

Microstructure control and mechanical properties of directionally solidified TiAl–Nb alloys

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Abstract: The double directional solidification (DS) technique was developed to control the lamellar microstructures in primary β TiAl–Nb alloys. Polysynthetically twinned (PST) crystals with lamellar boundaries parallel to or inclined 45° to the growth direction were achieved due to the complete peritectic transformation during directional solidification of the alloys with the dendritic solid/liquid interface. The PST crystals with aligned lamellar boundaries only parallel to the growth direction were produced when lamellar grains with lamellar boundaries in the same orientation were seeded by themselves under appropriate growth conditions. Low boron addition is harmful to align the lamellar orientation because of the growth of non-peritectic α phase. Due to the larger yttria particles and boride ribbons in the directionally solidified TiAl–Nb alloys, the tensile plastic elongations of the alloys are only close to 2%.

Key words: titanium aluminides; directional solidification; peritectic transformation; lamellar orientation; polysynthetically twinned crystals; mechanical properties

1 Introduction

TiAl–Nb alloys consisting of fully lamellar ($\alpha_2+\gamma$) microstructures are candidate materials for high-temperature structural applications due to their low density, good oxidation resistance and excellent high-temperature mechanical properties [1,2]. Addition of Nb can significantly improve the oxidation resistance and high-temperature strength, but cause a loss of room-temperature (RT) ductility of the TiAl alloys [3,4]. Low boron addition can refine the solidification microstructure and improve the mechanical properties [5]. Even so, the poor room-temperature ductility and fracture toughness still limit their applications in engineering. The mechanical properties of two-phase lamellar TiAl alloys are dependent on the lamellar orientation relative to the testing axis [6], so the directional solidification (DS) technique is developed to control the lamellar microstructures. By using the technique, columnar grains with aligned lamellar

microstructures [7] even polysynthetically twinned (PST) microstructures can be produced [8], which are very useful to improve the orientated mechanical properties.

Primary β TiAl alloys are important members in engineering. The addition of β -stabilizers, such as Nb, broadens the high-temperature β phase region towards the high Al composition side [9]. The peritectic solidification occurs during the DS process in the primary β TiAl alloys under a lower temperature gradient (G). The peritectic and solid-state transformations result in the directionally solidified TiAl alloys typically consisting of large lamellar grains with lamellar boundaries oriented between 0° and 45° to the growth direction [10]. Previous studies showed that the lamellar orientation in the alloys can be controlled by using an appropriate seed, such as the seed of Ti–43Al–3Si alloys [11]. However, the seeding route seems impractical for the application in engineering because of the different composition between the seed and the master ingot. In order to solve this problem, the full transformation to β phase at the solid/liquid interface [9,12] and the seed of

the similar composition as the precursor ingot were accordingly investigated [13]. Both of them need a higher G , which is usually achieved by a floating zone (FZ) process. Lamellar colonies in different orientations caused by the $\beta \rightarrow \alpha$ solid-state transformation make it almost unfeasible to use.

Recently, a double DS technique was developed, by which DS was performed on the alloys twice and the sample was inverted when the DS was repeated [14]. Using the technique, the lamellar microstructures of the primary β TiAl alloys can be successfully controlled by a Bridgman DS under a lower G . This study reports the progress in controlling the lamellar microstructures of primary β TiAl alloys. The results of recent studies on the DS of TiAl–Nb alloys and the room-temperature mechanical property tests were also summarized.

2 DS experimental methods and double DS technique

In order to remelt the TiAl–Nb alloys in a stable high-temperature environment, Bridgman type apparatus [15] was used to produce directionally solidified alloys. A considerable accurate measurement of G can be taken by a W–Re5/26 thermo-couple with a ceramic protective layer which was moved with the specimen of TiAl–Nb alloy during the DS process. Since the temperature gradient G is defined as a ratio of temperature to displacement, the value of G should be equal to the slope of the temperature curve. The typical temperature curve and the regression analysis results are shown in Fig. 1.

