Microstructural Development of Inconel 625 Nickel-Based Superalloy as Function of Laser Powder Bed Fusion Parameters

Sofia Nucci
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MICROSTRUCTURAL DEVELOPMENT OF INCONEL 625 NICKEL-BASED SUPERALLOY AS FUNCTION OF LASER POWDER BED FUSION PARAMETERS

by

SOFIA CAROLINA NUCCI
B.S. University of Central Florida, 2014

A thesis submitted in partial fulfillment of the requirements for the degree of Master of Science in the Department Materials Science and Engineering in the College of Engineering and Computer Science at the University of Central Florida Orlando, Florida

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ABSTRACT

Additive manufacturing (AM) allows fabrication of complex components with features that are impractical or impossible to achieve through conventional methods. Selective laser melting (SLM) powder bed fusion AM technology was selected for this study on Inconel 625, a widely utilized high-temperature alloy that is hard to machine. The present work investigates impact of laser power and scanning speed variations on the resulting characteristics of fabricated IN625 samples. Gas atomized metallic alloy powders were acquired and analyzed through laser diffraction to verify acceptable size distribution. Cubic samples were built with a range of laser scan speeds in 200 mm/s intervals for each laser power evaluated (125W, 200W, 275W, and 350W) while holding a constant 0.12 mm hatch spacing, 0.03 mm layer thickness, and 16-degree scan rotation angle. Archimedes’ method and optical image analysis were carried out to determine relative density of the samples. All laser powers evaluated yielded at least one sample with relative density above 99.7% as determined through both measurement techniques. Correlation of energy density with resulting sample porosity was identified with highest relative density values associated to energy densities in the 55 – 69 J/mm$^3$ range. Samples were sectioned and etched for examination of relevant microstructural features through optical and scanning electron microscopy; melt pools were measured and cell size approximated. Consistent cooling rate values in the order of $10^5 – 10^6 \, K/s$ were obtained from Rosenthal’s equation models and from secondary dendrite arm spacing calculation.
The author would like to dedicate this work to her family and friends who have provided support throughout the years leading up to this milestone.
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# TABLE OF CONTENTS

LIST OF FIGURES .................................................................................................................. viii

LIST OF TABLES ....................................................................................................................... x

CHAPTER 1: INTRODUCTION ................................................................................................... 1

CHAPTER 2: LITERATURE REVIEW .......................................................................................... 3

  2.1 Selective Laser Melting Additive Manufacturing .......................................................... 3
  2.2 As-Built Characteristics of AM Inconel 625 ................................................................. 5
  2.3 Heat Treatment of IN625 .............................................................................................. 7
  2.4 Microstructure & Mechanical Properties .................................................................... 10

CHAPTER 3: EXPERIMENTAL METHODS ................................................................................. 14

  3.1 Sample Fabrication ...................................................................................................... 14
  3.2 Microstructural Analysis .............................................................................................. 17

CHAPTER 4: RESULTS ................................................................................................................ 21

  4.1 Powder Feedstock Analysis ........................................................................................ 21
  4.2 Evaluation of Defects .................................................................................................. 22
  4.3 Dimensional Analysis of Melt Pool ............................................................................. 26
  4.4 Cellular Spacing and Cooling Rate Calculation ........................................................ 29

CHAPTER 5: DISCUSSION .......................................................................................................... 35

  5.1 Microstructural Analysis .............................................................................................. 35
  5.2 Theoretical Approximations ....................................................................................... 37
LIST OF FIGURES

Figure 1. IN625 Powder XEDS compositional analysis of (left) aggregate and (right) individual powder particles. .................................................................................................................................................................................. 14

Figure 2. Selective laser melting machine model 125HL by SLM Solutions Group AG. .......... 15

Figure 3. Schematic of sample cross-sectioning and identification of planes. ......................... 17

Figure 4. Sample density quantification via image analysis: (a) optical micrograph after thresholding, (b) processed image with flaws outlined. ........................................................................................................ 17

Figure 5. Schematic of melt pool measurement method and overlay on representative optical micrograph. ................................................................................................................................................................................................. 18

Figure 6. Representative SEM BSE image with overlay of lines utilized to count intersections between cellular gridlines as indicated by the red points. ................................................................. 19

Figure 7. Plot of IN625 powder size distribution. ........................................................................ 21

