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Competitive converging dendrites growth depended on dendrite spacing distribution of Ni-based bi-crystal superalloys

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ABSTRACT

The microstructure evolution and the dendrite spacing distribution were characterized by the minimum spanning tree method to investigate the relationship between the competitive converging dendrites growth and the dendrite spacing distribution in Ni-based bi-crystal superalloys with different withdrawal rates. The results indicated that the overgrowth was closely related with the dendrite spacing distribution determined by the withdrawal rate. As a selection criterion, the relative spacing at the grain boundary with respect to the spacing inside the favorable oriented (FO) grain had been identified. In particular the FO grain had been overgrown if its minimum spacing at the grain boundary had been smaller than the minimum spacing inside the FO grain. On the contrary the FO grain overgrew the unfavorable oriented (UO) grain if the maximum dendrite spacing at the grain boundary was larger than that inside the FO grain. Furthermore, the overgrowth speed was correlation with the ratio between the number of dendrite spacings at the grain boundary out of the stable range inside the FO grain and the total number of dendrite spacings at the grain boundary. The mechanism of competitive converging dendrites growth was experimentally validated that the dendrite spacing was adjusted to a stable range by the development of new FO dendrites or the elimination of existing FO dendrites.

1. Introduction

Ni-based single crystal (SX) superalloy is widely used as aircraft and power generation turbine engines blades ascribing to its excellent high temperature mechanical properties [1]. In order to successfully obtain SX component, the crystallographic orientation and defects need to be strictly controlled, which is actually the competitive dendrites growth. Therefore, the competitive dendrites growth has been one of the priorities for both theoretical and experimental investigation for decades [2–4]. The competitive growth is generally divided into converging growth and diverging growth. For diverging growth, it is widely accepted that favorable oriented (FO) dendrite overgrow unfavorable oriented (UO) dendrite during directional solidification [3–5]. However, in the case of converging growth, the overgrowth is still under controversy up to now [6–8].

In 1959 Walton and Chalmers first proposed a model based on the analysis of the difference in dendrite tip undercooling [3], which was schematically summarized by Rappaz et al., in 1994 [9,10], indicating that UO dendrite was blocked by FO dendrite for converging dendrites. In 2008, Zhou et al. found that UO dendrite was able to overgrow FO dendrite at low withdrawal rate caused by interdendritic solute interactions [4,11,12]. Using phase field simulation in two dimension (2D) and in-situ observation of transparent organic film experiment in 2012, Li et al. proposed a criterion for the unusual overgrowth of FO dendrite [7,13]. The unusual overgrowth only happened when the spacing between the FO dendrite at the grain boundary and the next FO dendrite was decreased to a certain level. This was confirmed by Takaki et al., in 2014 [14], who also showed the unusual overgrowth was a common phenomenon in metallic materials. According to Tourret' report in 2017 [15], the secondary orientation, defined as the azimuthal angles of rotation of the dendrite around its primary axis, could weakly influence the competitive converging dendrites growth in thin-sample directional solidification. However, a recent

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study showed that the needed critical withdrawal rate for bi-crystal coexisting was strongly affected by the azimuthal rotations in 3D space, mainly attributing to the dendrite insertion phenomenon which was ignored long time in 2D space [16]. Therefore, the mechanism of competitive converging dendrites growth may be different between 2D and 3D space. In despite of it was successfully used to explain the competitive converging dendrites growth [16,17], the direct evidence of the selection criterion proposed by Li et al. was still absent in 3D space.

A finite stable dendrite spacing range had been confirmed over the past few decades [18,19]. However, the traditional approach based on the average area was insufficient for capturing the dendrite spacing distribution. Some efforts had been performed to find a new method for accurately and conveniently characterizing the distribution of dendrite spacing [20,21]. The minimum spanning tree (MST), which was proposed by Dussert et al. to study the order and the disorder in a distribution of points [22], was becoming a commonly used approach in analyzing the range of the dendrite spacing [20–24]. The MST is a connected curve containing all dendrite centers without any closed loop, for which the sum of the edge weights is minimum. When it is applied to analyze the range of primary dendrite spacing, the MST branch length distribution is used to represent the primary dendrite spacing distribution. For example, by applied this MST method, Peng et al. had found that the primary dendrite spacing range was closely related with the growth history [25].

In this work, the competitive converging dendrites growth of Ni-based bi-crystal superalloy at different withdrawal rates were analyzed, then the dendrite spacing distributions at the grain boundary and in grain interior were characterized to clear the relationship between the dendrite spacing distribution and competitive growth and further to discuss the mechanism of competitive converging dendrites growth in 3D space.

