MICROSTRUCTURE EVOLUTION DURING ANNEALING OF CAST TiAl-BASED ALLOY

Alena KLIMOVÁ, Juraj LAPIN

Institute of Materials and Machine Mechanics, Slovak Academy of Sciences, Bratislava, Slovakia, EU,
ummsakli@savba.sk

Abstract

Microstructure evolution, Vickers hardness and Vickers microhardness were investigated in intermetallic TiAl-based alloy with nominal composition Ti-48Al-2Nb-1Cr-0.2Si (at.%). The samples prepared from as-received cast ingot and centrifugally cast turbocharger wheel were subjected to heat treatment consisting of isothermal annealing at temperatures ranging from 1150 to 1320 °C in protective argon atmosphere and air cooling to room temperature. The effect of contamination of the alloy resulting from induction melting in a ceramic crucible and centrifugal casting into ceramic mould on the microstructure evolution during annealing is discussed.

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

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 (Al_2O_3, Y_2O_3, 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 α_2 + γ lamellar grains and equiaxed γ grains, (iii) nearly lamellar and (iv) fully lamellar, composed of α_2 + 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 evolution of microstructure, Vickers hardness and microhardness during annealing followed by air cooling of cast Ti-48Al-2Nb-1Cr-0.2Si (at.%) alloy. The alloy was studied in the form of as-received cast ingot and centrifugally cast turbocharger wheel. Alloying with Cr and Nb was selected for better ductility and oxidation resistance of the studied alloy.

2. EXPERIMENTAL PROCEDURE

The intermetallic alloy with the nominal composition Ti-48Al-2Nb-1Cr-0.2Si (at.%) was supplied in the form of a cylindrical sample with a diameter of 55 mm and length of 30 mm cut from a vacuum arc remelted ingot prepared by GfE Germany and in the form of centrifugally cast turbocharger wheel prepared by CCN GROUP Castings, s.r.o., Považská Bystrica. The samples for heat treatment experiments were cut from the central part of the ingot and from the central part of the cast wheel to the blocks with dimensions of 10x10x15 mm^3. The heat treatment consisted of isothermal annealing at temperatures ranging from 1150 to 1320 °C in protective argon atmosphere for 4 hours and air cooling to room temperature.

Microstructural investigations of the samples before and after heat treatment were performed by optical microscopy (OM) and backscattered scanning electron microscopy (BSEM). The 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 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 H_2O, 10 ml HNO_3 and 3 ml HF.

Vickers hardness and Vickers microhardness measurements were performed on polished and slightly etched surfaces at an applied load of 98 N (HV10) and 0.49 N (HV_m), respectively. Interlamellar spacing and volume fraction of lamellar grains were determined from the digitalized micrographs using computer image analyser.

3. RESULTS AN DISCUSSION

3.1 Microstructure of the alloy before heat treatment

The microstructure of the as-received ingot is nearly lamellar, as shown in **Fig. 1a**. Equiaxed lamellar grains consist of α_2 (Ti_3Al phase with $D0_{19}$ crystal structure) and γ (TiAl phase with $L1_0$ crystal structure) lamellae. Single phase γ-grains are formed predominantly along the lamellar grain boundaries. In addition, the clusters of silicides were identified within the lamellar and γ grains. The microstructure of the turbocharger wheel is lamellar with occasional γ grains on the grain boundaries (**Fig. 1b**). Besides the silicides, which were observed in the wheel mainly in the interdendritic γ regions, Y_2O_3 particles were identified within lamellar colonies and in the interdendritic regions, as is shown in **Fig. 1c**.