Figure 8. Compilation of optical micrographs from XY and XZ planes for all samples organized with respect to laser power and scan speed. Optimal processing window indicates most dense samples for every laser power investigated (constant 120 µm hatch spacing, 30 µm layer thickness, and 16-degree scan rotation). .................................................................................................................................... 24

Figure 9. Density plotted as function of scan speed for various laser powers employed. ........ 25

Figure 10. Average density values determined from Archimedes’ method plotted as function of energy density for various laser powers employed. ................................................................................................................. 26

Figure 11. Optical micrographs of melt pools for samples built with 200W laser power and (a) 400 mm/s, (b) 800 mm/s, (c) 1200 mm/s, (d) 1600 mm/s. ........................................................................................................................................ 27
Figure 12. Melt pool dimensions plotted as function of laser scan speed for various laser powers employed...

Figure 13. SEM images of cellular structure for samples built with 125W laser power at (a) 200 mm/s, (b) 400 mm/s, (c) 600 mm/s, (d) 800 mm/s. .............................................................

Figure 14. Cell size plotted as function of laser scan speed for various laser powers employed. 31

Figure 15. Cooling rate plotted as function of laser scan speed for various laser powers employed. ............................................................. 34

Figure 16. SEM images of un-melted powder particles observed in samples built with (a) 350W laser power at 2000 mm/s, and (b) 200W laser power at 200 mm/s................................. 36

Figure 17. XRD patterns for (left) IN625 sample printed with 275W laser power, 1600mm/s scan speed, 0.12 mm hatch spacing, 0.03 mm layer thickness, and 16° scan rotation, (right) pure Nickel. ............................................................. 37

Figure 18. Plots of calculated cooling rate for samples printed with 200W laser power. SDAS results compared to Rosenthal model with (left) constant 0.57 absorptivity, (right) absorptivity between 0.36 and 0.96 increasing linearly between 0.2 and 0.5 laser specific energy (J/mm) [20]. .................................................................................................................................................. 39
LIST OF TABLES

Table 1. Print parameters utilized to generate the samples in this study .................................. 16
Table 2. IN625 powder size data. .................................................................................................. 21
Table 3. IN625 powder particle composition data ......................................................................... 22
Table 4. IN625 properties used in cooling rate calculation [20] ....................................................... 32
Table 5. Results of cooling rate calculations for each sample. ......................................................... 32
CHAPTER 1: INTRODUCTION

Over the past twenty years there have been significant developments in the constituent technologies required for additive manufacturing (AM) of metals, such as industrial lasers and powder feedstock processing, which have led to its adoption by industries for commercial production [1]. One of the key aspects of AM is that it allows creation of complex structures that are very costly to produce through casting or subtractive manufacturing, particularly for hard metals. Metallic components in the energy, aerospace, medical, and automotive fields are being designed and manufactured through AM due to its many advantages compared to traditional manufacturing procedures [1]. Manufacturing waste can be reduced with AM since only the required amount of raw material for a component is consumed and leftover powder particles can generally be reutilized [2]. AM also enables designers to change assemblies into more streamlined single-piece components which, in turn, can translate to reduced production times and lengthening of the operational life of the part.

Out of the many different AM methods available, this work focuses on Selective Laser Melting (SLM) Powder-Bed Fusion (PBF) technology. The complex interplay of the SLM parameters and variability of results with different alloys has led to significant research for determination of optimal build settings for specific materials. This study focuses on the alloy Inconel 625 (IN625), patented December 1964 after a decade of research on the basic Ni-Cr-Mo-Nb alloy system [3]. It is widely-utilized to this day because of its tensile strength, outstanding performance under fatigue and creep conditions, strong resistance to corrosion, and excellent weldability [3, 4]. The relatively low thermal conductivity and volume-specific heat make IN625 hard to cut through machining, a conventional manufacturing process [4]. These characteristics
make AM an attractive manufacturing method for IN625 components, however, the resulting properties differ from those of conventionally-processed IN625 alloy. Although SLM has been shown to satisfactorily process Nickel-based alloys, the fast cooling rate (in the order of $10^6$ K/s) associated with SLM does impact the microstructural development [2].

