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Influence of duplex ageing on secondary α precipitates and mechanical properties of the near β-Ti alloy Ti-55531

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ABSTRACT

This paper presents the results of studies on the microstructure and mechanical properties of a near β-Ti alloy processed with different ageing approaches. Influences of intragranular ultrafine α precipitates and grain boundary α layer on mechanical properties in Ti-55531 alloy were analyzed. The results indicate that the precipitation microstructure of the duplex aged alloy exhibits a uniform size and acicular α precipitated orientation shows an angle of approximately 60°. While the morphology of secondary α precipitates in single-step aged samples appear the irregular distribution and heterogeneous size. With the final ageing time increasing, the morphology of intragranular α precipitates and grain boundary α phase brings out a distinct coarsening. The single-step aged alloy can yield a better combination of the ultimate tensile strength (1265 MPa) and ductility (9%). However, the ultimate tensile strength of the duplex aged alloy even reaches 1368 MPa but it breaks at the stage of elastic deformation. The fracture mode of Ti-55531 alloy changes from predominantly dimple fracture in single-step aged samples to predominantly faceted and cleavage type fracture in duplex aged samples causing by the differences of intragranular α precipitates and the interface between grain boundary α layer and intragranular microstructure.

1. Introduction

Titanium alloys are widely applied in aviation due to their superior strength to weight ratio and excellent corrosion resistance within larger service temperature range [1, 2]. During last three decades, in addition to the well-known (α + β)-Ti alloy Ti-6Al-4V, the actual use of near/metastable β-Ti alloys is gradually increasing in terms of fasteners, springs and structural parts, and this is because of their high strength, deep hardenability and good tunability of mechanical properties [3]. The rich microstructure and phase transition behavior of near/metastable β-Ti alloys lead to the controllability of mechanical properties [4]. The tailor of performance can be realized by modifying the morphology, size and volume fraction of hexagonal α phase precipitated within the cubical β matrix. The evolution of precipitated α phase is affected by many factors, among which the critical factor is heat treated parameters, such as ageing temperature and heating rate [5–7]. Therefore, it is attractive that controlling the development and refinement of precipitated α phase in near/metastable β-Ti alloys to reach an excellent combination of strength and ductility.

It has been confirmed that through ageing resulting in ultra-fine secondary α precipitates can obtain higher strength in near/metastable β-Ti alloys [8–10]. Apart from the conventional single-step ageing process, duplex ageing and slow-heating ageing have been developed to attain the specific goal. By these means, it can provide sufficient time to low temperature decomposition of supersaturated β phase resulting in metastable phases, such as ω [11–14] and β′ [15, 16], which contribute to the homogeneous nucleation of secondary α precipitates. For example, under the slow-heating rate of 1–20 °C/min, the near β-Ti alloy Ti-5Al-5Mo-5V-3Cr could yield the microstructure of super-refined intragranular α phase precipitated within the residual β matrix [13, 14]. However, the evolution of grain boundary α phase with different ageing conditions and the influence of α phase on mechanical properties of the alloy were not concerned. On the other hand, it had been studied that duplex ageing of solute-rich β-Ti alloy Ti-15V-3Cr-3Al-3Sn and Ti-8V-6Cr-4Mo-4Zr-3Al can bring out a more uniform and finer distribution of secondary α precipitates compared to the single-step ageing [17, 18]. Pre-ageing at comparatively low temperature has an accelerating influence upon the precipitation process in metastable β-Ti alloys, which can result in an improvement of hardness and higher high-cycle-fatigue life [17]. Nevertheless, investigations on duplex ageing of β-Ti alloys

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contained relatively less β-stabilizing elements have been rarely reported. On this base, the effect of duplex ageing on secondary α precipitates and mechanical properties of a near β-Ti alloy would be investigated in this paper.

Ti-5Al-5Mo-5V-3Cr-1Zr (Ti-55531) alloy is a high strength near β-Ti alloy which has been utilized on fuse pins attaching the pylon to the wing of the Airbus A-380 [19]. The alloy is developed as an improved version of the Russian alloy Ti-5.7Al-4.8Mo-5.1V-1Cr-1Fe (VT-22) and designed to achieve deeper hardenability and better damage tolerant property. The microstructure characterization and thermophysical properties of the alloy have been fully studied [20–22]. However, the relationship between microstructure and mechanical properties is rarely analyzed [23], especially α precipitates evolution and tensile properties of the alloy with duplex ageing after the β solution treatment is not involved. In this work, the influence of duplex ageing on intragranular secondary α precipitated morphology and grain boundary α layers in Ti-55531 alloy is discussed, and the relationship between microstructure evolution and tensile properties is also analyzed. Subsequently, the fracture behavior of the duplex aged alloy is analyzed by combining the fractographic feature.