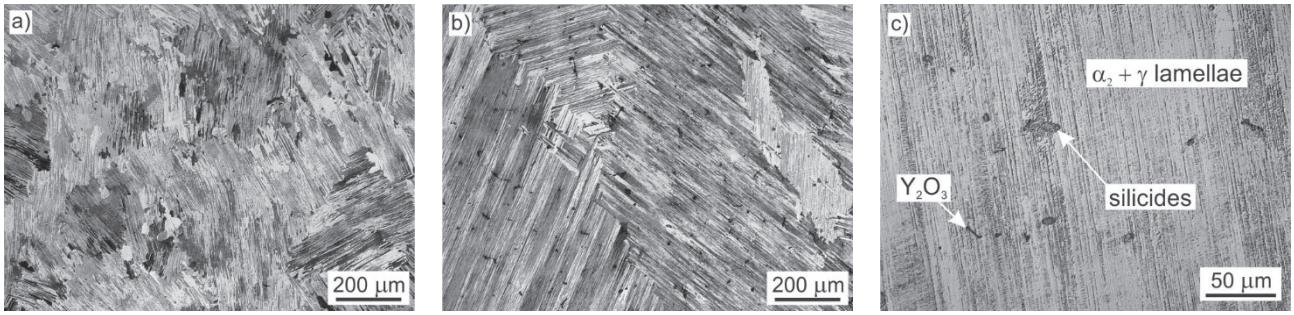

Fig. 1 Optical micrographs: (a) as-received ingot; (b) and (c) turbocharger wheel

Table 1 Chemical composition of the alloy

Sample	EDS (at.%)						LECO (wt. ppm)
	Ti	Al	Nb	Cr	Si	Y	O
As-received ingot	49.17	47.37	2.49	0.76	0.22	-	400
Turbocharger wheel	49.25	47.22	2.45	0.76	0.22	0.1	1800

Chemical composition of as-received ingot and turbocharger wheel measured by EDS and values of the oxygen content measured by the LECO ONH836 analyzer are summarized in **Table 1**. While the average content of the alloying elements in both types of castings is approximately the same, contamination of the turbocharger wheel with yttria (Y_2O_3) and oxygen, resulting from induction melting in a ceramic crucible and centrifugal casting into ceramic mould was detected.

3.2 Microstructure evolution during annealing

After annealing substantial differences between the microstructure of the samples prepared from the as-received ingot and from the turbocharger wheel were observed, as shown in **Fig. 2**. While the microstructure of the as-received ingot changes from duplex to nearly lamellar, the microstructure of the turbocharger wheel remains nearly lamellar after annealing in the studied temperature range. **Fig. 3** shows the evolution of the

volume fraction of lamellar grains V_{lg} with the temperature. It is clear that the annealing temperature has no substantial effect on the volume fraction of lamellar grains in the turbocharger wheel over the studied temperature range but significantly affects the volume fraction of lamellar grains in the as-received ingot. The duplex microstructure with prevailing γ grains after annealing at temperatures between 1150 and 1280°C of the as-received ingot abruptly changes to the nearly lamellar microstructure after annealing at the temperatures of 1300 and 1320 °C, as seen in **Fig. 3**. This sharp increase in V_{lg} of the as-received ingot can be explained by the transition from annealing in a thermodynamically stable α + γ field to the α-phase field at the temperatures above 1300°C [7].