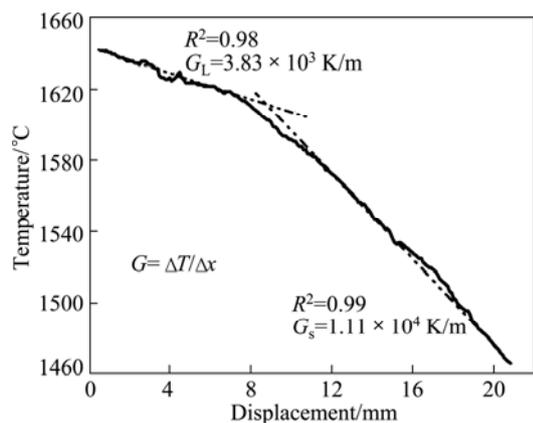


Fig. 1 Typical temperature curve and regression analysis during directional solidification, showing results of temperature gradient measurement by using Ti–45Al–8.5Nb alloys at growth rate of $10 \mu\text{m/s}$

Usually, CaO and Y_2O_3 crucibles [10] were used to produce the directionally solidified TiAl-based castings in order to reduce the reaction between the crucibles and the TiAl melts, but the applications were limited by the easy deliquescence and high hot brittleness, respectively [16]. In this study, yttria-moulded Al_2O_3 crucibles were used in the DS process. As shown in Fig. 2, the

interspace between the TiAl–Nb alloy and the Al_2O_3 crucible was filled with high-purity Y_2O_3 . After being agglomerated, the yttria mould was formed, which isolated the melting from the crucible completely. Irregular-shaped TiAl bars or ingots can be produced in the yttria mould, which possibly makes it very useful in processing compressor and turbine blades. However, long-term interactions between the TiAl–Nb melt and the mould during DS can result in the contamination of the melt by yttria inclusions. The size of the yttria particles decreases with increasing growth rate (v) and decreasing contact time. The size was less than $20 \mu\text{m}$ when v was greater than $30 \mu\text{m/s}$ [17].

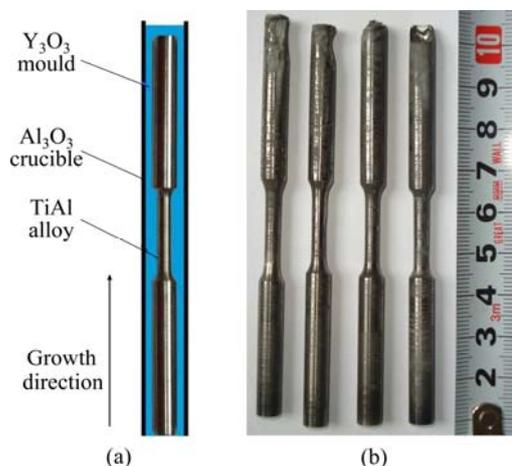


Fig. 2 Photos of yttria-moulded Al_2O_3 crucible (a) and directionally solidified specimens of irregular-shaped TiAl–Nb alloys (b)

The width of the oriented lamellar grain increased with the increase of the distance from the lower part of the directionally solidified ingot after the single DS process of the TiAl alloys [17,18]. In order to make the seeding technique more available, coarse lamellar grains, produced in the upper part of the single directionally-solidified ingot acted as seeds to control the lamellar orientation during the second DS. After the first DS, the samples were turned by 180° and the DS was repeated. No seeds with other compositions were used during the DS process. The double DS technique was very well suited for controlling the lamellar microstructures of the primary β TiAl alloys.