2. Materials and Experimental Procedure

Flat-sheet Ti-55531 alloy with thickness of 10 mm was provided by the Baosteel Group Corporation and used for all the experiments in this study. The β-trans temperature (T_β) of the alloy is 850 ± 5 °C measured by the method of metallographic observations. By electrical discharge machining (EDM) cutting, patches being suitable for microstructure observation were cut with dimensions 10 mm × 10 mm × 10 mm and samples with a gauge length of 18 mm and a cross section of 5 mm × 2 mm were prepared for the evaluation of tensile properties.

All specimens of the as-received Ti-55531 alloy were carried out on the same solution-treated at 900 °C/30 min and subsequently air cooled. In order to compare the changes of microstructure, traditional single-step ageing and duplex ageing are adopted simultaneously. Samples with single-step ageing were directly annealed at 600 °C for 30 min and 600 min. While the duplex aged samples were firstly pre-aged at 300 °C/120 min with water quenching, and then heating to 600 °C for 30 min and 600 min. Schematic illustration of different heat treatment approaches applied in this study was presented in Fig. 1. Detailed heat treatment processes of four groups of samples were shown in Table 1. The determination of temperature parameters is based on the thermophysical properties of Ti-55531 alloy as noted in Ref. [22] and the phase transformation kinetic during continuous heating by in-situ synchrotron x-ray diffraction observations in Ref. [11]. In order to eliminate the effect of heating rate on microstructure and mechanical properties of the alloy, samples were placed into the furnace at the target temperature.

Metallographic samples were prepared by the standard mechanical polishing method. The etchant of 2 vol% HF and 3 vol% HNO_3 in distilled water was used for the samples etched. The images of microstructure were characterized by FEI Quanta 250 Environmental Scanning Electron Microscope (ESEM) and Transmission Electron Microscope (TEM). ESEM observations were operated at 20 kV and a spot size of 3.5 and TEM observations were carried out on JEOL JEM 2100 at an accelerating voltage of 200 kV. An electrolyte containing 5% perchloric acid, 35% methanol and 60% normal butanol was applied for twin jet electro-polishing in the preparation for TEM specimens. The samples for tensile experiment were conducted on 50 kN Instron 8801 testing systems at a constant strain rate of $1.0 \times 10^{-4} s^{-1}$ and at room temperature. Three samples were tested for each condition and the average value was reported. Fractographic feature of the aged alloy was observed with ESEM.

Table 1
Sample ID and heat treatment schedules.

Sample ID	Heat treatment schedule
AC96-1	900 °C/30 min AC + 600 °C/30 min AC
AC96-2	900 °C/30 min AC + 600 °C/600 min AC
AC936-1	900 °C/30 min AC + 300 °C/120 min WQ + 600 °C/30 min AC
AC936-2	900 °C/30 min AC + 300 °C/120 min WQ + 600 °C/600 min AC

(AC: Ageing cooling, WQ: Water quenching)

3. Results

Fig. 2a shows optical microstructure of the as-received Ti-55531 alloy. Great quantities of spheroidal and virgate primary α phase are uniformly distributed in the β matrix, and the size of spheroidal α phase lies in the range of 2–4 μm. After β-solution treated at 900 °C, the initial primary α phase disappears into the matrix and the recrystallization microstructure of the alloy with large equiaxed β grains appears as shown in Fig. 2b. The recrystallized β grain size is estimated to be 120–290 μm. Under the same β-solution treatment, single-step ageing and duplex ageing are separately carried out, and subsequently microstructure and mechanical properties of the different aged alloys would be systematically studied and compared.

3.1. Microstructure of Aged Alloys

To start with, it is necessary to study the microstructure of the single-step aged alloy after the β solution treatment so as to provide reference for the duplex aged alloy. Fig. 3 shows the SEM microstructure of Ti-55531 alloy with single-step ageing for 30 min and 600 min. As can be seen, the microstructure of the alloy appears plenty of long acicular secondary α precipitates inside the β grain and continuous narrow grain boundary α phase. For the single-step ageing 30 min samples as shown in Fig. 3(a, b), there are multiple precipitated orientation of intragranular secondary α phase embedded in the

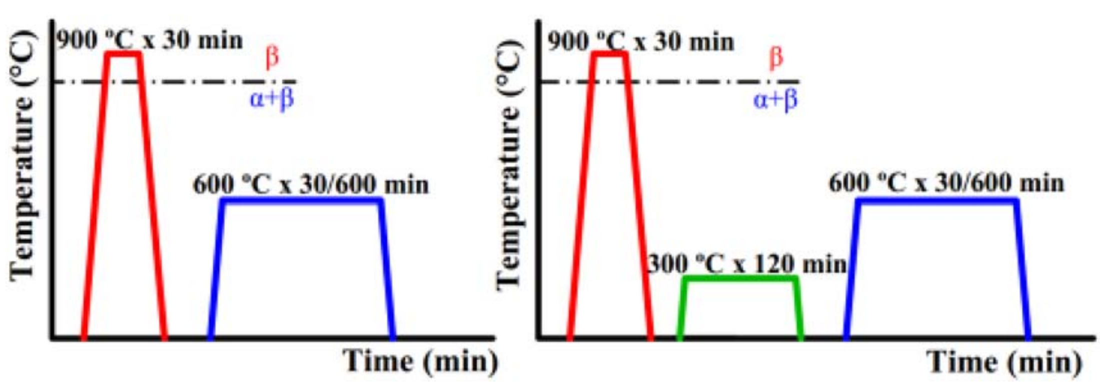

Fig. 1. Schematic illustration of different heat treatment approaches applied in this study.

