling rate 20~ 160 ℃/s.

3 OPTICAL OBSERVATIONS AND AFFECTING FACTORS

Figs. 2(a) and (b) respectively show the nucle

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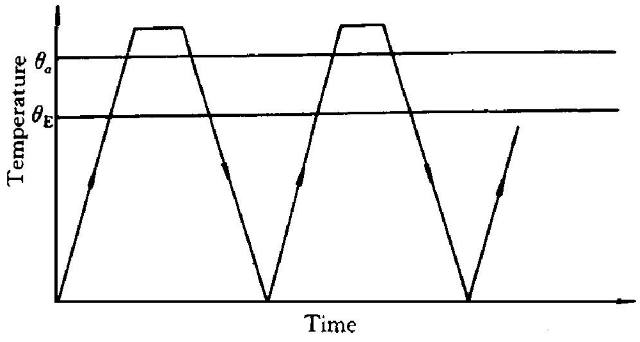

Fig. 1 Schematic diagram of cyclic heat treatment technology

ation of FL α_2/γ microstructure of cast TiAl-based alloy at grain boundaries and phase interfaces in simulation tests under rapid heating, short holding and relatively slow cooling conditions. Figs. 3(a) and (b) respectively show the nucleation and growth of FL α_2/γ microstructure of cast TiAl-based alloy at grain boundaries in induction heat treatment process.

By analyzing Figs. 2 and 3, combined with the results of the exploratory tests, the following conclusions can be drawn.

1) The nucleation of the cast TiAl-based alloy in the rapid heating cyclic heat treatment process belongs to phase transformation neocrystallization nucleation.

2) The effect of heating rate on the neocrystallization nucleation of the cast TiAl-based alloy is the largest. When the heating rate is smaller than 25 ℃/s and the holding temperature is below 1 310 ℃, no neocrystallization nucleation occurs.

3) When the holding temperature is below

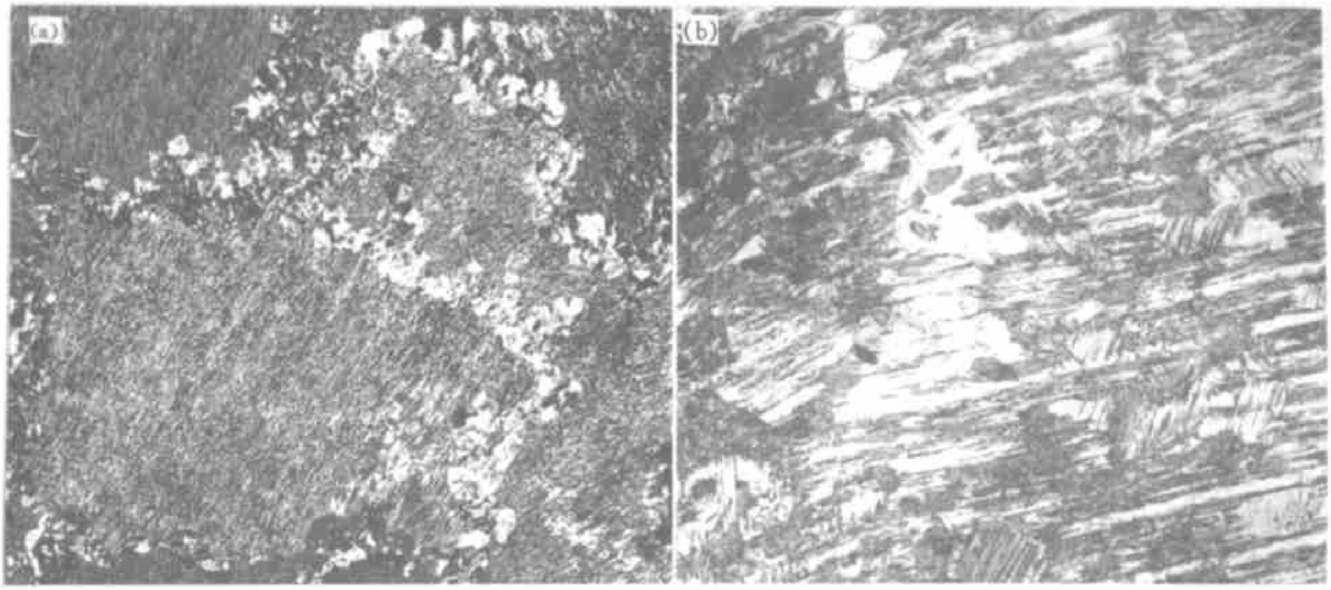

Fig. 2 Nucleation of FL microstructures of TiAl-based alloy in simulated heating cyclic heat treatment
(a) —At grain boundaries; (b) —At phase interface

