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Low-cycle fatigue behavior of Ti-6Mo-5V-3Al-2Fe alloy with various types of secondary α phase

Hao-Yu Zhang, Zhi-Peng Zhang, Zheng-Yuan Li, Jie Sun, Xin Che, Si-Qian Zhang, Yu Liang and Li-Jia Chen
1 School of Materials Science and Engineering, Shenyang University of Technology, Shenyang 110870, People’s Republic of China
2 State Key Laboratory of Rolling and Automation, Northeastern University, Shenyang 110819, People’s Republic of China
E-mail: zhanghaoyu@sut.edu.cn

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Abstract

Low-cycle fatigue (LCF) behavior of a novel near β titanium alloy Ti-6Mo-5V-3Al-2Fe (wt%) with various types of secondary α phase was investigated. Results indicate that No.1 microstructure containing crossed and fine intragranular α, as well as discontinuous GB α, is obtained through low temperature (480 °C) aging treatment. No.2 microstructure containing parallel and coarse intragranular α, as well as parallel WGB α, forms after high temperature (600 °C) aging treatment. Under all strain amplitudes, stress amplitudes and LCF life of the samples with No.1 microstructure are higher than those of the samples with No.2 microstructure. For the samples with No.1 microstructure, more and deeper secondary cracks and dimples form in fatigue crack propagation and fast fracture regions, respectively. Such fatigue fracture behavior reflects a longer LCF life.

1. Introduction

Near β titanium alloys are frequently used in aviation and aerospace applications due to their high specific strength, lightweight, excellent corrosion resistance, and fatigue crack growth resistance [1–3]. For example, near β titanium alloy Ti-10V-2Fe-3Al (Ti-1023) and Ti-5Al-5Mo-5V-3Cr (Ti-5553) are being used for the manufacture of landing gear for Boeing 777 and Airbus-350, respectively [4, 5]. As critical load structures, the near β titanium alloy components usually serve in the severe environments, such as cyclic loading, which can lead to fatigue crack and fracture within a short time. Particularly in aviation and aerospace industries, the fatigue failure of components is unpredictable, and can result in an overwhelming disaster [6]. Therefore, the low-cycle fatigue (LCF) property of near β titanium alloy has received increasing attention over the years [7–9].

Generally, for titanium alloys, microstructure regulation can be adopted to satisfy the requirement of mechanical property [10, 11]. Previous studies have attempted to reveal the underlying mechanism for the effect of microstructure features on LCF property and behavior. Xu et al [12] studied the effect of primary α phase and secondary α phase on LCF behavior of Ti-6Al-4V alloy. The cyclic softening/hardening behavior has been revealed to be determined by the dislocation behavior affected by α/β interfaces. Joseph et al [13] studied the dislocation interactions at the crack nucleation site of Ti-624Si alloy during LCF deformation. It is found that crack nucleated at the boundaries between primary α and two-phase regions, due to these two-phase regions providing a barrier to slip transfer. Zhou et al [14] obtained a novel kind of microstructure for Ti-6.5Al-3.5Mo-1.5Zr-0.3Si alloy by a new near β forging process, which contains equiaxed and lamellar α as well as transformed β matrix. Moreover, the LCF test shows that fatigue life of the alloy with such microstructure was higher than that of the alloy with conventional bimodal microstructure. However, the relationship between secondary α phase and LCF fracture mechanism is still unknown. Gaddam et al [15] investigated the LCF properties of Ti-6Al-2Sn-4Zr-2Mo alloy with different thicknesses of α-case layers. The experimental results indicate that the existence of α-case led to multiple crack initiation and significantly reduced LCF life. It can be seen from the works mentioned above that the unique microstructure features strongly affect the LCF behaviors in different titanium alloys.

