Microstructures, micro-segregation and solidification path of directionally solidified Ti-45Al-5Nb alloy

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Abstract: To investigate the effect of solidification parameters on the solidification path and microstructure evolution of Ti-45Al-5Nb (at.%) alloy, Bridgman-type directional solidification and thermodynamics calculations were performed on the alloy. The microstructures, micro-segregation and solidification path were investigated. The results show that the β phase is the primary phase of the alloy at growth rates of $5\text{--}20\ \mu\text{m}\cdot\text{s}^{-1}$ under the temperature gradients of $15\text{--}20\ \text{K}\cdot\text{mm}^{-1}$, and the primary phase is transformed into an α phase at relatively higher growth rates ($V > 20\ \mu\text{m}\cdot\text{s}^{-1}$). The mainly S-segregation and β -segregation can be observed in Ti-45Al-5Nb alloy at a growth rate of $10\ \mu\text{m}\cdot\text{s}^{-1}$ under a temperature gradient of $15\ \text{K}\cdot\text{mm}^{-1}$. The increase of temperature gradient to $20\ \text{K}\cdot\text{mm}^{-1}$ can eliminate β -segregation, but has no obvious effect on S-segregation. The results also show that 5 at.% Nb addition can expand the β phase region, increase the melting point of the alloy and induce the solidification path to become complicated. The equilibrium solidification path of Ti-45Al-5Nb alloy can be described as $L \xrightarrow{L\rightarrow\beta} L+\beta \xrightarrow{L+\beta\rightarrow\alpha} \alpha+\beta_R \xrightarrow{\beta\rightarrow\alpha} \alpha \xrightarrow{\alpha\rightarrow\gamma} \alpha+\gamma \xrightarrow{\alpha\rightarrow\alpha_2+\gamma} \gamma_R+(\alpha_2+\gamma)$, in which β_R and γ_R mean the residual β and γ .

Key words: TiAl-Nb alloy; directional solidification; microstructure; micro-segregation; solidification path

CLC numbers: TG146.23

Document code: A

Article ID: 1672-6421(2016)02-107-07

In the last two decades, TiAl-based alloys have attracted more and more attention due to their excellent properties, such as excellent high temperature properties, low density, high specific strength and high Young's modulus^[1, 2]. High Nb addition can significantly improve high temperature properties, but also causes a loss of room-temperature ductility of TiAl alloys^[3]. The mechanical properties of TiAl-based alloys are significantly dependent on their microstructures. The alloy, with fully lamellar microstructures consisting of TiAl (γ -phase) and Ti_3Al (α_2 -phase), exhibits a good combination of room temperature toughness and elevated temperature strength. However, the mechanical properties of fully lamellar TiAl-based alloys are extremely anisotropic with respect to the lamellar orientation of microstructures^[4, 5]. So the directional solidification (DS) technique is used to align the lamellar

microstructures. By using directional solidification technique, the columnar grains with aligned lamellar microstructures and poly-synthetically twinned (PST) microstructures can be obtained, which are helpful in improving the mechanical properties of TiAl-based alloys^[6, 7].

Recently, Ding et al^[8, 9] successfully controlled the lamellar orientation parallel to the growth direction in TiAl-Nb alloys with primary β phase by using double Bridgman DS technique, and proposed that closely-complete peritectic transformation is necessary to align the peritectic α phase and control the lamellar orientation. Liu et al^[10, 11] studied the microstructure evolution and micro-segregation pattern in directionally solidified Ti-46Al-8Nb alloy, and found that the lamellar orientation is parallel and 45° to the growth direction of β dendrite, and peritectic reaction can lead to significant chemical inhomogeneity. Previous studies also showed that high Nb addition changes the TiAl phase diagram greatly, which leads to the solidification behavior being different with binary TiAl alloys^[12]. The high Nb addition also has great effect on the solidification path and subsequent solid-state transformation, and usually leads to stabilization of β

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Received: 2016-01-08; Accepted: 2016-02-22

phase at lower temperature in the form of ordered B2 phase; which could greatly affect the mechanical properties of TiAl alloys^[13, 14]. However, few studies focus on the correlations between solidification parameters and solidification paths of TiAl-Nb intermetallic alloys. The intention of this paper is to investigate the effect of solidification parameters on the solidification path and microstructure evolution of Ti-45Al-5Nb alloy. The effects of solidification parameters on the primary phase, micro-segregation and solidification paths are discussed. The equilibrium and theoretical solidification path are established by using Thermo-Calc software and TT-TiAl database. These results will provide a reference for microstructure control and casting process design of TiAl intermetallics.

