EFFECT OF HEAT TREATMENTS WITH A POINTED COOLING ON THE MECHANICAL COMPRESSION PROPERTIES OF TiAl INTERMETALIC

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Abstract

The influence of the graded cooling heat treatments on a change in the structure and the room temperature mechanical compression properties of cast Ti-44Al-8Nb intermetallic were studied. The used heat treatments were consisting from heating to the temperature 1350 °C and following variable cooling. The coolings were the air cooling and the graded cooling which was composed of combination of furnace cooling and air cooling for selected two temperatures 1315 °C and 1200 °C. These selected temperatures were used from the measuring of the differential thermal analysis (DTA) which was used to determine the phase transformation temperatures. All heat treatments used remold the cast nearly lamellar structure \( \gamma/\alpha_2 \) to the fully lamellar uniform structure and decreased the average grain size to the half. The air cooling and the graded cooling with temperature 1315 °C were produced in higher yield stress and lower plasticity due the very thin lamellar thickness and planar grain boundary. While higher plasticity and lower yield stress are obtained using the graded cooling with temperature 1200 °C. It is caused by the very thick lamellar spacing and locked grain boundaries.

1. INTRODUCTION

Recently, applications using TiAl alloys as, e.g., gas engine turbines appeared, making profit from low density, high strength, and excellent corrosion properties at elevated temperatures. Nevertheless, the development of TiAl alloys is far from being finished. Applications are still limited by restricted machinability and low fracture toughness at room temperature. It is hoped to improve especially the fracture properties of the material at room temperature by increasing the Nb content at 8 at. %. This new class of TiAl alloys is now under testing worldwide [1].

The mechanical properties of \( \gamma \)-TiAl alloy are sensitive to its microstructure which can be changed to the heat treatment [2-6]. It has been well established that those alloys with a fine fully lamellar microstructure possess improved the ductility and toughness. Therefore, numerous studies [2-6] mainly TiAl-2Nb have been performed in heat treatments to control the microstructure through manipulating relevant phase transformations such as massive transformation, near \( \gamma \) transformation and discontinuous coarsening. To develop a fully lamellar microstructure usually requires cooling from the \( \alpha \) single phase field (see Fig. 1), but the most appropriate cooling rate for development of a microstructure with optimized mechanical

Fig. 1. Experimentally phase diagram of TiAl alloy for 8 at. % Nb, for reference, the binary TiAl – phase diagram is drawn by dotted lines [7].
properties (as creep, fatigue at higher temperatures) is unclear. It is known that reducing the lamellar interface spacing improves creep resistance [2, 3]. A simple technique to minimize the lamellar interface spacing is to cool relatively quickly from the $\alpha$ single phase region, e.g. air cooling. However, for many near $\gamma$-TiAl compositions, rapid cooling may give rise to the formation of massively transformed ($\gamma_M$), which may be deleterious to creep resistance [2]. Interlocked lamellae along lamellar grain boundaries may improve creep properties [2, 3]. Interlocked grain boundary structures result from relatively slow cooling (i.e. furnace cooling) from the $\alpha$ single phase field. The decomposition of the $\alpha$ phase during cooling of near $\gamma$-TiAl intermetallics has been widely studied [2-6].

In this paper, the microstructures of TiAl-8Nb and compression mechanical properties at room temperatures resulting from application of the pointed cooling are presented.

2. EXPERIMENTAL DETAILS

2.1 Experimental material

The second material is TiAl with 7.8 at.%Nb (7Nb alloy in the following text) in the form of a cylindrical ingot of 1070 mm in length and 70 mm in diameter was prepared by casting in the Flowserve company. The chemical composition of the alloy in at. % is: 44Al – 48Ti – 7.8Nb – 0.2Ni. The chemical composition does not vary within the ingot and the microstructure at the center differs from the ingot borders. Therefore, the cylindrical specimens were prepared only from the outer regions of the ingot.

Fig. 1a documents the grains of 0.1 to 1.2 mm in diameter. The fraction of single $\gamma$ phase islands is about 3 %, the rest of the volume contains lamellar substructure. Fine lamellar structure of $\gamma$ and $\alpha_2$ phases is shown in Fig. 1b. The $\gamma$ phase lamellas have average thickness of 0.97 $\mu$m. Using neutron diffraction, other minority phases were identified. Their volume fraction derived from the neutron diffraction analysis is: $\beta$ phase 2.8 %, $\beta_2$ phase 1.4 % and B2 phase 3.5 %. These minor phases were observed either in regions between lamellas $\gamma$ and $\alpha_2$ phases (see Fig. 1c) or in regions close to grain boundaries.
Fig. 1. Optical and electron micrographs of the cast structures TiAl alloy: (a) optical images of the specimen head, etched to reveal the disparate grain size in section perpendicular and (b) the detail of central part of a specimen heads; (c) TEM images of lamellar microstructures and (d) detail of region with minor phases in form of small spherical precipitates.

