TTT Curve of Ti-6Cr-5V-5Mo-4Al-1Nb Alloy

Song Yue¹²³, Cui Xuefei¹²³, Yu Yang¹²³, Song Xiaoyun¹²³, Luo Yumeng¹²³, Ye Wenjun¹²³, Hui Songxiao¹²³

¹State Key Laboratory of Nonferrous Metals and Process, GRINM Group Corpration Limited(GRINM),Beijing, China;
²GRIMAT Engineering Institute Co Ltd,Beijing, China;
³General Research Institute for Nonferrous Metals, Beijing, China

Abstract: In order to study the aging hardening characteristics and isothermal aging process of Ti-6Cr-5V-5Mo-4Al-1Nb alloy, the time-temperature-transformation (TTT) curve of the alloy was measured by aging hardness method, and the microstructure changes during isothermal process were analyzed by XRD and SEM. It was found that the alloy had a high aging response speed at other aging temperatures except for the slow aging at 300°C and 400°C. When the aging time is prolonged at medium and low temperatures, the alloy can also produce the same aging strengthening effect as that at medium temperature aging, indicating that the temperature window of the alloy aging is wide, the aging strengthening effect is good, and it has the operability of heat treatment strengthening, so it is an ideal structural material. The isothermal aging transformation curve of the alloy is C type, and it can be obtained from the figure that the nose temperature is about 545°C.

1. Introduction

Metastable β titanium alloy is widely used in important bearing structures of aviation materials due to its light weight, high specific strength and good hardenability, which has gradually attracted extensive attention of researchers[1-3]. For metastable β titanium alloy, the second phase strengthening is the main strengthening method. Through aging treatment, the strength of metastable β titanium alloy can be increased by 50-80%[4]. Therefore, it is of great reference significance to study the microstructure evolution law of metastable β titanium alloy during isothermal aging and determined the TTT curve of the alloy, so as to formulate appropriate aging process and improve the mechanical properties of the alloy. Many scholars have studied the isothermal aging process of metastable β. For example, Li Chenglin et al.[5] measured the nose temperature of Ti-6554 by metallographic method at 540°C, and found that α phase precipitated at this temperature for 5 min. Chang Hui et al.[6] determined the TTT curve of Ti-B19 by in-situ resistance method, and the nose temperature range was 500-550°C. Fuwen Chen et al.[7] determined the TTT diagram of Ti-55531 alloy mainly by in-situ thermal expansion method and supplemented by microstructure observation, which provided strong evidence for shortening the incubation time of β→α phase transformation process by pre-aging treatment.

The titanium alloy used in this experiment is a new metastable β titanium alloy developed based on the principle of diversified strengthening. At present, there are few reports on the isothermal transformation kinetics of the alloy. Therefore, in this paper, the hardness of the alloy after isothermal aging was tested to study the aging hardening characteristics, analyze the relationship between hardness
and aging temperature and aging time, and draw the TTT curve of the alloy.

2. Materials and Methods

The raw materials in this experiment were high-grade sponge Ti and some intermediate alloys. The raw materials were pressed into electrodes and then melted into ingots by three vacuum consumable arc furnaces. The chemical composition of the top, middle and bottom of the ingots was analyzed, and the average value is shown in Table 1.

| Element | Ti | Al | Cr | Mo | Nb | V | N | C | O | H |
|---------|----|----|----|----|----|---|---|---|---|---|
| wt%     | Bal.| 4.08 | 5.06 | 5.05 | 0.96 | 4.90 | 0.006 | 0.011 | 0.10 | <0.001 |

The sample used in this experiment was selected from the bar R/2 by wire cutting, and the sample size is Φ10x5mm. The original structure is shown in Figure 1. It can be seen that the αp is fully broken and dispersed in the β matrix. The phase transformation point of the alloy measured by quenching metallographic method is 810°C.