1 Experimental procedure

Master ingot of Ti-45Al-5Nb (at.%) alloy was prepared using Ti (99.96%), Al (99.98%) and Al-70Nb (wt.%) of commercial purity by the induction skull melting (ISM). The terminal composition is Ti-45.01Al-5.29Nb (at.%) based on chemical analysis. The DS samples were machined to cylindrical rods of 3 mm in diameter and 100 mm in length from the ingot using a wire cut electrical discharge machining. Then the rods were put into a high purity alumina crucible with a diameter of 4/5 mm (inside/outside diameter) and length of 120 mm. Yttria was coated on the inner surface of the alumina crucible to prevent reaction between the TiAl melt and alumina crucibles. DS experiments were performed in a Bridgman-type vacuum furnace^[15, 16].

Temperature profiles in the samples were measured by W/Re thermocouples which were placed near the alumina crucibles. One thermocouple was placed in the bottom of the samples which is near the solid/liquid interface. The other one was placed approximately 15 mm from the bottom of the samples which was in the liquid region. Temperature gradient (G) could be calculated from the measured temperature profiles. The temperature gradient (G) values was certainly dependent on the melt temperature and could be controlled as a constant of approximately 15 or 20 K·mm⁻¹. The samples were heated to the given temperature corresponding to the temperature gradient within 4 h followed by thermal stabilizing for 30 min. Then the samples were pulled at growth rates (V) of 5 μm·s⁻¹, 10 μm·s⁻¹ and 20 μm·s⁻¹, with temperature gradients of 15 K·mm⁻¹ and 20 K·mm⁻¹, respectively. After the DS, the samples were quenched in Ga-In-Sn liquid alloy to obtain the interface growth morphology.

The directionally solidified bars were cut longitudinally by a wire cut electrical discharge machining, polished using standard metallographic techniques; and etched with a solution of 10 ml HF + 10 ml HNO₃ + 180 ml H₂O. Detailed microstructures were examined using a scanning electron microscope (SEM) equipped with an energy dispersive spectrometer (EDS) in backscattered electron mode (BSE).

2 Results and discussion

2.1 Macro and microstructures of directional solidified bars

The longitudinal macrostructures of directionally solidified bars at different solidification parameters are shown in Fig. 1. There are four distinguished sections in DS bars, which are unmelted region (A), directional solidification region (B), mushy zone (C) and quenched region (D). In the un-melted region, the as-cast microstructure was retained since this section is placed into Ga-In-Sn alloy during the DS process. Columnar grains nucleated and grew competitively in DS region once pulling started. The number of the columnar grains increases with an increase in growth rate and temperature gradient, and decreases with an increase in distance from the lower part of the DS bar. After quenching, the remaining liquid solidified quickly and the morphology of the mushy zone can be reserved. Figure 2 shows the typical microstructures at quenched solid/liquid interfaces at different solidification parameters. A transient structure composed of cells and dendrites is formed at $V = 5 \mu\text{m}\cdot\text{s}^{-1}$, $G = 20 \text{ K}\cdot\text{mm}^{-1}$, as shown in Fig. 2(a). The dendritic arrays are well aligned along the growth direction, and the regular dendrites can be observed with increasing V , as shown in Fig. 2(b) and (c).

Fig. 1: Macrostructures of longitudinal section of directionally solidified bars at growth rate of (a) 5 μm·s⁻¹, (b) 10 μm·s⁻¹, (c) 20 μm·s⁻¹ with a temperature gradient of 20 K·mm⁻¹, and (d) 10 μm·s⁻¹ with a temperature gradient of 15 K·mm⁻¹. The letters show the different regions: A - unmelted region, B - directional solidification region, C - mushy zone, D - quenched region

Fig.2: Morphologies of solid-liquid interface at growth rate of (a) $5 \mu\text{m}\cdot\text{s}^{-1}$, (b) $10 \mu\text{m}\cdot\text{s}^{-1}$, (c) $20 \mu\text{m}\cdot\text{s}^{-1}$ with temperature gradient of $20 \text{ K}\cdot\text{mm}^{-1}$; and (d) $10 \mu\text{m}\cdot\text{s}^{-1}$ with temperature gradient of $15 \text{ K}\cdot\text{mm}^{-1}$

In order to identify the primary solidification phase (α or β), quenched technology is used to reserve the primary crystal morphology in the front of the solid/liquid interface. The primary phase is identified as β phase by noting that the secondary dendritic arms (SDAS) are perpendicular to primary dendritic arms (PDAS) at $V = 10 \mu\text{m}\cdot\text{s}^{-1}$, $G = 20 \text{ K}\cdot\text{mm}^{-1}$, as shown in Fig. 2(b). However, some secondary dendritic arms with orientation of 60° to primary dendritic arms can be observed when V is increased to $20 \mu\text{m}\cdot\text{s}^{-1}$, as shown in Fig. 2(c). It indicates that the solid/liquid interface is changed to mixed primary phases (α and β) as growth rate is increased. But there is no obvious transformation of primary phase as temperature gradient decreases from 20 to $15 \text{ K}\cdot\text{mm}^{-1}$ except primary dendrite arm spacing are significantly reduced.