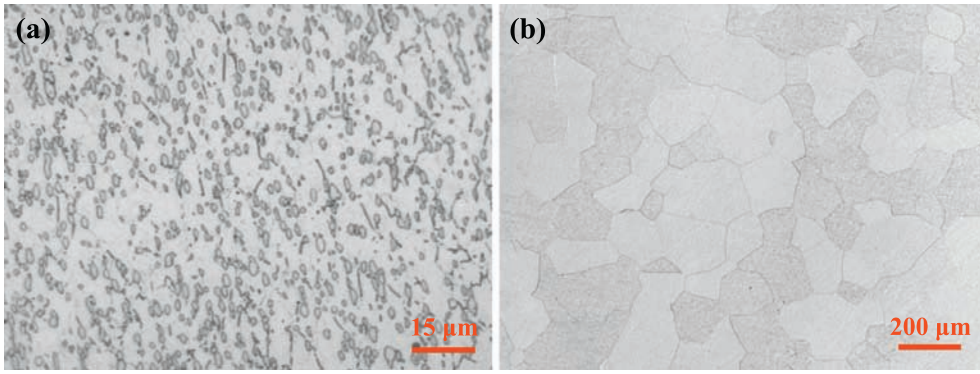

Fig. 2. Optical micrographs of Ti-55531 alloy under (a) as-received condition and (b) β solution-treated condition.

residual β matrix and the grain boundary α layer of the sample is very thin. The size and distribution of secondary α precipitates are inhomogeneous and disordered, including the size of more than 6 μm long and short only 0.2 μm. With the single-step ageing time increasing to 600 min, the morphology of acicular α precipitates have significant changes. The lamellar orientation is more uniform and the size difference is smaller. As for the grain boundary α layer, it appears clear and discontinuous along with longer ageing time. The formation of this microstructure feature in near β-Ti alloys could be caused by the rapid heating of the aged alloy and based on the pseudo-spinodal mechanism [24].

Next, the duplex aged microstructure of the alloy will be analyzed in detail. The micrographs of Ti-55531 alloy with duplex ageing for different time are shown in Fig. 4. As can be seen, the refinement of secondary α precipitates in the alloy is very noticeable after pre-aged at 300 °C/120 min. For the final ageing 30 min sample(Fig. 4(a, b)), the size and distribution of intragranular secondary α precipitates present much uniformly and orderly, and the length is estimated to less than 0.6 μm. The preferred growth orientation of acicular α precipitates in the alloy is more consistent in two directions, of which the angular separation exhibits approximately 60°. As for the grain boundary α layer, it presents a continuous narrow-band, and the precipitate free zone along the grain boundary can be insignificant. Similar ultrafine intragranular α precipitates are also obtained in the aged Ti-5553 alloy with a heating rate of 5 °C/min, but the preferred orientation of secondary α precipitates is undiscovered [13, 14]. With the final ageing increasing to 600 min, the coarsening state of intragranular and grain boundary α phase appears in the β matrix. As shown in Fig. 4c, morphology of secondary α precipitates changes from fine needle to irregular ellipse. For the triple grain boundary α phase, its morphology becomes abnormally coarse and develops into discontinuous particles. Under the pre-ageing condition, the morphology and sizes of intragranular secondary α precipitates and grain boundary α phase are largely sensitive with the final ageing time.

