Columnar to Equiaxed Transition in Peritectic TiAl Based Alloy Studied by a Power-Down Technique

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Abstract. Columnar to equiaxed transition (CET) was studied in a peritectic TiAl-based alloy with chemical composition Ti-45.1Al-4.9Nb-0.25C-0.2B (at.%). Solidification experiments were conducted in a Bridgman-type apparatus using cylindrical moulds made of high-purity Y_2O_3. The methodology containing appropriate etching and observations under flat light in stereo-microscope was used to identify the morphology of primary β phase grains and position of CET in the samples. All samples prepared by power down-technique showed sharp CET. The position of the CET measured from the beginning of the sample depends on the applied cooling rate and increases from approximately 65 mm to 115 mm by decreasing cooling rate from 50 to 15 K/min. Based on terrestrial experiments, the future work focused on microgravity and hypergravity CET experiments and numerical modeling is proposed. A Bridgman furnace front tracking method will be applied in future work to complement the experimental results here as part of the European Space Agency GRADECET programme. This modeling will input directly into planned microgravity and hypergravity CET experiments.

Introduction

In the recent years intermetallic TiAl-based alloys have been used for demanding applications in power engineering, aircraft and automotive industry, especially for processing of low pressure turbine blades for stationary gas turbines and aircraft engines and turbocharger wheels for petrol and diesel engines. Microstructure of large cast components from TiAl-based alloys usually consists of columnar grains growing from the mould surface and equiaxed grains formed in central zone of the castings [1, 2]. Besides from large anisotropy of mechanical properties resulting from such grain structure, columnar to equiaxed transition (CET) is also connected with formation of casting defects. The CET is connected to a change of microsegregation severity of alloying elements. Martorano and Capocchi [3] reported for copper alloys microsegregation severity in columnar dendrites than in equiaxed ones due to more extended homogenization of the columnar structure.

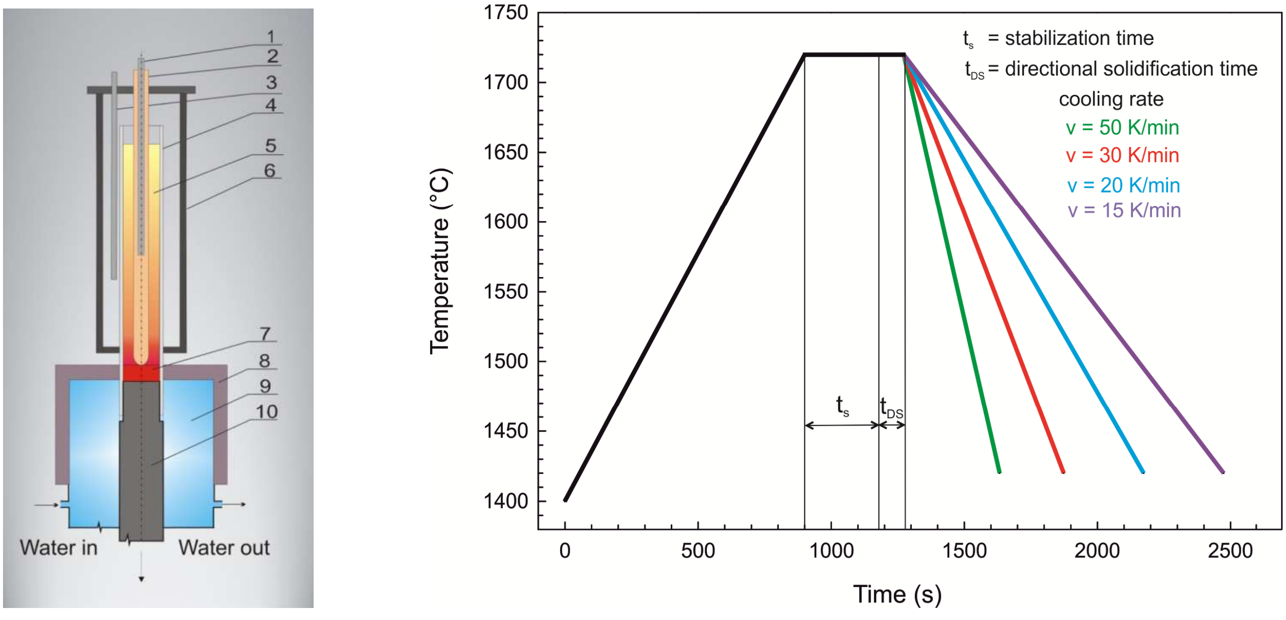
Various mechanisms and models have been proposed to explain CET during solidification of alloys [4-7]. There is a general consensus that the CET occurs when the moving front of columnar grains is blocked by equiaxed grains growing in the undercooled liquid ahead of this front, i.e. if the equiaxed grains are sufficient in size or number to arrest columnar grain growth. Regardless of the blocking mechanisms, experiments and theoretical models have shown that the CET is significantly affected by the number of equiaxed grains and the nucleation undercooling.

