Solidification microstructure selection maps for laser powder bed fusion of multicomponent alloys

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Abstract. Solidification Microstructure Selection (SMS) maps provide a simple yet effective approach to predict the non-equilibrium solidification microstructure and grain morphology during Additive Manufacturing. In this study, SMS maps have been created for the Inconel 625 (IN625) alloy processed by Laser Powder Bed Fusion (LPBF). Toward this end, theoretical solid growth models, a model of the Columnar to Equiaxed Transition (CET), interface response theory, thermal simulation results and computational thermodynamics are utilized. The predicted microstructures are compared both qualitatively and quantitatively to experimentally-obtained micrographs. The theoretical analysis was also compared to the earlier analytical calculation for Al-10Si-0.5Mg alloy to show how differences in thermophysical properties affect the microstructural predictions. The theoretical predictions are shown to be in good agreement with the experimental results in terms of the resulting microstructure and dendrite arm spacings. A discussion on the use of SMS maps, formed over a broad range of thermophysical conditions, to help guide industry in improving LPBF microstructure, is provided.

1. Introduction
Laser Powder Bed Fusion (LPBF) is a mature Additive Manufacturing (AM) process that uses metal powder as a raw material, along with a laser heat source, to fabricate end-use parts with complex geometries. In metallic components, the mechanical properties are significantly affected by the microstructure—grain size, morphology, and crystallographic orientation. Although metal LPBF has been recently adopted on an industrial scale, the knowledge to optimize the microstructure and consequently mechanical properties of the final product remains inadequate. In this regard, the advanced investigation between processing parameters, solidification microstructure, and mechanical properties is necessary [1].

LPBF is characterized by extremely high solidification rates, \( V \), due to the high laser scan speed, \( V_s \), on the order of 0.1-1.5 m/s, and micron-sized melt pools, which thus leads to high thermal gradients, \( G \), on the order of mega \( K/m \). The laser power needs to be high enough to avoid a lack of fusion during laser melting, but not too high to produce keyhole-induced defects [2]. Large values of \( G \) and \( V \) change the diffusion length and finally lead to non-equilibrium effects at the solid/liquid interface during solidification [3]. Due to this complicated heat transfer condition at small length scales during LPBF, in situ microstructural observation is challenging. This makes numerical methods—including Phase-Field method (PF) and Cellular Automata (CA) as well as analytical approaches i.e. solidification microstructure selection (SMS) maps [4,5]—an effective alternative for investigating the relationship between solidification parameters and microstructure. Although numerical simulation of solidification is a powerful method for predicting microstructure, these methods are quite computationally expensive,
especially for exploring structure-processing relationships [5]. Unlike PF and CA, SMS maps provide faster and more comprehensive predictions of microstructure in non-equilibrium solidification [4] and, being computationally efficient, allow for investigation over a wide range of compositions and growth velocities in a short time [4].

SMS maps are graphical charts that show the stable solidification microstructure and grain morphology under specific solidification conditions. They are useful for controlling solidification morphology through manipulating the processing parameters in order to obtain AM components with specific properties. Two types of SMS maps can be defined. The first type identifies the stability region of different solidification structures for a set of growth velocities and alloy compositions at a constant thermal gradient. For LPBF, the relevant microstructures include planar, dendritic, eutectic, and banded morphologies. The second type identifies the critical combination of interface velocity and thermal gradient at which the Columnar to Equiaxed Transition (CET) occurs for the dendritic morphology in an alloy with constant composition [3, 4, 6].

The analytical prediction of solidification morphologies is based on a set of growth models that describe the behaviour of the solid/liquid interface during non-equilibrium solidification. They were first proposed by Kurz et al. for various types of solid growth during directional solidification and laser treatment applications [3,7]. Utilizing Hunt’s analytical model for the CET [8], later extended by Gauman et al. [6] to describe this transition during non-equilibrium solidification, columnar and equiaxed morphologies were also included. These analytical models, combined with thermal and thermodynamic data, can be used to create SMS maps [4].

