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PREPARATION OF TWINNED DENDRITES OF Al-Zn ALLOY WITH HIGH Zn CONTENT

Twinned dendrites in Al-Zn alloy with high Zn content (40% wt.%) were successfully prepared by directional solidification. At different directional solidification rates (1000 and 1500 μm/s), microstructures and growth orientation variations of Al twinned dendrite and non-twinned dendrite were characterized. By using the inverted trapezoidal graphite sleeve at 1000 μm/s, Al twinned dendrite were formed to developed feather crystal structures in longitudinal section. Its primary and secondary twinned dendrite were grew along [110] direction. Moreover the deviation angle between [110] direction of Al twinned dendrite and the heat flow direction was about 27.15°. While not using the inverted trapezoidal graphite sleeve at 1000 and 1500 μm/s, Al dendrite was the non-twinned dendrite and the twinned dendrite was not appeared. The experimental results showed that the higher temperature gradient, a certain pulling rate and convection environment were the formation conditions of twinned dendrites.

Keywords: directional solidification, twinned dendrite, microstructure, growth orientation, the deviation angle

1. Introduction

Twinned dendrites, also are known as feathery grains, bicrystal, twinning crystal, basalt crystal, belong to a kind of growth twin [1-3]. It was initially found in the semi-continuous casting aluminium alloy. And it is a abnormal crystal as the third kind of dendrite in casting, which is completely different from the columnar and rod dendrite. The twinned dendrite is also called feathery dendrites [2], because its morphology is similar to the feather in two-dimensional microstructure. The current researches are showed that twinned dendrite is a casting structure existed under certain conditions. Because of the maximum growth rate along the axial direction, the growth direction of twinned dendrites paralleled with the axial direction is the strongest, which results in the other growth directions suppressing easily. In addition, since strong anisotropy characteristic and non-deform or fracture characteristic of twinned dendrites under the pressure processing process, the alloy microstructure and mechanical properties are optimized [4]. Therefore, the key of improving alloy properties is how to obtain larger areas of twinned dendrites in casting process, which is the important value on solidification theory research and industrial application.

It is generally believed that dendrite tip morphology of aluminium alloy, solute segregation and specific solidification conditions (temperature gradient, convection, etc.) are the important factors affecting formation and growth of twinned dendrite [5,6]. Most twinned dendrites are formed at the maximum shearing rate, which could lead strong convection. While the growth directions of dendrites trunks and side arms are related with the convection direction. That because the shearing stress caused by convection results in the formation of stacking faults, which could induce the twinned generating and twinned dendrite growth. Stacking fault formatting is a mistake in the periodic repeated stacking sequence of the crystal structure layer between some two layers, resulting in the wrong arrangement of atoms along the two sides of the interlayer plane (called the stacking fault surface) and changing the dendrite tip morphology. And during directional solidification, particularly in vertical Bridgman directional solidification, high temperature gradient and large solidify rate could easily cause solute distribution in disequilibrium at top and button sample, which leads the strong convection. Based on it, the stacking direction and stacking rate of atomic are changed, which results in the twinned dendrite interface appearing and twinned dendrite growth. On the other hand, twinned dendrite formation and growth are the result of the very steep temperature gradient and the narrow supercooled zone [7]. So according to the above analysises, when the superheat temperature of the melt is so higher, the cooling rate of ingot is faster and the heat flow is diffused from crystal surface to a single direction, twinned dendrites are easily prepared. Based on that, the current studies suggested that the high temperature gradient and strong convection are the necessary conditions for the formation of twinned dendrites.

