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ARTICLE

Decomposition Kinetics of Metastable β Phase in Ti-1300 Alloy Under Isothermal Conditions

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Abstract: Phase transformation kinetics of Ti-1300 alloy after solution treatment were investigated under isothermal conditions in the temperature range of 400 °C to 700 °C by dilatometric method. The decomposition kinetic equation of metastable β phase in the alloy was constructed under isothermal conditions by analyzing the experimental data in the theoretical frame of the Johnson-Mehl-Avrami (JMA) theory. The different values of K and n parameters were obtained in the temperature range of 400 °C to 420 °C and 500 °C to 700 °C, indicating that the mechanism of the phase transformation is different in different temperature ranges. The decomposition mechanism of metastable β phase is $\beta_m \rightarrow \beta' + \beta \rightarrow \alpha + \beta$ at the temperature of 400 °C to 420 °C, whereas the decomposition mechanism of metastable β phase is $\beta_m \rightarrow \alpha + \beta$ at the temperature of 500 °C to 700 °C. When the metastable β phase of Ti-1300 alloy was annealed isothermally at a given temperature, the amount of α phase increases firstly, and then reaches a stable value with increasing the holding time. Based on calculation and experimental results, time-temperature-transformation diagrams (TTT diagram) of Ti-1300 alloys were plotted for the metastable β phase decomposition under isothermal conditions from 500 °C to 700 °C. And the nose temperature of the TTT diagram of the alloy is around 600 °C for the Ti-1300 alloys.

Key words: decomposition kinetics; mechanism of phase transformation; Ti-1300 alloy; TTT diagram

Metastable β titanium alloys are widely used in aerospace and automotive industries due to their high specific strength, hardenability, corrosion resistance and toughness[1-4]. For the metastable β alloys, it was well known that the martensitic transformation cannot occur upon quenching to room temperature because they contain a large amount of stabilizing β phase elements. The mechanical properties of titanium alloy could be strengthened by the α phase precipitated from the matrix of the β phase during aging process. However, the size, morphology and amount of the α phase have a significant influence on the mechanical properties of the metastable β titanium alloy[5-8].

Many researches have been conducted to investigate the decomposition and microstructure evolution of metastable β and near β titanium alloys to improve the properties of these alloys[9-11]. Malinov et al[9] found that the mechanism of $\beta_m \rightarrow \alpha + \beta$ transformation in β21s alloy was depended strongly on aging temperature. At the temperature higher than 650 °C, the mechanism of the phase transformation was mainly the nucleation and growth of grain boundary α phase, while that was mainly homogeneous nucleation at the first stage and slow diffusion-controlled growth of very fine α plates at the second stage in the temperature range of 500 °C to 600 °C. The amount of α phase first increased with increasing aging time, and then reached an equilibrium value at a given aging temperature. Naveen et al[10] also reported that the mechanism of the phase transformation was a slow diffusion controlled growth of very fine α plates in Ti-15-3. Appolaire et al[11] developed a model to predict the kinetics of αGB and αWGB morphologies during isothermal treatments based on the nucleation and growth laws. These results show that the mechanism of phase transformation is different for different titanium alloys due to the difference in the chemical composition and aging temperature.

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Ti-1300 titanium alloy (Ti-5Al-4Mo-4V-4Gr-3Zr) is a new metastable titanium alloy with high strength and toughness, whose comprehensive performance is better than that of Ti-1023 alloy based on Refs. [12,13]. Recently, a large number of studies concentrated on investigating its hot working behavior, phase transformation, the relationship between microstructure and mechanical properties to obtain an optimum combination of mechanical properties, and a flaw stress model had been built with activation energy of 177.59 kJ[14-19]. Lu found many secondary α phases with acicular shape around 80~200 nm in width precipitate in β matrix, so that the alloy exhibits superior strength around 1640 MPa[20]. The microstructure and properties of the alloy are sensitive to the hot working history. Therefore, it is essential to investigate the heat treatment process and decomposition behavior of metastable β phase to optimize properties of Ti-1300 alloy.

