High-cycle fatigue properties at cryogenic temperatures in forged- and rolled-Ti–5% Al–2.5% Sn ELI alloys

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Abstract

High-cycle fatigue properties were investigated at 4, 77 and 293 K in Ti–5% Al–2.5% Sn ELI alloys, in which mean alpha grain sizes were about 30 µm in the rolled material and 80 µm in the forged material. The ultimate tensile strengths of both materials were almost same and increased with decreasing temperature. The fatigue strength of each material also tended to increase with decreasing temperature. At 293 K, the fatigue strength of each material was almost equivalent. At 4 and 77 K, however, the fatigue strength of the rolled material was higher than that of the forged material. Concerning the rolled material, the fatigue strengths at 10^6 cycles at 4 and 77 K were about 1.6 and 1.5 times higher than that at 293 K, respectively. On the other hand, in the forged material, it should be noted that the fatigue strengths in longer-life region (over 10^6 cycles) were almost equivalent not depending on test temperatures. Fatigue cracks initiated in the specimen interior independently of test temperatures and materials (we call this type of crack initiation ‘sub-surface crack initiation’) and formed facet-like structures at the sub-surface crack initiation sites at 4 and 77 K. The size of each facet-like structure corresponded closely to the grain size itself. The sizes of crack initiation sites were smaller in the rolled material than in the forged material. Since sub-surface cracks, which form facets or crack initiation sites, are supposed to act as defects, it is concluded that grain refinement leads to reduce the size of crack initiation site and this contributes effectively to improve the fatigue strength in high-cycle region at cryogenic temperatures.

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Keywords: Alpha-type titanium alloy; High-cycle fatigue properties; Cryogenic temperature; Grain size; Sub-surface crack initiation

1. Introduction

In recent years, the mechanical properties in structural materials used for Japanese-built launch vehicles have been studied with the aim of increasing their reliability [1,2]. Ti–5% Al–2.5% Sn ELI alloy, which is a representative alpha (α)-type titanium alloy, is used for liquid hydrogen turbo-pumps. The fatigue properties in this alloy need to be investigated in detail [1,3], since a fatigue fracture was found in the part of the liquid hydrogen turbo-pump which was made of this titanium alloy and it is supposed to have caused the failure of H-II rocket No. 8 [4,5].

As regards Ti–5% Al–2.5% Sn ELI alloy, it has been reported that fatigue cracks initiate in the test specimen interior (hereafter, we call this type of fatigue crack initiation ‘sub-surface crack initiation’) under uniaxial loading at liquid helium temperature (4 K) [6]. It has been also reported that the sub-surface crack initiation is associated with the microstructure rather than specific defects such as inclusions [7]. Additionally, the sub-surface crack initiation reduces the fatigue properties in the longer-life region in various materials including titanium alloys [8–22].

The present study examines the fatigue properties in forged and rolled Ti–5% Al–2.5% Sn ELI alloys with different α grain size at 4, 77 and 293 K, and fracture surface of each specimen. The fatigue properties in these alloys were discussed in terms of their correlation with the sub-surface crack initiation sites.

2. Experimental procedure

Ti–5% Al–2.5% Sn ELI alloy was cast into a cylindrical mold of 480 mm in diameter, hot-forged in the beta (β) region (1473 K heating) and then in the α + β region (1243 K heating). After these processes, the forged material
was annealed at 1073 K for 7.2 ks. It was then air-cooled and machined to 180 mm in diameter \( \times \) 113 mm long to provide the sample billets (hereafter, we call these samples ‘forged material’). For controlling \( \alpha \) grain size, some billets were hot-forged again and hot-rolled in \( \alpha + \beta \) region (1243 K heating) to 28 mm thickness plates. Finally, these plates were annealed at 1073 K for 7.2 ks, followed by air-cooling (hereafter, we call this samples ‘rolled material’). The chemical composition of the present material is shown in Table 1. The microstructures of these materials were observed by optical microscopy. Samples for optical microscopy were chemically etched with a solution of 2\% HF–8\% HNO\(_3\)–90\% H\(_2\)O. Mean \( \alpha \) grain size of each specimen was determined by comparing optical micrographs with the ASTM E112 grain size standards.