The majority of TiAl-based alloys containing about 44-45 at.% of Al and 5-10 at.% of Nb solidify through β-phase (Ti-based solid solution with cubic crystal structure), which transforms to α-phase (Ti-based solid solution with hexagonal crystal structure) during cooling. Formation of the α-phase from the β-phase is a complex problem, because the α-phase can be formed either through peritectic reaction and transformation in peritectic type alloys, or by solid state transformation from the single β-phase, resulting in the formation of different crystallographic orientation variants of the α-phase. The effect of peritectic reaction, which is part of the solidification path, is rather harmful for the grain refining process [8]. Therefore, the basic ternary systems are further alloyed by carbon and boron, which promote grain refinement. Low addition of boron increases the rate of heterogeneous nucleation of the α-phase during $\beta \rightarrow \alpha$ transformation leading to fine grain structure of the alloy. Nb, B, and C increase the stability of α grains against their growth on passing through the α single-phase field during cooling [9]. In addition, small amount of C improves high-temperature tensile and creep strength of these alloys [9].

The aim of this paper is to study CET in peritectic TiAl based alloy with nominal composition Ti-44.5Al-5Nb-0.2B-0.2C (at.%) alloy by power-down technique using Bridgman type apparatus at controlled cooling rates. Future complementary modeling activity is proposed.

Experimental Procedure

The intermetallic alloy, with the chemical composition Ti-45.1Al-4.9Nb-0.25B-0.2C (at.%) and oxygen content of 420 wtppm, was supplied in the form of vacuum arc remelted conical ingot with a diameter changing from 35 to 60 mm and a length of 310 mm. The ingot was cut to smaller blocks using electro spark machining, which were lathe machined to a diameter of 10 mm and a length of 150 mm, and put in dense cylindrical Y_2O_3 moulds (purity of 99.5%) with a diameter of 10/15 (inside/outside diameter in mm) and length of 170 mm. Power down experiments were performed in a modified Bridgman type apparatus described elsewhere [10, 11] under an argon atmosphere. Before solidification the vacuum chamber of the apparatus was evacuated to a pressure of 3 Pa, flushed with argon (purity 99.9995%) six times and then backfilled with argon at a pressure of 10 kPa, which was held constant during melting and solidification. The power down experiment consisted of: (i) heating of the sample to a temperature of 1993 K at a heating rate of 0.355 Ks^{-1}, (ii) stabilization at the temperature of 1993 K for 300 s, (iii) partial displacement of the sample from the



(a) (b)

Fig. 1. (a) Schematic of the Bridgman type apparatus: 1 - measuring thermocouple, 2 - ceramic tube, 3 - control thermocouple, 4 - Y_2O_3 crucible, 5 - solidifying sample, 6 - resistance heater, 7 - solid part of the sample, 8 - crystallizer, 9 - water bath, 10 - withdrawal rod. (b) Power-down experiments at four different cooling rates.

hot zone of the furnace into the crystallizer at a constant withdrawal rate of 2.78×10^{-4} ms^{-1} to a length of 20 mm, (iv) temperature decrease from 1993 to 1693 K at constant cooling rates ranging from 15 to 50 K/min and (v) furnace cooling of the sample from 1693 K to room temperature. Microstructural investigations were performed by optical microscopy (OM) and backscattered scanning electron microscopy (BSEM).

