HIGH CYCLE FATIGUE PROPERTIES OF Ti-6AL-4V ALLOYS

AT CRYOGENIC TEMPERATURES

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Abstract

Cryogenic systems require highly efficient structural materials. Titanium alloys are
blessed with many advantages like low permeability and low thermal conductivity as well as
high strength and low specific gravity. However, very few cryogenic fatigue data are available
probably because the test is extremely difficult. The present paper describes the high cycle
fatigue property of Ti-6Al-4V alloys at 293 K, 77 K and 4 K.

The Ti-6Al-4V alloys with 0.054, 0.104, and 0.135 mass% oxygen were tested.
Processing temperatures were in α+β region and finally mill-annealing was done. The
fatigue strength increased as the test temperature decreased, which can be accounted for by
increased strength at lower temperature. The rolled materials had higher fatigue strength than
the forged materials over the temperatures range tested and at given strength level,
independently of the oxygen content. This is believed to be due to the difference in the
morphology of primary α grains, since the mean grain size itself was almost the same but the
forged material had a "colony" structure in contrast with an equi-axed structure of the rolled
one.

Introduction

Recent R&D projects based on superconductivity and cryogenics have a wide range
of engineering applications like magnetic levitation car, electromagnetic thruster (ship), and
superconducting generator etc. Cryogenic structural materials should have a high fracture
toughness as well as a high yield strength [1]. In addition, a good fatigue strength is needed
[2], since the machines experience stop-run load cycles and they often have "moving"
components. And further some other properties are potentially demanded for better heat
insulation and more sound operation under high magnetic field (static or alternate).

Titanium (Ti) alloy has many advantages for the cryogenic applications. Its low
specific strength, strength-to-gravity ratio, and high yield strength are very favorable for high
efficiency of the "moving" machines. And further, the alloy is more blessed with the low
thermal conductivity, the extremely low magnetic permeability and the high electric resistivity
[3], compared with austenitic stainless steels.

Some of the present authors previously reported the tensile properties, fracture
toughness, and high cycle fatigue properties of a Ti-5Al-2.5Sn ELI (Extra-Low-Interstitials)
alloy [4]. The reduction of oxygen content yielded the high fracture toughness at 4 K.
Accordingly the Ti-5Al-2.5Sn ELI alloy showed an excellent combination of yield strength and fracture toughness at 4 K and an increased fatigue strength at lower temperature.

Ti-6Al-4V alloy is one of the most popular Ti alloys. The principal interest of the present authors is the probability of cryogenic use of this alloy. The previous paper [5] pointed out that the low temperature fracture toughness of the Ti-6Al-4V alloy was also highly dependent on the oxygen content. Reduction of oxygen suppressed the drop of low temperature fracture toughness. In the lowest oxygen alloy, therefore, the fracture toughness was in the same level at all the temperatures. The present paper reports the high cycle fatigue properties at low temperatures of the Ti-6Al-4V alloys.

Experimental Procedure

Test materials

Three Ti-6Al-4V alloys with different impurity levels were melted; namely a normal-grade one (Normal), an extra-low-interstitial grade one (ELI), and an extremely-low-interstitial grade one (Special ELI, abbreviated as SpELI here). They had different oxygen contents and the nominal oxygen content was 0.15, 0.10 and 0.05 mass%. In the SpELI alloy, no iron was added expecting a better toughness [6], although 0.2% iron was conventionally added in other two alloys. The chemical compositions are listed in Table I.

Ingots were forged finally in α + β region, and then a part of them was further rolled also in α + β region. The ingot with or without rolling is called "forged material" or "rolled material", respectively. All the materials were finally heat-treated for 7.2 ks at 973 K and air-cooled. Some details of the processing history are shown in Table II.