Figure 1 Original Microstructure of Alloys

In order to study the aging hardening effect of the alloy and determine the TTT curve, the sample was dissolved at 850°C for 30 min, and the aging scheme is shown in Table 2. Before heat treatment, the sample was coated with a layer of titanium alloy heat treatment antioxidant. After heat treatment, the sample was coarsely ground and finely ground, and then the hardness was tested with a Vickers hardness tester. Each sample was tested at five different positions. The average hardness value of the five positions was taken as the hardness value of the sample. XRD Analysis of Precipitates in Samples by MAX 2500X-ray Analyzer.

Table 2 Isothermal aging heat treatment scheme

| Aging Temperature (℃) | Holding Time (min) |
|-----------------------|--------------------|
| 300                   | 120,240,360,480,600,720,1440,1680 |
| 400                   | 60,120,180,240,300,420,720,1200,1560 |
| 450                   | 30,60,120,180,240,360,480,540,720 |
| 500                   | 10,20,30,60,120,180,240,300,360,420,480,540 |
| 520                   | 60,90,120,180,240 |
| 545                   | 10,30,60,120,180,240,300,360,420,480,540,600 |
| 570                   | 60,90,120,180,240,300,360 |
| 600                   | 30,60,120,180,240,300,360,420 |

3. Materials and Methods

3.1. Age Hardening Characteristics of Alloys

After the hardness test of the sample, the hardness-time curves of the alloy at different aging temperatures can be drawn as shown in Figure 2. In Figure 2, the hardness-time variation curve of the
alloy at different aging temperatures is shown. It can be seen from the figure that the aging hardening response speed of alloys at low temperatures (300°C and 400°C) is significantly lower than that at the medium and high temperatures. Among them, the value of hardness after aging at 300°C for different times fluctuates between 255 and 270 HV, which can be considered almost unchanged.

The samples were tested by XRD diffraction, and the test results are shown in Figure 3. It can be seen from Figure 3 that the types of phases in the alloy and the diffraction intensity of α phase do not change much. Figure 4 is the microstructure of the alloy after aging for different time. Compared with 10h, the amount of precipitation of α phase near the grain boundary increases after aging for 48h, and the no precipitate-free regions is more obvious. However, the amount of precipitation of α phase is still less.

Figure 2 Hardness-time variation curve of the alloy at different aging temperatures
(a) low temperature (b) middle-high temperature

Figure 3 XRD Test Results of Alloy Aged at 300 °C for Different Time
The reason for this phenomenon is that the aging temperature of 300 °C was relatively low, the degree of under cooling was relatively small, and the driving force for the nucleation was relatively low, and the inside of the alloy did not meet the conditions for the precipitation of new phases. When the aging temperature was increased to above 450 °C, the aging hardening response speed of the alloy has increased significantly, indicating that the temperature range of 450 °C-600 °C was the suitable aging temperature range of the alloy.

It can be seen that as the aging temperature increased, the peak hardness that the alloy could reach gradually decreased. This was because the higher the aging temperature, the α phase precipitated by the aging was easier to coarsen, resulting in the weakening of the aging effect. With the extension of the aging time, there were two situations in the hardness after aging. One was gradually becoming flat. For example, at the aging temperature of 500 °C, the hardness after 300 min tended to fluctuate within a certain range. Another situation was that over-aging occurred when reaching the peak hardness, and the hardness decreased with time. For example, at the aging temperature of 450 °C, the peak hardness was reached at about 600 minutes; at the aging temperature of 545 °C and 600 °C, the time to reach the peak hardness was about 300 minutes.

Within the experimental temperature range, in addition to the poor aging strengthening at the lowest temperature (300 °C) and the highest temperature (600 °C), even at a lower temperature of 400 °C, prolonging the aging time was able to produce a good aging strengthening effect, and there was no much different of the peak hardness at 450 °C, 500 °C, 520 °C, 545 °C and 570 °C. Above all, the alloy has a wide temperature window for aging and good aging strengthening effect, making it an ideal structural material.

