



Effect of solution plus aging heat treatment on microstructural evolution and mechanical properties of near- β titanium alloy

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Received 12 June 2018; accepted 8 January 2019

Abstract: The microstructural evolution, mechanical properties and fracture mechanism of a Ti-5Al-5Mo-5V-3Cr-1Zr (Ti-55531) alloy after solution (760–820 °C) plus aging (580–640 °C) treatments were investigated. The results show that the volume fraction of the primary α (α_p) phase decreases with the increase of solution temperature, and the length of the secondary α phase (α_s) decreases while its width increases with the increase of aging temperature. Yield and tensile strengths decrease with the increase of solution temperature, while increase with the increase of aging temperature. A good balance of tensile strength and ductility of the alloy is obtained under solution of 800 °C for 2 h plus aging of 640 °C for 8 h, in which the tensile strength is 1434 MPa and the elongation is 7.7%. The coarsening α_s phase makes crack propagation paths deflected and tortuous, which increases the crack propagation resistance and improves the ductility and fracture toughness.

Key words: Ti-5Al-5Mo-5V-3Cr-1Zr titanium alloy; hot treatment; solution; aging; microstructural evolution; mechanical properties; fracture mechanism

1 Introduction

Near- β titanium alloys have been widely used in the fields of aeronautics, astronautics and marine owing to their advantages of high specific strength ratio, excellent fracture toughness and good corrosion/oxidation resistance [1–3]. Compared with other near β titanium alloys, Ti-5Al-5Mo-5V-3Cr-1Zr (Ti-55531) is a relatively new near- β titanium alloy [4,5]. This alloy has some advantages of good hardenability, excellent strength and high fracture toughness, thus it becomes a suitable candidate for manufacturing the high strength airframe forgings [2,3]. In the aerospace industry, proper microstructures, excellent properties and good dimensional stability are required. To meet these requirements, the forgings are subjected to various heat treatments. During the heat treatment processes, the material has a complex microstructural evolution.

Moreover, its mechanical properties are sensitive to a minor variation of microstructures. Therefore, it is essential to clarify the relations among heat treatments, microstructures and mechanical properties.

CHEN et al [4] investigated the phase transformation between the α and β phases. They established a kinetics model of the α phase growth and dissolution. MANSHAD and DIPPENAR [6] studied the morphological evolution of α phase in a Ti-5553 alloy. They found that α phase was closely related to the aging temperature and time. CHEN et al [7] found that the finer acicular secondary α (α_s) phase can precipitate in β matrix during an aging stage. The size of α_s phase increased with the increase of aging temperature, and finally influenced the strength and ductility. According to the researches of ZHU et al [8] and ZHU et al [9], the morphology of α_s phase mainly depended on solution temperature. The lath α phase can improve the strength and plasticity, while the acicular α phase can reduce the

Foundation item: Project (SKLSP201853) supported by the Fund of the State Key Laboratory of Solidification Processing in NWPU, China; Project (51625505) supported by the National Science Fund for Distinguished Young Scholars of China; Project (U1537203) supported by the Key Program Project of the Joint Fund of Astronomy and National Natural Science Foundation of China; Project (KYQD1801) supported by the Scientific Research Foundation of Tianjin University of Technology and Education, China

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DOI: 10.1016/S1003-6326(19)65008-9

strength with no loss of ductility. DU et al [10] investigated the microstructural evolution and mechanical properties of a new β titanium alloy during solution plus aging treatment. Their results showed that the size and volume fraction of α_s phase were sensitive to temperature. ZHANG et al [11] studied the morphology evolution of the α_s phase of a Ti-7Mo-3Al-3Nb-3Cr alloy during isothermal heat treatment. ZHAO et al [12] showed that the phase boundaries of α_s phase increased with the increase of annealing temperature, while the volume fraction of primary α (α_p) phase decreased. XU et al [13] studied the microstructural evolution and tensile properties of a Ti-5553 alloy during solution plus aging treatment. They showed that the β solution treatment plus aging resulted in high strength, and the $\alpha + \beta$ solution treatment plus aging resulted in good combination of strength and ductility.

Although there has been some work on the same or similar alloys, the relation among the heat treatments, microstructural evolution and mechanical properties of the Ti-55531 alloy has not been studied exhaustively and systemically. Therefore, in this work, the microstructural evolution of the Ti-55531 alloy during heat treatments was studied, and their morphologies, sizes, volume fraction, precipitation location and growth modes were analyzed. Subsequently, the strength, ductility and fractography were evaluated. Finally, the effect of heat treatment on the mechanical properties and fracture mechanism was discussed.

2 Experimental

The as-received Ti-55531 alloy has a composition (in wt.%) of 5.21 aluminum (Al), 5.06 molybdenum (Mo), 4.96 vanadium (V), 3.01 chromium (Cr), 1.07 zirconium (Zr), 0.25 iron (Fe), 0.12 others, and balanced Ti. The β -transus temperature is 845 °C. The initial microstructure is shown in Fig. 1(a), which consists of a fine, bimodal $\alpha + \beta$ structure of approximately 45% (volume fraction) equiaxed α_p phase (denoted by white arrow) and 55% transformed β phase, where the α_p phase has an average grain diameter of 2–3 μm .