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Our previous work has designed a novel near β titanium alloy Ti-6Mo-5V-3Al-2Fe (wt%), whose ultimate tensile strength is higher than 1500 MPa after heat treatment [16]. Previous work has confirmed that three types of secondary α phase, in terms of precipitation sites, can be formed in Ti-6Mo-5V-3Al-2Fe alloy, i.e., grain boundary α that precipitates on β grain boundaries (GB α), Widmanstätten α that precipitates at vicinities of β grain boundaries (WGB α) and intragranular α that precipitates within β grains. As a near β titanium alloy expected to show potential for high-strength structural applications in aviation and aerospace industries, its LCF properties have not been investigated by far. Moreover, the effect of its microstructural features on LCF behavior is still unknown. In this present article, the LCF behavior and property of Ti-6Mo-5V-3Al-2Fe alloy under the influence of various types of secondary α phase were investigated. The results will provide an essential reference for the control of microstructure to improve the LCF performance of near β titanium alloy components.

2. Materials and methods

Sponge Ti, Mo-Al master alloy, pure Fe, and V-Al master alloy were used to prepare the Ti-6Mo-5V-3Al-2Fe alloy ingot. Raw materials were melted twice by vacuum arc remelting to obtain alloy ingot. The ingot was forged to plate with 100 mm in width and 30 mm in thickness. The plate went through multi-pass hot rolling to get a sheet with 6 mm in thickness. Samples used in this study were cut from the sheet.

Before aging treatment, samples were super-transus solution-treated for 0.5 h followed by water cooling. Various types of secondary α phase were obtained under different aging temperatures according to the works of literature [16], i.e., the super-transus solution-treated samples were aged at 480 °C and 600 °C for 4 h followed by air cooling, respectively. After aging treatment, two kinds of microstructure with different types of secondary α phase were obtained. Modified Kroll’s reagent (10 mL HF, 30 mL HNO3, and 80 mL H2O) was adopted to reveal microstructures. Then, microstructures were characterized by scanning electron microscopy (SEM, Hitachi Su8010, Hitachi, Japan). Further observation of the secondary α phase was carried out on transmission electron microscopy (TEM, JEOL Jem-2100, JEOL, Japan). TEM samples were cut in the centre of aged samples by electric sparking. Then, TEM samples were manually ground to 50 μm and twin-jet electropolished in a solution (6 vol% perchloric acid, 35 vol% n-butyl alcohol, and 59 vol% methanol) at −20 °C.

LCF tests of the aged samples were performed on the MTS landmark 370.10 servohydraulic test system. The samples for the LCF test are plate-like. The size of the experimental area is 5 mm in thickness, 10 mm in length, and 5 mm in width. The symmetrically axial LCF tests with a strain ratio \( R(\varepsilon_{\text{min}}/\varepsilon_{\text{max}}) \) of −1 were carried out under the control of strain amplitude at room temperature. A sine waveform with a frequency of 1.0 Hz was used during the LCF test. The total axial strains were measured by an extensometer with an axial gage length of 10 mm. Different strain amplitudes (0.2%, 0.4%, 0.6%, 0.8% and 1.0%) were selected to carry out the LCF tests. After LCF tests, the original data was transformed to the hysteresis loop and cyclic stress response curves, in order to analyze the LCF behavior of the alloy. The fractured fatigue samples were examined for fracture surface morphology on SEM. Besides, observation of dislocation was carried out on TEM.

3. Results

3.1. Microstructures containing various types of secondary α phase

After super-transus solution treatment, the microstructure of the alloy is almost composed of β phase, and the β grain size is about 100–150 μm. Figures 1(a) and (b) show the microstructures of the alloy aged at 480 °C and 600 °C for 4 h, respectively. After aged at 480 °C for 4 h, the microstructure of alloy contains two types of α precipitates. One is the fine intragranular α uniformly distributed within β grains, and the other is GB α distributed on β grain boundaries. When the aging temperature is 600 °C, intragranular α obviously coarsened so that the needle-like morphology can be clearly observed. In addition, mutually-parallel WGB α forms along the β grain boundaries. The various types of secondary α phase are shown in table 1. In the following part, the low temperature aged samples are referred as No.1 samples, and the corresponding microstructure containing fine intragranular α and GB α is designated as No.1 microstructure. The high temperature aged samples are referred to as No.2 samples, and the corresponding microstructure containing coarse intragranular α and GB α as well as WGB α is designated as No.2 microstructure.

