Research Article

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Effects of solution and aging treatment parameters on the microstructure evolution of Ti–10V–2Fe–3Al alloy

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Abstract: The solution-aging treatment parameters, including solution temperature, cooling rate and aging temperature, have significant influences on the microstructures and comprehensive mechanical properties of titanium alloy. In this work, the detailed microevolution behaviors of Ti–10V–2Fe–3Al alloy under different solution and aging conditions have been investigated through a series of heat-treatment experiments. The results of solution-treatment experiments reveal that the content of α_p -phase is reduced to zero as the solution temperature is raised to a certain $\alpha \rightarrow \beta$ critical transformation point. Recrystallized β -grains can be observed at the solution temperature of 820°C. In addition, the cooling way (air cooling or water cooling) has little influence on the microevolution behaviors for this alloy during the solution-treatment process. As for the solution-aging-treatment experiments, the results reveal that α_s -phases are precipitated from the

supersaturated β -phase, and the fraction of α_s -phase increases with increasing aging temperature. However, the precipitated α -grains intend to coalesce and coarsen as the aging temperature raises above 510°C. Therefore, the advocated solution-aging-treatment program is solution treatment at 820°C with air cooling followed by aging treatment at 510°C with air cooling.

Keywords: heat treatment, titanium alloy, microstructure evolution

1 Introduction

Titanium and its alloys have been extensively applied in aeronautics, astronautics and marine industry due to their excellent fracture toughness, good corrosion resistance and high strength-to-weight ratio [1,2]. Usually, the mechanical performance of titanium alloy components mainly depends on their microstructure features. Therefore, the desired combination of mechanical properties of titanium alloys can be obtained through adjusting the microstructures by means of suitable heat treatment processes such as solution-aging treatments [3,4], annealing treatments [5,6], etc.

Up till now, many researches have paid attention to the heat treatment process of titanium alloy. For example, Chong et al. [7] found that the refined β -grains can be obtained from fully martensite microstructures in a Ti–6Al–4V alloy by rapid heat treatment. Qiang et al. [8] have obtained the nanoscaled lamellar structure in an as-cast titanium alloy by the multistep heat treatment method. Ren et al. [9] investigated the aging process of Ti-5321 alloy and found that the lamellar α_s -phase coarsens with increasing aging temperature. By now, most researches only focused on the microevolution of single phase (α -phase or β -phase) during the heat treatment process of titanium alloy. Few studies have focused on the comprehensive microevolution regulation

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of titanium alloy during the heat treatment process, especially for Ti-10V-2Fe-3Al alloy.

Ti-10V-2Fe-3Al alloy, also called TB6 alloy, has inspired broad interests in the academic community and industry. It was firstly developed by TIMET Company in the 1970s to provide weight savings over Ti-6Al-4V alloy or high-strength steels in the forged aircraft components, such as the landing gear system of airplanes in A380 and Boeing 777 [10]. As a near β -titanium alloy, it is well known that the high strength of this alloy comes from the microstructure of precipitated fine α -phase particles (platelets) uniformly distributed on the β -phase matrix, which can be obtained by solution-aging treatment. During the heat treatment process, the β -phase may be transformed into primary α -phase (α_p), secondary α -phase (α_s), metastable ω -phase, etc. The pseudobinary phase diagram (Figure 1) can provide an accessorial analysis for the microevolution process of titanium alloy during the heat treatment process. In Figure 1, there are two types of phase transformation process corresponding to rapid cooling and relatively slow cooling way, respectively. When titanium alloy is cooled from the β -phase field to room temperature with a relatively slow cooling rate, the phase diagram made up of black lines is available. In other words, the phase diagram constructed with black lines can provide a reference for the phase transformation process as the cooling rate is relatively slow (like furnace cooling or air cooling). Correspondingly, as the cooling rate is rapid enough, the phase diagram constructed with blue lines is available. The M_s and M_f lines represent the start and end point of martensite

transformation, respectively. As the content of β -stabilizing element is near to the intersection of M_s line and X-axis, the quenched ω phase may be generated. For TB6 alloy, the saturated β -phase will be transformed into $\alpha + \beta$ phase at a relatively low cooling rate. The α' -phase or ω -phase may be generated under the condition of rapid cooling rate. In general, the kind and content of decompositions depend on the solution temperature, aging temperature and cooling rate. The morphology of α_p -phase can be adjusted by giving appropriate strain in the $\alpha + \beta$ phase field, and the morphology and distribution of α_s - or ω -phase can be altered by adjusting the heat treatment temperature and time [11].

