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Stability of cellular microstructure in laser powder bed fusion of 316L stainless steel

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ABSTRACT

Laser powder bed fusion additive manufacturing (L-PBF AM) offers great potential for local microstructure control. During this process, solidification occurs in conditions that are far from equilibrium and possesses – in the majority of cases – a strong directionality. In general, the size and morphology of the resulting microstructure is a function of two well-known parameters: the temperature gradient within the liquid phase (G) and the velocity of the solidification front (R). To provide guidance in selecting appropriate, systematically defined, process parameters for L-PBF of 316L stainless steel square pillars, we developed an intentionally simple thermal model to express these two parameters, G and R, as a function of selected process variables (laser scan speed, laser power) and material properties (thermal diffusivity). Results from both microstructural and mechanical characterization of the pillars indicate that high-strength, fully-dense parts with a highly oriented cellular microstructure can be obtained when using significantly different sets of process parameters. Furthermore, despite its simplicity, the numerical model correlates well with experimental evidence and confirms that rather than creating variable microstructures, the process parameter constraints actually lead to a stable cellular microstructure regardless of the wide process window studied.

1. Introduction

Microstructure tuning in metal-based additive manufacturing (AM) is fundamental in order to tailor location-specific mechanical properties of the alloys printed through this technology. It is therefore not surprising that an extensive body of literature has been dedicated to the understanding of solidification mechanics during both laser- and electron beam-melting of several commercially available alloys such as aluminum, steel, and nickel alloys. The study of solidification during metal additive manufacturing is not trivial, due to the complex, out-of-equilibrium nature of the process and the presence of the numerous physics-based phenomena that control the melt pool's heat and mass transport. King et al. [1] described the importance that several of these phenomena have in controlling the quality of additively manufactured parts: examples include laser absorption by the powder bed, convective fluid flow, surface tension-driven Plateau-Rayleigh instability, Marangoni thermocapillary convection, and recoil pressure.

In most cases, solidification during AM processes starts from the melt pool boundary and is directed inward (e.g., towards the center of the melt pool itself) following the well-known theory of directional solidification presented by Kurz and Fisher [2]. It is well established that directional solidification can be effectively described through the use of two distinct solidification parameters: the temperature gradient at the solid-liquid interface (G), commonly expressed in K/mm, and the growth rate of the solidifying front (R), expressed in mm/s. The product between these two quantities ($G{\cdot}R$, units of [K/s]) represents the cooling rate of the material within the solidification interval and therefore controls the scale of the resulting microstructure, with finer microstructures being achieved at higher cooling rates. On the other hand, the ratio between the temperature gradient and the growth rate (G/R, units of [Ksmm^{-2}]) controls the morphology of the solidified grains: as G/R is decreased, a transition from a planar solidification front to columnar cells, followed by columnar dendrites and finally by equiaxed dendrites is commonly observed. Several studies have been devoted to the quantification of these solidification parameters in order to predict resulting microstructure: Gäumann and coworkers [3] studied the directional solidification of CMSX-4, a nickel-based alloy, during laser welding, Wei et al. [4] investigated the solidification texture of Inconel 718 during directed energy deposition (DED) process, Dehoff et al. [5] focused instead on controlling dendrite morphology of the same alloy (Inconel 718) during electron beam melting (EBM), and finally Cloots et al. [6,7] devoted multiple studies to the microstructural characteristics of the IN738LC alloy manufactured through laser-powder bed fusion (L-PBF). In some cases, a high G/R ratio is sought, in

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order to induce the formation of a highly-oriented microstructure. This type of microstructure proves beneficial against creep failure, such as in the case of nickel-based high-temperature alloys, as demonstrated by Chen and coworkers [8] who proved that susceptibility to liquation cracking was greatly reduced when a highly oriented microstructure was obtained. In other cases, the interest is in achieving a low *G/R* ratio and thus attaining homogeneous nucleation from within the melt pool. This behavior is achieved by targeting the so-called Columnar-to-Equiaxed Transition (CET) and is especially sought for precipitation-strengthened alloys that are susceptible to solidification cracking (e.g., aluminum 6000 and 7000 series alloys, and γ' and γ''-reinforced nickel alloys). Solidification cracking tends to occur in alloys with large solidification intervals when isolated pockets of interdendritic liquid are trapped between solidified regions. Coniglio and Cross [9] explained in detail how the volumetric shrinkage due to both phase change and thermal contraction produce cavities that can span across the entire length of columnar grains, easily leading to cracks that can cross tens of printed layers in additively manufactured components. Moreover, Dehoff et al. [5] demonstrated that achieving CET and forming equiaxed grains is possible in EBM through the modification of the scanning strategy (e.g., through variations in electron beam current and speed function) while [10] further corroborated these results showing that – aside from beam current and speed function – the process parameter with the greatest effect on the formation of equiaxed grains is the preheat temperature of the powder bed. The high substrate temperatures achieved in commercially available EBM systems (1275 °C for IN718 presented by Raghavan et al. [10] allow lower thermal gradients and therefore lower *G/R* ratios, thus favoring spontaneous nucleation of equiaxed grains from within the melt pool. In a complementary study on DED-processed IN718, Chen et al. [8] demonstrated how active water cooling of the substrate enhances the epitaxy of the columnar dendrites by increasing thermal gradient *G*.