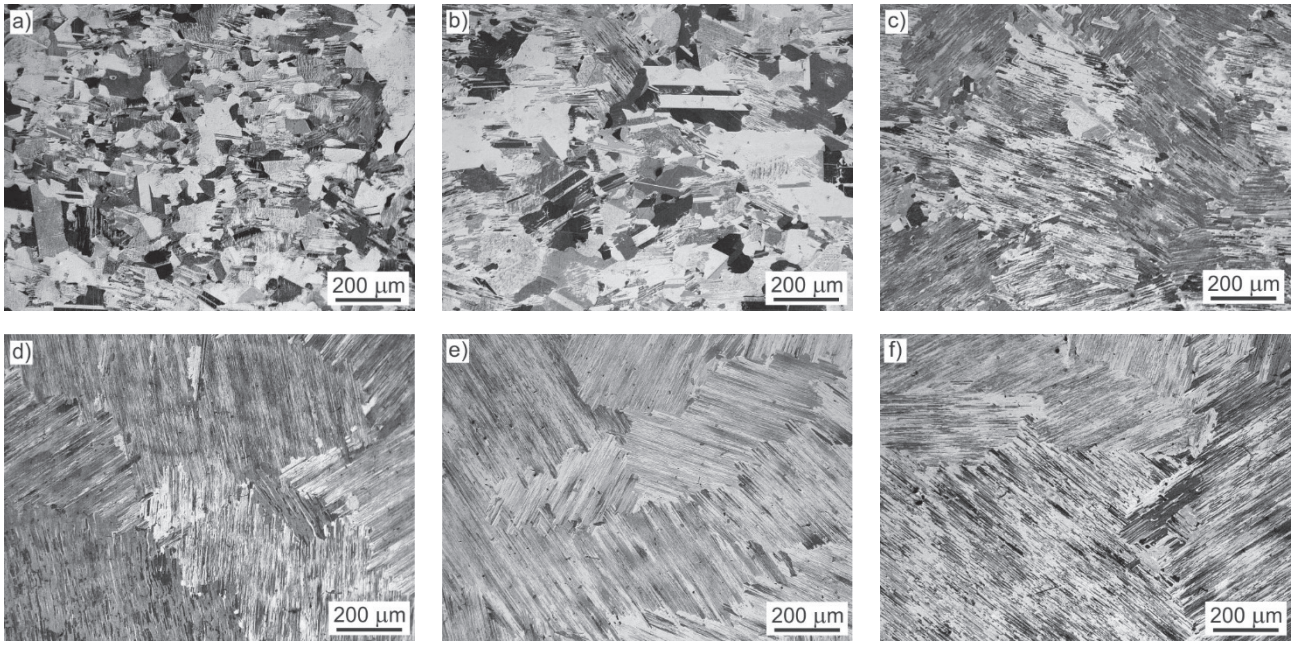

Fig. 2 Optical micrographs after heat treatment: as-received ingot (a) at 1150 °C, (b) at 1280 °C, (c) at 1320 °C and turbocharger wheel (d) at 1150 °C, (e) at 1280 °C, (f) at 1320 °C

In the turbocharger wheel the ratio between prevailing lamellar and occasional γ grains remained after all studied temperatures nearly the same, but the interlamellar α_2-α_2 spacing increased with the temperature from 1.0 µm after annealing at 1150 °C to 1.7 µm at 1320 °C, as shown in **Fig. 4**. Increase of the interlamellar α_2-α_2 spacing with the temperature is most likely connected with the changes in the volume fraction of stable α and γ-phases with the temperature, and with a decrease of cooling rates during air cooling from higher annealing temperatures.

Stabilization of the lamellar structure in the heat treated samples from the turbocharger wheel can be attributed to the contamination of the alloy by oxygen. Content of the oxygen in the turbocharger wheel increased more than four times in comparison with its content in as-received ingot, reaching the value of 1800 wt. ppm, as is shown in **Table 1**. Solubility of oxygen in γ and α_2-phase is significantly different. Concentration of O in the γ phase is typically found to be 250-300 at. ppm, while up to the 6 at.% has been reported in the α_2 lamellae [5]. Because low solubility of oxygen in the γ-phase cannot be explained by thermodynamic evaluation of Ti-Al-O system, which supposes solubility up to 3 at.% of oxygen in the γ-phase, the following explanation interpreted as chemical effect was suggested by Menand *et al.* [8]. Oxygen atoms occupy octahedral interstitial sites in the closed-packed structures. Despite the same size of these sites in γ ($L1_0$ structure) and α_2 ($D0_{19}$ structure), oxygen atoms are preferentially located in the α_2-phase, because they prefer octahedral interstitial sites surrounded by six titanium atoms, which are present in the α_2 but not in the γ [8]. Limited solubility of oxygen in the γ-phase causes stabilization of the α_2 lamellae and inhibits formation of the γ grains during annealing at

the studied temperatures. As was 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 γ-phase is minimized because of stabilization effect of oxygen on the lamellar structure at such high oxygen contents.

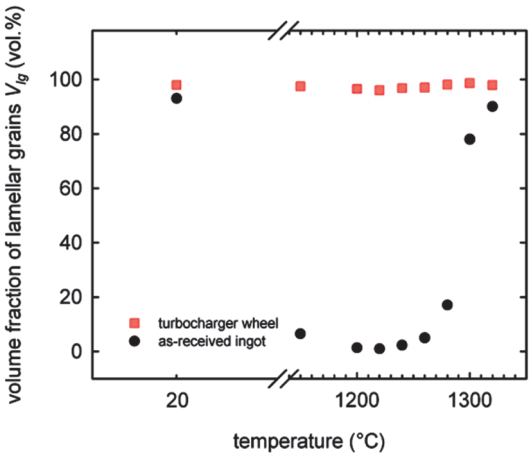

Fig. 3 Evolution of the volume fraction of lamellar grains V_{lg} with the annealing temperature

