Melt Pools, Process Parameters, and Defects in LPBF UNS N07718

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Chapter 1: Abstract

In this work, mathematically derived relations from the Rosenthal equation are used to predict the melt pool dimensions of UNS N07718 samples created using the laser powder bed fusion (LPBF) process. Various laser powers (P) and scan speeds (V) were used to create the samples in this work; however, it was determined that the recommended standard conditions result in keyhole-shaped melt pools. The predicted dimensions are then compared to the measured values on plots where $(P/V)^{1/2}$ is on the x-axis. Another mathematical relation is derived to predict the groove depth between samples with varying hatch spacing, where the melt pool is approximated by two half-ellipses with different minor axis values. Actual measurements of the groove depth are compared to the predictions on plots where the ratio of hatch spacing (H) to melt pool width (W) is on the x-axis. This work also utilized a previously derived geometrical relation to predict which of the fabricated samples were expected to have lack-of-fusion (LOF) porosity.

The other part of this work explores the formation of both incorporated and surface aluminum oxides. Incorporated and surface oxide concentration are plotted against certain variables to determine potential correlations. Incorporated oxide area fraction was also used as a means of determining whether the location where a part is built on the build plate affects the defect concentration. The idea of oxide flotation in a molten UNS N07718 matrix is tested by conducting analysis of the oxygen content at the top and bottom of tall samples. Finally, compositional analysis was done on samples made with different powders to determine whether elemental composition differences could explain the variance in hot cracking behavior seen.

Chapter 2: Project Motivation

The motivation for the work presented in this document is to understand the connection between process parameters used to create UNS N07718 parts via laser powder bed fusion (LPBF) and the resulting melt pool geometries, surface profilometries, porosity, and oxide formation. Measurement of melt pool geometries provides input for a simple mathematical relation to determine whether lack-of-fusion porosity will be present in a sample. The measurements also help with determination of the keyholing boundary for UNS N07718, where keyhole porosity becomes a concern. Furthermore, the melt pools associated with keyholing provide insight into oxide residence inside and on the top surface of parts. Keyhole-shaped melt pools exhibit an increase in the vapor plume size, which allows for more oxidized spatter to form. The surface profilometry, which is correlated with the cap height of newly deposited material and extent of overlap of consecutive melt pool tracks, may then explain how the oxides are reworked into the sample as it is being built. Ultimately, oxides and porosity decrease the density of the final part and serve as initiation sites for fatigue cracks that limit the performance of the material. Therefore, this work is needed to establish a connection between process parameters and results with the aim of being able to predict expected oxide and porosity concentration in LPBF UNS N07718 parts.

Chapter 3: Background and Literature Review

3.1 Background Information

3.1.1 About UNS N07718

Superalloys are heat-resisting alloys based on nickel, nickel-iron, or cobalt that exhibit a combination of mechanical strength and resistance to surface degradation [1]. Nickel-based alloys are utilized in load-bearing applications at temperatures more than 80% of their incipient melting temperatures, a fraction that is higher than that for any other class of engineering alloys [2]. The principal characteristics of nickel as an alloy base are the high phase stability of the nickel FCC, γ matrix and the capability to be strengthened [3]. This stability can be further improved by alloying with chromium and/or aluminum [3]. UNS N07718 is a nickel-based superalloy with additions of chromium, niobium, and molybdenum to overcome cracking problems during welding [2]. The typical composition of UNS N07718 powder is shown in Table 3.1 as well as measured compositions for powders and samples used in this work.

Element	Ni	Cr	Fe	Nb	Mo	Ti	Al
Typical WT %	50 - 55	17 – 21	15 – 21	4.75 - 5.5	2.8 - 3.3	0.75 – 1.15	0.2 - 0.8
LOC 12	54.2	19.3	17.1	5.38	3.68	1.01	0.593
PAC 5	54.5	18.7	17.9	5.59	3.13	0.974	0.394
PRAX 5	53.3	19.8	18.8	5.40	3.21	0.911	0.572
PAC PWDR	51.2	19.7	18.5	5.48	3.19	1.11	0.480
PRAX PWDR	53.2	19.0	18.2	5.06	3.01	0.940	0.520

 Table 3.1: Chemical Composition of UNS N07718 Powder Alloy [4]

Superalloys derive their strength mostly from solid-solution hardeners, such as iron and chromium, and precipitated phases. For UNS N07718, these are primarily the γ ' and γ " phases [5]. The γ ' phase is a principal strengthening phase in many nickel-base superalloys. Its crystal structure is FCC (ordered L1₂) and the formulas seen in UNS N07718 are Ni₃Al and Ni₃Ti. The γ " phase is a principal strengthening phase particular to UNS N07718 and usually precipitates on the {100} planes. Its crystal structure is BCT (ordered DO₂₂), and the formula seen in UNS N07718 is Ni₃Nb [6].

Unfortunately, some of the elements added to increase the favorable formation of γ ' and γ '' can participate in the formation of detrimental phases. The η phase is found in superalloys with high

ratios of titanium to aluminum. Its crystal structure is HCP (ordered DO₂₄) and the formula seen in UNS N07718 is Ni₃Ti. The δ phase is observed in over-aged UNS N07718 and has an acicular shape when formed. Its crystal structure is orthorhombic (DO_A) and the formula seen is Ni₃Nb. Laves phase is typically found in UNS N07718 as well [7]. It has a hexagonal crystal structure, and the high concentration of niobium may allow for the formation of Fe₂Nb [6]. These phases are brittle and can therefore lower the rupture strength and ductility of UNS N07718 [5]. However, the stability of γ " and reduction in the amount of δ can be obtained via appropriate chemical manipulation [8]. Other detrimental phases, such as σ and μ , are not likely to form in UNS N07718.

Use of Thermo-Calc to create phase diagrams for Ni-based superalloys approaches the levels of accuracy usually associated with experimental measurement. The field of computer aided thermodynamics makes it possible to predict the phase behavior of multi-component alloys [9, 10]. Below is a pseudo-binary phase diagram generated for UNS N07718. The mass fraction of niobium is on the x-axis and temperature is on the y-axis. A similar diagram was found from literature for comparison.



Figure 3.1: Pseudo-Binary Phase Diagram of UNS N07718: Thermo-Calc (left) and From Literature (right) [11]

The diagrams in Figure 3.1 compare favorably. It has been shown experimentally that the cooling rate influences the segregation of niobium [12]. The cooling rates for laser melting are high ($\sim 10^5$ - 10^6 K/s), which may allow for the formation of Laves phase in laser powder bed fusion via the significant segregation of niobium [13].

3.1.2 Laser Powder Bed Fusion (LPBF)

The emergence of additive manufacturing has been great for the production of UNS N07718. Typically, UNS N07718 is manufactured at very high temperatures; because at room temperature, excessive tool wear, low material removal rates, and poor surface finish are major concerns [14,15]. For this work, the systems used to generate the parts for analysis and testing utilize the laser powder bed fusion additive process. Those systems are an EOS M290 located at Carnegie Mellon University's NextManufacturing Center and an SLM Solutions machine located at NASA Langley Research Center (LaRC). In this process, a computer aided design (CAD) file is turned into a finalized part through the following steps: 1) A CAD file is either created from scratch, downloaded or created from laser scanning an existing part, 2) The CAD file is input into the planning software, where it can be sliced, the part can be oriented, and support structures can be added, 3) The build file, which contains everything needed to build the part, is created, 4) The build is input into the LPBF machine for printing, 5) During fabrication, layers of fine metal powder are deposited by a powder bed and a high power laser fully melts the powder together, and 6) The printed part undergoes any necessary post processing before it is finished [16].

The geometry of the generated melt pools is a major topic for the additive manufacturing of metals. When the laser interacts with the substrate, a melt pool of a certain width and depth is formed as seen in Figure 3.2 (left) [17]. The process parameters chosen, have a direct effect on the sizes of these features. The main process parameters discussed in literature are the power of the laser and its scanning speed. However, two other parameters play a significant role. These would be the hatch spacing (distance between the centers of consecutive melt pools) and layer thickness (vertical distance that the build plate moves), as shown in Figure 3.2 (right) [18].



Figure 3.2: Schematic of Melt Pool Width and Depth (left) [14], Hatch Spacing and Layer Thickness (right) [15]

In fact, plots of laser power versus scanning speed can be broken into regions delineating characteristic melting. At high laser power and low scanning velocity, melt pools will begin to take on a keyhole shape. The aspect ratio of the resulting melt pool can be calculated as the ratio of the melt pool depth to half its width. If the aspect ratio is less than 1, then the melt pool formation mode is called conduction mode [19]; however, if the ratio is greater than 1, then the melt pools

switch to a keyhole mode, where the intensity of the laser beam at the center is high enough to cause deep penetration of the melt pool and vaporization of the material. Typically, a laser intensity of $\sim 0.5 - 1$ MW/cm² is enough to cause keyholing in most metals. Increasing the laser intensity further leads to the aspect ratio exceeding 2.5, which shifts the melt pool into severe keyholing [19]. Although being in the keyholing area of P-V space will guarantee full melting of the material, it also could introduce spherically shaped pores of entrapped gas. Keyhole porosity is dependent on the equilibrium between the two opposing pressures acting on the keyhole. Recoil pressure, which occurs due to the evaporation of the molten metal and its ejection in the form of a vapor, induces a pressure to keep the keyhole open. Conversely, Marangoni convection due to surface tension, resulting from the temperature gradient across the melt pool surface, acts to close the keyhole [20]. As the vapor cavity diameter increases, the recoil pressure becomes greater than the Marangoni convection, causing a stable open keyhole. The vapor cavity of the melt pool becomes very unstable, and thus the cavity pinches off spherical pores that are solidified in the material before escaping. However, as the vapor cavity increases beyond the laser beam diameter, the recoil pressure decreases and is overcome by the surface tension leading to the closure of the keyhole. In such cases, the keyhole might not form pores [21].

On the opposite side of the spectrum, there is the lack-of-fusion region where the material is printed with low laser power and high scanning velocity. This area is of concern due to incomplete melting of the material since the speed of the laser will not allow for enough energy to be put into the material to cause full melting. Another area of concern is the balling (or bead-up) region, which is located at high laser powers and scanning velocities; however, this work will not touch on that region. Overall, these areas of concern outline what is termed the process window for a given material. A schematic outlining these regions of P-V space is shown in Figure 3.3 below.