1 Experiment

The materials used in this study were hot-rolled Ti-1300 bars with a diameter of 12 mm, which was provided by Northwest Institute for Non-ferrous Metal Research. The β transus temperature of Ti-1300 alloy was measured to be about 830±5 °C by a metallographic method. The cylindrical specimens with 10 mm in height and 4 mm in diameter were machined from the as-received bars. The thermal expansion amount in length of the specimen during isothermal aging were measured using a Bähr DIL-805A/D dilatometer with induction heating device. The process of heating and insulation was carried out under vacuum (0.05 Pa), then followed by cooling with high purity argon atmosphere (99.999%) to avoid oxidation. The samples were solution treated at 845 °C for 10 min, and then aged at 400, 450, 500, 600, 650 and 700 °C for different time. Metallographic specimens were prepared by grinding and polishing, and then chemically etched with a solution of 10% hydrofluoric acid (HF), 20% nitric acid (HNO_3), and 70% distilled water (H_2O). The microstructure of the samples was observed by a Leica DMI5000M optical microscope and a SUPRA 40 scanning electron microscope (SEM). Phase analysis was conducted with a D8 ADVANCEX X-ray diffraction (XRD), with Cu Kα radiation at 45 kV and 40 mA. Microstructure and phase structure of the specimens was analyzed with a Tecnai G2 F20 field emission transmission electron microscope after mechanically thinning and ion polishing.

2 Results and Discussion

2.1 Microstructure and phase composition of the samples after solution treatment

Fig.1 shows the microstructure and XRD pattern of Ti-1300 alloy after quenching from the β phase field at 845 °C for 10 min. The average grain size of the alloy is measured to be 48 μm, as shown in Fig.1a. It is seen from Fig.1b that the microstructure of the alloy consists mainly of β phase due to rich β-stabilizing elements to hinder transformation martensitically upon quenching to room temperature[12].

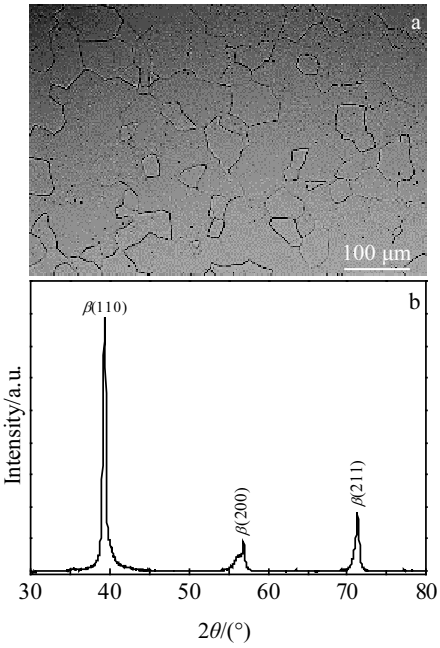

Fig.1 Microstructure (a) and XRD pattern (b) of Ti-1300 alloy after solution treatment at 845 °C for 10 min

2.2 Dilatometric behaviors

Fig.2 shows the variation of specimen length versus holding time for the Ti-1300 alloy during aging treatment at 400, 420, 500, 550, 600, 650 and 700 °C. The lengths of the specimens decrease slightly with the increasing aging time, then decrease pronouncedly, and finally decrease slightly for the samples aging-treated at 400 and 420 °C in Fig.2a. When the aging temperature is higher than 420 °C, the lengths of the specimens firstly decrease significantly with the increasing aging time, and then decrease slightly. It is well known that the length of metallic materials should be constant under isothermal conditions when the process of solid phase transformation cannot occur. Accordingly, it can be considered that the phase transformation is almost completed when the lengths are nearly constant. The lengths of specimens are almost constant in the early holding period at 400 and 420 °C, indicating that the phase transformation is impossible to occur in this stage. However, similar tendencies are found in the curves of specimens aging-treated at 500, 550, 600, 650 and 700 °C. Moreover, more time would be required to finish phase transformation when the alloys are aged at lower temperature in the range from 500 °C to 700 °C, and the rate of change in length is very fast as further increasing the aging temperature. Ti-1300 alloy which is a metastable β titanium alloy could be decomposed into ω, β' and α phase during aging process[1]. The $\beta_m \rightarrow \omega$, β' and α transformations are able to result in contraction of dilatometric curve[21]. Thus, the