This study aims to improve the AM design space for the popular multi-component Ni alloy Inconel 625 (IN625) processed by LPBF by combining knowledge of microstructure development gained through analytical predictive approaches and experimentation. The SMS maps were created for non-equilibrium solidification of this alloy. The microstructure of LPBF processed material, specifically the primary-dendrite-arm-spacing, was then characterized to validate the SMS maps both qualitatively and quantitatively. Finally, SMS map results for IN625 have been compared with previously published results for AlSi10Mg alloy [4]. These maps can be used to show how microstructure and properties are influenced by changing in solidification parameters and composition.

2. Creation of SMS maps

2.1. Description of material and phase formation

IN625 is a nickel-based superalloy which is strengthened mainly by solid solution strengthening via niobium, molybdenum, and chromium within the nickel-based (γ-FCC) matrix [9]. The nominal chemical composition of IN625 based on the ASTM F3056 standard is shown in Table 1 [10]. This alloy shows outstanding corrosion and oxidation resistance in elevated temperature/corrosive atmosphere conditions, as well as excellent yield strength, creep strength and fatigue strength due to the formation of intermetallic precipitates including γ”-Ni$_3$(Nb; Ti), δ -Ni$_3$(Nb; Mo), laves-(Ni; Cr; Fe)$_3$(Nb; Mo; Ti) and MC carbides in the γ-FCC matrix [9,11].

| Element | C  | Mn | Si | P  | S  | Cr | Co | Mo | Nb | Ti | Al | Fe | Ni  |
|---------|----|----|----|----|----|----|----|----|----|----|----|----|-----|
| Min (wt%) |    | -  | -  | -  | -  | 20.00 | -  | 8.00 | 3.15 | -  | -  | -  | Remainder |
| Max (wt%) | 0.10 | 0.50 | 0.50 | 0.015 | 0.015 | 23.00 | 1.00 | 10.00 | 4.15 | 0.40 | 0.40 | 5.00 |     |

Figure 1 shows an isopleth section of the Ni-Cr-Nb-Fe- Mo phase diagram with 21 wt% Cr, 5 wt% Fe, 9 wt% Mo, and 0.8 wt% Co calculated via the Thermo-Calc software using the TCNi9 database [12]. The composition of IN625 is shown by the dashed line. As can be seen, equilibrium solidification of this alloy results in a single solid phase, γ-FCC. However, based on the Scheil solidification path [12] and previous experiments, the microstructure of IN625 parts fabricated via LPBF contain multiple
phases: $\gamma''$-Ni$_3$Nb, laves, and (on rare occasions) $\delta$-Ni$_3$Nb in addition to $\gamma$-FCC(Ni-Cr) [9, 13-14]. As can be seen in figure 1, the $\gamma''$-Ni$_3$Nb and laves phases must have formed through a eutectic reaction.

2.2. Analytical calculation

The aforementioned Kurz’ and his colleagues’ non-equilibrium growth models and interface response theory, i.e. the relationship between the solid/liquid interface temperature and velocity, as well as Gaumann’s CET model, have been employed to create the type (1) and type (2) SMS ideas. These models and calculation procedures have been documented in the literature [3,7,8] and most-recently summarized in [4] and are not completely repeated, but they are briefly discussed in this paper.

The general idea is based on calculating the undercooling of the S/L interface for all possible solidification growth morphologies while taking into account the effect of interface velocity on thermodynamic parameters in order to estimate the interface temperature over a range of $C_{0,i}$, $G$, $V$, and finally, to predict the microstructure under non-equilibrium solidification conditions. The stable morphology is then the one with the highest interface temperature. The interface temperatures for planar, dendritic, and eutectic growth, i.e. the relevant morphologies for the IN625 system, are shown below,