Figure 3.3: Diagram of P-V Space

Operation within the process window is recommended to achieve parts that undergo full melting and minimize the amount of porosity left in the sample.

3.1.3 Relative Stability of Oxides

During building of the samples, the chamber was filled with an inert gas (argon), so that reactions between the surroundings and the specimen are eliminated. Unfortunately, the argon gas used was not completely pure and did contain 0.1% oxygen. Therefore, oxidation of the material during building did occur, which resulted in the formation of oxides. Oxides are detrimental to part performance as they serve as initiation sites for cracking under fatigue and may also have an effect on the part surface as it is being fabricated. Therefore, it is important to understand their formation. For UNS N07718 parts, the main oxide created is alumina and the reason for this can be explained using a Richardson-Ellingham diagram.

Ellingham diagrams are graphs based on the principle that the thermodynamic possibility of a reaction occurring is dependent on the sign of the change in Gibbs free energy ($\Delta G = \Delta H - T\Delta S$), where ΔH is the change in enthalpy and ΔS is the change in entropy. These diagrams plot the standard Gibbs free energy change for oxidation reactions as a function of temperature. For comparison, all reactions considered are for the same quantity of oxygen, which is typically one mole. An Ellingham diagram for the major metals in UNS N07718 is shown below in Figure 3.4.

The lines are drawn for pure elements (whereas the alloy is a solution, with lower activities), but serve to indicate the general trend.



Figure 3.4: Ellingham Diagram for Metals in UNS N07718

The curves in Ellingham diagrams are straight lines with positive slopes, where the slope is proportional to the change in entropy. As the position of a metal's line moves further down in the diagram, the greater is the stability of that oxide. Also, in comparing two metals at a given temperature, the metal with the lower Gibbs free energy of oxidation on the diagram will reduce the oxide of the higher Gibbs free energy. Applying this information to the graph in Figure 3.4, one can see that formation of aluminum oxide is favorable for all temperatures up to 2000 °C since this reaction has the lowest change in Gibbs free energy. As higher temperatures are approached, the line for titanium oxidation becomes closer to that of aluminum. Therefore, it might be possible to see formation of aluminum titanates in the fabrication of UNS N07718 parts as well.

Since alumina is present in and on printed UNS N07718 parts, there are three possible formation mechanisms to consider. The first is direct oxidation of the melt pool by the 0.1% oxygen in the chamber gas. This possibility was tested by estimating the oxide thickness which can form on the melt pool surface before solidification [22]. This was based on the limiting case of rate control by

mass transfer of oxygen in the gas phase to the melt pool surface. The calculations estimated an oxide thickness of around 1 nm resulting from direct melt pool oxidation, which is small compared to the estimated average thickness of the deposited oxide of 500 nm for UNS N07718. Therefore, it was concluded that this mechanism is not the main cause of oxide inclusion formation in LPBF UNS N07718 parts.

The second mechanism is oxidation of hot, liquid spatter ejected from the melt pool as it travels through the chamber atmosphere. It has been noted that for spatter to oxidize, both the reaction rate and temperature need to be high enough [22]. Like the first mechanism, the oxidation of aluminum from molten UNS N07718 is limited by oxygen mass transfer to the spatter. However, it was calculated that the reaction is rapid for droplets up to tens of microns in size, which is corroborated by the observed oxides in and on top of built UNS N07718 parts being less than 10 microns in size [23]. Therefore, it was concluded that spatter oxidation is likely to contribute to the oxides seen in LPBF UNS N07718 parts.

The final mechanism considered is that of reworking of the existing oxide film on powder particles. Although this film is thin, the melt pool temperature may be high enough to cause agglomeration of these films into thicker particles [22]. It has been shown that the surfaces of samples built with UNS N07718 powder exhibited some streaks of oxides close to and parallel with the melt pool edges [23], which can also be seen in Figure 5.8. Therefore, it appears likely that the oxide on powder, and the oxide deposited by spatter, can be reworked by melting.

3.2 <u>Literature Review</u>

3.2.1 Rosenthal Equation

The Rosenthal equation is a solution to the moving heat source problem. The solution makes some simplifying assumptions including: 1) conduction as the main mode of heat transfer (ignoring convection and radiation), 2) constant thermal properties, 3) a semi-infinite build plate thickness, 4) a point heat source, and 5) a steady-state solution [24]. When applying these to additive manufacturing, the assumptions are fair to make. These assumptions lead to the following equation:

$$T = T_0 + \frac{\varepsilon P}{2\pi Rk} \exp\left(\frac{-V(\xi + R)}{2\alpha}\right)$$
(1)

Using further mathematics, we can develop an equation that gives a predicted melt pool width based on the power (P) and velocity (V) of the laser. Because of the assumption that heat-transfer

in the melt pool is by conduction only, the resulting shape of the melt pool cross section is semicircular and allows for the equation below showing the association between melt pool width (W) and depth (D) [25].

$$W \approx \sqrt{\frac{8\varepsilon P}{e\pi\rho CV\Delta T}} \text{ and } D = \frac{W}{2}$$
 (2)

The Rosenthal relation was chosen as the theoretical model for this work, because comparisons of the dimensions obtained using this relation and actual melt pool measurements have been made with good results [26]. The results from the Rosenthal relation are also similar to those achieved using a finite element model that considers temperature-dependent properties, the Gaussian distribution of the heat source, and heat losses from natural convection and radiation [27]. Therefore, the ability to predict melt pool dimensions without having to incorporate a plethora of temperature-dependent variables made calculations easier and faster without sacrificing accuracy, especially at low energy input levels.

Table 3.2 below explains what each of the variables are in the above equations (where $\Delta T = T_f - T_0$) and lists the values of the material properties for UNS N07718 [28]. It must be noted that the absorptivity of UNS N07718 varies depending on the wavelength of the laser, but the value of 0.35 agrees well with data seen in literature, for non-keyholing conditions [29,30]. Once keyholing begins, the absorptivity has been shown to increase quickly and approach values of approximately 0.8. Over this range of absorptivities, there is a linearly correlated increase in melt pool width, as well as a more drastic linear increase in melt pool depth, as the absorptivity increases [31]. Table 3.2: Material Properties of UNS N07718

Variable	Definition	Value
α	Thermal diffusivity [m ² /s]	2.80 * 10 ⁻⁶
С	Heat capacity [J/kg*K]	435
3	Absorptivity [-]	0.35
k	Thermal conductivity [W/m*K]	10
ρ	Density [kg/m ³]	8200
T_0	Initial temperature of the base plate [°C]	25, 80
T _f	Melting temperature [°C]	1298

3.2.2 Index Relation

To predict whether lack-of-fusion porosity will be present in a sample, a mathematical relation was developed as follows [25], with Figure 3.5 showing important variables. First, the shape of

the cross-section of a melt pool is approximated as two half-ellipses. The half-ellipse outlining the re-melted region has a (horizontal) major axis of length W (melt-pool width) and a (vertical) minor axis length of (2D-2R), where D is the total melt pool depth, and R the height of the "cap" (newly deposited material). The half-ellipse describing the cap has the same major axis length (W) as the re-melted region, and a minor axis length of 2R. The vertical dimension of the region of overlap between two adjacent melt pools is L^* . As the figure below shows, L^* is the sum of y_2 (overlap in cap region) and y_1 (overlap in re-melted region). The values of y_1 and y_2 follow from the equation of an ellipse; for example, y_2 is the y-coordinate of the upper ellipse when the x-coordinate is equal to H/2:



Figure 3.5: Schematic Showing Important Variables for LOF Porosity Prediction

$$\left(\frac{y_2}{R}\right)^2 = 1 - \left(\frac{\frac{H}{2}}{\frac{W}{2}}\right)^2 = 1 - \left(\frac{H}{W}\right)^2 \rightarrow y_2 = R\sqrt{1 - \left(\frac{H}{W}\right)^2}$$

Similarly, $y_1 = (D - R)\sqrt{1 - \left(\frac{H}{W}\right)^2}$
The total overlap depth is given by $L^* = y_1 + y_2 = D\sqrt{1 - \left(\frac{H}{W}\right)^2}$

The criterion of full melting is that the layer thickness (L) must be smaller than L^* ;

This can be rewritten as follows:
$$L \le D \sqrt{1 - \left(\frac{H}{W}\right)^2} \to \left(\frac{L}{D}\right)^2 \le 1 - \left(\frac{H}{W}\right)^2$$

This can then be rearranged into the final relationship:

$$\left(\frac{L}{D}\right)^2 + \left(\frac{H}{W}\right)^2 \le 1 \tag{3}$$

The final mathematical relation developed, known as the index relation, will be used to accurately predict lack-of-fusion porosity in LPBF of UNS N07718 parts. If the index is a number less than or equal to one, the relation predicts for there to be no lack-of-fusion porosity as the melt pool depth (D) and width (W) are larger than the layer thickness (L) and hatch spacing (H), respectively. Only when the index is larger than one does the relation predict lack-of-fusion porosity to be present [26].

3.2.3 Groove Depth Derivation

As a part is being built, each melt pool contains previously deposited material that is re-melted. Newly deposited material, which is the powder melted into the pool, then forms a "cap" on top of the previously deposited layers. Both the re-melted material and the cap are assumed to be half-ellipses, with their horizontal major axis equal to the melt pool width and located at the surface of the previously deposited layer. This assumption was found to approximate many real melt pools and simplifies the mathematical derivation [25]. Figure 3.6 provides a diagram of overlapping, adjacent melt pools and defines important variables.