To investigate the effects of heat treatment on the microstructural evolution and mechanical properties, cubic (dimensions of 15 mm in length, 13 mm in width and 5 mm in depth) and standard tensile samples (see Fig. 1(b)) were electro-discharged from the received material. These samples were firstly solution-treated at 760, 780, 800 and 820 °C for 0.5, 2 and 4 h, respectively. Then, the samples solution-treated at 800 °C for 2 h were subjected to aging treatment at 580, 600, 620 and 640 °C for 6, 8 and 10 h, followed by air cooling (corresponding

to cooling rate of 100 °C/min) [14]. The detailed heat treatment procedures are shown in Table 1.

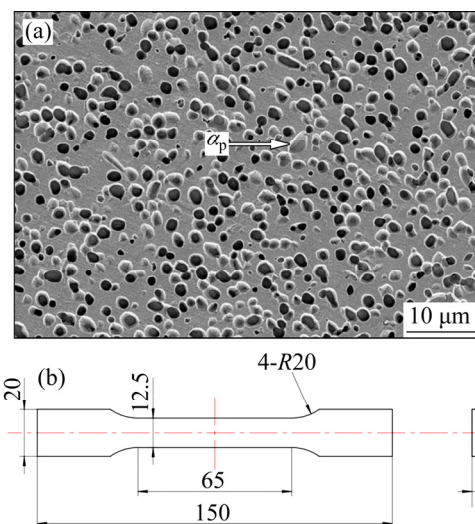


Fig. 1 Initial microstructure of as-received Ti-55531 alloy (a) and sizes of standard tensile sample (unit: mm) (b)

Table 1 Solution treatment and solution (800 °C, 2 h) plus aging treatment parameters of Ti-55531 alloy

Solution temperature/°C	Solution time/h
760, 780, 800, 820	0.5, 2, 4
Aging temperature/°C	Aging time/h
580, 600, 620, 640	6, 8, 10

Cooling methods for solution and aging treatment are air cooling

After the heat treatment, the cubic samples were metallographically polished and etched with a solution of 1.5 mL hydrofluoric acid + 3 mL nitric acid + 100 mL water for 10–15 s. The OLYMPUS/PMG3 optical microscope (OM) and Zeiss Sigma 500 scanning electron microscope (SEM) were used to observe the microstructure. Quantitative measurements of grain size and volume fraction of each phase were conducted.

The standard tensile samples were tested using an MTS810 material test system (according to standard GR/T 228–2002). After the tensile tests, the yield strength ($R_{p0.2}$), tensile strength (R_m), breaking strength (R_b), uniform elongation (A_u) and failure elongation (A_f) were measured. The fractography was analyzed by the SEM to identify the fracture mechanism.

3 Results and discussion

3.1 Microstructural evolution

3.1.1 Effect of solution temperature and time

Figures 2(a) to (d) show the SEM micrographs of the Ti-55531 alloy at solution temperatures of 760, 780, 800 and 820 °C for 2 h, respectively. The α_p grains are

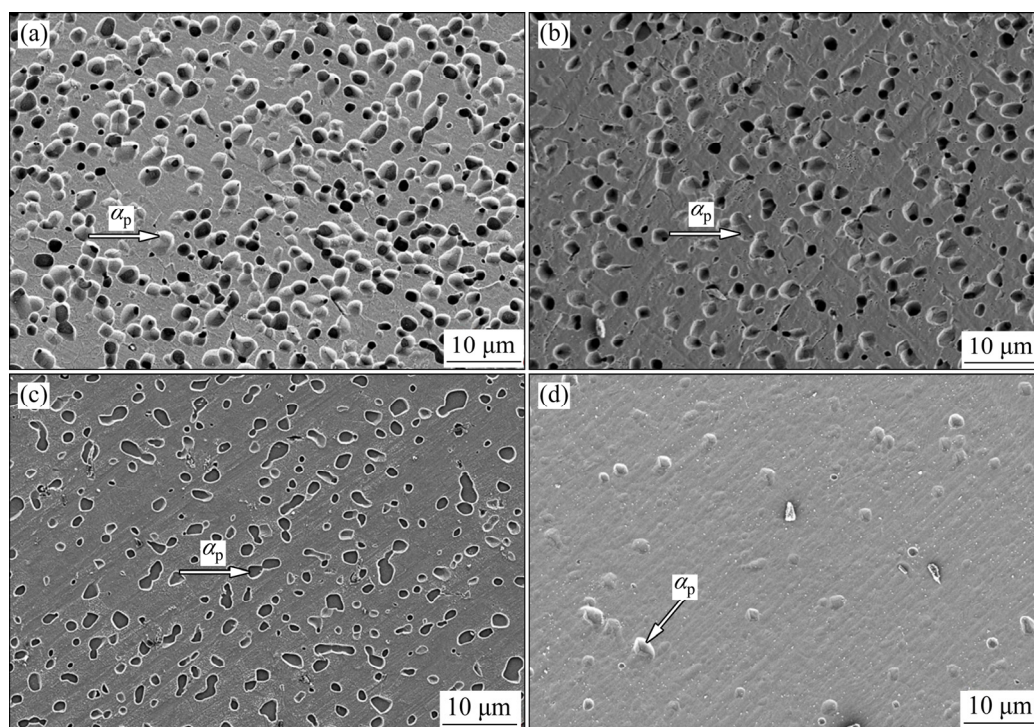


Fig. 2 Microstructural evolution of Ti-55531 alloy solution-treated at different temperatures for 2 h followed by air cooling: (a) 760 °C; (b) 780 °C; (c) 800 °C; (d) 820 °C

equiaxed and their boundaries are straight. The static coarsening of the α_p phase is not apparent. This is because the content of β -stabilizing elements is higher and Mo equivalence of the alloy is about 12.8 [10], which has a significant dragged effect on the grain coarsening [15–17].

