Effect of microstructure on low-cycle fatigue property of Ti6321 alloy

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Abstract

The low-cycle-fatigue behavior of Ti6321 alloy with bimodal and lamellar microstructure have been investigated by analyzing of strain-cycles curve under different stress amplitudes. The results show that under the stress amplitude of 600 MPa, the fatigue life of lamellar microstructure is 6644 times, and the fatigue life of bimodal microstructure is 16252, which is more than twice that of the former. What’s more, two dislocation models of initiation and growth of cracks were demonstrated by analysis of fatigue fracture. The bimodal microstructure have the higher dislocation density and the shorter effective dislocation slip distance, which could hinder the initiation and propagation of fatigue cracks. The findings of low-cycle-fatigue property can provide data support and theoretical guidance for the safety assessment of titanium alloy pressure structures.

1. Introduction

Titanium and titanium alloys, owing to their high specific strength and excellent corrosion resistance which can effectively improve the safety and reliability of marine engineering equipment [1], have growing application in the field of deep-sea resource exploitation, seawater desalination and ship equipment. However, due to the lack of systematic supportive data of service performance in the marine environment, no evaluation system for marine titanium alloys has yet been established, restricting the application of titanium alloys in marine engineering [2]. With the development for higher-performance, lower-cost, deeper-dive and longer-life marine equipment, the demand of titanium alloys has become increasingly exigent [3]. Ti6321 alloys, a near-α titanium alloy developed for deep-sea submersibles, ships and offshore oil platforms with a nominal composition of Ti-6Al-3Nb-2Zr-1Mo, has outstanding properties such as corrosion resistance, toughness and solder ability compared with the Ti6Al4V alloy. Deep-sea equipment, such as deep-sea submersibles, often suffer from the alternating cyclic loading of seawater pressure when floating or diving during operation, leading to the damage due to low-cycle fatigue of pressure hull, and thus affecting the safety of deep-sea equipment [4–7].

Figure 1 shows the crack source of Ti6321 alloy pressure hull with a feature of fatigue failure. It demonstrated that the crack failure resulted from the accumulation of cracks in the deformation process of the hull. The process of floating and diving was studied in a simulated deep-sea environment by alternating dynamic external pressure, which produced the alternating stress resulting in the partial fatigue failure. The initiation of fatigue cracks is one of the most important stages in the fatigue fracture processes of metals. A large number of metallographic observations were carried out to elucidate the micro mechanisms responsible for crack initiation. The site of crack initiation varies depending on the microstructures of the material involved and types of applied stresses. The research on the initiation and propagation of short fatigue cracks in metals and alloys has received increasing attention, because the subject has great importance both for the distinct behaviour of short cracks that cannot be interpreted by conventional theories, and for engineering interests since the stage of short-crack damage may occupy a large portion of total fatigue life.

As a consequence, the fatigue property of titanium alloy under cyclic loading is the important basis of life evaluation and safety checking of high-pressure structures. With the higher performance requirement of the
pressure hull materials applied for submersibles to cope with the more harsh marine engineering, the low-cycle-fatigue of titanium alloys is an important subject in the design and verification of deep-sea pressure structures [8–13].

The mechanical properties and stress corrosion of Ti6321 were extensively researched to further enhance its comprehensive property. However, there are few related reports on the low-cycle fatigue of Ti6321 alloy as a new type titanium alloy. Therefore, to study the effect of microstructure on low-cycle fatigue properties of Ti6321 alloy, the phase transformation point of Ti6321 alloy was measured by calculation method in this paper to establish the heat treatment process obtaining different microstructures. To calculate accurately for the phase transformation point, the calculated result was compared with the experimental results by different thermal analysis (DTA) methods. Furthermore, low cycle fatigue behavior of Ti6321 alloy with bimodal and lamellar microstructure was investigated by analysis of strain-cycles curve with different stress amplitudes. In addition, the fracture morphology of the specimen was analyzed using scanning electron microscopy in order to further interpret the mechanism of fatigue fracture. The dislocation models of initiation and growth of cracks were supposed to establish in this paper. The dislocation models of initiation and growth of cracks were illustrated by analyzing the fatigue cracks on high-cycle fatigue (HCF) properties [8, 9]. The aim of the present paper is to investigate the effects of different microstructure on the low-cycle-fatigue behavior of Ti6321 alloy. And the dislocation models of initiation and growth of cracks were illustrated by examining the microstructure and fracture in detail, so as to provide a basis for developing new high strength titanium alloy with optimized low cycle fatigue properties.