For process conditions where the melt pools fully overlap laterally (H < W) and in the vertical direction, the height of the cap (R) is found from the melt pool geometry. The derivation for this variable is shown below. First, one can start with the double integration necessary to find the area under an ellipse with a major axis length of 2a and minor axis length of 2b:

$$Area = \int_{-a}^{a} \int_{-b\sqrt{a^2 - x^2}/a}^{b\sqrt{a^2 - x^2}/a} dy \, dx$$

Next, one must change the limits of integration for y to y = 0 and $y = b\sqrt{a^2 - x^2}/a$, since only the top half of the ellipse is being considered.

$$\rightarrow Area = \int_{-a}^{a} \int_{0}^{b\sqrt{a^{2}-x^{2}/a}} dy \, dx = \int_{-a}^{a} \frac{b}{a} \sqrt{a^{2}-x^{2}} \, dx$$
$$\rightarrow Area = \frac{b}{a} \left\{ \frac{1}{2} \left[x\sqrt{a^{2}-x^{2}} + a^{2} \sin^{-1}\left(\frac{x}{|a|}\right) \right] \right\}_{x=-a}^{a}$$

Next, one must change the limits of integration for x to x = 0 and x = H/2 since the only part of the ellipse under consideration is from the center of the ellipse to the location of the groove depth along the major axis. One must also note that a = W/2 and b = R for this derivation as seen in Figure 3.6. Finally, the area under the ellipse being considered must equal to area of material deposited, which is simply HL/2.

$$\rightarrow Area = \frac{2R}{W} \left\{ \frac{1}{2} \left[x \sqrt{\left(\frac{W}{2}\right)^2 - x^2} + \left(\frac{W}{2}\right)^2 \sin^{-1}\left(\frac{2x}{W}\right) \right] \right\}_{x=0}^{H/2} = \frac{HL}{2}$$
$$\rightarrow Area = \frac{2R}{W} \left\{ \frac{1}{2} \left[\left(\frac{H}{2}\right) \sqrt{\left(\frac{W}{2}\right)^2 - \left(\frac{H}{2}\right)^2} + \left(\frac{W}{2}\right)^2 \sin^{-1}\left(\frac{H}{W}\right) \right] \right\} = \frac{HL}{2}$$

Simplifying the above relation leads to the relationship shown in the equation below [25].

$$R = \frac{2L}{\sqrt{1 - (H/W)^2} + (W/H)\sin^{-1}(\frac{H}{W})}$$
(4)

Now, this result can be pushed further to find the depth of the groove located between the overlapping melt pools. By utilizing the result for the cap height (R), the equation below was derived for the groove depth (GD), where y_2 is the y-coordinate of the upper ellipse when the x-coordinate is equal to H/2 (shown in Figure 3.5).

$$\left(\frac{y_2}{R}\right)^2 = 1 - \left(\frac{\frac{H}{2}}{\frac{W}{2}}\right)^2 = 1 - (H/W)^2 \to y_2 = R\sqrt{1 - (H/W)^2}$$
$$GD = R - y_2 = R(1 - \sqrt{1 - (H/W)^2}) \tag{5}$$

As mentioned previously, the surfaces of samples built with UNS N07718 powder exhibited some streaks of oxides close to and parallel with the melt pool edges [23]. The location of the groove depth is at the melt pool boundaries and may play a role as part of the surface profile in the reworking and residence of oxides. Therefore, it is important to characterize whether the groove depth can be controlled by variation of the extent of overlap between melt pools.

3.2.4 Melt Pool Size Measurements

In looking through literature, there were different approaches to measurement of melt pools for LPBF of UNS N07718. *Cheng et al.* measured melt pool widths of UNS N07718 fabricated using a Concept Laser M1 machine. The measurements were done on processed images taken via thermal imaging of the samples. Melt pool widths were gathered at various heights during the construction of the builds as well as lengths. However, this process was only done for a constant power of 185 W and three speeds: 400 mm/s, 600 mm/s, and 800 mm/s. They ultimately found the melt pool width to remain constant throughout the height of a sample and noted a decrease in the width with increasing laser speed [32]. The main differences of note are that there is no measurement of melt pool depth nor is there a wide breadth of P-V combinations to measure.

Sadowski et al. constructed 24 UNS N07718 bases using an EOS M280 machine. The laser power (285 W), scan speed (960 mm/s), hatch spacing (0.11 mm), beam diameter (75 µm), and beam offset (0.015 mm) were chosen to achieve consistent base microstructures and good foundations for the subsequent scan lines of interest. Each of the bases had 10 equally spaced scan lines on top that were created using different sets of laser power and scan speed. The bases were then sectioned, polished, and etched to reveal the melt pools for measurement. Images were taken both from a cross-sectional and top view for measurement of the melt pool/line widths; and only the cross-sectional view allowed for measurement of the melt pool depths. Ultimately, they found both dimensions to increase with increasing laser power and increase with decreasing laser speed. Their work also investigated potential correlations with ratio of power to velocity, or laser energy density (LED), and concluded that LED values above 0.21 J/mm result in nearly perfectly filled lines, but also sacrifice dimensional accuracy [33]. *Sadowski et al.* took an interesting approach of analyzing melt tracks on top of built parts, which is not done in the work presented in this document. They also showed correlation between LED and melt pool dimensions, which will allow for comparison between their data and the data gathered in this work.

Scime et al. covered a wide range of P-V space with powers between 100 W and 370 W and beam velocities between 200 mm/s and 1400 mm/s. Their work was also done on an EOS M290 machine with a surface ground UNS N07718 plate to maintain a consistent powder layer thickness. Both melt pool widths and depths were measured using ImageJ software. However, all the measurements in their work were done on 20-mm long melt tracks that were spaced 500 µm apart, and not on built samples like the work presented in this document. They also made use of the Rosenthal relation for width, but the material properties input into the relation were evaluated at 1427 °C from thermophysical data to roughly fit their results [34]. Furthermore, the initial temperature input was the preheat temperature of 80 °C and the melting temperature was taken to be the liquidus temperature (1336 °C) of UNS N07718. Figure 3.7 below shows the P-V space diagram generated by *Scime et al.*



Figure 3.7: P-V Diagram for UNS N07718 by Scime et al.

This process window provides a good idea of what to expect, but there are some important distinctions to make when comparing these results to those in this work. First, it must again be stated that these are measurements taken from melt tracks that have no extent of overlap. In fact, the 500 μ m hatch spacing was chosen to ensure that adjacent melt pools did not overlap. This lack of overlap also eliminates the possible effects of the denudation zones of adjacent melt pools, which can extend between 100 μ m and 200 μ m from the center of the melt pool [35]. Finally, it is noted that *Scime et al.* chose to define severe keyholing as when the ratio of the melt pool depth to half of the melt pool width is greater than 2.5.

Measurements of UNS N06625 melt pools generated via LPBF can also be examined, since its composition and performance are like that of UNS N07718. *Ghosh et al.* conducted an experiment where seven combinations of power (49 W, 122 W, 195 W) and scan speed (200 mm/s, 500 mm/s, 800 mm/s) were used to melt single laser tracks on a solid UNS N06625 build plate using a commercial LPBF machine. The width of the laser tracks was measured ex-situ by confocal laser scanning microscopy. The track depths were also measured once the tracks were cross-sectioned and polished prior to etching with aqua regia [36]. Like the work from *Scime et al.*, single melt tracks were analyzed as opposed to completely built samples; but this work did not feature any powder on the base substrate. There was also a small number of P-V combinations explored compared to what is presented in this document.

Criales et al. performed analysis of melt pools on built parts of UNS N06625. The samples analyzed investigated three factors each with three levels associated: laser power (169 W, 182 W, 195 W), speed (725 mm/s, 800 mm/s, 875 mm/s), and hatch spacing (0.09 mm, 0.10 mm, 0.11 mm). These parameters were chosen so that the energy density fell within limits that showed acceptable builds. After electro-polishing the cross-section of samples, a digital optical microscope was used to obtain images of the melt pools. Instead of measuring the melt pools in the last deposited row of material, the measurements in this work were taken at the center of the cross-section. A mathematical equation was developed for shape analysis that allowed for the measurement of the melt pool dimensions [37]. This work is comparable to what is presented in this document, but the choice of melt pools to measure as well as the criterion for parameter selection differ.

3.2.5 Quantitative Prediction of Lack-of-Fusion Porosity

Tang et al. developed the relation shown in section 3.2.2 above that provides a geometrical means of determining whether a part built using LPBF will contain lack-of-fusion porosity. To test the efficacy of the relation, relative density and porosity data was gathered from literature on different materials, such as AlSi10Mg, MS1, and Ti64. In comparing the results, the plots show that the model was able to match the experimental results with very minimal percent error [25]. Based on the success of this model, this work seeks to identify whether it can be used to also predict the lack-of-fusion porosity formation in UNS N07718 samples produced via LPBF.

3.2.6 Previous CT Measurements of Porosity in LPBF Builds

In a paper by *Cunningham et al.*, synchrotron-based x-ray microtomography was used to characterize the effect of process variables on porosity formation in LPBF of Ti64. Their work encompassed analysis of 13 samples with different combinations of laser power, scan speed, and hatch spacing. The chosen method of analysis provided the means for calculating the volume fraction of both keyholing and lack-of-fusion pores. Their work also made use of the geometrical model for lack-of-fusion prediction from *Tang et al.* and they concluded that the presence of lack-of-fusion porosity did scale with the overlap depth between melt pools [38]. This work seeks to follow the same methodology laid out by *Cunningham et al.* to analyze samples of UNS N07718 for porosity quantification.

In a more recent paper, *Georgilas et al.* conducted both area and volume fraction analyses to determine the porosity in UNS N07718 samples created using a pulsed LPBF process. A series of 16 cubes were built with varying laser power (170-200 W) and exposure time (90-120 μ s) using an AM250 machine built by Renishaw. For volume fraction analysis, micro-CT scans were performed using the Zeiss XRADIA-410. Their results showed that both increasing the laser power at a constant exposure time and increasing the exposure time at a constant laser power led to a decrease in the amount of porosity. It was also noted that the two types of porosity detected were entrapped gas and lack-of-fusion porosity [39].

3.2.7 Previous Measurements of Inclusions in LPBF Material

A paper from *Yu et al.* studied the volume fraction of alumina formation inside of UNS N07718 samples constructed via selective laser melting (SLM) and electron beam melting (EBM). Cube samples were manufactured for the SLM process in an EOS machine with 99.9999% argon in the chamber atmosphere. Once built, backscattered electron images of the sample cross-sections were analyzed using ImageJ software. For the SLM produced specimen, the area fraction of alumina inclusions was found to be 0.269%. Further analysis was done on a TEM, which revealed that SLM processing results in a large number density of small alumina precipitates with a mean size of 42.5 nm [40]. The process used in their work is similar to what is done in this work, except the inclusion analysis done for this work was on an SEM, not a TEM. In addition to the quantification of incorporated alumina formation, this work also examined alumina area fraction coverage on the top surface of manufactured parts. Although their work also investigated oxygen content, it was not done with the intent of showing a variation throughout the height of a sample.