Table I Chemical compositions of Ti-6Al-4V alloys tested in the present study in mass%

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Al</th>
<th>V</th>
<th>Fe</th>
<th>O</th>
<th>N</th>
<th>H</th>
<th>C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Normal</td>
<td>6.34</td>
<td>4.23</td>
<td>0.199</td>
<td>0.135</td>
<td>0.0071</td>
<td>0.0053</td>
<td>0.011</td>
</tr>
<tr>
<td>ELI</td>
<td>6.23</td>
<td>4.25</td>
<td>0.200</td>
<td>0.104</td>
<td>0.0035</td>
<td>0.0032</td>
<td>0.011</td>
</tr>
<tr>
<td>SpELI</td>
<td>5.97</td>
<td>4.12</td>
<td>0.028</td>
<td>0.054</td>
<td>0.0019</td>
<td>0.0055</td>
<td>0.024</td>
</tr>
</tbody>
</table>

Table II Process of forging, rolling, and heat treatment

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Forging</th>
<th>Rolling</th>
<th>Heat Treatment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Normal</td>
<td>α + β (75 x 85)→α + β (70 x 70)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>ELI</td>
<td>α + β (115 x 120)→α + β (70 x 70)</td>
<td>α + β (28φ)</td>
<td>973 K, 7.2 ks</td>
</tr>
<tr>
<td>SpELI</td>
<td>β (170φ)→α + β (70 x 70)</td>
<td></td>
<td>Air Cooled</td>
</tr>
</tbody>
</table>

# Forging or rolling temperatures are indicated as "α + β" which accounts for a temperature in the α + β region. The final section size in one heat series is shown in parenthesis (mm).
Figure 1 - SEM photographs of microstructure. The print planes are on the transverse section of the fatigue test pieces.

**Microstructure**

A finer and more homogeneous microstructure was expected for the rolled material because of the heavier reduction in forming. Figure 1 represents the SEM photographs of microstructure. The forged materials have lamellar microstructure principally composed of elongated or plate-like α and β (or transformed β) platelet. The formation of "colony", namely the region in which α plates are aligned, is seen especially in ELI and SpELI alloys. The mean width of a grain was 5.0, 4.0, and 1.9 µm in Normal, ELI, and SpELI, respectively. In the rolled materials, the α grain becomes globular. The mean diameter of α grain was 4.0, 4.0, and 2.8 µm in Normal, ELI, and SpELI, respectively.

Rolling did not always make α grains finer. The Normal and the ELI alloys had almost the same α grain size. The SpELI alloy had the finest microstructure. However, the reason is not made clear.

**High cycle fatigue test**

Hourglass type unnotched test pieces with a waist diameter of 4.5 mm were machined in the L-direction for both the forged and the rolled materials. S-N curves at 293, 77, and 4 K were determined using the cryogenic fatigue test machine [7]. In obtaining the S-N curves, the estimation of a million cycles fatigue strength (MFS) was intended. The test machine was servo-hydraulic and its dynamic capacity was ±50 kN. Load control test was done in a sinusoidal wave with a minimum-to-maximum load ratio, R=0.01 at 4 Hz at 4 K and at 10 - 20 Hz at 77 and 293 K.
Results and Discussion

High cycle fatigue property

All the S-N curves obtained in the present study are described in Figures 2 to 4. In these materials, two kinds of fatigue crack initiation site were observed. One was at the specimen surface, and the other was in the specimen interior. SEM photographs of the latter cases are presented in Figure 5. In Figs. 2 to 4, therefore, the crack initiation sites are classified into "Surface" or "Internal".
Figure 5  SEM micrographs of subsurface crack initiation sites and sub-cracks for Ti-6Al-4V forged materials: (a) Normal, 4K, 1,235MPa, (b) ELI, 4K, 944MPa, (c) ELI, 77K, 601MPa, (d) Normal, 4K, 1,235MPa, (e) Normal, 4K, 1,058MPa, and (f) Normal, 4K, 1,147MPa.

Figure 6  Comparison of a million-cycle-fatigue-strength between forged materials and rolled materials as a function of (a) test temperature and (b) yield strength.

