EFFECT OF ANNEALING ON MICROSTRUCTURE AND PROPERTIES OF YTTRIUM ALLOYED INTERMETALLICS Ti-47Al

Tomáš ČEGAN, Miroslav KURSA, Kateřina KONEČNÁ, Daniel PETLÁK

Vysoká škola báňská – Technical University of Ostrava, Faculty of Metallurgy and Materials Engineering, 17. listopadu 15, 708 33 Ostrava - Poruba, Czech Republic, tomas.cegan@vsb.cz

Abstract

Ti-Al intermetallic alloys, especially γ-TiAl alloys, are perspective materials for high-temperature applications in aerospace, automotive and power industries. However, their mechanical properties highly depend on the obtained microstructure. It was proved that the refinement of grain size leads to improvement of mechanical properties. However, process parameters and alloy composition should be regulated in the narrow limits to achieve a low porosity and a microstructure required in the solid state. The influence of annealing at different temperatures and different cooling conditions on the microstructure of the Ti-47Al binary alloy and also on the samples alloyed with yttrium in range from 0.1 to 0.4 at. % was studied in this article. It was achieved several type of microstructures – fully lamellar, nearly lamellar and duplex type. It was compared the influence of yttrium as refining element on the different types of structures and also the influence of this element on microhardness.

Keywords: TiAl alloys; microstructure; yttrium; annealing; microhardness

1. INTRODUCTION

Alloys based on γ-TiAl are a unique type of material. They are characterized by low density (3.8 g/cm³) and good strength at elevated temperatures. This is ideal for using in the components of engines used in the automotive and aerospace industries, where they could replace the currently used nickel alloys and titanium based alloys [1, 2]. There are many different methods of preparation of alloys based on γ-TiAl. The most commonly used methods are [3]: arc melting in a vacuum (VAR), plasma melting (PAM), vacuum induction melting (ISM). However, preparation of appropriate ingots is very complicated. Titanium aluminides are highly susceptible to reactions with refractory materials of crucibles and also with oxygen or other gases (N₂, H₂) during the melting. This can leads to the reduction in ductility [4], or even to change the primary crystalization phase from β to α [5].

Plasma melting in water-cooled copper crystallizers under a dynamic argon atmosphere allows the preparation of high purity alloys with small content of gases and impurities. However, the influence of different temperature gradients in the ingots during solidification causes that products of plasma melting exhibit inhomogeneous composition and different types of microstructures in various parts of the products [3, 6]. Another problems for the further using of alloys based on γ-TiAl are the low formability and low ductility at room temperature, which is their typical feature. Many efforts have been devoted to increase the plastic properties of TiAl alloys. It was designed and tested a number of different methods such as alloying by elements, which precipitates causes reduction of grain size and interlamellar spacing. Yttrium is one of the elements, which refines the microstructure [7, 8]. Affect the resulting structure of the alloys can also achieved by annealing in α + γ region of the binary diagram (see Fig. 1). This type of annealing causes change to the duplex microstructure. This type of microstructure exhibits higher ductility at low temperatures, but lower resistance to creep [2, 9]. Annealing in the α region causes the microstructure changes to fully lamellar, which exhibit higher creep resistance and lower ductility [2]. The aforementioned implies, that the properties
of alloys based on $\gamma$-TiAl are very sensitive to changes in microstructure and appropriate microstructure modification can increase the mechanical properties.

The aim of this article is to describe the effect of annealing at various temperatures on the microstructure of plasma-melted alloy Ti-47Al and influence of alloying by small additions of yttrium for the purpose of grain refinement. The possibility of increasing homogeneity of the composition and microstructure of alloys by appropriate heat treatments and influence on microhardness were investigated.

