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# Influence of crystal orientation on cellular growth of a nickel-base single crystal superalloy

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ABSTRACT

The cellular pattern evolution during directional solidification of a nickel-base single crystal superalloy has been studied in different crystallographic orientations using re-oriented seed crystals. Under the same thermal gradient and solidification velocity, the microstructures of differently oriented cellular single crystals are schematically investigated. It is concluded that the cellular growth direction is less affected by the seed orientation and depends on the heat flow, and is usually along heat flow direction. Cellular interface stability and microstructure are greatly influenced by the crystallographic orientation. When increasing the misorientation, the cell spacing increases correspondingly and the cellular growth interface becomes more unstable.

## 1. Introduction

Single crystal superalloys are extensively used as blades in air-engines and land based gas turbines for its excellent high temperature strength and creep strength. The superior mechanical properties of nickel-base superalloys are derived largely from the microstructure and precipitated phase [1–3]. Many researchers have focused on the dendrite growth [4–7], due to the advanced mechanical properties of dendritic structure for industrial blades. Actually, cell is another important microstructure for nickel-base single crystal superalloys. The cellular growth state can be well indicated by the growth direction, growth interface and cellular envelop. Consequently, understanding of the development of solidified cells is of great importance.

For the array of cellular growth, many simplified analytical models have been proposed [8–10]. These models are mainly as function of growth rate ($R$) and thermal gradient ($G$), which lead to expressions on the basis of the growth of symmetric cells. Nickel-base superalloys with face centered cubic (fcc) crystallographic structure have a preferred growth direction, which would play an important role during the crystal growth. Ma [11] and He [12] indicated that the crystal orientation affected the stability of planar interface for nickel-base superalloys. It is established that crystal orientation determines the dendritic growth direction, while the growth direction of cellular crystal is almost dependent on the heat flow direction [13,14]. Plenty of studies have validated that the solidification rate can greatly affect the cellular microstructure. However, few references are concerned about the effect of crystal orientation on cellular growth [15]. Thus, in the present paper, influence of crystal orientation on the cellular interface stability and microstructure was investigated in a nickel-base superalloy.

## 2. Experimental

A first generation nickel-base single crystal superalloy DD407 was used in this work. The nominal composition of this alloy is listed in Table 1. Single crystal samples were produced by means of seeding technique in a directional solidification vacuum furnace. Differently oriented seeds with a diameter of 4 mm were cut from mater single crystals. Both of the seeds and the master alloy used in the experiments are DD407. The thermal gradient at the solid–liquid interface was measured to be 360 K/cm, and a withdrawal rate of 6 $\mu$m/s was selected to obtain cellular interface. After drawing downward to 40 mm length, quenching was performed immediately.

All specimens for metallographic examination were cut parallel or perpendicular to the withdrawal direction. Then the sections were etched with a solution of 14 mL HCl, 21 mL $H_2O$ and 8 g $FeCl_3$ for optical observation. Micrographic examinations were made on a LEICA DM4000M microscope. The orientation of each crystal was examined by a rotation X-ray diffraction method on a D/MAX-2400 X-ray diffractometer. Energy dispersive spectroscopy (EDS) was conducted using a ZEISS SUPRA 55 field emission scanning electron microscope (SEM) to investigate the solute distribution.

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**Table 1**
Nominal composition of the Ni-base superalloy DD407.

| Element | Cr | Co | Mo | W | Al | Ti | Ta | Ni |
|---|---|---|---|---|---|---|---|---|
| (wt.%) | 7.82 | 5.34 | 2.25 | 4.88 | 6.02 | 1.94 | 3.49 | Bal. |

## 3. Results and discussion

### 3.1. Influence of crystal orientation on cellular growth direction

Fig. 1 shows the different initial transitional zones of melt-back seeds. Single crystals were obtained by remelting the master alloy and a bottom seed. Half of the seed was unmelted to grow new crystal. The relationship between growth direction of the seed and heat flow direction was depicted in Fig. 1. The unmelted seeds exhibit different growth directions, which are related to the preferred orientation. It has been indicated that the primary dendrite growth direction is along ⟨0 0 1⟩ direction for superalloys. In Fig. 1(a), the ⟨0 0 1⟩ direction deviates from heat flow direction with a small angle, while cellular direction is almost along heat flow direction. However, as shown in Fig. 1(b) and (c), when the primary dendrites of the seeds deviate from the heat flow direction with larger angles, the formed cellular arms still grow along the heat flow direction. It is obviously different from the dendrite growth which was found in the previous experiments [13,14]. The cell growth direction is determined by the heat flow and irrelative to the preferred orientation of the seed. Esaka et al. [16] depicted the growth direction of cell and dendrite in succinonitrile (SCN) in the case of constrained growth. The growth direction changes from heat flow direction to the preferred orientation with increasing of growth velocity at a constant thermal gradient. It also revealed that the anisotropy progressively increased with the increasing growth velocity. Cells grow in the direction perpendicular to the solid–liquid interface as the growth direction is not constrained to ⟨0 0 1⟩ by anisotropy.