3 Lamellar microstructure control

The lamellar microstructures are produced from parent α -phase following the Blackburn orientation relationship [19] of $(0001)_\alpha // \{111\}_\gamma$ and $\langle 11\bar{2}0 \rangle_\alpha // \langle 110 \rangle_\gamma$, so the control of the lamellar orientation can be accomplished by controlling the orientation of the high-temperature α phase. As indicated by YAMAGUCHI et al [20], the primary β TiAl alloys can be seeded by using

the α -seeding technique. A rotated α -seed crystal with lamellar boundaries aligned parallel to the DS growth direction is placed at the bottom of the crucible. During the DS process, the top part of the seed is melted, and subsequent growth occurs from the seed crystal, causing the orientation of the seed crystal reproduced in the solidifying alloy. Using the conventional α -seeding technique, a planar solid/liquid interface during the seeding DS is required to prevent the α -dendrite growth, which tends to lead the lamellar microstructures oriented perpendicular to the growth direction. The nucleation of the primary β phase can be suppressed, and the interdendritic α phase is seeded when the $L+\beta+\alpha$ three-phase region exists. Using the double DS technique, however, the dendritic solid/liquid interface under a lower G is available to control the lamellar orientation in the following details.

3.1 Lamellar microstructure controlled by complete peritectic transformation

As peritectic solidification occurs at lower G/v ratios, the dendritic structure with peritectic α enveloped primary β dendrite is generated. The peritectic α envelops the β phase by peritectic reaction at the

positions in the growth direction behind the β tips. Peritectic transformation occurs when the properitectic β is isolated by peritectic α . The peritectic α can be thickened by direct precipitation in interdendritic liquid as well as dissolving the primary β dendrite towards its arm. Usually, the β dendrite is partially peritectically transformed into one α grain, and the residual β is subsequently transformed directly into several α grains by the solid-state transformation $\beta \rightarrow \alpha$, which induces the formations of α variants with different orientations according to the Burgers orientation relationship [21]. However, the closely complete peritectic transformation can lead to producing the parent- α phase as a whole in both dendritic and interdendritic regions, which can be aligned accordingly with the lamellar orientation in primary β TiAl alloys [22].

According to St JOHN and HOGAN [23], the peritectic transformation tends to be complete in a peritectic system, such as TiAl, in which the peritectic composition range always includes peritectic point C_p , but the alloying composition and the solidification conditions are crucial to obtain the complete peritectic transformation [22]. Figure 3 presents the BSE microstructures in quenched mushy zones in longitudinal