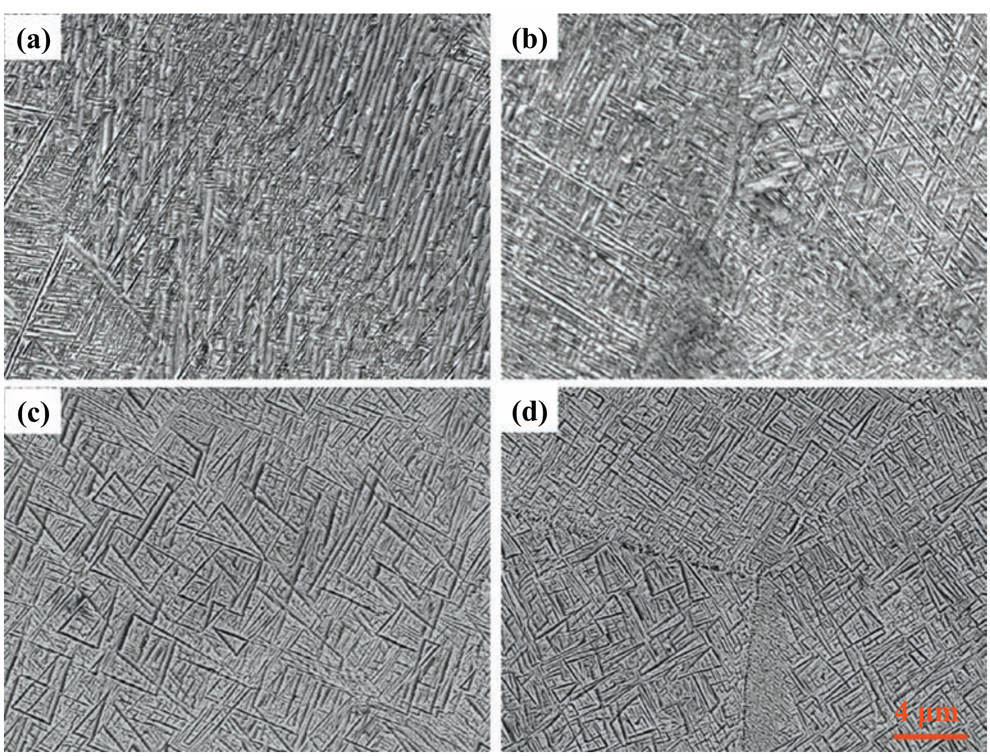

Fig. 3. SEM micrographs of the sample AC96-1: (a) intragranular α precipitates and (b) grain boundary α phase, and the AC96-2: (c) intragranular α precipitates and (d) grain boundary α phase.
agranular α precipitates and (d) grain boundary α phase.

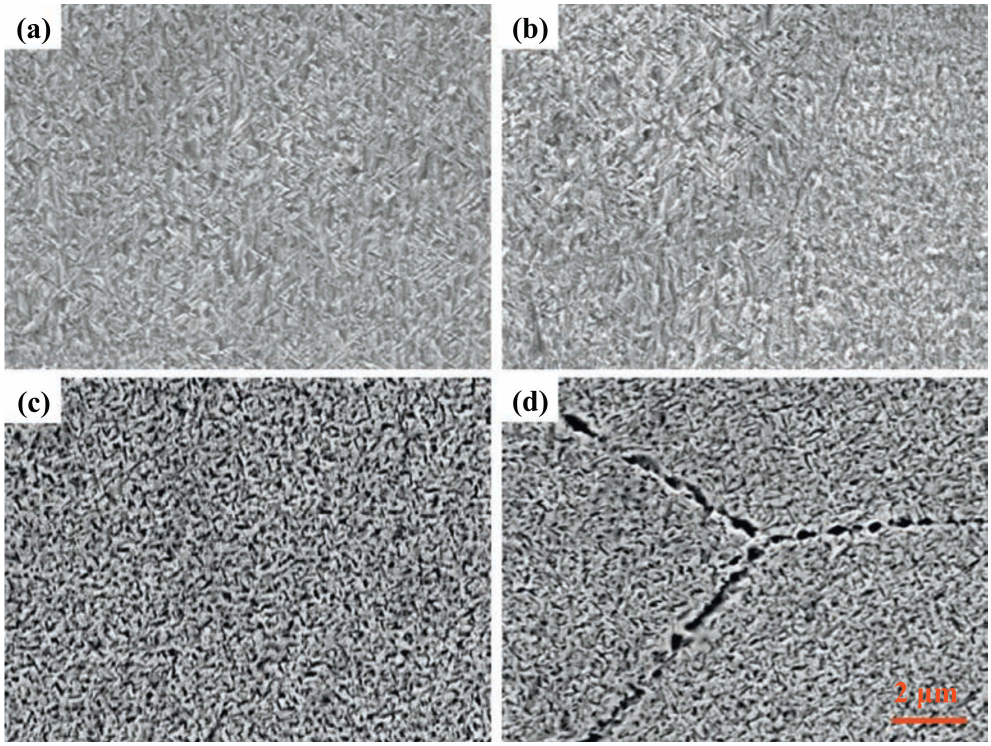

Fig. 4. SEM micrographs of the sample AC936-1: (a) intragranular α precipitates and (b) grain boundary α phase, and the AC936-2: (c) intragranular α precipitates and (d) grain boundary α phase.

3.2. Mechanical Properties of Aged Alloys

The matching of strength and ductility in near β-Ti alloys is remarkably influenced by its microstructure feature which is largely controlled by thermal treatment regimes [5]. In this paper, Ti-55531 alloy with different ageing paths obtains diversified microstructures, such as the morphology of intragranular α precipitates and grain boundary α layer, which provide the possibility for the regulation of mechanical properties.

