The Microstructure and Creep Behavior of a Boron-Modified Ti-15Al-33Nb (at%) Alloy

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Abstract

The affect of boron (B) on the microstructure and creep behavior of a Ti-15Al-33Nb (at%) alloy was investigated. In addition to the normal constituent phases present in the monolithic alloy, the B-modified alloy contained borides enriched in titanium and niobium. These borides were present in the form of needles/laths up to 50 µm long and 10 µm wide which took up 5-9% of the volume. Constant load, tensile-creep experiments were performed in the stress range of 150-340 MPa and the temperature range of 650-710°C, in both air and vacuum environments. An addition of 0.5 at% B did not improve the creep resistance of the monolithic alloy, while the addition of 5 at% B significantly improved the creep resistance.

Introduction

The addition of trace amounts of B to conventional titanium (Ti) alloys, such as Ti-6Al-4V (wt%), has been shown to decrease the as-cast grain size by approximately an order of magnitude [1]. This drastic reduction in the as-cast grain size leads to significant benefits including increasing yield strength while reducing or avoiding time spent on expensive and energy intensive thermomechanical processing. The addition of B also leads to the stabilization of titanium boride (TiB) whiskers within the conventional $\alpha + \beta$ microstructure [1,2]. The presence of this TiB phase within these microstructures has lead to substantial increases in room temperature (RT) strength and Young’s modulus, while maintaining adequate elongation-to-failure values [2]. Ti-Al-Nb alloys differ from conventional titanium alloys in that their constituent phases may include the ordered intermetallic orthorhombic (O) phase based on Ti$_2$AlNb, the ordered HCP intermetallic $\alpha_2$ phase based on Ti$_3$Al, and the BCC phase whose ordering is dependent upon composition [3]. Such microstructures have shown potential to exceed the elevated-temperature capabilities of conventional $\alpha + \beta$ Ti-alloys. In particular a Ti-15Al-33Nb (at%) alloy has been shown to exhibit an attractive balance of elevated-temperature tensile and creep strength while maintaining RT strength and elongations comparable to Ti-6Al-4V (wt%) [4]. However, relatively few studies have focused on investigating the effects of B additions on the microstructure and mechanical properties of Ti-Al-Nb alloys [5-9]. The purpose of this work was to determine the affect of varying B additions on the microstructural features and the impact this causes on the creep deformation behavior of a Ti-15Al-33Nb (at%) alloy [henceforth all compositions will be given in atomic percent unless otherwise noted]. It is noted that no previous work has been performed with regard to B modification of this alloy.

Experimental

Conventional ingot metallurgy and thermomechanical processing routes were employed to produce both monolithic Ti-15Al-33Nb and Ti-15Al-33Nb-0.5B rolled sheet materials. The monolithic material was cast into a 140 mm diameter ingot using double vacuum-arc-remelting
while the Ti-15Al-33Nb-0.5B ingot was cast into an approximately 250 gram button-shaped melt. All the casting and subsequent forging and rolling operations were performed at RTI International (Niles, Ohio). The monolithic alloy was hot forged and rolled at 899°C and the Ti-15Al-33Nb-0.5B alloy was hot forged and rolled at 975°C. The BCC-transus temperature for this alloy is 980°C [4], thus all thermomechanical processing steps were performed below the BCC transus which enabled the resulting fine-grained microstructure.

The Ti-15Al-33Nb-5B material was induction-skull melted at Flowserve (Dayton, Ohio). The elements were mixed in the correct stoichiometric amounts and then melted and cast into an ingot of 70 mm diameter and 500 mm length. The ingot was then hot isostatically pressed (HIP) for four hours at a pressure of 172 MPa and temperature of 900°C. No subsequent thermomechanical processing was performed on this material.