The samples for optical microscopy were chemically etched in a reagent of 100 ml H_2O, 10 ml HNO_3 and 3 ml HF. Analysis of grain structure of CET samples consisted of polishing of the samples and etching them in Kroll's reagent for subsequent observation and imaging under the stereo-microscope using (a) grazing light at about 45° angle and (b) flat light at about 0° with the light beam parallel to the sample surface. All images were taken at 8x magnification. Under grazing light, the samples were rotated successively into various positions, thus bringing individual grains, one by one, into optimum reflection conditions (white). The contour of the individual grains was extracted pretty accurately, one after the other, from the appropriate images. Under flat light, the dendrite structure of the primary β(Ti) phase was revealed with sufficient quality.

Results

Fig. 2 shows the typical macrostructure of the samples prepared by power down technique at a cooling rate of 30 K/min. The macrostructure consists of four different zones designated as initial, columnar, coarse equiaxed and radial. The initial zone with an average length of 12 mm represents non-melted part of the sample composed of equiaxed grains. The columnar zone is composed of columnar grains which were formed by the initial 20 mm of directional solidification at a growth rate of $V = 2.78\times10^{-4}$ ms^{-1} and an approximate temperature gradient in liquid at the solid liquid interface of $G_L = 5.5\times10^3$ Km^{-1} followed by the columnar grains formed during power-down cycle at a constant cooling rate of 30 K/min. The coarse equiaxed zone is composed of large equiaxed grains with various crystallographic orientations formed in the liquid during solidification. The radial zone is composed of the grains nucleating at the crucible wall a growing in a radial direction towards the centre of the sample during cooling.

Fig. 3 shows grain structure of the CET sample prepared at a cooling rate of 30 K/min. Under grazing light, the resulting map shows the α(Ti) grains, i.e. the grains formed through peritectic reaction/transformation in the deep mushy zone of the sample, see Fig. 3a. Under flat light, the dendrite structure of the primary β(Ti) phase grains can be clearly seen, as shown in Fig. 3b. It is

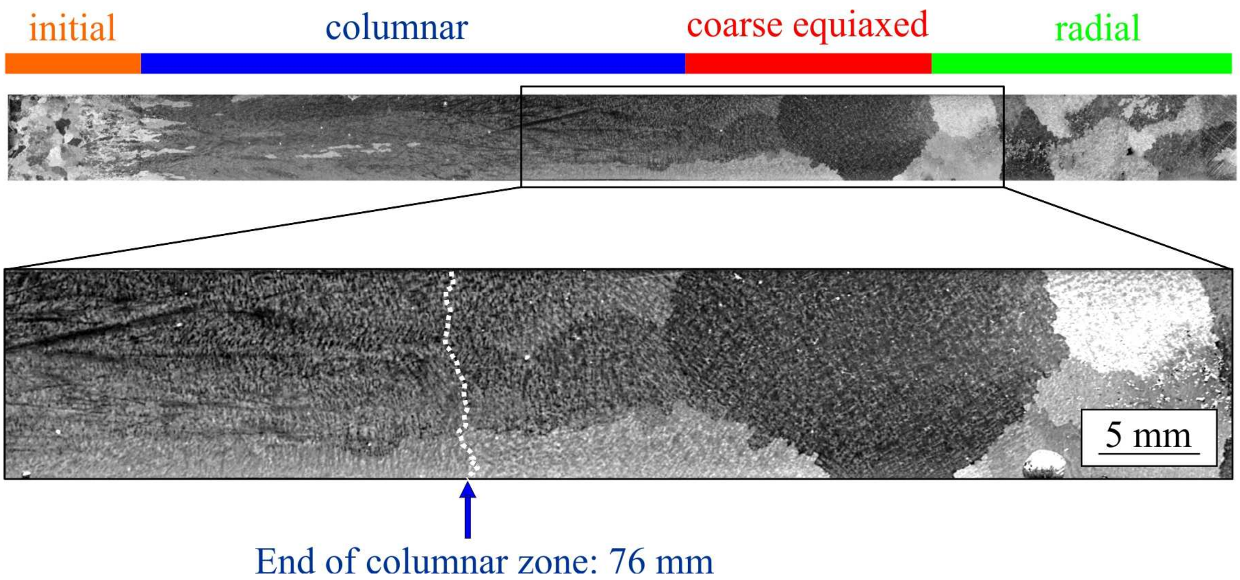


Fig. 2. Macrostructure of longitudinal section of CET sample prepared power-down technique at a constant cooling rate of 30 K/min.