However, the alloy element composition is another important effecting factor. As Al element has the weak anisotropic in Al-Zn alloy, which is easily formed a rough interface not

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conducive to the formation of twinned dendrites [8]. While Zn is the hcp structure, which is contributed to the stacking faults formatting and easily induced twinned dendrite formatting in the fcc structure. And Gonzales and Rappaz [9] recently found that with the Zn content increasing, the solid-liquid interface energy anisotropy was changed, then the growth direction would be turned from [100] direction of conventional dendrite to [110] direction of twinned dendrite. Meanwhile, the present study [10] showed that when Zn content was below 25 wt. %, the dendrites remained its [100] direction. And above 60 wt. %, it remained [110] direction of the twinned dendrite growth direction. That was because the overall anisotropy of the stiffness plot for 25 wt. % was much weaker than that around 60 wt pct. So twinned dendrite would be grown easily along [100] direction in crystallography in below 25 wt. % Zn alloy and along [110] direction in crystallography in below 65 wt. % Zn alloy. It means that between 25 wt. % to 60 wt. % of Zn content, the twinned dendrite might be no longer formed in Al-Zn alloy. While some recent results [8-10] were showed that the twinned dendrites could be prepared in below 40 wt. % Zn alloy, but no appeared above 40 wt. % Zn alloy, which became the bottleneck of the Al-Zn alloy preparation

Thus, in this work, to solve the bottleneck of the twinned dendrites preparation in Al-Zn alloy, we have selected Al-40 wt. %Zn alloy between 25 wt. % to 60 wt. % of Zn content and designed a new design method to obtain the high temperature gradient and strong convection. Using the inverted trapezoidal graphite insulating sleeve, twinned dendrites in high Zn content alloy with were successfully produced during directional solidification. Moreover, the microstructure of twinned dendrites was characterized, dendrite growth orientation was investigated and analysed by the electron back-scattered diffraction (EBSD) analysis method.

2. Experimental procedures

2.1. Materials and device

Al-40 wt.% Zn alloy used in this study was prepared in a vacuum induction melting furnace with purity aluminium (99.9 wt.%) and zinc (99.99 wt.%). The composition of the ingot measured by chemical titration is Al-40.4 wt.% Zn. The cast sample were enveloped in high purity graphite tube with a inner diameter of 7 mm and length 120 mm. Directional solidification experiments were carried out using a Bridgman vertical vacuum furnace described elsewhere [11]. In directional solidification process, the sample was heated by a graphite heater at 650ºC and then held isothermal for 15 min. Subsequently, the sample was moved downwards at 1000 and 1500 μm/s. In order to keep the solid/liquid interface, when the directional solidification distance reached 80 mm, the sample was quenched into a liquid Ga-In-Sn pool.

To obtain the higher temperature gradient and convection environment, we have improved the design of directional solidification experimental device shown in Fig. 1a. On the basis of conventional Bridgman directional solidification, we used the inverted trapezoidal graphite sleeve to hold the sample heated temperature higher before it was pulled into liquid metal. Thus the higher temperature gradient could be got. Meanwhile, because of well heat conduction and dissipation characteristics of the high purity graphite, in this work, the high purity graphite tube was used to obtain higher cooling rate and convection environment. To compare this design method, we have also done the other experiments without the inverted trapezoidal graphite sleeve at 1000 and 1500 μm/s shown in Fig. 1b.