Gaspar et al. conducted extensive analysis on spatter production during LPBF of UNS N07718. Their work discusses the mechanisms of spatter formation, which include melt-election spatter, hot entrained spatter, and vaporization of the material. They also considered the role oxygen in the chamber plays in spatter production by determining the equilibrium partial pressure of oxygen threshold below which oxidation of aluminum should not occur [41].

Zhao et al. unravel that the bulk explosion of a tongue-like protrusion forming on the front keyhole wall induces the ligamentation of the molten metal at the keyhole rims and the subsequent spattering of extremely fast droplets. Their work summarizes four common sequential events that lead to spattering: (1) transformation of the keyhole morphology from a letter "J"-like shape to a reverse-triangle-like shape, (2) formation and evolution of a tongue-like protrusion from the front keyhole wall, (3) local curvature changes around the keyhole rims, and (4) formation and evolution of melt ligaments and spatters. After the explosion of the tongue-like protrusion, melt ligaments emerge at the keyhole rims and then rise, neck, and break up into spatters. Five detectable spatters were generated and categorized into two groups: forward-flying group and backward-flying group. Different from the forward-flying spatters, which travelled almost straight at nearly constant speeds after they detached from the melt pool, the backward-flying spatters exhibited curved trajectories of the backward-flying spatters became nearly straight [42].

Chapter 4: Hypotheses

4.1 <u>Hypothesis #1: Melt Pool Geometry</u>

• When operating in the process window of P-V space, the melt pool geometry for UNS N07718 can be predicted by the Rosenthal equation to within 10% error

It was determined that prior work has been done on melt pool measurements for UNS N07718, but mainly for single melt tracks either directly on the base plate or on a single layer of powder. The work in this document seeks to analyze melt pools in multi-layered samples and add the element of prediction by calculating melt pool dimensions using Rosenthal relations.

- 4.2 <u>Hypothesis #2: Groove Depth</u>
 - Variation of the hatch spacing affects the size of grooves that form between melt pools as predicted from a geometric model

As mentioned in section 3.1.3, in looking at SEM images of the top surfaces of samples built with UNS N07718 powder, many exhibited some streaks of oxides close to and parallel with the melt pool edges. Since the location of the groove depth is at the melt pool boundaries, it may play a role, as part of the surface profile, in the reworking and residence of oxides. Therefore, it is important to characterize whether the groove depth can be controlled by variation of the extent of overlap between melt pools.

4.3 <u>Hypothesis #3: Lack-of-Fusion Porosity</u>

• The previously established model for predicting lack-of-fusion porosity is also valid for laser powder-bed fusion of UNS N07718

This hypothesis is derived from the work of *Tang et al.* in developing a geometry-based simulation model to predict the volume fraction of unmelted material in parts built by LPBF. The materials tested previously include MS1 (maraging steel), AlSi10Mg, and TiAl6V4. Comparison of the model to several sets of experimental data from literature showed its ability to correctly identify process conditions that will lead to lack-of-fusion porosity [22]. This hypothesis seeks to test whether the model can provide the same insight for UNS N07718 samples made by LPBF.

4.4 Hypothesis #4: Alumina Formation

• There is a greater concentration of aluminum oxide formation nearer the top of a build than at the bottom

As a part is being built in LPBF, melting of the newly deposited powder is also accompanied by re-melting of the material in previous layers. This fact led to the idea that as a part is being built, the alumina may float on top of the melt pool since its density is lower than that of the liquid metal. Therefore, the concentration of aluminum oxide will be higher nearer the top of a build since those layers experience less re-melting than the ones toward the bottom or middle of the part.

4.5 Hypothesis #5: Location Effect

• The location where a part is built on the build plate has an appreciable effect on the defect concentration

For this hypothesis, the thought is that as the laser is moved around the build plate by a pair of mirrors driven by galvo motors [43], the shape of the beam that interacts with the surface of the part changes depending on where the part is located. Therefore, this change in shape could have some effect on the melting behavior of the powder, which in turn could lead to an appreciable difference in concentration of defects such as oxides and pores.

4.6 Hypothesis #6: Hot Cracking

• The percentage of boron in the composition of UNS N07718 has an appreciable effect on the hot cracking susceptibility of the material as predicted by the Kou model.

It was noted that some of the samples fabricated for this work showcased hot cracking behavior. After GDMS analysis was done on a couple of samples made from different powders, the boron concentration was found to be a full magnitude greater in one sample than the other. Therefore, it is hypothesized that this change could influence the solidification range of the material and subsequently the hot cracking behavior.

Chapter 5: Experimental Methods

5.1 Experimental Design

Table 5.1 below contains all the information pertaining to samples that were created for this work, including process parameters, and predicted quantities.

Table 5.1: All Sample Information ("GD" = groove depth)

Camala ID	DII1	DUO	DUD	DII4	CD5	CDC	CD7
Sample ID	BHI	BH2	BH3	BH4	GD5	GD6	GD/
Power (P) [W]	285	285	285	285	285	285	285
Speed (V) [mm/s]	480	720	960	1200	960	960	960
Hatch Spacing (H) [mm]	0.11	0.11	0.11	0.11	0.05	0.09	0.10
Layer Thickness (L) [mm]	0.04	0.04	0.04	0.04	0.04	0.04	0.04
Layer Rotation [°]	90	90	90	90	90	90	90
Sample Height [mm]	25	25	25	25	10	10	10
Predicted Width [µm]	207	169	146	131	146	146	146
Predicted Depth [µm]	103.5	84.5	73	65.5	73	73	73
Predicted GD [µm]	-	-	-	-	2.4	6.3	8.1
Predicted Index [-]	0.43	0.65	0.86	1.08	0.42	0.68	0.77
Sample ID	GD8	GD9	GD10	GD11	KEY7	KEY8	LOC
Power (P) [W]	285	285	285	285	285	285	285
Speed (V) [mm/s]	960	960	960	960	480	480	960
Hatch Spacing (H) [mm]	0.11	0.12	0.13	0.14	0.13	0.15	0.11
Layer Thickness (L) [mm]	0.04	0.04	0.04	0.04	0.04	0.04	0.04
Layer Rotation [°]	90	90	90	90	67	67	67
Sample Height [mm]	10	10	10	10	10	10	10
Predicted Width [µm]	146	146	146	146	207	207	146
Predicted Depth [µm]	73	73	73	73	104	104	73
Predicted GD [µm]	11.2	15.7	21	30.5	-	-	-
Predicted Index [-]	0.86	0.97	1.09	1.21	0.54	0.67	0.86
Sample ID	OS	PAC	PRAX	TA1	TA2	TA3	TA4
Power (P) [W]	285	200	200	150	200	300	300
Speed (V) [mm/s]	960	900	900	400	530	800	1200
Hatch Spacing (H) [mm]	0.11	0.12	0.12	0.11	0.11	0.11	0.11
Layer Thickness (L) [mm]	0.04	0.03	0.03	0.04	0.04	0.04	0.04
Layer Rotation [°]	90	0*	0*	90	90	90	90
Sample Height [mm]	10	10	10	10	10	10	10
Predicted Width [um]	146	127	127	165	165	165	134
Predicted Depth [um]	73	63	63	82.5	82.5	82.5	67
Predicted GD [um]	_	-	-	_	_	-	_
Predicted Index [-]	0.86	1.12	1.12	0.68	0.68	0.68	1.29
Sample ID	S1/S7	S2/S8	S3/S9	S4	S5	S 6	

Power (P) [W]	285	285	285	285	285	285
Speed (V) [mm/s]	960	960	960	960	960	960
Hatch Spacing (H) [mm]	0.11	0.14	0.17	0.11	0.14	0.17
Layer Thickness (L) [mm]	0.08	0.08	0.08	0.12	0.12	0.12
Layer Rotation [°]	90	90	90	90	90	90
Sample Height [mm]	10/25	10/25	10/25	10	10	10
Predicted Width [µm]	150	150	150	150	150	150
Predicted Depth [µm]	75	75	75	75	75	75
Predicted GD [µm]	23.5	49.2	_	36.5	73.8	-
Predicted Index [-]	1.68	2.01	2.42	3.1	3.43	3.84

There are a few things to note about the information in the table above: the standard process parameters recommended by EOS for building LPBF UNS N07718 parts are P = 285 W, V = 960 mm/s, H = 0.11 mm, and L = 0.04 mm; the PAC and PRAX samples were built with different powders and the laser starting at a 45° angle, but the rotation between subsequent layers is 0°; the predicted melt pool widths and depths were found using the Rosenthal relations defined in section 3.2.1; the predicted groove depths were obtained using the equation shown in section 3.2.3, where W is the predicted melt pool width; and the predicted index is tabulated using the relation derived in section 3.2.2, where W is again the predicted melt pool width and D is the predicted melt pool depth. All these predictions were made with an absorptivity value of 0.35 being used.

For melt pool measurements, the idea was to create a series of samples that spanned the process window of P-V space for UNS N07718. Figure 5.1 shows the variety of P-V combinations used to construct samples in this work. Samples with an index value less than one and a 90° rotation between layers were good conditions to test, because these samples were predicted to undergo full melting and have the melt pools aligned for easy measurement. The melt pools measured in this work are not single beads, and instead are measured using the top row of samples built to specific heights.



Figure 5.1: Map of P-V Space Showing Combinations Used for Samples, Standard Conditions Indicated by Red Dot

The thought behind probing groove depth is that as two overlapping melt pools are pushed closer together, the local minimum residing between the melt pools should shrink. To test this notion, parts with increasing hatch spacing were created, which allowed for the potential extent of change in the depth to be quantified and compared to mathematical predictions.

To investigate the ability to predict lack-of-fusion porosity with the index relation, samples were built with predicted index values of both less than one and greater than one. The conditions chosen correlate with the index equation and will therefore determine whether this geometrical criterion applies for predicting lack-of-fusion porosity in LPBF of UNS N07718 parts.

Since alumina is less dense than the surrounding molten UNS N07718 matrix, the belief is that these oxides will continue to float upward as a specimen is being built. To explore this idea, analysis of the incorporated and surface oxide concentration was performed on a multitude of samples. Also, a few samples were built to a height of 25 mm to allow for analysis of oxygen content throughout the height. These samples were constructed with different P-V combinations to ascertain whether region of P-V space influences the results.