3.2. Observations of secondary α phase

As shown in figure 2, the secondary α phase of No.1 and No.2 microstructure could be clearly observed by TEM. The intragranular α with high aspect ratios could be found within β grains in both No.1 and No.2 microstructures. According to figure 2(a), in No.1 microstructure, the intragranular α exhibits small size (~80 nm in width) and narrow inter-particle spacing (~100 nm). Moreover, the precipitated orientations of intragranular α crossed with each other. According to figure 2(b), in No.2 microstructure, the size (~360 nm in...
width) and inter-particle spacing (∼180 nm) of intragranular α are obviously bigger than those in the No.1 microstructure. In addition, precipitated intragranular α has a specific orientation and keep parallel distribution. From figure 2(c), it can be seen that GB α with an average width of 150 nm located along prior β grain boundaries in No.1 microstructure. However, it is different from the conventional continuous GB α formed in other near β titanium alloys [17, 18]. The GB α is discontinuous in No.1 microstructure. From figure 2(d), besides GB α, mutually parallel WGB α formed at β grain boundaries and grew into the internal areas of prior β grains in No.2 microstructure.

### 3.3. Cyclic stress response behavior

The cyclic stress response curves of No.1 and No.2 samples are shown in figure 3. At the same strain amplitude, stress amplitudes of the No.1 samples are overall higher than that of the No.2 samples. To quantitatively characterize the softening or hardening behavior during LCF tests, the softening/hardening degree η is introduced in this work. The η is associated with the variation of stress amplitude for each strain amplitude and is calculated as equation (1) [19].

$$\eta = \frac{\sigma_{i+1} - \sigma_i}{\sigma_{i+1}} (i = 2, 3, 4,...)$$  

where $\sigma_{ini}$ is the stress amplitude of the first cycle, and $\sigma_i$ is the stress amplitude of the $i$th cycle after the second cycle. $\eta > 0$ means cyclic softening behavior. In contrast, $\eta < 0$ means cyclic hardening behavior. The softening/hardening degrees with number of cycles at different strain amplitudes of No.1 and No.2 samples are

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**Table 1. Types of secondary α phase.**

| Microstructure type | Aging temperature | Secondary α phase               |
|---------------------|-------------------|---------------------------------|
| No.1                | 480 °C            | (fine) intragranular α, GB α    |
| No.2                | 600 °C            | (coarse) intragranular α, GB α, WGB α |

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Figure 1. Microstructures of alloys aged at (a) 480 °C and (b) 600 °C for 4 h.

Figure 2. TEM images of secondary α phase. (a) Intragranular α of No.1 microstructure, (b) intragranular α of No.2 microstructure, (c) GB α of No.1 microstructure, (d) GB α and WGB α of No.2 microstructure.
shown in figure 4. According to figure 4(a), for No.1 samples, at strain amplitudes of 0.2% to 0.8%, the cyclic stress responses show cyclic hardening with a variable degrees. However, there is a continuous slight cyclic softening from beginning till failure at a high strain amplitude of 1.0%. According to figure 4(b), for No.2 samples, at low strain amplitudes of 0.2% to 0.6%, the cyclic stress responses show continuous cyclic hardening, but the hardening degrees are very low. When the high strain amplitudes of 0.8% and 1.0% are used, the cyclic stress responses exhibit an obviously cyclic softening trend.