At 760 °C (see Fig. 2(a)), the volume fraction of the α_p is reduced to 35%–40% compared with the initial value of 45% (Fig. 1(a)). At 800 °C, its volume fraction decreases sharply (Fig. 2(c)). Most of the α_p phase is dissolved into the matrix. At the solution temperature of 820 °C (Fig. 2(d)), most of the α_p phase disappears. The volume fraction of the α_p phase is less than 5%.

Figure 3 shows the SEM micrographs of the Ti-55531 alloy solution-treated at 780 °C for 0.5–4 h. The α_p phase still keeps equiaxed and its grain size has no change. However, the volume fraction of α_p phase has a little change with the solution time. The volume fraction is 35%–40% at 0.5 h (Fig. 3(a)). With increasing solution time to 4 h (Fig. 3(c)), this value is reduced to 30%.

3.1.2 Effect of aging temperature and time

The Ti-55531 alloy was solution-treated at 800 °C for 2 h, followed by aging at 580–640 °C (20 °C interval) for 6–10 h (2 h interval). Figures 4(a₁, a₂) display the microstructures aging-treated at 580 °C for 8 h. It is seen that the equiaxed α_p phase is around the original grain

boundaries. The lamellar α_s phase precipitates inside the β grains. The α_s phase is cross-distributed irregularly and the orientation is random, thus the orientation is different from one to other. The precipitation process usually consists of two stages, nucleation and growth. When the cooling rate is less than 100 °C/min, the α_s phase preferentially nucleates at the β/β grain boundaries (GB) or the α/β interface (PI), because these zones have high energy for the α_s nucleation [11]. In this study, the samples were cooled in air, which produced a high driving force for the α_s nucleation. At aging temperature of 600 °C (Figs. 4(b₁, b₂)), the α_p phase still has no change, but the α_s/β_r (retained beta phase) interface becomes clearer, obvious coarsening of α_s lamella takes place. At 620 °C (Figs. 4(c₁, c₂)) and 640 °C (Figs. 4(d₁, d₂)), the coarsening phenomenon is more pronounced, the width of α_s phase increases while the interlamellar spacing decreases.

The coarsening of lamellar α_s phase is a thermally activated process, and the coarsening rate depends on the diffusivity of solute atoms. Therefore, the coarsening of the α_s phase is more pronounced at higher temperatures. Two mechanisms, namely boundary splitting and termination migration, can interpret the coarsening, which are graphically illustrated in Figs. 5 and 6 [15], respectively. During the initial stage, the boundary splitting mechanism is dominant. Some defect zones

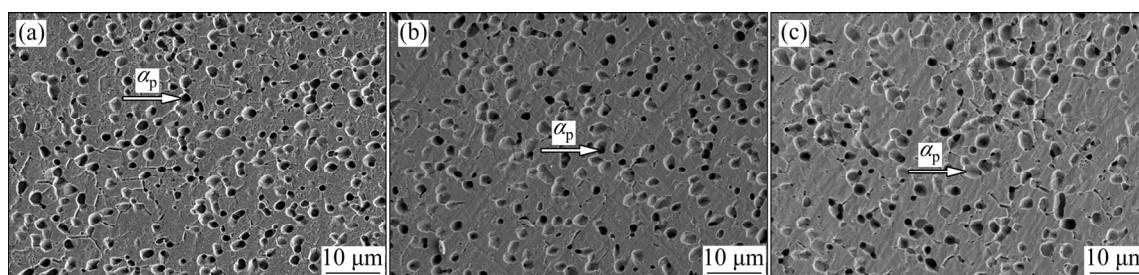


Fig. 3 Microstructural evolution of Ti-55531 alloy solution-treated at 780 °C for different time followed by air cooling: (a) 0.5 h; (b) 2 h; (c) 4 h

