Solidification microstructure during selective laser melting of Ni based superalloy: experiment and mesoscopic modelling

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Abstract. A set of single track laser melting experiments was performed in a selective laser melting (SLM). The tracks were done on an Inconel 718 plate with various laser scan velocities at a constant laser power of 150 W. The geometries of the molten pool (MP), as well as the solidified dendrite structures, i.e., primary and secondary dendrite arm spacing (PDAS and SDAS), in the cross sections of the molten path were characterized to evaluate the effect of the laser scan velocity during SLM. Moreover, the local solidification thermal conditions (cooling rate \( R^* \), tip growth velocity \( V^* \) and temperature gradient \( G^* \)) at the MP bottom were deduced from the SDAS and the geometries of the molten pool. Finally, the mesoscopic envelope model was used to simulate the PDAS selection of the columnar dendrite growth in the molten pool. The simulated results were compared with the experimental data, and a good agreement was achieved under different laser scan velocities.

1. Introduction
Inconel 718, a nickel based superalloy, is widely used in many industrial applications, such as turbine blades in aerospace and automobile industries, combustion chambers and nuclear reactors, due to its excellent mechanical properties, oxidation resistance and great corrosion resistance at high temperatures [1–3]. However, it is very difficult to manufacture products of Inconel 718 by conventional machining techniques due to its high hardness and low thermal conductivity [4,5]. Thus, the novel additive manufacturing (AM) technologies will have a significant potential in the applications of Inconel 718, especially for the components with complex structures and high dimensional specifications.

Selective laser melting (SLM) is a commonly used additive manufacturing technique, which generally consists of the melting of small volumes of powders in a localized region and the subsequent rapid solidification of the molten pool (MP) [6,7]. Many aspects of SLM and other AM processes are being widely investigated using both experimental and modelling methods. Until now, most of these research focused on the heat transfer and liquid flow in the MP [8], on defects [9], on the properties of the final product [1–3] and also on the microstructure evolution during AM process [6], which are on a scale the grains. However, there is a lack of studies on the formation of the solidified dendrite structure.
(substructure of the grain) during SLM, which is crucial for controlling the grain texture, the segregation, and ultimately the material properties [10]. The solidification during SLM is a highly complex process and is significantly affected by various conditions, such as the spot size, the power and the velocity of the laser [11]. Therefore, it is necessary to elucidate the effects of these conditions on the solidification microstructure.

The traditional approach for researching the SLM process is using the final layer-by-layer buildup, which is highly time consuming and costly. As a substitute, the single track laser melting experiment was used in some investigations such as the correlation of the MP geometries and the SLM parameters, which greatly reduced the time and cost [12]. In the aspect of solidification characterization, due to the epitaxial dendrite growth between the layers [7], the dendrite structure of the final buildup is mostly controlled by the first layer. Thus, it is reasonable to utilize the single track experiment to investigate the solidification behavior in AM process, and also to estimate the solidification conditions which could be very difficult to be obtained from experiment but would be necessary to be used in the simulation.

In this study, a set of single track laser melting experiments was performed on Inconel 718 plate under various laser scan velocity at a constant laser power of 150 W to evaluate the effect of the laser scan velocity on the MP geometries and on the characterization of the solidified dendritic structures. Moreover, the mesoscopic envelope model was used to simulate the primary arm spacing (PDAS) selection of the columnar dendrite growth in the MP, using the solidification thermal conditions deduced from the MP geometries and the solidified structures. The simulation results were compared with the experimental data.

2. Single track laser melting experiments
A set of single track laser melting experiments was performed on the same Inconel 718 plate under six various laser scan velocities from 50 to 300 mm/s using a commercial laser powder bed fusion machine ReaLizer SLM 250. A continuous laser with the wave length of 1060-1100 nm was used. The spot size of the laser was around 35 µm and the laser power was constant at 150 W. The dimensions of the plate were approximately 150 × 150 × 5 mm. The original microstructure of this plate was characterized as equiaxed grains structure without specific texture. The length for each laser scan velocity is around 140 mm and the distance between the neighbor single tracks is 2 mm to avoid the interference from each other. The samples were named by the scan velocity, i.e., V50 means the sample with the scan velocity of 50 mm/s. After the SLM process, the samples for metallographic analysis were cross sectioned perpendicular to the laser scan direction, at the position of more than 15 mm from the start of the laser, at which the MP reached a steady regime. The samples were mechanically polished with diamond suspension as the final step to identify the dendritic microstructure and then examined by scanning electron microscopy (SEM).

