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Precipitation Behavior of Orthorhombic Phase in Ti-22Al-25Nb Alloy during Slow Cooling Aging Treatment and Its Effect on Tensile Properties

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Abstract: The precipitation behavior of the orthorhombic (O) phase during the slow cooling aging treatment of Ti-22Al-25Nb (at.%) alloy was investigated by microstructural characterization. Then the effect of O phase precipitation on the tensile properties was studied by room-temperature tensile tests. The results showed that the precipitation of the O phase transformed from both grain boundaries and intragranular to only grain boundaries with the temperature increasing. The nucleation mechanism of the O phase from intergranular is composed of sympathetic nucleation and interface instability nucleation. In addition, the results of tensile tests indicated that the ultimate tensile strength of the alloy decreases as the precipitation of the O phase increases. Meanwhile, from the tensile results, it is concluded that the optimum heat treatment process is slow cooling after aging at 950 °C for 1 h.

Keywords: Ti-22Al-25Nb alloy; orthorhombic phase; tensile properties; heat treatment

1. Introduction

As an important high temperature structural metal, Ti₂AlNb-based alloys have low density, high specific strength, high temperature strength, and excellent creep resistance [1–3]. In comparison with the conventional γ -TiAl-based alloys which suffer from brittleness and poor fracture toughness [4], Ti₂AlNb-based alloys have better plasticity at room temperature, giving rise to outstanding application potential in aero engines and aerospace [4,5].

Generally, Ti₂AlNb-based alloys are usually composed of 12–25 at.% Al and 12–38 at.% Nb. This leads to the fairly complicated phase constituents. Except the hcp (hexagonal close-packed) Ti₃Al phase and bcc (body-centered cubic) B2 phase [6–8], the orthorhombic Ti₂AlNb phase (O phase) was observed in the Ti-25Al-12.5Nb (at.%) alloy in 1988 for the first time [3]. Subsequent studies revealed that the O phase has important effects on room temperature plasticity, creep resistance, and high temperature strength of Ti₃Al-based alloys [9]. The plastic deformation of O phase is mainly carried by (001) $\langle 110 \rangle$ slip systems, as well as twinning on (101) planes [1]. Although the alloys with a single O phase were found to have poor room-temperature ductility and toughness, the O phase as a second phase has a positive effect on the mechanical properties. Therefore, the dual-phase (O + B2) Ti₂AlNb alloys have attracted intensive attention in the past two decades due to their superior mechanical properties [10–12]. Wang et al. reported that after thermomechanical processing followed by solid solution and aging at the B2 + O region, a lamellar microstructure was obtained which

exhibited excellent tensile strength at room and elevated temperatures [13–15]. However, due to the complicated phase transition pathways, the microstructure of Ti₂AlNb-based alloys is quite sensitive to the thermomechanical parameters. As a consequence, the level of thermomechanical processing maturity of Ti₂AlNb-based alloys only reaches 3–5 to date [16]. Former researches have shown that the behaviors and the mechanism of phase transitions are related to the temperatures of processing, solution, and age-treatments [17]. Thus, it is of significance to reveal the relationship among the heat-treatment temperatures, microstructure, and mechanical properties, especially the effect of precipitation behavior of the O phase on mechanical properties.

In this paper, the effect of heat treatment parameters on the precipitation behavior of the O phase during the slow cooling aging treatments of a Ti-22Al-25Nb alloy was studied by the quantitative metallographic statistics based on the microstructure images, and the nucleation mechanism of the O phase was clarified. In addition, the relationship between the content of the O phase and its tensile properties was also established.

2. Experimental

Table 1 shows the actual composition of Ti-22Al-25Nb (at.%) alloy. The alloy was prepared by hot rolling and then cut into samples with a dimension of 9 mm × 7 mm × 6 mm by wire electrical discharge machining. A tubular furnace was used for heat treatment experiments. The involved parameters are shown in Figure 1. The samples were heated to 1080 °C with a rate of 8 °C/min, held for 15 min, and then cooled slowly to different temperatures (900 °C, 930 °C, 950 °C) with a rate of 5 °C/min. After that, the samples were held for 0.5 h, 1 h, 1.5 h, and 2 h, respectively, and were then water-quenched to room temperature.

Table 1. The actual composition of Ti-22Al-25Nb alloy used in the present work (wt.%).

Rolled Plate	Ti	Al	Nb	C	N	H	O
wt. %	Bal.	10.11	41.25	0.0051	0.0031	0.002	0.0471

Figure 1. The schematic illustration of the heat treatment process.