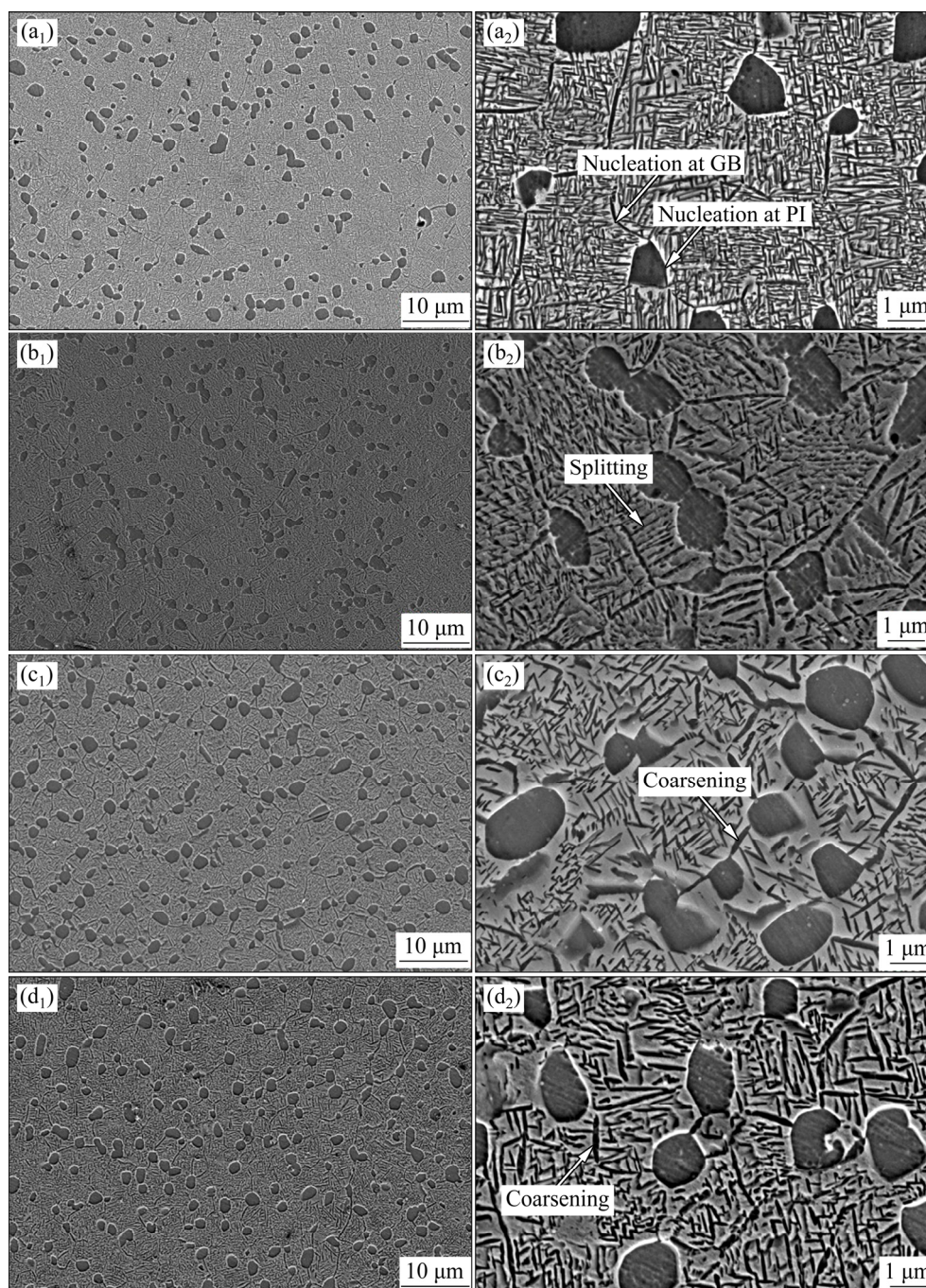


Fig. 4 Microstructural evolution of Ti-55531 alloy aging treated at 580 °C (a₁, a₂), 600 °C (b₁, b₂), 620 °C (c₁, c₂) and 640 °C (d₁, d₂) for 8 h followed by air cooling

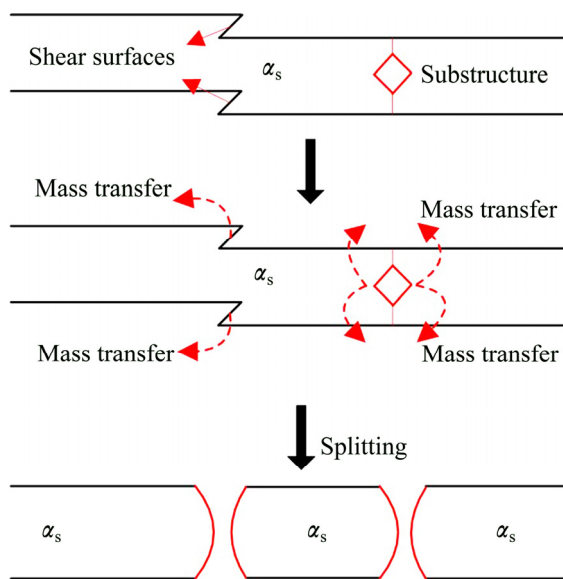


Fig. 5 Illustration of boundary splitting mechanism [15]

such as shear surfaces and substructures are produced inside the α_s phase. The mass transfers from these zones to adjacent flat surfaces to reduce surface energy. Therefore, the nature of the boundary splitting is the fade away of inside boundary of α phase and incuneation of β phase [15,17]. Then, the lamellar α_s phase is split and begins to coarsen.

The termination migration is another important mechanism to explain the coarsening. This theory was firstly proposed for the coarsening behavior of pearlite in steels [18,19], which has been successfully applied to explaining the coarsening of lamellar α_s phase in titanium alloys [20,21]. For the lamellar α_s phase, the curvature in the termination location is higher than that in other flat locations, which results in a higher potential energy at these positions. In this case, a chemical potential curvature can be formed between the termination and other flat position, which drives the solute atoms to migrate from the termination to other place, finally resulting in the dissolution of termination tips and the coarsening of the lamellar microstructure [15].