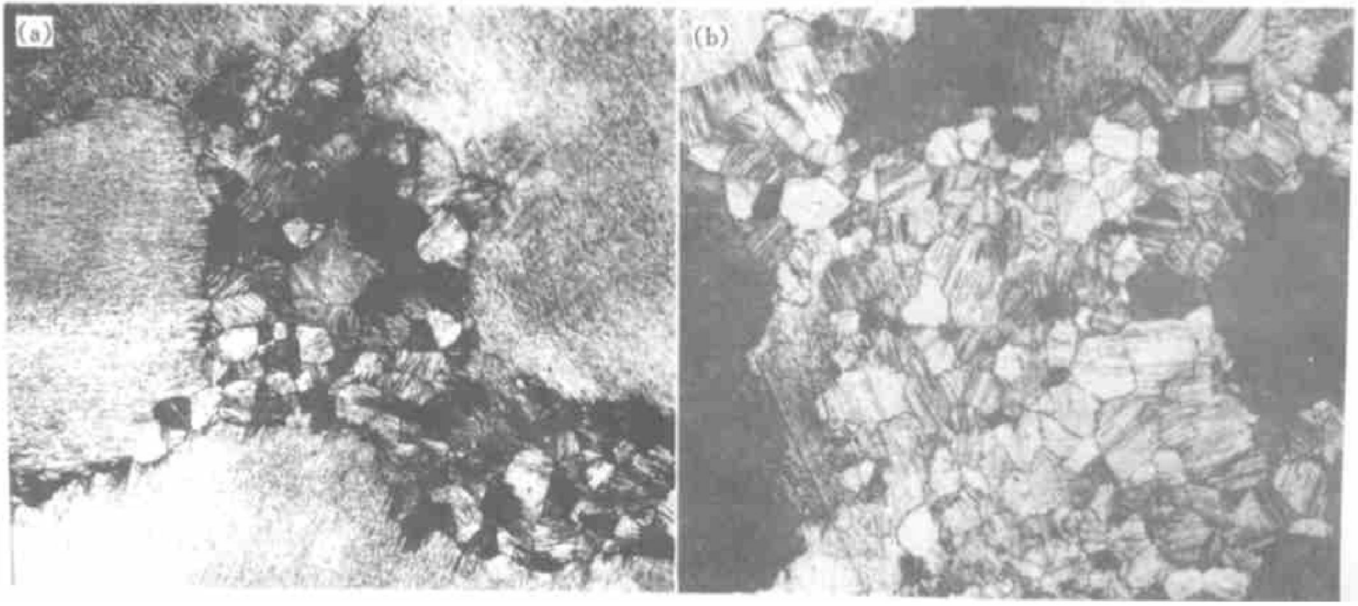

Fig. 3 Neocrystallization nucleation and growth of FL microstructure in induction heating cyclic heat treatment of cast TiAl-based alloy

1340 ℃, the effect of holding time (2s~ 5min) on nucleation and growth of the cast TiAl-based alloy is slight. For the convenience of microstructure control, short holding time is recommended.

4) When the cooling rate is slower than 50~ 80 ℃/s, FFL α_2/γ microstructure can be obtained.

5) When the cycling number is 5 times, FL α_2/γ microstructure can be obtained. In consideration of the other factors, 3~ 7 times cycling is proper.

4 CHARACTERISTICS OF $\alpha+\gamma\rightarrow\alpha$ TRANSFORMATION ON CONTINUOUS HEATING

4.1 Degree of superheating occurs and it increases with increasing heating rate

The continuous heating process can be decomposed into a series of isothermal process with short holding time. Each very small time section ($\Delta\tau_i$) corresponds to a certain temperature (T_i), and it is known from the isothermal transformation diagram that each temperature (T_i) corresponds to a certain inoculation period (Z_i) (Fig. 4). Therefore, in the temperature range in which the α/γ lamellar microstructure decomposes, $\Delta\tau_i/Z_i$ at a certain temperature T_i expresses the inoculation effect or inoculation period consumption (I_p). The I_p from T_α to T_n can be expressed by

$$I_p = \frac{\Delta\tau_1}{Z_1} + \frac{\Delta\tau_2}{Z_2} + \cdots + \frac{\Delta\tau_n}{Z_n} = \sum_{i=1}^{n}\frac{\Delta\tau_i}{Z_i} \qquad (1)$$

When $\Delta\tau_i$ approaches zero, expression (1) can be rewritten as

$$I_p(T_n) = \int_{\tau_0}^{\tau_n}\frac{d\tau}{Z(T)} = \int_{T_\alpha}^{T_n}\frac{d\tau/dT}{Z(T)}dT \qquad (2)$$

where T_α is the temperature at which ($\alpha+\gamma$) and α reaches equilibrium; $d\tau/dT$ is the reciprocal of heating rate.

