POSSIBILITIES OF AFFECTING THE LAMELLAR MICROSTRUCTURE OF TI-46AL-5NB-1W ALLOY BY DIRECTIONAL CRYSTALLISATION

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MOŽNOSTI OVLIVNĚNÍ LAMELÁRNÍ MIKROSTRUKTURY SLITINY TI-46AL-5NB-1W POMOCÍ SMĚROVÉ KRYSTALIZACE

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Abstrakt

Intermetalické slitiny vycházející z γ-TiAl dvoufázových slitin jsou vyhledávanými materiály pro předpokládané aplikace v leteckém a automobilovém průmyslu. Jejich příznivé mechanické vlastnosti jako jsou nízká hustota, dobrá korozní odolnost jsou doprovázeny nízkou houževnatostí a obtížnou metalurgickou přípravou. Jednou z možností jak zlepšit houževnatost slitiny Ti-46Al-5Nb-1W (at.%) spočívá v přeměně mikrostruktury na mikrostrukturu lamelární, a to například metodou směrové krystalizace. Tento experiment je popsán v článku. Zvolená slitina Ti-46Al-5Nb-1W (at.%) byla připravena plazmovým tavením v plazmové peci. Nehomogenní mikrostruktura byla upravena vakuově-indukčním tavením s následným odlitím do grafitových kokil. Odlitky byly podrobeny směrové krystalizaci. Rychlosti ochlazování byly konstantní a pohybovaly se v rozmezí 20 mm/h až 425 mm/h. Experiment byl prováděn v keramických korundových trubicích Al_2O_3, ve kterých byly vloženy odlité vzorky slitiny. Mikrostruktura směrově krystalizovaných vzorků byly podrobena metalografickému zkoumání. Bylo zjištěno, že lamelární mikrostruktura je tvořena střídajícími se lamelami fází α_2 a γ. Ve vzorcích byly detekovány keramické částice Al_2O_3. Dosažená lamelární mikrostruktura byla zdokumentována. Byl změřen obsah kyslíku a dusíku v závislosti na parametrech směrové krystalizace. Pomocí DTA analýzy byla určena teplota tavení slitiny.

Abstract

Intermetallic alloys based on γ-TiAl two-phases alloys are sought-after materials for assumed applications in aerospace and automotive industries. Their favourable properties, such as low density and good corrosion resistance, are accompanied on the other hand by low toughness and very difficult metallurgy. One of the possibilities to improve the toughness of Ti-46Al-5Nb-1W (at.%) alloy consists in change of their microstructure into lamellar microstructure, which can be reached moreover by directional crystallisation. This experiment is described in this paper. Samples of the Ti-46Al-5Nb-1W (at.%) alloy were prepared by plasma melting. Then the samples were remelted in vacuum-induction furnace because of the low homogeneity. Cast samples were subjected to directional crystallization. Cooling rates were constant and ranged from 20 mm/h to 425 mm/h. Directional crystallization has been accomplished in ceramic tubes made of corundum – Al_2O_3. The samples were studied by

metallographic analysis. Lamellar microstructure of the samples was found to consist of α_2- and γ-phase lamellas. Moreover, ceramic particles Al_2O_3 were found to be present in the samples. Distribution of the alloying elements in the samples was homogenous. Obtained lamellar microstructures are documented, contents of nitrogen and oxygen in dependence on parameters of crystallisation were verified and temperature of melting of this alloy was measured on DTA analyser.

Key words: γ-TiAl, titanium aluminide, directional solidification, directional crystallisation, microstructure.

1. Introduction

Duplex γ-TiAl intermetallic alloys are characterised by favourable properties, thanks to which their research and development is currently being developed more and more. They are considered to be the most interesting materials for high-temperature applications in aerospace and automotive industries, such as blades high-pressure compressors of aircraft engines, blades of low-pressure stage of combustions turbines, diffusers, exhaust valves and action wheels of turbo blowers. Attractiveness of this alloy consists particularly in its low density (ρ~3800 kg/m^3), high specific strength, high specific rigidity, high-temperature strength and resistance to oxidation [1].

The following microstructures can occur in the alloy:

- fully lamellar
- almost lamellar
- duplex
- almost gamma [2].

Out of many microstructures that can be developed in Ti-Al alloys, the fully lamellar or almost lamellar microstructure consisting of the γ-phases (TiAl) and α_2 (Ti_3Al) is very promising for structural use. This structure offers higher resistance to creep, higher resistance to brittle fracture and higher fracture strength than duplex structure [2,3].