2.2 The DTA measurement

The temperatures of relevant invariant reactions (phase transformation temperatures as $T_\alpha$) were determined by differential thermal analysis (DTA) measurements of cast specimens with original structure (Fig. 1). Netzsch DSC 404 C Pegasus was used for the measurements. A sample mass of max 40 mg was put into Al$_2$O$_3$ crucible under argon atmosphere and used for the measurements with Cr as reference material. The heating rate 10 K min$^{-1}$ and 20 K min$^{-1}$ were employed for calibration and also for the measurements in the temperature range 400÷1,500 °C. The temperatures of the invariant phase reactions were usually taken from the extrapolated peak-onset on heating. The accuracy of all given temperatures is ±2 °C. Fig. 3 shows example of measured DTA curves with determined temperatures and marked structures obtained from water quenching, EBSD analyses and the experimetaly determined phase diagram (see Fig. 1 from [7]). The phase transformation temperatures as the eutectic temperature $T_{\text{e}}$=1,042 °C, the $\alpha$ transus $T_\alpha$=1,268 °C and the $\beta$ transus $T_\beta$=1,365 °C were determined.
2.3 Heat treatments

The samples (Fig. 1a) with diameter 12 mm and height 8 mm were inserted to preheated furnace at 1 350 °C / 25÷35 minutes and then different cool down (Fig. 2). Three heat treatments with different cool down were done: (i) air cooling; (ii) pointed cool consisting from furnace cooling to 1 315 °C and air cooling; (iii) pointed cool consisting from furnace cooling to 1 200 °C and air cooling. The temperature rate of furnace cooling was 20 K min⁻¹. After the heat treatments of specimens the structure was observed by light microscopy and scanning electron microscopy (SEM) on metallographic surface. Preparation included polishing and etching in 12 ml H₂O+25 ml glycerol+12 ml HNO₃+1 ml HF.

2.4 Compression tests

Block specimens with 3.1 mm length of edges and 6 mm height were sectioned by spark-cutting machine from cylindrical specimens (Fig. 1a). The surface of specimens was mechanically and then electrolytically polished. Compression tests were performed under displacement control using MTS servohydraulic machines. The strain rate of 2.5x10⁻³ s⁻¹ was kept constant in all tests. The tests were performed at room temperature in air.

3. RESULTS

3.1 Microstructures after heat treatments

When the specimens were air cooled from temperature 1 350 °C the near lamellar microstructure changed to fully lamellar and modifies from rugged grains (as-cast in Fig. 1b) to polyhedron grains with planar grain boundaries – see Fig. 4. The average grain size was twice smaller then as-cast structure (see Table 1). Essentially, during the air cool the lamellae thickness of the γ phase is finer than as-cast state. Furnace cooling to 1 315 °C and air cooling, results in a fully lamellar structure (see Fig. 5), with the almost same average grain size and shape grains as air cooling (see Table 1) with thicker lamellas as air cooling. After furnace cooling to the 1 200 °C followed air cooling, the fully lamellar structure (see Fig. 6) was had interlocked grain boundary with variety grain size (see Table 1) but approximately the same size as previous heat treatments. Due to the bimodal furnace/air stepped cooled had the dual lamellar structure. Thicker lamellae were resulting from furnace cooling and thin lamellae formation by air cooling. Furthermore,
in Fig. 6b many of the finer air cooled lamellae span the entire grain, indicating that a second lamellar nucleation event has occurred during air cooling.

3.2 Compression mechanical properties

According to the compression stress–strain curves, the several compression yield stresses as \( \sigma_{0.1} \), \( \sigma_{0.2} \), the largest ultimate compression strength (breaking stress) \( \sigma_{\text{UCS}} \) and ultimate compression strain \( \varepsilon_{\text{UCS}} \) correspond to \( \sigma_{\text{UCS}} \) of all heat treatments specimens and as-cast sample were calculated by means of many tests. The typical compression stress-strain curves are shown in Fig. 7 and the results of all tests are listed in Table 1. The test was done when the specimen rupture (the rapid drop of the end compression curve). It is clear from Table 1 and Fig. 7 that the compression yield stresses of air cooled and furnace cooled to 1 315 °C specimens are higher than furnace cooled to 1 200 °C and as-cast specimens, while the compression strain \( \varepsilon_{\text{UCS}} \) was opposite character. The compression strain of the as-cast specimens was twice bigger then air cooled specimens. The same effect was in furnace cooled to 1 200 °C and furnace cooled to 1 315 °C specimens. The heat treatments as air cooled and furnace cooled to 1 315 °C was lower compression ultimate strength \( \sigma_{\text{UCS}} \) than as-cast and furnace cooled to 1 200 °C. In the stress–strain curves the elastic and the plastic region can be found. The compression stress–strain curves in the plastics region were approximated by the Hollomon power law:

\[
\sigma = K \varepsilon^n
\]

where \( K \) is hardening coefficient and \( n \) is hardening exponent. These material parameters were evaluated by linear regression analysis and their values are listed in Table 1. The hardening mechanism on the compression stress–strain curves is nearly the same because the hardening exponents \( n \) are almost identical, for used heat treatments, and the smaller differences was caused by different lamella thickness (see Table 1).