Let the heating rate be a constant, and express it by v_h, then $dT/d\tau = v_h$. Thus Eqn. (2) can be further simplified into

$$I_p(T_n) = \frac{1}{v_h}\int_{T_\alpha}^{T_n}\frac{dT}{Z(T)} \qquad (3)$$

Only when $I_p = 1$, can the inoculation process be completed, consequently $\alpha+\gamma\rightarrow\alpha$ transformation occurs. It is clear from Fig. 4 that when the temperature is above T_α, there exists

$$Z_1 > Z_2 > Z_3 > \cdots > Z_n \qquad (4)$$

Therefore,

$$\frac{1}{Z_1} < \frac{1}{Z_2} < \frac{1}{Z_3} < \cdots < \frac{1}{Z_n} \qquad (5)$$

Let $\Delta\tau_1 = \Delta\tau_2 = \Delta\tau_3 = \cdots = \Delta\tau_n$ and $\Delta\tau_1 + \Delta\tau_2 + \Delta\tau_3 + \cdots + \Delta\tau_n = Z_n$, then

$$\frac{\Delta\tau_1}{Z_1} + \frac{\Delta\tau_2}{Z_2} + \frac{\Delta\tau_3}{Z_3} + \cdots + \frac{\Delta\tau_n}{Z_n} < \frac{\Delta\tau_1 + \Delta\tau_2 + \cdots + \Delta\tau_n}{Z_n} = 1$$

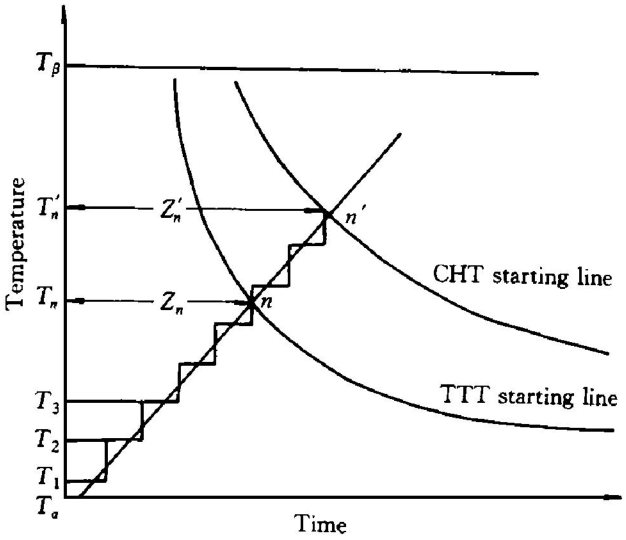

Fig. 4 Schematic diagram showing comparison between starting line of CHT and that of TTT

that is

$$\frac{1}{v_h}\int_{T_\alpha}^{T_n}\frac{dT}{Z(T)} < 1 \qquad (6)$$

In other words, when the continuous heating curve at v_h intersects the starting line of the isothermal transformation diagram of $\alpha+\gamma\rightarrow\alpha$ (corresponding to T_n and Z_n), the $\alpha+\gamma\rightarrow\alpha$ transformation will not occur. Only through further heating to a higher temperature (T'_n), to satisfy $\frac{1}{v_h}\int_{T_\alpha}^{T'_n}\frac{dT}{Z(T)} = 1$, can the $\alpha+\gamma\rightarrow\alpha$ transformation start. Therefore, for the continuous heating process, the starting point of transformation is located at the upper right side of the intersection between the continuous heating curve and the point corresponding to time temperature transformation (TTT) starting line, thus the continuous heating transformation (CHT) starting line will be located at the upper right side of the TTT starting line.