The engineering stress-strain curves of the aged Ti-55531 alloy are shown in Fig. 5. As can be observed, the samples with single-step ageing achieve an excellent matching of strength and ductility, while the duplex aged alloys obtain higher tensile strength but terrible ductility. For the final ageing 30 min samples(Fig. 5a), the ultimate tensile strength of the AC96-1 sample is 1265 MPa with an acceptable elongation 9%, while the AC936-1 sample obtains the ultimate tensile strength of 1368 MPa but breaks at the stage of elastic deformation. In other words, the ultimate tensile strength of the duplex aged alloy increases by 103 MPa but the elongation decreases significantly. With the final ageing time increasing to 600 min, the ultimate tensile strength of the AC96-2 sample varies little but the elongation decreases to 6%, while the ultimate tensile strength of the AC936-2 sample reduces to 1212 MPa but still breaks without any plastic deformation. The tensile strength and elongation of Ti-55531 alloy change obviously with the final ageing time increasing, which can attribute to the coarsening of microstructure.

3.3. Fractography of Aged Alloys

To identify the effect of precipitation path of α phase on the fracture mechanisms, single-step and duplex aged specimens were selected for the fractography analysis. Fig. 6 shows the fracture surface of the samples aged in different paths. As can be seen, the fracture morphology of single-step and duplex aged alloys is obvious differences. According to Fig. 6(a, b), the fractographs of the single-step aged alloys shows dimple fracture accompanied with secondary cracking. Lots of fine ductile dimples in fracture surface represent the good ductility of the alloy, and crack path appears along grain boundaries as well as

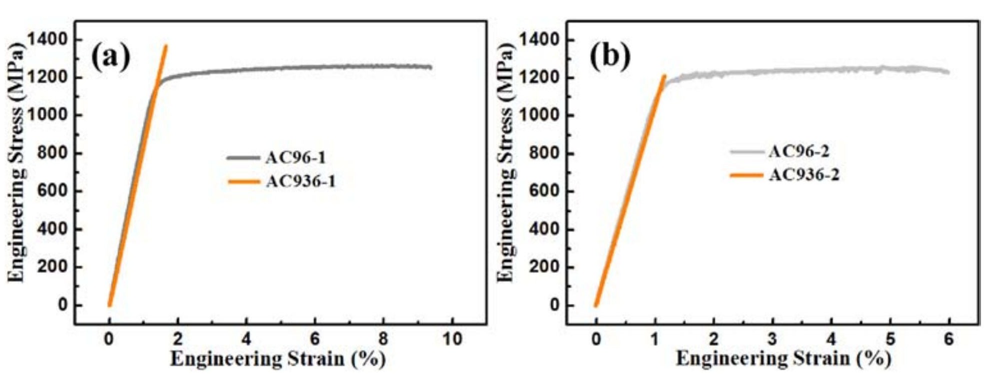

Fig. 5. Tensile engineering stress-strain curves of Ti-55531 alloy with various heat treatment conditions. The experiments were carried on at room temperature and strain rate of $1.0 \times 10^{-4}\,s^{-1}$.

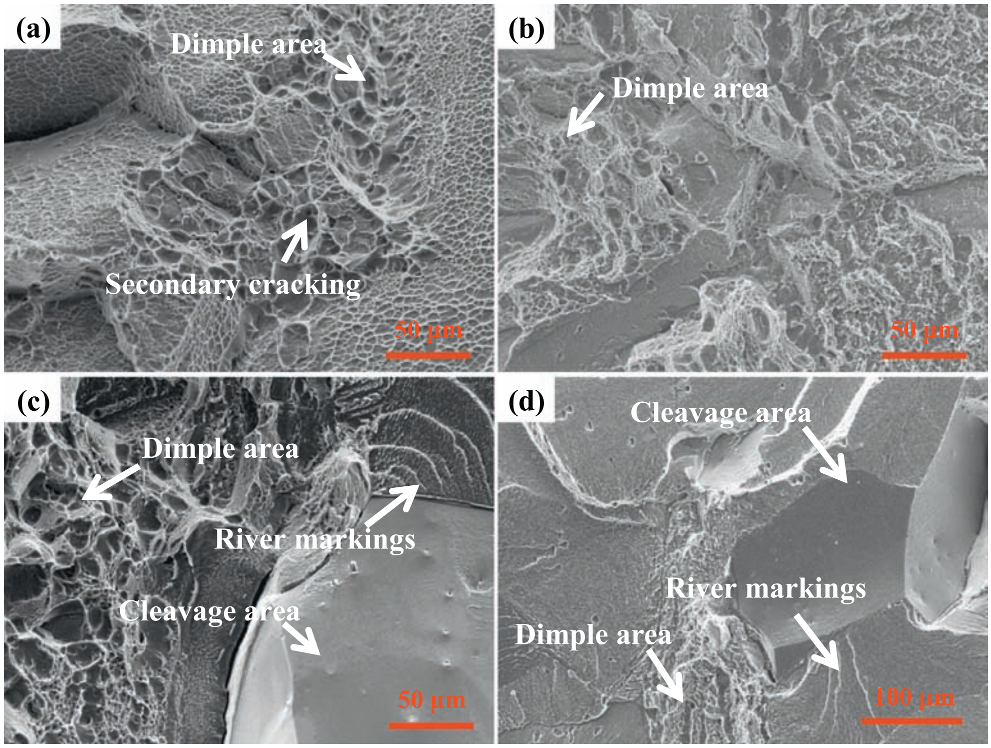

Fig. 6. SEM images of fracture surfaces of Ti-55531 alloy after β solution treatment plus various ageing ways: (a) AC96-1, (b) AC96-2, (c) AC936-1, (d) AC936-2.