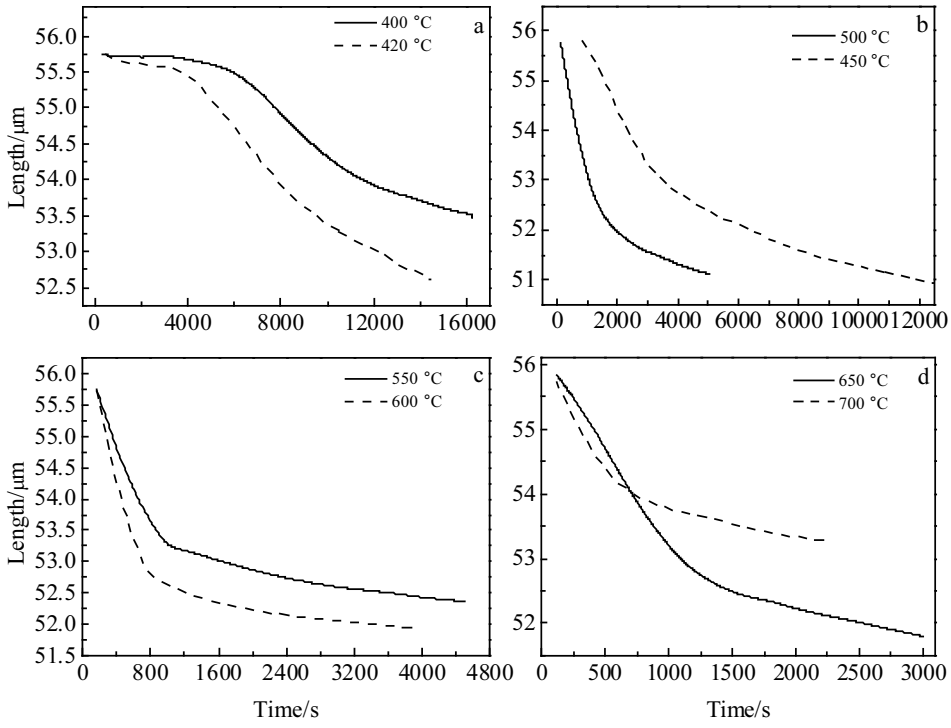

Fig.2 Variation of length vs time of solution treated Ti-1300 alloy during aging treatment at 400, 420 °C (a), 450, 500 °C (b), 550, 600 °C (c), and 650, 700 °C (d)

lengths shrink faster at first and then become relatively slow in the isothermal aging process, which indicate that the phase transformation is produced quickly in early stage and then the rate of phase transformation gradually slows down at the later stage.

2.3 Decomposition kinetics of Ti-1300 alloys

The degree of phase transformation is linear with the length of the polycrystalline titanium alloy when the composition of the alloy is uniform[22]. The change in length of the Ti-1300 alloy during isothermal aging process was analyzed by lever rule, as shown in Fig.3. The relative change in length of Ti-1300 alloys at any isothermal moment in the aging process (y) can be calculate by[18]:

$$y = \frac{\Delta l_t}{\Delta l_f} = (l_s - l_t)/(l_s - l_f) \quad (1)$$

where, Δl_t is the variation in the length of the sample at transformation time t, Δl_f is the total variation in sample length during the phase transformation process, l_s is the length of the sample at the beginning of the phase transformation, l_t is the length of the specimen at transformation time t and l_f is the length of the specimen at the end of the phase transformation.

According to dilatometric results, the relationship between volume fraction of metastable β phase and the aging time can be determined by formula (1), and then the metastable β_m decomposition dynamic equation and kinetic parameters of

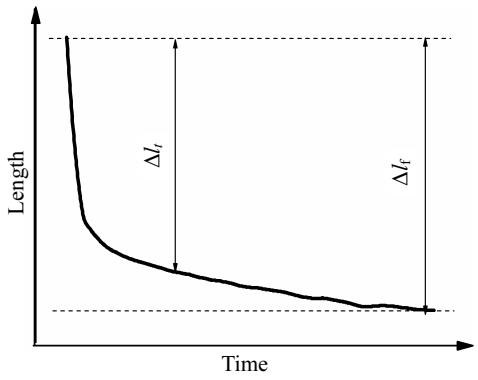

Fig.3 Variation of length vs time during aging treatment of solution treated Ti-1300 alloy

Ti-1300 alloy can be obtained during the isothermal aging process.