3.4. LCF behavior

The curves of total strain amplitude versus reversals to failure are shown in figure 5(a). The LCF life feature can be characterized using the Coffin-Manson relationship between the plastic strain amplitude ($\Delta \varepsilon_p/2$) and the number of reversals to failure ($2N_f$), as equation (2) [20].

$$\Delta \varepsilon_p/2 = \varepsilon'f (2N_f)^c$$

where $\varepsilon'$ and $c$ are fatigue ductility coefficient and exponent, respectively. The plastic strain amplitudes are determined from the half-life hysteresis loops for all strain amplitudes. The Coffin-Mason plots of log ($\Delta \varepsilon_p/2$) versus log ($2N_f$) are shown in figure 5(b). Using equation (2) to fit the plots of figure 5, a linear relationship can be obtained between $\Delta \varepsilon_p/2$ and $2N_f$. According to figures 5(a) and (b), it can be found that the fatigue life of both No.1 and No.2 samples is increased with the decrease of strain amplitude. Under all strain amplitudes, the fatigue life of No.1 samples is significantly higher than that of No.2 samples at the same strain amplitude. It can be found that the present alloys exhibit different LCF life features under the influence of the secondary $\alpha$ phase.
3.5. Fatigue fracture behavior

The fracture surface morphologies at low and high strain amplitudes are shown in figure 6. It can be seen that the fatigue cracks all initiate at the free surface of samples, and the fatigue fracture surfaces can be all divided into three regions: fatigue crack initiation region (as showed by white arrows), fatigue crack propagation region (as surrounded by white dashed line) and fast fracture region. For both No.1 and No.2 samples, there is less fatigue crack initiation site at low strain amplitude (figures 6(a) and (b)). But more fatigue cracks initiated at the free surface under high strain amplitude (figures 6(c) and (d)). In addition, the area of fatigue crack propagation region at high strain amplitude is smaller than that at low strain amplitude. Correspondingly, the area of fast fracture region at high strain amplitude is larger than that at low strain amplitude.

Figure 7 shows the fatigue crack propagation region morphologies of No.1 and No.2 samples at the same strain amplitude. It can be observed that there are secondary cracks in the fatigue crack propagation region of
both No.1 and No.2 samples. By comparing figures 7(a) and (b), it can be found that the number of secondary cracks in the fatigue crack propagation region of No. 1 sample is larger than that of No. 2 sample. Moreover, the secondary cracks of No.1 sample are obviously deeper. Figure 8 shows the fast fracture region morphologies of No.1 and No.2 samples at low and high strain amplitudes. For No.1 and No.2 samples, there are plenty of dimples in the fast fracture regions. In addition, the number of dimples is larger, and the depth is deeper at low strain amplitude. At the same strain amplitude, the dimples of No.1 sample are deeper than those of No.2 sample. It can be speculated that the difference between secondary α phases can affect LCF crack initiation and propagation features.

Figure 7. Fatigue crack propagation regions at strain amplitude of 0.4%. (a) No.1 sample, (b) No.2 sample.

Figure 8. Fast fracture regions. (a) No.1 sample at low strain amplitude (0.4%), (b) No.2 sample at low strain amplitude (0.4%), (c) No.1 sample at high strain amplitude (1.0%), (d) No.2 sample at high strain amplitude (1.0%).
4. Discussion

In general, for near β titanium alloys, the phase transformation driving force is the dominating factor for precipitation during aging treatment [21]. Low aging temperature (480 °C) provides a high undercooling degree and brings about a large transformation driving force and a high nucleation rate. In addition, low elements diffusion rate caused by low aging temperature results in the low growth rate of precipitates. As a result, finely intragranular α forms in No.1 microstructure after the low temperature aging treatment. However, high aging temperature (600 °C) provides a large driving force for the intragranular α growth but reduces the nucleation driving force due to the relatively lower undercooling. Accordingly, in the case of No.2 microstructure, the number of intragranular α reduces, and the size of precipitated intragranular α increases. Besides, the inter-space among intragranular α becomes larger after being aged at a higher temperature. Predominant orientation of parallel intragranular α in No.2 microstructure can be attributed to the variant selection effect during heterogeneous nucleation of the secondary α phase. Song et al [22] reported a similar phenomenon, which occurred in Ti-10Mo-8V-1Fe-3.5Al alloy. They considered that the transformation strain of secondary α phase could be effectively accommodated with the dislocation strain field and resulted in preferable nucleation and growth of intragranular α. GB α forms preferentially during both low and high temperatures aging treatment, due to the supply of nucleation sites by large amounts of defects existing at prior β grain boundaries. Literature [23] assumed that the discontinuity of GB α attributed to the release of lattice distortion energy during aging treatment. With the formation of GB α, β-stabilizers get enriched at the vicinity of β grain boundaries. When the aging temperature is relatively low, the vicinity of β grain boundaries is chemically stable due to the enrichment of β-stabilizers. However, higher aging temperature enhances the diffusion of β-stabilizers, resulting in the stability decrease of the zone near the β grain boundaries. In addition, the high aging temperature can provide much driving force for precipitation. As a result, in No.2 microstructure, WGB α forms at the vicinity of β grain boundaries after high temperature aging treatment.