Chemical Analysis was performed using Inductively Coupled Plasma Optical Emission Spectroscopy and Inert Gas Fluorescence. In order to produce thermally stable microstructures each of the processed materials was subjected to an identical heat treatment (HT), which could be described as 1005°C/3h/FC/855°C/8h/CC/650°C/FC, where FC indicates furnace cooling at 10°C/minute and CC indicates control cooling at 1°C/minute. Samples for microstructural evaluation were sectioned from the processed alloys and metallographically polished through a final finish of colloidal silica, which contained an average particle size of 60 nm. The average chemical composition of the boride needles in the Ti-15Al-33Nb-5B alloy was determined using a JEOL JXA-8200 Electron Microprobe (EMP), where over 20 needles were characterized. Phase volume fractions were determined from the Backscattered Electron (BSE) SEM images using ImageJ image analysis software. Grain size was determined using the mean line intercept method [10].

Dogbone shaped samples were cut from the processed materials using an Electrodischarge Machine (EDM). The EDM affected surface layers were removed through mechanical polishing prior to creep testing. ATS lever arm creep frames were used for the constant load tensile creep tests. ATS single zone furnaces were used, and temperatures were maintained within ±3°C of the targeted test temperature. Creep strain was monitored during the tests using a linear variable differential transformer that was connected to a 25.4 mm gage length ATS high-temperature extensometer. The extensometer was attached directly to the gage length of each sample. A servohydraulic thermal-mechanical testing machine was also used in order to perform tensile creep experiments in vacuum (10^{-7} torr) in order to determine the effect of environment on creep deformation. For such experiments, a high temperature quartz-armed extensometer was attached directly to the sample’s gage section. The samples were allowed to soak at the creep temperature for one hour prior to applying load. After the minimum creep rate had been achieved, either the load or temperature was increased, or the creep test was discontinued. The applied stresses ranged from 150-340 MPa and the temperatures ranged from 650-710°C.

Results

Microstructure

The chemical compositions of each the alloys are presented in Table 1 and the measured phase volume fractions and grain sizes are presented in Table 2. It is noted the targeted compositions were maintained adequately well in all the alloys, and that the oxygen content was kept extremely low in the Ti-15Al-33Nb-5B material. The average composition of the borides in the Ti-15Al-33Nb-5B material was 46.8B-29.0Nb-23.0Ti-1.2Al. Photomicrographs of the as-processed (AP) and heat-treated (HT) microstructures are presented in Figure 1.
Figure 1. BSE SEM images of Ti-15Al-33Nb-xB materials: (a) Ti-15Al-33Nb AP, (b) Ti-15Al-33Nb HT, (c) Ti-15Al-33Nb-0.5B AP, (d) Ti-15Al-33Nb-0.5B HT, (e) Ti-15Al-33Nb-5B AP, and (f) Ti-15Al-33Nb-5B HT. The $\alpha_2$ phase is the darkest phase, the O-phase is the gray lath phase, the $\beta$ phase is the light matrix phase, and the borides in (e) and (f) are the bright needles.

Table 1. Measured Chemical Composition of Ti-15Al-33Nb-xB Alloys

<table>
<thead>
<tr>
<th>Nominal Composition [at.%]</th>
<th>Ti [at.%]</th>
<th>Al [at.%]</th>
<th>Nb [at.%]</th>
<th>B [at.%]</th>
<th>O [ppm]</th>
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</thead>
<tbody>
<tr>
<td>Ti-15Al-33Nb</td>
<td>51.4</td>
<td>15.3</td>
<td>33.3</td>
<td>0</td>
<td>1100</td>
</tr>
<tr>
<td>Ti-15Al-33Nb-0.5B</td>
<td>52.4</td>
<td>14.3</td>
<td>32.9</td>
<td>0.4</td>
<td>1450</td>
</tr>
<tr>
<td>Ti-15Al-33Nb-5B</td>
<td>45.3</td>
<td>15.9</td>
<td>33.4</td>
<td>5.3</td>
<td>130</td>
</tr>
</tbody>
</table>
Table 2. Measured Phase Volume Fractions and Grain Size