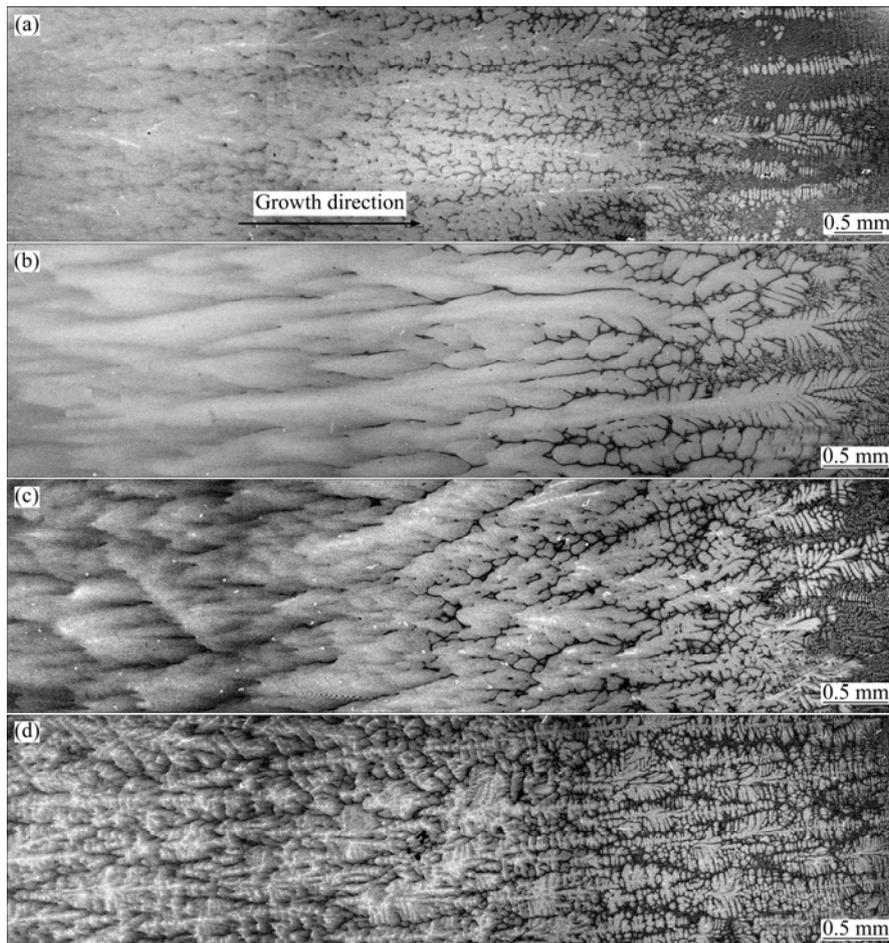


Fig. 3 BSE microstructures in quenched mushy zones in longitudinal sections, showing directly solidified α from liquid in interdendritic regions in Ti-45Al-6Nb-0.3B alloys (a), α -leading solidification at low v (b), complete peritectic transformation in dendritic regions at medium v (c) and incomplete peritectic transformation at high v (d) in Ti-46Al-5Nb alloys

sections of the directionally solidified TiAl–Nb alloys with and without boron addition. At the solid/liquid interface, the dendrites in white contrast shown in Figs. 3(a), (c) and (d) are the primary β ones. The β dendrites exhibit a white contrast because the fine β -segregations can be formed by the $\beta \rightarrow \alpha$ solid-state transformation during quenching [15]. Figure 3(b) shows the growth morphology of α -leading solidification. The leading phase in the solidified microstructures can be easily determined by examining the side arm orientation of the dendrite spine due to its crystal structural difference between cubic β phase and hexagonal α phase. The included angles 60° and 90° between the primary and secondary dendritic arms are corresponding to the leading phases α and β , respectively. In the alloys with low boron addition (Fig. 3(a)), the boride particles can nucleate from the liquid in interdendritic regions before solidification of the α phase, which play the role of active catalyst for α nucleation and make α grow non-peritectically. In the alloys without low boron addition, if the growth rate v is low, as shown in Fig. 3(b), the α phase can be possibly selected as the leading solidification phase during the DS process [24]. The incomplete peritectic transformation occurs at a high v , which is characterized by the formation of obvious β -segregations in dendritic regions. The solid-state $\beta \rightarrow \alpha$ transformation in residual β dendritic regions should be responsible for the formation of the β -segregations. Because Al is concentrated in the dendritic regions, the γ phases can even form when v is high enough [17]. Appropriate v is needed to grow the primary β with appropriate dendrite spacing and the completely transform peritectically the β dendrite (Fig. 3(c)).

The PST crystals should be easily achieved if the alloy composition is close to C_p from the hypo-peritectic side [25]. When the solidification temperature drops below the peritectic point, α phase precipitates and covers the primarily solidified β phase. For hyper-peritectic alloys, the solute concentration of α solid is always less than that of the original liquid. The α phase solidified directly from the liquid can be suppressed. Although the volume fraction of the primary β phase in hyper-peritectic alloys is possibly less than that in hypo-peritectic alloys and then easily completely transforms peritectically into α phase, the α phase may solidify non-peritectically from the liquid, which results in unsuccessful controlling of the lamellar orientation. On the other hand, the peritectic transformation should be easier to be completed when the alloy composition is closer to C_p under the consideration of non-equilibrium solidification.