through the grains. Large deflection of the crack path indicates higher fluctuation depth on the fracture surface, which requires large amount of energy for crack propagation [25]. However, as shown in Fig. 6(c, d), the fracture surface of the duplex aged samples exhibits the combination of cleavage and dimples failure, and some river markings are displayed in the cleavage facets. The morphology of fracture surface consists of flat intergranular failure and shallow dimples, which show the crack source occurs at the grain boundaries and extends mostly along the grains. The facets correspond to low plasticity intergranular fracture and dimpled zones correspond to transgranular fracture.

4. Discussion

In near β-Ti alloys after the β solution treatment plus ageing, the morphology of α precipitates controls the strength level and the β grain size determines the ductility [26]. In this paper, all samples undergo the same solution treatment under 900 °C/30 min, so the influence of the β grain size is insignificant. Based on the investigation of tensile properties, duplex ageing samples own higher strength but poor ductility, which can be attributed to the microstructural features of ultrafine secondary α precipitates and grain boundary α phase. Ultrafine α precipitates are responsible for high strength and grain boundary α phase is responsible for low ductility, which is consistent to the fractograph analysis of the samples. Then, the influence of intragranular ultrafine secondary α precipitates and grain boundary α phase on the mechanical properties of Ti-55531 alloy would be fully studied from the following aspects (Table 2).

Table 2
Tensile properties of the aged Ti-55531 alloy at room temperature.

Sample ID	σ_b(MPa)	$\sigma_{0.2}$(MPa)	δ(%)
AC96-1	1265 ± 5	1173 ± 4	9 ± 0.7
AC96-2	1261 ± 3	1186 ± 5	6 ± 0.9
AC936-1	1368 ± 11	–	2 ± 0.4
AC936-2	1212 ± 7	–	1 ± 0.6

4.1. Effect of Ultrafine Secondary α Precipitates on Mechanical Properties

To reap benefits from the uniform dispersion of ultrafine α precipitates, it is essential to have a thorough understanding of the process and mechanisms on the β-to-α phase transformation. Various microstructural features of intragranular secondary α precipitates in Ti-55531 alloy with final-ageing at 600 °C/30 min is shown in Fig. 7. As can be seen, the effect of pre-ageing treatment on the morphology, distribution and size of intragranular α phase is noticeable, which could be attributed to the difference of the composition of β phase before the final-ageing. Fig. 8 shows the bright-field TEM images of the specimens before the final-ageing, as well as selected-area electron diffraction patterns responding to the samples viewed along the $[110]_\beta$ zone axis. As can be identified, there is no α phase present in both samples, observed by the diffuse spots at the $1/2\{112\}_\beta$ positions. For the β solution sample, extremely faint diffuse streaks are observed through the primary $\{112\}_\beta$ reflections. Coupled with the pre-ageing treatment at 300 °C/120 min, diffuse streaks are strengthened and weak diffuse spots appear through the primary $\{112\}_\beta$ reflection, which could be ascribed to the increasing of the amount of embryos ω [27]. The embryos ω phase generating in nanoscale spinodal decomposition of the β matrix increases the nucleated sites of α precipitates which achieve the refinement of intragranular α phase [28].

The effect of various microstructures on the fracture of (α + β)-Ti alloys had been carried out on prior β grain size and morphology of α lath [29, 30], but the role of ultrafine α precipitates on the fracture was few investigated. For a given microstructural feature of a near β-Ti alloy, including intragranular α precipitates into the β matrix, a crack can deflect past suitably inclined α platelets or cut through the platelets depending on the minimization of energy needed to propagate through the microstructure [31]. As for the microstructure of Ti-55531 alloy in this paper, the crack deflects past suitably inclined α lamella which can be affirmed from the fractographs shown in Fig. 6. Also, the propagating of a crack is affected by the inter-lamellar spacing and aspect ratio of acicular α precipitates. As can be seen in Fig. 7, the microstructural feature of the sample AC96-1 is uneven spacing and size of