Let v_{h1} and v_{h2} be two arbitrary heating rates ($v_{h2} > v_{h1}$), from the above analyses, one can obtain

$$\int_{T_\alpha}^{T'_{n1}}\frac{dT}{Z(T)} = v_{h1} \qquad (7)$$

and

$$\int_{T_\alpha}^{T'_{n2}}\frac{dT}{Z(T)} = v_{h2} \qquad (8)$$

From expressions (7) and (8), one can obtain

$$\int_{T_\alpha}^{T'_{n2}}\frac{dT}{Z(T)} - \int_{T_\alpha}^{T'_{n1}}\frac{dT}{Z(T)} = v_{h2} - v_{h1} \qquad (9)$$

namely

$$\int_{T'_{n1}}^{T'_{n2}}\frac{dT}{Z(T)} = v_{h2} - v_{h1} \qquad (10)$$

According to the Cauchy Intermediate Value Theorem, one has

$$T'_{n2} - T'_{n1} = Z(T_0)(v_{h2} - v_{h1}) > 0 \qquad (11)$$

where T_0 is a certain value between T'_{n1} and T'_{n2}, thus

$$(T_{n2}' - T_\alpha) - (T_{n1}' - T_\alpha) > 0$$

or

$$\Delta T_2' - \Delta T_1' > 0 \tag{12}$$

Therefore it can be concluded that the degree of superheating increases with heating rate.

4.2 Transformation rate of $\alpha+\gamma\rightarrow\alpha$ increases with heating rate and is completed in a temperature range

Fig. 5 schematically shows the $\alpha+\gamma\rightarrow\alpha$ transformation on continuous heating. The intersection points express the starting and finishing time and temperature at various heating rates. It is clear that the higher the heating rate is, the higher the starting and finishing temperature are, and the shorter the transformation time is, i. e. the faster the formation of α phase is, and the transformation is completed in a certain temperature range.

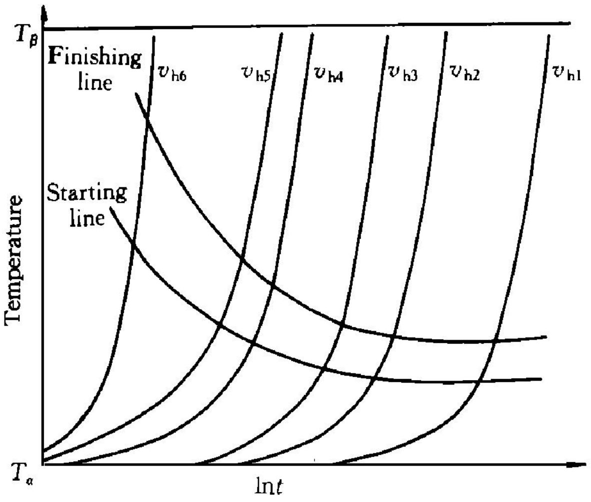

Fig. 5 Schematic diagram showing effects of heating rate (v_h) on starting line and finishing line of $\alpha+\gamma\rightarrow\alpha$ transformation ($v_{h1} < v_{h2} < v_{h3} < v_{h4} < v_{h5} < v_{h6}$)

Above T_α, the nucleation rate of α phase can be expressed by

$$I \propto \exp(-Q/kT) \cdot \exp(-\Delta G^*/kT) \tag{13}$$

where I is nucleation rate, Q is diffusion activation energy of atoms, ΔG^* is nucleation work, k is Boltzmann's constant, and T is absolute temperature.

On one hand, the value of Q changes very slightly with temperature and can be considered a constant, therefore $\exp(-Q/kT)$ increases with temperature. On the other hand, ΔG^* is inversely proportional to $(\Delta T)^2$, therefore when the temperature rises, $\exp(-\Delta G^*/kT)$ also increases. As a result, when the heating rate rises, because of superheating, the starting temperature of the $\alpha+\gamma\rightarrow\alpha$ transformation moves to a higher temperature, which is beneficial to fast nucleation.

The growth rate of α phase can be expressed by

$$v_G \propto \Delta G_v / T \cdot \exp(-Q/kT) \tag{14}$$

The change of $\exp(-Q/kT)$ is faster than that of $\Delta G_v/T$, therefore the growth rate of the α phase increases with temperature.

The above analyses account for the reasons why rapid heating can refine the grains and shorten the transformation time.

5 MODEL FOR NUCLEATION AND GROWTH OF FL MICROSTRUCTURE

Different from the conventional heat treatments and the cyclic heat treatments at relatively slow heating rates, the rapid heating cyclic heat treatment can rapidly and directly refine the coarse cast lamellar microstructure, and the refining effect is very obvious. The optical observations show that the nucleation mainly occurs at the grain boundaries, which can be shown by Fig. 6.

Regardless of the anisotropy of the interface energy, the nuclei of the new phases formed at grain boundaries should be like double lens, i. e. the shape of Fig. 6(a).