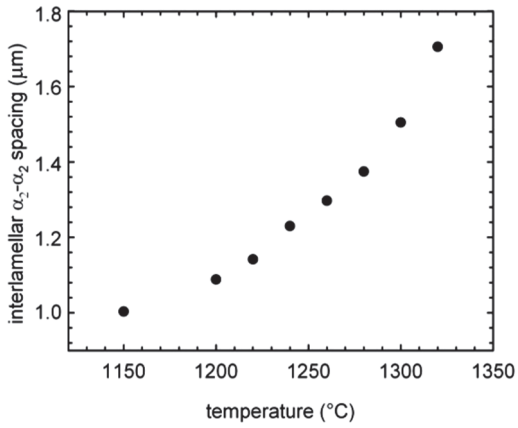

Fig. 4 Evolution of the interlamellar α_2-α_2 spacing with the annealing temperature in the turbocharger wheel

3.3 Evolution of the hardness and microhardness during annealing

Vickers hardness HV10 of the samples from the turbocharger wheel decreases with increasing annealing temperature as shown in **Fig. 5**. Because V_{lg} in the samples from turbocharger wheel after annealing at all studied temperatures remained above 96 vol.%, decrease of HV10 with the temperature can be explained by the transformation inside the lamellar structure. This assumption was approved by the measurements of microhardness HV_m in the lamellar grains. The evolution of HV_m in the lamellar grains with the temperature plotted in **Fig. 6** shows the same decreasing tendency as HV10. Direct relationship between microhardness and interlamellar α_2-α_2 spacing has already been proved by Lapin et al. [9, 10] for directionally solidified alloys. We can suppose that also in studied cast lamellar structure the decrease of HV_m with the temperature is closely related to the observed increase of the interlamellar α_2-α_2 spacing with the annealing temperature (**Fig. 4**).

Vickers hardness HV10 of the samples from the as-received ingot increases with increasing annealing temperature (**Fig. 5**), which can be explained by the increase of V_{lg} with the temperature at the expense of softer γ grains (**Fig. 3**). Due to heterogeneity of lamellae and small size and amount of lamellar grains (max. 17 vol.%), which remained in the structure after heat treatment at the temperatures from 1150 to 1280 °C, only microhardness of lamellar grains after heat treatment at 1300 and 1320 °C could be reliably measured. Their microhardness was comparable with the microhardness of the lamellar grains in the turbocharger wheel after the heat treatment at the same temperatures, as shown in **Fig. 6**. The microhardness of γ grains does not depend on the annealing temperature and was measured to be (257 ± 7) HV_m.

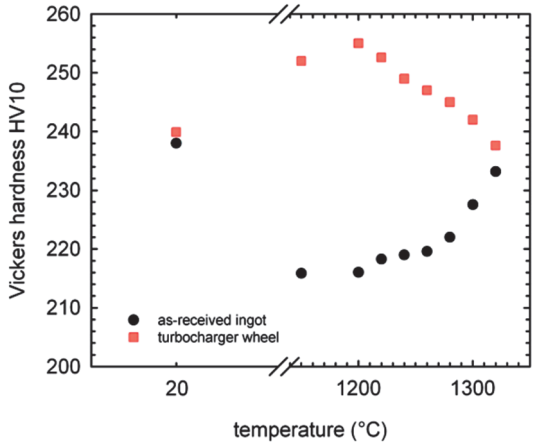

Fig. 5 Evolution of the Vickers hardness with the annealing temperature

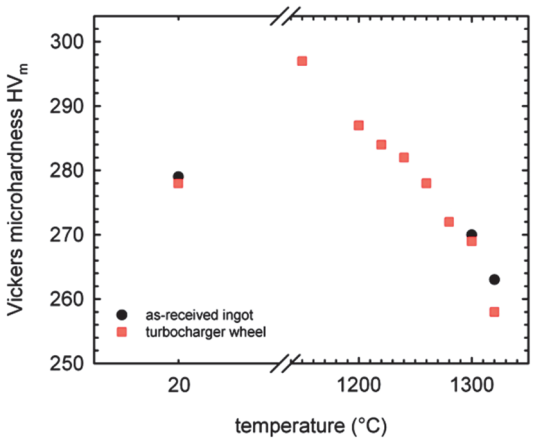

Fig. 6 Evolution of the Vickers microhardness of the lamellar grains with the annealing temperature

CONCLUSIONS

The study of microstructure evolution during heat treatment of cast TiAl-based alloy with nominal composition Ti-48Al-2Nb-1Cr-0.2Si (at.%) could be summarized as follows:

1) While the microstructure of the as-received ingot changed from duplex to nearly lamellar with increasing annealing temperature from 1150 to 1320 °C, the microstructure of the centrifugally cast turbocharger wheel remained nearly lamellar within this studied temperature range.
2) Duplex microstructure with prevailing γ grains formed during heat treatments at annealing temperatures between 1150 and 1280 °C in the as-received ingot abruptly changes to the nearly lamellar microstructure after annealing at the temperatures above 1280 °C. The volume fraction of lamellar grains in the turbocharger wheel remains above 96 vol.% during heat treatment at all studied temperatures. Interlamellar α_2-α_2 spacing in the turbocharger wheel increases from 1.0 to 1.7 μm with increasing annealing temperature from 1150 °C to 1320 °C.
3) Decrease of the Vickers hardness and Vickers microhardness with increasing annealing temperature in the turbocharger wheel can be explained by increasing interlamellar α_2-α_2 spacing. The increase of the Vickers hardness with increasing annealing temperature in the as-received ingot can be related to the increase of the volume fraction of lamellar grains at the expense of softer γ grains.

ACKNOWLEDGEMENTS

The financial support of the Slovak Research and Development Agency under the contract APVV-0434-10 and the Slovak Grant Agency for Science under the contract VEGA 2/0149/13 is acknowledged. The authors would like to thank to Ján Čičman, Executive Manager of CCN GROUP Castings, s.r.o. for supplying of the experimental material.

REFERENCES

[1] ZOLLINGER, J., LAPIN, J., DALOZ, D., COMBEAU, H. Influence of oxygen on solidification behaviour of cast TiAl-based alloys. *Intermetallics*, 2007, Vol. 15, pp. 1343-1350.

[2] LAPIN, J., GABALCOVÁ, Z., PELACHOVÁ, T. Effect of Y_2O_3 crucible on contamination of directionally solidified intermetallic Ti-46Al-8Nb alloy. *Intermetallics*, 2011, Vol. 19, pp. 396-403.

[3] LAPIN, J., ONDRÚŠ, Ľ., NAZMY, M. Directional solidification of intermetallic Ti-46Al-2W-0.5Si alloy in alumina moulds. *Intermetallics*, 2002, Vol. 10, pp. 1019-1031.

[4] TETSUI, T., KOBAYASHI, T., UENO, T., HARADA, H. Consideration of the influence of contamination from oxide crucibles on TiAl cast material, and the possibility of achieving low-purity TiAl precision cast turbine wheels. *Intermetallics*, 2012, Vol. 31, pp. 274-281.

[5] LAMIRAND, M., BONNENTIEN, J.L., GUÉRIN, S., FERRIÈRE, G. , CHEVALIER, J.P. Effects of interstitial oxygen on microstructure and mechanical properties of Ti-48Al-2Cr-2Nb with fully lamellar and duplex microstructures, *Metallurgical and Materials Transactions A*, 2006, Vol. 37, pp. 2369-2378.

[6] LAMIRAND, M., BONNENTIEN, J.L., GUÉRIN, S., FERRIÈRE, G. , CHEVALIER, J.P. Relative effects of chromium and niobium on microstructure and mechanical properties as a function of oxygen content in TiAl alloys. *Scripta Materialia*, 2007, Vol. 56, pp. 325-328.

[7] LU, B., HUANG, L., LIU, Y., LIANG X., LIU, B., HE, Y., LI, H. Evolution of lamellar structure in Ti−47Al−2Nb−2Cr−0.2W alloy sheet, *Transactions of Nonferrous Metals Society of China*, 2013, Vol. 23, pp.1293-1298.

[8] MENAND, A., HUGUET, A., NÉRAC-PARTAIX, A. Interstitial solubility in γ and α_2 phases of TiAl-based alloys. *Acta Materialia*, 1996, Vol. 44, No.12, pp. 4729-4737.

[9] LAPIN, J. Effect of lamellar structure on microhardness and yield stress of directionally solidified intermetallic Ti-46Al-2W-0.5Si alloy. *Journal of Materials Science Letters*, 2003, Vol. 22, No. 10, pp. 747-749.

[10] LAPIN, J., GABALCOVÁ, Z., BAJANA, O. The effect of microstructure on mechanical properties of directionally solidified intermetallic Ti-46Al-8Nb alloy. *Kovove Mater.*, 2009, Vol. 47, No. 3, pp. 159-167.