### 2. Materials and experimental procedure

The ingot of Ti6321 alloy in the experiment was casted three times using vacuum arc re-melting method, the chemical compositions of which are shown in table 1. Titanium alloy ingots are forged, stamped and rolled to titanium plate. Figure 2(a) shows the rolled microstructure of optical microscope (OM), while figure 2(b) is the amplifying image of OM. It can be seen from figure 2 that the coarse structure consists of large number of lamellar α phase and little β phase, which is typical Widmanstätten structure.

The microstructure was observed through OLYMPUS GX71 metallographic microscope. The solution used for etching metallographic samples of Ti6321 alloy has a composition that the volume ratio of hydrofluoric acid, nitric acid and water was 1:3:20. The etching time was 6s. Subsequently, the etched sample was cleaned by ultrasonic vibration to remove the remaining impurities on the specimen’s surface. The volume fraction and size of each phase were measured by using pro-imaging metallographic analysis software and Image Pro Plus (IPP) professional image analysis software.

![Figure 1. The crack source of Ti6321 alloy pressure hull.](image)

**Table 1.** Chemical composition of Ti631 alloy (wt%).

|   | Al  | Nb  | Zr  | Mo  | C   | O   | H   | N   | Fe  | Si  | Ti  |
|---|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
|   | 6.16| 2.90| 2.08| 1.04| 0.004| 0.079| 0.0008| 0.0098| 0.024| 0.013| Bal.|

![Amplification](image)
The low-cycle fatigue test was carried out in the electro-hydraulic servo low-cycle fatigue testing machine (MTS370). The selected direction of the sample was parallel to the rolling direction of the substrate. The specific size of the sample is shown in figure 3. The control stress level is respectively 500, 600, 700, 800 MPa, and the stress ratio is $-1$. In the loading mode of the usual symmetric cycle-pressure load, the criterion of titanium alloy material failure is the fatigue fracture of specimen in the room temperature. Trapezoidal wave was adopted in this test. Besides, the load frequency and the holding time is 0.1 Hz and 2s, respectively.

3. Results and discussion

Ti6321 alloy is specially designed for the deep-sea submersible and pressure structures, which contains a variety of chemical compositions to improve the corrosion resistance and toughness. The compositions and impurity element affect the phase transformation point of Ti6321 alloy, which is the critical temperature of $\alpha + \beta$ phase to $\beta$ phase transition, and is the key parameter to obtain different microstructures by heat treatment. Therefore, to study the effect of different microstructures on low-cycle fatigue properties of Ti6321 alloy, the phase transformation point should be measured, and then make sure the heat treatment temperature to acquire the bimodal and lamellar microstructure based on the phase transformation point.

3.1. Calculation for phase transformation point of Ti6321 alloy

It is important to accurately calculate the phase transformation point in order to determinate the heat treatment process obtaining different microstructures of Ti6321 alloy. The phase transition temperature of titanium alloy is affected by the type and content of alloy and impurity elements. The calculation method selected in the experiment is to calculate the phase transition point of Ti6321 alloy according to the influence of each element on the temperature of titanium alloy phase transition.

The main alloy elements of Ti6321 alloy are Ti, Al, Nb, Zr and Mo. Among them, the solid solubility of Al, an $\alpha$ stabilizing element, can reach 7.5% in $\alpha$-Ti, which can significantly improve the temperature of phase transition point. Nb and Mo, stabilizing elements of $\beta$ phase, have infinite solubility in $\beta$-Ti, but also finite solubility in $\alpha$-Ti, which can significantly reduce the phase transition temperature of titanium alloy. Zr is a common neutral element in titanium alloy, which has a high solubility in both $\alpha$-Ti and $\beta$-Ti, and has little

![Figure 2. Rolled microstructure of optical microscope (a) and the amplifying image of OM (b).](image1)

![Figure 3. Dimension of the sample for low-cycle fatigue test (All dimensions are in mm).](image2)
The effect of element content on phase transition temperature of Ti6321 alloy.