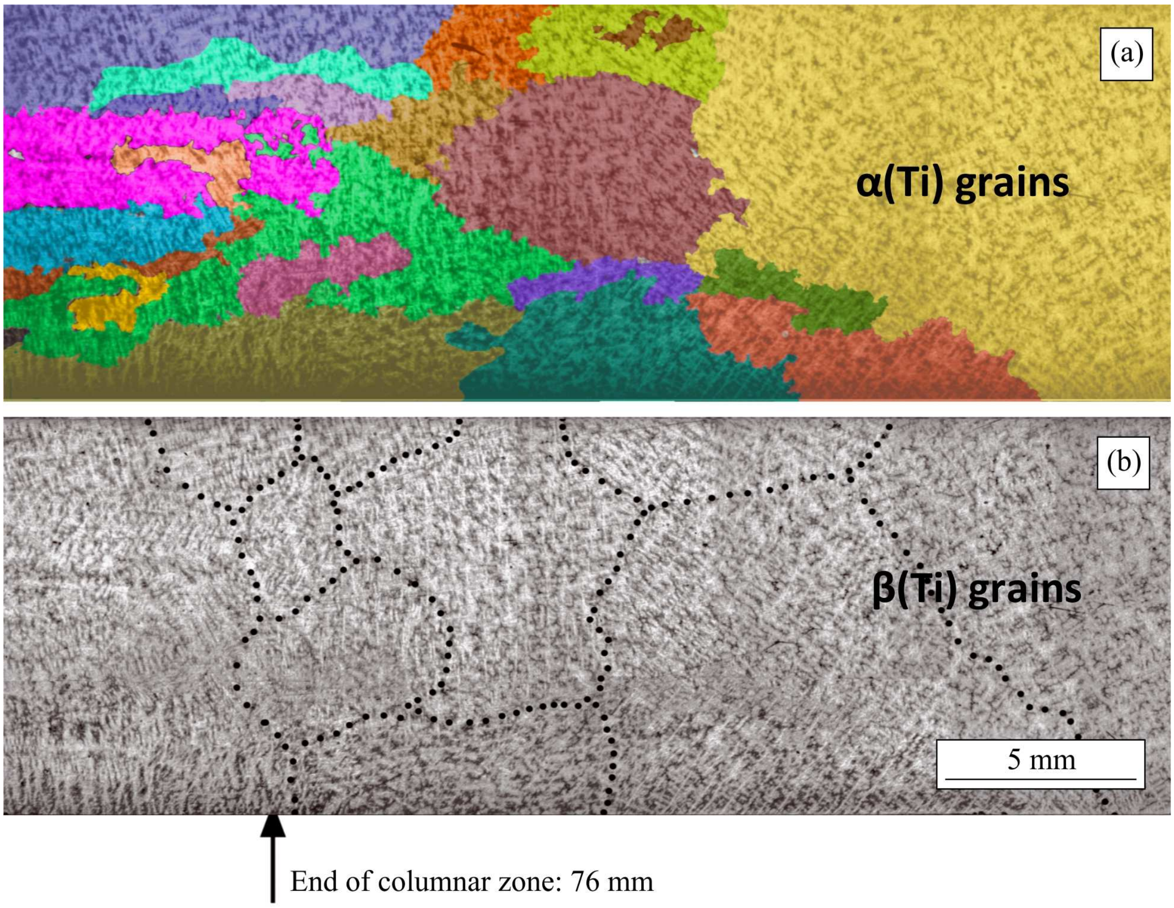


Fig. 3. Grain structure of a CET sample prepared by power-down technique at a cooling rate of 30 K/min: (a) columnar and equiaxed α(Ti) grains in the vicinity of CET, (b) columnar and equiaxed β(Ti) grains in the vicinity of the CET.

clear that the α grain structure of the sample differs significantly from that of the primary β phase grains. In addition, apparently mixed region between the columnar and equiaxed zones composed of columnar and equiaxed grains is missing in the structure composed of the primary β(Ti) phase grains. The studied alloy shows sharp CET when the primary grain structure is correctly analyzed.
All samples prepared by power down-technique showed clear CET. The position of the CET measured from the beginning of the sample depends on the cooling rate and achieves 65, 76, 85 and 115 mm for the cooling rate of 50, 30, 20 and 15 K/min, respectively.