In TiAl-Nb alloys, the solidification path becomes complex compared with binary alloys, due to the addition of β phase stabilizing element Nb, which results in the liquid+ α + β phase coexistence region appearing in the phase diagram^[17], as shown in Fig. 3. The mixed primary phase is expected when cooling into L+ α + β three phase region. In addition, the variation of primary phase with growth rate and temperature gradient can be explained according to interfacial temperature response theory. The schematic of interfacial temperature response function with growth rate and temperature gradient is shown

Fig. 3: Schematic phase diagram of ternary Ti-Al-M systems^[17] (M is a stabilizing element)

Fig. 4: Schematic of interfacial temperature response function with growth rate and temperature gradient^[19]

in Fig. 4^[18]. When $V_1 < V < V_3$, β cellular/dendrite growth is stable, while α cellular/dendrite growth is becoming stable as $V_3 < V < V_4$. In this research, the transformation of primary phase happens as $G = 20 \text{ K}\cdot\text{mm}^{-1}$, $V = 20 \mu\text{m}\cdot\text{s}^{-1}$, therefore, it can be deduced that the growth rate of $20 \mu\text{m}\cdot\text{s}^{-1}$ is located about in V_3 . At the growth rate higher than $20 \mu\text{m}\cdot\text{s}^{-1}$, the α phase has the higher interface response temperature and it will be the primary phase.

2.2 Solute segregation and lamellar orientations

Figure 5 shows the microstructure of the mushy zone and the corresponding solute distribution at $V = 10 \mu\text{m}\cdot\text{s}^{-1}$ and $G = 15 \text{ K}\cdot\text{mm}^{-1}$. EDS analysis shows that the primary β phase is rich in Ti ($\sim 42.74 \text{ at.}\%$), while the quenching liquid inter-dendritic regions contain high Al ($\sim 49.46 \text{ at.}\%$) and low Nb ($\sim 3.52 \text{ at.}\%$), as shown in Fig. 5(a). The solute distribution above demonstrates the segregation characterization of Ti-45Al-5Nb alloy named S-segregation during solidification^[13]. The white strip-shapes inside dendritic arm in Fig. 5(c) are enriched in Nb element named β segregation, which can be attributed to the solute segregation during the $\beta \rightarrow$

Fig. 5: Morphologies and solute distribution of mushy zone in Ti-45Al-5Nb alloy during directional solidification at a growth rate of $10 \mu\text{m}\cdot\text{s}^{-1}$ and temperature gradient of $15 \text{ K}\cdot\text{mm}^{-1}$: (a) microstructure of mushy zone, (b) solute distribution of mushy zone, (c) retained β phase in mushy zone, (d) composition of retained β phase

α transformation. The solute Al diffuses to α while Nb goes in the opposite direction to retained β phase during transformation. This tendency leads to enriching in Nb and depletion in Al at the core of the dendrites and the boundaries between transformed α grains.

Figure 6 shows the microstructures and solute distribution of the mushy zone and the directional solidification region at $V =$

$10 \mu\text{m}\cdot\text{s}^{-1}$ and $G=20 \text{ K}\cdot\text{mm}^{-1}$. The dendritic growth behavior and S-segregation with higher Nb and lower Al contained in dendritic core can be observed, but there is no obvious β -segregation in the mushy zone during solidification. It indicates that the higher temperature gradient can increase the diffusion of Nb and Al elements during $\beta \rightarrow \alpha$ transformation, which can reduce

Fig. 6: Morphologies and solute distribution of Ti-45Al-5Nb alloy during directional solidification at a growth rate of $10 \mu\text{m}\cdot\text{s}^{-1}$ and temperature gradient of $20 \text{ K}\cdot\text{mm}^{-1}$: (a) and (b) mushy zone, (c) and (d) directional solidification zone

the degree of Nb and Al enrichment. Additionally, with a large temperature gradient the richening of Nb in the primary β phase will be reduced. When the temperature gradient is increased, more Al is enriching the liquid near the S/L zone, which is helpful for the formation of α phase, and the α phase may even become the primary phase. It also indicates the solidification path can be modified with changes in the solidification parameters.