| Type of element | Name of element | Content of element (wt%) | Difference | Accumulation |
|-----------------|-----------------|--------------------------|------------|--------------|
| Stable elements of α phase | Al | 0 ~ 2.0 | +14.5 °C/1.0% | +29.0 °C |
| | | 2.0 ~ 27.0 | +23.0 °C/1.0% | +143.0 °C |
| | N | 0 ~ 0.5 | +5.5 °C/0.01% | — |
| | O | 0 ~ 1.0 | +2.0 °C/0.01% | — |
| | C | 0 ~ 0.15 | +2.0 °C/0.01% | +3.0 °C |
| Stable elements of β phase | Mo | 0 ~ 5.0 | −5.5 °C/1.0% | −27.5 °C |
| | Fe | 0 ~ 15.0 | −16.5 °C/1.0% | — |
| | Nd | 0 ~ 10.0 | −8.5 °C/1.0% | — |
| | Si | 0 ~ 0.45 | −1.0 °C/0.1% | — |
| | H | 0 ~ 5.0 | −5.5 °C/0.01% | — |
| Neutral element | Zr | 0 ~ 10.0 | −2.0 °C/1.0% | — |

Influence of element on the temperature of transition α + β phase to β phase

\[
T_{\alpha + \beta/\beta} = 885 °C + \sum M_i \times M_{id}
\]  

Where \( M_i \) is the content of element and \( M_{id} \) is the influence difference of chemical elements on the temperature of transition \( \alpha + \beta \) phase to \( \beta \) phase.

Considering the content of elements measured in the table 1, the process can be calculated by,

(i) Effects of α stabilizing elements on the temperature of phase transition

\[ \text{Al: } 2\% \times (14.5 °C/1.0\%) + (6.16 - 2.0\%) \times (+23.0 °C/1.0\%) = +124.68 °C \]

\[ \text{N: } 0.0098\% \times (+5.5 °C/0.01\%) = +5.39 °C \]

\[ \text{O: } 0.079\% \times (+2.0 °C/0.01\%) = +15.8 °C \]

\[ \text{C: } 0.004\% \times (+2.0 °C/0.01\%) = +0.8 °C \]

(ii) Effects of β stabilizing elements on the temperature of phase transition

\[ \text{Nb: } 2.9\% \times (-8.5 °C/1.0\%) = -24.65 °C \]

\[ \text{Mo: } 1.04\% \times (-5.5 °C/1.0\%) = -5.72 °C \]

\[ \text{Fe: } 0.024\% \times (-16.5 °C/1.0\%) = -0.4 °C \]

\[ \text{Si: } 0.013\% \times (-1.0 °C/0.1\%) = -0.13 °C \]

\[ \text{H: } 0.0008\% \times (-5.5 °C/0.01\%) = -0.44 °C \]

(iii) Effects of neutral element on the temperature of phase transition

\[ \text{Zr: } 2.08\% \times (-2.0 °C/1.0\%) = -4.16 °C \]

Therefore, Substituting into equation (1), the phase transformation point of Ti6321 alloy is calculated as follow:

\[ T_{\alpha + \beta/\beta} = 885 °C + 124.68 °C + 5.39 °C + 15.8 °C + 0.8 °C - 24.65 °C - 5.72 °C - 0.13 °C - 0.44 °C - 4.16 °C = 996.17 °C \]

And then compared with the experimental results by different thermal analysis (DTA) method, the calculation method is reliable to predict the phase transformation point of Ti6321 alloy in order to establish the heat treatment process obtaining different microstructures. The bimodal and lamellar microstructure used in this low-cycle-fatigue experiment were obtained by solid solution and aging treatment below (980 °C/1 h/AC + 5 50 °C/4 h/AC) and above (1020 °C/1 h/AC + 5 50 °C/4 h AC⁻¹) the β phase transformation point, respectively.

Figure 4(a) shows the bimodal microstructure of Ti6321 alloy with primary equiaxed α phase and partially transformed β phase containing acicular α. Among that, the average size of primary equiaxed α phase is 8.6 μm,
which accounts for 46.8%. Figure 4(b) shows the lamellar microstructure presenting distinct original boundary of $\beta$ phase and the platelet thickness of secondary $\alpha$ phase is $0.5 \sim 1.6 \mu m$.

3.2. Relationship of Strain amplitudes and fatigue life to different microstructures
The fatigue life curves of lamellar and bimodal microstructures in $500 \sim 800$ MPa under different stress amplitudes are shown in figure 5. It can be found that for either bimodal or lamellar microstructure, low-cycle fatigue life decreases significantly with the total stress amplitude increases. Under the same stress amplitude, the bimodal specimen showed a longer fatigue life. Among them, under the stress amplitude of 600 MPa, the fatigue life of lamellar microstructure is 6644 times, and the fatigue life of bimodal microstructure is 16252, which is more than twice that of the former; Under the stress amplitude of 750 MPa, the fatigue life of lamellar microstructure is 529 times, and the fatigue life of bimodal microstructure reaches 850 times. It can be found that under the high stress amplitude, the gap of fatigue life is narrowing.

