

Article

Influence of Crystal Orientation on the History Dependence of Primary Dendrite Arm Spacing in Ni-Based Single-Crystal Superalloy

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Abstract

Using a liquid metal cooling (LMC) high-gradient directional solidification furnace, this study compared primary dendrite arm spacing (PDAS) for nominal $\langle 001 \rangle$ deviation groups of 0° , 20° , and 45° under constant withdrawal rates and rate transitions between 15 and 100 $\mu\text{m/s}$. History dependence was evaluated by comparing the post-transition steady-region PDAS with the constant-rate reference at the same nominal current condition and normalizing the difference by the axial standard deviation of that reference. Under constant-rate growth, 30 mm was used as a common practical boundary for the relatively stable region. At 15 $\mu\text{m/s}$, steady-region PDAS increased from $182.1 \pm 2.7 \mu\text{m}$ at 0° to $204.9 \pm 2.7 \mu\text{m}$ at 20° and $259.2 \pm 2.5 \mu\text{m}$ at 45° . At 100 $\mu\text{m/s}$, both misoriented groups remained above the 0° value, but the ordering of 20° and 45° was not monotonic. The rate-transition effect exceeded the axial reference variability for the 0° group at both target rates, whereas the effects for the nominal 20° and 45° groups remained within that variability. The observed morphologies are consistent with orientation-dependent trunk replacement by higher-order branching. These results show that crystal orientation and process path should be considered jointly when controlling PDAS.

Keywords: Ni-based single-crystal superalloy; directional solidification; crystal orientation; primary dendrite arm spacing; history dependence

1. Introduction

Ni-based single-crystal superalloys exhibit excellent high-temperature mechanical properties, good resistance to hot corrosion, and high microstructural stability, making them the preferred materials for aero-engine turbine blades [1]. With the development of the aviation industry, increasing amounts of refractory elements, such as W, Re, and Ta, have been added to these alloys to improve the service performance of single-crystal blades [2,3]. However, owing to the low diffusion coefficients of refractory elements, the microsegregation induced during directional solidification cannot be completely eliminated by homogenization heat treatment. Severe residual segregation therefore prevents the high-temperature capability of Ni-based single-crystal superalloys from being fully exploited [4–6].

The average primary dendrite arm spacing (PDAS) is one of the most important characteristic length scales of directionally solidified microstructures and has a significant influence on the homogenization heat treatment of Ni-based single-crystal superalloys [7–9].



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Zhang et al. reduced the PDAS by increasing the withdrawal rate during directional solidification, thereby facilitating the homogenization heat treatment of a third-generation Ni-based single-crystal superalloy and improving its high-temperature creep properties [10]. Zhang et al. found that increasing the PDAS of the Ni-based single-crystal superalloy DD26 significantly enhanced the anisotropy of its mechanical properties [11]. Extensive theoretical analyses and experimental investigations have demonstrated that, under given solidification conditions, PDAS can vary over a relatively wide allowable range and that its average value depends on the solidification history [12,13]. Hunt et al. proposed that the upper limit of the primary dendrite or cellular spacing is twice its lower limit [14]. Huang et al. found that the ratio of the upper to lower limits of PDAS in Ni-based single-crystal superalloys was greater than 2 and that, under identical steady-state growth conditions, decreasing the PDAS effectively reduced the degree of microsegregation [15]. Liu et al. further reported that the upper limit of the average PDAS in Ni-based single-crystal superalloys could be as high as 8.5 times its lower limit [16]. Therefore, reducing the average PDAS by optimizing the solidification history is expected to provide an effective approach for improving the mechanical properties of single-crystal blades.

Previous studies of PDAS history dependence have focused on dendrites oriented along the $\langle 001 \rangle$ direction. In production castings, however, the crystal orientation commonly deviates from $\langle 001 \rangle$, which changes dendrite growth morphology and may alter spacing selection [17,18]. It is therefore unclear whether the behavior reported for $\langle 001 \rangle$ -oriented dendrites applies to misoriented crystals. In the present study, seeding was used to prepare $\langle 001 \rangle$ -oriented specimens and specimens misoriented by 20° and 45° . Their PDAS values were measured under constant withdrawal rates and two reciprocal rate-transition schedules. The study tests these two misorientation angles only; it does not assume that they represent every non- $\langle 001 \rangle$ orientation.