<table>
<thead>
<tr>
<th>Composition</th>
<th>Condition</th>
<th>(\alpha_2) Vp</th>
<th>O Vp</th>
<th>Bcc Vp</th>
<th>Boride Vp</th>
<th>Grain Size* [(\mu)m]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti-15Al-33Nb</td>
<td>AP</td>
<td>10</td>
<td>0</td>
<td>90</td>
<td>0</td>
<td>3</td>
</tr>
<tr>
<td>Ti-15Al-33Nb</td>
<td>HT</td>
<td>1</td>
<td>44</td>
<td>55</td>
<td>0</td>
<td>120 +/- 32</td>
</tr>
<tr>
<td>Ti-15Al-33Nb-0.5B</td>
<td>AP</td>
<td>22</td>
<td>0</td>
<td>78</td>
<td>0</td>
<td>11 +/- 4</td>
</tr>
<tr>
<td>Ti-15Al-33Nb-0.5B</td>
<td>HT</td>
<td>0</td>
<td>69</td>
<td>31</td>
<td>0</td>
<td>130 +/- 29</td>
</tr>
<tr>
<td>Ti-15Al-33Nb-5B</td>
<td>AP</td>
<td>1</td>
<td>69</td>
<td>21</td>
<td>9</td>
<td>48 +/- 12</td>
</tr>
<tr>
<td>Ti-15Al-33Nb-5B</td>
<td>HT</td>
<td>4</td>
<td>65</td>
<td>26</td>
<td>5</td>
<td>63 +/- 13</td>
</tr>
</tbody>
</table>

* Grain Size represents the prior \(\beta\) grain size except for the Ti-15Al-33Nb AP material where both the O and \(\beta\) grain sizes were averaged.

**Creep**

The minimum creep rates were determined by applying a linear least squares regression curve fit to the raw strain versus time data obtained during the creep experiments. Figure 2 (a) and (b) display typical load jump and temperature jump experimental data. Creep stress exponents (n) and apparent activation energies (Qapp) were calculated from the measured minimum creep rates in order to suggest the dominant creep deformation mechanism in the steady-state creep regime for each composition. The stress dependence of the minimum creep rate for each composition is displayed in Figure 3 (a) and the creep strain versus time behavior at 650°C is given in Figure 3 (b). Comparing the minimum creep rates for identical applied stresses and temperatures, the Ti-15Al-33Nb-5B alloy exhibited the lowest creep rates while the Ti-15Al-33Nb-0.5B alloy exhibited the greatest creep rates. For example, at 172 MPa/650°C the minimum creep rates were 7.6x10^{-9} s^{-1}, 1.6x10^{-8} s^{-1}, and 2.3x10^{-9} s^{-1} for the monolithic, 0.5B, and 5B alloys, respectively. Thus, the 5B alloy exhibited an order of magnitude lower minimum creep rate than the 0.5B alloy. Table 3 lists the calculated n and Qapp values for the stress and temperature ranges examined.

![Figure 2](image-url)  
(a) Creep data obtained directly from experiment for two separate Ti-15Al-33Nb-0.5B samples: (a) data obtained from a load jump experiment at \(T = 650°C\) and (b) data obtained from a temperature jump experiment at \(\sigma = 250\) MPa.
Figure 3. (a) Minimum creep rate versus stress at $T = 650^\circ\text{C}$ and (b) creep strain versus time for test conditions of $\sigma = 250-275 \text{ MPa}/650^\circ\text{C}$ in air.

Table 3. Measured Creep Stress Exponents and Activation Energies for the Heat-Treated Materials

<table>
<thead>
<tr>
<th>Composition</th>
<th>$\sigma/T \ [\text{MPa}/^\circ\text{C}]$</th>
<th>$n$</th>
<th>$\sigma/T \ [\text{MPa}/^\circ\text{C}]$</th>
<th>$Q_{\text{app}} \ [\text{kJ/mol}]$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti-15Al-33Nb</td>
<td>148-226/650</td>
<td>2.5</td>
<td>150/650-710</td>
<td>163</td>
</tr>
<tr>
<td></td>
<td>226-299/650</td>
<td>8.7</td>
<td>273/650-690</td>
<td>288</td>
</tr>
<tr>
<td>Ti-15Al-33Nb-0.5B</td>
<td>125-200/650</td>
<td>2.2</td>
<td>150/650-710</td>
<td>236</td>
</tr>
<tr>
<td></td>
<td>200-250/650</td>
<td>5.5</td>
<td>250/650-690</td>
<td>320</td>
</tr>
<tr>
<td>Ti-15Al-33Nb-5B</td>
<td>172-340/650</td>
<td>3.2</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Discussion