3.2 Lamellar microstructure controlled by self-seeding DS

When the Bridgman DS is initiated, there exist

inevitably three regions in the lower section of the directionally solidified ingot, which are named annealing, transition and melting regions, respectively [14]. The high-temperature β and α phases are reproduced from the previous as-cast microstructure in the transition region. The annealing temperature in the transition region should be increased from the $\beta + \alpha$ to the $L + \beta + \alpha$, eventually to the $L + \beta$ phase-region. Generally, the dendritic growth is related to the specimen history [26]. During the single DS, some β phases with inhomogeneous dendrite spacing formed in the conventional casting process can be reproduced and continue their growths towards the liquid in the transition region. Under a given growth condition, a stable growth can be reached when the appropriate dendrite spacing is achieved. The dynamical changing of the dendrite spacing may eventually result in the stable growth which needs a long distance to reach. This is also the possible reason why the width of the orientated columnar grain usually increases with the increase of the distance from the lower part of the directionally solidified ingot after the single DS process in the TiAl alloys.

Using the double DS technique, the coarse lamellar grains produced in the upper section of the single directionally-solidified ingot play the role as β -seeds. The recovered β -phases, which are qualified with appropriate dendrite spacing and preferred orientation in the transition region, should continue their growths towards the liquid. Although the grain is not a conventional α -seed, the PST crystal can still be produced by the double DS technique due to the complete peritectic transformation in both dendritic and interdendritic regions. Figure 4 shows the optical microstructure of the PST crystal in Ti–46Al–5Nb alloys produced by using the double DS technique. The lamellar microstructures in the crystal are aligned parallelly to the growth direction. Because of the preferred orientation of primary β and the orientation relationship between the β and α phases [21], the PST crystal, in which the lamellar microstructures are aligned parallelly or inclined 45° to the growth direction, can be produced in this way. The same conditions in the first and the repeated DS processes are very important to seed β phase. It suppresses the nucleation of new β phases in interdendritic regions and continues the growths of β dendrites with the same dendrite spacing at the beginning of the second DS process.

The complete peritectic transformation is difficult to achieve in both dendritic and interdendritic regions, but much easy to achieve only in the dendritic region. In order to align the lamellar microstructures with only one possible orientation, the lamellar seed with lamellar boundaries parallel to the growth direction should be chosen in the upper section of the single directionally-

solidified ingot. Figure 5 shows the BSE microstructures between transition and DS regions after the second DS in Ti–46Al–5Nb alloy. The lamellar orientation parallel to the growth direction does not change from the transition to DS region. The lamellar microstructures are aligned in both dendritic and interdendritic regions. During the second DS, the high-temperature β and α phases are recovered from the single directionally- solidified microstructures when the $L+\beta+\alpha$ three-phase region exists. Subsequently, the β phase can continue its growth, whereas the interdendritic α phase is easily seeded by itself in the conventional α -seeding way near the interface of two regions. Rejection of Al from the β dendrites should result in the preferred growth of the seeded α around the β dendrites and then thickening towards the dendritic arms by the peritectic transformation. Because the primary β dendrites are

completely transformed peritectically into the seeded α phase, the lamellar microstructures, even in the angled β dendritic regions, can be aligned parallelly to the growth direction by using the double DS technique.

However, the double DS technique seems not suitable for the TiAl–Nb alloys with low boron addition. Figure 6 presents the typical macrostructure of the directionally solidified Ti–45Al–6Nb–0.3B alloy after the second DS process. Due to the competitive growth of equiaxed α in the interdendritic region, a distinguished transition region with combined equiaxed and columnar grains disturbs the continuous growth of lamellar grain in the early stage of the second DS. The microstructures are not desirable to improve the room temperature ductility. Neither the refined columnar grains nor the aligned lamellar microstructures can be produced in the stable growth stage.