2. Experiment

2.1. Experimental procedure

A third generation Ni-based single crystal superalloy was used in this study. The nominal composition of this alloy consisted of Ni-3.44Cr-4.0Re-7.8Ta-8.93Co-1.53Mo-5.75W-6.0Al-0.2Ti (wt. %). The single-crystal seeds with specific orientation were cut into half-cylinders, marked as Seed A and Seed B, with 4 mm diameter × 20 mm height, which were put together in the case of converging grains. One Seed A was a FO grain, which the secondary orientation deviated from the grain boundary 20°, another Seed B was a UO grain, where the mis-orientation of $\langle 001 \rangle$ from sample axis was 20°. Solidification of bi-crystal samples were carried out at different withdrawal rates (50, 100, and 200 μm/s) under a constant temperature gradient (~170 K/cm) in a modified Bridgeman-type furnace. The detail description could be found in Ref. [16]. Following directional solidification, the samples were transversally sectioned, polished, and etched with a solution of HNO_3, HF and H_3O_8 (volume ratio 1:2:3) for further analysis. The microstructure of the samples was observed with optical microscopy (DM-4000M; Leica, Berlin, Germany). A professional image analysis software (Image Pro Plus 6.0) was used to measure the grain area and capture the position of dendrite centers.

2.2. Typical statistical analysis process of the MST method

To carry out the statistical analysis on the dendrite spacing distribution using the MST method, there were three steps should be done: the identification of the centers of dendrites, obtaining the MST branches, and then study of the distribution of dendrite spacing respected by the MST branch. Since the competitive growth was mainly affected by the position of dendrite tip, the dendrite spacing in this study specially represented the spacing between neighboring dendrite tips. The bi-crystal sample, which was directional solidified at the withdrawal rate of 50 μm/s, was chosen as an example to clearly illustrate the typical analysis process of dendrite spacing distribution. The transverse view at a distance of 5 mm from the re-melt interface was presented in Fig. 1a. Fig. 1b showed the distribution of the dendrite centers. The X-Y coordinates of the dendrite centers were obtained by manually capturing them on the magnified images of each dendrite assisted by Image Pro Plus software. Fig. 1c displayed the corresponding MST realized by a *Prim* algorithm in Matlab software, which represented the shortest total length of the branches to connect all nodes in Fig. 1b. It was clear that dendrite distribution was non-uniform in cross section as shown in Fig. 1b-c. The average value of the branch lengths (λ) and the corresponding standard deviation (σ^*) obtained from the MST were used to provide a statistical measurement of the nearest-neighbor distribution of dendrites. Fig. 1d showed the frequency distribution of dendrite spacings corresponding to the MST map shown in Fig. 1c and a Gaussian fit through the data, where the frequency (F) was given by:

$$F = A_0 \exp\Big(-0.5((X - A_1)/A_2)^2\Big) \quad (1)$$

Where A_0 was the amplitude, A_1 was the center position, and A_2 was the width of the fit curve. The r^2 parameter (correlation coefficient) of the Gaussian fit was 0.9775. Therefore, the experimentally determined dendrite spacing distribution showed a good fit to the normal distribution. It should be noted that it also could be fitted well using Gaussian two-peak function or Gaussian three-peak function with corresponding to the r^2 parameter of 0.999 and 0.995, respectively. However, it was difficult to find the relationship between distribution of dendrite spacing and competitive growth using this common analysis method. Therefore, in the next section, the dendrite spacing distribution would be analyzed in grain interior and at the grain boundary, respectively.

3. Results

3.1. The competitive growth process

Fig. 2 showed the microstructure evolution of the Ni-based bi-crystal samples with different withdrawal rates during directional solidification. The black solid lines indicated the grain boundaries of bi-crystal, and white dot lines indicated the original positions of the grain boundaries at re-melt interface. The inset was a schematic diagram showing the orientation relationship between the FO Seed A and the UO Seed B. When the withdrawal rate was set as 50 μm/s, the grain boundary gradually moved from middle position towards Grain A so that both the area of Grain A and the number of FO dendrites in cross section were decreased as shown in Fig. 2(a1~a3). It indicated that the FO Grain A was overgrown by UO Grain B during directional solidification. When the withdrawal rate was increased to 100 μm/s, the corresponding microstructure evolution of bi-crystal sample was displayed in Fig. 2(b1~b3). The motion behavior of the grain boundary became complex, where the grain boundary not only moved towards Grain A, but also a part of the grain boundary approached Grain B. It could be concluded that not only FO dendrites were overgrown by UO dendrites on one position, but also UO dendrites were overgrown by FO dendrites on the other position simultaneity. Furthermore, when the withdrawal rate was reached to 200 μm/s, the grain boundary mainly moved towards Grain B resulting in the increase of both the area of Gran A