Figure 7 shows the microstructural evolution of the Ti-55531 alloy at different aging time. The α_s

phase displays the boundary splitting and subsequent coarsening behavior. At 6 h (Figs. 7(a₁, a₂)), the equiaxed α_p phase precipitates around the β grain boundaries, the lamellar α_s phase is arranged randomly inside the β grains, and the α_s phase boundaries are not clear. At the aging time of 8 h (Figs. 7(b₁, b₂)), the lamellar α_s phase evolves into a short rod-like structure. The length and width of the short rod-like α_s phase decrease and increase, respectively, compared with the initial values. At 10 h, the coarsening phenomenon is more pronounced, which is displayed in Figs. 7(c₁, c₂).

3.2 Mechanical properties

The mechanical properties of the Ti-55531 alloy under different heat treatments are listed in Tables 2 and 3. It is seen that the mechanical properties are sensitive to temperature and time. The variation of yield strength ($R_{p0.2}$), tensile strength (R_m) and elongation with temperature and time is graphically illustrated in Figs. 8 and 9, respectively. The yield strength $R_{p0.2}$ and tensile strength R_m increase with the decrease of solution temperature, as shown in Fig. 8(a), while these values decrease with the solution time. This phenomenon may be related to the α_p dissolution and to the β phase coarsening during the solution treatment [22]. The volume fraction of the α_p phase decreases with the increase of solution temperature. The α_p phase with a HCP (hexagonal close packed) structure has a higher deformation resistance than β phase with BCC (body-centered cubic) structure, thus the strength of the alloy decreases with increasing temperature. In addition, the grain boundary area decreases with the growth of β phase grains, which can largely reduce the deformation resistance. The variation of the elongation with solution temperature and time is depicted in Fig. 8(b). The elongation increases with increasing solution time at the initial stage; however, it decreases with further increasing solution time. This may be caused by static coarsening. During the initial stage, the microstructure gradually becomes uniform, thus the material displays a good ductility. However, the sizes of grains begin to increase dramatically with the increase of solution time, which can reduce the ductility of material.

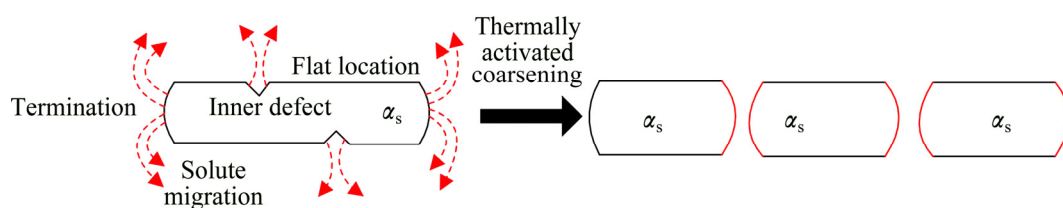


Fig. 6 Graphical illustration of termination migration mechanism [15]