The decomposition of metastable β_m phase in the isothermal aging process is a typical nucleation-diffusion controlled phase transformation at certain temperature, and thus the kinetics of decomposition of metastable β_m phase in the Ti-1300 alloy can be characterized by classical Johnson-Mehl-Avrami equation. Therefore, according to the variation of the length shown in Fig.3, the degree of β_m

decomposition Ti-1300 alloy during isothermal aging can be expressed as[23]:

$$f = y = \frac{l_s - l_t}{l_s - l_f} = 1 - \exp(-Kt^n) \quad (2)$$

where, f is the degree of β_m decomposition at isothermal aging time t, K is the reaction rate constant decided by the phase transformation temperature, phase transformation free energy, interface energy and other parameters[15]; n is the Avrami exponent that be used to characterize the nucleation and growth mechanism of new phase in the β_m decomposition and t is isothermal time. Thus, Eq. (2) can be rewritten as:

$$1-f=\exp(-Kt^n) \quad (3)$$

Eq. (3) was used to analyze experimental data by means of logarithmic plots,

$$\ln\left(\ln\left(\frac{1}{1-f}\right)\right) = \ln K + n\ln t \quad (4)$$

Based on Eq. (4), the K and n at different aging temperatures could be derived for Ti-1300 alloy. The plots of ln(ln(1/(1–f))) against ln$t$ are presented in Fig.4 when Ti-1300 alloys were aged at aging temperature from 400 °C to 700 °C. The slope of the fitted straight line represents the value of Avrami exponent n and the intercept represents the value of K. Such plots are presented in Fig.5 for 420 °C (Fig.5a) and 550 °C (Fig.5b). Similarly, according to experimental data, the values of n and K for all studied temperatures could be obtained and are presented in Table 1. Remarkable distinction of the Avrami exponent n and reaction rate constant K could be seen for Ti-1300 alloy at different aging temperatures. These mean that JMA theory can be applied to characterize the kinetics of the β_m decomposition in Ti-1300 alloy under isothermal conditions.

The ln(ln(1/(1–f))) was basically linearly related to the ln$t$ when Ti-1300 alloys were aged at aging temperature 400, 420 and 700 °C. The value of n is 3.495, 2.74 and 1.25, respectively. Different values of n indicate different decomposition mechanism at corresponding temperature[24,25]. It also

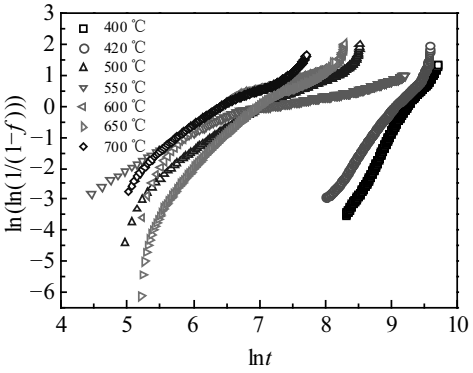

Fig.4 Plots of ln(ln(1/(1–f)))-ln$t$ for aging treatment at different temperatures of Ti-1300 alloy

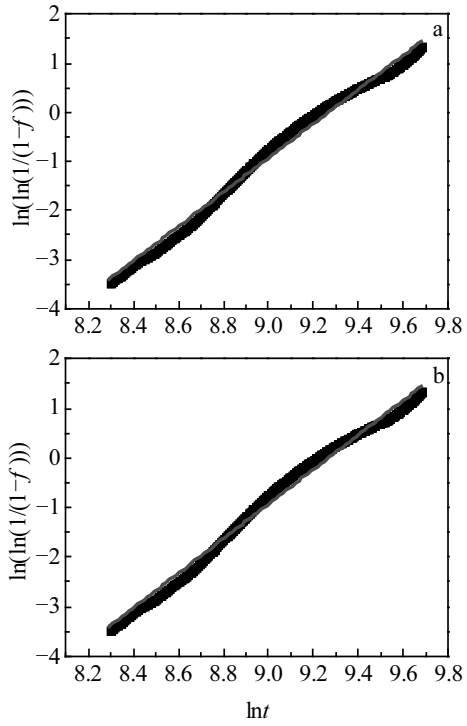

Fig.5 Plots of ln(ln(1/(1–f)))-ln$t$ for deriving the JMA parameters for Ti-1300 alloy at 420 °C (a) and 550 °C (b)