$$T_{PL} = T_{m} \sum_{i} C_{0,i} m_{i}^{r} \frac{V}{\mu_{k,i}}$$

$$T_{D} = T_{liq} - \Delta T_{tip} \quad \text{with} \quad \Delta T_{tip} = \sum_{i} \left( \frac{k_{i} \Delta T_{0,i} \mu_{k,i}}{1-(1-k_{i})\mu_{k,i}} + C_{0,i}(m_{i}^{r} - m_{i}) + \frac{V}{\mu_{k,i}} \right) + \frac{2R}{\Gamma} + \frac{GD}{V}$$

$$T_{E} = T_{eut} - \Delta T_{eut} \quad \text{with} \quad \Delta T_{eut} = \Gamma \delta V + \frac{\delta}{\kappa_{i}}$$

where $T_{PL}$, $T_{D}$, and $T_{E}$ are the planar, dendritic, and eutectic interface temperatures, $\Delta T_{tip}$ and $\Delta T_{eut}$ are the dendrite tip and eutectic solidification undercooling, $T_{eut}$, $T_{liq}$, and $T_{m}$ are the equilibrium eutectic, liquidus, and pure metal melting temperatures, $V$ is the solidification velocity, $D_{l}$ is the diffusion coefficient in the liquid, $R$ the dendrite tip radius, $\Gamma$ the Gibbs-Thomson coefficient, $\mu_{k,i}$ the kinetic attachment, $\delta$ the lamellar spacing, and $C_{0,i}$, $m_{i}^{r}$ and $k_{i}^{r}$ the initial composition, velocity-dependent liquidus slope, and velocity-dependent partition coefficient. The $i$ subscript refers to the alloying element. The terms $m_{i}^{r}$ and $k_{i}^{r}$ are calculated via Aziz’ model [15] for solute redistribution during non-equilibrium solidification as $m_{i}^{r} = m_{0,i}(1 + (k_{i}^{r} - k_{i}^{r})(L - k_{i}^{r}))$ and $k_{i}^{r} = (k_{i}^{r} + Pe) / (1 + Pe)$ where $Pe$ is the Péclet number, $m_{i}$ and $k_{i}$ are the equilibrium liquidus slope and partition coefficient for element $i$.

The general equation of the CET model is defined as

$$G = \frac{1}{1+n} \sqrt{\frac{4mN_{0}}{3ln(1-\phi)}} \Delta T_{n} \left( 1 + \frac{\Delta T_{n}^{n-1}}{\Delta T_{n}^{n-1}} \right)$$

Figure 1. Isopleth section of Ni-Cr-Nb-Fe-Mo phase diagram with 21 wt% Cr, 5 wt% Fe, 9 wt% Mo, and 0.8 wt% Co - calculated via the Thermo-Calc software [12].
where $\Delta T_n$ and $N_0$ are the nucleation undercooling and nucleation density of the equiaxed grains, $n$ is a constant and $\phi$ is the volume fraction of the equiaxed grains.

Previous studies of LPBF processed IN625 have shown that the microstructure contains the $\gamma$-FCC(Ni-Cr) phase as a matrix with the texture in $<100>$ as well as $\gamma''$-Ni$_3$Nb and laves precipitates. Potentially, each of these phases could form during non-equilibrium solidification and as such the possible solidification microstructures that must be considered include: Planar- $\gamma$-FCC(Ni-Cr), dendrite-$\gamma$-FCC(Ni-Cr), planar- $\gamma''$-Ni$_3$Nb, primary- $\gamma''$-Ni$_3$Nb, planar-laves, primary-laves, eutectic ($\gamma$- $\gamma''$), eutectic ($\gamma$-laves) and the banded structure. Of these nine different microstructures, only four are probable, Planar- $\gamma$-FCC(Ni-Cr), dendrite- $\gamma$-FCC(Ni-Cr), eutectic ($\gamma$- $\gamma''$), and eutectic ( $\gamma$-laves), given the alloys' low Nb content. Thus, only these four microstructures will be considered within the calculation of SMS maps.