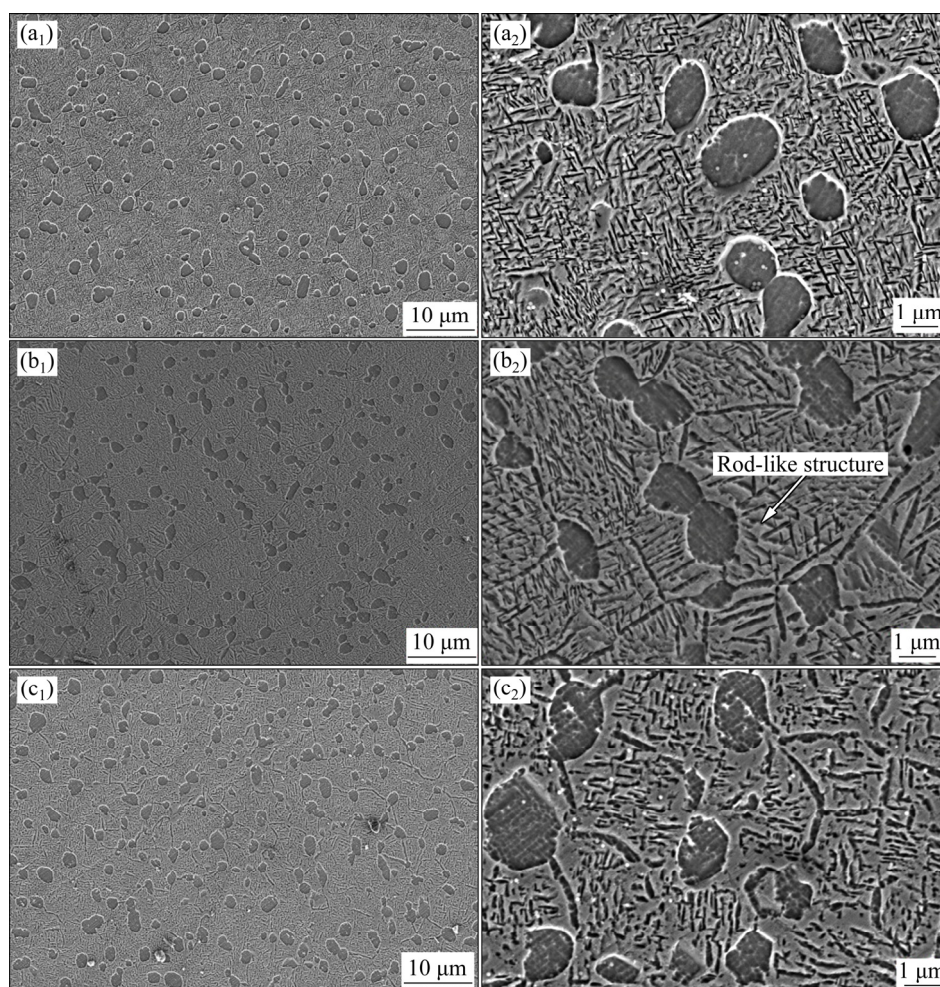


Fig. 7 Microstructural evolution of Ti-55531 alloy aging-treated at 600 °C for 6 h (a₁, a₂), 8 h (b₁, b₂) and 10 h (c₁, c₂) followed by air cooling

Table 2 Tensile properties of Ti-55531 alloy under different solution conditions

Temperature/°C	Time/h	$R_{p0.2}$ /MPa	R_m /MPa	Elongation/%
760	0.5	1031	1049	5.1
	2	1003	1018	8.7
	4	900	931	3.1
800	0.5	947	954	4.8
	2	872	949	5.1
	4	832	913	3.6

Table 3 Tensile properties of Ti-55531 alloy solution-treated at 800 °C for 2 h under different aging conditions

Temperature/°C	Time/h	$R_{p0.2}$ /MPa	R_m /MPa	Elongation/%
640	6	1704	1734	2.5
	8	1360	1435	7.7
	10	953	1206	5.8
580	6	1189	1455	1.5
	8	1217	1352	3.5
	10	1125	1259	2.4

The variation of strength and ductility of the alloy with aging conditions is shown in Fig. 9(a). The strength can be greatly improved after the aging treatment. For instance, the values of $R_{p0.2}$ and R_m reach 1704 and 1734 MPa, respectively, after being aging-treated at 640 °C for 6 h. Generally, the strength increases with increasing aging temperature and decreasing aging time. It has been reported that the α_s phase is an important factor influencing the strength and ductility of titanium alloys [23]. Although the volume fraction of the α_s phase decreases slightly with increasing aging temperature, its sizes including length and width increase largely, which can increase the interface area of α/β . The α/β interface acts as an effective dislocation barrier during the plastic deformation. Additionally, the space of the α_s phase is another factor determining the strength of the alloy. The strength of titanium alloy is inversely proportional to the space, which can be expressed as $S=a+bl^{-1}$, where S is the strength of a alloy, a and b are constants, l is the space between two neighboring lamellar α_s phases. The size of the α_s phase increases, while the space decreases

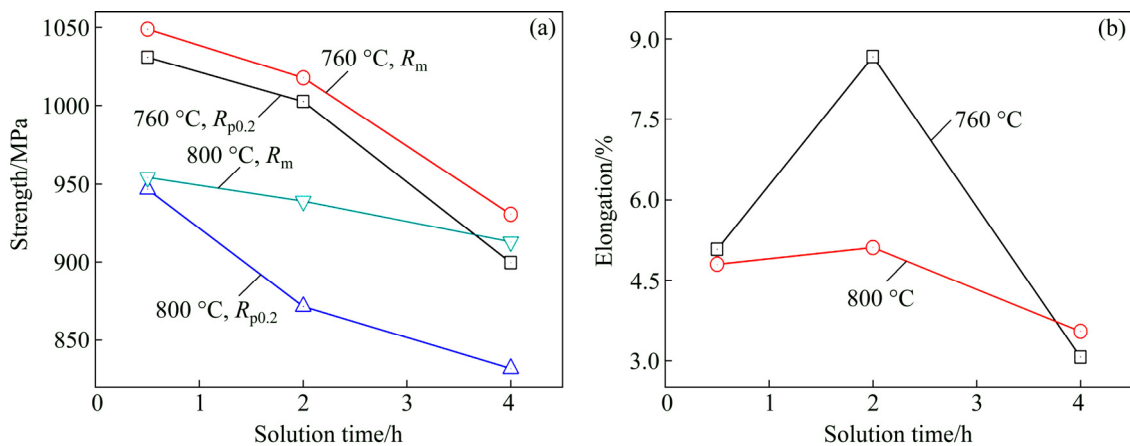


Fig. 8 Variation of strength (a) and ductility (b) of Ti-55531 alloy with solution temperature and time

