Subsurface Fracture in High-Cycle Fatigue at Cryogenic Temperatures in Ti-5%Al-2.5%Sn Extra Low Interstitial Alloy*¹

Yoshinori Ono*², Tetsumi Yuri, Hideshi Sumiyoshi, Saburo Matsuoka and Toshio Ogata

Materials Information Technology Station, National Institute for Materials Science (MITS/NIMS), Tsukuba 305-0047, Japan

High-cycle fatigue properties were investigated for Ti-5%Al-2.5%Sn extra low interstitial alloy with a mean α grain size of 80 μm , which had been used for liquid hydrogen turbo-pump of Japanese-built launch vehicle. The fatigue strengths at cryogenic temperatures of 4 K and 77 K do not increase in proportion to increments of the ultimate tensile strength, and come to be lower than that at 293 K around 10^7 cycles. Observations by optical microscopy and scanning electron microscopy reveal that fatigue cracks initiate in the specimen interior and grow transgranularly, not depending on the test temperature. At the subsurface crack initiation sites, several facet-like structures are formed at cryogenic temperatures, while there are no facet-like structures at 293 K. Since localized deformation occurs at cryogenic temperatures, the subsurface cracks form facet-like structures and are supposed to initiate in the early stage of the fatigue life. As the result, the fatigue strength deteriorates at cryogenic temperatures.

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1. Introduction

In recent years, the mechanical properties of structural materials used for Japanese-built launch vehicles have been studied with the aim of increasing their reliability.¹⁾ As a part of this research program, NIMS SPACE USE MATERIALS STRENGTH DATA SHEETS (Nos. 0–2) were published in February 2003. Ti-5%Al-2.5%Sn extra low interstitial (ELI) alloy is one of the object materials and is used for liquid hydrogen turbo-pumps. The fatigue properties in this alloy need to be investigated in detail,¹⁾ since a fatigue fracture was found in a part of the liquid hydrogen turbo-pump and it was considered to have caused the failure of H-II launch vehicle No. 8.²⁾

In this study, which is related to the DATA SHEET No. 1, the high-cycle fatigue properties in Ti-5%Al-2.5%Sn ELI alloy were investigated at 4 K, 77 K and 293 K, and discussed in terms of how they correlate with the subsurface crack initiation.

2. Experimental Procedure

The present material was cast into a $\phi 480$ mm cylindrical mold, hot-forged in the β region (1473 K heating) and then in the $\alpha + \beta$ region (1243 K heating). After these processes, the forged material was annealed at 1073 K for 7.2 ks, followed by air-cooling. It was then machined to $\phi 180$ mm $\times$ 113 mm to provide the sample billets. The chemical composition of the present material is shown in Table 1. The microstructure

of this alloy was observed with an optical microscope. Tensile tests and fatigue tests were carried out at 4 K, 77 K, and 293 K. Tensile tests were carried out using cylindrical specimens 6.25 mm in diameter and 35 mm in gauge length and at an initial strain rate of $2.4 \times 10^{-4} \text{ s}^{-1}$. The fatigue test specimens had an hourglass-type with a minimum diameter of 6 mm. Fatigue tests were performed using sinusoidal waveform loading and uniaxial tension-tension loading under stress ratio (R) of 0.01. Test frequencies of 4 Hz at 4 K and 10 Hz at 77 K 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).

3. Results and Discussion

3.1 Microstructure and tensile properties

Figure 1 is an optical micrograph of the present material. The microstructure consists of α and retained β phases. The mean size of α grains is about 80 μm . Retained β phase is present along grain boundaries or within the α grains.

Fig. 1 Optical micrograph of Ti-5%Al-2.5%Sn ELI alloy used: Annealed at 1073 K for 7.2 ks, followed by air-cooling.

Table 1 Chemical composition of Ti-5%Al-2.5%Sn ELI alloy used (mass%).

Al	Sn	Fe	O	H	N	Ti
4.82	2.40	0.210	0.060	0.008	0.004	bal.

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*²Researcher, Cryogenic Materials Group.

Table 2 Tensile properties in Ti-5%Al-2.5%Sn ELI alloy used.

Strength (MPa)	4 K	77 K	293 K
0.2% proof stress	1262	1139	693
Ultimate tensile strength	1390	1206	740

The 0.2% proof stress and the ultimate tensile strength of the present material are summarized in Table 2. These properties increased with decreasing temperature and both properties at 4 K and 77 K are about 1.8 times and 1.6 times higher than those at 293 K, respectively.

3.2 Fatigue properties

Figure 2 shows S-N diagrams for the present material. In low-cycle region, the fatigue strength is higher at cryogenic temperatures than at 293 K, similarly to the ultimate tensile strength. In longer-life region, however, the fatigue strengths at 4 K and 77 K come to be lower than that at 293 K.

Figure 3 shows the relationship between ratio of the stress amplitude to the ultimate tensile strength (σ_a/σ_u) and number of cycles to failure (N_f) at 4 K, 77 K and 293 K. The σ_a/σ_u vs.

Fig. 2 S-N diagrams at 4 K, 77 K and 293 K for Ti-5%Al-2.5%Sn ELI alloy used.

Fig. 3 Relationship between ratio of stress amplitude to ultimate tensile strength (σ_a/σ_u) and number of cycles to failure (N_f).