Tensile tests and fatigue tests were carried out at 4, 77, and 293 K. Tensile tests were carried out using cylindrical specimens of 6.25 mm in diameter and 35 mm in gauge length and at an initial strain rate of \( 2.4 \times 10^{-4} \) s\(^{-1}\). The fatigue test specimens had an hourglass-type with a minimum diameter of 4.5 or 6 mm. To verify the influence of sampling location and anisotropy of the billets on its properties, tensile specimens of the forged material were taken from both the inner part and the outer part of the billets as well as in the axial and radial directions [1]. Fatigue specimens of the forged material were cut from only the inner part and in the radial direction, since these specimens were large [1]. On the other hand, the tensile test specimens of the rolled material were cut parallel to transverse direction (TD) and rolling direction (RD) of the plate. The fatigue specimens of the rolled material were cut parallel only to TD of the plate. Fatigue tests were carried out using sinusoidal waveform loading and uniaxial loading under stress ratio (\( R \)) of 0.01. Test frequencies of 4 Hz at 4 K and 10 Hz at 77 and 293 K were chosen to minimize specimen temperature rise. Fracture surfaces and crack initiation sites of failed specimens were observed by scanning electron microscopy (SEM).

### Table 1

| Al   | Sn   | Fe  | O    | H    | N    | Ti   |
|------|------|-----|------|------|------|------|
| 4.82 | 2.40 | 0.21 | 0.06 | 0.008| 0.004| bal. |

3. Experimental results

#### 3.1. Microstructure and tensile properties

Fig. 1 shows optical micrographs of the Ti–5\% Al–2.5\% Sn ELI alloys used in this study. The microstructures of the present materials consist of \( \alpha \) and retained \( \beta \) phases. The mean sizes of \( \alpha \) grains are about 80 \( \mu m \) in the forged material (a) and 30 \( \mu m \) in the rolled material (b). Retained \( \beta \) phase exists on grain boundaries or in the \( \alpha \) grains in both materials. Additionally, retained \( \beta \) phase exists almost parallel to RD in the rolled material. Retained \( \beta \) phase has been confirmed by electron probe microanalysis to be an iron-rich phase.

The 0.2\% proof stress and the ultimate tensile strength of the present materials are summarized in Table 2. Data for the forged material are averages since there was no notable difference among data according to sampling location or anisotropy [1]. In the rolled material, there was also no pronounced difference among data depending on sampling direction. The 0.2\% proof stress and the ultimate tensile strength of both materials increased with decreasing temperature, and both properties at 4 and 77 K are about

![Fig. 1. Optical micrographs of Ti–5\% Al–2.5\% Sn ELI alloys used: Forged (a) and forged and rolled (b) in the \( \alpha + \beta \) region (1243 K heating) before annealing at 1073 K for 7.2 ks, followed by air-cooling.](image-url)
1.8 and 1.6 times higher than those at 293 K, respectively. The tensile properties in the present materials are almost the same level although microstructure of each material is different.

3.2. Fatigue properties

Fig. 2 shows S–N diagram at 4, 77 and 293 K for the present materials. The relevant numerical data of stress amplitude ($\sigma_a$) and number of cycles to failure ($N_f$) are listed in Table 3. The fatigue strength of each material tends to increase with decreasing temperature. At 293 K, the fatigue strength of each material is almost same. At 4 and 77 K, however, the fatigue strength of the rolled material is higher than that of the forged material. Concerning the rolled material, the fatigue strengths at $10^6$ cycles at 4 and 77 K are about 1.6 and 1.5 times higher than that at 293 K, respectively. On the other hand, in the forged material, it should be noted that fatigue strengths over $10^6$ cycles are about 300 MPa not depending on test temperatures. This result indicates that the low-temperature fatigue strength in high-cycle region of the material with coarser grains does not increase in response to increments in tensile strength.

3.3. Fractography

Figs. 3–6 are typical SEM micrographs showing the fracture surfaces of the present materials fatigue-tested at 4 and 293 K. Photographs (a) show the fracture surfaces with a low magnification and photographs (b) show the vicinity of fatigue crack initiation sites. In the present materials, fatigue cracks initiated in the specimen interior irrespective of materials or test temperatures. However, aspects of the fatigue crack initiation sites are quite different depending on test temperature. At 4 K, a limited number of crystallographic facet-like structures (hereafter, we abbreviate this term to ‘facets’) were formed at the fatigue crack initiation sites, and each facet was inclined with respect to the stress axis (Figs. 3(b) and 4(b)). These aspects of the fatigue crack initiation site were also observed in the specimens tested at 77 K. As regards facets observed in the specimens tested at cryogenic temperatures, size of each facet was clearly smaller in the rolled material than in the forged material, and the facet size of each material was found to correspond closely to the grain size itself. The size of the fatigue crack initiation site, which consists of several facets, was evaluated in $\sqrt{\text{area}}$ and summarized in Table 3. The $\sqrt{\text{area}}$ values are smaller in the rolled material than in the forged material, and 2–4 times larger than the grain size itself in each material.

