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Acta Materialia 60 (2012) 498-506



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### Microstructural control of TiAl-Nb alloys by directional solidification

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> Received 15 August 2011; received in revised form 4 October 2011; accepted 5 October 2011 Available online 22 November 2011

#### Abstract

The lamellar microstructure of TiAl–Nb alloys with and without low boron additions is controlled using double directional solidification (DS). In alloys without the addition of boron, the  $\beta$  phase is seeded during double DS. Complete peritectic transformation occurs in both the dendritic and interdendritic regions, which can lead to the successful alignment of both the high-temperature  $\alpha$  phase and the lamellar microstructures. Well-aligned lamellar microstructures can be easily achieved if the alloy composition is close to the peritectic point on the hypo-peritectic side. In alloys with low boron additions, however, the competitive growth of the  $\alpha$  phase breaks the continuity of the lamellar microstructure in the region ahead of stable growth, which finally results in columnar grain coarsening and unsuccessful alignment of the lamellar microstructures.

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Keywords: Titanium aluminides; Directional solidification; Peritectic solidification; Crystal growth; Grain refining

#### 1. Introduction

As a substitute for nickel-based superalloys, TiAl–Nb alloys are potential candidates for new-generation aeroengine applications due to their low density, excellent high-temperature mechanical properties and good oxidation resistance [1–4]. However, their poor room temperature (RT) ductility and fracture toughness limit their engineering applications. In terms of creep resistance and fracture toughness, TiAl alloys with a fully lamellar ( $\alpha_2 + \gamma$ ) microstructure show superior mechanical properties compared with alloys composed of other microstructures, especially when the lamellar microstructure is aligned parallel to the loading axis. Therefore, directional solidification (DS) was developed to control the lamellar microstructures consisting of TiAl ( $\gamma$ ) and Ti<sub>3</sub>Al ( $\alpha_2$ ) in TiAl alloys [5–8].

A microstructure consisting of refined columnar grains or a single lamellar grain (polysynthetically twinned or PST crystals) is expected when using DS, because it eliminates transverse grain boundaries and improves the RT and high-temperature mechanical properties of TiAl alloys. Grain refinement can also be achieved in the alloys with low boron additions [9–11]; however, very few research findings have been reported on directionally solidified microstructures in TiAl alloys with low boron additions. PST crystals usually grow from an appropriate seed, such as the seed of Ti–43 Al–3 Si alloys [6,12–14], but the seeding route seems impractical for forming directionally solidified ingots with a near-net shape, particularly for ingots used in turbocharger wheels or engine valves.

Recently, we were able to successfully control the lamellar orientation in TiAl alloys solidified through their primary  $\beta$  phase by using the double Bridgman DS technique [15]. DS was performed on the alloys twice, and the sample was inverted before the second DS step. The single lamellar grain, produced in the upper part of the directionally solidified ingot during the first DS process, served as a seed that controlled the lamellar orientation of the initial stage of the second DS process. A low temperature gradient was required and dendritic growth morphology was developed

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<sup>1359-6454/\$36.00</sup>  $\odot$  2011 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved. doi:10.1016/j.actamat.2011.10.009

during DS. The main benefit of the seeding technique is that the seed has the same composition as the master ingot.

In this study, double DS is performed on the TiAl–Nb alloys with or without low boron addition to control the lamellar microstructures of the columnar grains or to produce a single lamellar grain, respectively. The feasibility and effectiveness of lamellar microstructure control in these alloys are investigated. The purpose of this paper is to explain the seeding mechanism of double DS in the primary  $\beta$  TiAl alloys. Additionally, the optimal alloy composition to obtain alignment of the lamellar microstructure using double DS is discussed.