It is generally accepted that the cyclic stress response behavior and LCF life feature are mainly determined by the underlying LCF crack initiation and propagation [24, 25]. Figure 9 shows the TEM images of fractured fatigue samples. High dislocation density zones can be observed near the α/β interface in both No.1 and No.2 microstructures. It can be ascribed to that α and β phases have different hardness and undergo different deformation behavior, and result that the dislocation motions caused by plastic deformation are inhibited at α/β interface. Thus, two LCF crack nucleation mechanisms can be reasonably speculated as follows.

1. Compared with the β matrix strengthened by intragranular α, the GB α is relatively softer [26]. During LCF deformation, the activated dislocations pile up at the interfaces between GB α and β phase. Moreover, the differences of stress distribution along with zones near β grain boundaries, resulting from the differences in response to elastic and plastic deformation between prior β grains, make the plastic zone to be constrained mechanically to the vicinity of β grain boundaries [27]. This phenomenon can be more obvious with the existence of softer GB α. Thus, the crack nucleation is easy to occur near the β grain boundaries.

2. When stress or strain is high enough, multiple slip systems can be activated within β grains. Due to the considerable lattice mismatch, the interfaces formed by intragranular α and β matrix can act as effective dislocation barriers. The stress concentration due to dislocation aggregation at the interfaces of intragranular α and β matrix may also cause crack nucleation.

Figure 9. TEM images of the samples after fatigue tests. (a) No.1 microstructure, (b) No.2 microstructure.
At low strain amplitudes, the slip within $\beta$ grains is relatively difficult. Thus, the strain accumulation at the interfaces between GB $\alpha$ and $\beta$ phase is reasonably considered to be responsible for the LCF crack initiation. The LCF cracks initiate at grain boundaries and propagate along GB $\alpha$. However, GB $\alpha$ is discontinuous in No.1 microstructure (figure 2(c)). Therefore, cracks must transfer across the grain boundaries as the absence of GB $\alpha$. The variational crack front profile can consume large energy and provide a positive contribution to crack growth resistance [23]. However, in No.2 microstructure, because of the formation of WGB $\alpha$ (figure 2(d)), the LCF cracks propagate not only along GB $\alpha$ but also WGB $\alpha$ [16]. Therefore, the existence of WGB $\alpha$ promotes LCF cracks propagation. As a result, the No.1 samples present higher stress amplitude and fatigue life during LCF deformation at low strain amplitudes.

At high strain amplitudes, the stress is high enough to activate all of the LCF crack nucleation mechanisms mentioned above. The LCF cracks nucleate at multiple sites, i.e., the interfaces between GB $\alpha$ and $\beta$ phase as well as the interfaces between intragranular $\alpha$ and $\beta$ matrix. As a result, more fatigue cracks initiate from the free surface at high strain amplitudes (figures 6(c) and (d)). The significant stress amplitude difference between No.1 and No.2 microstructure can be attributed to the variant selection phenomenon of intragranular $\alpha$. It is difficult for dislocations in $\beta$ matrix to break through the barriers imposed by mutually-crossed $\alpha$ platelets. However, it is relatively easy for dislocations to bypass parallelly arranged $\alpha$ platelets [22]. Thus, dislocation glide is likely to be more easily accomplished in the matrix with parallel intragranular $\alpha$ (No.2 microstructure, figure 2(b)) than with mutually-crossed intragranular $\alpha$ (No.1 microstructure, figure 2(a)). In addition, the wider inter-particle spacing of No.2 microstructure can provide a longer effective slip range. As a result, the stress amplitudes of No.2 samples are lower than that of No.1 samples at high strain amplitudes.