Microstructure

The addition of 0.5 B to the Ti-15Al-33Nb alloy did not produce a significantly different grain size, and no boride needles were visible within the microstructure in either the AP or HT condition. This suggests that if a eutectic composition exists for this alloy system, as found for the TiB system [1,2], 0.5 B is below the eutectic composition for this alloy and 0.5 B is soluble within one or more phases in the microstructure. After the heat treatment, both the monolithic and 0.5 B microstructures contained prior $\beta$ grain sizes between 120-130 $\mu$m, suggesting that 0.5 B did not significantly affect the BCC-transus temperature. The increased volume fraction of the $\alpha_2$ phase within the AP microstructure is an effect of processing at 975$^\circ$C, and further confirms that a two phase $\alpha_2 + \beta$ phase field exists below the $\beta$ phase field as was suggested in previous work [3,4].

The addition of 5 B significantly altered the microstructure. Boride needles were evident throughout the AP and HT microstructures ranging in length from 3-50$\mu$m and width up to 10 $\mu$m. The prior $\beta$ grain size in the 5B samples was significantly reduced when compared to the monolithic and Ti-15Al-33Nb-0.5B alloys, see Table 2. In fact, the Ti-15Al-33Nb-5B material exhibited a grain size half that of the monolithic and Ti-15Al-33Nb-0.5B alloys after heat treatment. This reduction in grain size may be attributed to either the B addition raising the BCC-transus temperature of the alloy, and/or the boride needles pinning grain growth. The small volume of $\alpha_2$ phase was observed to be secluded predominately to prior $\beta$ grain boundaries within these samples, which could also contribute to pinning grains.

The EMP data from the boride phase in the Ti-15Al-33Nb-5B material suggests a phase composition close to $\text{B}_2\text{TiNb}$. This is significant due to the fact that B additions to Ti-6Al-4V (wt%) alloys have produced boride whiskers with a composition of TiB or TiB$_2$ [1,2].

Creep
The addition of 0.5B increased the minimum creep rates while the addition of 5B decreased the minimum creep rates. Based on the calculated n values (see Table 3 and Figure 3 (a)), at least two dominant creep mechanisms are suggested to be active as a function of stress. Grain boundary sliding is suggested in the low stress regime and dislocation climb is suggested in the high stress regime. The Ti-15Al-33Nb-5B material displayed a constant n value of 3.2 over the entire stress range studied. It is noted that the addition of 0.5B increased the minimum creep rates over the examined stress and temperature range, but did not drastically change the transition stress between the two apparent deformation mechanisms. The onset of dislocation climb controlled creep is suggested to occur at 200 MPa in the Ti-15Al-33Nb-0.5B alloy, while it is not suggested to occur until 250 MPa in the monolithic alloy. The increased creep resistance of the Ti-15Al-33Nb-5B material may be attributed to the presence of the boride particles impeding grain boundary sliding and providing a substantial obstacle for dislocation climb. It is noted that Emura and Hagiwara have also observed improved creep resistance due to B addition in a Ti-22Al-27Nb(at.%)/6.5 mass% TiB alloy [11]. Understanding why B has such a significant effect on the microstructure and creep behavior of Ti-Al-Nb alloys will be the focus of ongoing studies.

Summary

The effect of B additions on the microstructure and creep behavior of a Ti-15Al-33Nb (at%) alloy was investigated. The addition of 0.5 at% B did not significantly alter the grain size, and this concentration of B was suggested to be soluble within one or more of the constituent phases. Boride needles approximately 3-50 µm in length with a measured average composition of 46.8B-29.0Nb-23.0Ti-1.2Al were observed in the Ti-15Al-33Nb-5B (at%) alloy in small volume fractions (5-9%). The Ti-15Al-33Nb-0.5B alloy was shown to have worse creep resistance than the monolithic alloy, while the Ti-15Al-33Nb-5B material displayed superior creep resistance over the monolithic alloy. Grain boundary sliding and dislocation climb controlled creep were the suggested deformation mechanisms based upon calculated creep stress exponents and apparent activation energies.

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References