### 3.2. Influence of crystal orientation on the interface stability

The cell microstructures at the quenched liquid–solid regions are shown in Fig. 2. It can be seen that the crystal orientation has great influence on the cellular interface. The interface becomes more instable with increasing misorientation between the preferred orientation and heat flow direction under fixed cooling rate. In Fig. 2(a), the misorientation of the crystal is 4° and the ⟨0 0 1⟩ direction is close to the heat flow direction, and the cellular tip is semicircular and symmetrical. Although the cellular arms grow along the heat flow direction, the cellular tips become spiculate and incline to the preferred orientation as the misorientation increases to 8° (Fig. 2(b)). When the misorientation increases to 19° (Fig. 2(c)), the cellular tips deviate from the heat flow with a larger angle and become more asymmetrical. The cell arms are coarser and side-flanges are found in the growth front direction, suggesting that the interface becomes more unstable.

Previous experimental results have revealed that the cellular growth is mainly influenced by $G$ and $R$. The cellular interface would become more unstable with increased $R$ and decreased $G$ [17–19]. It was found that the instability of cellular interface of SCN increases with the increasing of misorientation [20]. Furthermore, as the misorientation increases, the cells could grow along ⟨0 0 1⟩ and side branches propagate faster in the direction close to the one of the heat-flow. Trivedi and Mason [21] has investigated the effects of interface kinetics anisotropy on the cellular direction in the pivalic acid-(PVA)-ethanol system in which significant anisotropy in interface kinetics and interfacial free energy are present. The results indicate the cellular growth is governed by the relative effects of heat flow and anisotropic property of the crystal. As compared with the results that observed in the model alloys, the cellular growth of nickel-base superalloy is influenced by the anisotropy of interface energy, for which the cells continuously grow with a rough interface. In our case, the anisotropy of surface energy would add a contribution to the appearing slope of the cellular tips.

### 3.3. Influence of crystal orientation on the cellular microstructure

Fig. 3 illustrates the cellular microstructures of single crystals with different orientations. Hexagonal cells uniformly distribute throughout the whole transverse planes. It is found that the cellular microstructure of single crystals changes from fine to coarse ones, and the cell spacing increases with the increase of misorientation. It is well known that the cell spacing is determined by the heat emission condition at the solidification interface, which mostly depends on cooling rate during directional solidification.

Kurz and Fisher [22] proposed a theoretical model to characterize the cell spacing based on imposed processing parameters. It demonstrated that the cell spacing was mainly dependent on $G$ and

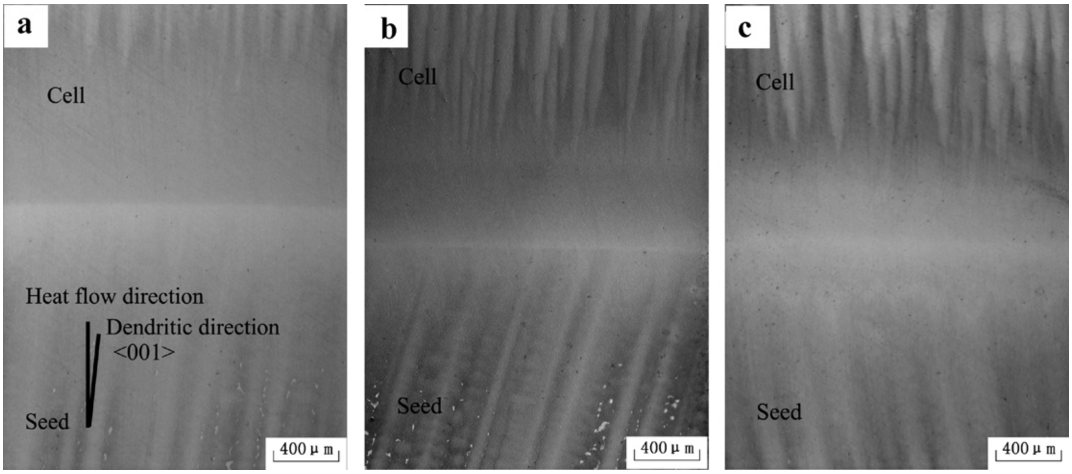

**Fig. 1.** Optical micrographs of three different initial transitional zones for differently oriented seeds. (a) $\Phi = 4°$; (b) $\Phi = 8°$; (c) $\Phi = 19°$.