On the other hand, at 293 K, unlike those observed at cryogenic temperatures, there were no facets that can be clearly identified as crack initiation sites in both materials (Figs. 5(b) and 6(b)).
Figs. 3(c) and 5(c) show SEM images of the crack propagation region in the forged materials tested at 4 and 293 K, respectively. They were observed at about 1 and 1.5 mm distance from each fatigue crack initiation site as shown Figs. 3(a) and 5(a), respectively. In both specimens, ductile striations were formed independently of test temperature. The same aspects of them were also observed in the rolled material. These results mean that the present materials themselves do not exhibit brittle fracture at cryogenic temperatures, although the brittle-like facets were observed at the sub-surface crack initiation sites.

4. Discussion

It is known that there is a good correlation between fatigue limit ($\sigma_w$: fatigue strength at $10^7$ cycles) and the ultimate tensile strength ($\sigma_u$) in case where the fatigue crack initiates at the specimen surface, and this has been characterized by the following empirical equation [20,21,23]:

$$\sigma_w = C\sigma_u$$  \hspace{1cm} (1)

where $C$ is the constant. At room temperature (293 K), $C$ is 0.522 under $R = -1$ on various tempered martensitic steels and aluminum alloys [23], and $C$ is 0.419 under $R = 0$ on various tempered martensitic steels [21]. Reflecting this relationship for fatigue limit, the relationship between the stress amplitude to the ultimate tensile strength ($\sigma_a/\sigma_u$) and number of cycles to failure ($N_f$) is expressed as an unique curve for the materials in which the fatigue crack initiates at the specimen surface [23].

Fig. 7 shows the relationships between $\sigma_a/\sigma_u$ and $N_f$ of the present materials. At 293 K, $\sigma_a/\sigma_u$ values of both materials are nearly equivalent. The $\sigma_a/\sigma_u$ at $10^6$ cycles is about 0.365 in each materials (Fig. 7) and is somewhat lower than that of steels ($C = 0.419$). This result and SEM images shown in Figs. 5(b) and 6(b) represent that, in case where there are no facets that can be clearly identified as
crack initiation sites, the fatigue properties show almost same tendency with those of the materials in which the surface crack initiation occurs. Therefore, most of the fatigue life of the present materials is supposed to be spent in crack initiating at 293 K Eylon et al. [24] investigated the effect of microstructure on crack initiation behaviour in Ti–6% Al–4% V alloy using flat center-notched specimens. They reported that the number of cycles for stable crack propagation could be neglected compared to the number of cycles for crack initiation, especially near fatigue limits, whereas Ns was much smaller than Nf.

On the other hand, at 4 and 77 K where the facets were formed at the crack initiation sites, σf/σu vs. Nf curves are

Fig. 5. SEM micrographs of fracture surface (a), fatigue crack initiation site (b) and fatigue crack propagation region in about 1.5 mm distance from sub-surface crack initiation site (c) for Ti–5% Al–2.5% Sn ELI alloy fatigue tested at 293 K: α grain size is 80 μm, σu = 275 MPa, Nf = 7,186,900 cycles.

Fig. 6. SEM micrographs of fracture surface (a) and fatigue crack initiation site (b) for Ti–5% Al–2.5% Sn ELI alloy fatigue tested at 293 K: α grain size is 30 μm, σu = 281 MPa, Nf = 109,440 cycles.

Fig. 7. Relationships between ratio of stress amplitude (σa) to ultimate tensile strength (σu) and number of cycles to failure.
lower than those at 293 K in both materials. In particular, \( \sigma_a/\sigma_u \) decreases drastically in the forged material. These results suggest that the sub-surface cracks, which form facets, initiate in the early stage of the fatigue life even at lower stress level and act as defects at cryogenic temperature. This crack initiation behaviour lead to shorten the fatigue life and then results in the lower \( \sigma_a/\sigma_u \) at cryogenic temperature.