The neocrystallization nucleation of the TiAl-based alloy is likely to occur at the grain boundaries, which is due to the fact that the grain boundaries have the following advantages.

1) The grain boundaries have higher energy when compared with the interiors of the grains.

2) The strain energy increment for the grain boundary nucleation is relatively small because the atoms at the grain boundaries are much disordered.

3) It is easy for the atoms to diffuse along the

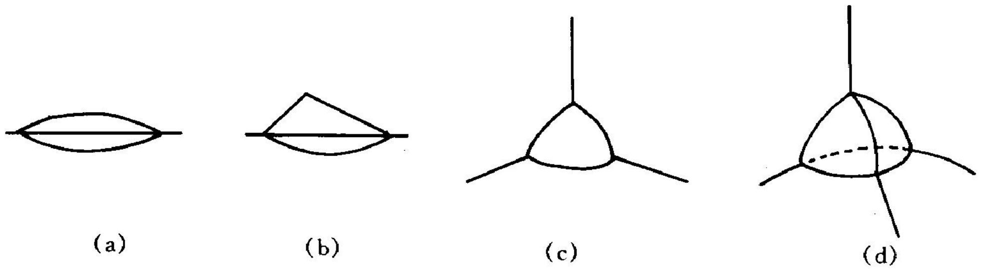

Fig. 6 Nucleations at grain boundaries of FFL microstructures of TiAl-based alloy under rapid heating cyclic heat treatments

grain boundaries.

4) It is likely for the grain boundaries to be rich in the elements needed for the formation of new phases.

Generally, it is not easy for the neocrystallization nucleation to occur at the phase interfaces, because there exists strict coherent relationship between the α and γ phases of the TiAl-based alloys, and the interfacial energy is low. But in the rapid heating cyclic heat treatment process, the practical holding temperature is higher due to strong superheating, and belongs to the range in which the α phase is very stable. There occurs the decomposition of the γ phase when held in the α phase field, thus making the α/γ interface protrude into the γ phase and forming small convex regions of the α phase. They will be transformed into fine α_2/γ lamellae on the following cooling. Fig. 7 shows the process of the formation of α phase nuclei at the α/γ interfaces and the transformation of the α phase nuclei into α_2/γ lamellae on cooling. The newly formed α_2/γ lamellae represent an angle of 60° or 120° with the primary lamellae. This is caused by that the new phases form favorably in the form of lamellae on special habit planes along special habit directions of the matrix so as to reduce the transformation resistance.

Based on the optical observation results and the above analyses, a model is proposed for explaining the formation of FFL α_2/γ microstructure in the rapid heating cyclic heat treatment process, as shown in Fig. 8.

It has been proved[18] that the formation of lamellar microstructure of TiAl alloys is not through eutectoid reaction but through the precipitation of the α lamellae on disordered α or ordered α_2 matrix, as shown in Fig. 9.

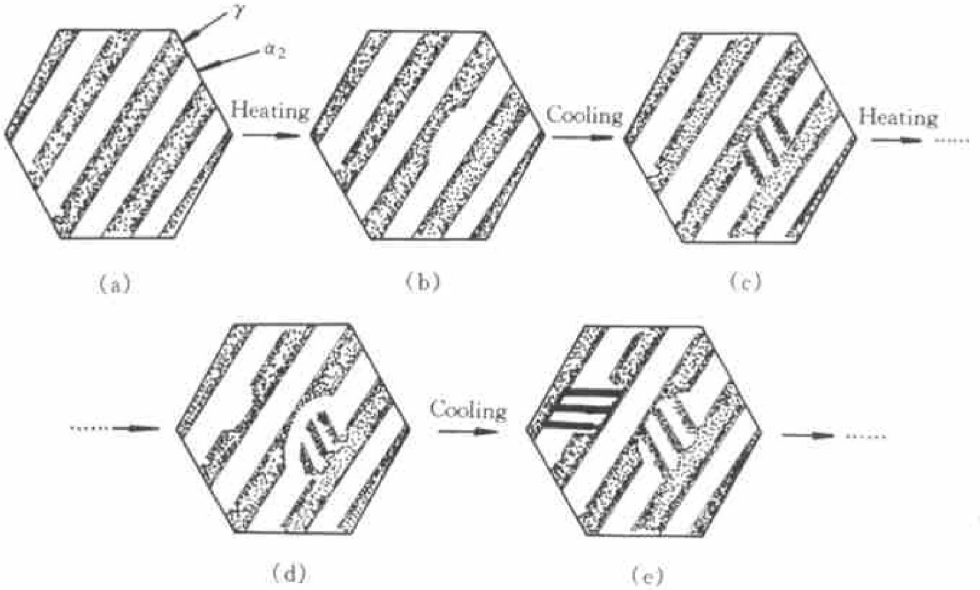

Fig. 7 Nucleation and growth at phase interfaces of FFL microstructures in rapid heating cyclic heat treatment process