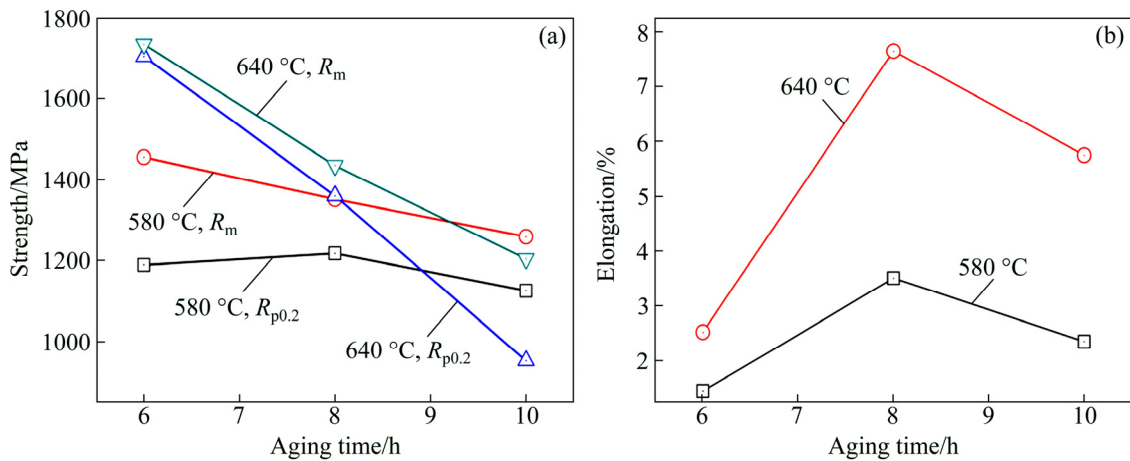


Fig. 9 Variation of strength (a) and ductility (b) of Ti-55531 with aging temperature and time

with the increase of the aging temperature, thus the strength of the alloy increases as well.

α_s phase also influences the elongation of the alloy. The elongation increases with the increase of aging temperature, as shown in Fig. 9(b). This is because the size of α_s phase increases, whereas its volume fraction decreases with the increase of the aging temperature. At the beginning of deformation, the stress and strain are distributed uniformly in the matrix. However, the strain localization takes place with further deformation. In particular, small α_s phase is harder to deform plastically, so that strain localization is extreme, which can lead to the macroscopic brittleness. Thus, the elongation of the alloy is improved by the sacrifice of its strength. The elongation increases firstly and then decreases with the increase of aging time. The variation trend is similar to that with solution treatment.

3.3 Fracture mechanism

The fractographs of the alloy solution-treated at 760 °C for different time are shown in Fig. 10(a). In Fig. 10(a), the fracture surface can be identified as a

typical dimpled region, where the dimples become elongated, but the morphologies of these dimples are more uniform and the average diameter of the dimples is about 5 μm . Some micro-voids can also be found, as remarked by red arrows. These characteristics represent a typical ductile fracture mode and indicate good ductility of the alloy.

The fractograph at solution temperature of 800 °C is shown in Fig. 10(b). The fracture surface includes many deep dimples and some micro-voids, which shows that the ductile failure mode is still dominant. The average diameter of the dimples is about 10 μm . Moreover, some facet surfaces can be observed, which demonstrates that the fracture mode of the alloy under this condition may be a combination of ductile and brittle modes. This may be caused by some brittle or second phases in the β matrix. The stress and strain localization between the brittle phases and β matrix induce some brittle fractures during tensile process.

Figure 11 shows the fractographs of the alloy aging-treated at 580 °C for 6–10 h. The fracture surfaces include many deep dimples and some micro-voids. This

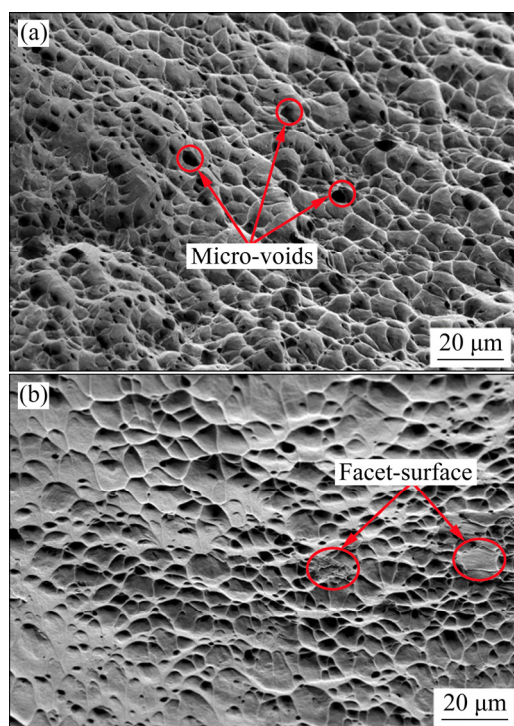


Fig. 10 Fractographs of alloy solution-treated at 760 °C (a) and 800 °C (b) for 0.5 h

indicates that the ductile fracture is still a dominant mechanism. However, there are still some differences among these fractographs. After being aging-treated at 580 °C for 6 h, the fracture surface is relatively flat, as shown in Fig. 11(a). With the increase of aging time, the fracture surface shows some obvious ups and downs, even a big fracture step (see Fig. 11(c)). Moreover, the dimples are not uniform, and their sizes around the fracture step are smaller than those in other regions.

The effect of aging temperature on the fractograph is investigated, and the results are shown in Fig. 12. The fracture surfaces are covered with many deep and dense dimples and some micro-voids. In Fig. 12(a), where the aging condition is 580 °C for 10 h, the fracture surface shows relatively flat, but there are still some smaller ups and downs. At aging temperature of 640 °C (Fig. 12(b)), the ups and downs become more obvious, even a big fracture step can be observed, and the dimples become more uniform and deep than those at 580 °C. This indicates that the crack propagation is difficult and more energy is required for cracks to bypass the α_s phase. The crack propagation is controlled by the α_s phase. The coarsening of α_s phase takes place easily at higher aging temperatures. The alloy containing a coarser α_s phase usually has a rougher crack front profile, which can lead to a higher crack propagation resistance, and finally the increase of the ductility and fracture toughness.