2. Materials and Methods

The experimental material was a low-Re third-generation Ni-based single-crystal superalloy with the nominal composition (wt.%): 4.3 Cr, 9.0 Co, 2.0 Mo, 8.0 W, 7.5 Ta, 4.6 Re, 0.1 Hf, 5.6 Al, 0.5 Nb, and balance Ni. The master-alloy composition was not independently verified by chemical analysis before directional solidification; the values are therefore identified as nominal throughout. The principal comparisons are made within the same nominal alloy material under matched processing conditions and do not require an independently known absolute composition to calculate relative PDAS differences. Actual chemistry may nevertheless affect absolute PDAS and generalization to another melt. Cylindrical seeds ($\Phi 4$ mm $\times$ 20 mm) were cut from a bulk as-cast single crystal along the directions in Figure 1, and the master-alloy rods were $\Phi 4$ mm $\times$ 100 mm. The experimental matrix comprised 12 orientation-schedule combinations: three nominal orientation groups for each of the four schedules in Table 1, with one independently solidified rod for each combination.

Single crystals were prepared in an LMC high-gradient directional solidification furnace. The quoted approximately 200 K/cm is the nominal axial gradient associated with this furnace's fixed hot-zone, thermal-barrier, and liquid-metal-cooling configuration, rather than a gradient derived from PDAS or measured in situ at each dendrite tip. All runs used the same furnace configuration: 1550 °C setpoint, 20 min hold, 4 mm rod geometry, and liquid-metal cooling bath; only the withdrawal-rate schedule was changed. Prior thermal analyses show that cooling configuration and casting geometry strongly influence G , while withdrawal speed can also shift the solidification front and alter its local gradient [19,20]. With fixed boundary conditions and uniform small-diameter rods, an approximately steady, comparable nominal gradient is a reasonable working approximation in the downstream

steady region, not a measurement proving that the local interface G was exactly 200 K/cm at every height or identical at both speeds. On that stated approximation, $G \cdot V$ is about 0.30 K/s at $15\text{ }\mu\text{m/s}$ and 2.0 K/s at $100\text{ }\mu\text{m/s}$; these are nominal cooling-rate proxies rather than measured local cooling rates.

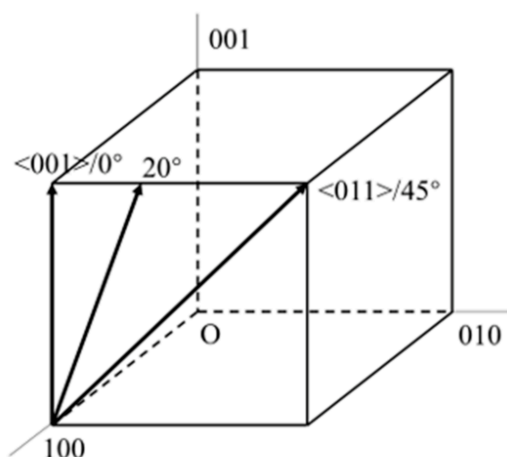


Figure 1. Schematic diagram of seeding cutting direction.

Table 1. Withdrawal schedules for the directional solidification experiments.

Scheme	First Stage		Second Stage		Transition Position (mm)
	Withdrawal Rate ($\mu\text{m/s}$)	Withdrawal Time (s)	Withdrawal Rate ($\mu\text{m/s}$)	Withdrawal Time (s)	
1	15	6000			
2	100	900			
3	15	3200	100	420	48
4	100	480	15	2800	48

The seed and master alloy were loaded sequentially into a high-purity Al_2O_3 crucible fixed to the withdrawal rod. The chamber was evacuated below $6.7 \times 10^{-2}\text{ Pa}$, heated to $1550\text{ }^\circ\text{C}$, and held for 20 min before withdrawal. The withdrawal-rates used in this study are shown in Table 1, where Schemes 1 and 2 employ a constant withdrawal-rate, while Schemes 3 and 4 employ a variable withdrawal-rate. For Schemes 3 and 4, the withdrawal unit switched mechanically and directly between its preset speeds of 15 and $100\text{ }\mu\text{m/s}$ when the calculated withdrawal distance reached 48 mm . No intermediate-speed segment, dwell, or programmed acceleration/deceleration ramp was imposed; the change is operationally instantaneous in the schedule. The physical switching duration was not measured, and the present PDAS sampling does not resolve the immediate transient. Multiplication of preset rate by scheduled time gives 90 mm for Schemes 1 and 2, and 48 mm before plus 42 mm after the switch for Schemes 3 and 4.