3.2 Drawing of TTT curve

Since the precipitation amount of α phase will affect the hardness of the alloy, the hardness can reflect the change of the precipitation amount of the phase to a certain extent. At the same time, considering
the error range of the hardness combined with the microstructure of the alloy, the TTT curve of the alloy is plotted as shown in Figure 5:

![Figure 5 The TTT curve of alloy](image)

Generally speaking, when isothermal aging at a higher temperature (e.g. the experimental temperature of 600°C in this paper), the diffusion rate of solute atoms was relatively fast, but the degree of under cooling was small, and the driving force for phase variation was insufficient, leading to a slower phase transition speed. When processing at a low temperature (e.g. 450 °C), the diffusion rate of solute atoms was small and the transition speed was relatively slow. While when processing at the intermediate temperature (500–550°C), both the driving force of semi-stable β phase decomposition and the diffusion rate of solute atoms had relatively good matching effects on the phase transition process, so the phase transition speed also reached the extreme in this temperature range[8]. Therefore, the graph was showing a "C" shape, it can be observed from the Figure 5 that the alloy's nose temperature was about 545°C.

4. Conclusion
In this paper, Ti-6Cr-5V-5Mo-4Al-1Nb alloy was used as the research object. The TTT curve of the alloy was measured by hardness method. The microstructure changes of the alloy during isothermal process were analyzed by XRD and SEM. The main conclusions are as follows:

(1) In addition to the slow aging at 300°C and 400°C, the alloy has high aging response speed at other aging temperatures
(2) When the aging time is prolonged at medium and low temperatures, the alloy can also produce the same aging strengthening effect as that at medium temperature aging, indicating that the temperature window of the alloy aging is relatively broad, the aging strengthening effect is good, and it has the operability of heat treatment strengthening, so it is an ideal structural material.
(3) The isothermal aging transformation curve of the alloy is "C" type, and it can be obtained from the figure that the nose temperature is about 545 °C.

Acknowledgements
This paper is one of the phased achievements of the National Key R&D Program of China of the National Social Science Fund ( 2017YFB0306204 ). The authors is grateful to the Fund for its support.

References
[1] Nyakana S L, Fanning J C, Boyer R R. (2005) Quick reference guide for β titanium alloys in the 00s[J]. Journal of Materials Engineering and Performance, 14(6): 799-811.
[2] Banerjee D, Williams J C. (2013) Perspectives on titanium science and technology[J]. Acta Materialia, 61(3): 844-879.
[3] Boyer R R, Briggs R D. (2005) The use of β titanium alloys in the aerospace industry[J]. (2005)
Journal of Materials Engineering and Performance, 14(6): 681-685.

[4] Leyens, C, Peters, M. (2003) Titanium and titanium alloys: fundamentals and applications[M]. New Jersey: John Wiley & Sons

[5] Li C L, Yu Y, Hui S X, et al. (2010) Determination of TTT curve of Ti-6554 titanium alloy [J]. Journal of Nonferrous Metals of China, 20(S1): 560-564.

[6] Chang H, Zhou L. (2006) $\beta \rightarrow \alpha + \beta$ isothermal transformation kinetics of Ti-B19 titanium alloy [J]. Rare metal materials and engineering, 35(11): 1695-1699.

[7] Chen F, Xu G, Zhang X, et al. (2017) Isothermal kinetics of $\beta \leftrightarrow \alpha$ transformation in Ti-55531 alloy influenced by phase composition and microstructure[J]. Materials & Design, 130: 302-316.

[8] Wang Q J, Sun Y, Shuang Y X, et al. (2019) Ageing mechanism and phase transformation kinetics of new $\beta$ titanium alloy [J]. Rare metals, 43(1): 1103-1108.

[9] Naveen M, Santhosh R, Geetha M, et al. (2014) Experimental study and computer modelling of the $\beta \rightarrow \alpha + \beta$ phase transformation in Ti15-3 alloy under isothermal conditions[J]. Journal of Alloys & Compounds, 616: 607-613.