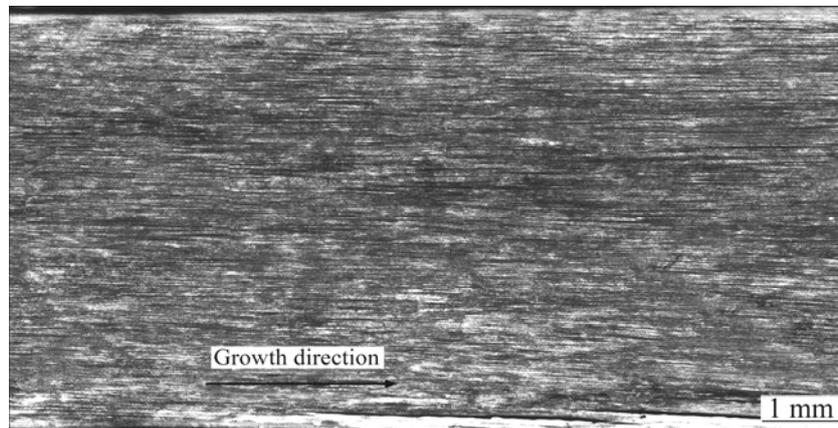


Fig. 4 Optical microstructure of PST crystal in Ti–46Al–5Nb alloy produced using double DS technique, showing aligned lamellar boundaries parallel to growth direction

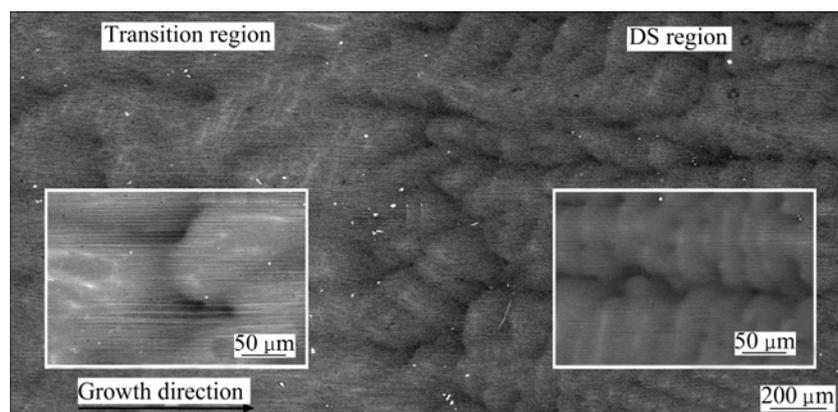


Fig. 5 BSE microstructures between transition and DS regions after second DS in Ti–46Al–5Nb alloy, showing unchanged lamellar orientation in both transition and DS regions, inserts shows high-magnitude microstructures in transition and DS regions, respectively

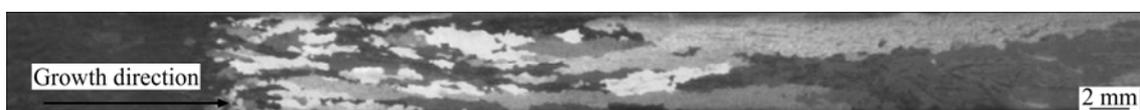


Fig. 6 Typical macrostructure of directionally solidified Ti–45Al–6Nb–0.3B alloy after second DS process at growth rate of 30 $\mu\text{m/s}$

4 Tensile properties and fracture of directionally solidified TiAl–Nb alloys

Although the lamellar microstructures can be controlled by the DS technique, the RT mechanical properties of the directionally solidified TiAl–Nb alloys are still not highly improved because of the yttria inclusions. Most of their tensile plastic elongations are only close to 2%. Consequently, further studies on the crucible mould for DS of TiAl–Nb alloys should be conducted. Some typical microstructures of fracture surfaces after tensile tests are shown in Fig. 7.