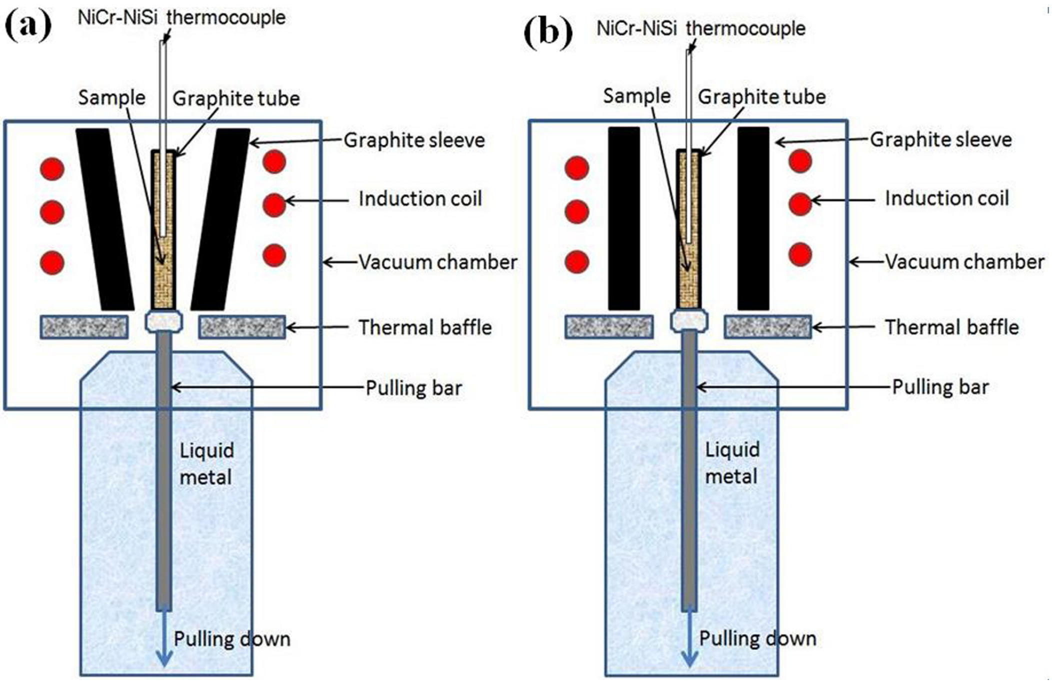

Fig. 1. Schematic diagram of two type of directional solidification experimental device: (a) with the inverted trapezoidal graphite sleeve, (b) without the inverted trapezoidal graphite sleeve

2.2. Characterization

The directionally solidified samples obtained in the experiments were then sectioned horizontally and vertically, respectively. And the specimens polished and etched using solvent Keller of H_2O (95 mL)+ HNO_3 (2.5 mL) + HCl (1.5 mL) + HF (1 mL) for about 10s. The scanning electron microscopy (SEM, JSM-6390A) were employed to photograph the specimens microstructures. The growth orientations were investigated by the electron back-scattered diffraction (EBSD) in scanning electron microscopy (SEM, Zeiss Supra 55) equipped with the Channel 5 EBSD system (HKL Technology-Oxford instrument).

3. Results and discussions

3.1. The temperature gradient measured in different devices

To explain higher temperature gradient by using the inverted trapezoidal graphite sleeve, in this experimental section, the temperature gradient (G) was measured using the NiCr-NiSi thermocouple at the pulling rates of 1000 μm/s with the inverted trapezoidal graphite sleeve and 1000, 1500 μm/s without the inverted trapezoidal graphite sleeve. Figure 1 also shows the schematic of the temperature gradient measurement in different devices and figure 2 is the experimental data in different devices and different rates. The NiCr-NiSi thermocouple was embedded inside middle of the crucibles, respectively. Under directional solidification process, the sample was preheated by a graphite heater at 650ºC for 20 minutes. Then the NiCr-NiSi thermocouple and the sample were moved downwards synchronously. At the same time, the temperature curves were measured by the thermometric instruments shown in Fig. 2. The thermal gradient was calculated by the Eq. (1):

$$G = \frac{T_L - T_S}{V \cdot t} \tag{1}$$

where T_L is the liquidus temperature, T_S is the solidus temperature, V is the pulling rate, and t is the time of the melt cooling from the liquidus temperature to the solidus temperature. By calculation, the thermal gradient used the inverted trapezoidal graphite sleeve was about 336 K/cm at 1000 μm/s, which is obviously higher than the value of 248 K/cm at 1000 μm/s and 231 K/cm at 1500 μm/s without the inverted trapezoidal graphite sleeve. This result proves that through using the inverted trapezoidal graphite sleeve, the higher temperature gradient could be got, which is good for obtaining the twinned dendrites.