Unfortunately, most of the commonly available L-PBF machines only allow build plate preheat temperatures up to 200 °C and therefore achieving CET might prove more difficult for this process when compared to EBM. Recently, Martin and coworkers [11] were successful at forming equiaxed microstructures in additively manufactured aluminum alloys by using grain refining nanoparticle additions to lower the energy barrier for homogeneous nucleation from within the melt. Alternatively, modifications of the spatial energy distribution of the laser beam has yielded successful microstructure control in L-PBF: for instance, Roehling et al. [12] used an elliptical beam shape to promote CET of 316L stainless steel, while Cloots et al. [6] adopted a "doughnut-shaped" beam laser source to control solidification of IN738LC. In both cases the temperature distribution in the melt pool was significantly different from that produced by a conventional, round Gaussian laser beam, and homogeneous nucleation seemed thus to be favored. Nevertheless, a systematic study on microstructural conditioning of L-PBF-processed materials through the control of selected process parameters is currently lacking.

We have chosen to study L-PBF-processed austenitic stainless steel 316L because it commonly solidifies with a columnar cell morphology and the degree of epitaxy of this microstructure also seems controllable by changes in the power of the laser used; Niendorf et al. [13] have in fact highlighted the microstructural differences of 316L stainless steel processed using a 1 kW high power laser vs a conventional 400 W laser. Nevertheless, a systematic and quantitative correlation between process parameters (e.g., laser power, scan speed) and solidification parameters (*G, R*) was not presented in their work and, to the best of our knowledge, has not been previously investigated. The purpose of the present work is therefore to show how a relatively simple, yet validated, analytical thermal model can provide quantitative relationships between process parameters and solidification parameters and thereby provide guidance to predict the resulting microstructure in terms of length scale, morphology, and orientation with a sufficient degree of accuracy. Moreover, the results presented in this work were obtained without requiring cumbersome computational efforts but were reduced to simple calculations based on a "spreadsheet" simulation. The model can also be swiftly adapted to other materials as long as their basic physical properties are known. The corresponding experimental matrix was equally straightforward, designed to systematically explore the broadest possible process window available in L-PBF, without the complexity of changing laser beam shape or intensity.

Table 1
List of process parameters and material properties for 316L stainless steel used in Rosenthal's solution to compute temperature distribution in the melt pool.

Property	Value	Units	Ref.
Laser power (P)	100, 150, 200, 250, 300, 350	W	
Scan speed (v)	500, 800, 1400, 2200, 3000	mm/s	
Substrate temperature (T_0)	200	°C	
Melting temperature (T_m)	1375	°C	[21]
Thermal conductivity (k)	29.5	W/mK	[22]
Density (ρ)	7318	kg/m^3	[23]
Thermal diffusivity (α)	0.05	cm^2/s	[16]

2. Experimental methods

2.1. Model for calculation of solidification parameters

In order to gain insight into the expected temperature distribution both inside and outside the melt pool, a steady-state (e.g., "quasi-stationary") temperature field was computed using the well-known analytical solution for a moving point heat source introduced by Rosenthal [14]. To effectively employ this solution, it is necessary to introduce the following assumptions: latent heat of solidification is neglected, as well as convective and radiative cooling into the surroundings. Fluid flow within the melted region is also ignored and thermal properties are assumed constant throughout the calculations (Table 1), their values are taken at the melting point because this is the temperature at which most of the energy is delivered to the material. Although these simplifications may seem significant, Mukherjee et al. [15] have recently shown good agreement between experiments and a model analogous to the one used in this work. Furthermore, Rubenchik and coworkers [16] have explained how the presence of a powder layer can also be ignored without committing significant error (both the time and energy required to melt the powder layer alone are negligible when compared to those required to melt the underlying substrate), hence the physical properties listed in Table 1 are taken as bulk material values. Rosenthal's approach also considers the laser beam energy as focused in one point, thus yielding a non-physical infinite temperature value under the laser spot. More realistic analytical models would include a Gaussian energy distribution around the center of the laser beam, as utilized in the well-known solution proposed by Eagar and Tsai [17]. Nevertheless, Hunziker et al. [18] demonstrated how, at sufficient distance from the laser source (e.g., at the solid-liquid interface towards the cooler portion of the melt pool), the differences in temperature distribution between Rosenthal's solution and Eagar-Tsai's become irrelevant, and the first solution can be used as an approximation of the latter. Despite the simplifying assumptions described above, the present work shows how realistic approximations of temperature distribution, as well as cooling rates and solidification parameters are achievable with very little computational effort. An example is provided in Fig. 1, where typical cooling rates within the solidification interval are displayed with respect to the linear energy input (laser power *P* divided by scan speed *v*) for different parameter combinations. Results from experimental measurements and a heat conduction model developed in COMSOL by Scipioni Bertoli et al. [19] are shown along with results from a more complex heat transfer and fluid flow model developed by Mukherjee et al. [15] and along with results from the present work using Rosenthal's solution, the details of which are provided below. As