#### 2. Experimental details

Ti-46 Al-5 Nb and Ti-45 Al-6 Nb-0.3 B alloys were supplied in the form of two cast cylindrical ingots, which were produced using induction skull melting (ISM). The ingots had exact chemical compositions of Ti-46.20 Al-4.78 Nb or Ti-44.64 Al-5.91 Nb-0.31 B (at.%), and their oxygen contents were less than 600 ppm. Two sets of bars that were 5.5 mm in diameter and 80 mm in length were cut from each ingot. Each bar was placed into an alumina crucible with a yttria mold isolating the alloy from the crucible. The Bridgman type apparatus [16] was employed to produce the directionally solidified bars under the protection of 380 Pa highpurity argon. After heating to the desired temperature and holding for 30 min, the bar was directionally solidified under a temperature gradient of  $G = 5 \times 10^3 \text{ K m}^{-1}$  and at a growth rate of 10-50  $\mu$ m s<sup>-1</sup>. The surface layers of the directionally solidified bars were removed by machining. After the first DS step, one set of samples was turned by 180° lengthwise and the DS was repeated under the same conditions as the first DS process. The directionally solidified bars were sectioned longitudinally and transversely and then polished using standard metallographic techniques. The microstructure and composition were analyzed by field-emission scanning electron microscopy (FESEM) using a Zeiss SUPRA 55 in back-scattered electron (BSE) imaging mode and equipped with an energy dispersive X-ray spectroscopy (EDS) detector. After etching in a solution of 5 ml HF, 10 ml HNO<sub>3</sub> and 85 ml H<sub>2</sub>O, optical microscopy (OM) and SEM were used to characterize the macrostructures and the lamellar microstructures. The lamellar structures were further examined using transmission electron microscopy (TEM), and the textures of the longitudinal microstructures in different regions were determined using X-ray diffraction techniques.

#### 3. Results and discussion

## 3.1. Microstructural control in TiAl–Nb alloys without boron addition

Fig. 1a and b shows the longitudinal macrostructures of the directionally solidified Ti–46 Al–5 Nb bars after the single and the double DS processes, respectively, at a growth rate of  $30 \ \mu m \ s^{-1}$ . As observed in Fig. 1a, an annealing region (region A) consisting of equiaxed grains is observed at the lower part of the bar after the single DS process. The alloys are unmelted but annealed in this region during DS. The columnar grains are found in the DS region (region B). Some grains continue to grow from region A to region B. The columnar grains are mostly at inclined angles to the growth direction, and the ones with suitable crystallographic orientations can significantly grow in the columnar region. Only one grain can be produced in the upper part of the bar. After the second growth, however, only one grain is observed in region A and B in Fig. 1b.

Fig. 2 presents the optical microstructures of the directionally solidified Ti–46 Al–5 Nb bar after double DS at a growth rate of  $30 \ \mu m \ s^{-1}$ . The optical microstructure in the longitudinal section of the bar is shown in Fig. 2a. Fig. 2b and d shows the magnified optical microstructures in the areas marked in Fig. 2a. Fig. 3c shows the typical optical microstructure in the transverse section of the bar. Only one lamellar grain is observed in the transverse section. In the annealing region, the lamellar boundary is inclined at ~30° to the growth direction. In the whole DS region, however, the lamellar boundaries are aligned parallel to the growth direction. The turning position of the lamellar orientation is almost at the same cross-section of the sample.

Fig. 3 shows the BSE microstructures in the transverse section and in the area marked in Fig. 2b. Some  $Y_2O_3$  particles in white contrast can be observed in Fig. 3. The particles were incorporated into the melt due to thermal impaction and convection applied to the yttria mold during the DS processes [17]. Due to the partition of solute, K > 1, Al is rejected into the interdendritic regions. Al segregation, in a dark-grey contrast, can be formed in the interdendritic regions [16]. Partial segregation with the morphology



Fig. 1. Longitudinal macrostructure of the directionally solidified Ti–46 Al–5 Nb bars after the single (a) and double (b) DS processes at a growth rate of  $30 \,\mu m \, s^{-1}$ .

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