To perform the necessary calculations, material physical properties are needed. Thermodynamic data, including the equilibrium liquidus slope ($m$), partition coefficient ($k$), liquidus temperature ($T_{liq}$), and solidus temperature ($T_s$) are calculated with Thermo-Calc software using TCNi9 database [12]. Other needed properties were taken from the available literature. Furthermore, the thermal gradient of the LPBF melt pool must be known. Although sophisticated methods exist, we have used the Rosenthal solution [16]—an analytical solution to the heat conduction equation developed for welding that is able to estimate the temperature field around a moving heat source—to match the desire for a computationally efficient prediction of microstructure during LPBF. The general Rosenthal solution formula, equation (5), was utilized to predict the temperature history:

$$T = T_0 + \frac{\lambda P}{2kta} \exp \left[-\frac{V_s(x+r)}{2a}\right]$$

where $T_0$ is the temperature far from the heat source, $r$ is the radial distance from the moving point, $\lambda$ is absorptivity, $k$ and $\alpha$ are thermal conductivity and diffusivity, and $P$ is laser power. The thermal gradient for LPBF processing of IN625 is estimated to be $10^6$ to $10^{10}$ K/m within a meltpool when utilizing $P=200$ W and $V_s=1100$ mm/s as scanning power and velocity during single track LPBF processing.

3. LPBF single track experiments

3.1. Laser powder bed fusion experiment

In order to verify the microstructures predicted by the SMS maps, a single-track LPBF experiment has been performed utilizing the EOSINT M280 machine equipped with a 400 W Ytterbium fiber laser. The IN625 powder had a size distribution of 15-45$\mu$m and was processed using a power of $P=200$ W and scan velocity of $V_s=1100$ mm/s.

3.2. Characterization

After fabrication via AM, samples were prepared for metallography. First, samples were cut transverse cross-sectionally, mounted, polished and then etched chemically using aqua regia 3 HCl: 1 HNO$_3$. Then, the melt pool microstructure was investigated by a KEYENCE optical microscope (OM) and a JEOL JSM-7000F Scanning Electron Microscope (SEM) equipped with an Oxford AZtecHKL Electron Backscatter Diffraction (EBSD) detector.

4. Results

4.1. Qualitative comparison of the SMS maps of LPBF IN625 with experimental findings

Figure 2(a) shows the interface responses of the four solidification microstructures that are expected to form during LPBF processing of IN625 as a function of interface velocity at the thermal gradient of $2\times10^7$ K/m. Based on the maximum interface approach, dendrite-$\gamma$-FCC(Ni-Cr) has the most stable morphology in the processing range of LPBF, as expected. The information contained in figure 2(a) can
be used to create the SMS Maps. Figures 2(b) and 2(c) show the type 1 and type 2 maps, respectively, as well as the LPBF processing conditions. As can be seen in these maps, the dendritic $\gamma$-FCC(Ni-Cr) and banded-FCC(Ni-Cr) solidification microstructure, as well as fully columnar grains, are expected to form during LPBF processing of IN625. Figure 3(a) shows an OM micrograph of the transverse cross-

**Figure 2.** (a) The variation of interface temperature with solidification velocity for possible growth morphologies of the IN625 alloy at $G=2\times10^7$K/m, (b) Type (1) SMS map for the IN625 alloy at $G = 2\times10^7$K/m over a range of composition for Nb, (c) Type (2) SMS map for the IN625 alloy.

**Figure 3.** (a) OM (b) SEM micrographs, and (c) EBSD image of the transverse cross-section of IN625 single track melt with powder at scan velocity= 1100 mm/s and laser power= 200 W. The hump represents the deposited layer. Figures 3(b) and 3(c) provide the corresponding high-resolution SEM micrograph and the EBSD orientation map of the melt pool. As can be seen in figure 3(b), the microstructure consists of dendritic $\gamma$-FCC(Ni-Cr) with a small amount of $\gamma''$-Ni$_3$Nb and laves participates within the inter-dendritic region. As a result of the non-equilibrium
solidification process, the dendritic structure contains mainly primary arms with very short secondary arms that result in the structure appearing to have a cellular morphology. Further, at the base of the melt pool, figure 3(c), the columnar grains appear to have the same orientation as the substrate, providing evidence that they grew epitaxially towards the centre of the deposit. The epitaxial growth is related to the partial melting of the substrate while the elongated morphology is a result of the high thermal gradient and localized directional heat extraction at the edge of the melt pool.