Effect of test temperature and strength level

Temperature decrease produced an increase in strength, and it is generally said that the fatigue strength is proportional to the tensile strength. Hence a simple analogy leads to a speculation that the fatigue strength is increased at lower temperature. It is obviously true for the rolled materials irrespective of the oxygen level; the S-N curves shift to higher stress level at lower temperature and three temperature curves are nearly parallel. In the forged materials, on the other hand, there is almost no gap between the S-N curves at 77 K and 4 K. In the
Normal alloy, the gap becomes narrower as the number of cycles to failure increases and the three temperature curves are supposed to overlap at around 5 million cycles.

In Figure 6 (a), one million cycles fatigue strength (MFS) is plotted as a function of test temperature. As far as the MFS is concerned, the MFS increases with a decrease in temperature and the rolled material is superior to the forged material at all the temperatures. Especially at 4 K, the difference in the MFS between two materials is distinctly large.

Ratio of yield strength to ultimate tensile strength was higher than 95% at all the temperatures for three alloys. Therefore, the interrelation between strength and MFS is described in terms of yield strength vs. MFS as in Figure 6 (b), since yield strength is one of the most important measures in the selection of candidate materials. Anyway almost the same plotting was done when the tensile strength was taken as the abscissa.

As seen in Figure 6 (b), the plots form two separate groups when the processing is taken as a parameter. In other words, the MFS of the rolled material is higher than that of the forged material at a given yield strength. Roughly speaking, either in the rolled material or in the forged material, the MFS is proportional to the yield strength over the temperature range investigated. And the dependence of MFS on yield strength is less in the forged material than in the rolled material. This corresponds to the above-mentioned result in Figure 6. Although more detailed comparison leads to a different conclusion that at 4 K the SpELI alloy, having the lowest yield strength, showed a higher MFS than other two alloys, this is considered to be no major concern here except the fact that the SpELI alloy had the maximum MFS at 4 K.

Microstructural factors and fatigue strength

Texture. A. Sommer et al. said that in textured Ti-6Al-4V alloy, the alternating stress parallel to c-axis introduced longer fatigue lives than that perpendicular to c-axis [8]. Figure 7 shows the simplified X-rays analysis of texture, namely X-rays intensities of three major peaks from prismatic plane, (1010)α basal plane, (0002)α, and pyramidal plane, (1011)α are shown in relative ratios to normalized total intensity,

\[ I_\alpha = \frac{1}{3} (I_{(1010)\alpha} + I_{(0002)\alpha} + \frac{1}{4} I_{(1011)\alpha}) \]

This equation is based on an assumption that the ratio of the intensities is equal to 1 : 1 : 4 where the calculated ratio is 23.2 : 25.0 : 100 for randomly-oriented-polycrystalline pure-Ti (Cu-Kα). Therefore the maximum of relative intensity, I/ I_\alpha, is 3.0 and the larger I/ I_\alpha shows the more preferred orientation. From Figure 7, it is concluded that all the materials have the similarly textured microstructure in which the prismatic plane is parallel to the principal stress, that is, the c-axis is perpendicular to the principal stress. The texture is further accentuated in the rolled materials. Hence the difference in fatigue strength between the forged and the rolled materials can not be explained in terms of texture.