3.3. Analysis of fracture morphology
As the cyclic softening characteristics of Ti6321 alloy are most obvious under the stress amplitude condition of 750 MPa, which are chosen to analysis the fracture morphology. The fatigue fracture morphology of the alloy bimodal and lamellar microstructure samples under this stress amplitude condition is shown in figure 6. It can be found that the fatigue fracture of bimodal and lamellar microstructure samples is similar, which is composed of crack source (region A), crack propagation region (region B) and transient fracture region (region C).

In the low-cycle fatigue experiment, due to adopting the stress control and large stress amplitude, once the crack is formed, it will quickly generate macro crack and accelerate its growth until material failure, so the crack source (region A) and crack propagation region (region B) occupy a smaller area in the whole fatigue section. It
can be found that, on the other hand, compared with the lamellar microstructure samples, the crack source area of bimodal samples is larger and the macroscopic fracture morphology is relatively flat. At the same time, the crack source region of bimodal microstructure is clear and smooth, and around the source of crack the crack propagation outward growth in a radial pattern.

There are obvious differences in the crack source region, crack propagation region and transient fracture region of bimodal microstructure. In comparison, the macroscopic fracture morphology of lamellar microstructure is relatively rough, and the geometric surface on the fracture surface of the sample is larger leaf-like or block-like structure, as shown in figure 6(b) (region D), which is related to the size and shape of $\beta$ phase in lamellar microstructure.

The micro fracture morphology of bimodal and lamellar microstructure samples is shown in figure 7.

Around the crack source, the initial crack propagation of samples are all in the form of the radial outward growth for bimodal and lamellar microstructure. Due to the large extension resistance, the extension deflect along the long axis direction of the secondary $\alpha$ cluster, when the initial crack encounters the $\alpha$ cluster which is approximately perpendicular to the direction of crack propagation. The deflection of crack recurs and the secondary cracks easily appears while the initial crack encounters the next $\alpha$ cluster which is approximately perpendicular to the direction of crack propagation.

In the crack propagation region of bimodal microstructure sample, many cleavage steps of the brittle fracture characteristics occur, which are related to the transfer $\beta$ phase (that is the secondary $\alpha$ phase). However, many tearing ridges consisting of many flat areas occur in the crack propagation region of lamellar microstructure specimen.

In the transient fracture region, the fracture surfaces present the morphology of dimple in the specimens of two kinds of microstructures. Because of the effect of stress on the specimens, a large number of micro pores were produced inside the materials, and the pores gradually grew up along with the development of plastic deformation. As a result, the specimens show the fracture morphology of micropores coalescence. But under the same stress level the dimples of bimodal microstructure is deeper than the lamellar microstructure, which illustrates the worse plasticity of lamellar microstructure Ti6321 alloy material.

To further interpret the mechanism of fatigue fracture, the cross-section morphology of fatigue fracture along the fatigue crack propagation direction is shown in the figure 8. It can be seen from the figure that the fracture forms of both bimodal and lamellar microstructure samples are composed of transgranular and intergranular fracture. Because of the longer fatigue life and more cycle-index, $\alpha$ phase showed signs of being elongated in the fracture process of bimodal microstructure samples. The crack expanded along the equiaxial $\alpha$ phase and transfer $\beta$ phase, while the fracture form of lamellar microstructure samples was along and across the lamellar layer $\alpha$ phase.

As we know, initiation and propagation of short fatigue cracks are important stages of material damage [11]. Furthermore, this stage accounts for eighty percent of the total fatigue life [12]. It is generally considered that if the length crack does not meet the validity requirement of linear elastic fracture mechanics, it is called the short crack. Because the stage of short-crack initiation and propagation occupy a large portion of total material’s fatigue life, it has great importance both for engineering interests and for the distinct behavior of short cracks, which cannot be interpreted by conventional theories.

The existing research results show that the microstructure of material has a distinct impact on the initiation of short fatigue cracks. In addition, the initiation of short fatigue cracks is also related to local plastic
deformation, or slide, under periodic loads. The local hardening in the material surface is caused by the plastic deformation of micro zone under the action of alternating stress. When the hardening reaches the saturation value, the continuous movement of dislocation will trigger the metastable dislocation pile-up group, which can

Figure 7. Morphologies of the fatigue fracture surfaces of Ti6321 alloy: (a) BM and (b) LM.