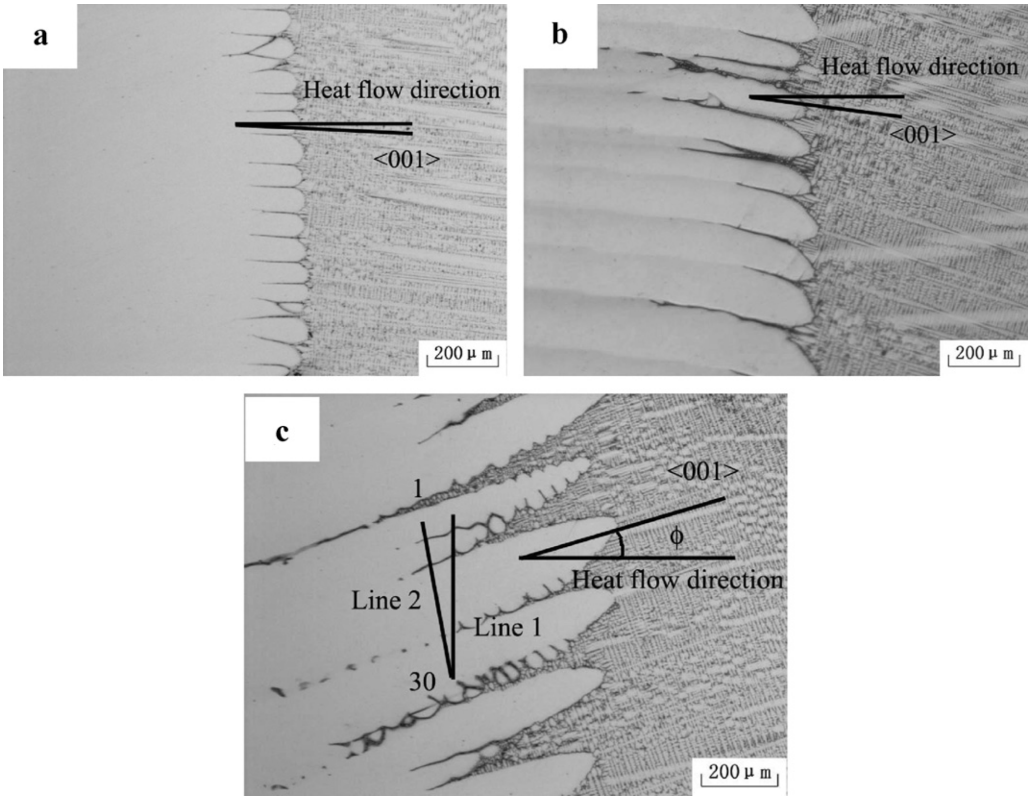

**Fig. 2.** Influence of misorientation on the cellular interface: (a) $\Phi = 4^\circ$; (b) $\Phi = 8^\circ$; (c) $\Phi = 19^\circ$.