The samples were sectioned along the longitudinal direction for microstructure characterization by X-ray diffraction (XRD, PANalytical X'Pert PRO, CuK α , (PANalytical B. V, Almelo, Netherlands),

optical microscopy (OM, OLYMPUS/PMG3, Olympus Corporation, Tokyo, Japan), and scanning electron microscope (SEM, ZEISS SUPRA55, Carl Zeiss AG, Jena, Germany) equipped with electron backscatter diffraction (EBSD) system. After mechanically polished, the samples for OM were etched in the etching solutions of $\text{HF:HNO}_3:\text{H}_2\text{O} = 1:3:8$. The SEM and EBSD samples were electrochemically polished for 18–30 s at 20–25 V and about 18 °C in the solution of 6 vol% perchloric acid + 34 vol% butanol + 60 vol% methanol. The quantitative metallographic analysis was performed using the Image-Pro Plus 5.0 (IPP) software (Media Cybernetics, MD, USA). The grain size was evaluated by using the line-intercept method according to GB/T 6394-2017. The EBSD analysis was conducted with a step of 0.2 μm and analyzed by the HKL-Channel 5 software (HKL, Oxford, UK).

For tensile tests, the samples with a dimension of 65 mm $\times$ 10 mm $\times$ 8 mm were machined from the as-rolled sheet and then machined into a gauge dimension of $\Phi 5$ mm $\times$ 30 mm after various heat treatments, as shown in Figure 2. The tensile tests were carried out on an Instron-3362 machine (Instron, Boston, MA, USA) with a constant speed of rate of 0.001 mm/s. At least three sets of tensile tests were conducted at each condition.

Figure 2. Machining drawing of the tensile specimens.

3. Results and Discussion

3.1. Initial Microstructure

Figure 3a shows the XRD patterns of the as-rolled sheet, it can be concluded that the initial microstructure mainly consists of the O phase and B2 phase. In Figure 3b, it can be seen that the initial grain size of the B2 phase is relatively large with an average size of about 160 μm . According to the BSE (back-scattered electron) image in Figure 3c, numerous O phases with acicular shape are precipitated in the matrix of the B2 phase, while the sizes are far from identical.

From Figure 3d–f, it can be seen that the microstructure after solution treatment sample consists of a single B2 phase with grain sizes ranging from 60 to 350 μm . Obviously, the mean grain size is larger than that in the initial state, indicating inevitable grain growth when the solution is treated in the single-phase region.

Figure 3. Initial microstructure (a–c) and the one after solution treatment at 1080 °C for 15 min (d–f) for the rolled Ti-22Al-25Nb alloy. (a,d) XRD patterns, (b,e) optical microscopy (OM) images, (c,f) back-scattered electron (BSE) images.

3.2. Precipitation Behavior of the O Phase

To understand the precipitation behavior of the O phase, the heat treatment of Ti-22Al-25Nb alloy in Figure 1, named as the slow cooling aging, was carried out. Obviously, the heat-treated microstructure at 900–950 °C/0.5 h is mainly composed of the B2 phase and O phase as shown in Figure 4a–c. The precipitation of the O phase transformed from both grain boundaries and intragranular to only grain boundaries with the temperature increase. When the aging temperature is below 950 °C, the acicular O phase is distributed in the intragranular and grain boundaries. According to the diagram of Ti-22Al-xNb phase [18], the approximate phase regions of the alloy were $B2 + O < 955\text{ °C} < B2 + O + \alpha_2 < 1010\text{ °C} < B2 + \alpha_2 < 1065\text{ °C} < B2$, which indicates that 900 °C and 930 °C is far from the transition point of the B2 + O two-phase region and the B2 + O + α_2 three-phase region (950–960 °C). The higher degree of subcooling can achieve the higher driving force. Accordingly, the O phase tends to nucleate simultaneously in the grain boundary and intragranular. The average size of the O phase at 930 °C (75 μm) is larger than that at 900 °C (45 μm). With the increase of temperature, the O phase nucleates preferentially at grain boundary. The driving force for coarsening derived from the interfacial energy [19,20]. However, the globular O phase only exists at grain boundaries when the aging temperature reaches 950 °C. The driving force of spheroidization is provided by the reduction in interface energy [21]. The volume fraction of precipitated O phase is counted by IPP. The statistical results are shown in Figure 4d. It can be seen that the volume fraction of the O phase reached more than 50% at 900 °C, while it is less than 5% at 950 °C, indicating that the precipitation of the O phase decreased significantly as the temperature increased.