Finally, several samples were built with the recommended standard conditions and placed at various locations across the build plate as shown in Figure 6.24. The thought is that building parts at the center of the build plate with the standard parameters minimizes the defect concentration

within the finished part. Therefore, creating parts using the same conditions in other locations will allow for investigation of variance in oxide and porosity concentration, which could lead to a meaningful result.

5.2 <u>Sample Preparation</u>

Once the samples were finished being built, they were cut from the build plate using a Wire Electrical Discharge Machine. This same machine was then used to section each sample parallel to the build direction, and perpendicular to the travel direction of the laser beam if a 90° rotation between layers was specified. The sectioned samples were then hot mounted in Konductomet with the cut side visible. Before placing the samples in a scanning electron microscope (SEM) for analysis, each puck was polished using the following sequence with diamond polish suspension on a Struers auto-polisher: 9 μ m (MD Largo pad) \rightarrow 3 μ m (MD Dac pad) \rightarrow 1 μ m (MD Nap pad) \rightarrow 0.25 μ m (MD Nap pad). Finally, to measure the melt pools, samples were etched either via swabbing with glyceregia (15 mL HCl, 10 mL glycerol, 5 mL HNO₃) for ~20 seconds or in a 10% solution of oxalic acid using a potential of 4 volts for 5 seconds.

5.3 Measurement Techniques

5.3.1 Optical Microscopy

An optical microscope is a commonly used system of lenses and visible light that generates magnified images of small objects. For the microscope used in this work, the etched samples were placed upside-down on the stage for viewing through an eyepiece camera. Images of the top row of melt pools deposited were taken at 10X magnification, which allowed for the measurement of the melt pool dimensions via an image analysis tool (ImageJ). Ultimately, the determined melt pool dimensions from this analysis examine whether those predicted by the Rosenthal relations are accurate to within 10% error.

5.3.2 3D Optical Metrology via Alicona

To investigate surface profilometry, the samples were placed under the Alicona, which is an instrument that utilizes 3D optical metrology. Figure 5.2 below shows a schematic of how the machine works.



Figure 5.2: Schematic Detailing 3D Optical Metrology

In the Alicona, a beam of light is projected onto a small area of the surface and that light is then reflected from the surface to the optical system of the microscope. The light is directed by a beam splitter onto the active area of a photoelectric detector, which then forms an image containing photometric and geometric information. The Alicona then makes use of focus-variation microscopy, which works by vertically scanning a part surface within a z-range in which the surface lies. The part surface is detected by calculating the maximum focus variation value within the scan range. The equation used to do this is shown below in Figure 5.3.

$$F_{z}(x, y) = FM(reg_{w}(I_{z}(x, y)))$$

$$FM = \frac{1}{n^2} \sum_{reg_w(I_z, x, y)} (GV_i - \overline{GV})^2$$



Figure 5.3: Formulas Utilized by Alicona

Next, an algorithm adjusts the focus for each image and stiches them together to form a 3D representation. Figure 5.4 shows an example of the final rendering.



Figure 5.4: Surface Rendering of Sample Surface

Use of the Alicona allowed for investigation into the hypothesis that variation of the hatch spacing can affect the depth of the groove between melt pools. Advantages of using this machine include being able to gather information without destroying the samples as well as being able to gather a plethora of measurements per image taken. To measure the groove depths, multiple horizontal lines were drawn across the 3D renderings to generate 2D profiles. The x- and z-coordinates of these profiles were then exported into Excel. Once in Excel, a MatLab script was used to locate all the local minima and maxima within the 2D profile. Their coordinates were then located along the profile to find the correct minima and maxima to use for measurement of the groove depth. To verify that the location of these points along the profile were good data points to use, the difference between the x-coordinates was also calculated and compared to the hatch spacing for each sample. Finally, the average of the left-hand and right-hand subtraction of the minima from the maxima was done to find the average groove depth for each sample.

5.3.3 Micro-CT Scanning

Micro-computed tomography (micro-CT) scanning is a 3D imaging technique that utilizes X-rays to see inside an object slice by slice. A series of 2D X-ray images is captured and reconstructed into 2D cross-sectional slices. The process is like that of a CT (or CAT) scan that one gets at a hospital, but on a much smaller scale. The other difference of note is that with CAT scans, the X-ray machine revolves around the patient; however, with micro-CT scanning, the sample rotates while the X-ray beam remains stationary.

For this work, a pink x-ray beam was aimed at a rotating sample with a 1 mm x 1 mm crosssectional area. The pink beam, which allows more wavelengths to pass than a monochromatic beam, hits the sample and projects a 2D image onto an X-ray detector. The sample is then rotated through a total of 180° , which allows for ~1500 radiographs to be taken (i.e., 1 radiograph per 0.12°). Variation in density and atomic number of the elements in the material affect the intensity of the radiograph that is projected onto the detector. These radiographs are then combined into a sinogram that is then backprojected to form a 3D visual of the sample. An example of the experimental setup is shown below in Figure 5.5.



Figure 5.5: Schematic of Micro-CT Scanning Setup

The main advantage of micro-CT scanning is that it is a non-destructive process. A non-destructive process enables analysis of a sample without altering or destroying the sample during testing. It also provides the ability to gain insight into internal features of the sample, such as porosity, by way of a 3D rendering. Earlier attempts at measuring porosity were made using an SEM, but the sample preservation and accuracy of micro-CT scanning allow for a much more in-depth analysis. The use of this technique will provide the means for determining whether the index relation can be used to predict the extent of lack-of-fusion porosity in LPBF UNS N07718 samples; and it also allows for characterization of keyholing porosity.

5.3.4 Inclusion Analysis on ASPEX Explorer and MIRA3 SEM

Before elaborating on the specifics of the inclusion analysis, it is important to discuss the two main operating modes of a scanning electron microscope: backscattered electron imaging (BSEI) and secondary electron imaging (SEI).

BSEI involves high energy electrons originating from an electron beam and undergoing elastic scattering interactions with atoms in an interaction volume. These electrons are backscattered out of the sample and into a solid-state detector that is usually located above the specimen, concentric with the electron beam. The detectors, which typically consist of symmetrically divided parts, are positioned this way to maximize the collection of backscattered electrons. When all parts of the detector are enabled, the resulting contrast in the image corresponds to the atomic number of the elements in the sample. Heavy elements (higher atomic number) have larger electron clouds than lighter (lower atomic number) elements, meaning they produce a higher signal and appear brighter in the obtained image. Alternatively, one could enable only particular quadrants of the detector to gather topographical information.

SEI encompasses the collection of low energy secondary electrons that are ejected from valence bands of atoms by inelastic scattering. In contrast to backscattered electrons, secondary electrons originate from a smaller interaction volume nearer the surface of the sample. These secondary electrons are commonly detected by an Everhart-Thornley detector (ETD). This device is comprised of a scintillator within a positively biased Faraday cage that attracts the electrons. The ETD is positioned on the side of an SEM chamber to boost the efficiency of detecting secondary electrons. The scintillator is more positively biased than the Faraday cage by greater than 10 kV, which allows for the electrons to be accelerated towards the scintillator [44]. This acceleration gives the electrons sufficient energy to cause the scintillator to emit flashes of light that result in an image. The brightness of the image is controlled by the number of secondary electrons reaching the detector, so one can either increase the beam diameter or beam voltage to generate more signal and brighten the image. Also, if the beam enters the sample at an increasing angle of incidence, then the interaction volume increases while the "escape" distance of one side of the beam decreases. The net result of this is an increase in the number of secondary electrons emitted from the sample and explains why steep surfaces and edges tend to appear brighter than flat surfaces in the image. Overall, SEI is very good for gathering information about the topography of the sample surface, but BSEI is better for learning more about sample composition.

In this work, an ASPEX Explorer SEM was first utilized to conduct an inclusion analysis on prepared samples. BSEI was used to set a threshold to distinguish between the UNS N07718 matrix and other defects, such as inclusions, pores, or silicon carbide scratches. Since the inclusions being detected were aluminum oxides, a piece of aluminum tape was placed on the edge of each mounted sample to help with setting the contrast threshold. Figure 5.6 below shows an example of the difference in contrast between the aluminum tape and UNS N07718 matrix containing elements with higher atomic number.



Figure 5.6: BSEI Showing Contrast Between Al Tape and UNS N07718 Matrix

Once an appropriate threshold was set, the system was programmed to analyze a 4-6 mm² region located near the center of the cross-sectional area of each specimen. Using a line scan, as shown in Figure 5.7 below, the Explorer was able to image anything that fell within the specified threshold and conduct an EDS on what was detected. Subsequent samples were analyzed on the MIRA3 using INCA Feature software with the same accelerating voltage of 20 kV that was used on the Explorer, but the area examined was 9 mm².


Figure 5.7: Backscattered Electron Line Scan Over a Pore

Energy-dispersive X-ray spectroscopy (EDS or EDX) is an analytical technique used for elemental analysis of a sample. An atom has electrons surrounding its nucleus at discrete energy levels or shells. As an electron beam interacts with the atoms in a sample, an inner electron may be excited and ejected from its shell, leaving an electron hole. As a result, an electron from a higher energy shell will fill in the vacancy and the difference in energy between the higher-energy shell and lower-energy shell is converted into an X-ray. The number and energy of the X-rays emitted from a specimen are measured by an energy-dispersive spectrometer. Since the energies of the X-rays are equal to the difference in energy of the two shells of the emitting element, EDS allows for the composition of a sample to be measured. In reference to this work, the EDS allowed for the distinction between alumina inclusions, pores, and silicon carbide scratches remaining from sample preparation. Overall, this technique helped in testing the hypothesis of location where a sample is built influencing the defect concentration present in the part.

5.3.5 SEM Image Analysis using ImageJ

For this analysis, BSEI was done of samples using a Quanta600 SEM. The reason for using BSEI is because it provided the contrast needed to distinguish between the alumina forming on the top surface of samples and the rest of the matrix. Figure 5.8 below shows an example image taken for one of the samples.