N_f curve is expressed as a unique curve for the materials in which the fatigue crack initiates at the specimen surface.³⁾ For the present material, the σ_a/σ_u vs. N_f curves at 4 K and 77 K are markedly lower than that at 293 K. This result means that the fatigue strength of the present material deteriorates at cryogenic temperature.

3.3 Fractography

Figures 4 and 5 are typical SEM micrographs showing the crack initiation sites of the specimens fatigue-tested at 77 K

Fig. 4 SEM images showing the fracture surface (a) and the vicinity of the subsurface crack initiation site (b) in the specimen fatigue-tested at 77 K; $\sigma_a = 400$ MPa, $N_f = 43960$.

Fig. 5 SEM micrograph showing the vicinity of the subsurface crack initiation site in the specimen fatigue-tested at 293 K; $\sigma_a = 275$ MPa, $N_f = 7186900$.

and 293 K, respectively. The aspects of the crack initiation sites of the specimens failed at 4 K and 77 K are almost same. At 4 K and 77 K, fatigue cracks initiate internally (Fig. 4) and form several facet-like structures (hereafter, we abbreviate each facet-like structure to just 'facet') at the fatigue crack initiation site, not depending on stress amplitude level. As regards the subsurface crack initiation in Ti-5%Al-2.5%Sn ELI alloy, Nagai *et al.*⁴⁾ also reported that facets were formed at the fatigue crack initiation site of the specimen fatigue-tested at 4 K. In the specimens tested at 293 K, the fatigue cracks are also initiated internally (Fig. 5). However, unlike those observed at cryogenic temperatures, there is no facet that can be clearly identified at the crack initiation site.

Figures 6 and 7 represent the microstructure under the subsurface crack initiation sites of the specimens fatigue-tested at 77 K and 293 K, respectively. These figures correspond to Figs. 4 and 5. Figure 6 reveals that, in the specimens tested at 77 K, ① facets (*i.e.* subsurface cracks) are inclined with respect to the stress axis, ② subsurface cracks are formed except for the main crack and retained in the specimen interior, ③ facets (*i.e.* subsurface cracks along the main crack) and retained subsurface cracks are formed through transgranular crack initiation and growth. Moreover, another observations of the microstructure shows that the number of the retained subsurface cracks tends to decrease at lower stress amplitude. On the other hand, in the specimens

Fig. 6 Optical micrographs showing the microstructure under the subsurface crack initiation site in the specimen fatigue-tested at 77 K; $\sigma_a = 400$ MPa, $N_f = 43960$. Photograph (b) is the magnified image of the square area in photograph (a).

Fig. 7 Optical micrograph showing the microstructure under the subsurface crack initiation site in the specimen fatigue-tested at 293 K ($\sigma_a = 275$ MPa, $N_f = 7186900$).

tested at 293 K (Fig. 7), the subsurface cracks are also found to grow transgranularly. However, the fracture surface and retained subsurface cracks appear more ductile than those of the specimen tested at 77 K.

On the materials in which the fatigue crack initiate at the specimen surface, the extrusion-intrusion mechanism⁵⁾ explains the fatigue crack initiation behavior. In this mechanism, the fatigue crack initiates within persistent slip bands, which are formed through cyclic deformation. Similar mechanism can be introduced to the subsurface crack initiation in the present material, since slip bands are actually observed parallel to main crack or ahead of retained subsurface cracks in the specimen tested at 77 K (Figs. 6(a) and (b)). Umezawa *et al.*⁶⁾ reported that coplanar dislocation arrays were distributed in α grains in Ti-5%Al-2.5%Sn ELI alloy fatigue-tested at cryogenic temperatures. Since this localized deformation introduces stress or strain concentration in the vicinity of grain boundaries or $\alpha - \beta$ interfaces, subsurface cracks would initiate easily at cryogenic temperature. In fact, subsurface cracks look like initiating in the vicinity of grain boundaries or $\alpha - \beta$ interfaces in Fig. 6. Therefore, at cryogenic temperature, the subsurface crack is supposed to initiate in the early stage of the fatigue life even at lower stress level, and then they act as defects and lead to shorten the fatigue life (Fig. 3). Moreover, this mechanism on subsurface crack initiation also explains that the number of the retained subsurface cracks tend to decrease at lower stress amplitude and the subsurface cracks form facet-like structures.

Since it is easier to activate plural slip systems at 293 K than at cryogenic temperature,⁷⁾ the stress or strain concentration can be easily relieved at 293 K and the fracture surface appears more ductile than those of the specimen tested at 77 K (Fig. 5).

4. Conclusions

Fatigue properties and fracture surfaces of the specimens were investigated in Ti-5%Al-2.5%Sn ELI alloy, which is used for Japanese-built launch vehicles. The results obtained are as follows:

- (1) Fatigue strength does not increase in proportion to increments of the ultimate tensile strength at cryogenic temperatures. At around 10^7 cycles, the fatigue strength is lower at cryogenic temperatures than at 293 K. At cryogenic temperature, it is supposed that the subsurface crack initiates in the early stage of the fatigue life even at lower stress level because of the localized deformation. These subsurface cracks would act as defects and lead to shorten the fatigue life.
- (2) Fatigue cracks initiate in the specimen interior and are formed transgranularly, not depending on test temperature. At 4 K and 77 K, several facets are formed at the subsurface crack initiation site, while there are no facets that can be clearly identified as crack initiation sites at 293 K.

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