Figure 5 shows the heat-treated microstructure under various conditions. Distinctly, as the aging time increases, the precipitation of the O phase is promoted. The nucleation and growth of the O phase can occur both in the intragranular and grain boundaries. The quantitation metallographic statistic was performed based on the images. The results in Figure 6 demonstrated that the volume fraction of the acicular O phase increases with the decrease of aging temperature and the increase of time. In addition, the volume fraction of the O phase increases obviously at 900 °C during the initial 0.5 h. Then the growth rate decreases quickly during the subsequent aging process. Similarly, the volume fraction of the O phase increases steadily at 930 °C with the increasing time. However, the growth rate is constant and is lower than that at 900 °C/0.5 h. When the aging temperature increased to close to the transition point of the B2 + O two-phase region and the B2 + O + α_2 three-phase region, defined as $T_{2 \rightarrow 3}$, such as

950 °C, there is nearly no nucleation of O phase occurring at the initial aging stage until the aging time extended to 1.5 h. The volume fraction of the O phase increases heavily during the subsequent process. Furthermore, the precipitation rate of the O phase reached the highest point at 900 °C/0.5 h.

Figure 4. Microstructure of Ti-22Al-25Nb alloy held for 30 min at different temperatures. (a) 900 °C, (b) 930 °C, (c) 950 °C, (d) statistical results for the volume fraction of the orthorhombic (O) phase.

Figure 5. The OM images of the heat-treated Ti-22Al-25Nb alloy at (a) 900 °C/1 h, (b) 900 °C/1.5 h, (c) 900 °C/2 h, (d) 930 °C/1 h, (e) 930 °C/1.5 h, (f) 930 °C/2 h, (g) 950 °C/1 h, (h) 950 °C/1.5 h, and (i) 950 °C/2 h.

Figure 6. The volume fraction of the acicular O phase varies with temperature and time.

Due to the limitation of OM magnification, the heat-treated samples at 900–950 °C/1–2 h were selected for SEM observations. The corresponding microstructure is shown in Figure 7. It is found that the acicular O phase which precipitates at 900 °C/1 h can grow up into the lath cluster with thicknesses of 10–50 μm when holding at 900 °C for 2 h. At 930 °C/1 h, the O phase precipitates along the grain boundaries at a certain angle and then continues to nucleate and grow up nearly perpendicular to the O phase which has already precipitated. When the aging time increases to 2 h, it can be seen that the O phase precipitates discontinuously. The O phase is broken and refined due to the collision of O phase laths. Moreover, almost all O phases precipitate along the grain boundaries at 950 °C.

Figure 7. The BSE images of the heat-treated Ti-22Al-25Nb alloy at (a) 900 °C/1 h, (b) 930 °C/1 h, (c) 950 °C/1 h, (d) 900 °C/2 h, (e) 930 °C/2 h, (f) 950 °C/2 h.

3.3. Nucleation Mechanism of the O Phase

In this work, two kinds of nucleation mechanisms of the O phase from grain boundaries are observed—sympathetic nucleation and interface instability nucleation [22]. The continuous α layers along the β/β grain boundary denoted α_{GB} , and Widmanstätten plates colonies denoted α_{WGB} [23,24]. Sympathetic nucleation will lead to different orientations between α_{WGB} and α_{GB} , while α_{WGB} grows up from α_{GB} grain with the same orientation due to interface instability. The orientation relationship (OR) between the O phase and the B2 phase satisfies the well-known Burgers OR, $\{001\}_O // \{110\}_{B2}$, $\langle 110 \rangle_O // \langle 111 \rangle_{B2}$ [8,25]. Under ideal conditions, there are 12 variants of the O phase with different orientations that can precipitate in the B2 matrix [26]. However, these variants will not precipitate with the same probability due to various reasons, and the variants selection of the O phase occurs during the process of nucleation and growth [27,28]. The EBSD measurements of the orientation parameters were performed on a triangle grain boundary of B2 phase obtained at 900 °C/0.5 h. Figure 8a,b shows the inverse pole figures (IPF) of the O phase and B2 matrix respectively, and Figure 8c shows the pole figures (PF) of the sites marked in Figure 8a,b. The O phase precipitated from the grain boundaries is named O_{GB} . O_{G1} , O_{G2} , and O_{G3} refer to the O_{GB} . Moreover, it was found that some O phase variants nucleate in the vicinity of O_{GB} and then grow into the β grains. O1 is a variant nucleated at O_{G1} , and O2, O3 are the variants nucleated at O_{G2} . Obviously, the orientation of O_{G1} is consistent with that of O1 as shown in Figure 8c. This is a typical interface instability nucleation behavior. Furthermore, the ORs of O1, O_{G1} , and b1 satisfy the Burgers ORs. However, the O phase variants precipitated in b2, such as O2 and O3, have different orientations with O_{G2} . It is also strong evidence that there are two kinds of nucleation mechanisms of the O phase from grain boundaries—sympathetic nucleation and interface instability nucleation. Through the analysis, six kinds of variants precipitated in b2 have a certain orientation relationship with b2 grains, $(001)O // (110)B2, [111]B2 // [110]O$. The O_{G3} and b3 grains also satisfy the above orientation relationship.