Discussion

During directional growth in a positive temperature gradient – due to the anisotropy of properties such as solid-liquid interface energy and growth kinetics – the dendrites grow in the crystallographic direction that is the closest to the direction of heat flow. In the case of the β primary solidification phase, the columnar dendrites grow in [001] crystallographic direction. Such preferred crystallographic growth direction of the β phase with bcc crystal structure determines growth directions of the {011} planes, which grow with an angle of 0° or 45° to the growth direction. When the β phase transforms to the α phase, through the solid phase transformation, the crystallographic orientation relationship $(110)_\beta || (0001)_\alpha$ is maintained between coexisting phases. In peritectic TiAl-based alloys, the α phase is formed through peritectic reaction/transformation of the type: liquid + bcc β(Ti) → hcp α(Ti). This means that in our case the grain morphology revealed by the etching is not the one of the β primary solidification phase, but one of the α phase formed by solid state transformation and peritectic reaction/transformation. Hence, the grain structure shown in Fig. 3a has no direct relationship to the primary dendrite grain structure and cannot be related to the mechanisms and position of CET in the samples. Therefore, it is necessary to reveal the original primary β phase grain structure to assess correctly the CET phenomena in peritectic TiAl-based alloys. The peritectic α phase forms subgrain structure within large α grains indicating the growth at unconstrained conditions. However, it is still not clear whether the nucleation and growth of the α phase is sensitive to the local temperature gradient.

Future Work

The GRADECET project (GRAvity Dependence of the CET in Peritectic TiAl alloys) aims to study the influence of gravity on the CET formation for the peritectic TiAl based alloy. The gravity scenarios to be considered include terrestrial gravity (1 g), microgravity (approx. 10^{-6} g), and hyper gravity (> 1g to 20 g).

Planned Power-Down Experiments. The CET will be investigated in the same alloy at different cooling rates. Both higher and lower cooling rates will be applied to the alloy. This planned research partially represents the terrestrial gravity study.

Planned Modeling Activity. The Bridgman furnace, used in the experiments discussed here, has been the focus of a study [12] to measure the heat transfer conditions occurring in the Ti-Al samples. Experimental temperature data, measured along the axis of a stationary sample, was used to estimate axial and radial heat flux from the sample and to estimate heat transfer coefficients in hot and cold regions of the furnace. Subsequently, the estimated heat transfer coefficient will be used to model the power down experiments described herein. The Bridgman furnace front tracking model (BFFTM) of Mooney et al. [13] will be adapted for this purpose. The focus of the modeling exercise will be to perform a CET analysis [14] and to generate a Hunt plot [4] for each of the power down experiments.

Microgravity Experiment. Plans are underway to develop a furnace that will be used in the MAXUS 9 sounding rocket campaign. The sounding rocket, to be launched by the Swedish Space Corporation, will provide approximately 10 minutes of high quality microgravity. This activity will be a continuation of the microgravity solidification study performed on a similar alloy on the MAXUS 8 sounding rocket [15].

Hyper Gravity Experiments. The furnace module that will be used for the microgravity experiments will also be installed onto the Large Diameter Centrifuge (LDC), which resides at the ESTEC facility in the Netherlands [16]. The LDC has the capacity to provide increased gravity levels up to and including 20 g. Solidification experiments for the Ti-Al will be performed.

Conclusions

Columnar to equiaxed transition (CET) was studied in a peritectic TiAl-based alloy with the chemical composition Ti-45.1Al-4.9Nb-0.25C-0.2B (at.%). All samples prepared by power down-technique in Bridgman type apparatus showed sharp CET when the primary β phase grain structure was revealed and analyzed correctly. The analysis of the α phase grain structure formed by solid phase transformation of the primary β phase and peritectic reaction/transformation indicate an apparent mixed zone of columnar and equiaxed grains between the columnar and equiaxed zones of the samples. The position of the CET, measured from the cold end of the sample, depends on the applied cooling rate and increases from approximately 65 to 115 mm by decreasing cooling rate from 50 to 15 K/min. A Bridgman furnace front tracking method will be applied in future work to complement the experimental results here as part of the European Space Agency GRADECET programme. This modeling will input directly into other planned microgravity and hypergravity CET experiments under the GRADECET project.

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