After solidification, the rods were macroetched with $\text{H}_2\text{O}_2 + \text{HCl}$ (1:2) to confirm single-crystal integrity. For the constant-rate schedules, one transverse section was analyzed at each 10 mm increment from 10 to 100 mm . The entire usable polished cross-section was used for the area method at each height (nominal area for a 4 mm diameter section, 12.57 mm^2); the displayed micrographs are representative fields rather than additional specimen replicates. A primary dendrite core center was assigned to the geometric center of a continuous primary trunk profile, including the center of a cross-shaped profile for the 0° group and the central trunk of a bird-like profile for the misoriented groups. Isolated secondary/tertiary-arm profiles and eutectic pools were not counted. To avoid specimen-edge bias, a core was included only when its center lay inside the calibrated

usable boundary; partial profiles centered outside that boundary were excluded. PDAS was calculated as the square root of S/N , where S is the calibrated usable area and N is the included number of primary-core centers.

The constant-rate steady-region summaries use the eight axial positions from 30 to 100 mm (section positions $n = 8$) and report the mean and sample standard deviation across those positions. Because these positions come from one rod, they quantify axial variability and are not independent solidification replicates. Post-transition values are reported descriptively for the available final steady region; inferential p -values are not presented.

To assess history dependence, each post-transition steady-region PDAS value was compared with the constant-rate reference for the same nominal orientation and final withdrawal rate: the 100-to-15 $\mu\text{m/s}$ path was compared with constant 15 $\mu\text{m/s}$, and the 15-to-100 $\mu\text{m/s}$ path with constant 100 $\mu\text{m/s}$. Thus the current nominal rate and furnace settings are matched, while the preceding rate differs. We report $\Delta\lambda = \lambda_t - \lambda_c$ and the descriptive index $H = |\Delta\lambda|/s_c$, where λ_t is the post-transition steady-region PDAS value of the rate-transition rod; λ_c is the mean PDAS across the eight axial positions (30–100 mm) of the constant-rate rod matched for nominal orientation and final withdrawal rate. s_c is the sample stand deviation over the eight axial positions (30–100 mm) of the corresponding constant-rate rod. We call a difference resolved from ordinary axial reference variability when $H > 1$; $H \leq 1$ means that a retained difference was not resolved by this descriptive criterion. This threshold is a transparent scale comparison, not a hypothesis test or proof of zero effect; one independently solidified rod per combination precludes a between-casting significance test.

Crystal orientation was characterized with an Apreo 2 field-emission scanning electron microscope equipped with EBSD (Thermo Fisher Scientific, Waltham, MA, USA). Maps covered $200\ \mu\text{m} \times 200\ \mu\text{m}$ with a $20\ \mu\text{m}$ step and were processed in OIM Analysis. The axial deviation was defined as the minimum angle between the specimen-axis direction and the symmetry-equivalent cubic $\langle 001 \rangle$ axes: $\theta = \min \arccos(|z_s \cdot g c_i|)$, where z_s is the specimen-axis unit vector, g transforms crystal directions into the specimen frame, and c_i denotes $[100]$, $[010]$, or $[100]$. Visual comparison of the $\langle 001 \rangle$ pole positions in Figure 2 with the intended seeded directions indicates an offset of less than approximately 2° for each of the nominal 0° , 20° , and 45° groups. This is an estimate from the displayed pole figures, not a pixel-level map average or a calibrated angular uncertainty.