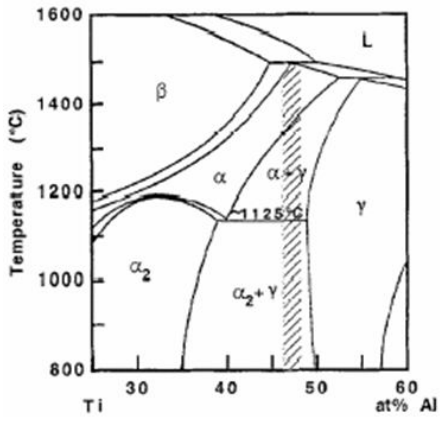

Fig.1 Central part of the binary diagram Ti-Al. Area of duplex alloys is marked.

Binary phase diagram (Fig.1), proposed by McCullough, is used as basic type for explanation of phase transformation in investigated alloys. According to this phase diagram,

solidification of investigated alloys occurs in principle from the area α, which corresponds to hexagonal phase of structure of the types A3. Disintegration of α-phase during the following cooling offers possibilities for forming of two arranged phases, γ (TiAl) and α_2 (Ti_3Al). γ-phase corresponds to tetragonally arranged phase of structure of the type $L1_0$ with ratio *c/a* of approximately 1,02. α_2-phase results from reaction of arrangement of the α-phase and it corresponds to hexagonal structure of the type DO_{19} .

Duplex lamellar structure is formed in the range specified by this reaction:

$$\alpha \rightarrow \alpha+\gamma \rightarrow \alpha_2+\gamma \text{ [4].}$$

Resulting microstructure can be influenced by chemical composition, heat treatment and finally also by directional crystallisation. Directional crystallisation is originally a refining method, based on various effective distribution coefficients of admixtures in the basic matter.

One of possibility manners of affecting the structure and thus also properties of titanium alloys is control of their crystallisation process - directional crystallisation, which enables influencing of structure and of properties of final product by speed of crystallisation. Hence comes the objective of this article, dealing with evaluation of impact of various speeds of directional crystallisation on structure, oxygen and nitrogen contents, formation of inclusions of ceramic particles and interaction of the melt material of fusion crucible.

Development of technology of directional crystallisation was motivated by an effort to control morphology of grain boundaries in order to improve resistance to fracture. This technology was successfully applied in production of turbine blades with columnar or mono-crystalline structure. Process of modification of structure at crystallisation can be performed in two ways. The first method is zonal melting, which is method when only one defined part of the ingot is melted, i.e. narrow zone with given width. Melted zone travels through the ingot and it has two boundaries: (I) smelting front and (II) solidification front, where crystallisation occurs. The second method is Bridgman's method. Its principle is that phase boundary crystal-melt is slowly passing through melted ingot. This is realised with melt contained in ceramic mold. This affects purity of final product, since particularly in case of titanium alloys there occurs, due to their high reactivity, a contamination of the alloy by ceramic mold [5,6,7].

2. Experiment

2.1 Preparation of the alloy

Objective of the experiment is evaluation of impact of directional crystallisation on final microstructure, and determination of how various reaction times influence oxygen and nitrogen contents in the alloy and formation of Al_2O_3 based inclusions from ceramic particles during reaction.

The selected alloy Ti-46Al-5Nb-1W (at%) was prepared in laboratory conditions of the department of non-ferrous metals, refining and recycling. The alloy was at the first stage plasma re-melted in plasma furnace. Charge was put into the copper mold, which is cooled by water flowing at high speed. Advantagaes of this method of melting (heating) is the fact that the alloy is not contaminated by material of fusion crucible. Low homogeneity of the alloy can be considered as main disadvantage of this method. Composition of ingot changed significantly along its cross-section. After smelting of certain zone by plasma burner it is intensively cooled by water, which passes through the copper tray. This causes formation of non-homogenous structure. It is possible to ensure higher homogeneity by longer time of melting or by subsequent re-melting in another type of furnace.

