Dendritic Packing and Porosity Formation within Single Crystal CMSX-4® Ni-Base Superalloy

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Abstract. Specialised imaging techniques are instrumental for extracting the wealth of information locked within as-cast microstructures. Recently, a novel characterisation tool (Shape-Limited Primary Spacing) was developed for determining primary dendrite arm spacing (PDAS), dendritic packing pattern and porosity formation within single crystal microstructures. In this work, the algorithm is applied to a single crystal CMSX-4® Ni-base superalloy to investigate the relationship between dendritic packing pattern and condensation porosity. It is concluded that porosity formation is reduced when regular organised dendritic packing patterns form. This work elucidates new unknown relationships between packing and porosity and may help to develop improved process-property models for defect prediction.

1. Introduction

Single crystal casting is a critical metal manufacturing technique used throughout the aerospace industry. High strength turbine blades produced via single crystal casting possess significantly higher creep resistance in comparison to directionally-solidified and equiaxed counterparts [1]. A continual goal for materials scientists is to increase jet engine performance and efficiency. Single crystal Ni-base super alloys contain significant levels of microsegregation; a result of their complex chemistry. The primary dendrite arm spacing (PDAS) is a key length scale used for characterisation of microsegregation between dendritic and the interdendritic regions. A large PDAS corresponds to increased microsegregation which results in the formation of low melting point secondary phase eutectics, as well as incoherent precipitates and pores in the interdendritic region. It is these secondary phases and defects which determine the properties of the as-cast material and its high temperature performance [2].

Porosity formation is an unavoidable phenomenon in any cast microstructure. It is a result of thermal strains and shrinkage which occurs due to lack of liquid feeding at the last to solidify regions [3]. In single crystal samples, porosity is localised in the interdendritic regions of the array [4]. This microporosity acts as crack initiation sites and is severely detrimental to material mechanical properties. Particularly important to turbine blade performance is the detrimental impact of porosity on fatigue life [5]. Classically, porosity characterisation constitutes identifying the pore type, be it a condensation, diffusion or deformation related formation. This involves careful microscale analysis to quantify the surface, shape and pore roughness to draw conclusions about the formation mechanisms. Pore formation is dictated by the liquid feeding capability to the interdendritic region, therefore, it is intrinsically related to the arrangement of the local dendritic neighbourhood. Dendritic packing patterns are a result of the bulk solidification conditions. Consequently, porosity formation must also be closely related to global variations in solidification conditions.
In this work, a recently developed Shape-Limited Primary Spacing (SLPS) algorithm [6] is applied to a single crystal CMSX-4® microstructure to relate dendritic packing patterns and porosity formation. The main aim of this work is to prove porosity and dendritic packing patterns are related; thus, improving the understanding of porosity formation mechanisms. The end goal is to improve single crystal modelling capability through further understanding pore formation pathways. This will reduce scatter of as-cast/solutionised mechanical property data, thereby allowing turbine blade design service stresses to be raised.

2. Experimental procedure

A single crystal as-cast CMSX-4® bar of 9.4 mm diameter was sectioned using a metallographic precision cutter and mounted in Conducto-Mount. The samples were prepared for Scanning Electron Microscopy (SEM) through standard metallographic preparation and polished using 1µm diamond suspension. The sample was imaged in backscattered electron (BSE) mode using a FEI 650 Quanta SEM facility at the University of Leicester (20 keV, 5.0 spot, 1.92mm HF, 10.1 mm WD) to visualise the microsegregation between the dendritic core and interdendritic region. The final image was comprised of a 24 × 24 grid of individual SEM images taken at 238 × magnification with 20 µs scan speed and encompassed the full horizontal plane to the test bar. The image was loaded into an automatic dendritic core detection software, DenMap, and the exact core and porosity pixel positions were located [7]. The dendritic packing and local PDAS were calculated using the novel Shape-Limited Primary Spacing (SLPS) algorithm [6] which is based on Voronoi area and regular polygon shapes (figure 1). The algorithm identifies the dendrites whose diffusion fields interact locally with a central dendrite. This method of true nearest neighbour (TNN) determination is applied to each dendrite within the single crystal array. The porosity area and location are indexed and overlapped on each local packing pattern (Voronoi shape) such that each pore belongs to one polygon. This allows local and bulk spacing and packing to be related to pore location.

\[ N_3 \quad N_4 \quad N_5 \quad N_6 \quad N_7 \quad N_8 \quad N_9 \]

Figure 1. Dendritic packing patterns. Coordination number, \( N \), indicates the true nearest neighbouring dendrites determined by the Shape-Limited Primary Spacing (SLPS) algorithm.