3.2. Microstructure and orientation of twinned dendrites at 1000 μm/s

According to the design of directional solidification experimental device shown in Fig. 1, through the pulling rate at 1000 μm/s, higher temperature gradient and convection condition could be obtained. Then the twinned dendrites were finally prepared successfully in high Zn content Al-Zn alloy (40 wt.% Zn), as shown in Fig. 3. Figure 3a is the twinned dendrite microstructure in longitudinal section at directional solidification rate of 1000 m/s. It could be seen that the developed feather crystal structures of Al phase were formed inside the Al-Zn alloy [12]. The growth direction of twinned dendrite was consistent and clearly different form the other non-twinned dendrite. This is mainly due to the higher temperature gradient and strong convection environment by the inverted trapezoidal graphite sleeve and higher pulling rate. On the other hand, the growth direction of primary twinned dendrite trunk was not parallel to the pulling direction (the heat flow direction). And once the secondary twinned dendrite branch was formed, it would be grown faster than other non-twinned dendrite. Then

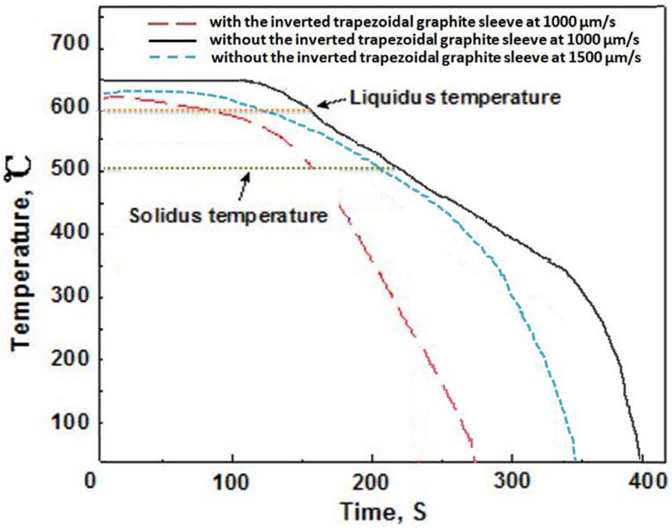

Fig. 2. The experimental data of temperature in different devices

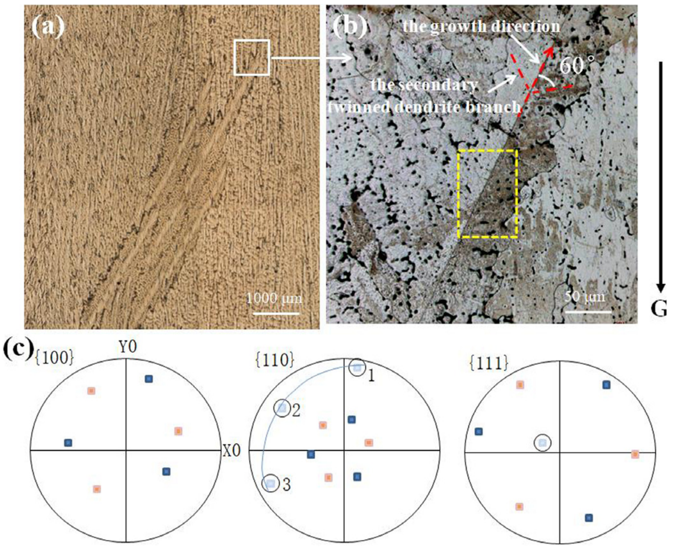

Fig. 3. Directionally solidified microstructures in longitudinal section and orientation of Al-40% Zn alloy at 1000 μm/s: (a) the twinned dendrite microstructure, (b) the enlarged map of white area in (a), and (c) pole figure of Al twinned dendrite

it grew quickly at high temperature gradient and convection environment. Figure 3b is the enlarged map of white area in (a). The symmetrical interface was the twinned interface and primary twinned dendrite was shown obviously, such as red dotted lines. Both sides of the secondary twinned dendrite branch were grew symmetrically along the twinned interface, and its growth direction was about 60° with the twinned interface. In addition, the twinned dendrites had obvious grain boundaries in Fig. 3b.