As the second stage of metallurgical preparation were used melting in vacuum-induction furnace followed by casting of samples under argon atmosphere. Melting in induction furnace was necessary in order to obtain higher homogeneity of the alloy. Eddy currents mix intensively melted metal and ensure thus homogenous distribution of alloying elements. Induction melting enables better temperature equalisation than other methods of melting. Corundum crucible for melting was chosen. Ingots were re-melted in high-frequency vacuum-induction furnace. After melting of charge the melt was cast into graphite ingot molds. Castings had form of bars with diameter 10 mm and length 100 mm.

2.2 Directional crystallisation

Directional crystallisation was made in the device of the Bridgman's type. Top oxidic layer was removed from the sample by turning and the sample was then inserted into the ceramic tube Luxal 203. Directional crystallisation was realised under protective argon atmosphere. Argon purity was 4N5. The device consists of heating part of furnace and movable equipment. Tube containing melt is fixed to this movable equipment In dependence on selected speed of crystallisation this equipment pulls out the tube with melt at a constant speed from hot zone through water cooled copper mold and ensures thus the required crystallisation of the melt in accordance with pre-set parameters. We chose for the alloy Ti-46Al-5Nb-1W the melting temperature T_M=1650°C. Dwell at this temperature was 900 s and then shifting began. We chose for shifting five constant speeds, 20 mm/h, 50 mm/h, 100 mm/h, 200 mm/h and 425 mm/h. After removal of tubes with crystallised melt from the furnace space the samples were taken out and cut longitudinally by saw with carbide disc. The samples were then sealed with Dentacryl resin and prepared by grinding and polishing for metallographic observation. Composition of the etching agent used was: HNO_3, HF and distilled water. Etching time was from 5 to 10 seconds. Observation of structure was performed on optical metallografic microscope Olympus GX-51.

2.3 Analyses of gases, oxygen, nitrogen.

For envisaged high-temperature applications the critical point is contents of gases in material. It can influence very significantly mechanical properties. That's why measurement of gas contents in samples was included. The samples were cut to dimensions of approx. 4x4x5 mm. Analyses were performed by Division of testing laboratories at VÚHŽ Dobrá.

2.4 Differential thermal analysis

DTA (differential thermal analysis) is a method used for investigation of properties of substances based on exact measurement of temperature difference of investigated and reference sample during heating or cooling. Measurement of temperature of melt was made at the department of physical chemistry and theory of technological processes with use of experimental equipment SETARAM SETSYS 18_{TM}.

Differential thermal analysis (DTA) is dynamic temperature analytical method, at which there are observed temperature effects of investigated sample, accompanying its physical or chemical changes at its continuous linear heating or cooling. This method is used for measurement of temperature differences between investigated sample and reference sample, which occur during their simultaneous heating, which is linear function of time. Area of peak at

heating indicates amount of heat absorbed by the sample, at cooling there occurs on the contrary delivery of heat from sample to its environment, surface of peak represents amount of transferred heat. Apart from determination of temperatures of phase transformations (plotting of phase diagram) it is possible with use of enthalpy of transformations to determine from the measured peaks also activation energy necessary for realisation of the given transformation [8].

3. Results

Pictures of microstructures of individual samples are given in Fig.2 and 3. They were taken from longitudinal sections of samples. It is obvious from these photos that directional crystallisation has substantial impact on microstructure. Microstructure of samples depends on such parameters as speed of crystallisation, with which time of reaction is connected, and temperature gradient in the melt. At the process of directional crystallisation we are able to control crystallisation of the sample by selecting speed of crystallisation.

Fig.2 Lamellar microstructure of the sample of alloy Ti-46Al-5Nb-1W (at %). a) speed of crystallisation 20 mm/h. b) speed of crystallisation 50 mm/h.

Temperature gradient in the melt remains the same all the time. This means that differences, which can be seen on pictures, reflect various speeds of crystallisation. Microstructure is in all cases formed by lamellar grains. Lamellas of γ- and α_2- phases alternate in them. Grains are oriented longitudinally in direction of heat removal. Lamellas in them are oriented in direction predominantly perpendicular or almost perpendicular to this direction. Structure became more refined with increasing speed of crystallisation (in the interval from 20 to 425 mm/h), see Fig.2a), b) and 3a), b), c).