indicates that transformation mechanism of Ti-1300 alloy does not change at the aging temperature of 400, 420, and 700 °C. However, the plots can be divided into two segments at aging temperature 500, 550, 600 and 650 °C, and the different slope straight line to fit and different fitting lines can be obtained. These represent different mechanisms of decomposition. The value of n varies between 1.06~2.2 in the early stage at aging temperature 500, 550, 600 and 650 °C. However, there are an obvious tendency for change of the line slops to lower values between 0.5~1.38 as the aging time increases. The results show that the transformation mechanism of Ti-1300 alloy alters during the process of the β_m decomposition that take place at 500, 550, 600 and 650 °C. In the early stage, the mechanism of the decomposition in the alloy is that β grain boundaries are the nucleation sites and the α phase has a plate-like morphology[9]. And similar reports have been found in TC21, Ti-B19 and Ti 15-3 alloys[10,23,26].

The kinetic equation of aging treatment at different temperatures of Ti-1300 alloy was calculated using the JMA parameters which are listed in Table 1. The degree of metastable β phase decomposition at 400, 500, 550, 600, 650 and 700°C was calculated. Fig.6 shows the variation in the degree of metastable β phase decomposition of the alloys from the calculated volume fractions (from the JMA theory) and the experimental values with the aging time at different aging temperatures. It can be seen that there is only a slight distinction

Table 1 *n* and *K* of JMA equation of metastable *β* phase decomposition of solution treated Ti-1300 alloy

Temperature/°C	Early stage		Later stage	
	n	*K*	*n*	*K*
400	3.495	8.66×10^{-15}	3.495	8.66×10^{-15}
420	2.74	1.535×10^{-11}	2.74	1.535×10^{-11}
500	1.59	1.421×10^{-5}	0.962	1.114×10^{-3}
550	1.236	3.37×10^{-4}	0.756	6.819×10^{-3}
600	1.06	1.462×10^{-3}	0.5	5.03×10^{-2}
650	2.2	2.03×10^{-7}	1.38	3.3×10^{-4}
700	1.25	2.63×10^{-4}	1.25	2.63×10^{-4}

between the calculated and experimental values. In a word, there is a good agreement between the experimental and calculated fraction of metastable *β* phase decomposition in the alloys during isothermal aging treatment at different temperatures. This indicates that the JMA parameters can be used in the aging treatment practice of the Ti-1300 alloy to predict the course of the *β* phase decomposition.

In order to further identify decomposition pattern of metastable *β* phase in Ti-1300 alloy during isothermal aging, the microstructure evolution have been investigated by TEM and SEM analysis. Fig.7 shows TEM microstructures of Ti-1300 alloys aging-treated at 400 °C for 1 h and 4 h. It is seen that *β*′ precipitates with a bcc crystal structure exist in the matrix of the alloy at an aging time of 1 h. The reason for it is that alloying elements partition is produced during the early stage of isothermal aging at 400 °C, resulting in β_{lean} and β_{rich}; both of them are bcc phases; however, the magnitude of distortion of the bcc lattice in the coherent disordered precipitates (β_{lean}) is much larger than that of the bcc lattice of the matrix (β_{rich}) [1]. The metastable particles *β*′ are found in highly concentrated alloys. But *β*′ phase, having high free energy, is a typical transitional phase, and it is possible to transfer to more stable phase at higher temperature or for longer holding time. When the aging time is 4 h, a large number of *α* platelets distribute in the matrix, as shown in Fig.7b. Thus, the decomposition mechanism of the Ti-1300 alloy is $\beta_m\rightarrow\beta'+\beta$ in the early stages, and $\beta_m\rightarrow\beta'+\beta\rightarrow\alpha+\beta$ mechanism is dominated in later time during aging process at 400 °C. In addition, a large number of *α* platelets were observed in the Ti-1300 specimen aging-treatment at 550 °C for 30 and 90 min, as shown in Fig.8. And the volume fraction of *α* phase in the alloy aging-treated at 550 °C for 30 min is less than that for 90 min. Fig.9 shows XRD patterns of Ti-1300 alloy after isother- mal aging-treated at 550 °C for different time. It can be directly observed that the volume fraction of *α* platelets increases firstly with increasing hold time during isothermal aging treatment, and then reaches an equilibrium value. This is the main reason for the two segments of plots between ln(ln(1/(1–*f*))) and ln*t* at 550 °C. In other words, the nucleation and growth of the $\beta_m\rightarrow\alpha+\beta$ phase transformation is mainly produced during the first stage of the plots, and the coarsening of *α* platelets is formed during the latter stages.