Through further micro EBSD analysis of the yellow region in Fig. 3b, it could be determined that the prepared dendrite was twinned dendrite, of which twinned interface was (111) plane. The primary and secondary twinned dendrite were grew along [110] direction [13,14] shown in Fig. 3c, which was in accordance with the results of Gonzales and Rappaz [9,10]. Through above analysis, it was found that under some special conditions, such as high temperature gradient and strong convection environment, the twinned dendrite in Al-Zn alloy with high Zn content was successfully obtained by directional solidification.

3.3. The deviation angle of twinned dendrites at 1000 μm/s

From the above analysis, we found that the growth direction of primary twinned dendrite trunk was not parallel to the pulling direction (the heat flow direction) shown in Fig. 3, and there had a certain deviation angle between these two directions. Thus the deviation angle of twinned dendrite was investigated and characterized by the EBSD analysis [15]. Figure 4 shows the EBSD maps in the transverse section, the corresponding (110)-pole figures and inverse pole figure of Al twinned dendrite at 1000 μm/s, respectively. The Al twinned dendrite could be observed in Fig. 4a, which was different from other non-twinned dendrite. The centre position of the (110)-pole figure was the heat flow direction of directional solidification (Fig. 4b), in which three poles were sited near the centre and circle.

Because of Al phase belonged to cubic crystal system, it was easily deduced that Al primary twinned dendrite had oriented with its [110] crystal direction [10,16], which was accord with the results of Fig. 3. And one pole near centre was not at the centre, which could be inferred that its [110] growth direction of primary twinned dendrite trunk was not parallel to the heat flow direction. Then the deviation angle between [110] direction of Al twinned dendrite and the heat flow direction was about 27.15° by testing. Meanwhile, in the inverse pole figure of Fig. 4c, the preferred growth orientation of Al twinned dendrite was also only its [110] direction, agreeing well with the results in Fig. 3.

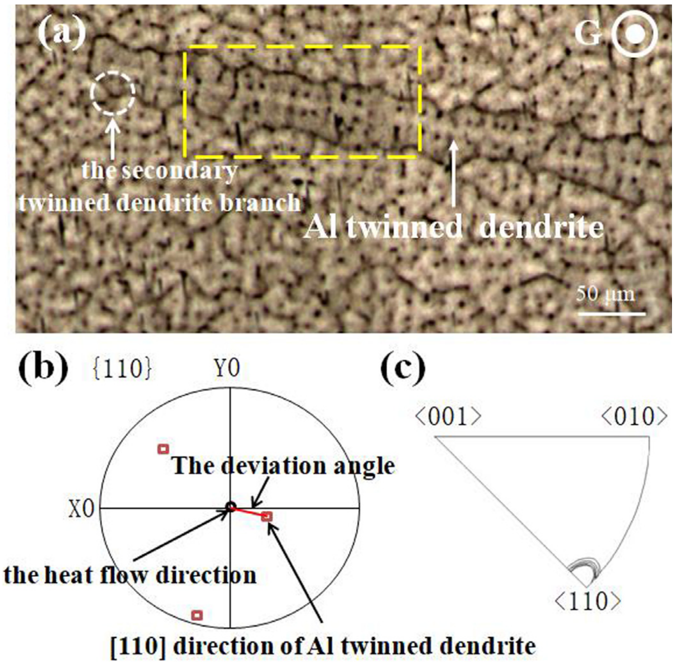

Fig. 4. The deviation angle of twinned dendrites at 1000 μm/s: (a) the Al twinned dendrite microstructure in transverse section, (b) the corresponding (110)-pole figure of Al twinned dendrite, and (c) the inverse pole figure of Al twinned dendrite