![Figure 7 - X-rays relative intensities of major peaks of α phase. (L: Plane parallel to principal stress, T: Plane perpendicular to principal stress. Solid symbols: Forged materials, and Open: Rolled materials.)](image-url)
Internal Initiation Site. Internal initiation prevailed more at lower cyclic stress as seen in Figs. 2 to 4. The site was composed of facet-like unit(s) and inclined by several ten degrees to the principal stress axis. The size became larger at lower stress as seen in Figure 5 (single, plural, and aggregate). Figure 8 shows the size varies between several µm and several ten µm. Here the size was defined as the minor axis of an orthographic projection of the initiation site on the main crack propagating plane, fs. The presence of subcracks near the main crack and its coalescence under lower stress, shown in Figure 5 (d)-(f), demonstrate that the first stage of fatigue is the subcracking of microstructure and the subcracks grow until a main crack forms originating at one of the subcracks. Figure 9 rearranges the data in Figure 8, when a critical condition like $\Delta K_1 = k \Delta \sigma / fs$ is assumed where $\Delta \sigma$ is cyclic stress range. The value of $\Delta \sigma / fs$ is in a narrow range around the mean value of 2.98 MPa/µm. Strictly speaking, the coefficient k does not always take a constant value. The value depends on the shape and location of the initiation site. When the shape is taken into account, the smaller fs tends to have a higher k value due to its lower aspect ratio. However, the coefficient k can be roughly estimated to be around 2. Then $\Delta K_1$ becomes about 6 MPa/µm. For the Ti-6Al-4V alloy, $\Delta K_{th}$ is slightly lower than 10MPa/µm at 4K [9] and $K_{IC}$ is greater than 30 MPa/µm at 4K [10]. Hence it is considered that a main crack starts to grow when the initiation site grows up to a critical size determined for a given cyclic stress by a critical condition.

The α grain is a subcracking microstructure. As shown in Figure 5, subcracks were produced in α grains. Table III shows a comparison of point analysis data for α, β phases and facets in the initiation site. The chemical compositions of the facet are very similar to those of the α phase. In addition, the size and shape corresponded to those of the α grains. Namely, in the forged materials the sites were composed of elongated facet(s) and, on the other hand, in

### Table III  EDS analyses of α, β and internal initiation facet in the Normal alloy.

<table>
<thead>
<tr>
<th>Region</th>
<th>Concentration (mass%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Al</td>
</tr>
<tr>
<td>α phase</td>
<td>5.4 - 6.9</td>
</tr>
<tr>
<td>β phase</td>
<td>4.1 - 5.9</td>
</tr>
<tr>
<td>initiation facet</td>
<td>4.8 - 7.3</td>
</tr>
</tbody>
</table>

Figure 8 - Relationship between internal crack size and peak cyclic stress at 4K for Ti-6Al-4V alloys.

Figure 9 - Relationship between cyclic stress range $\Delta \sigma$ and $\Delta \sigma / fs$ for Ti-6Al-4V alloys. 'fs' is the size of the internal crack site.
the rolled materials the facets were rather globular. Hence, the facet in the initiation site is attributed to an α grain.

Some reported that finer primary α grain size produced higher fatigue strength [11]. In the forged material, the primary α grains in a colony are believed to be crystallographically aligned and act as a single path for dislocation moving [12]. In that case, the mean slip length becomes several times of primary α grain width in the forged material. In the rolled material, on the other hand, the mean slip length is considered to be of an order of single primary α grain size. Accordingly, the forged material has a longer slip length than the rolled material, which may introduce higher stress localization and easier crack initiation at the same applied stress and this leads to lower fatigue strength.

From these consideration, it is concluded that the significant difference in the fatigue strength was caused by the morphological change in their microstructure. In other words, the globular α grain microstructure produces a higher fatigue strength than the microstructure in which plate-like or elongated α grains of the same size form "colonies".

**Summary**

The cryogenic high cycle fatigue properties were investigated for Ti-6Al-4V alloys with varied oxygen level; nominally 0.05, 0.10, and 0.15 mass %. The alloys were prepared both in the as-forged material and in the rolled material.

Mean primary α grain size of the lowest oxygen alloy was smallest. Although rolling did not always produce the finer α grain size, the process changed the morphology of α grains. Namely the globular grains were obtained in the rolled material in place of plate-like α grains forming "colony" in the forged one.

The fatigue strength increased as the test temperature decreased. This can be accounted for by increased strength at lower temperature. The fatigue strength of the rolled material was superior to that of the forged one especially at 4 K. At a given strength level, the former was higher than the latter over the temperature range investigated. This is believed to be ascribed to the difference in a grain morphology.

**References**