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Influence of thermal stabilization treatment on the subsequent microstructure development during directional solidification of a Ti–46Al–5Nb alloy

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ABSTRACT

Directional solidification (DS) experiments with thermal stabilization (TS) treatments were performed on Ti–46Al–5Nb (at.%) alloys in a Bridgman-type furnace using a quenching technology. Influence of the TS treatment on mushy zone and directional growth afterwards were investigated. The results show that the length of the mushy zone decreases but the β dendrite spacing in directional growth significantly increases with increasing TS time. During the DS process, β dendrite spacing is more homogeneous and its growth direction is more inclined to parallel to the axial direction with increase of the TS time. Al solute concentration in the mushy zone in a steady-state is always lower than that in original as-cast alloys. The mushy zone with the columnar β and α grains is easily produced after TS treatment on the alloys with microstructures of the directional dendrite segregation morphology before DS starting. TS treatment results in the redistribute of solute Al thus changes the phase constituent in the mushy zone. An appropriate TS is necessary to produce the L + β + α region in the mushy zone, which is of great benefit to control DS microstructures of TiAl peritectic alloys.

1. Introduction

As a representative of the third generation γ-TiAl alloys, niobium-rich γ-TiAl based alloys are potential candidates for high-temperature structural applications because of their excellent high temperature properties, good oxidation resistance and creep resistance [1,2]. However, their applications are mainly hindered by the poor ductility and toughness at room temperature. Directional solidification (DS) technology allows a close control of solidification path and crystal orientation to improve mechanical properties of the alloys, which has been attracted extensive attentions in recent years [3,4].

Because of the existence of an axial temperature gradient during DS, the mushy zone mixed liquid and solid phases is produced at the position between the as-cast solid zone and the complete liquid zone. Directional crystal grows in a way of forward movement of the mushy zone into the complete liquid zone, thus, the crystal growth has a degree of inheritance from the mushy zone. Initial transition stage of a DS is relevant to the crystal growth state in initial mushy zone before starting the DS. Previous studies show that, as a result of the existence of the mushy zone, the initial solid/liquid interface between the mushy zone and the complete liquid phase zone is corrugated [5], and the solute concentration in the complete liquid zone is deviated from the original alloy composition [5,6], which can lead to solute redistribution thus change the phase constituent in the mushy zones. Additionally, microstructure in the steady-state stage is closely related to nucleation and competitive growth in initial transition stage of the DS [7–9]. Therefore, a holding stage named as thermal stabilization (TS) treatment is necessary before DS starting for modifying the crystal growth state and morphology in the initial mushy zone, which may affect microstructure development during directional growth later. The TS treatment is very important to control DS microstructures, especially in TiAl alloys with complicated phase transitions during cooling. However, very limited research results concerning TS treatment were reported in TiAl alloys.

In this work, a preliminary study on the effects of the TS treatment on the mushy zone and the microstructures of DS in Ti–46Al–5Nb alloys is conducted. The aim of this work is try to understand the microstructure development affected by TS

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treatment and specimen history in the initial stage as well as in steady-state stage of the DS.

2. Experimental procedures

Ti–46Al–5Nb (at.%) alloys with the exact chemical composition of Ti-46.20Al-4.78Nb were supplied in the form of a cast cylindrical ingot, which was produced by using induction skull melting (ISM). A set of bars with 6.3 mm in diameter and 100 mm in length were cut from the ingot. Each bar was placed into an alumina crucible coated by yttria, then proceeded with TS and DS treatments, which ended by quenching after remelted in a Bridgman-type DS furnace. All bars were divided into three groups. The first group (G1) bars were directly performed a quenching (Q) after TS treatment for 0, 30 and 60 min, respectively. The second group (G2) bars were directionally solidified for 50 mm at a growth rate of 50 μm/s, then quenched after TS treatment for 30 min and 60 min. After TS treatments for 0 min, 30 min and 60 min, the third group (G3) bars were directionally solidified at a growth rate of 50 μm/s, then quenched after DS to a constant length of 50 mm. Table 1 summarizes the different treatment processes on the three group bars. The temperature gradient in liquid was G_L~1 K/mm in each treatment.

All the three group bars were sectioned longitudinally. After polished using standard metallographic techniques, longitudinal microstructure analyses were studied with the use of a field emission scanning electron microscope (FEM) adopting the back-scattered electron (BSE) imaging mode. In addition, energy dispersive X-ray analysis (EDX) was employed to analyze the composition distribution in quenched mushy zone. Variable temperature sectional view of TiAl–5Nb alloy was calculated by *Thermo-Calc* software to analyze the influence of TS treatment on Al solute diffusion in the complete liquid zone.