2. EXPERIMENT

Five alloys with a nominal composition Ti-47Al and different amounts of yttrium additions (0-0.4 at. %) were prepared by plasma melting in a water-cooled copper mould. As charge were used master alloys, which were prepared by vacuum induction melting in corundum crucible with $Y_2O_3$ coating. This coating was applied to prevent contamination of alloys by $Al_2O_3$ particles. Preparation of master alloys by this method was selected in order to increase homogeneity. Plasma melting was carried out in the dynamic argon atmosphere with flow rate of 27 l/min for 60 s for each alloy. Used current density was 600 A and voltage 54 V. Purity of argon was 4N6. The products of plasma melting were oval samples. These samples were cut in half vertically and the cuts were taken for metallographic examination. Subsequently, they were cut in half again, so that from each oval sample remained four almost identical parts. These samples were subsequently sealed in quartz tubes and evacuated to 1-2 Pa. Consequently they were processed by annealing for 4 h at various temperatures and different cooling rates: 900 °C ($\alpha_2 + \gamma$ region, HT1) - slowly cooled inside the furnace, 1200 °C ($\alpha$+ $\gamma$ region, HT2) - slowly cooled inside the furnace, 1200 °C ($\alpha$+ $\gamma$ region, HT3) – quenched by inserting the ampoule into a container with water, 1370 °C ($\alpha$ region, HT4) - cooled slowly inside the furnace and quenched by inserting the ampoule into a container with water at 750 °C. The alloys were observed by optical microscopy on the microscope Olympus GX51 equipped with digital camera Olympus DP12 (OM), scanning electron microscopy in mode of scattered electrons (BSE) on the microscope SEM JEOL JSM - 6490LV, equipped with a probe EDS INCA X – ACT and on the microscope QUANTA FEG 450 with a probe EDAX APOLLO X. Measurement of grain size and inter-lamellar spaces were performed with using the program analySIS auto in images taken by optical microscopy. Micro-hardness measurements were performed on micro-hardness tester FM-ARS 9000 at a load of 100 g for 7 s. Decrease of microhardness between bottom and upper part of samples was measured with an arrangement of the points of measurement into a matrix in the upper part and bottom part of cut of samples. Samples were prepared for metallographic observation by standard methods of grinding and polishing and used etchant was a mixture
of 50 ml H$_2$O, 40 ml HNO$_3$ a 10 ml HF on visualisation of macrostructure and 50 ml H$_2$O, 3 ml HNO$_3$ a 1.5 ml HF on visualisation of microstructure.

![Fig. 3 Microstructure of sample Ti-47Al (OM) a) bottom part b) upper part](image)

![Fig. 4 Cluster of Y$_2$O$_3$ particles (BSE)](image)

![Fig. 5 BSE image of as-cast alloy Ti-47Al-0.4Y](image)

3. RESULTS

<table>
<thead>
<tr>
<th>Table 1 Analysed composition [at. %]</th>
<th>O</th>
<th>Al</th>
<th>Ti</th>
<th>Y</th>
</tr>
</thead>
<tbody>
<tr>
<td>Element 1</td>
<td>64.14</td>
<td>0.43</td>
<td>35.43</td>
<td></td>
</tr>
<tr>
<td>Element 2</td>
<td>51.2</td>
<td>13.94</td>
<td>15.24</td>
<td>19.62</td>
</tr>
<tr>
<td>Element 3</td>
<td>43.85</td>
<td>21.13</td>
<td>19.42</td>
<td>15.6</td>
</tr>
<tr>
<td>Element 4</td>
<td>51.1</td>
<td>48.9</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Element 5</td>
<td>46.2</td>
<td>53.8</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Fig. 2 shows the typical macrostructure of the plasma melted ingots. Three different regions can be well distinguished on the transverse section: (i) region of fine columnar grains at the bottom, (ii) region of coarse columnar grains in the middle and (iii) fine equiaxed grains in the upper part. In Fig. 3 are shown two different types of structures that were observed in all prepared as-cast samples. At the bottom, which has been in contact with the copper crystallizer, was occurred fully lamellar structure consisting of alternating lamellas $\alpha_2$ and $\gamma$ phase. In the middle and upper parts was occurred a typical dendritic structure consisting of lamellar dendrites and interdendritic $\gamma$-phase. The amount of interdendritic $\gamma$ phase increases toward the upper edge of the cut of samples. Establishment of this type of structure can be explained by rapid solidification of $\alpha$ grains upwards from the bottom of the samples. The $\alpha$ phase grains have a higher content of titanium compared with the nominal composition of alloys (see Fig. 1). These results to the enrichment of the remaining melt with aluminum [5, 11]. Thereafter $\gamma$ phase was created by peritectic reaction of $\alpha$ with liquid to produce $\gamma$ at the border of $\alpha$ grains. The $\alpha$ grains subsequently transformed to the lamellar structure of $\alpha_2$ and $\gamma$.

Nonmetallic particles were observed in all prepared alloys. These particles are shown in Fig. 4. Their size ranged up to 50 $\mu$m and they were occurred mainly in clusters at the bottom of the cut of samples.
chemical composition analyzed by EDS was approximately 35 at. % Y and 65 at. % O. Besides yttrium and oxygen were other elements contents in the particles negligible. These particles got into the samples during the preparation of master alloys from protective coating probably by mechanical erosion of the melt [12]. Apart from particles released from the protective coating was observed another phase rich on yttrium in the prepared alloys with yttrium addition. These particles reached usually smaller dimensions than 2 μm and had mostly ellipsoidal or elongated shape. Their EDS analysis revealed that the composition is similar to the particles in the master alloys, which has been described in another article [13] and corresponds to Y₂O₃. These phases were occurred mainly in the inter-dendritic spaces, but also within the lamellar grains, which can be seen in Fig. 5, where yttrium-rich phases are shown by white colour. Results of composition analysis in marked points are shown in Tab. 1.