During the fatigue crack propagation stage, the stress concentration due to dislocation aggregation at the interfaces between intragranular $\alpha$ and $\beta$ matrix promotes micro-cracks formation. The fine and crossed intragranular $\alpha$ of No.1 microstructure provides more $\alpha/\beta$ interfaces and higher stress levels than those in No.2 microstructure. It contributes to more sites and higher energy for micro-cracks formation. As a result, more and deeper secondary cracks form in the fatigue crack propagation region of No.1 samples (figure 7). As schematically shown in figure 10, during main fatigue crack propagation, the formation of more and deeper secondary cracks in No.1 samples causes more energy consumption and reduces the main crack propagation rate and results in longer LCF life for No.1 samples.

During the fast fracture stage, the samples are closely subjected to unidirectional loading rather than cyclic loading. Similar to the tensile fracture surface, the fast fracture regions of the No.1 and No.2 samples are all covered by lots of dimples. The fracture surfaces of No.1 samples tend to be rougher, and the quantity and depth of dimples are larger (figure 8). The rough crack profile consumes large energy and provides a positive contribution to crack growth resistance compared to the smooth crack path [23]. This is another explicit reflection of longer LCF life for the No.1 samples.

To sum up, for both No.1 and No.2 microstructures, the crack initiation is mainly caused by stress concentration due to dislocation aggregation at $\alpha/\beta$ interface. No.1 and No.2 microstructures show different crack initiation and propagation features due to their different secondary $\alpha$ phase compositions. For No.1 microstructure, discontinuous GB $\alpha$ consumes more energy during cracks transfer across. Besides, mutually-crossed and fine intragranular $\alpha$ provides more $\alpha/\beta$ interfaces and narrow slip range and results in the formation of more and deeper secondary cracks, which reduced the main crack propagation rate. In contrast, for No.2 microstructure, WGB $\alpha$ promotes LCF cracks propagation. Besides, the parallelly arranged intragranular $\alpha$ with wider inter-particle spacing has smaller resistance to dislocation glide and secondary cracks growth. These different fatigue cracks initiation and propagation features between No.1 and No.2 microstructures are right the underlying reasons for the differences in stress amplitude and LCF life.

![Figure 10](image)

**Figure 10.** Two-dimension schematic illustration showing the effect of secondary crack on main crack propagation for No.1 and No.2 samples.
5. Conclusions

In this work, low-cycle fatigue behavior of Ti-6Mo-5V-3Al-2Fe alloy with various types of secondary α phase was investigated. The following conclusions can be drawn from this work.

(1) After low temperature (480 °C) aging treatment, the discontinuous GB α formed on prior β grain boundaries and crossed finely intragranular α precipitated within β grains. After high temperature (600 °C) aging treatment, WGB α formed in the vicinity of β grain boundaries and parallel and coarse intragranular α precipitated within β grains.

(2) The discontinuous GB α provided more contribution to crack growth resistance. The crossed and fine intragranular α provided more dislocation slip barriers and inhibited the effective slip range. At the same strain amplitude, the stress amplitude and fatigue life of samples with discontinuous GB α and crossed intragranular α were overall higher than those of the samples with WGB α and parallel intragranular α.

(3) For the samples with discontinuous GB α and crossed intragranular α, more and deeper secondary cracks and dimples appeared on the fracture surfaces. This is the direct reflection of longer fatigue life for the samples with such microstructure.

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ORCID iDs

Hao-Yu Zhang https://orcid.org/0000-0001-5070-7547
Zheng-Yuan Li https://orcid.org/0000-0002-1515-4008
Xin Che https://orcid.org/0000-0003-0645-855X

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