Figure 5.8: BSEI of Sample BH1 from Quanta600

ImageJ (aka Fiji) is a very powerful image analysis tool. The goal of this analysis was to provide insight into and measurement of the percentage of area that the aluminum oxides were covering the top surface of samples built using various processing parameters. An image like the one above was uploaded into the software and converted into an 8-bit image. This conversion then allowed for easy thresholding of the image into black and white, so the area of coverage could be calculated. Manual adjustments of the threshold set were done if needed and then an analysis of particles followed. The *Analyze Particles* function in ImageJ not only counts the total number of particles within the threshold, but also calculates the area of each particle. Figure 5.9 below shows the 8-bit and particle images for sample BH1 shown above.



Figure 5.9: 8-bit Image of Sample BH1 (left); Particle Analysis Image of Sample BH1 (right)

Ultimately, this analysis provides some insight into the hypothesis of alumina concentration being greater nearer the top surface of UNS N07718 LPBF parts; it also helps with investigating the effect of location and whether the alumina coverage shows any variation.

5.3.6 Glow Discharge Mass Spectrometry (GDMS)

This type of spectrometry involves a sample being ionized in a plasma (a partially ionized gas) so that its elemental composition can be obtained to a detection limit on the order of parts per billion (ppb). In this process, which typically occurs at a reduced pressure of 0.1-1 torr with Argon as the surrounding gas, an electrical discharge of ~1 kV is applied between two electrodes, where the sample being analyzed is the cathode. The voltage applied creates a gas breakdown into positive ions and electrons. The positive ions are then accelerated toward the cathode and bombard it, resulting in emission of electrons and release of atoms from the sample. Both the electrons and atoms arrive in the plasma, where the sample material is ionized and the electrons re-pair with Argon gas ions (resulting in a glow via excitation collisions). The ionized material is then extracted and characterized by its mass-to-charge ratio by a mass spectrometer. Figure 5.10 is a diagram depicting the GDMS process.



Figure 5.10: Diagram of the Main Processes Occurring in GDMS

A few of the fabricated samples displayed interesting hot cracking behavior and the thought was that the composition of the powder used may be the reason for this phenomenon. Hence, the decision to utilize GMDS as a means of quantifying the concentration of boron (B) and sulfur (S), which could affect the hot cracking behavior.

5.3.7 Thermal Extraction (TE)

TE measures the presence of gaseous elements (C, H, O, N, and S) in solid materials from ppm to percentage levels. A furnace at high temperature is required to rapidly heat the specimen and convert certain elements into volatile forms so that separation, detection, and measurement can occur.

For carbon (C) and sulfur (S), measurement is based on combustion and infrared detection during TE. The solid material is completely oxidized by combustion in an oxygen plasma typically above 2000 °C. The sample is placed in a high frequency induction furnace inside of a ceramic crucible. Then various gases are released during combustion and their quantity is measured by infrared detectors. The amount of carbon monoxide (CO) and carbon dioxide (CO₂) generated determines the carbon content, while the amount of sulfur dioxide (SO₂) determines the sulfur content.

Nitrogen (N), oxygen (O), and hydrogen (H) are measured using inert gas fusion. In this process, the specimen is placed in a graphite crucible inside of a furnace, where it is placed between

electrodes. The furnace is then purged with an inert gas, such as argon, and a high current is passed through the crucible causing a large temperature increase. Upon heating, CO, CO₂, N₂, and H₂ are released and directed toward the appropriate detectors.

This method of analysis seeks to provide insight into whether oxide concentration is greater nearer the top of LPBF UNS N07718 parts. The analysis was conducted with the top and bottom halves of the 25 mm tall samples that were generated with different process parameters. This designed variance in process parameters will allow for comprehension of whether the region of P-V space in which a sample is created influences the oxide concentration as well.

Chapter 6: Results and Discussion

6.1 <u>Melt Pool Geometry</u>

Figure 6.1 below shows an optical image taken of a sample built with the standard conditions specified by EOS for fabrication of UNS N07718 parts via LPBF (P = 285 W, V = 960 mm/s).



Figure 6.1: Melt Pools Revealed After Etching (Standard UNS N07718 Parameters)

In the figure, the black outline surrounding the boundary of the melt pools is very different from the blue ellipse, which is the predicted melt pool size from the Rosenthal relations. The shape of the melt pools indicates that they are keyhole-shaped, which explains why the measured widths and depths were consistently larger than what was predicted using the Rosenthal relations. Figure 6.2 and Figure 6.3 below show these melt pool measurements and their percent error relative to the Rosenthal predictions for samples fabricated with a 40-micron layer thickness.



Figure 6.2: (a) Average Melt Pool Width and Percent Error Relative to Rosenthal vs Speed; (b) Average Melt Pool Width and Percent Error Relative to Rosenthal vs Hatch Spacing



Figure 6.3: (a) Average Melt Pool Depth and Percent Error Relative to Rosenthal vs Speed; (b) Average Melt Pool Depth and Percent Error Relative to Rosenthal vs Hatch Spacing

From the plots above, one can see that as the scanning speed used to fabricate a sample is increased, both the melt pool width and depth decrease. This makes sense as the amount of energy input into a spot along the melt track should decrease, since the time spent dwelling in that location decreases with increasing scanning speed. It must also be noted that the percent errors relative to the Rosenthal equation for the plots involving width are all below 20%; however, the percent errors for the plots involving depth are consistently over 100%. This large increase in depth compared to the predicted values is indicative that keyhole-shaped melt pools, like those in Figure 6.1, were being generated.

Another interesting observation concerns the red bar marked 960 and the green bar marked 110 in the above figures. The samples measured for these data points were built with the standard condition processing parameters. However, the only difference between them was their height; sample BH3 (red bar marked 960) was built to be 25 mm tall, whereas sample GD8 (green bar marked 110) was built to be 10 mm tall. In looking at the plots, the melt pool width and depth measurements for BH3 (W = 174 μ m, D = 173 μ m) are larger than those for GD8 (W = 164 μ m, D = 158 μ m). The explanation for this is most likely heat being built up vertically as the sample is fabricated. Denudation may also play a role in that its clearing of powder from subsequent melt tracks would allow for mostly remelting of the substrate to occur in each deposited layer. The concept of denudation will be discussed further later in this section.

After measuring the melt pool dimensions, the melt pool cross-sectional areas were also measured and used as a means of estimating the effective absorptivity. To do this, the percent error equation shown below was utilized.

$$Percent \ Error_{Ros} = 100 * \left[\frac{\left((EA/0.35) * A_{Ros}\right) - A_{Measured}}{A_{Measured}}\right]$$
(6)

In the equation, EA is the effective absorptivity, A_{Ros} is the area predicted using the Rosenthal dimensions, and $A_{Measured}$ is the actual measured area of the given melt pool. Once all the melt pool areas for a given sample were measured, the equation above was iterated to find the effective absorptivity value that minimized the error to as close to zero as possible. Figure 6.4 below shows the results plotted as a function of scanning speed.



Figure 6.4: Effective Absorptivity as a Function of Scanning Speed

In the figure above, the samples plotted had the same process parameters other than scanning speed (P = 285 W, H = 0.11 mm, L = 0.04 mm). It can immediately be noted that the increase in scanning speed leads to a decrease in effective absorptivity, which then leads to the formation of smaller melt pool areas. There are a couple of samples that have effective absorptivities just exceeding a value of one, which should not be possible. However, the overall premise is still clear, which is that slower scanning speeds correlate with larger effective absorptivities.

Now since the Rosenthal relation predicts a correlation between the melt pool dimensions and $(P/V)^{1/2}$, the measured widths were plotted against this variable to compare the results. Figure 6.5 shows those results along with results obtained from papers by *Sadowski et al.* and *Scime et al.*; and Figure 6.6 shows a similar plot done for melt pool depths.



Figure 6.5: Comparison of Measured Melt Pool Widths to Rosenthal Predictions



Figure 6.6: Comparison of Measured Melt Pool Depths to Rosenthal Predictions

The results from *Sadowski et al.* and *Scime et al.* better fit the Rosenthal prediction. However, because both data sets were measurements of single tracks, as opposed to melt pools from the top layer of a fabricated sample, the effect of continuous denudation is mitigated. The effect of denudation on the deposition pattern produced by multiple, overlapping tracks has been studied in

the past [45], [46] and is relevant when considering a complete additive manufacturing process. The dominant driving force for denuding powder near a melt track is the entrainment of particles by surrounding gas flow. The flow is induced by the intensive evaporation that occurs within the laser spot and pressure drop inside the associated vapor jet due to the Bernoulli effect. The vapordriven entrainment causes particles to not only be incorporated into the melt track, an effect intimately related to final bead size of the track, but also to eject vertically and rearward relative to laser scan direction and redistribute elsewhere on the powder bed [19]. Yadroitsev et al. [47] showed that denudation leads to a decrease in track height with successive scans which is strongly affected by hatch spacing. Thus, when interpreting the single-track data presented here, it is important to consider how denudation plays a role in determining optimal process parameters and scan strategies.

From Figure 6.5, one can also see that despite being built with the same power and scanning velocity, there is still a range of melt pool widths that is obtained at a $(P/V)^{1/2} = 0.545 (W/mm)^{1/2}$. This can potentially be explained by variation in hatch spacing. As shown in Figure 6.2b, the melt pool width does decrease as the hatch spacing increases, which is likely attributed to smaller hatch spacings allowing for more heat build up to occur since the extent of overlap between melt pools is greater. The other variable to consider is layer thickness. Figure 6.7 below is a plot of the average melt pool width as a function of layer thickness for two different hatch spacings.



Figure 6.7: Average Melt Pool Width vs Layer Thickness

From the figure, there is generally no difference made to the average melt pool width by variation of layer thickness; however, there is an interesting spike that arises for the 80-micron layer thickness at a 110-micron hatch spacing. Further investigation into this data point must be done to see whether this spike is actually a common occurrence or an anomaly.

Since the melt pool dimensions were larger than predicted, then the index values calculated for all the samples analyzed in this work back in Table 5.1 were redone with the correct values. A few of those samples were predicted to contain some lack-of-fusion porosity, but those predictions were made with the Rosenthal widths and depths, not the measured dimensions. Recalculation of these values for the samples previously predicted to experience lack-of-fusion was done and the results are shown in Table 6.1.