Fig. 6 Macrostructures of samples a) Ti-47Al-0.4Y after HT1 b) Ti-47Al-0.4Y after HT3 c) Ti-47Al after HT4 d) Ti-47Al-0.4Y after HT4

Fig. 7 BSE image of alloy Ti-47Al-0.4Y after HT3

Fig. 8 BSE image of alloy Ti-47Al-0.4Y after HT2

Fig. 6a shows macrostructure of sample Ti-47Al-0.4Y after heat treatment in the α₂ + γ region. By comparing this image with Fig. 1 can be concluded that annealing in the α₂ + γ field significantly was not affected the size and shape of grains. Observation of microstructure was not found greater differences compared to as-cast alloys. Fig 6b shows macrostructure of sample Ti-47Al-0.4Y after heat treatment at 1200 °C and quenched by inserting the ampoule into a container with water. In this image can be also recognized that the annealing in the α + γ field also significantly was not affected the size and shape of grains. The characteristic microstructures obtained by heat treating of samples in the α + γ field are shown in the Figs. 7 and 8. In the SEM microstructures shown in Fig. 8 the following constituents are identified: γ-phase (dark regions); α₂ (thicker white regions with 60 at. % Ti) and lamellar structure, remaining from dendrites. The amount of remaining dendrites increases toward the upper edge of cut of samples. No significant changes of composition of remaining lamellar dendrites and yttrium rich phases, were not occurred. It is clear that the volume fraction of dendrites after 4 h at 1200 °C decreased, showing that solution of α₂ + γ dendrites has progressed, but for any samples the remaining dendrites were not completely removed. The remaining dendrites were also observed in samples after HT3, but lamellar structure in the dendrites aren’t so apparent
23. - 25. 5. 2012, Brno, Czech Republic, EU

Due to rapid cooling, in Fig. 6c and 6d are shown macrostructures of samples Ti-47Al and Ti-47Al-0.4Y after annealing in the α field. From these images is evident that grains of unalloyed sample are very large and grains of alloyed sample Ti-47Al-0.4Y are finer after HT4. Variations of grain size with addition of yttrium in samples after HT4 and in as-casted samples are shown in Fig. 9. From this graph is clear that refining effect of yttrium has higher efficiency in samples after HT4 than in the as-cast samples and samples after other heat treatments. Microstructure of unalloyed and HT4 samples consisted of coarse fully lamellar grains without remaining dendrites and interdendritic spaces. However, samples alloyed with yttrium consisted of lamellar grains and small amount of γ phase between grains in bottom part. The amount of γ phase increased with increasing yttrium content and increased from bottom to the top of the cut of samples. This means that the additions of Y had apparently stabilized the dendritic structure. Phases rich on yttrium were observed mainly in γ phase (see Fig. 10). The average lamellar spacing of the HT4 samples obviously increased from 0.2 to 1 μm contrary to the as-cast samples. Fig. 9 shows the dependence of yttrium content on lamellar spacing of HT4 samples. The average lamellar spacing decreased from 1.4 μm to 0.8 μm with increasing content of yttrium. The grain and lamellar refining effects of Y were attributed to the precipitation of Y oxides. These oxides may act as nucleation clusters during solidification and recrystallisation. Increased heterogenous nucleation site is the main factor that is relevant to the microstructural refinement in the Y-containing TiAl-based alloys [8]. The microhardness evolution in as-cast samples shows Fig. 11. It follows from this diagram that micro-hardness decreased with increasing content of yttrium and increased from...
upper part (distance 0 in Fig. 11) to bottom (distance 12 mm in Fig. 11). The decrease of microhardness was caused by inhomogeneous composition. Fig. 12 shows dependence of microhardness decrease $\Delta HV$ between bottom and upper part of samples after different heat treatment. It is clear that the most decreasing tendency of $\Delta HV$ after different heat treatment had samples after HT2 and HT4, in which $\Delta HV$ decreased from 95-140 in as-cast samples to 10-40 in HT4 samples and to 5-70 in HT2 samples. This means that microstructure can be partially homogenised by annealing in $\alpha + \gamma$ or in $\alpha$ field.

CONCLUSIONS

Three different types of microstructure were obtained by different heat treatments and different yttrium addition: fully lamellar, nearly lamellar and duplex. Refining effect of yttrium has the highest efficiency in samples after annealing at 1370 °C for four hours. The most homogenized microstructure was occurred in samples after same heat treatment.

ACKNOWLEDGEMENT

This paper was created within the project No. CZ.1.05/2.1.00/01.0040 "Regional Materials Science and Technology Centre" within the frame of the operation programme "Research and Development for Innovations" financed by the Structural Funds and from the state budget of the Czech Republic.

REFERENCES