In structure there are visible also dark particles, Fig.3 d). These are ceramic particles Al_2O_3 (determined by energy dispersion micro-analyser EDAX), which resulted from reaction of the melt with corundum tube during crystallisation. The difference between density of particles Al_2O_3 and density of the melt Ti-46Al-5Nb-1W is imperceptible, that's why these particles float in the melt. Thanks to this the moving boundary solidus-liquidus ensured a uniform distribution of these particles. The differences of the shape of particles are also perceptible. There occur particles of latticed shape, fine particles and coarse particles of irregular shape, see Fig.3 d). Large latticed particles occurred in the sample with speed of crystallisation 20 mm/h. Shorter reaction time caused its different shape and smaller size. Number of particles and their size

decreased with increasing speed of crystallisation. This is caused by shorter reaction time between the melt and ceramic tube. Particles occurred at higher speeds of crystallisation in a fine-grin form, and they created sporadically clusters.

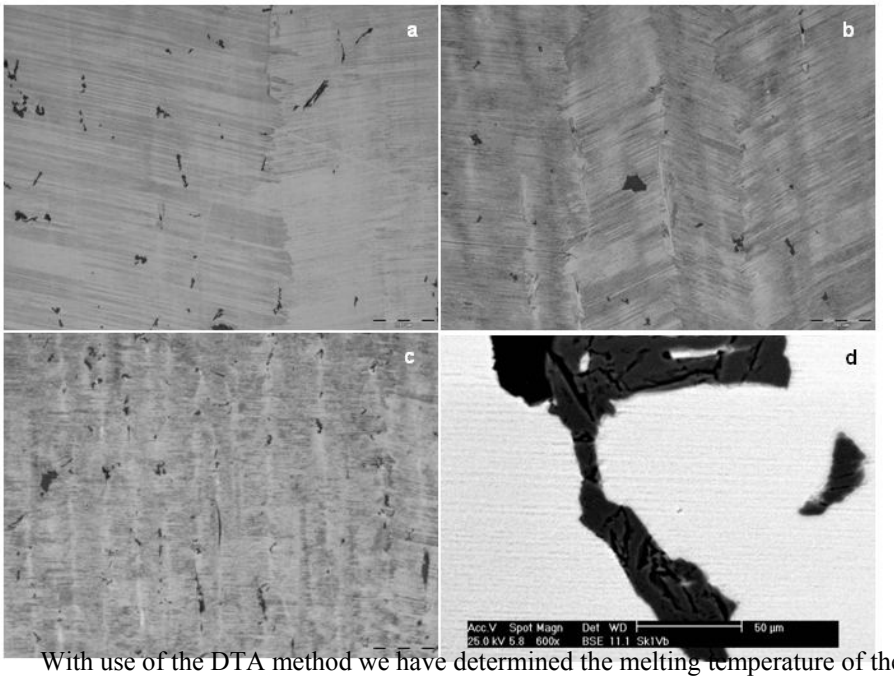

Fig.3 Lamellar microstructure of the sample of alloy Ti-46Al-5Nb-1W (at %). a) speed of crystallisation 100 mm/h. b) speed of crystallisation 200 mm/h. c) speed of crystallisation 425 mm/h. d) ceramic particles Al_2O_3, magnification 600x.

With use of the DTA method we have determined the melting temperature of the alloy Ti-46Al-5Nb-

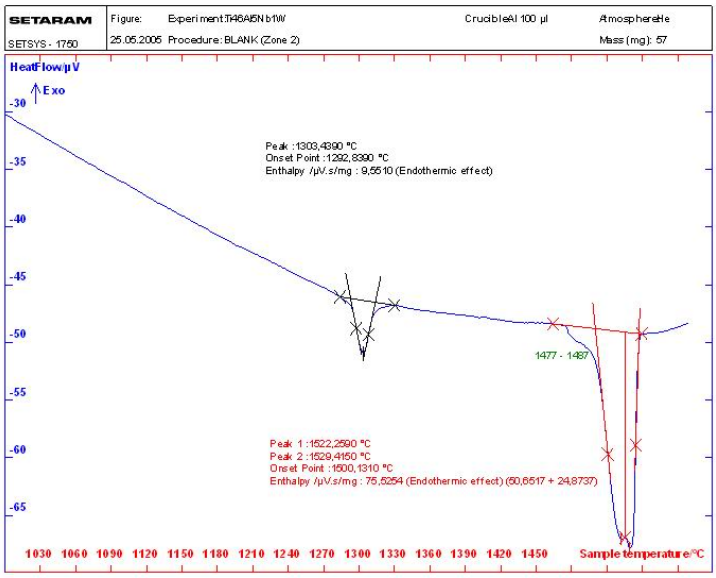

Fig.4 Final DTA diagram of the alloy Ti-46Al-5Nb-1W.