3.4. Formation conditions analysis of twinned dendrites

Due to the above results in this work, we considered that the formation conditions of twinned dendrites in Al-Zn alloy with high Zn content formed were higher temperature gradient and a certain pulling rate [17,18]. That is because the higher temperature gradient could lead the convection. Firstly, higher temperature gradient could provide a large temperature difference in the vertical direction to form the thermosolutal convection, which provided the formation condition for twinned dendrite formed. On the other hand, twinned dendrites have larger dynamic growth advantages than non-twinned dendrite. Once higher temperature gradient could provide higher temperature before solid-liquid interface. Then twinned dendrites would be formed and grown rapidly. The pulling rate also brought the thermosolutal convection inside the melt, and the pulling rate higher the convection degree more. In particular, in vertical directional solidification, because the densities of Al and Zn were different, with solidification rate increasing, it would cause the solute segregation inside the melt and led the thermosolutal convection. To a certain extent, it is beneficial for atom stacking along the twinned interface and promoting twinned dendrites formed. But the convection degree caused by the pulling rate was not the larger the better, because the larger thermosolutal convection would make the solid-liquid interface instability and resulted in dendrite remelting, destroying and affecting the twinned dendrite tip forming. Moreover, the solidification time was too short at higher pulling rate, then the twinned dendrites would be grown rapidly, which limited the twinned dendrites growing up. Therefore, a certain pulling rate is advantageous for the formation and growth of twinned dendrites in Al-Zn alloy with high Zn content.

3.5. Microstructure and orientation of non-twinned dendrites at 1000 μm/s

The above results were indicated that higher temperature gradient and convection environment were the formation conditions of twinned dendrites of Al-Zn alloy with high Zn content. So to prove the role of the inverted trapezoidal graphite sleeve, the experiment without the inverted trapezoidal graphite sleeve at 1000 μm/s had also been done shown in Fig. 5. Figure 5a is the non-twinned dendrites microstructure in longitudinal section at directional solidification rate of 1000 μm/s [19]. It could be seen easily that it were non-twinned dendrites and there were not the developed feather crystal structures formed inside the Al-Zn alloy. The dendrites were all grew regularly along the heat flux direction, which was perpendicular to the quenching interface. In addition the angle between primary and secondary dendrite branch was not 60° in Fig. 5b. The results of EBSD analysis showed that the growth direction of primary dendrite was [100] direction not [110] direction. And the twinned interface was not appeared, as shown in Fig. 5c. Then through the above analysis, it can be determined that without the inverted trapezoidal graphite sleeve at 1000 μm/s the twinned dendrites could not be prepared.

3.6. Microstructure and orientation of non-twinned dendrites at 1500 μm/s

In this work, we continue to increase the solidification rate to see whether twinned dendrites could be obtained by solidification rate increasing. Figure 6a is the non-twinned

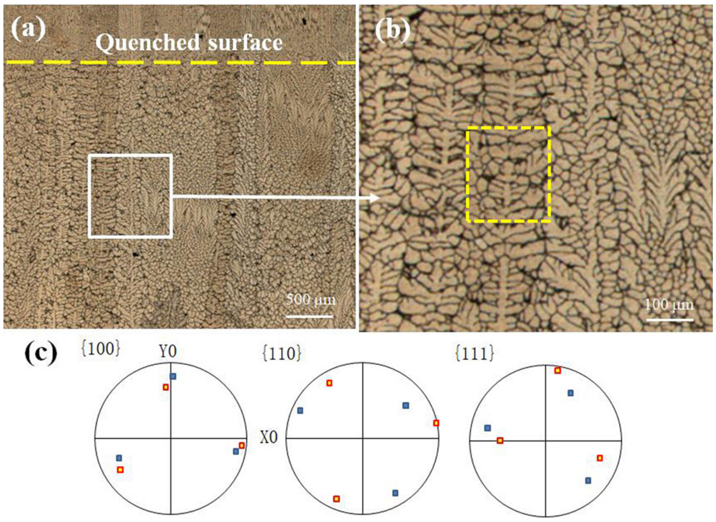

Fig. 5. Directionally solidified microstructures in longitudinal section and orientation of Al-40% Zn alloy at 1000 μm/s without the inverted trapezoidal graphite sleeve: (a) the non-twinned dendrite microstructure, (b) the enlarged map of white area in (a), and (c) pole figure of Al non-twinned dendrite