The main objective of this work was to evaluate impact of laser power and scan speed variables on the resulting characteristics of AM-fabricated IN625. SLM parameters were systematically modified to observe changes in melting mode and solidification outcomes. Development of melt pool geometry and resulting microstructure were documented for all samples. Analysis of porosity with respect to parameter variation was carried out to identify an optimal processing window. Melt pool depth and width were measured and size of cellular morphology approximated. Models from welding metallurgy were employed to evaluate their effectiveness in approximating experimental data for melt pool dimensions and cooling rate.
CHAPTER 2: LITERATURE REVIEW

2.1 Selective Laser Melting Additive Manufacturing

Manufacturing through SLM requires a CAD model of the component, properly oriented and considering structural supports, in order to program the build parameters. In the case of PBF, the laser scans on a layer of deposited powder particles and fuses together the powders on the scanned area. Parts are built directly onto an interchangeable base plate of the same material and must be cut off from the plate upon build completion. The base plate moves vertically downward, by a programmed height corresponding to the selected layer thickness, then the powder re-coater or spreader goes over the work area to deposit raw material. At the end of the build process, the part is surrounded by unfused powder particles that can be re-utilized for another printing run. This setup is enclosed in a unit that allows control of the gaseous atmosphere because atmospheric air has high oxygen content which can be detrimental to the final product. Depending on the material being printed, Nitrogen or Argon gas flood the chamber during the build process in order to reduce likelihood of oxidation. Capabilities offered with the machines vary by brand, but some type of continuous monitoring for layer quality is generally offered to allow for early detection of potential defects in the build.

Due to the unique characteristics of different materials, the processing parameters have to be specifically adjusted to the material in order to obtain fully densified parts without major defects. The most important variables to control, in terms of their impact on the final product, are laser power (W), scan speed (mm/s), layer or slice thickness (mm), hatch spacing (mm), and scan rotation angle from layer to layer. Additional parameters include the pattern of the laser path, focus
point of the laser to control the spot size, and base plate heating temperature. Energy Density as defined in Equation (1) [5] is commonly calculated to describe the processing parameters in a normalized manner that simplifies comparison of the variable combinations when analyzing sample data.

\[
\text{Energy Density (ED)} = \frac{\text{Laser Power}}{\text{Scan Speed} \times \text{Hatch Spacing} \times \text{Slice Thickness}}
\]  

The amount of energy transferred to the powder, mainly determined by the laser power and scan speed, affects the amount of melting that can be achieved and thus the resulting structure [6]. Hence, the higher scan speeds desirable to reduce building time can only be utilized if the laser power is sufficiently strong to achieve complete melting. Partial melting results in a porous and typically brittle structure of coarse agglomerates, sometimes un-melted particle cores baked together, which is undesirable [6]. Furthermore, defects such as melt pool break-up, bead formation, and denudation of adjacent powder bed worsen with increasing laser scan speed and power due to greater material ejection from shear gas flow [7]. A general trend is that cooling rates decrease with increasing laser power for a constant scan speed, but cooling rates generally increase with increasing scan speed [7].

The final qualities of AM parts are also greatly influenced by the shape, size distribution, surface morphology, composition, and flowability of the powders; typical size range for SLM powders is 10 to 60 µm [1]. It has become a widely accepted industrial practice to reutilize leftover powder from previous builds, but it is worth noting that some variabilities in microstructure and composition compared to virgin powder have been observed even though the particle size and shape do not exhibit significant changes [8]. Powder particle size distribution affects packing and thus density of the printed part, additionally it shall be considered with respect to layer thickness to ensure proper powder deposition. Hatch spacing is the distance between adjacent laser scans.
and is important in achieving a fully densified build, if the spacing is too large the melt pools will not overlap and thus cause porosity in the structure. A study by Marchese et al. [9] observed that reducing hatch spacing resulted in improved densification and hardness while high scanning speed induced the opposite, more porosity and lower hardness. Scan rotation angle, also referred to as hatch angle, between the directions of consecutive layer scans plays a significant role in the isotropy of mechanical properties for some materials [6]. Different rotation angles between layers in the scan strategy also affect the melting and solidification rates, namely the thermal cycling, throughout the process and thus impact the coarseness of the final microstructure [9]. It is worth noting that the surface roughness varies with the angle of the built part and cantilevered surfaces showed higher average roughness values on the bottom side (downskin) due to large volumes of attached partially melted particles [10]. Depending on the application requirements of the manufactured component this surface quality could be advantageous or detrimental, so post-processing of the surface may be necessary.