Sample ID	BH4	GD10	GD11	TA4
Index with Rosenthal Dimensions	1.08	1.09	1.21	1.29
Index with Measured Dimensions	0.81	0.78	0.93	0.92

Table 6.1: Recalculated Index Values

Since the index is now less than one for these samples, that means none of the parts in this work were in the lack-of-fusion condition. Therefore, this means that the build rates for fabrication can be increased beyond the EOS recommendation for the material without introducing lack-of-fusion porosity. Below is a figure showing the build rates for samples in this work versus their index value. Amongst the population of blue dots are some red dots that identify the standard condition build rate.



Figure 6.8: Build Rate vs Index

The plot above shows that it is possible to increase the build rate, while keeping the index relation below one. One can manipulate either the hatch spacing (H), the layer thickness (L), or the velocity (V); but it should be noted that while increasing the layer thickness does increase the build rate, it also can lead to lack-of-fusion defects when combined with high enough hatch spacing. This can be seen in Figure 6.9 below, which contains optical micrographs showing lack-of-fusion defects. These images are of samples S3, S5, and S6, whose process parameter data can be found in Table 5.1 above. It should also be noted that the index relation predicts these samples to contain lack-of-fusion porosity even when using the measured melt pool widths and depths.



Figure 6.9: LOF Porosity in Samples S3 (top left), S5 (top right), and S6 (bottom)

Increasing the layer thickness also leads to much rougher surfaces as seen in the following profiles.



Figure 6.10: Comparison of Measured Profile to Calculated Profile

The blue line in the figure above is the measured profile for a sample with the following parameters: P = 285 W, V = 960 mm/s, H = 0.17 mm, and L = 0.08 mm. The overlayed red lines show the calculated profile for the same sample. One can see that the measured profile shows much more fluctuation from melt pool to melt pool as well as overall variation in height across the sample. This can be better seen in the Figure 6.11 below, which contains the same information in a 3D rendering of the surface.



Figure 6.11: 3D Rendering of Surface Made with 80-micron Layer Thickness

Simulations of the melt pool overlap for some of the samples were done using the measured dimensions to estimate the expected amount of lack-of-fusion porosity. The melt-pool sizes were assumed to be normally distributed, according to the measured average depth and width and their standard deviations [48]. The results, shown in Table 6.2, agree with the new index values by predicting the percentage of lack-of-fusion porosity to be zero. The table also gives the average number of times each volume is melted, with LOC13 having the standard melting conditions. Table 6.2: Melt Pool Simulation Results

Sample /	Simulated meltpools	Sample / Count /	Simulated meltpools
Count / %LOF	(1 mm side length)	%LOF	(1 mm side length)
TA1 7.4 0		KEY7 20.9 0	

TA2 8.3 0	KEY8 11.8 0		
TA3 7.5 0	LOC13 5.0 0		
TA4 1.9 0	Notes: "Count" is the average number of times that the material is melted during building. "%LOF" is the calculated volume percentage unmelted material (zero in all cases). For the "TA" samples (at left) the hatch rotation was 90°; the simulated plane is at 45° to both scan directions. The hatch rotation was 67° for the other builds (in right-hand column).		

Further corroboration of this was achieved by looking at the μ CT results of sample TA4, shown in Figure 6.12, which does not exhibit any of the periodic spacing of irregularly shaped porosity that is characteristic of lack-of-fusion pores. Ultimately, this result indicates that repeated remelting is happening under the standard conditions, which means that faster builds would be possible with full melting. This has been shown for LPBF of other materials, such as AlSi10Mg and Ti64, as well [38,48].



Figure 6.12: Micro-CT Results for Sample TA4

Although the melt pool dimensions are larger than what was predicted and exhibit keyhole shapes, this does not necessarily mean that keyholing porosity will be deposited in the samples. Figure 6.13 below contains an image showing the melt pools for samples TA1, TA2, and TA3. These melt pools are clearly very similar and keyhole-shaped; yet the amount of porosity in samples TA2 and TA3 was found to be ~0.5 - 1.25%, whereas sample TA1 has almost 4% porosity.



Figure 6.13: Melt Pools for Samples TA1 (top left), TA2 (top right), and TA3 (bottom)



A plot of the index versus the porosity measured via μ CT scanning shows that it is possible to have keyhole-shaped melt pools without increasing the porosity drastically.

Figure 6.14: Plot of Index Versus Measured Porosity via µCT Scanning

In addition to the melt pool measurements, groove depth measurements were also gathered and compared to predictions made using the relation derived in section 3.2.3. Since the relation is a function of the ratio of hatch spacing to melt pool width, the measured and calculated values were plotted against this variable as shown in Figure 6.15. In the figure, the blue data points correlate with the blue line, which is the calculated groove depth for samples fabricated with a 40-micron layer thickness; the orange data points correlate with the orange line, which is the calculated groove depth for samples fabricated with a point correlates with the yellow line, which is the calculated groove depth for a sample fabricated with a 120-micron layer thickness.



Figure 6.15: Groove Depth Plotted Versus H/W

The measured groove depths compare well with those predicted using the derived relation, with some quantitative differences. For the samples fabricated with a 40-micron layer thickness, the groove depth was generally lower than what was predicted from the calculation. Wetting of the previously deposited solid bead by the liquid metal in the melt pool is likely to have changed the shape of the groove. Liquid metal strongly wets solid metal of the same composition; this would have caused the contact point between the melt pool and the solid bead to move up the solid bead, rounding the bottom of the groove, and decreasing the groove depth. Some evidence of rounding of the bottom of the groove is evident in the measured profiles. However, perturbations in the surface profile (which cannot be explained by surface tension alone) are much more evident. An example of the perturbations in such a profile is shown in the figure below for sample GD11 and a three-dimensional rendering of the surface can be found in Figure 6.17. The blue line in the figure below is the measured profile generated with the following parameters: P = 285 W, V = 960 mm/s, H = 0.14 mm, and L = 0.04 mm. The overlayed red lines show the calculated profile for the same sample.



Figure 6.16: Example Surface Profile for GD11



Figure 6.17: 3D Rendering of Surface of Sample GD11

There is also another interesting result shown for the samples made with an 80-micron layer thickness. Two sets of samples were created using two different process parameter combinations. However, within each set the only difference between the samples was their overall height. Both sets exhibited the same phenomena of the shorter sample having a larger groove depth than the

taller sample. Interestingly, both taller samples showcased smaller and more consistent melt pool widths as evidenced by the rightward shift in H/W and smaller 90% confidence intervals.

6.2 Oxide Concentration

Aluminum oxides were found to reside inside of samples built and on the top surface. The composition of these inclusions is discussed below.



Figure 6.18: EDS Spectrum of Alumina Inclusion

Figure 6.18 above shows an example of an EDS spectrum of an alumina inclusion taken at a beam energy of 15 kV. The peaks for aluminum, oxygen, and titanium have been marked since these are the main elements found in the detected inclusions. For these oxides, the average ratio of titanium to aluminum is ~0.013 after correcting for aluminum and titanium in the matrix; and the general distribution of this ratio can be seen in the Figure 6.19c below. The following equation was used to correct the aluminum and titanium percentages. Although written in terms of %Al, simply replacing these terms with %Ti allows for the calculation of the %Ti_{Corrected}:

$\% Al_{corrected} = \% Al_{Analyzed} - (\% Ni_{Analyzed} / \% Ni_{Matrix}) * \% Al_{Matrix}$

The values for Al_{Matrix} and Ti_{Matrix} were obtained by plotting the analyzed aluminum and titanium percentages against the analyzed percentage of nickel; and the values associated with the highest percentage of nickel were chosen. The value used for the Ni_{Matrix} was 50.5. Most of the inclusions maintain a Ti/Al ratio between 0.003 and 0.023; however, this histogram is essentially showcasing that the Ti/Al ratio is zero. Therefore, we can expect to see only alumina formation in these samples.





Figure 6.19: (a) %Al (analyzed) vs %Ni (analyzed); (b) %Ti (analyzed) vs %Ni (analyzed); (c) Histogram of Ti/Al Ratios of Inclusions

The concentration and morphology of the oxides varied between the inside of the fabricated parts and the top surface. Those incorporated within the samples were small and resulted in concentrations on the level of ppm; whereas those on the surface were larger and covered a substantial fraction of the surface. It was also noted that the top surface oxides formed streaks along the boundaries between melt tracks as shown in Figure 5.8. The extent of this is quantified in the figure below.



Figure 6.20: Extent of Oxides in Streaks on Top Surface

Figure 6.20 above shows that most of the oxides residing on the top surface are part of the streaks. For most of the samples, both the oxide streak area fraction and total oxide area fraction are nearly in a 1:1 ratio. For the samples with larger area fractions, the ratio does drop a little bit. It may be that the severe keyholing of these samples both increased the amount of oxide on the surface and its distribution across the surface, especially when considering the volatility of the melt pools. Overall, the oxide streak area accounts for at least 70% of the total oxide area on the top surface. In examining the amount of surface that the oxides covered, it was noticed that the area fraction increased under keyholing conditions. Figure 6.21 below is a plot of the area fraction of coverage on the surface against the ratio of hatch spacing to melt pool width; and the numbers next to the dots on the plot are the ratio of the melt pool depth to half of the melt pool width. The H/W ratio was chosen because it appears likely that the oxide deposited by spatter can be reworked by melting; so, as H/W decreases, the volume of material being remelted increases, thus allowing for more hot, oxidized spatter to form. The one anomaly in the results is sample GD5, but it must be mentioned that measurements of the melt pool widths for this sample were rather difficult considering the extent of overlap that occurs with a hatch spacing of only 50 μ m.



Figure 6.21: Area Fraction of Surface Oxides

The BH samples show an interesting trend that as one moves away from the smaller H/W ratios that are typically associated with keyholing, the area fraction of oxide coverage decreases. This makes sense as keyholing results in more vaporization of the material, which allows for more spatter to be produced and oxidized in the chamber atmosphere before falling back onto the sample surface. Another way to analyze at this is by looking at the numbers next to the dots, which are the ratio of the melt pool depth to half the melt pool width. We can see that as the ratio goes beyond 2.5, the severe keyholing that occurs leads to a noticeable increase in the surface oxide concentration.

Incorporated oxide concentration may correlate with the number of melting events that occurs for a given layer of deposited material. The number of times a layer is remelted is calculated by multiplying $\pi/4$ by the ratios of melt pool width to hatch spacing and melt pool depth to layer thickness. Figure 6.22 shows the area fraction of incorporated oxide formation plotted against this variable.