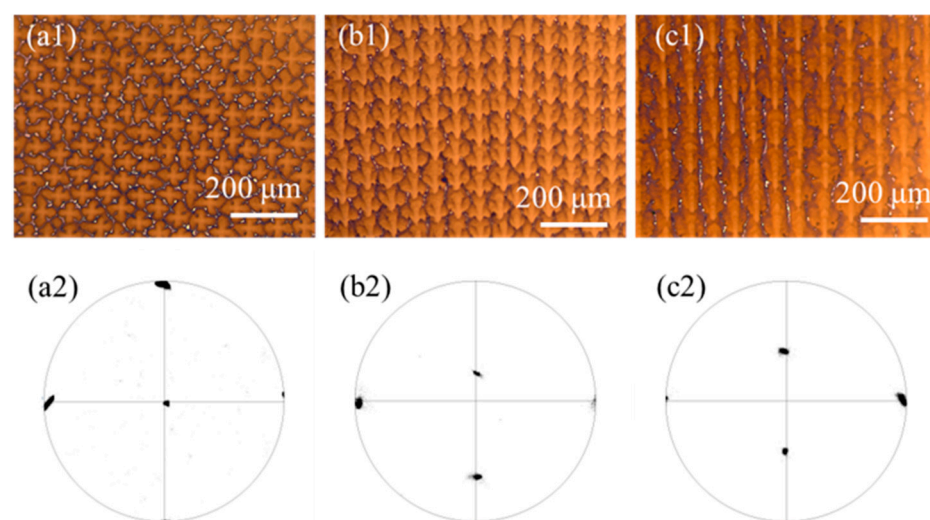


Figure 2. Cross-sectional microstructures (a1–c1) and corresponding pole figures (a2–c2) for specimens withdrawn at 15 $\mu\text{m/s}$: (a1,a2) nominal 0° deviation from $\langle 001 \rangle$; (b1,b2) nominal 20° deviation; and (c1,c2) nominal 45° deviation.

3. Results and Discussion

3.1. Dendrite Growth Morphology and Orientation

Figure 2 shows transverse microstructures and pole figures for the three nominal orientation groups at 15 $\mu\text{m/s}$. The pole positions confirm the intended progression from an approximately axial $\langle 001 \rangle$ group toward increasingly misoriented groups and suggest that each group's actual deviation lies within approximately 2° of its nominal target. All groups contained dendrites and interdendritic eutectic constituents. The 0° group showed a comparatively uniform array and a cross-shaped transverse morphology. The 20° and 45° groups showed increasingly linear arrangements and asymmetric, bird-like profiles. These ex situ morphologies are consistent with preferential $\langle 001 \rangle$ growth: an inclined primary trunk intersects the transverse plane asymmetrically, and favorably directed side branches can develop into replacement trunks.

At 100 $\mu\text{m/s}$, the dendritic microstructure was refined while the orientation-dependent transverse and longitudinal morphologies remained qualitatively similar (Figure 3). Interdendritic eutectic constituents were not resolved in the displayed fields. Primary trunks were nearly parallel to the specimen axis in the 0° group and inclined in the two misoriented groups. The nominal 45° group showed a V-shaped longitudinal morphology. This behavior is consistent with earlier observations of tilted dendrites [21] and with recent reports that secondary branches and tertiary-arm development can regulate aligned primary dendrite patterns [22,23].