2.2 As-Built Characteristics of AM Inconel 625

Due to the localized high heat input and thermal cycling intrinsic to the SLM building process, the resulting structure often has elevated residual stress, heterogeneous metastable microstructures, and nonequilibrium elemental compositions or phase distributions [11]. This requires evaluation of specific post-processing options to obtain the standard IN625 alloy characteristics from the AM-processed alloy, both a challenge and an opportunity to optimize the properties for specific applications. Analysis of the as-built AM microstructure becomes necessary to understand the starting point.
Pronounced grain elongation along the build direction with a range of non-uniform crystallographic orientations within the grains is observed in AM-built IN625 samples, contrasting with the equiaxed grain structure of wrought IN625 [11]. The phenomenon of grain growth in the direction of heat extraction was observed consistently for IN625 as well as other AM alloys [1, 11, 12]. These grains are able to grow epitaxially, thus intersecting multiple melt pools along the build direction [9]. In a study with unspecified AM-build parameters by Kreitberg et al. [12], as-built and stress-relieved IN625 samples exhibited strong build-orientation dependency for yield and ultimate tensile strength values; suggesting that grain elongation leads to anisotropy. However, an evaluation of Nickel-based alloy properties from different studies found no clear trend in anisotropy despite the columnar grains [1]; indicating that there could be other factors affecting the directionality of mechanical properties. This observation is most likely due to the variability in results with interplay of different AM build parameters, especially since the scan rotation utilized to produce parts is not always disclosed and could affect isotropy.

The face-centered cubic (FCC) crystal structure of the starting IN625 powder is preserved through the AM process, however, Zhang et al. [11] observed asymmetric peaks in the XRD data of as-built material which were presumably caused by localized elastic strains and compositional gradients. Both of these phenomena can be attributed to the fast cooling rates associated with the SLM process, typically in the order of $10^6 ^\circ\text{C} /s$ [13]. For comparison, conventionally cast alloys solidify with a cooling rate on the order of 1 to 1000 $^\circ\text{C} /s$ [14]. The fast solidification in AM leads to high concentration of tangled dislocations which in turn cause localized strains and variation in lattice parameters for the crystal structure [15]. This resulting residual stress in as-built AM IN625 parts is linked to elevated hardness values compared to the conventionally manufactured equivalent [4]. Furthermore, rapid cooling causes compositional gradients in the form of elemental
micro-segregations due to solute rejection under nonequilibrium solidification conditions [11]. This is observed consistently in many studies through formation of dendrite cores enriched in Ni and Cr with a deficit of those elements in the inter-dendritic regions, which in turn show enrichment of solute elements Nb and Mo [8, 11, 14]. The importance of this phenomenon is that, upon analyzing local compositions, the resulting mass fractions do not consistently meet the chemically allowable values mandated for IN625 [11]. These deviations from the alloy specifications can lead to deviations in the material behavior. For example, a study by Marchese et al. [15] identified Nb-rich MC carbides of 10 to 50 nm in size formed during solidification inside dendrite cores through the eutectic reaction (L → γ + MC), presumably due to very high cooling rates trapping solute in the core. Observing the solidification structure, primary dendrite spacing in the AM build is on the order of 1 µm while secondary dendrite arm spacing, approximately 300 nm, is harder to differentiate due to cooling rate near the transition point to cellular solidification [14]. Dendrite arm spacing for a traditional casting is 100 to 300 µm, comparatively two orders of magnitude larger than obtained with AM [13]. These differences between additive and conventional processing of the alloy have a significant impact on the properties and macroscopic behavior of the final product.