Figure 8. Morphologies of fatigue fracture section: (a) BM and (b) LM.
cause dislocation collapse and create new slides. As a result, the resident slip bands are formed. The dispersed short cracks possess an orientation preference, which is associated with the crystalline orientation of the relevant slip system. Under the action of alternating load, the slide on the resident slip band will lead to the phenomenon that the local area of the resident slip band is above or below the surface of grain, namely the jagged shape, which is called as the ‘extrusion’ and ‘intrusion’. The accumulation of this process will create the initiation of short fatigue cracks. The schematic for this process is shown in the figure 9. Two dislocation models of initiation and growth of cracks were illustrated. The condition of the growth of the crack embryo will be treated from the viewpoint of energy balance. If the stored strain energy due to dislocations accumulated after n-cycles becomes equal to the surface energy, the layers of dislocation dipoles can be transformed into a free surface.

Figure 9. Two types of crack initiation: (a) Crack initiation from vacancy dipoles or extrusion, (i) crack embryo, (ii) subsequent growth; (b) Crack initiation from intrusion, (i) crack embryo, (ii) subsequent growth.

Figure 10. TEM image of transform β in bimodal microstructure.

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Figure 10 shows the TEM image of bimodal. There is a higher dislocation density in the primary α phase of bimodal, and α phase accounts for a larger percentage of the whole bimodal microstructure, which plays an important role in blocking for the crack initiation stage so as to enhance ability to resist fatigue crack. In addition, the dispersed short cracks possess an orientation preference, which is associated with the crystalline orientation of the relevant slip system. The complexity of crack initiation is related to the effective dislocation slip distance which is determined by the size of α cluster commanded of primary β grains. The size of primary β grains is smaller and the effective dislocation slip distance is shorter in the bimodal microstructure so that the critical shear stress crack initiation required become larger. By contrast, because the primary β grains is coarser in the lamellar microstructure (figure 2(b)), the dislocation slip easily and the effective dislocation slip distance is longer. Due to the irreversibility of slipping, the dislocations tend to plug at the boundary of β grains or at boundary of the secondary α phase, which results in stress concentration. As a result, the fatigue cracks initiated more easily in the lamellar microstructure samples.
4. Methodology

(i) Ti6321 alloy is a new type titanium alloy developed for deep-sea submersibles. The composition of the titanium alloy contain more chemical element. To accurately calculate the phase transformation point, the method of calculation and experiment were integrated into account. By comparing the DTA experimental results, the calculation method is reliable to predict the phase transformation point of Ti6321 alloy.

(ii) Low-cycle fatigue behavior of Ti6321 alloy with bimodal and lamellar microstructure was investigated by analyzing the strain-cycle curves with different stress amplitudes.

(iii) Two dislocation models of initiation and growth of cracks were illustrated by analyzing fracture morphology and TEM images.

5. Conclusions

The fatigue property of titanium alloy under cyclic loading is the important basis of life evaluation and safety checking of pressure structures. There are few related reports on the low-cycle-fatigue of Ti6321 alloy as a new type titanium alloy. The site of crack initiation varies depending on the microstructures of the material involved and types of applied stresses. In this paper, low-cycle fatigue behavior of Ti6321 alloy with bimodal and lamellar microstructure was studied with different stress amplitudes. Two dislocation models of initiation and growth of cracks were illustrated by analyzing of fracture morphology and TEM image.

Results indicated that the volume fraction of equiaxed $\alpha$ phase is about 26.6% for the bimodal microstructure, while the average grain size is approximately 7.8 $\mu$m. At the same time, the thickness of $\alpha$ phase layer is about 0.5 ~ 2.0 $\mu$m for lamellar microstructure. To accurately calculate the phase transformation point is important to establish the heat treatment process obtaining different microstructures of Ti6321 alloy. Furthermore, it can provide supporting data and theoretical basis for the safety assessment of titanium alloy pressure structures.

(1) Low-cycle-fatigue life increases with the decrease of total stress amplitude;

(2) Under the same stress amplitude, the bimodal specimen showed a longer fatigue life;

(3) The initiation and propagation of short fatigue cracks accounts for a high percentage of the total fatigue life. The presence of equiaxed $\alpha$-phase with high dislocation density could also hinder the initiation and propagation of fatigue cracks for the bimodal microstructure;

(4) Fracture morphology is flat and smooth for the sample with bimodal microstructure, however, some geometric facets associated with the original coarse-grained $\beta$ are found in the sample with lamellar microstructure.

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

Disclosure statement

No potential conflict of interest was reported by the authors.

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Data availability statement

All data generated or analyzed during this study are included in this published article.
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