Figure 6.22: Area Fraction of Incorporated Oxides Versus Number of Times Remelted

These results show that there is no difference made to the incorporated area fraction of oxides by increasing the number of melting events. This ultimately means that other factors besides the number of melting events contribute to the development of incorporated oxides.

This leads into the idea that the oxide concentration is greater nearer the top of a build, which was tested by analyzing the oxygen and oxide content of the bottom and top halves of 25 mm tall parts as outlined in the figure below. Each of the halves analyzed were 5 mm x 10 mm x 12.5 mm.



Figure 6.23: Bottom (blue dashed line) and Top (red dashed line) Halves Analyzed by LECO and Feature Analysis

Table 6.3 below shows the results of a LECO analysis performed on the BH samples and a feature analysis done on the MIRA3. The 9 mm² area analyzed by the feature analysis is outlined by black dashed lines as seen in the cross-sectional image on the right side of Figure 6.23.

Table 6.3: Oxygen and Oxide Content in Top and Botto	om of Samples
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Sample ID	BH1 (Top)	BH2 (Top)	BH3 (Top)	BH4 (Top)
Oxygen [ppm]	200	170	160	170
Oxide [ppm]	-	-	75.95	173.75
Sample ID	BH1 (Bottom)	BH2 (Bottom)	BH3 (Bottom)	BH4 (Bottom)
Oxygen [ppm]	150	150	160	180
Oxide [ppm]	-	-	68.83	202.27

The results do not show any appreciable difference in the oxide concentration between the top and bottom halves of samples. This means that the increase in area fraction of oxides found on the surface of the parts must be a layer-by-layer development, as opposed to being a general increase in the concentration as the sample is built.

In fact, one can calculate the expected oxygen content that comes from the surface oxides. *Ohtsuki et al.* estimated the average thickness of the surface oxides to be 0.5 μ m [23]; so, one can use this information along with the surface coverage percentage, layer thickness (40 μ m), density of alumina (3.99 g/cm³), density of UNS N07718 (8.10 g/cm³), and mass fraction of oxygen in alumina (48/102 = 0.47) to estimate the oxygen content from the surface. The equation below shows the calculation of the oxygen content from the surface for the average surface coverage of 10%.

$$M_{O_2} surface = (0.1) * \frac{0.5 \,\mu m}{40 \,\mu m} * \frac{3.99 \,g/cm^3}{8.10 \,g/cm^3} * (0.47) = 290 \,ppm \tag{7}$$

This calculation was done again using the maximum surface coverage, which reached ~31.5%. Therefore, the range of the oxygen content from the surface is between 290 - 910 ppm. Combining this with the 100 ppm of oxygen content coming from the powder [23], leads us to conclude that the amount of oxygen content should be ~400 ppm on average. However, the results of the LECO analysis show an oxygen content just below 200 ppm. So, it may be possible to remove some of the oxide that is forming with the most likely mechanism being the re-coater blade.

Finally, an investigation into whether location plays a role in defect concentration in UNS N07718 parts built via LPBF was conducted. Figure 6.24 depicts a top view of the samples on the build plate and the incorporated area fraction of inclusions measured has been placed above (or below in some cases) the sample it corresponds to in the figure.



Figure 6.24: Top View of 1st Build Plate

Of the samples designed for this location study, they all seem to have very similar incorporated area fractions of oxides, except for the sample located near the front left of the build plate. One reason for the spike in incorporated oxide concentration for this sample may have to do with the flow of argon in the chamber. Argon flows from the back of the chamber (red line) to the front (blue line) and into an extraction nozzle that is located at the front left of the chamber. This means that any oxidized material in the chamber is flowing directly toward this sample, which may have landed on the sample and resulted in the increase in area fraction.

6.3 Hot Cracking

After imaging, it was noted that the samples from NASA Langley exhibited more hot cracking behavior than those built at CMU. These cracks are likely to be the sites of fatigue crack initiation, which is very detrimental to the performance of the material. Examples of these cracks are shown in Figure 6.25 below.



Figure 6.25: Top Surface of Samples Created Using Praxair Powder

To investigate this behavior further, we had samples from both sources sent out for compositional analysis to see if there was any variance in elemental presence that could explain this oddity. Glow Discharge Mass Spectrometry (GDMS) was done to detect the presence of boron and sulfur; and Thermal Extraction (TE) was done to identify the concentrations of carbon, nitrogen, and oxygen. Table 6.4 below shows the resulting information for the samples analyzed.

 Table 6.4: Compositional Analysis Results

Sample ID	C [ppm]	N [ppm]	O [ppm]	B [ppm]	S [ppm]
KEY8	370	130	170	2.6	24
PAC	550	340	140	32	10
PRAX	330	130	200	-	-

Sample KEY8 was created at CMU using powder provided by EOS, and the PAC and PRAX samples were built at NASA LaRC using different powders (PAC = Powder Alloy Company, PRAX = Praxiar). The table above shows some interesting distinctions between the composition of the samples, notably with PAC having a higher carbon and nitrogen content than the others. There is also a magnitude of ten difference in the boron concentrations between samples. The effects of boron and carbon on the solidification range of the material are possibly the reason for the change in hot cracking behavior. Kelly studied the compositional effects on the weldability of cast UNS N07718 and he found that boron was the most detrimental element in that it appeared to aggravate the weld heat-affected zone (HAZ) liquation cracking [49]. Chen et al. noted in their study of cast UNS N07718 with various compositions that boron tended to promote Laves phase

formation. They found that high boron (~0.01 wt%) and niobium content enhanced the formation of Laves phase, which lowers the liquation temperature of the alloy [50].

Upon running Scheil simulations using Thermo-Calc for the boron concentrations listed in the Table 6.4 above, the results showed that boron had no effect on the solidification range of the material. This is probably because the concentration of boron in both samples listed above is too low. However, the concentration of carbon and nitrogen does seem to have an effect as shown in the plot below.



Figure 6.26: Kou Criterion for Crack Susceptibility

Kou et el. developed an index that can be used to predict the crack susceptibility of an alloy qualitatively. The index is based on the slope of the curve generated when temperature is plotted versus the square root of the fraction solid and is written out thusly: $|dT/d(f_s^{1/2})|$ near $f_s^{1/2} = 1$. When comparing two curves, the alloy generating the curve with the steeper slope when the fraction solid is between 0.87 and 0.94 is the more likely alloy to experience cracking [51]. Applying this to the plot above, one can see that the PAC sample has a slightly steeper curve within the specified range; therefore, it is more susceptible to cracking.

Chapter 7: Conclusions

7.1 <u>Hypothesis #1: Melt Pool Geometry</u>

• When operating in the process window of P-V space, the melt pool geometry for UNS N07718 can be predicted by the Rosenthal equation to within 10% error

This hypothesis is supported, but only by one sample, as the other samples created to measure the melt pools for comparison to the predictions from the Rosenthal relations were all keyhole shaped. The prediction must be tested against non-keyholing melt pools for an accurate comparison to be made. Sample TA4 was the only sample printed in conduction-mode melting and both the melt pool width and depth measurements were within 10% error. More samples would need to be fabricated to increase support for this hypothesis.

7.2 Hypothesis #2: Groove Depth

• Variation of the hatch spacing affects the size of grooves that form between melt pools

This hypothesis is supported by the results obtained from this work. The groove depth was found to vary as a function of the ratio of the hatch spacing to melt pool width. Furthermore, these depths were very comparable to those predicted using a mathematical relation, with the discrepancies partially explained by wetting between the solid and liquid metal.

7.3 <u>Hypothesis #3: Lack-of-Fusion Porosity</u>

• The previously established model for predicting lack-of-fusion porosity works for laser powder-bed fusion of UNS N07718

This hypothesis is supported. Once the actual melt pool dimensions were obtained, the index relation correctly predicted that none of the samples created in this work would showcase lack-of-fusion porosity. This was further verified by simulations done using the measured dimensions to estimate the expected amount of lack-of-fusion porosity, where the melt-pool sizes were assumed to be normally distributed, according to the measured average depth and width and their standard deviations. Also, results from μ CT scanning did not show any periodic formation of porosity that is characteristic of lack-of-fusion pores.

7.4 Hypothesis #4: Alumina Formation

• There is a greater concentration of aluminum oxide formation nearer the top of a build than at the bottom

This hypothesis is not supported. The results do not show any appreciable difference in the oxide concentration between the top and bottom halves of samples. This means that the increase in area fraction of oxides found on the surface of the parts must be a layer-by-layer development, as opposed to being a general increase in the concentration as the sample is built.

7.5 Hypothesis #5: Location Effect

• The location where a part is built on the build plate has an appreciable effect on the defect concentration

Because of the increase in incorporated area fraction of oxides for a sample located at the front left of the build plate, this hypothesis is supported. However, more samples would need to be fabricated to increase support for this hypothesis.

Hypothesis #6: Hot Cracking

• The percentage of boron in the composition of UNS N07718 has an appreciable effect on the hot cracking susceptibility of the material as predicted by the Kou model

The results from the Kou model conclude that this hypothesis is not supported. The small concentration of boron in UNS N07718 has no effect on its solidification range or cracking behavior. However, the concentrations of carbon and nitrogen were found to have some effect on the cracking behavior of the alloy as shown in Figure 6.26.

Chapter 8: Contribution and Publication Plan

8.1 Contribution

Overall, this work has shown that when it comes to fabricating samples and qualifying parts, process parameter selection plays a very important role in the outcome of the part. It was noted that the standard conditions for creation of UNS N07718 parts resulted in keyhole-shaped melt pools. Therefore, it was found to be possible to generate samples at faster build rates without sacrificing complete melting of the material. However, increasing the layer thickness to achieve this faster build rate may not be the best idea. While the samples are created faster, there is a sacrifice made regarding surface finish of the part. This work also showed that calculation of the groove depth between melt pools is possible and compares well to actual measurements when using a 40-micron layer thickness. Finally, this work showed that severe keyholing results in the increase in the concentration of pores and oxides in and on top of parts.

8.2 Publication Plan

As for publications, some of the work I have done is already published in an ASTM manuscript, which has been cited as reference [23]. On top of this publication, the plan is to have two papers derive from this document: one on melt pool geometry prediction using mathematical relations and the other on correlations between processing parameters and incorporated and surface oxide formation.

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