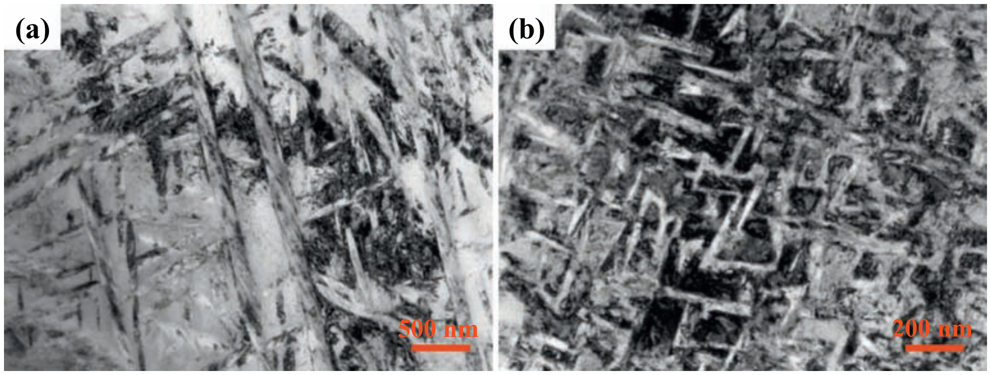

Fig. 7. High-magnification images of (a) AC96-1 and (b) AC936-1 showing intragranular acicular α precipitates.

lamellar α precipitates as well as nonuniform distribution, which results that the propagating crack has a large plastic zone size to travel. While the sample AC936-1 has homogeneous size and relatively parallel arrangement of acicular α precipitates as well as narrow and small plastic zone size, thus propagating crack encounters large deflection and spends more energy deviating past the platelets. In this case, with analogous grain boundary α phase, the intragranular strength of the alloy with pre-ageing is higher in comparison to the sample AC96-1.

With the final-ageing time increasing, the aspect ratio of intragranular α precipitates reduces significantly and the size of the plastic zone increases in the sample AC936-2 (Fig. 4c), leading that the crack spends less energy deflecting past the platelets. As a result, the strength of the pre-ageing alloy decreases. However, the distribution and inter-lamellar spacing of intragranular α precipitates in the sample AC96-2 changes little which results the tensile strength fluctuating faintly.

4.2. Effect of Grain Boundary α Phase on Mechanical Properties

For near β-Ti alloys, the grain boundary α phase generally appears continuous layer morphology due to the phase nucleating and developing preferentially at initial β grain boundaries [32–34]. The schematic illustration of the interface of grain boundary and intragranular microstructure in near β-Ti alloys under different heat treatment conditions is shown in Fig. 9. Under solution treatment above the β-trans temperature, the primary α phase of Ti-55531 alloy gradually dissolves into the β matrix and the β grain grows with the solution time increasing. During samples carried out the isothermal ageing at 600 °C, large amounts of grain boundary α nucleus nucleate and grow to connect with each other, which results in the formation of grain boundaries allotriomorphs [33]. Simultaneously, intragranular α precipitates also nucleate and develop owing to the rapid heating rate of ageing. While the interface microstructure of the single-step aged sample shows the nonuniform size and distribution of intragranular acicular α precipitates, resulting in distinct lamella spacing and undulating interface, which can assimilate more the plastic dissipation. Under the condition of homogeneous nucleation of secondary α precipitates, the duplex aged sample appears the ultrafine and uniform of acicular α precipitates, leading to a more flat interface between intragranular precipitates and grain boundary. When the tensile stress is applied, the interface of the single-step aged sample is more easily deformed and exhibits certain plasticity. However, the duplex aged interface need higher stress to generate deformation which leads to the void formation and expansion largely along the interface, and consequently fractured intergranular.

Based on the formation process of grain boundary α layer, the natural feature of grain boundary α surface shows the surface instability and the fluctuation of concentration of elements, which are beneficial to the morphological transformation with the isothermal ageing time increasing [35]. With the final ageing time increasing to 600 min in this paper, it is clearly seen that grain boundary α layer gradually develop into discontinuous and appeared α colonies, and the intragranular precipitates show different degrees of coarsening. However, there is no fundamental change in the interface between grain boundary and intragranular precipitates, and so the fracture mode of the alloy has not changed.

Generally, grain boundary α phase can also influence the tensile properties of the alloy through its width and continuity. In the fully lamellar microstructure of both (α + β) and β titanium alloys, continuous layers of α phase at prior β grain boundaries can be deleterious in mechanical properties [32]. While discontinuous grain boundary α colonies, obtained through altering the conventional β solution treatment to step-solution treatment, can effectively improve the ductility of a near β titanium alloy [33]. Based analysis results of microstructure