In view of the complexity and the variety of microstructure transformation during the heat treatment process, a comprehensive evaluation of the microstructure evolution behaviors of this alloy is a critical issue worthy to be investigated. Therefore, the aims of this study are to investigate the phase transformation of TB6 alloy under different solution and aging treatment conditions as well as to find an advocated solution-aging-treatment program to provide production guidance.

2 Material and experiments

The material investigated in the present study is a commercial Ti-10V-2Fe-3Al alloy with chemical compositions (wt%) of 0.041C-0.0538Si-5.095V-1.147Fe-5.087Al-5.303Mo-1.1Cr-0.005Zr-(bal.)Ti. Sixteen cylindrical specimens with 10 mm in diameter and 12 mm in length were separated from an as-rolled rob by electric spark linear cutting. The original microstructures of these specimens are shown in Figure 2. In order to investigate the effects of solution temperature and cooling way on microstructure transformation of this alloy, the first batch of specimens was solution-treated at varying temperatures and different cooling ways. Considering the phase transformation temperature of this alloy as $805 \pm 25^\circ\text{C}$ (varying with the content of β -stabilizing element) [12], the solution temperature was set as 700, 760, 820 and 880°C . For the sake of ensuring the atoms diffuse sufficiently, these specimens were held at a specific solution temperature for 2 h. Subsequently, these specimens were cooled to room temperature by water or air to analyze the impact of cooling way on microstructures. The concrete experimental program of the solution treatment process has been listed in Table 1. All the solution-treated specimens were

Figure 1: Pseudobinary phase diagram of titanium alloy.

Figure 2: Original microstructures for TB6 alloy.

sectioned paralleled to the central axis, and the sectioned surfaces were grinded and polished on an automatic grinding-polishing machine. Finally, these treated surfaces were etched with 2% HF + 4% HNO₃, and the microstructures were characterized by conventional optical microscopy.

In order to further investigate the effects of solution-aging treatment on microstructures of this alloy, the remaining specimens were divided into two groups. In the first group, the specimens had been solution-treated first at different solution temperature. Then, they were aging-treated under a consistent aging temperature. For the second group, in order to investigate the effects of aging temperature on microstructures of this alloy, the specimens were solution-treated first at a consistent solution temperature, and then, they were aging-treated at different aging temperatures. During the aging treatment process, all the specimens were soaked in specific temperatures for 8 h to ensure the atoms diffuse sufficiently. The concrete experimental program of the solution-aging treatment process has been listed in Table 2. All the solution-aging-treated samples were characterized by the same way as mentioned before. The instrument applied in these heat-treatment experiments is a box-type atmosphere furnace MXQ1400-30.

3 Results and discussion

3.1 Effects of solution treatment on the microstructures of TB6 alloy

3.1.1 Microstructure evolution of TB6 alloy at various solution temperatures

The microstructures of solution-treated specimens with water-cooling way are shown in Figure 3. In Figure 3(a), the microstructures consist of equiaxed β -grains, basket-weave α_p -phases and lamellar α_p -phases. As the solution temperature is raised to 760°C, most of the lamellar α_p -phases are dissolved into the β -phase, and some newly formed α_{GB} phases appear around the primary β -grain boundaries, which infers that basket-weave α_p -phases are more stable than lamellar α_p -phases. According to Hua et al. [13], the α_p -phases intend to be formed at the β -grain boundaries because the β -grain boundaries can offer more habit plates for the nucleation and growth of α -phases. Comparing Figure 3(a) and (b), it can be seen that the β -grains in Figure 3 are “cleaner” than those in Figure 3(a), which means a plenty of basket-weave α_p -phases have been dissolved into the β -phase at higher solution temperatures. According to Figure 3(c), as the solution temperature is increased to 820°C, the

Table 1: Experiment program of solution treatment for TB6 alloy

Number	Solution temperature/°C	Holding time/h	Way of cooling	Number	Solution temperature/°C	Holding time/h	Way of cooling
1	700	2	ac	5	820	2	ac
2	700	2	wc	6	820	2	wc
3	760	2	ac	7	880	2	ac
4	760	2	wc	8	880	2	wc

