EFFECT OF OXYGEN ON THE MICROSTRUCTURE OF ANNEALED TiAl BASED ALLOY

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Abstract

The effect of oxygen on the microstructure of cast intermetallic TiAl-based alloy with nominal composition Ti-48Al-2Nb-1Cr-0.2Si (at.%) was studied after isothermal annealing at temperatures ranging from 1150 to 1320 °C followed by free air cooling to room temperature. The samples for annealing were prepared from an ingot with an oxygen content of 400 wt. ppm and from cast turbocharger wheels with two different amounts of oxygen of 1000 and 1800 wt. ppm. While the microstructure of the alloy with the oxygen content of 400 wt. ppm changes from duplex one composed of lamellar α2+γ and single phase γ grains to nearly lamellar with increasing annealing temperature, the microstructure of the annealed samples with oxygen content of 1000 and 1800 wt. ppm remains nearly lamellar within the studied temperature range. The evolution of the Vickers hardness with the annealing temperature is discussed from the point of view of volume fraction of single phase γ grains. The effect of oxygen on the Vickers hardness of studied castings is described.

Keywords: TiAl, oxygen, heat treatment, microstructure, hardness

1. INTRODUCTION

TiAl-based alloys belong to the attractive progressive light-weight materials for high-temperature structural applications in the aerospace and automotive industries. Despite the protective atmosphere during melting and casting that represent most common metallurgical preparation of the components from these alloys, the reaction between the molten alloy and the ceramic crucibles or moulds (Al2O3, Y2O3, CaO) used in these processes is practically unavoidable [1, 2, 3]. The contamination of the alloy by oxygen and oxide particles during melting and casting has a negative impact on room temperature ductility of cast components [4, 5].

Four basic types of microstructure are formed in the cast TiAl-based alloys: (i) near γ, (ii) duplex, composed of α2 + γ lamellar grains and equiaxed γ grains, (iii) nearly lamellar and (iv) fully lamellar, composed of α2 + γ lamellar grains. The duplex type of microstructure is more ductile at room temperature than the lamellar type, while lamellar microstructure is tougher and more creep resistant than the duplex microstructure [5, 6]. The resulting microstructure of the cast component depends not only on the chemical composition and parameters of the casting process, but is significantly influenced by the subsequent heat treatments. Cast components are usually subjected to hot isostatic pressing in α + γ phase field to eliminate cast porosity.

The aim of the present work was to investigate the effect of oxygen on the microstructure and Vickers hardness after annealing followed by free air cooling of cast Ti-48Al-2Nb-1Cr-0.2Si (at.%) alloy. The alloy was studied in the form of cast ingot and centrifugally cast turbocharger wheels with different amounts of oxygen. Alloving with Cr and Nb was selected for better ductility and oxidation resistance of the studied alloy.

2. EXPERIMENTAL PROCEDURE

Experimental TiAl-based alloy with nominal composition Ti-48Al-2Nb-1Cr-0.2Si (at.%) was supplied in two forms: (i) initial ingot with a diameter of 55 mm and length of 30 mm and (ii) two centrifugally cast turbocharger wheels prepared by CCN GROUP Castings, s.r.o., Považská Bystrica. The samples for heat treatment were cut from the central part of the ingot and from the central part of the as-cast wheels to the blocks with dimensions of 10x10x15 mm³. The samples were subjected to isothermal annealing at
temperatures between 1150 and 1320 °C in protective argon atmosphere for 4 hours with subsequent free air cooling to room temperature. Microstructural investigations of the samples before and after heat treatment were performed by optical microscopy (OM), backscattered scanning electron microscopy (BSEM) and X-ray diffraction analysis (XRD). Chemical composition of the alloy was analysed by energy-dispersive spectrometry (EDS) using JSM-7600F scanning electron microscope equipped with EDS detector. For measurements of oxygen content, LECO ONH836 Elemental Analyzer was used. OM, BSEM, XRD and EDS samples were prepared using standard grinding and polishing metallographic techniques. After mechanical polishing the samples for optical microscopy were chemically etched in a reagent consisting of 100 ml H2O, 10 ml HNO3 and 3 ml HF. Vickers hardness measurements were performed on polished and slightly etched surfaces at an applied load of 98 N (HV10). Volume fraction of γ grains was determined from the digitalized micrographs using computer image analyser.

3. RESULTS

3.1 Chemical composition and microstructure of the alloy before heat treatment

Chemical composition of the initial ingot (I400) and cast turbocharger wheels (W1000 and W1800) measured by EDS and values of the oxygen content measured by the LECO ONH836 analyzer are summarized in Tab. 1. While the average content of the alloying elements in all castings is approximately the same, increase of yttrium and oxygen content in the turbocharger wheels resulting from induction melting in a ceramic crucible and centrifugal casting into ceramic mould is observed in the samples.