Fatigue limits (\( \sigma_u \)) in the case of fish-eye fractures, in which the sub-surface fatigue crack originates from internal inclusions, were accurately estimated by applying Murakami’s equation [20]:

\[
\sigma_u' = \frac{1.56(HV + 120)}{(\sqrt{\text{area}})^{16}} \left[ 1 - R \cdot \frac{1}{2} \right]
\]

where \( R = \sigma_{\min}/\sigma_{\max} \) and \( \alpha = 0.226 + HV \times 10^{-4} \). HV is Vickers hardness in kgf/mm\(^2\) and \( \sqrt{\text{area}} \) is the inclusion size of fracture origin in \( \mu \text{m} \). Moreover, Furuya et al. [18] reported that a relationship between \( \sigma_a/\sigma_w' \) and \( N_i \) is shown as an unique curve independently of inclusion sizes in high-strength steels.

Fig. 8 shows \( \sigma_a/\sigma_w' \) vs. \( N_i \) curves of the present materials at 4 and 77 K. We used \( \sqrt{\text{area}} \) of the fatigue crack initiation site, since the sub-surface cracks, which consist of several facets, are supposed to act as defects same as inclusions. HV values at cryogenic temperatures of the present materials were estimated as follows: At 293 K, HV values are 270 in both materials and ratios of the ultimate strength \( \sigma_u \) to HV are 2.74 in the forged materials and 2.78 in the rolled materials. We assumed that these relationships between \( \sigma_u \) and HV could be obtained even at cryogenic temperatures, and estimated HV values at 4 and 77 K of the present materials.

The fatigue life plots in the normal S–N diagram (Fig. 2) were almost expressed as an unique curve in the modified S–N diagram (Fig. 8). This diagram indicates that the S–N curves at 4 and 77 K of both materials slide in the vertical direction to fit the fatigue limits at the same level. However, there is somewhat difference in the values of \( \sigma_a/\sigma_w' \) in both materials, and they tend to be still higher in the rolled material than in the forged material. This is supposed to result from the difference in the microstructure, especially dispersion condition of \( \beta \), as observed in Fig. 2. Size, volume or morphology of \( \beta \) is supposed to affect the fatigue properties, since it has been discussed that \( \beta \) is expected to affect the subsurface crack initiation behaviour [10,13]. Although we have to take them into consideration to evaluate the fatigue properties, on the result of root-area analysis as shown in Fig. 8, it can be concluded that grain refinement leads to reduce the size of crack initiation site, and this is supposed to be the main factor on the improvement of the fatigue strength in high-cycle region at 4 and 77 K. In other words, it is important to refine the \( \alpha \) grains for obtaining the better fatigue strength at cryogenic temperature in Ti–5% Al–2.5% Sn ELI alloy.

5. Conclusions

High-cycle fatigue properties were investigated at 4, 77 and 293 K in forged- and rolled-Ti–5% Al–2.5% Sn ELI alloys with a mean grain size of 30 and 80 \( \mu \text{m} \), respectively. The fatigue properties of these alloys are discussed in terms of how they correlate with the fatigue crack initiation sites. The results obtained are as follows

1. The 0.2% proof stress and the ultimate tensile strength of both materials increase with decreasing temperature.

2. At 293 K, the fatigue strengths of both materials are almost equivalent. On the other hand, at cryogenic temperatures, the high-cycle fatigue strength is higher in the rolled material than in the forged material. Concerning the forged material with 80 \( \mu \text{m} \) grains, the fatigue strengths in longer-life region (over \( 10^6 \) cycles) are almost equivalent not depending on test temperatures.

3. Fatigue cracks initiate in the specimen interior independently of test temperatures and materials. At 4 and 77 K, facet-like structures are formed at the sub-surface crack initiation sites. Sizes of facets and crack initiation sites are smaller in the rolled material with 30 \( \mu \text{m} \) grains than in the forged material with 80 \( \mu \text{m} \) grains. On the other hand, at 293 K, there are no facets that can be clearly identified as crack initiation sites.

4. Grain refinement leads to reduce the size of crack initiation site, and this contributes effectively to improve the fatigue strength in high-cycle region at cryogenic temperatures.
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