3. Results

The packing results are illustrated in figure 2(a), the scale bar indicates the type of dendritic packing pattern. The top right of figure 2(a) demonstrates refined connected hexagonal packing (\( N_6 \)). The bottom left of figure 2(a) a combination of irregular geometry triangular (\( N_3 \)), square (\( N_4 \)), pentagonal (\( N_5 \)), heptagonal (\( N_7 \)), octagonal (\( N_8 \)) and nonagonal (\( N_9 \)) packing. Figure 2(b) illustrates the detected pore size within figure 2(a). Pore areas of about \( \sim 50 \mu m \) are the most frequently detected. Table 1 indicates the \( N_6 \) region (section 1) obtains a more refined PDAS, six times smaller pore frequency and a smaller average and maximum pore size than the irregular packed region (section 2). The bulk of the pores are congregated on the left-hand side of the sample (figure 3(a)). There is a region of increased pore frequency shown in green in the bottom half of figure 3(a). The upper right-hand side demonstrates almost no pores, which also corresponds to the hexagonally packed, \( N_6 \), region in figure 2(a). \( N_6 \) packing over the entire sample demonstrates the smallest pore frequency per shape out of all determined coordination numbers, \( N \). A pore area distribution plot is illustrated in figure 3(c). Similarly, to figure 3(a) the largest pore sizes are congregated in a band across the bottom half of the sample in figure 3(c). The \( N \) with the largest pores is determined as the triangularly packed.
N_3 dendrites. The smallest pore area per shape area is determined as N_8 which is closely followed by N_6.

Figure 2. (a) Shape-Limited Primary Spacing (SLPS) Voronoi packing map. Key data for the highlighted sections (1 & 2) are illustrated in Table 1. (b) Pore frequency vs pore area for the entire sample in figure 2(a).

Table 1. Mean PDAS and pore data for section 1 & 2 from figure 2(a).

| Region | Packing   | Mean PDAS (μm) | Pore Frequency | Average Pore Size (μm²) | Maximum Pore Size (μm²) |
|--------|-----------|----------------|----------------|-------------------------|-------------------------|
| 1      | Hexagonal | 311.91         | 17             | 65.11                   | 269.09                  |
| 2      | Irregular | 367.56         | 102            | 79.28                   | 981.19                  |
4. Discussion

A variety of dendritic packing has been determined across the entire microstructure (figure 2(a)). There exists a clear hexagonally packed, $N_6$, region in the top right of the micrograph, and an irregular packed region in the bottom left. Interestingly, the majority of the microporosity resides in the irregularly packed regions, illustrated figure 3(a,c). In fact, in the irregularly packed region in section 2, there exists six times the microporosity as the regular packed hexagonal region (table 1). Porosity is known to be intrinsically linked to fluid flow during solidification [8]. Therefore, the organised arrangement of dendrites in the hexagonal configuration is likely to allow sufficient liquid feed to the last to solidify regions which in turn reduces the propensity for microporosity formation.

Porosity formation within the $N_3$ packing was the second most frequent observed within the sample (figure 3(b)) and it obtained the largest pore area per packing size (figure 3(d)). Interestingly, $N_3$ dendrites surround the higher order $N_6$ and $N_7$ packing patterns which also witness the second and third largest pore frequencies, respectively (figure 3(b)). These packing patterns are all congregated together on one side of the sample. It follows that porosity mainly forms between the $N_3$, $N_7$ and $N_8$ dendrites. The $N_7$ and $N_8$ configurations have the largest associated areas on the micrograph, clearly seen in figure 2(a). These arrangements with large PDAS indicate areas rich in interdendritic partitioning elements such as Ni, Ti, Ta, and Al [9]. Consequently, the close packed areas with small PDAS indicate regions rich in dendritic core elements such as Co, W and Re [10]. It follows, that the largest and most frequent porosity forms between the $N_3$ and $N_7$ and $N_8$ dendrites. Therefore, the most detrimental porosity to material mechanical properties forms when large interdendritic rich regions are neighboured by correspondingly smaller core element rich regions. To fill shrinkage voids, melt must flow downwards between neighbouring dendrites [11]. The irregular packed shapes inhibit the ability of the liquid to flow and fill these voids. It follows, that the more irregular the dendritic packing arrangement the more liquid feed is restricted and the higher the propensity for porosity formation.

The temperature profile across a sample during solidification is uneven. A strong radial thermal gradient component exists due to the nature of the directional solidification process [12]. Therefore, the solidification rate across a sample section varies, with one side of the sample solidifying more quickly than the other. Interestingly, a band of pores exists across the microstructure which finds itself between the regular and irregular packed regions (figure 3(a)). This porosity band witnesses not only the highest frequency of pores but also the largest (figure 3c). Therefore, microporosity formation may be intrinsically linked to differential rates of thermal contraction due to solidification strain.
disparity in freezing point which exists between the separate regions may induce a tensile stress in the sample which manifests in these oversized pores. Further investigation into these zones is needed to establish the exact relationship between thermal contraction, shrinkage and microsegregation. However, DenMap, SLPS and the shown pore-packing analysis are capable of locating and characterising these crucial zones.

5. Conclusions
A novel packing and porosity algorithm, Shape-Limited Primary Spacing (SLPS) is applied to study a CMSX-4® single crystal Ni-base superalloy. The new technique has elucidated unknown relationships between dendritic packing patterns and the corresponding porosity formation. For successful modelling of defect prediction, a full understanding of the influence of solidification conditions on the propensity for defect formation is required. SLPS enables rapid characterisation of local PDAS, packing patterns and defects, within single crystal alloys. Now, for the first-time single crystals can be characterised in a way that is repeatable, comparable, and accurate. Therefore, enabling development of sophisticated defect prediction models, machine learning algorithms and eventually A.I.

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6. References
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