3. Mesoscopic envelope model
The mesoscopic envelope model, developed by Steinbach et al. [13], is a powerful simulation tool for dendritic solidification which bridges the gap between microscopic and macroscopic approaches. The core idea of this model is the description of a dendritic grain by its envelope - a virtual smooth surface that links the tips of the actively growing dendrite branches. The growth velocity of the envelope growth can thus be calculated from the velocities of the dendrite tips. The growth of the dendrite tips is determined from the local supersaturation of the liquid in the proximity of the envelope by a stagnant-film formulation of a tip model [14]. The branched dendritic structure inside the envelope is not resolved; the interior of the envelope is rather described in a volume-averaged sense by a phase-fraction field and other volume-averaged quantities. The phase change that determines the evolution of the structure inside the growing envelope is controlled by the solute exchange with the surroundings of the grain. We used the in-house mesoscopic model code CrystalFOAM, developed on the OpenFOAM finite-volume platform. More detailed description about this model could be found in our previous studies [14].
4. Results and discussion

4.1. Effect of the laser scan velocity on the MP morphologies

Figure 1(a) shows the SEM image of the laser track cross section of V50. Only V50 was chosen as a representative one because the MP shape is almost the same for all the parameters used in this study. A typical semi-ellipse-shaped MP could be observed as shown by the red dash line in figure 1(a). Thus, the width (W) and depth (D) of the semi-ellipse were used to describe the MP geometry. Figure 1(b) shows the profiles of the width and depth of the MP as a function of the laser scan velocity (V_L). It’s clear that the MP size is highly sensitive to V_L. With the increase of V_L, both W and D gradually decrease: from W=320 µm, D=94 µm at V_L=50 mm/s, to W=180 µm, D=60 µm at V_L=300 mm/s. This can be easily explained by the decrease of heat input with the increase of V_L.

4.2. Dendrite structure in the MP

The solidified grain structure in the MP of V50 can also be observed in figure 1(a). Figure 1(c) shows the corresponding microstructure zoomed in from the bottom of the MP as indicated by the yellow box in figure 1(a). It clearly shows a dendritic solidification structure under the present laser remelting conditions. Accordingly, the mean primary and secondary dendrite arm spacing (PDAS and SDAS) could be measured from the images. The measured results of the PDAS and SDAS are shown in table 1. Only V300 and V50 are included because only the samples with the highest and lowest V_L were representatively chosen for the characterization and the simulation in the following sections.

| Sample | V_L (m/s) | PDAS (µm) | SDAS (µm) | R* (K/s) | D/L | V* (m/s) | G* (K/m) |
|--------|-----------|-----------|-----------|----------|-----|----------|---------|
| V50    | 0.05      | 1.20      | 0.78      | 5.0e5    | 0.40| 0.02     | 2.5e7   |
| V300   | 0.30      | 0.56      | 0.32      | 6.7e6    | 0.30| 0.09     | 7.5e7   |

From the measured SDAS, the local cooling rate R* could be estimated by [10]:

\[
SDAS = 5.5 \left( -\frac{\Gamma_1 D_1 \ln \left( \frac{C_{\text{out}}}{C_0} \right)}{m_1 (1 - k_0) (C_{\text{out}} - C_0)} \right)^{\frac{1}{3}} \left( \frac{\Delta T_0'}{R^*} \right)^{\frac{1}{3}}
\]
where $\Gamma_{sl}$ is the Gibbs-Thomson coefficient, $D_l$ the diffusion coefficient in liquid, $C_o$ the concentration in the liquid, $C_{eut}$ the concentration in the liquid at the eutectic point, $m_l$ the liquidus slope, $k_0$ the binary equilibrium partition coefficient and $\Delta T'_0$ the non-equilibrium solidification range. By simplifying the Inconel 718 as a Ni-$5.2\text{Nb}$ binary system, these parameters are shown in table 2, which were calculated by fitting the solidification path of Inconel 718 using Thermodcalc and from reference [15]. The estimated $R^*$ by equations (1) are shown in table 1. The results indicate that both the PDAS and the SDAS decrease, while $R^*$ increases, with increasing $V_L$, which could be expected.