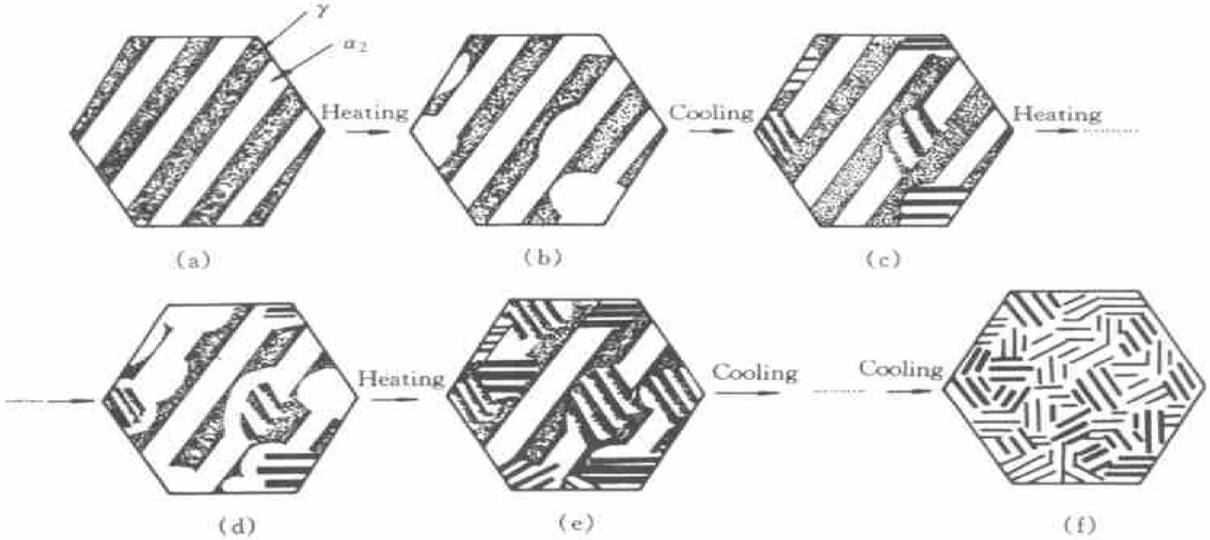

Fig. 8 Model for explaining nucleation and growth of FFL α_2/γ microstructure in rapid heating cyclic heat treatment process

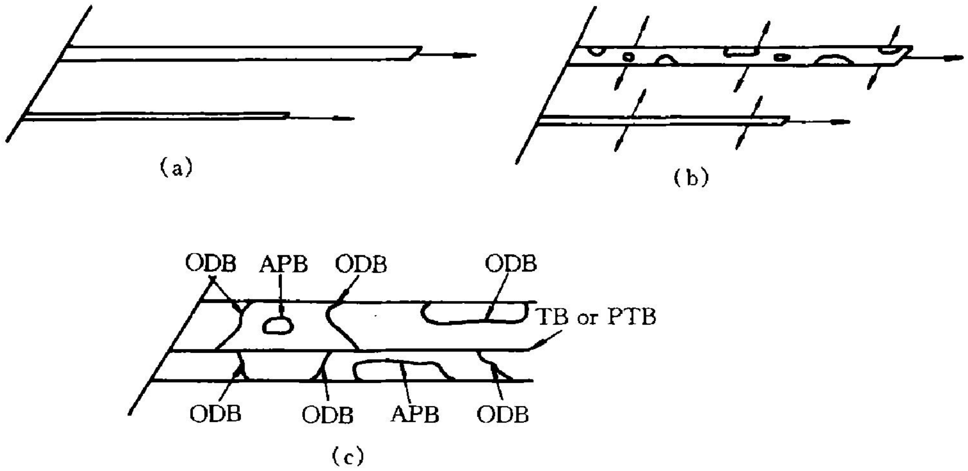

Fig. 9 Schematic representation of formation sequence of γ lamellae
(TB—Twin boundary; PTB—Partial TB; ODB—Order domain boundary; APB—Antiphase boundary)

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