The $(\alpha_2+\gamma)$ lamellar structure in the directional solidification region can be observed in Fig. 6(c). The micro-segregation, mainly S-segregation still exists but has been reduced after a

series of phase transformations compared to the mushy zone. Figure 7 shows the lamellar structure in directional solidification region with different solidification parameters. The micro-segregation amplitude increases with an increase in growth rate and temperature gradient due to the lower diffusion of Nb and Al elements. Through the results above, the solidification parameters have great influence on the micro-segregation such as S-segregation and β -segregation, and the decrease of the micro-segregation can be attributed to the lower growth rate and higher temperature gradient.

Fig. 7: BSE images of lamellar structure of directionally solidified Ti-45Al-5Nb alloy in directional solidification region at different solidification parameters: (a) $V=5 \mu\text{m}\cdot\text{s}^{-1}$, $G=20 \text{ K}\cdot\text{mm}^{-1}$, (b) $V=10 \mu\text{m}\cdot\text{s}^{-1}$, $G=20 \text{ K}\cdot\text{mm}^{-1}$, (c) $V=20 \mu\text{m}\cdot\text{s}^{-1}$, $G=20 \text{ K}\cdot\text{mm}^{-1}$, (d) $V=10 \mu\text{m}\cdot\text{s}^{-1}$, $G=15 \text{ K}\cdot\text{mm}^{-1}$

Figure 7 shows the BSE images taken from the DS region with steadily grown columnar grains at different DS parameters. The lamellar structure can be observed and the lamellar orientations are shown by the arrows. The lamellar orientations are aligned about 45° to growth direction in cases shown in Fig. 7(b) and (c). However, the orientation in Fig. 7(a) and (d) are aligned about 30° with growth direction. The lamellar structure is not formed from the liquid but from solid-state phase transformation, and the lamellar structure of TiAl alloys consists of γ and α_2 phases with the $(111)\gamma/(0001)\alpha_2$ orientation relationship^[4,19]. When the β phase is formed during solidification, the resulting lamellar structure is oriented parallel and at 45° to the growth direction since the β phase grows along $\langle 001 \rangle$ and the HCP α phase is formed from β phase following the Burgers orientation relationship^[4,20].

In Fig. 7(a) and (d), the orientation of the lamellar is aligned about 30° to the growth direction, not 45° . We all know, the preferred growing orientation of primary β phase is $\langle 001 \rangle$, after solid transformation (β to lamellar of γ and α_2), the orientation of lamellar is aligned about 45° to the directional solidification direction. This deviation is attributed to the initial formation of the

lamellar structure and the competitive nucleation and growth of crystals during initial solidification. In seeding DS technology, the top of the seeds melts firstly, inducing the orientation of the seeding crystals to be repeated in solidifying alloy during subsequent solidification. The final lamellar orientation seeded by the unmelted region grains can be observed, as shown in Fig. 1. The orientation in un-melted regions is stochastic, so the final lamellar orientation may deviate from parallel or 45° to the growth direction.

2.3 Solidification path of Ti-45Al-5Nb alloy

According to the discussion in section 2.1, the solidification path for Ti-45Al-5Nb in the present condition with lower growth rates ($V < 20 \mu\text{m}\cdot\text{s}^{-1}$) under a constant temperature gradient ($G=20 \text{ K}\cdot\text{mm}^{-1}$) can be described as:

In higher growth rates ($V > 20 \mu\text{m}\cdot\text{s}^{-1}$) under a constant temperature gradient ($G=20 \text{ K}\cdot\text{mm}^{-1}$) can be described as:

In order to compare with the equilibrium solidification, the equilibrium solidification path is investigated by using both

Thermal-Calc software and TT-TiAl database. The ternary equilibrium phase diagram of Ti-45Al-5Nb alloy is established with the boundary condition of total number of moles equal to one under atmospheric pressure. Figure 8 shows the vertical section of a calculated ternary phase diagram of Ti-45Al-5Nb alloy. Compared with the equilibrium phase diagram of Ti-Al phase diagram, the main differences manifest as follows: the liquidus rises from 1,530 °C to about 1,600 °C; the peritectic transformation temperature increases to 1,505 °C; the β phase region moves right and the α phase region gets smaller; the solid-state transformation temperature decreases to 1,090 °C. Briefly, the addition of 5at.% Nb mainly expands the β phase region and increases the melting point of the alloy, inducing the solidification path becomes complicated. In order to investigate the equilibrium solidification path precisely, the isothermal section and its amplification are intercepted at different temperatures, as shown in Fig. 9. Figure 9(a) shows the isothermal section of the phase diagram at 1,600 °C, the solid β phase is precipitated directly from the liquid L in the Al content of 45at.%. The peritectic reaction $L+\beta\rightarrow\alpha$ can be observed at the range of 1,400 °C–1,500 °C in isothermal section of the phase diagram, as shown in Fig. 9(b). The solid-state phase transformation $\beta\rightarrow\alpha$ process happens in the range of 1,100 °C –1,400 °C, as shown in Figs. 9(c) and (d).