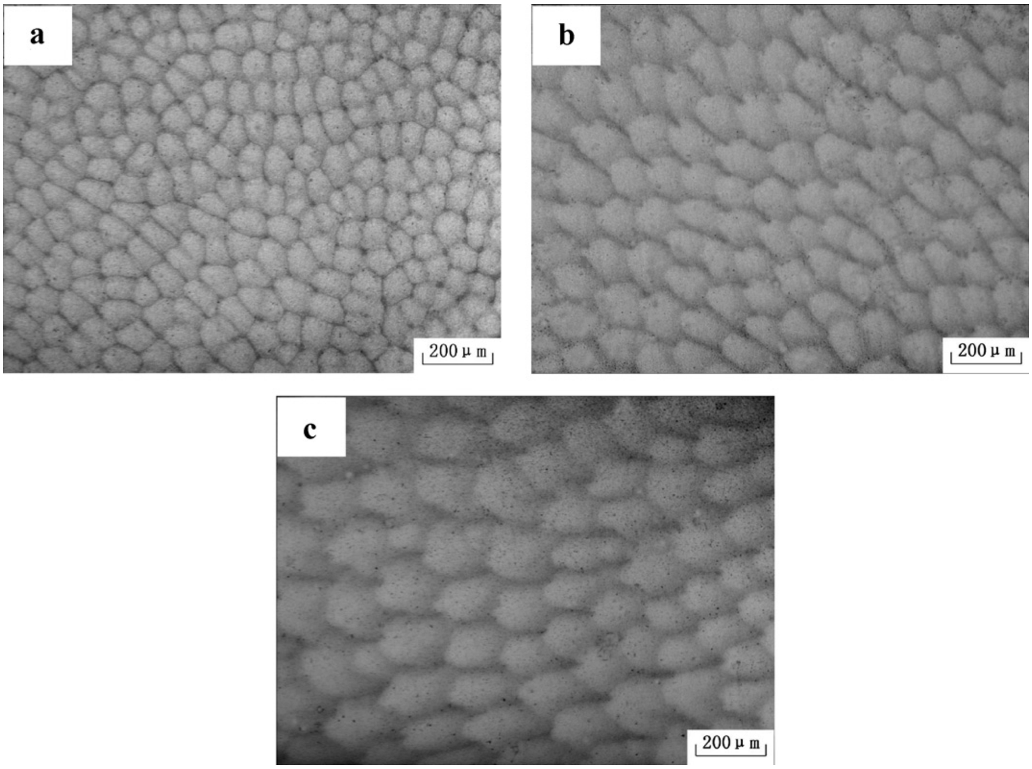

**Fig. 3.** Microstructures of differently oriented cells: (a) $\Phi = 4^\circ$; (b) $\Phi = 8^\circ$; (c) $\Phi = 19^\circ$.

*R*, meanwhile no parameter was related to crystal orientation for one alloy. The numerical results from Hunt showed that the cellular spacing was correspondingly influenced when surface energy was introduced, which was because the thermal gradient and low growth velocities were quite sensitive to the amount of anisotropy [23]. Grugel and Zhou [24] described the relationship between the dendrite spacing and crystal orientation through the transition of *G* and *R* along the heat flow direction. However, the model is not