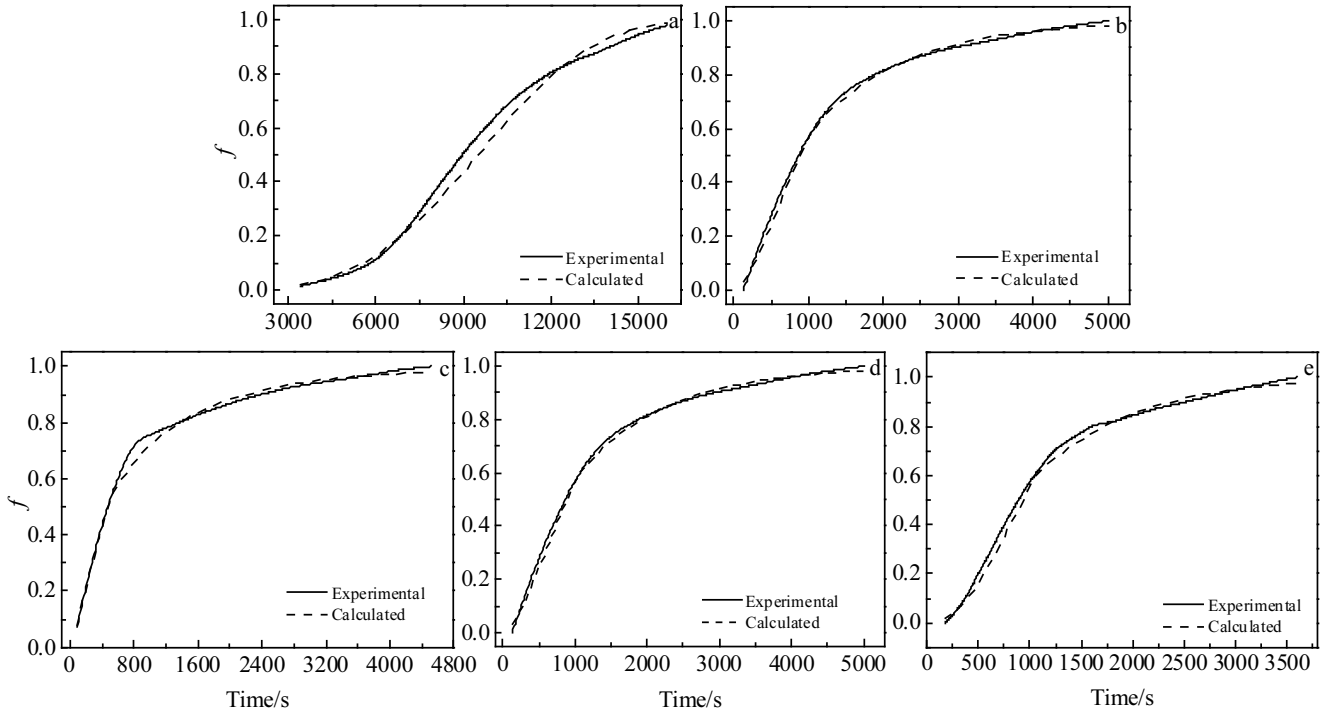

Fig.6 Comparison of calculated phase transformation kinetics with experimental results of Ti-1300 alloy isothermal aged between 400 °C to 700 °C: (a) 400 °C, (b) 500 °C, (c) 550 °C, (d) 600 °C, and (e) 650 °C