With use of the DTA method we have determined the melting temperature of the alloy Ti-46Al-5Nb-1W. It follows from the Fig. 4, that this reading is not unequivocal due to complexity of diagram. Melting temperature falls within the range from 1522°C to 1529°C.

Determination of contents of gases in the samples after directional crystallisation was made at the VÚHŽ Dobrá on the analyser LECO TC. The results can be seen on diagrams, see Figures 5, 6. It is obvious from these results that contents of gas in the sample depend on duration of time, during which the melt was exposed to reaction with ceramic tube made of Al_2O_3. It is known from literature that oxygen contents have influence on resulting mechanical properties. Higher values of oxygen content result in lower toughness and higher strength of lamellar alloy TiAl, since higher contents of oxygen reduces mobility of dislocations.

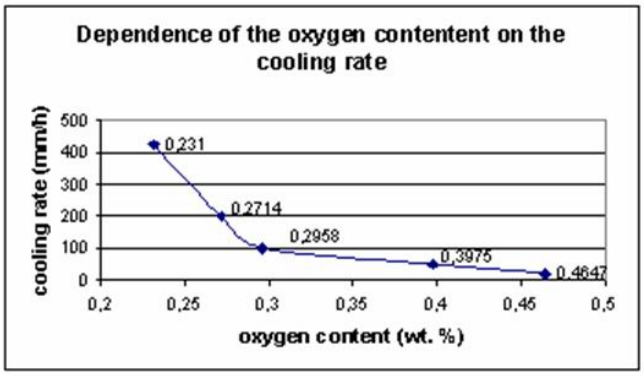

Fig.5 Dependence of oxygen contents on speed of crystallisation of the alloy Ti-46Al-5Nb-1W .

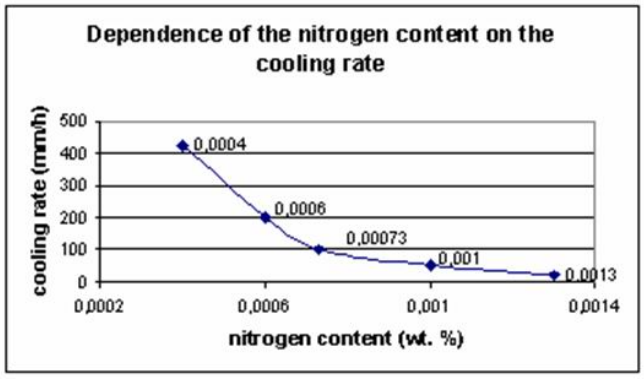

Fig.6 Dependence of nitrogen contents on speed of crystallisation of the alloy Ti-46Al-5Nb-1W.

4. Conclusion

This work was devoted to evaluation of impact of speed of directional crystallisation on final structure of the samples made of the alloy Ti-46Al-5Nb-1W (at%). It is obvious from the obtained results that speed of crystallisation significantly influences the structure, since higher speed of crystallisation causes more refined microstructure. The speeds used at experiment varied within the interval from 20 mm/h to 425 mm/h. Microstructure is formed by lamellar grains, which are oriented in parallel with direction of heat removal. In lamellar grains

there alternate lamellas of the γ-phases (TiAl) and α_2 (Ti_3Al). These are oriented predominantly perpendicularly or almost perpendicularly to the direction of heat removal.

In structure there can be seen particles, which were determined by micro-analyser EDAX as Al_2O_3 particles. They are result of reaction between the melt and ceramic tubes made of Al_2O_3 in the course of directional crystallisation. Their shape and number in the alloy also varied in dependence on speed of directional crystallisation. At longer reaction time (V = 20 mm/h) the particles were of latticed shape. At shorter reaction time the size of particles decreased.

Contents of oxygen and nitrogen were measured in the alloy. The measured values show dependence on time, during which the alloy was in melted condition. The slower was speed of the crystallisation, the higher was contents of oxygen and nitrogen.

Acknowledgements

The presented results were obtained within the frame of solution of the GAČR grant project No. 106/03/0984 „Metallurgical possibilities of modification of properties of inter-metallic compounds of the system Ti-Al and Ti-Al-Me“ and of the research project MSM 6198910013 „Processes of preparation and properties of highly pure and structural defined materials”.

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