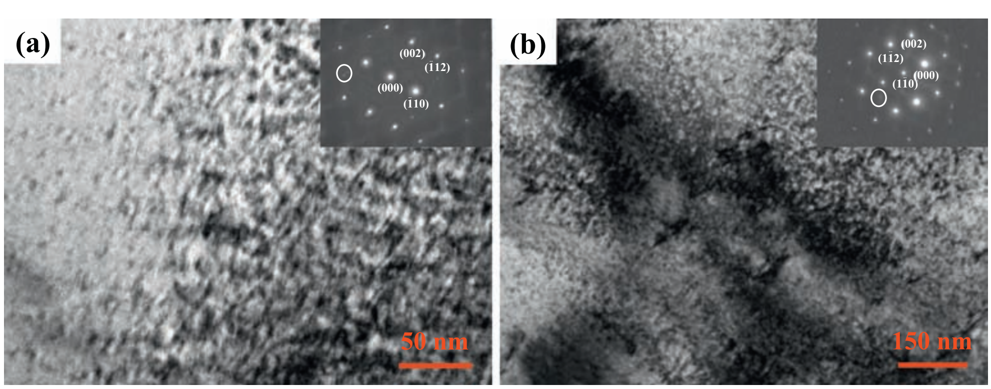

Fig. 8. Bright-field TEM images of Ti-55531 alloy after (a) the β solution treatment with air cooling and (b) the β solution treatment plus pre-ageing at 300 °C/120 min. Selected-area diffraction patterns were taken along the $[110]_\beta$ zone axis.

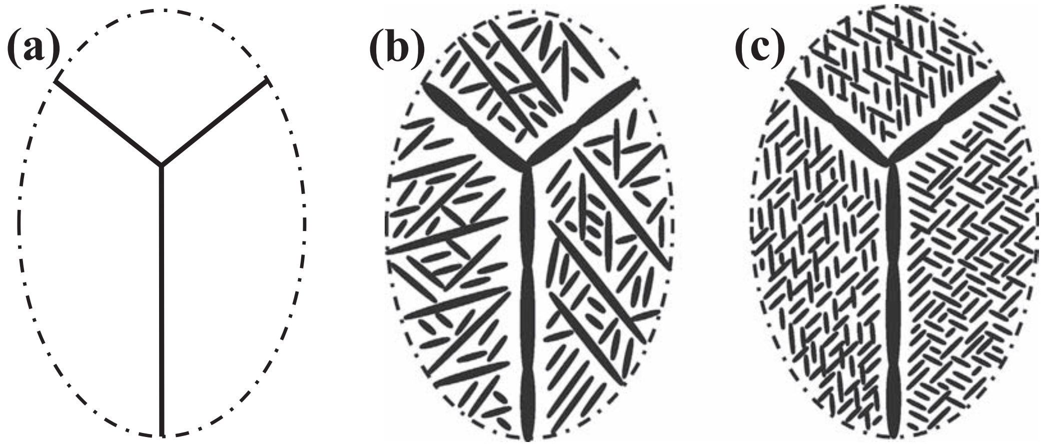

Fig. 9. Schematic illustration of the interface of grain boundary and intragranular microstructure under different heat treatment conditions: (a) Initial β grain boundary after the β solution treatment, (b) The single-step aged interface of grain boundary α phase and intragranular precipitates, (c) The duplex aged interface of grain boundary α phase and intragranular precipitates.

and mechanical properties in this paper, the Ti-55531 alloy can be benefited from the ultrafine intragranular α precipitates. If yielded the microstructure of discontinuous grain boundary α colonies before intragranular ultrafine α precipitates, the alloy may obtain an optimum combination of higher strength and ductility. This may be done through the heat treatment process of step-solution treatment plus dual-step ageing. Between discontinuous grain boundary α colonies, it appears mounts of ultrafine acicular α precipitates which may enhance the ductility of the grain boundary. In addition, the influence of size and morphology of discontinuous grain boundary α phase on the tensile properties is indistinct. As far as we know, there is still a huge room for systematic researches on the mechanism of grain boundary α phase to strengthen and toughen the mechanical properties.

5. Conclusions

In this investigation, duplex aged microstructure and mechanical properties of a near β titanium alloy Ti-55531 were studied. The influence of intragranular α precipitates and grain boundary α phase on mechanical properties was fully analyzed, respectively. The results can be summarized as:

(1) Duplex ageing has a significant effect on the precipitation behavior of secondary α phase in Ti-55531 alloy. Compared against the irregular distribution and heterogeneous size of secondary α precipitates in single-step aged samples, the precipitation microstructure of the duplex aged alloy exhibits a uniform size and acicular α precipitated orientation shows an angle of approximately 60°. With the final ageing time increasing, the morphology of intragranular α precipitates and grain boundary α phase appears a distinct coarsening.

(2) The combination of strength and ductility for Ti-55531 alloy is changed by duplex ageing. The single-step aged alloy can yield a better combination of the ultimate tensile strength (1265 MPa) and ductility (9%), while the ultimate tensile strength of the duplex aged alloy can reach 1368 MPa but it breaks at the stage of elastic deformation.

(3) Based on the differences of intragranular α precipitates and the interface between grain boundary α layer and intragranular microstructure, the fracture mode of Ti-55531 alloy changes from predominantly dimple fracture in single-step aged samples to predominantly faceted and cleavage type fracture in duplex aged samples.

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