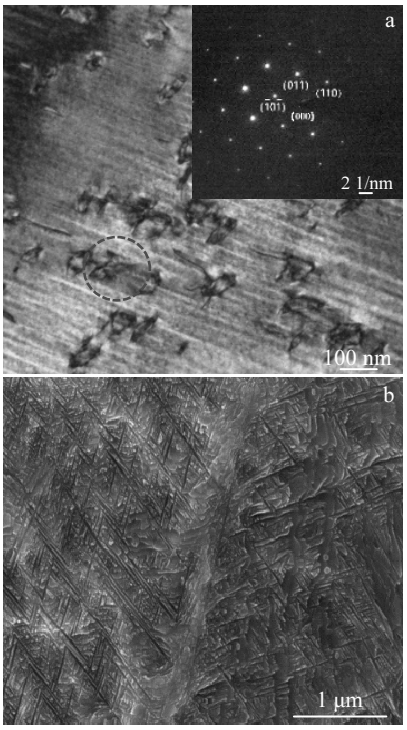

Fig.7 Bright field TEM micrograph and corresponding SAED pattern of the specimen aged at 400 °C for 1 h (a) and SEM micrograph of the specimen aged at 400 °C for 4 h (b)

2.4 Time-temperature-transformation diagram (TTT diagram)

The time-temperature-transformation diagram of $\beta_m \rightarrow \alpha+\beta$ transformation in Ti-1300 alloys, which can be used in heat treatment practice, were plotted based on the JMA equation. The diagrams reflect the relationship between the hold time and the temperature and the degree of metastable β decomposition to be attained. On the basis of the dilatometric results and the above JMA equation, the time-temperature-transformation diagram of the alloys can be plotted in Fig.10. Because times corresponding to the start and the end of the transformation are difficult to be measured experimentally, the start and the end of the $\beta_m \rightarrow \alpha+\beta$ transformation is defined as the volume fraction of 5% and 95% during phase transformation, respectively. It must be emphasized that the end of the transformation should be regarded as the degree of $\beta_m \rightarrow \alpha+\beta$ transformation and not as 95% of α phase. The middle curve corresponds to 50% of transformation completion. For instance, the final microstructure is a mixture of α and β phases when Ti-1300 alloy were isothermal aging-treated at 550 °C. As can be seen in Fig.10, the nose temperature of the time-temperature-transformation diagram is around 600 °C. It is well known that the $\beta_m \rightarrow \alpha+\beta$ transformation is a typical diffusion solid-transition. Thus, the rate of the $\beta_m \rightarrow \alpha+\beta$ transformation is controlled by the amount of undercooling ΔT

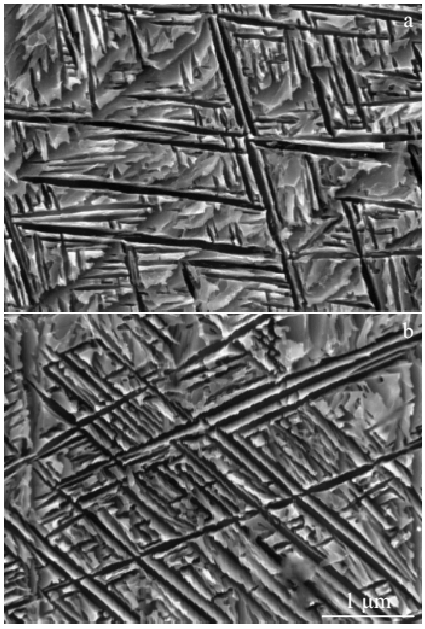

Fig.8 Microstructures of Ti-1300 alloy after isothermal aging at 550 °C for different time: (a) 30 min and (b) 90 min