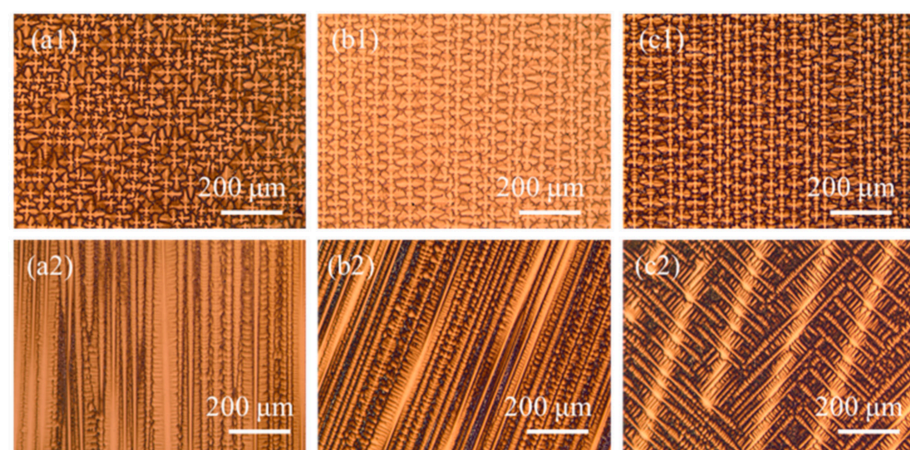


Figure 3. Cross-sectional (a1–c1) and longitudinal (a2–c2) microstructures for specimens withdrawn at 100 $\mu\text{m/s}$: (a1,a2) nominal 0° deviation from $\langle 001 \rangle$; (b1,b2) nominal 20° deviation; and (c1,c2) nominal 45° deviation.

3.2. Effect of Crystal Orientation on PDAS at a Constant Withdrawal Rate

Figure 4 shows PDAS as a function of solidification distance. All six constant-rate series decreased during the early portion and then fluctuated within a narrower band. A common boundary of 30 mm was therefore used to summarize the relatively stable region for all groups. From 30 to 100 mm, the mean PDAS values were 182.1 ± 2.7 , 204.9 ± 2.7 , and 259.2 ± 2.5 μm for nominal deviations of 0° , 20° , and 45° , respectively, at 15 $\mu\text{m/s}$. At 100 $\mu\text{m/s}$, the corresponding values were 110.1 ± 2.0 , 120.5 ± 2.1 , and 115.5 ± 2.1 μm . Thus, the 15 $\mu\text{m/s}$ series increased monotonically with nominal deviation and agrees qualitatively with the orientation trend reported recently for DD6 [22]. At 100 $\mu\text{m/s}$, both misoriented groups remained above the 0° group, but 20° exceeded 45° ; the angular ranking was therefore not monotonic. Increasing rate reduced both the absolute PDAS and the spread among orientation groups, but it did not eliminate orientation sensitivity.

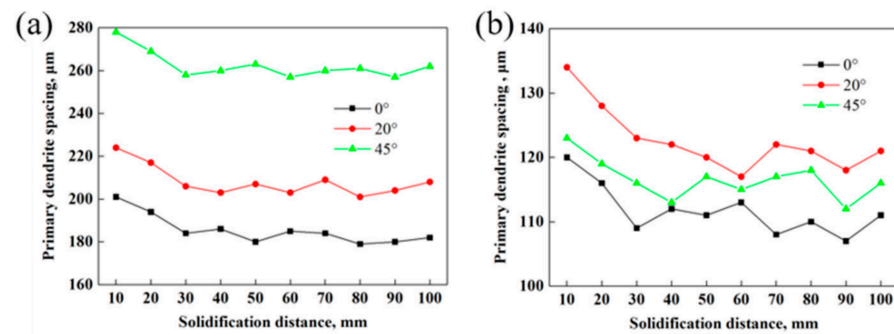


Figure 4. Variation in primary dendrite arm spacing with solidification distance for the three nominal orientation groups at constant withdrawal rates of (a) 15 $\mu\text{m/s}$ and (b) 100 $\mu\text{m/s}$.

3.3. Effect of Crystal Orientation on PDAS Under Withdrawal Rate Transition Conditions

Classical steady-field models relate PDAS to current growth conditions, especially withdrawal rate and temperature gradient [24,25]. Array adjustment, however, occurs through discrete events such as trunk elimination, lateral migration, and branch-mediated replacement, so different paths can lead to different spacings at the same nominal final condition [12–15]. Here, the path effect is quantified by $\Delta\lambda$ and H using the matched-rate comparison and descriptive criterion defined in Section 2. This comparison separates a retained history effect from ordinary axial variation in the constant-rate reference; it does not establish statistical significance across independently repeated castings.