Fig. 8: Calculated ternary phase diagram of Ti-45Al-5Nb alloy in vertical section

According to the results above, the equilibrium solidification of Ti-45Al-5Nb alloy is as follows: the primary β phase will form firstly by $L\rightarrow\beta$ at equilibrium solidification; the peritectic reaction $L+\beta\rightarrow\alpha$ will occur between primary β phase and retained liquid as the temperature decreases; since the retained liquid is limited, the residual β phase will transform into α phase by solid-state transformation; after the solid phase is completely converted into α -phase, when the temperature is further decreased, $\alpha\rightarrow\gamma$ solid phase transformation occurs in the range of 1,100 °C –1,400 °C, and then the alloy turns into the $\alpha+\gamma$ two-phase region; the retained α phase is decomposed by $\alpha\rightarrow\gamma+\alpha_2$ eutectoid transformation at eutectoid transformation temperature, eventually forming $\gamma+\alpha_2$ two-phase structure. Therefore, the equilibrium

Fig. 9: Isothermal sections of Ti-45Al-5Nb ternary system at different temperatures: (a) 1,600 °C, (b) 1,500 °C, (c) 1,400 °C, (d) 1,100 °C, and (a1), (b1), (c1) and (d1) are amplification of (a), (b), (c) and (d)

solidification path of Ti-45Al-5Nb alloy can be described as:

There are some obvious differences between the calculated equilibrium solidification path and the non-equilibrium solidification path found in the experiment. When the growth rate (V) increases and the temperature gradient (G) decreases, the equilibrium solidification path gradually changes into the non-equilibrium solidification path, and the primary phase will be different. At a relatively higher G/V , the primary phase will be β phase and the resulting lamellar structure will be oriented 45° or parallel to the growth direction. At a relatively lower G/V , the primary phase will be α phase and the resulting lamellar structure will be perpendicular to the growth direction. Therefore, at a relatively lower G/V , the preferred method for lamellar orientation controlling is by seeding technology, since the final lamellar structures will be perpendicular to growth direction when α phase is the primary

phase of the alloy. The equilibrium solidification path and higher G/V solidification path are similar with the β phase as the primary phase. Thus the regular directional solidification is appropriate for lamellar orientation control, since lamellar orientations will be aligned about 45° or parallel to the growth direction, and the comprehensive properties will be obtained.

3 Conclusions

In the present research, microstructures, micro-segregation and solidification path of Ti-45Al-5Nb alloy were investigated by thermodynamics calculation and directionally solidified technology under different solidification parameters. The following conclusions were obtained:

(1) The β phase is the primary phase of the alloy at the growth rates of $5 \mu\text{m}\cdot\text{s}^{-1}$ to $20 \mu\text{m}\cdot\text{s}^{-1}$ and the temperature gradients of $15 \mu\text{m}\cdot\text{s}^{-1}$ to $20 \text{K}\cdot\text{mm}^{-1}$. The primary phase is transformed into α phase at a relatively higher growth rate ($V > 20 \mu\text{m}\cdot\text{s}^{-1}$).

(2) The mainly S- and β -segregation, corresponding to solute Nb enrichment in the primary β dendrite during solidification and $\beta \rightarrow \alpha$ respectively, can be observed in the Ti-45Al-5Nb alloy at a growth rate of $10 \mu\text{m}\cdot\text{s}^{-1}$ and the temperature gradient of $15 \text{K}\cdot\text{mm}^{-1}$. The increase of temperature gradient to $20 \text{K}\cdot\text{mm}^{-1}$ can eliminate β -segregation by increasing the diffusion of Nb element during $\beta \rightarrow \alpha$ transformation.

(3) The addition of 5at.% Nb mainly expands the β phase region and increases the melting point of the alloy; inducing the solidification path becomes complicated. The equilibrium solidification path of Ti-45Al-5Nb alloy can be described as:

(4) At a relatively lower G/V , the preferred method for lamellar orientation control is by seeding technology. At a relatively higher G/V , the regular directional solidification is appropriate for lamellar orientation control.

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