<table>
<thead>
<tr>
<th>Sample</th>
<th>EDS (at.%)</th>
<th>LECO (wt. ppm)</th>
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<tbody>
<tr>
<td></td>
<td>Ti</td>
<td>Al</td>
</tr>
<tr>
<td>I400</td>
<td>49.17</td>
<td>47.37</td>
</tr>
<tr>
<td>W1000</td>
<td>49.31</td>
<td>47.24</td>
</tr>
<tr>
<td>W1800</td>
<td>49.25</td>
<td>47.22</td>
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Fig. 1 Microstructure of the samples I400, W1000 and W1800 before annealing: (a) I400 (OM); (b) W1000 (OM); (c) W1800 (OM); (d) single phase γ regions, clusters of silicides and Y2O3 particles in the sample W1800 (BSEM); (e) clusters of silicides and Y2O3 particles in the sample W1800 (OM); (f) γ grains at the lamellar grain boundaries in the sample W1000 (OM)
The microstructure of the sample I400 is nearly lamellar, as shown in Fig. 1a. Equiaxed lamellar grains consist of \( \alpha_2 \) (Ti_3Al phase with \( D0_{19} \) crystal structure) and \( \gamma \) (TiAl phase with \( L1_0 \) crystal structure) lamellae. Single phase \( \gamma \) grains are formed predominantly along the lamellar grain boundaries and their volume fraction is measured to be 7 vol. %. Besides the \( \gamma \) grains at the lamellar grains boundaries, single \( \gamma \) regions with a diameter up to 50 \( \mu \)m and the same crystallographic orientation as the \( \gamma \)-phase in the neighboring lamellae are observed within the lamellar colonies. These \( \gamma \) regions with diffusive boundaries and very random occurrence are either original interdendritic \( \gamma \) regions or are formed during solid-state transformation/dissolution of the \( \alpha \)-phase during cooling of the castings. In addition, the clusters of silicides are identified in the structure.

Figs. 1b and 1c show the typical microstructure of the samples W1000 and W1800 that consists largely of the columnar grains composed of \( \alpha_2 \) + \( \gamma \) lamellae with occasional appearance of single phase \( \gamma \) regions, clusters of silicides and \( Y_2O_3 \) particles, as shown in Figs. 1d and 1e. At the boundaries of the lamellar grains the single phase \( \gamma \) grains are formed (Fig. 1f). Volume fraction of the single phase \( \gamma \) grains is measured to be 5 and 2 vol. % in the sample W1000 and W1800, respectively.

### 3.2 Microstructure and Vickers hardness of annealed alloy

After annealing the substantial differences between the microstructure of the studied samples were observed, as shown in Fig. 2.

![Fig. 2 Microstructure of the samples I400, W1000 and W1800 after annealing at, (OM): (a) 1150 °C, I400; (b) 1280 °C, I400; (c) 1320 °C, I400; (d) 1150 °C, W1000; (e) 1280 °C, W1000; (f) 1320 °C, W1000; (g) 1150 °C, W1800; (h) 1280 °C, W1800; (i) 1320 °C, W1800](image-url)
While the microstructure of the sample I400 changes from duplex to nearly lamellar with the increasing annealing temperature, the microstructure of the samples W1000 and W1800 remains nearly lamellar after annealing in the studied temperature range. Fig. 3 shows the evolution of the volume fraction of γ grains $V_{\gamma g}$ with the annealing temperature. From the diagram is clear that the annealing temperature has no substantial effect on the volume fraction of lamellar grains in the samples W1000 and W1800 over the studied temperature range but significantly affects the volume fraction of γ grains in I400. The duplex microstructure with prevailing γ grains after annealing at temperatures between 1150 and $1280^\circ C$ of the sample I400 abruptly changes to the nearly lamellar microstructure after annealing at the temperatures of 1300 and $1320^\circ C$, as seen in Fig. 3. This sharp decrease in $V_{\gamma g}$ of the initial ingot can be explained by the transition from annealing in a thermodynamically stable $\alpha + \gamma$ field to the $\alpha$-phase field at the temperatures above $1300^\circ C$ [7]. In the samples W1000 and W1800 the volume fraction of single γ grains remains after all studied temperatures nearly constant, but its value in W1000 is higher than in W1800. Formation of the γ grains at the lamellar grains boundaries during annealing is accompanied by the formation of particles and thickened lamellae (Fig. 4) with higher amount of Cr and lower content of Al, as is shown in the Tab. 2. Results of the XRD analysis didn’t prove the presence of β phase, so we assume, that these Cr enriched particles and thickened lamellae consist predominantly of $\alpha_2$-phase and are formed due to ejection of the oxygen from the γ grains during their growth because of much lower solubility of oxygen in the γ-phase than in $\alpha$-phase which will be discussed further.