Figure 5 and Table 2 compare constant-rate and post-transition values at the same target rate. For the nominal 0° group, the transition value differed from the constant reference by $-8.0\ \mu\text{m}$ (-4.4% , $H = 3.0$) at 15 $\mu\text{m/s}$ and by $+4.2\ \mu\text{m}$ ($+3.8\%$, $H = 2.1$) at 100 $\mu\text{m/s}$; both effects exceeded the axial s_c of the corresponding reference. For the nominal 20° group, the changes were $-1.9\ \mu\text{m}$ (-0.9% , $H = 0.7$) and $+0.7\ \mu\text{m}$ ($+0.6\%$, $H = 0.3$), respectively. For the nominal 45° group, they were $0.0\ \mu\text{m}$ ($H = 0.0$) and $+0.1\ \mu\text{m}$ ($+0.1\%$, $H = 0.0$), respectively. Thus, the rate-history effect was resolved from axial variability for the nominal 0° group at both target rates but not for the two misoriented groups under the tested conditions.

Table 2. Descriptive comparison of steady-region PDAS under constant-rate and rate-transition conditions.

Target Rate ($\mu\text{m/s}$)	Nominal Deviation (deg)	Reference Mean PADS (μm)	After Transition (μm)	$\Delta\lambda$ (μm (%))	$H = \Delta\lambda /s_c$
15	0	182.1 ± 2.7	174.1	-8.0 (-4.4)	3.0
15	20	204.9 ± 2.7	203.0	-1.9 (-0.9)	0.7
15	45	259.2 ± 2.5	259.2	0.0 (0.0)	0.0
100	0	110.1 ± 2.0	114.3	$+4.2$ ($+3.8$)	2.1
100	20	120.5 ± 2.1	121.2	$+0.7$ ($+0.6$)	0.3
100	45	115.5 ± 2.1	115.6	$+0.1$ ($+0.1$)	0.0

Note: s_c is the sample standard deviation across the eight constant-rate section positions from 30 to 100 mm (section positions $n = 8$). H is a descriptive effect index, not an inferential test. Because one independently solidified specimen was used per orientation-schedule combination, section positions are not treated as independent specimen replicates.

3.4. Mechanism of the Effect of Crystal Orientation on the History Dependence of PDAS

Ma and Sahm [12] treated primary-trunk growth and side-arm development as coupled but distinct contributions to spacing selection. For an approximately $\langle 001 \rangle$ -aligned array, changing withdrawal rate alters tip and arm kinetics before it changes the lateral

positions of existing trunks. The array subsequently adjusts through trunk elimination, lateral migration, and tertiary branching, and the discreteness of these events can preserve a memory of the preceding array. This framework is consistent with the resolved path effects for the nominal 0° group, but the present measurements do not identify which adjustment event dominates.

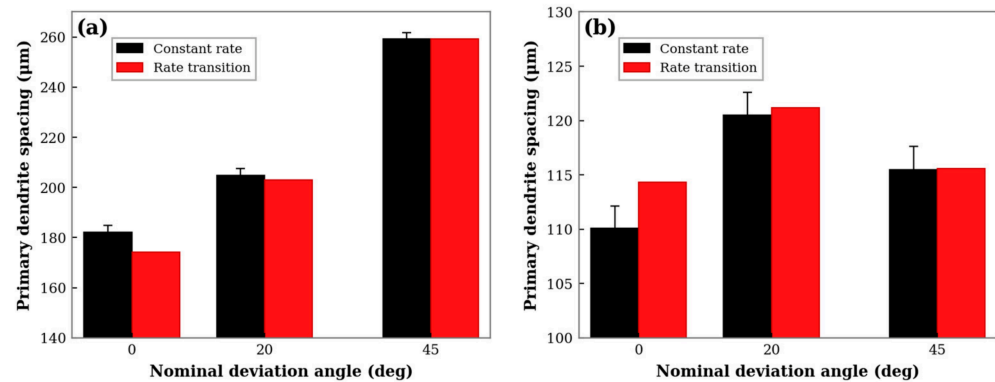


Figure 5. Influence of withdrawal-rate history on steady-region primary dendrite arm spacing at target rates of (a) 15 μm/s and (b) 100 μm/s. Error bars on the constant-rate bars show the sample s_c across the eight axial positions from 30 to 100 mm; no inferential error bar is assigned to the single-specimen post-transition summary.