Comparing figure 3(c) to the SMS map prediction in figure 2(c), it can be seen that while dendritic $\gamma$-FCC(Ni-Cr) is correctly predicted by the SMS map, the formation of the $\gamma''$-Ni$_3$Nb and laves participates is not anticipated from the analytical solution. On one hand, the phase diagram indicates that the $\gamma''$-Ni$_3$Nb and laves precipitates form as a solid-state transformation, and thus it is reasonable to not see them on the SMS map since only solidified microstructures can be predicted via the SMS map approach. On the other hand, Scheil solidification predicts their formation as a eutectic reaction. However, as the amount of Nb (4 wt%) in IN625 is much smaller than the eutectic composition (10wt% Nb for eutectic ($\gamma$- $\gamma''$) and 20wt%Nb for eutectic ($\gamma$-laves)) it is unlikely that the eutectic reaction is taking place. In fact, the SMS Type 1 map (figure 2(b)) shows that these phases precipitate as a eutectic only for a high amount of Nb (larger than 11 wt%) and at interface velocities much lower than the LPBF processing range. Based on these two points, it is likely that the $\gamma''$-Ni$_3$Nb and laves precipitates formed during solid-state phase transformation. The bonded $\gamma$-FCC(Ni-Cr) was also expected to form ahead of the columnar dendrites. However, it was not observed in the microstructure. This is likely because, as shown in figure 3(c), equiaxed dendrites have formed instead ahead of the columnar dendrites. Finally, comparing figure 3(c) to the SMS map prediction in figure 2(c), it would appear that while columnar solidification is correctly predicted by the SMS map towards the base of the melt pool, the formation of equiaxed grains was not anticipated by the analytical solution. These equiaxed grains have nucleated ahead of the columnar front due to a drop in the thermal gradient at the end of melt pool’s solidification. The calculation shown in figure 2(c) indicates that a CET will occur when the thermal gradient is less than approx. $5 \times 10^5$ K/m. Thus, it is likely that the thermal gradient near the top of the melt pool is significantly reduced as compared to what is predicted via the Rosenthal equation.

4.2. Quantitative comparison of primary arm spacing between experiment and calculation

Based on knowledge of the thermal gradient, Kurz and Fisher’s model [17] can be used to quantify the Primary Dendrite Arm Spacing (PDAS) during dendritic growth,

$$\lambda = 4.3 \left( \frac{\Delta T}{\alpha} \right)^{1/3} \left( \frac{D_f}{V_k \Delta T_0} \right)^{1/2}$$

where $\lambda$ is the primary arm spacing, $k$ the equilibrium partition coefficient, and $\Delta T_0$ and $\Delta T$ the equilibrium and non-equilibrium solidification range. The thermophysical parameters are listed in table 2, while the non-equilibrium solidification interval is given by $\Delta T = m^2 C_0 (R^2 - 1) / k^2$.