Table 2: Experimental program of solution-aging treatment for TB6 alloy

Number	Solution temperature/°C	Holding time/h	Way of cooling	Aging temperature/°C	Holding time/h	Way of cooling
9	700	2	ac	510	8	ac
10	760	2	ac	510	8	ac
11	820	2	ac	510	8	ac
12	880	2	ac	510	8	ac
13	820	2	ac	480	8	ac
14	820	2	ac	510	8	ac
15	820	2	ac	540	8	ac
16	820	2	ac	570	8	ac

microstructure only consists of fine and equiaxed β -grains. The temperature of 820°C is the phase transformation temperature, and meanwhile, it is the recrystallized temperature of β -phase. As the solution temperature is further increased to 880°C, the β -grains begin coalescing and coarsening rapidly, and the quantity of grain boundaries begins to reduce. In summary, when the solution temperature decreases below 820°C, the content of the dissolved α_p -phases increases with increasing solution temperature, and the α_p -phases intend to be gathered at the β -grain boundaries. When the solution temperature reaches 820°C, the α_p -phases dissolve completely and then recrystallized β -grains

appear. As the solution temperature raises above 820°C, the β -grains begin coarsening rapidly.

It is well known that the solution treatment of titanium alloy can promote the strength and hardness at the cost of reduction in plasticity and fracture toughness, while the solution-aging treatment will provide an excellent comprehensive mechanical property for the titanium alloy components. Thus, the heat-treatment process of titanium alloy will generally include solution and aging process, and the solution-treatment process can offer various microstructures for the following aging process. As for this titanium alloy, the advocated solution temperature is 820°C where fine saturated

Figure 3: Microstructures of sample solutions treated at different temperatures with water cooling: (a) 700°C, wc; (b) 760°C, wc; (c) 820°C, wc and (d) 880°C, wc.

β -grains can be obtained. As for the impact of various solution-treated microstructures on the microevolution of titanium alloy during the subsequent aging process, the following sections will discuss it in detail.

3.1.2 Microstructure evolution of TB6 alloy under different cooling ways

The microstructures of samples solution-treated under the air-cooling condition are shown in Figure 4. Comparing Figure 4 with Figure 3, it can be found that the microstructures of samples solution treated with air cooling are more uniform and clear than those that are treated with water cooling. This may result from the relatively slow cooling rate which provides enough time for the growth of new phases. In Figure 4(a), there are a lot of α_p -phases distributed on β -grains and around β -grain boundaries (α_{GB} -phases), while the lamellar α_p -phases cannot be observed. With increasing solution temperatures, the content of α_{GB} -phases and α_p -phases reduces, and a part of basket-weave α_p -phases intend to be spheroidized (Figure 4b). When the solution temperature raises above the phase transformation point, all the α -phases are dissolved into β -matrix (Figure 4c and d).

In general, the difference in microevolutions of TB6 alloy under the air-cooling and water-cooling conditions is minor. As for the air-cooling condition, the microstructures are more uniform because the air-cooling way can provide sufficient time for the homogenization of the solution microstructures. In addition, according to the phase diagram (Figure 1), α' phase may be formed at the condition of water cooling. It is well known that the existence of α' phase has negative impacts on the plasticity and fracture toughness of the material. Therefore, the air-cooling way is a better option to obtain desirable microstructures.

3.2 Effects of solution-aging treatment parameters on the microevolutions of TB6 alloy

3.2.1 Varying solution temperatures and fixed aging temperature

During the aging treatment process of titanium alloy, the second phase (strengthening phase) particles will be precipitated from the saturated β -phase. The second

Figure 4: Microstructures of sample solutions treated at different temperatures with air cooling: (a) 700°C, ac; (b) 760°C, ac; (c) 820°C, ac and (d) 880°C, ac.

phase particles can hinder the migration of dislocation, which results in the promotion of strength, and meanwhile, it will have a little influence on the plasticity and fracture toughness of the material. Figure 5 revealed the microstructures of specimens solution-aging-treated under different solution temperatures and consistent aging temperatures. In Figure 5a, there are lamellar α -phases and precipitated α -phases (α_s) within β -grains. With the increasing solution temperature, more and more α_p -phases are dissolved into β -grains during the solution process, which results in the increase of β -stabilizing element. Accordingly, the content of the precipitated α_s -phases increases. In addition, according to Figure 5b, it can be found that a part of α_s -phases intend to be congregated along with the β -grain boundaries. This phenomenon is more obvious in Figure 5c. As the solution temperature is further increased to 880°C, although all the α_p -phases have been completely dissolved into β -grains during the primary solution process, the content of α_s -phases is still small. According to Zheng [14], the α_s -phases can be formed at the special plates of β -grains. And, grain boundaries can provide various lattice arrangements. Thus, the abnormal phenomenon may be a result of relatively few β -grain boundaries in Figure 5(d), which

cannot provide enough sites for the precipitation of α_s -phases. According to the solution-treated microstructures, it can be found that the recrystallized β -grains appear as the solution temperature is 820°C. The density of β -grain boundaries has been increased rapidly. It is well known that the new phases intend to nucleate and grow at the grain boundaries. As the solution temperature is raised to 880°C, the β -grains grow up rapidly. The density of β -grain boundaries is reduced, which results in the reduction of nucleation sites of α_s -phases. Therefore, in order to obtain fine and uniform microstructures after aging treatment, the primary solution microstructures must be fine saturated β -grains, and the solution temperature of 820°C can meet the requirement.