Figure 8. The inverse pole figures (IPF) maps (a) of O phase and (b) of B2 phase. (c) The $\{110\}$, $\langle 111 \rangle$ pole figures of B2 phase and the $\{001\}$, $\langle 110 \rangle$ pole figures of O variants.

3.4. Tensile Properties

Based on the above analysis, the volume fraction of the O phase increases with the decrease of aging temperature and the increase of time, as shown in Figure 6. To figure out the effect of the O phase on mechanical properties of Ti-22Al-25Nb alloy, room-temperature tensile tests of the heat-treated samples were carried out. Figure 9a–c shows the tensile curve of the samples at different conditions. It can be seen that the elongation of the alloy at 900 °C is lower than that of at 930 °C and 950 °C after aging for 0.5 h. As it was known, the O phase suffers from a lower number of available slip systems compared with the B2 structure [29]. The high-volume fraction of the O phase was considered to be the probable reason for this exhibition at 900 °C [30]. It can be seen that the elongation tends to increase with time at 900 °C. It can be interpreted that most of the O phase preferentially precipitates at the grain boundaries and then grows into the B2 matrix. During the subsequent aging process, the preferentially precipitated O phase grows up and gathers into a lath cluster. This O phase cluster is larger in size than the individual O phase, and the plasticity of the large-size O phase is superior to that of the small-size O phase [13,31]. The elongation of the alloy decreases with the prolongation of aging time due to the gradual precipitation of O phase at 930 °C and 950 °C. Compared with this, the initial precipitation amount of the O phase at 900 °C has reached 50%, and then the growth of the O phase is the main process. At 930 °C and 950 °C, the precipitation process of the O phase is the dominant, the size of the precipitated O phase is relatively large, but its volume fraction is lower, which cannot grow into clusters. Thus, the elongation of the alloy decreases with the increase of O phase precipitation. Figure 9d shows the relationship between the ultimate strength (UTS) and the temperature of the alloy. It can be seen that the UTS of the samples heat-treated at 900 °C is the lowest. On the contrary, the volume fraction of the O phase at 900 °C is the highest in the same aging time. With the increase in the aging temperature, the volume fraction of the O phase decreased. At the early stage of aging, the UTS of the alloy at different temperatures varies slightly, such as 0.5 h. With the extension of aging time, the precipitation of the O phase increased. In the subsequent aging process, the decrease rate of UTS at 900 °C is greater than that of at 930 °C. Boehlert [30] found that when the volume fraction of the O phase reaches 28%, the UTS of the alloy with the B2+O microstructure is higher than that of the full B2 microstructure for the Ti-12Al-38Nb alloy. The precipitation of the O phase can strengthen the matrix, but the corresponding plasticity will lose a little. When the volume fraction of the O phase reaches 79%, both strength and plasticity will reduce significantly. In addition, the thickness of lamellar O affects the tensile properties, as has been known that the coarse lamellar O exhibits poor strengthening effect compared with the fine lamellar O [32]. It can be seen from Figure 7a, the thickness of lamellar O gradually increased with the increase of time at 900 °C. Therefore, although the volume fraction of the O phase increased with the increase of time, the UTS of the alloy decreased. When aging at 950 °C, the content of the O phase is very small at the initial aging stage. As mentioned previously, a small amount of the O phase can strengthen the matrix, but when the volume fraction of the O phase increases to a certain extent, the strength of the alloy will be sacrificed. This is the reason why the strength experiences the first increase then decrease process at 950 °C. The strength of the alloy reaches the highest at 950 °C/1 h.

Figure 9. The effect of B2 slow cooling to aging treatment to ambient tensile curve for Ti-22Al-25Nb alloy: (a) 900 °C/0–2 h, (b) 930 °C/0–2 h, (c) 950 °C/0–2 h, (d) the ultimate strength (UTS) of the alloy at different temperatures.

4. Conclusions

- (1) The acicular O phase precipitated from both the grain boundaries and the intragranular when aging at 900 °C. Increasing the temperature to 930 °C, the acicular O phase tends to precipitate from the grain boundaries. In addition, the average size of the O phase at 930 °C is larger than that at 900 °C. When aging at 950 °C, only a few globular O phases could be found at grain boundaries.
- (2) Under the condition of B2 slow cooling aging, there are two modes of nucleation mechanism of O phase from grain boundaries—sympathetic nucleation and interface instability nucleation.
- (3) With the increase of aging temperature, the volume fraction of the O phase decreased and the strength of the alloy increased. The strength of the alloy decreases with the increase of aging time when aging at 900 °C and 930 °C. When aging at 950 °C, strength experiences a first increase then decrease process. The reasonable match between strength could be obtained at 950 °C/1 h.

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