4. DISCUSSION

The heat treatment used in the present work produces fine fully lamellar structures with almost identical grain size which differ mainly in the grain boundary morphology and lamella thickness. The differences influence mechanical properties [5], in our case the compression behaviour at room temperature. From this point of view, structures produced by air cooling and furnace cooling to 1 315 °C followed by air cooling were more strength but less plastic than as-cast structures and the structure obtained by furnace cooling to 1 200 °C followed by air cooling. This can be explained by and blocking of moving dislocations in the grain boundary and lamella spacing. When the grain boundaries are planar and the lamella thickness is small then the dislocation moving is difficult. Such an alloy have higher yield stress but low plasticity in comparison with structures produced both by air cooling and furnace cooling to 1 315 °C followed by air cooling shown in Fig. 4 and Fig. 5. But lower yield stress and great plasticity can be found in the case of as-cast structures (Fig. 1) and structure (Fig. 6) given by furnace cooling to 1 200 °C and air with the rugged grains and thick lamellae. However, structures produced by furnace cooling to 1 200 °C followed by air cooling have better creep resistance because the degradation creep mechanism is grain boundary sliding [3] and grain boundaries are interlocked [2, 3] as seen in Fig. 6.

Nevertheless, next creep and fatigue tests are needed to decide which heat treatment is better.
Fig. 4. Microstructure after the heat treatment 1 350 °C / 25 min → Air: (a) optical image of polyhedron grains; (b) SEM images of lamellar microstructure in the one grain.

Fig. 5. Optical image of TiAl alloy microstructure after the pointed cool heat treatment 1 350 °C / 35 min → furnace to 1 315 °C → Air: (a) polyhedron grains; (b) detail of lamellar microstructure.

Fig. 6. Optical image of TiAl alloy microstructure after the pointed cool heat treatment 1 350 °C / 35 min → furnace to 1200 °C → Air: (a) interlocked grain boundary; (b) detail of lamellar microstructure.
Table 1. Results from image analysis and compression mechanical properties of the all tests.

<table>
<thead>
<tr>
<th></th>
<th>as-cast</th>
<th>1350°C Air</th>
<th>1350°C F. 1315°C A.</th>
<th>1350°C F. 1200°C A.</th>
</tr>
</thead>
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<tr>
<td>Grain size, [μm]</td>
<td>898.2 ± 412.2</td>
<td>624.9 ±150.9</td>
<td>637.3 ±159.5</td>
<td>657.8 ±239.9</td>
</tr>
<tr>
<td>Lamellar thick., [μm]</td>
<td>0.97 ± 0.25</td>
<td>0.51 ± 0.14</td>
<td>0.73 ± 0.18</td>
<td>1.02 ± 0.24</td>
</tr>
<tr>
<td>σ0.1 [MPa]</td>
<td>598 ±36.5</td>
<td>798 ±50.5</td>
<td>1026 ±0.9</td>
<td>585 ±40.5</td>
</tr>
<tr>
<td>σ0.2 [MPa]</td>
<td>642 ±45.5</td>
<td>861 ±46</td>
<td>1117 ±1</td>
<td>621 ±35</td>
</tr>
<tr>
<td>σUCS [MPa]</td>
<td>1797 ±100</td>
<td>1498 ±53</td>
<td>1577 ±71</td>
<td>1626 ±188</td>
</tr>
<tr>
<td>εUCS [%]</td>
<td>25.9 ± 1.7</td>
<td>13.4 ± 2.2</td>
<td>12.4 ± 0.9</td>
<td>25.1 ± 1.3</td>
</tr>
<tr>
<td>K [MPa]</td>
<td>3067 ±3107</td>
<td>3107 ±3416</td>
<td>3860 ±25</td>
<td></td>
</tr>
<tr>
<td>n</td>
<td>0.448 ± 0.354</td>
<td>0.354 ± 0.347</td>
<td>0.347 ± 0.541</td>
<td></td>
</tr>
</tbody>
</table>

6. CONCLUSIONS

The structure of near γ-TiAl fully lamellar morphology and grain boundary shape via a pointed cool heat treatment has been investigated and the influence of the microstructural features on compression mechanical properties has been identified in TiAl-8Nb intermetallic. The main conclusions can be summarized as follows:

(i) Important phase transformation temperatures including the α and β transus and the eutectic temperature were obtained using the DTA measurement.

(ii) All the used heat treatments remould the as-cast nearly lamellar structure to the fully lamellar uniform structure and result in the decrease of an average grain size to the half.

(iii) The heat treatment, consisting both of the air cooling and the pointed cool heat treatment (furnace cooling to 1 315 °C followed by air cooling) improve the yield stresses and decrease plasticity due the very thin lamellar thickness and planar grain boundary.

(iv) Pointed cooling from the α+β2 region, by furnace to 1 200 °C followed by air cooling, results in improving plasticity due to thicker lamellar spacing and locked grain boundaries.

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LITERATURA