Table 2. The thermophysical properties of Inconel 718.

| $\Gamma_{sl}$ [15] | $D_l$ [15] | $C_o$ | $C_{eut}$ | $m_l$ | $k_0$ | $\Delta T'_0$ | $K$ [15] | $\alpha$ [15] |
|-------------------|------------|-------|----------|-------|------|--------------|--------|-------------|
| (Km)              | (m²/s)     | (at.%)| (at.%)   | (K/at%)| (K)  | (K)          | (W/m-K) | (m²/s)      |
| 3.65e-7           | 3e-9       | 5.2   | 18.56    | 16.00 | 0.33 | 145          | 30.1   | 5.5e-6      |

4.3. Estimation of the local tip growth velocity $V^*$

In order to simulate the columnar solidification structure with the mesoscopic model, it is important to get one more solidification conditions except $R^*$. Figure 2(a) shows the typical shape of the MP in the longitudinal section during the SLM process. It can be clarified that time the solidification front needed from the bottom of the MP to the surface (from B to C with the distance of the MP depth, D) should be the same with the time needed from the left boundary to the same position (from A to C with the distance of the MP length, L), as shown in figure 2(a). By assuming a constant tip growth velocity, the local velocity at the MP bottom, $V^*$, could be estimated from $V_L$, L and D by $V^* = D V_L / L$. Thus, it will be necessary to get the MP length during SLM process.

Figure 2. (a) Schematic diagram of the MP in the longitudinal section during SLM. (b, c) The Rosenthal’s solution of the MP on the plate surface for $V_{50}$ and $V_{300}$ during SLM, respectively. The red lines in (b) and (c) are the liquidus temperature of Inconel 718.

Rosenthal proposed an analytic solution for the temperature field around a moving point heat source, which travels over a semi-infinite medium along the positive x direction at velocity $v$ [16]:

$$T = T_0 + \frac{P}{2\pi Kr} \exp \left( -\frac{v(x + r)}{2\alpha} \right)$$  \hspace{1cm} (2)

where $T_0$ is the room temperature 298 K, $P$ the laser power, $K$ the thermal conductivity of the medium, $r$ the radial distance from the moving point, $\alpha$ the thermal diffusivity. Accordingly, the width,
W, and length, L, of the MP can be approximated. Figure 2(b) and (c) show Rosenthal’s solutions for V50 and V300 using the thermophysical properties of Inconel 718 in table 2, from which the ratio L/W of the MP was roughly estimated. With the experimentally measured W in section 4.1, the MP length L could be deduced. Correspondingly, the V* could be estimated by V*=DV/L, followed by the local temperature gradient, G*, by the equation G*=R*/V*, as shown in table 1. As expected, for both samples, V* is lower than V_L, and decreases with decreasing V_L.

4.4. Simulation of the dendrite structure by mesoscopic envelope (MS) model

Two simulations were performed on the simplified Ni-Nb binary system, according to the corresponding local solidification thermal conditions at the MP bottom of V50 and V300 in table 1 and the thermophysical properties of Inconel 718 in table 2. The widths of the domain (Ws) were chosen as 5 µm and 2.4 µm, with the grid sizes of 0.025 µm and 0.006 µm, for V*=0.02 m/s and V*=0.09 m/s, respectively. The moving frame computational domain is used, in which minimum distance between the most advanced tip and the top boundary is maintained. A symmetry boundary condition was used at the vertical sides and a fixed concentration C_0 was imposed at the top boundary.

The dendrites growth initiated from a planar envelope placed at the bottom of the domain at an initial supersaturation corresponding to the required V*. During an initial transient, the initial planar envelope destabilizes, protuberances with a small spacing appear and then the competition of the branches takes place until achieving a stable PDAS. The growth lengths to reach the steady state in the simulations are 20.5 µm and 4.3 µm for the cases V*=0.02 m/s and V*=0.09 m/s, respectively. The simulated PDASs in the steady state are shown in figures 3(a) and (b). The predicted PDASs are 0.84 µm and 0.28 µm for the cases V*=0.02 m/s and V*=0.09 m/s, respectively. The results by MS model can be compared to
classic geometrical models, such as two models developed by Hunt [17] and Kurz and Fisher [18], which give an average PDAS for steady growth as a function of $G^*$ and $V^*$, i.e., proportional to $G^{*0.5}V^{*-0.25}$. The comparison of the predicted PDASs by MS model with the two geometrical models is shown in figure 3(c), as well as the experimental ones. The results indicate that the MS model can predict the PDAS values and the PDAS variation with the $V^*$ in the selected regions and parameters.

5. Conclusion and perspectives
The effect of the laser scan velocity on the MP geometries and the solidified dendrite structure in the MP during the SLM of Inconel 718 was investigated by the single track laser melting experiments. Moreover, a simplified estimation of the local solidification conditions, such as cooling rate $R^*$, tip growth velocity $V^*$ and temperature gradient $G^*$, was proposed. In this estimation, the $R^*$, $V^*$ and $G^*$ were deduced from the SDAS and the MP geometries. Finally, using the estimated $R^*$, $V^*$ and $G^*$ as the input, the mesoscopic envelope model was used to simulate the PDAS selection of the columnar dendrite growth in the MP under two laser scan velocities. A good agreement between the experiment and the mesoscopic envelope model was achieved for both cases.

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