![Fig. 3](image1.png)

**Fig. 3** Evolution of the volume fraction of γ grains $V_{\gamma g}$ with the annealing temperature

![Fig. 4](image2.png)

**Fig. 4** Microstructure of the sample W1000: (a) as-cast (BSEM); (b) after annealing at $1260^\circ C$ (BSEM) and (c) after annealing at $1240^\circ C$ (OM)

<table>
<thead>
<tr>
<th></th>
<th>EDS (at.%)</th>
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<tbody>
<tr>
<td></td>
<td>Ti</td>
</tr>
<tr>
<td>γ grains</td>
<td>48.45 ± 0.32</td>
</tr>
<tr>
<td>$\alpha_2$ particles</td>
<td>55.90 ± 1.56</td>
</tr>
</tbody>
</table>

**Table 2** Chemical composition of γ grains and $\alpha_2$ particles in the sample W1000 after heat treatment
Vickers hardness HV10 of the annealed samples from the ingot with 400 wt. ppm (I400) increases with increasing annealing temperature, as is shown in Fig. 5, which can be explained by the decrease of $V_{\gamma g}$ with the temperature at the expense of harder lamellar grains (Fig. 3). Vickers hardness HV10 of the samples from the turbocharger wheels (W1000 and W1800) decreases with increasing annealing temperature, as shown in Fig. 5. Because the microstructure in the both wheels samples after annealing at all studied temperatures remained nearly lamellar with nearly the same volume fraction of single $\gamma$ grains (Fig. 3), decrease of HV10 with the temperature can be explained only by the transformation inside the lamellar structure. This assumption was approved by the measurement of microhardness $HV_m$ and interlamellar $\alpha_2-\alpha_2$ spacing in the lamellar grains of the wheel with 1800 wt. ppm, discussed elsewhere [8].

4. DISCUSSION

Stabilization of the lamellar structure in the annealed samples W1000 and W1800 can be attributed to the contamination of the alloy by oxygen. Content of the oxygen in the sample W1000 increased more than twice and in sample W1800 more than four times in comparison with its content in as-received ingot I400, as is shown in Tab. 1. Solubility of oxygen in the $\gamma$ and $\alpha_2$-phase is significantly different. Concentration of O in the $\gamma$ phase is typically found to be 250-300 at. ppm, while up to the 6 at.% has been reported in the $\alpha_2$ lamellae [5]. Because low solubility of oxygen in the $\gamma$-phase cannot be explained by thermodynamic evaluation of Ti-Al-O system, which supposes solubility up to 3 at.% of oxygen in the $\gamma$-phase, Menand et al. [9] suggested that oxygen atoms occupy octahedral interstitial sites in the closed-packed structures. Despite the same size of these sites in $\gamma$ ($L1_0$ structure) and $\alpha_2$ ($DO_{19}$ structure), oxygen atoms are preferentially located in the $\alpha_2$-phase, because they prefer octahedral interstitial sites surrounded by six titanium atoms, which are present in the $\alpha_2$ but not in the $\gamma$. Limited solubility of oxygen in the $\gamma$-phase causes stabilization of the $\alpha_2$ lamellae and inhibits formation of the $\gamma$ grains during annealing at the studied temperatures. As has been already proved by Lamirand et al. [6] in ternary and quaternary alloys contaminated with 1000-2000 wt. ppm of oxygen, also stabilization effect of Cr on the $\gamma$-phase is minimized because of large stabilization effect of oxygen on the lamellar structure at such high oxygen contents.

Considerably lower values of $V_{\gamma g}$ in the samples W1000 and W1800 when compared to that of I400 at all studied temperatures clearly indicate that the contamination of the studied alloy by oxygen and yttrium affects significantly solid phase transformations and hinders formation of duplex type of microstructure. In addition, this contamination increases significantly the Vickers hardness of the samples W1000 and W1800 during annealing at the temperatures up to 1300 °C when compared to that of I400 samples. However, in spite of the fact that HV10 measurements show lower average hardness values of W1000 than those of W1800, these differences are within standard deviations of the measurements. Hence, increase of oxygen content from 1000 to 1800 wt. ppm does not affect the microstructure and Vickers hardness after annealing at the temperatures between 1150 and 1320 °C to such a great extent as the increase from 400 to 1000 wt. ppm.
CONCLUSIONS

The study of the effect of the oxygen on the microstructure of annealed TiAl-based alloy with nominal composition Ti-48Al-2Nb-1Cr-0.2Si (at.%) can be summarized as follows:

1. Duplex type of microstructure with prevailing \( \gamma \) grains formed during annealing at the temperatures between 1150 and 1280 °C of the samples with oxygen content of 400 wt ppm changes to the nearly lamellar microstructure after annealing at the temperatures above 1300 °C.

2. The contamination of the studied alloy by oxygen from 1000 to 1800 wt ppm affects significantly solid phase transformations and hinders formation of duplex type of microstructure during annealing at temperatures ranging from 1150 to 1320 °C.

3. The contamination by oxygen of 1000 and 1800 wt ppm increases significantly Vickers hardness of the samples when compared to that of the samples with oxygen content of 400 wt ppm. Average Vickers hardness values of samples with oxygen content of 1000 wt ppm are lower than those of samples with 1800 wt ppm but these differences are within standard deviations of the measurements.

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