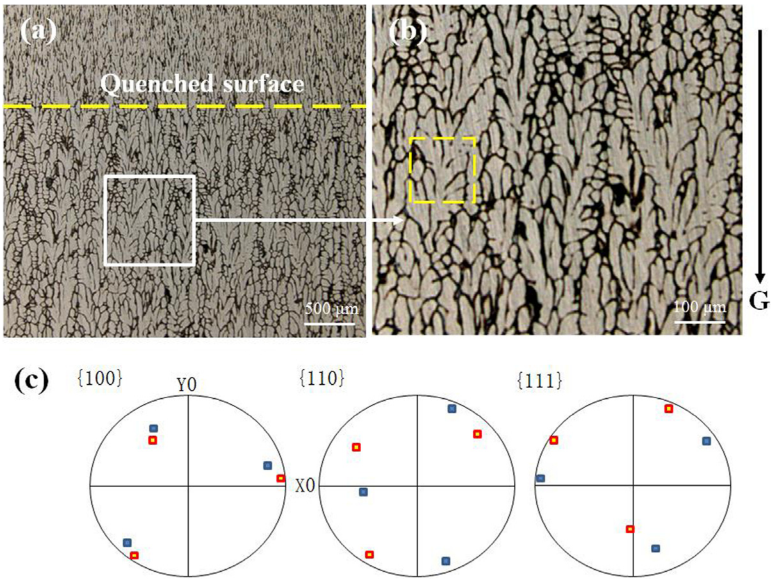

Fig. 6. Directionally solidified microstructures in longitudinal section and orientation of Al-40%Zn alloy at 1500 μm/s without the inverted trapezoidal graphite sleeve: (a) the non-twinned dendrite microstructure, (b) the enlarged map of white area in (a), and (c) pole figure of Al non-twinned dendrite

dendrites microstructure in longitudinal section at directional solidification rate of 1500 μm/s without the inverted trapezoidal graphite sleeve. There were also not the developed twinned dendrite appeared. The non-twinned dendrites were all grew regularly along the heat flux direction too. Seen from Figure 6c, the growth direction of primary dendrite was [100] direction not [110] direction. So we considered that it is not possible to increase solidification rate blindly to get twinned dendrites. The higher temperature gradient, a certain pulling rate and convection environment were the formation conditions of twinned dendrites.

In this work, using the inverted trapezoidal graphite insulating sleeve, twinned dendrites in Al-Zn alloy with high Zn content (40 wt.%) were successfully produced during directional solidification. The results showed that higher temperature gradient, a certain pulling rate and convection environment were the formation conditions of twinned dendrites of Al-Zn alloy with high Zn content.

4. Conclusions

Twinned dendrites in Al-Zn alloy with high Zn content (40% wt.%) were successfully prepared by using the inverted trapezoidal graphite insulating sleeve during directional solidification. The higher temperature gradient, a certain pulling rate and convection environment were the formation conditions of twinned dendrites. The developed feather crystal structures of Al phase were formed inside the Al-40% Zn alloy. Its primary and secondary twinned dendrite were grew along [110] direction. Moreover the deviation angle between [110] direction of Al twinned dendrite and the heat flow direction was about 27.15°. A brief description for formation conditions of twinned dendrites was given. While the other non-twinned dendrites formed in other pulling rate without the inverted trapezoidal graphite sleeve was consistent and clearly different form Al twinned dendrite.

Acknowledgments

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