Original Article

Orientation controlling of Ni-based single-crystal superalloy by a novel method: grain selection assisted by un-melted reused seed

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A new grain selection method assisted by un-melted short seed was proposed to control the primary and secondary orientations of Ni-based single-crystal superalloy. The results showed that the use of short seeds could effectively avoid the formation of the melt back mush zone. Although a few stray grains were formed at the upper of the short seed because Al2O3 and SiO2 particles might act as nucleation sites, an expected grain with the smallest misorientation was predominant in starter block. Finally, the orientations of the Ni-based single crystal superalloy component could be not only well controlled with a smaller misorientation about 2.1°, but also a paper-thin substance mixed Al2O3 and SiO2 particles was partly covered with the surface of the un-melted short seed, which made this seed convenient to separate and reuse resulting in the reduction of cost.

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1. Introduction

Ni-based single crystal superalloy is widely used as aircraft and power generation turbine engines blades ascribing to its excellent high temperature mechanical properties [1]. Because of the anisotropy of mechanical properties, the ⟨001⟩ direction as the primary orientation needs to be controlled paralleling with the load direction to obtain the maximize low-cycle fatigue life during thermal cycling [2].

Generally, in order to get the ⟨001⟩ preferred orientation for preparing Ni-based single crystal superalloy component during directional solidification, two basic ways are always used: grain selection and seeding technique. Grain selection method is used to obtain Ni-based single crystal superalloy by the competitive growth between grains in grain selector constituted by the starter block and the spiral passage [3–5]. It is convenient and economical to prepare Ni-based single crystal superalloy components within 15° off the ⟨001⟩ orientation [6]. However, the low accuracy for primary orientation control

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and the incapable of secondary orientation control prohibit the wide use of grain selection method in some circumstances where the accurate orientation is strictly required for properties assurance [7]. Therefore, the seeding method is further introduced [8], that a pre-fabricated seed with the desired orientations located at the bottom of mold is partly melted back during the soak period and subsequently transfers its orientations to a new grain [9]. By seeding method, both of the primary orientation and the secondary orientation can be accurately controlled to obtain the better mechanical properties [10,11]. However, two defects are always unavoidable for the seeding method. Firstly, it is expensive to fabricate expectedly defect-free oriented seed. Secondly, the success rate for the production of Ni-based single crystal superalloy components is low because the stray grains are always formed at the melt-back region. Although some efforts were attempted to improve the success rate of Ni-based single crystal superalloy components, for example, a “pig tail” method could be introduced between the seed and the component to eliminate stray grains [12], this improvement increased the production cost of Ni-based single crystal superalloy components and limited the wide application of seeding method as an important factor.

In order to overcome the disadvantages of grain selection and seeding methods mentioned above, a novel grain selection method assisted by un-melted short seed was proposed to control the orientation of Ni-based single crystal superalloy component. By means of this method, both of required primary and secondary orientations not only could be well controlled, but also the production cost of Ni-based single crystal superalloy component was approximately equal to that of traditional grain selection method. The detailed mechanism was further analyzed and the corresponding microstructures were characterized using the metallurgical microscope and the scanning electron microscopy (SEM) with an energy dispersive spectroscopy (EDS) and an electron backscatter diffraction (EBSD).

2. Experimental procedure

The material used in this work was a third-generation single crystal superalloy DD33, the nominal composition was listed as shown in Table 1. An upper surface of cylinder-shaped short seed with (001) orientation and 4 mm height was polished and etched by a solution of 14% HNO₃, 28% HF and 58% H₂O₃ for observing microstructure. Then, the short seed was plugged into the mold from the opening bottom in a modified Bridgman furnace, and the etching surface was upper. The short seed was rotated so that its secondary orientation was consistent with that of the required Ni-based single crystal superalloy component. Then, the mold with seed was mounted on a water-cooled copper chill plate in a modified Bridgman furnace. During directional solidification, the furnace chamber was evacuated to a partial pressure of approximately 10⁻³ bar, and the ceramic mold with seed was preheated to 1550 °C for 10-20 min by graphite heating component. After the ingot was melted, the liquid melt was poured into the preheated mold cavity at 1500 °C and held for 10-20 min to stabilize. Finally, the ceramic mold was withdrawn from the furnace at a pre-determined withdrawal rate of 100 μm/s. More details about this directional solidification process could be given in Ref. [12]. In order to avoid damage to the seed surface, the mold was carefully removed from the casting body after withdrawing. Then, the upper surface of seed was washed by the ultrasonic to eliminate the floating dust. Leica DM-4000 optical microscope and ZEIS SUPRA 55 SEM equipped with EDS and EBSD were used to observe the microstructures of the samples and seeds.