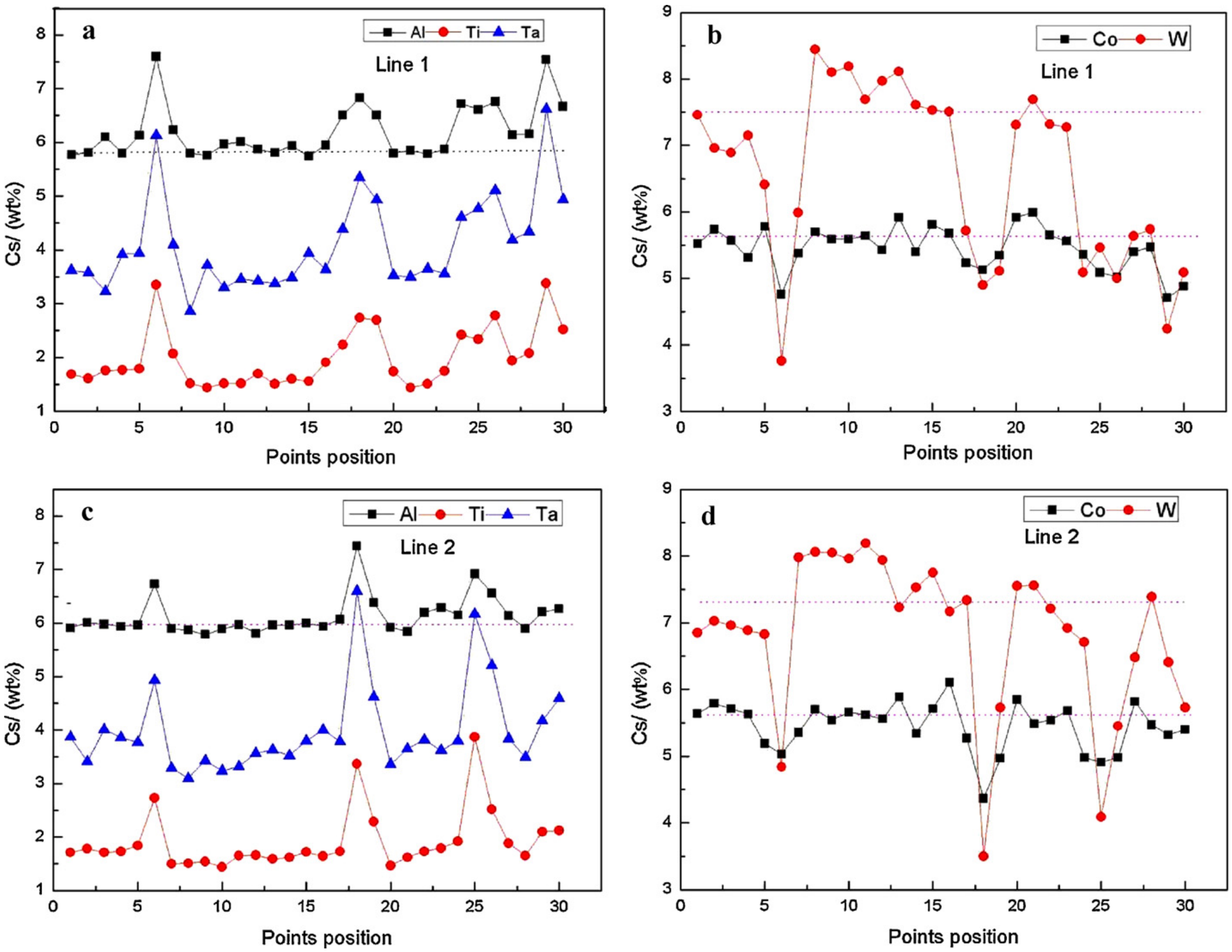

**Fig. 4.** Distribution of different solute elements along different directions at the quenched interface: (a) and (b) in line 1; (c) and (d) in line 2.

fit to the present experimental results, in which the function is based on simple model alloys. Nickel-base single crystal superalloys are complex alloys that contain several solute elements and complicated precipitated phases. The influence of interface energy anisotropy is evident, but the value could not be accurately measured in present time. Therefore, more systematic researches are needed for the foundation of the relationship between cell spacing and misorientation.

Fig. 4 illustrates the solute distribution in front of the solidification interface. Two lines are marked in Fig. 2(c) that line 1 is the distribution pattern of solutes perpendicular to the withdrawal direction, and line 2 is the distribution pattern of solutes perpendicular to the cell tip growth direction. 30 points selected in every line were measured. Al, Ti and Ta are positive segregation elements that segregate to the interdendritic regions, while W and Co are negative segregation elements that segregate to the dendrite cores. The data in line 1 reveal that the concentration of positive segregation elements from point 1 to 30 tends to increase (Fig. 4(a)). However, for negative segregation elements, the concentration along point 1 to 30 decreases gradually (Fig. 4(b)). As can be observed in Fig. 4(c) and (d), all the solutes almost uniformly distribute along line 2 direction. Both of the solute distribution patterns illustrate that the concentration distribution in the transverse direction of the cell growth interface is asymmetric.

As solute enrichment at different positions influences the crystal growth, extensive work has been done to predict the microsegregation in cellular and dendritic microstructures. Our previous research [25] has found that the solute distribution is influenced by crystal orientation in dendritic single crystals. The results of Ma showed that the solute segregation profiles in ⟨0 0 1⟩ orientation is flatter than that in ⟨0 1 1⟩ orientation across a dendritic cell, which illustrate the solute segregation in ⟨0 1 1⟩ direction is more severe [26]. The solute distribution in the vicinity of the cell tip is important during the crystal growth process. For cell tips direction with deviation from the heat flow direction, the solute elements in a misoriented cell section no longer uniformly distributed along radial direction in the crystal. Furthermore, non-symmetrical distribution of the solute in the misaligned cells was observed, which resulted in a higher growth rate of the side branches. The concentration gradient in front of the growth interface of the cells is larger than that at the opposite direction, which leads to the coarse flange in the growth direction and depressed flange at the back of the cellular trunks. An analysis of solute distribution around dendrite trunks for title dendrite was studied, and similar growth phenomena for secondary branching and solute distribution were checked [27].

To describe such tip growth behavior in cellular structure with misorientation, the anisotropy of crystal growth should be taken into account. The curvature undercooling in the growth depends on both the magnitude and anisotropy of the solid–liquid interfacial energy, while the surface energy is extremely sensitive to minor variations in alloy composition and these changes are not easy to predict [28]. Meanwhile, it was found that the magnitude of anisotropy has a marked effect on growth direction when the growth rate is low [29]. Studies are in progress to quantitatively analyze the cellular growth and the magnitude of the effect of anisotropy.

## 4. Conclusions

In this work, we have studied the influence of crystal orientation on the cellular growth during directional solidification of a nickel-base superalloy. Although the cellular growth direction is along heat flow direction and less affected by the seed orientation, the cell tip growth is influenced by the crystal orientation and inclines to $\langle 0\,0\,1 \rangle$ direction. Larger misorientation increases the cell spacing and aggravates the instability of the cellular interface, which is because that the solute distribution around the cells is affected by crystal orientation.

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