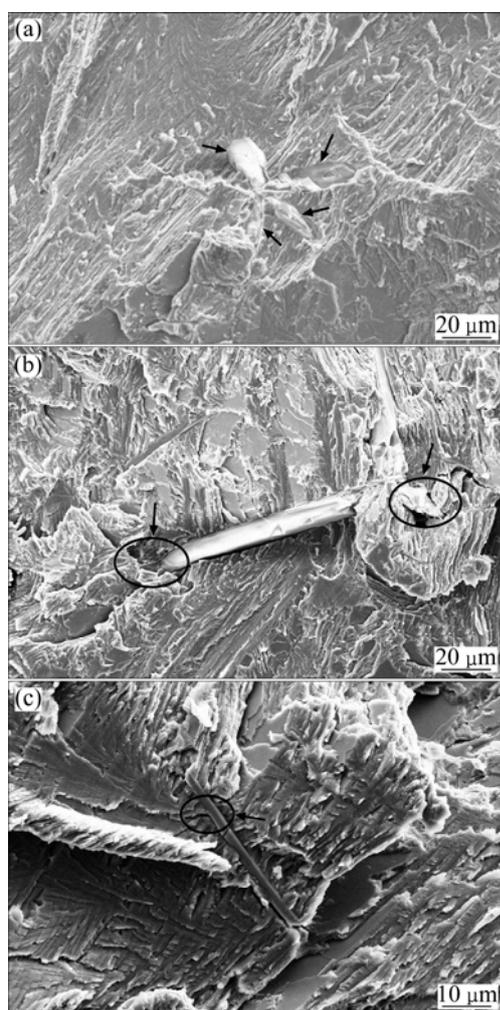


Fig. 7 Typical SEM fractographs of directionally solidified TiAl–Nb alloys, showing yttria particles at tip corner of fracture step (a) and microcracks formed along interfaces of yttria/matrix (b) in TiAl–Nb alloys, and long boride ribbons connected to radial stripe in TiAl–Nb alloys with boron addition (c)

The RT fracture is dominantly transgranular cleavage-like failure. The yttria particles and boride ribbons are frequently observed on the fracture surfaces,

and the tops of them are usually connected to the cracks or the radial stripe. The particles are likely to cause stress concentration, especially when they lie in the tensile specimens with the broad surface perpendicular to the tensile axis. The cracks in the poorly orientated particles can spread through their whole lengths instantaneously due to the brittle nature. Larger yttria particles and boride ribbons are detrimental to the tensile ductility of the directionally solidified TiAl–Nb alloys.

5 Conclusions

1) The lamellar microstructures can be aligned due to the complete peritectic transformation during DS of the alloys with dendritic solid/liquid interface under a lower temperature gradient G . Appropriate growth rate is needed to transform the β dendrites completely by the growth of the peritectic α phase. Low boron addition is harmful to align lamellar orientation due to the growth of non-peritectic α phase.

2) The double DS can successfully control the lamellar microstructures in primary β TiAl alloys. The coarse lamellar grains produced in the upper section of the directionally solidified ingot after the single DS act as β -seeds.

3) Due to the complete peritectic transformation in both dendritic and interdendritic regions, the PST microstructures with lamellar boundaries parallel or inclined 45° to the growth direction can be achieved by the double DS technique. The aligned lamellar microstructures parallel to the growth direction can be produced when the lamellar grains with the lamellar boundaries in the same orientation are seeded by themselves under appropriate growth conditions. The double DS technique is not suitable for the TiAl–Nb alloys with boron addition.

4) Due to the larger yttria particles and the boride ribbons in the directionally solidified TiAl–Nb alloys, the tensile plastic elongations of the alloys are only close to 2%.

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定向凝固 TiAl–Nb 合金的显微组织控制与力学性能

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摘要: 利用二次定向凝固技术控制初凝固 β 相 TiAl–Nb 合金的片层组织。由于合金定向凝固过程中发生完全包晶转变, 在枝晶固液生长界面条件下, 即可获得片层方向与生长方向成 0° 或 45° 夹角的多孪晶合成晶体(PST)。在适当的生长条件下, 当片层方向与生长方向平行的片层团在二次定向凝固过程中发生籽晶作用时, 可以控制 PST 晶体内部的片层方向仅与生长方向平行。合金中低含量 B 的加入会导致非包晶 α 相的生长, 从而不利于控制片层方向。定向凝固 TiAl–Nb 合金中存在较大的氧化钨颗粒与条状硼化物颗粒, 导致其室温拉伸延伸率仅接近 2%。

关键词: TiAl 合金; 定向凝固; 包晶转变; 片层方向; 多孪晶合成晶体; 力学性能

(Edited by FANG Jing-hua)