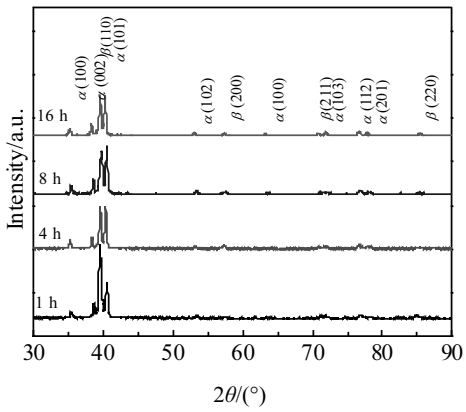

Fig.9 XRD patterns of the samples at 550 °C for different time

and activation energy for diffusion ΔG[27]. The rate of $\beta_m \rightarrow \alpha+\beta$ phase transformation decreases with increasing aging temperature when the temperature is over 600 °C. The reason for it is that over the nose temperature, the $\beta_m \rightarrow \alpha+\beta$ transformation rate is mainly controlled by ΔT, and the amount of undercooling decreases with increasing aging temperature, resulting in that the nucleation of α phase needs more time. At the temperature lower than 600°C, the $\beta_m \rightarrow \alpha+\beta$ transformation is mainly controlled by activation energy for diffusion which increases with the decreasing aging temperature. As the transformation temperature decreases in this range, the transition rate changes from fast to slow. Therefore, the iso-

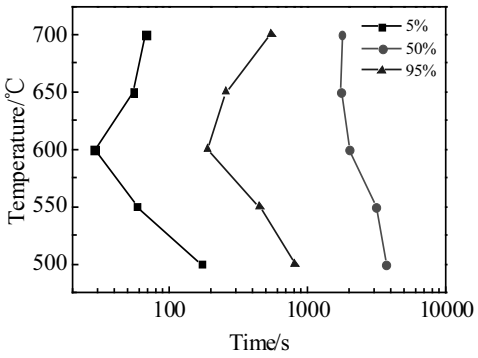

Fig.10 Calculated TTT diagrams for Ti-1300 alloy under isothermal conditions

thermal transformation diagram for Ti-1300 alloy presents C-shape.

3 Conclusions

1) The decomposition kinetics of metastable β phase in Ti-1300 alloy were modeled under isothermal conditions in the theoretical frame of the JMA equation. There is a good agreement between the experimentally measured and the calculated decomposed fractions of metastable β phase.

2) The decomposition mechanism of metastable β phase for Ti-1300 alloy is different under isothermal aging treatment at different temperatures. The mechanism of the decomposition in the alloy is that β grain boundaries are the nucleation sites in the early stage when the specimens are aged at 500, 550, 600 and 650 °C.

3) Based on calculation and experimental results, time-temperature-transformation diagrams (TTT diagram) of Ti-1300 alloys were plotted for the metastable β phase decomposition under isothermal conditions. And the nose temperature of the TTT diagram of the alloy is around 600 °C for the Ti-1300 alloys.

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等温条件下 Ti-1300 合金亚稳 β 相的分解动力学

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摘　要：通过高精度膨胀法研究了固溶态 Ti-1300 合金在 400~700 ℃等温条件下相变动力学。研究表明：固溶态 Ti-1300 合金中亚稳 β 相的分解动力学可用 Johnson-Mehl-Avrami（JMA）方程表征，并获得 400~700 ℃温度范围内 JMA 方程的特征参数 K 和 n，一定程度上反映了合金中亚稳 β 相的分解机制。当 Ti-1300 合金在 400~420 ℃时效时，亚稳 β 相的分解方式主要为 $\beta_m \rightarrow \beta' + \beta \rightarrow \alpha + \beta$；当合金在 500~700 ℃时效时，亚稳 β 相的分解方式主要为 $\beta_m \rightarrow \alpha + \beta$；同时在等温条件下，时效初期 α 相的形核率较快，且含量迅速增加，后期达到一定量后保持稳定。根据计算和试验结果，得到了 Ti-1300 合金在 500~700 ℃等温条件下亚稳 β 相的分解的 TTT 曲线，鼻尖温度约为 600 ℃。

关键词：分解动力学；相变机制；Ti-1300 合金；TTT 曲线

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