| Parameter | Unit |
|-----------|------|
| Initial composition, $C_0$ | 21Cr-9Mo-5Fe-4.1Nb-0.8Co wt% |
| Liquidus slopes for: $m_{\text{Nb}}, m_{\text{Nb}, \text{laves}}, m_{\text{Nb}, \text{Ni}_3\text{Nb}}$ | 1199, 1356, 737 K wt%$^{-1}$ |
| Partition coefficients for: $k_{\text{Nb}}, k_{\text{Nb}, \text{laves}}, k_{\text{Nb}, \text{Ni}_3\text{Nb}}$ | 0.5, 0.48, 0.37 - |
| Gibbs-Thomson coefficient for: $\Gamma_{\gamma}, \Gamma_{\text{laves}}, \Gamma_{\text{Ni}_3\text{Nb}}$ | $10^{-7}$, $2 \times 10^{-7}$, $2 \times 10^{-7}$ mK |
| Diffusion coefficient in the liquid, $D_f$ | $3 \times 10^{-9}$ m$^2$s$^{-1}$ |
| Material property, $n$ | 3.4 - |
| Nucleation undercooling of equiaxed grains, $\Delta T_n$ | 1.5 K |
| Nucleation density of equiaxed grains, $N_0$ | $2 \times 10^{15}$ m$^{-3}$ |
| Equilibrium solidification range, $\Delta T_0$ | 75 K |
Figure 4(a) shows a comparison between the experimentally measured PDAS and the values calculated with equation (6). The calculated values are shown by the solid and dotted lines, for two thermal gradients. The experimentally-measured points are given by black squares. As expected, the calculation shows that an increase in the growth velocity and thermal gradient decreases the PDAS. For the range of conditions [19] seen in LPBF processing, the calculated PDAS at $G=10^7$ K/m and $G=2 \times 10^7$ K/m are seen to change from 0.75 to 1.08 $\mu$m and 0.5 to 0.76 $\mu$m respectively for different growth velocities. These values match quite closely the experimental points, with locally-averaged measurements of 0.4 and 0.7 $\mu$m at two different positions within the melt pool, given uncertainties in materials properties especially $D_l$ and the potential for Marangoni-induced convection. The PDAS is seen to decrease from the melt pool edge to the center due to the increase in the solidification velocity and the decrease in the thermal gradient.

Figure 4. (a) Predicted PDAS over a range of growth velocity at the thermal gradient of $G=10^7$ and $2 \times 10^7$ K/m (b) experimental PDAS.

4.3. Comparison of IN625 and AlSi10Mg SMS maps
SMS maps can provide guidance to improve LPBF processed microstructure and understand phase formation. To give an example, the differences between the SMS maps of IN625, and AlSi10Mg [4], figure 5, are striking, owing to the different thermal and thermodynamic properties and compositions.

Figure 5. SMS maps for the AlSi10Mg alloy (a) Type (1) at $G = 10^6$K/m and (b) Type (2) over a range of Si compositions [4].
AlSi10Mg has a near-eutectic composition (12wt% Si), which leads to the formation of the eutectic microstructure. However, in IN625, the composition is far from the eutectic composition and as a result, the only predicted phase is dendritic γ-FCC(Ni-Cr). Furthermore, the grain morphology for LBPF processed AlSi10Mg is expected to be both columnar and equiaxed, whereas only the formation of columnar grains is predicted for IN625. Different parameters can affect the critical $G$ in the columnar to equiaxed transition, such as type and composition of alloying elements, thermodynamic parameters (liquidus slope and partition coefficient), nucleation density, and nucleation undercooling. The overall contribution of these parameters produces a lower critical $G$ for CET in IN625.

5. Conclusion
The modes of solidification and grain morphology formed during LPBF processing are dependent on the $G$, $V$, and initial composition of the material. $G$ and $V$ are a function of not only processing parameters but also the position within the melt pool. Due to the high $G$ and $V$ during LBPF processing, the formation of planar, eutectic, columnar dendrite, bands and equiaxed dendrite are expected. The SMS maps predict that columnar dendritic and banded microstructure form in LPBF microstructure of IN625. However, columnar to equiaxed transition has been observed in the experimentally micrograph of this alloy; this can be related to the drop in the thermal gradient in the center of the melt to the lower value than the predicted value by the Rosenthal solution. Since the heat source is moving, the amount of $G$ and $V$ are changing in the melt pool, which leads to the uneven distribution of solidification modes, grain size and PDAS in the single melt pool. The microstructure of LPBF processed IN625 mostly contains columnar dendrites with a PDAS ranging from 0.4 to 0.7 µm, in agreement with the analytical calculation results in terms of both the mode of solidification and primary arm spacing.

Aknowledgement
The authors acknowledge funding from the National Sciences and Engineering Council of Canada, and experimental support from McMaster’s Additive Manufacturing Research Group.

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