For inclined arrays, the longitudinal and transverse microstructures are consistent with repeated trunk replacement by favorably oriented higher-order branches. Yuan et al. directly linked oriented secondary branches and tertiary-arm development to changes in primary dendrite patterns [23], and Yang et al. reported migration of the positions at which tertiary dendrites can transform into primary dendrites as orientation changes [22]. These studies make branch-mediated replacement a plausible interpretation of Figure 6b. Nevertheless, our evidence is *ex situ* and two-dimensional: no time-resolved branching frequency, trunk-replacement count, or three-dimensional connectivity was measured.

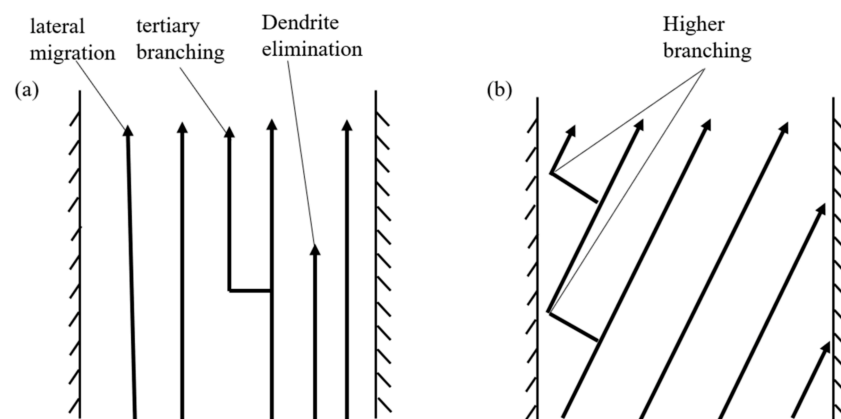


Figure 6. Schematic diagram of dendrite spacing selection mode. (a) $\langle 001 \rangle$ orientation, (b) non- $\langle 001 \rangle$ orientation.

The reduced absolute orientation spread at 100 μm/s can be interpreted in terms of faster growth kinetics. Using the approximately common nominal G as a working approximation, raising V increases nominal $G \cdot V$ from about 0.30 to 2.0 K/s and shortens the solutal diffusion length ahead of a tip (scaling as D/V). The resulting finer array and shorter interaction distance can reduce the time and space available for orientation-dependent side branches to modify neighboring trunks. Because actual interface G was

not measured at both speeds, this kinetic argument is conditional on the shared-boundary approximation rather than a demonstration of identical local gradients. It is consistent with the refinement in Figures 3 and 4 and recent phase-field work linking PDAS to growth and mushy-zone conditions [26], but the nonmonotonic 20°/45° ordering and transition results show that high rate does not make crystallographic orientation irrelevant.

3.5. Implications for Turbine-Blade Process Control

The results imply that PDAS control in single-crystal blades should not rely on instantaneous withdrawal rate alone. For nearly <001>-aligned regions, the preceding rate path should be recorded and rate changes should be designed with sufficient downstream distance for array readjustment. For misoriented regions, the present data do not justify assuming history independence; instead, orientation-specific response maps are needed because both the baseline PDAS and the sign of the transition effect changed with nominal angle and target rate. Increasing withdrawal rate refined PDAS and compressed the orientation spread, but it did not remove orientation sensitivity. A practical control strategy would combine seed/airfoil EBSD information, commanded rate history, and calibrated thermal-field estimates rather than using a single global PDAS-rate relation. Translation to a full blade will also require accounting for cross-sectional thermal nonuniformity and liquidus-isotherm shape, which can create local PDAS variations even at one nominal withdrawal rate [27].

4. Conclusions

- (1) Under the six constant-rate conditions examined, average PDAS reached a relative plateau by approximately 30 mm.
- (2) Increasing the withdrawal rate from 15 to 100 $\mu\text{m/s}$ reduced PDAS. At a given rate, PDAS increased across the tested orientations from <001> to 20° and 45° deviation.
- (3) The post-transition PDAS for the <001> sample outside the corresponding constant rate plateau range for both rate-transition directions, demonstrating history dependence under the tested conditions. The values for the 20° and 45° specimens remained within their reference ranges; no history dependence was detected for these two angles.

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Conflicts of Interest: Authors Licheng Du was employed by the company AECC Aero Science & Technology Co., Ltd. The remaining author declares that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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