3. Results

Fig. 1(a) and (b) show the typical microstructures in the region ahead of TS treatment area in experimental bars. The axial direction is horizontal. Dark grey regions show Al-segregations where Al is rejected by primary β phase [10]. The equiaxial dendrite segregation morphology is observed in G1 and G3 bars, while the directional dendrite segregation morphology is observed in axial direction in G2 bars.

Fig. 2 shows microstructures of the quenched mushy zone in G1 bars with different TS time. As shown in Fig. 2, microstructures in the mushy zone consist of bright grey β phase and dark grey liquid phase for all the TS treatments [11]. The size of β dendrite increases but the volume fraction of liquid decreases with increase of temperature in each of the mushy zones. The β dendrites tend to coarsen with increase of the TS time. The high temperature interface, β/liquid interface marked by T_L and the low temperature interface, solid-state phase transformation interface marked by T_S are presented in quenched mushy zone. The β/liquid interface migrates downward to a lower temperature zone with increase of the TS time from 0 to 60 min.

Table 1
Experimental bars with different treatment processes.

Sample group	Treatment process	TS treatment time
G1	TS + Q	0, 30, 60 min
G2	DS + TS + Q	30, 60 min
G3	TS + DS + Q	0, 30, 60 min

Fig. 3 shows microstructures of the quenched mushy zone in G2 bars with different TS time. The length of quenched mushy zone decreases when the TS time increases from 30 min to 60 min. Light grey contrast α phase can be observed in lower temperature section of the mushy zones. A small three-phase mixture region consisting of liquid, β and α phase is presented in Fig. 3a. As shown in Fig. 3b, the columnar β and α are observed in the mushy zone. The volume fraction of the liquid phase almost reaches to zero in the mushy zone, which suggests that it reaches to a closely-steady state during TS treatment [12].

Fig. 4 shows the Al-concentration profiles in longitudinal and transversal directions in the quenched mushy zone in Fig. 3b. The Al-concentration of the solid phase along the cylinder axis in the mushy zone increases from 43.2 at.% at the β/liquid interface to 45.5 at.% at solid-state phase transformation interface, however, the Al-concentration along the transverse direction varies in a small range and the average content is measured as 43.5 ± 0.20 at.%. The Al solute variation between β phase and α phase is not observed. It should be noted that the Al concentration of the mushy zone decreases to a lower value than that of 46.2 at% in original as-cast alloys.

Fig. 5 shows microstructures in the initial transition stage in the G3 bars with different TS time. The initial interface of directional growth, marked by dark dotted line T_L, is corresponding to the high temperature interface in initial mushy zone before DS starting. As shown in Fig. 5, with increase of the TS time from 0 to 60 min, the number of columnar dendrites at the upside of the T_L interface decreases, and the dendrite morphology exhibits more regularly. The initial directional growth interface also migrates downward to a lower temperature side, which is similar to that in Fig. 2 and Fig. 3. Length of the initial mushy zone, which is summarized in Table 2, decreases with increasing TS time in G3 bars.

Fig. 6 shows microstructures at the quenched solid/liquid interfaces and in steady-state zones in G3 bars with different TS time. As shown in Fig. 6, with increase of the TS time, the more regular β dendrite morphology and the larger primary dendrite arm spacing are observed at the quenched solid/liquid interfaces (Fig. 6a, b and c) as well as in the steady-state zones (Fig. 6d, e and f). The β dendrite spacing is more homogeneous for a longer TS treatment time. Both of the primary and secondary dendrites tend to coarsen when the TS time increases. Statistics on the primary dendrite arm spacing in steady state zones are also listed in Table 2. The primary dendrite arm spacing increases significantly with increasing TS time.

4. Discussion

A typical mushy zone forms between the non-molten solid zone and the complete liquid zone after remelting because of one-dimensional temperature gradient existence. Before starting DS, the mushy zone is a transition region that will connect the original microstructure of as-cast alloy with the directional growth microstructure. The crystals are going to grow on the basis of the high temperature interface of the mushy zone during DS, which inherits crystal state and orientation formed in the TS stage. So the mushy zone is subjected to the TS treatment, and plays an important role in microstructure control during DS.

TS treatment intensively acts on the redistribute of solute Al in the mushy zone. Fig. 7 shows schematic representation of the mushy zone created by remelting of the alloy when a temperature gradient is imposed. As shown in Fig. 7, three parameters, the temperature region (ΔT) with both solid phase and liquid phase in the diagram, the temperature gradient (G) and the length of mushy zone in the sample (L_m), can be calculated by $L_m = \Delta T/G$. It is indicated that the length of mushy zone is dependent on the range