3. Results and discussion

Removing the shell, it was found that the short seed with 4 mm height was naturally departed from the casting without any extra loading force. The image was showed in Fig. 1a and b was the macroscopic morphology on the upper surface of seed, showing that it was partly covered by a paper-thin
substance. In order to confirm the composition of this paper-thin substance, its microstructure was further observed by SEM as shown in Fig. 1c. It was clear that two different contracts could be clearly presented on the upper surface of seed, marked point A and point B. EDS was used to analyze the compositions of point A and point B, and the results were shown in Table 2. It was seen that a large portion of O, Al and Si elements were enriched at point A, indicating that this paper-thin substance was mainly mixture of Al₂O₃ and SiO₂ particles, which might comes from the heated shell. And, the matrix element Ni was the main composition of point B, indicating that the upper surface of seed was partly exposed.

Based on the SEM/EDS analysis results in Fig. 1 and Table 2, it was obvious that there were some Al₂O₃ and SiO₂ substances on the upper surface of seed. Therefore, after pouring, these Al₂O₃ and SiO₂ particles directly contacted the molten Ni melt. It is well known that the Al₂O₃ particle has a melting point of about 2054 °C, and the corresponding crystal structure is hexagonal close packing (HCP) structure with R-3c space group and has a = 0.475 nm, c = 1.299 nm lattice parameter. And, Ni has a melting point of about 1453 °C and a face-centered cubic (FCC) crystal structure. Similar to the TiB₂ (HCP structure)-Al (FCC structure) system in Al alloys where the TiB₂ particles acted as the nucleation sites of Al grains [13], these Al₂O₃ particles might have a similar HCP–FCC orientation relationship with Ni grains: \([110]_{\text{al}} // [110]_{\text{Ni}}, \{0001\}_{\text{al}} // \{111\}_{\text{Ni}}\). Therefore, the corresponding lattice misfit between Al₂O₃ particles and Ni grains could be calculated as about only 4.7%. In addition, for the other oxide (SiO₂ particles) on the upper surface of seed, its melting point is about 1650 °C and the corresponding crystal structure is FCC and has a = 0.716 nm lattice parameter. Similar to the MgAl₂O₄ (FCC structure)-Al system where MgAl₂O₄ particles could act as the nucleation sites of Al grains [14], the SiO₂ particles might have also a cubic–cubic orientation relationship with Ni grains: \([110]_{\text{SiO}_2} // [110]_{\text{Ni}}, \{111\}_{\text{SiO}_2} // \{111\}_{\text{Ni}}\). Consequently, the lattice misfit between SiO₂ particles and Ni grains could be calculated as about only 1.56%.

Based on the analysis above, it was clear that the misfits of Al₂O₃/Ni and SiO₂/Ni systems were well below the limitation given for a good nucleation agent, which was about 12% suggested by Bramfitt [15]. It indicated that the nucleation barriers of Al₂O₃ and SiO₂ particles were lower for Ni grains. Therefore, when the molten Ni-based superalloy melt was poured into the shell and contacted this paper-thin substances on the upper surface of seed, these Al₂O₃ and SiO₂ particles might act as the nucleation sites of Ni grains, resulting in the formation of some stray grains. Simultaneously, it should be noted that, due to the existence of this oxide–metal interface, the short seed was easily peeled off from the Ni-based single crystal superalloy component after directional solidification. Generally, when the grain selection technique was used to produce Ni-based single crystal superalloy component, many random grains always nucleated at the bottom of starter block [16,17]. As the alloy melt poured into the casting cavity, it was rapidly cooled because the heat was conducted to a cooler seed. Once the melt temperature was lower than the liquidus temperature, a part of melt which contacted the upper surface of exposed seed could directly epitaxially grow from the seed with a same orientation. However, the other part of melt which contacted this paper-thin Al₂O₃ and SiO₂ substances might act the nucleation sites of Ni grains and grew up to form some stray grains once the required undercooling was reached during directional solidification. Therefore, the cross-sectional microstructure at the bottom of starter block with the short seed was observed and the corresponding crystal orientations were analyzed by means of EBSD technology. The results were displayed in Fig. 2. It was found that 11 grains were nucleated at the bottom of starter block as shown in Fig. 2a. Fig. 2b displayed the crystallography orientations of these nucleated grains in the form of an inverse pole figure. It was obvious that Grain A had a roughly same crystallography orientation with the original seed, which was closed to \((001)\) orientation, indicating that Grain A might directly grow epitaxially from the exposed surface of the short seed. However, the orientations of other 10 grains were randomly distributed in the inverse pole figure, which should nucleate on the paper-thin Al₂O₃ and SiO₂ substances. It was clear that Grain A was the favorably orientated grain while the other 10 grains were the unfavorably orientated grains, and Grain A occupied the maximum size with about 41.28% area fraction as shown in Fig. 2.

