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ARTICLE

Homogeneous Microstructure of Bulk K4169 Superalloy Obtained by Stable Undercooling

Zhang Keren[1,2], Xie Faqin[1], Hu Rui[2], Li Jinshan[2], Wu Xiangqing[1]

[1] *School of Aeronautics, Northwestern Polytechnical University, Xi'an 710072, China;* [2] *State Key Laboratory of Solidification Processing, Northwestern Polytechnical University, Xi'an 710072, China*

Abstract: By improving the method of B_2O_3 glass purification combined with cycle superheating based on traditional undercooling method, a stable undercooling above 200 K of 100 g bulk K4169 superalloy was obtained, and the largest undercooling went to 271 K. The effects of purification and superheating temperature on undercooling have been discussed. Moreover, the three-dimensional numerical analysis of temperature field inside the undercooled melt has been presented, and the maximum temperature deviation in different parts is only 14 K before nucleation. In contrast with the as-solidified microstructure, the grain size will increase with the increment of nucleating temperature. Generally, the difference of microstructure in 100 g undercooled K4169 superalloy is tiny and the average grain size of the superalloy with 271 K undercooling is 12±2 μm.

Key words: K4169 superalloy; undercooling; purification; temperature field; microstructure; homogeneity

The microstructure of undercooled melt after rapid solidification is one of the most important fundamental subject in various materials [1-3]. The so-called undercooling refers to the phenomenon of pure liquid metal or alloy below a certain temperature range without crystallization or solidification. Molten glass purification combined with cycle superheating is one of the methods which can achieve reasonable undercooling successfully [4-6].

To date, high undercooling in bulk alloys has been widely investigated [7,8]. Previous researches only focused on the relationship between superheating, holding time and undercooling. However, some details of the undercooling process have not been studied systematically yet, such as the replacement of purification and the way of cycle superheating, and thus the undercooling degree is obtained randomly. Furthermore, with the increase of melt size, the melt needs longer time to cool down and the external part cools faster than the center. So the inhomogeneous temperature field inside the undercooling melt leads to the forming of inhomogeneous microstructure [9].

As one of the most widely used superalloys in modern industry, K4169 superalloy shows outstanding performance in elevated temperature environment, and it's widely used as the critical structural components in gas-turbine engines. Therefore, lots of investigations have been carried out for a better understanding on K4169 superalloy [10-13] and the research of undercooled solidification of K4169 is indispensable. In the present work, the high undercooling of K4169 superalloy has been achieved by a method of replacement of molten glass combined with optimized cycle superheating. And the temperature field in the cooling process of large size melt was simulated to figure out the way how the temperature distribution affects the uniformity of microstructure.

1 Experiment

The undercooling experiment was conducted by a glass purification method in a high frequency induction unit with a coil mounted in atmosphere. The glass flux was B_2O_3 which was dehydrated at 1073 K for 5 h in advance. In each

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Corresponding author: Hu Rui, Ph. D., Professor, State Key Laboratory of Solidification Processing, Northwestern Polytechnical University, Xi'an 710072, P. R. China, Tel: 0086-29-88491764, E-mail: rhu@nwpu.edu.cn

experiment, a bulk K4169 superalloy of 100 g was placed in a quartz crucible of 20 mm diameter and covered with B_2O_3 powder on the top and the bottom. Then the crucible was placed in the coil of the high frequency induction unit. Firstly the sample was heated to 773~873 K until completely enwrapped by the molten B_2O_3. Then the temperature increased to 1821~1881 K which was 200~260 K above the liquidus temperature (T_L) and held for 3~5 min. After that the temperature decreased to T_L+30 K and held for 3~5 min. The process was cycled 2~3 times to generate different undercooling.

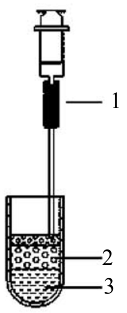
In each cycle, the negative pressure suction device was used to replace the molten B_2O_3, as illustrated in Fig.1. The device can extract molten B_2O_3 from the upper edge of the quartz crucible, following by addition of same amount of fresh B_2O_3. When the interface between alloy melt and B_2O_3 was stable and there were no air bubbles, it was time to stop heating. The thermal behaviors of samples were monitored by a double-color pyrometer with ±5 K accuracy and 100 ms response time.

The solidified samples were cut along the longitudinal section, polished and etched using a solution of 10 g $CuCl_2$ +100 mL HCl +100 mL alcohol. The as-solidified microstructures of 5 parts on the longitudinal section were observed by Olympus PMG3 metallographic microscope (OM), which are marked by a, b, c, d and e as shown in Fig.2. The B_2O_3 was analyzed by X-ray diffraction (XRD).

2 Results and Discussion

2.1 Influencing factors of purification

2.1.1 Influence of B_2O_3 on purification



1-negative pressure suction device, 2-molten B_2O_3, 3-alloy melt

Fig.1 Principle diagram of negative pressure suction device

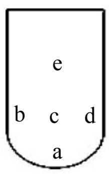


Fig.2 Longitudinal section of the superalloy

The selection of purification agent is very important for achieving high undercooling[14]. The reason why B_2O_3 is widely used can be ascribed to that the molten B_2O_3 in amorphous state won't provide heterogeneous nucleus, meanwhile the sticky molten B_2O_3 can eliminate heterogeneous nucleation particles through physical absorption, and it can also melt some impurities through chemical reaction to achieve physicochemical purification [15]. In addition, the molten B_2O_3 can not only protect the melt from atmosphere to avoid the formation of oxide film, but also work as a high-damping isolation layer to eliminate the interference from external vibration. However, the amorphous B_2O_3 will be crystallized at high temperature by absorbing large numbers of metal ions such as Fe^{2+} and Fe^{3+} in alloy melt, and the viscosity of molten B_2O_3 will be decreased. To some extent, the crystallization of B_2O_3 provides heterogeneous nucleuses which may lead to the large decrease of undercooling [16]. Fig.3 shows the XRD patterns of B_2O_3 after and before the undercooling experiment. It can be seen that a crystallization peak appears obviously in the used B_2O_3, however, in the unused B_2O_3 there appears a broad diffraction peak which is a typical symbol of amorphous state. It suggests that B_2O_3 will be seriously crystallized after participating the purification for a long time. Thus, it is not sufficient to add B_2O_3 in bulk alloys only once during the undercooling experiment and it is indispensable to replace the molten B_2O_3 by adopting the negative pressure suction device as shown in Fig.1.

2.1.2 Influence of superheating on purification

Normally it is difficult to get a desirable undercooling by superheating only once, and thus the method of cycle superheating is usually adopted, which means that the alloy is superheated for a few minutes and then cooled down till solidification in each cycle. Nevertheless, this kind of cycle superheating is not most suitable, due to the fact that the alloy will be segregated with B_2O_3 during solidification and exposed to the air during the process of reheating. In order to avoid the oxidation, the temperature of alloy melt should be cooled to T_L+30 K for 1~3 min and then reheated to

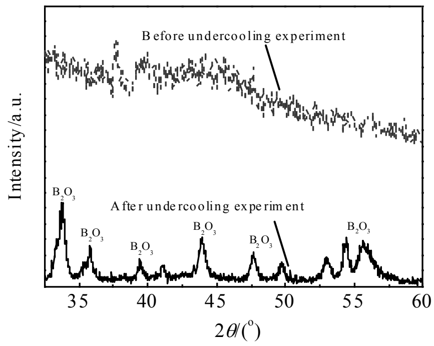


Fig.3 XRD patterns of B_2O_3 before and after undercooling experiment