3.2.2 Fixed solution temperature and varying aging temperature

It is well known that not only the primary solution microstructures have great impact on the microevolution of aging samples, but the aging temperature is a key factor to decide the final microstructures of the aging samples. Hence, the effects of aging temperature on the microevolution of this titanium alloy are worthy to be

Figure 5: Microstructures of sample solutions aging-treated at solution temperatures of (a) 700°C, (b) 760°C, (c) 820°C and (d) 880°C and the aging temperature of 510°C.

Figure 6: Microstructures of sample solutions aging-treated at the solution temperature of 820°C and aging temperatures of (a) 480°C, (b) 510°C, (c) 540°C and (d) 570°C.

investigated. Here, in order to keep the other variants consistent, the specimens to be aging-treated have been solution-treated at the solution temperature of 820°C.

Figure 6 revealed the results of solution-aging treatment at the solution temperature of 820°C and aging temperatures of 480°C, 510°C, 540°C and 570°C. Apparently,

as the aging temperature increases, more α_s -phases are precipitated. In Figure 6a, it can be seen that only a few acicular α_s -phases appear at β -grain boundaries. As the aging temperature increases, the acicular α_s -phases intend to be transformed into short-rod α_s -phases, and meanwhile, more acicular α_s -phases are precipitated from the saturated β -grains (Figure 6b). With the aging temperature further increased to 540°C, fine and diffusive α_s -phase particles are precipitated from the β -matrix. When the aging temperature is reached to 570°C, the α_s -phases begin to coalesce and coarsen rapidly, which leads to the nonuniform distribution of α_s -phases (Figure 6d). Besides, there are some bright fields in Figure 6d. These phases are precipitated and gathered α_s -phases. They are different from the diffusive α_s -phases precipitated from the saturated β -grain. These α_s -phases are intended to nucleate at the original β -grain boundaries, and they grow along a special direction. According to reference [14], only nucleation on the habit planes and growth along with the packed direction, the special α_s -phases (a kind of α_{GB} -phases) can be generated. Because the content of the α_{GB} -phases is small, they will have little influence on the final mechanical properties of titanium alloy components. Therefore, it is a reasonable and effective retort to ignore the influences of the special α_{GB} -phases.

Actually, the coarse α_s -phases have a negative influence on the plasticity and fracture toughness of titanium alloy. Because α -phase with hcp structure has only three slip systems, the coordination of deformation is difficult. Thus, the locally coarse α_s -phases will intend to result in stress concentration. Through the analysis of the aging-treated microstructures, the aging temperature of 540°C is the desired aging temperature, where fine and uniformly distributed α_s -phases can be obtained.

4 Conclusions

The microstructures of solution- and aging-treated samples at different solution temperatures, cooling rate and aging temperature have been analyzed, and the following conclusions can be drawn.

- (1) The microstructures of TB6 titanium alloy can be adjusted by solution treatment. As the solution temperature decreases below 820°C, the content of lamellar α_p -phases reduces and basket-weave α_p -phases intend to be spheroidized with increasing solution temperature. When the solution temperature is further increased to 820°C, the recrystallized

β -grains can be observed. As the solution temperature raises above 820°C, the β -grains begin coarsening rapidly. Consequently, the desired solution temperature should be set at 820°C where fine and supersaturated β -grains can be obtained.

- (2) The differences of microevolution under air cooling and water cooling are minor. The microstructures of samples treated under the air-cooling condition are more uniform and clear than those that are treated with water cooling.
- (3) Aging temperature has a significant influence on the microstructural evolution of TB6 alloy. During the aging process, more α_s -phases are precipitated from the supersaturated β -grains with increasing aging temperature. However, as the aging temperature raises above 540°C, the precipitated α_s -phase particles begin coalescing and coarsening rapidly.
- (4) For this alloy, the advocated solution-aging treatment program is solution treatment at 820°C with air cooling followed by aging treatment at 510°C with air cooling. Under such solution-aging-treatment condition, the uniform and diffusive strengthening phase particles are distributed on equiaxed and fine β -grains.

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