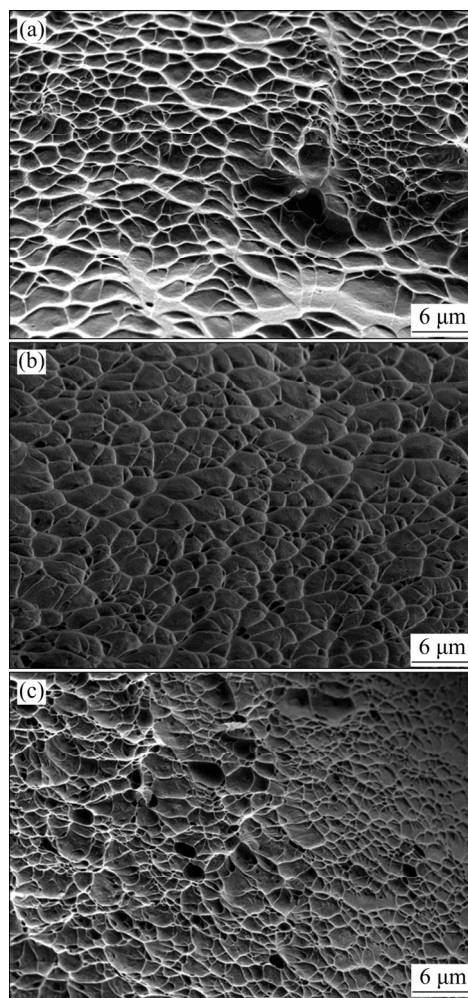


Fig. 11 Fractographs of alloy solution-treated at 800 °C for 2 h plus aging-treated at 580 °C for 6 h (a), 8 h (b) and 10 h (c)

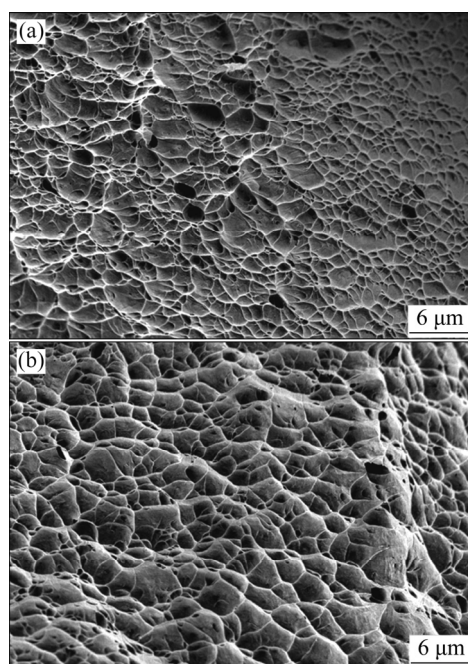


Fig. 12 Fractographs of alloy solution-treated at 800 °C for 2 h plus aging-treated at 580 °C (a) and 640 °C (b) for 10 h

4 Conclusions

(1) The volume fraction of the α_p phase decreases with the increase of solution temperature, while the size and morphology are insensitive to the solution temperature.

(2) The α_s phase nucleates at the β grain boundaries and α/β phase interface and within β grains during the aging treatment. The coarsening of lamellar α_s phase can take place during the aging treatment, the length decreases and width increases with the increase of aging temperature.

(3) The yield and tensile strengths increase with increasing aging temperature. A good balance of strength and ductility is reached after the solution treatment at 800 °C for 2 h plus aging at 640 for 8 h, in which the tensile strength is 1434 MPa and the elongation is 7.5%.

(4) The coarsening lamellar α_s phase makes the crack propagation paths more deflected and tortuous, which increases the crack propagation resistance and finally improves the ductility and fracture toughness.

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固溶–时效热处理工艺对 近 β 钛合金显微组织演化与力学性能的影响

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摘 要: 研究钛合金 Ti-5Al-5Mo-5V-3Cr-1Zr(Ti-55531)在不同固溶(760~820 °C)与时效(580~640 °C)热处理条件下的显微组织演化、力学性能及断裂机理。结果表明, 初生 α 相(α_p)的体积分数随固溶温度的升高而降低, 而次生 α 相(α_s)的长度随时效温度升高而降低, 其宽度则随时效温度升高而增加。Ti-55531 合金的屈服强度和抗拉强度随固溶温度升高而降低, 但随时效温度的升高而增大。合金在 800 °C 固溶 2 h、640 °C 时效 8 h 的条件下获得的抗拉强度(1434 MPa)与韧性(伸长率 7.7%)达到最优匹配。随时效温度 and 时间的增加, α_s 相发生粗化, 使微观裂纹扩展路径变得曲折、崎岖, 从而提高裂纹扩展阻力, 最终提高合金的韧性与断裂韧性。

关键词: Ti-5Al-5Mo-5V-3Cr-1Zr 钛合金; 热处理; 固溶; 时效; 显微组织演化; 力学性能; 断裂机理

(Edited by Wei-ping CHEN)