Generally, the single crystal selected by grain selector originated from the grain located at the bottom of starter block. Therefore, Esaka et al. [3] believed that a main function of starter block was to obtain well-oriented \((001)\) texture. Previous research had indicated that when the withdraw rate was set as 100 μm/s, the favorably oriented grain would present a competitive advantage to eliminate the other unfavorably oriented grains during directional solidification [18]. Therefore, the favorably oriented Grain A with the smallest misorientation in Fig. 2 was predominant in the microstructure and its area would be bigger and bigger at starter black. Consequently, Grain A was highly likely to occupy the inner wall of the grain selector where the grain was usually selected as a final single crystal. Finally, the orientation of obtained Ni-based single crystal superalloy component was possible to present the same orientation with Grain A. Fig. 3 was the orientations of Ni-based single crystal superalloy component and the used short seed in the inverse pole figures, showing that the crystal orientation of Ni-based single crystal superalloy component was basically consistent with that of this used short seed. However, it should be noted that the final orientation of selected single crystal had still a little deviation from the orientation of pre-set seed with a misorientation about 2.1° in the growth direction as shown in Fig. 3. The reason might
be that the dendrite which laterally grew at the paper-thin mold materials was possible to bend downwards as the solidification time elapsed as a result of the thermal loading from the thermal contraction between mold materials and metal \[19\], and a random misorientation might occur as a successive dendrite branching in grain selector \[20\].

As it was known that a melt back mush zone always occurred at the upper surface of seed because the temperature here was slightly lower than that of molten melt. Generally, the temperature at the upper surface of seed should be positively related to the height of seed. The lower the height of seed was, the larger the temperature difference between the seed and the melt was, which resulted in a larger cooling rate at the upper surface of seed. Therefore, three seeds with different heights were used to further clarify the advantage of short seeds in this method. Polishing these seeds after directional solidification, the cross-sectional microstructures at the upper surface of seeds with 4 mm, 10 mm and 15 mm heights were showed in Fig. 4, where the microstructure of the original seed was also presented in Fig. 4a. It was clear that the microstructures of these seeds after preparing Ni-based single crystal superalloy components had no obvious difference with that of the original seed (Fig. 4a), when the heights of seeds were 4 mm and 10 mm where the area fraction of γ/γ′ eutectic still remained at about 4.5% as shown in Fig. 4b and c. However, when the seed height was set as 15 mm, the γ/γ′ eutectic had basically been dissolved because the holding time has more than 50 min (holding temperature for 30 min prior to homogenizing for 20 min) in a thermal region as shown in Fig. 4d. However, it was clear that the primary dendrite spacing of all seeds with different heights were basically consistent with that of the original seed in microstructure, indicating that the upper surface of these seeds were basically not dissolved during directional solidification. Unlike the traditional seeding method where the upper portion of seed was partly melted to form a melt back mush zone during the holding temperature period, this re-melting phenomenon and the melt back mush zone did not occur in all used seeds in current study. A main reason should be that the height of the used seeds was sharply below than that of the traditional seed (about 50 mm height).
Fig. 4 – Cross sectional optical microscopy images of seeds. (a) Original microstructure and microstructures after experiment with different seed height 4 mm (b), 10 mm (c) and 15 mm (c).

[12], and then the upper surface of seeds was not heated to the solidus temperature. In other words, if the height of seeds was too high, the melt back mush zone would be inevitable to be presented resulting in the formation of some stray grains.

Using the traditional grain selection method, the crystallographic (001) direction of final Ni-based single crystal superalloy component could be controlled in misorientation angle of 15° by optimizing the size of the grain selector and process parameter [6]. However, the secondary orientation of final Ni-based single crystal superalloy component was random. In this new developed method, because the final Ni-based single crystal superalloy component epitaxially grew from the original seed, its primary and secondary orientations were well controlled with a small misorientation angle despite that the result of the secondary orientation controlling was not revealed here. In addition, a natural separation between the seed and the Ni-based single crystal superalloy component made this un-melted short seed convenient to reuse after polishing and etching the upper surface resulting in the reduction of cost. In a word, the grain selection method assisted by un-melted short seed could control the crystallographic orientation of final Ni-based single crystal superalloy component without an obvious increase of cost.

4. Conclusion

A novel grain selection method assisted by un-melted short seed was proposed to control the orientations of Ni-based single-crystal superalloy component. Some results were summarized as follows:

(1) The use of short seeds could effectively avoid the formation of the melt back mush zone. Although a few stray grains were still formed at the upper of the un-melted seed because Al₂O₃ and SiO₂ particles from the shell materials acted as nucleation sites, an expected grain with the smallest misorientation and the largest area was still predominant in the microstructure in starter block. It might further present a competitive advantage to eliminate the other unfavorably oriented grains during directional solidification to occupy the inner wall of the grain selector as a final single crystal.

(2) Compared with the traditional grain selection method, the primary and secondary orientations of the final single crystal component could be not only well controlled with a smaller misorientation, but also a paper-thin substance mixed Al₂O₃ and SiO₂ particles partly covered the surface of the short seed, which made this seed convenient to separate and reuse resulting in the reduction of cost.

Conflicts of interest

The authors declare no conflicts of interest.

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