2.3 Heat Treatment of IN625

Studies of as-built IN625 parts produced with AM revealed macroscopic residual stresses exceeding 750 MPa, so stress relief heat treatment with careful monitoring of microstructural evolution is recommended [11]. Reevaluation of standard heat treatment cycles becomes necessary for AM parts due to the local discrepancies in composition impacting the equilibrium fractions of the precipitate phases [8]. Observed variation in composition from the starting point and within
the different areas of the build is significant, for example localized Nb and Mo concentrations can be as high as 9 and 11 mass percent respectively though the feedstock powder contained 3.79% Nb and 8.83% Mo [8]. Considering these deviations, a study by Zhang et al. [11] produced a calculated equilibrium isopleth phase diagram for IN625 over a range of 3% to 10% Nb mass fraction and temperatures of 600°C to 1400°C for visualization of differences to be expected in heat treatment results compared to conventionally manufactured IN625. Since as-built AM parts display this wide variation in local composition, the phase diagram informs that heat treatment results will not be consistent across the entire part unless the microstructure is homogenized. Furthermore, simulations of precipitation kinetics of standard IN625 feedstock powder compared with one typical composition found in AM IN625 revealed that, while the transformation sequence persists, both γ” and δ phases emerge much sooner in the AM alloy [11]. The following findings from several studies on various heat treatments, both ageing and solutioning, of IN625 samples provide more wholistic observations.

One manufacturer of laser powder bed fusion equipment suggests 1 hour at 870°C direct ageing for stress relief of as-built parts, but a study showed that while it does significantly reduce residual stresses it also leads to formation of deleterious phases [14]. Examination of microstructural evolution of AM IN625 directly aged at 870°C for periods of 0.5, 1, 4, and 8 hours with SEM and TEM revealed formation of precipitates, needle-shaped δ-phase and smaller globular MC carbides, and increasing volume fraction with longer heat treatment time duration [11]. Direct ageing at 800°C for 1 hour is proposed by Lass et al. [14] as an alternative to minimize precipitation of detrimental phases, maintaining similar composition maps to the as-built sample, while still reducing residual stresses. Sample analysis showed that less than 1% volume fraction of δ-phase precipitates form after 1 hour at 800°C and increase to roughly 6% after 4 hours, but in
both cases the precipitate volume is less than formed after 1 hour at 870°C [14]. Residual macro-
stress in these AM IN625 samples was analyzed with neutron diffraction; the nearly 800 MPa of
the as-built condition were reduced to roughly 200 MPa after 1 hour at 800°C while in the same
time at 870°C these reached around 100 MPa [14]. Hence, the majority of the stress relief can be
achieved with the lower temperature and perhaps even more reduction could be obtained with
additional time at the same 800°C.

Another study by Stoudt et al. [8] analyzed XRD patterns of samples directly aged for
duration of 1 hour at 700°C, 800°C, 870°C, and 950°C; the pattern for 700°C was virtually
identical to the as-built condition exhibiting no identifiable peaks aside from those of FCC phase.
The 800°C sample pattern began to differ with two small peaks near 46 deg 2θ associated with the
orthorhombic Ni\textsubscript{3}Nb δ-phase and, as expected from observation of precipitates through SEM
images, these peaks become more pronounced (greater intensity) in the 870°C sample pattern [8].
A change in overall character for the sample aged at 950°C is observed through SEM, suggesting
initial stages of homogenization, while the XRD pattern revealed a reduction in the intensity of the
peaks near 46 deg 2θ compared to the 870°C sample and the appearance of small (Nb, Mo)C matrix
carbide peaks near 42.5 deg and 44.5 deg [8]. This change in the XRD peaks for heat treatment at
950°C is consistent with observations of a study by Zhang et al. [11] which conducted heat
treatment at 1150°C for 1h on a specimen previously aged at 870°C for 1h. The elemental
distribution homogenized after 1h at 1150°C resulting in a single-phase FCC structure, high-
resolution synchrotron XRD analysis demonstrated complete dissolution of δ-phase precipitates
from the previous 870°C ageing [11]. Furthermore, the resulting FCC lattice parameter was 0.0167
Å larger than that for another sample aged at 870°C for 8 hours which suggests that the heavy
elements in the δ-phase became part of the solid solution in a homogenized matrix [11]. This
homogenization points to solutioning heat treatment as a means to mitigate the impacts of localized compositions on the kinetics of microstructure evolution in as-built AM parts.

2.4 Microstructure & Mechanical Properties

Samples of IN625 produced with AM display columnar grains along the build direction with both cellular and columnar dendritic structures, high density of tangled dislocations, segregation of solute elements, and Nb-rich MC carbides with no evidence of δ and Laves phases [15]. These general characteristics describe the as-built microstructure of IN625 processed via SLM, serving as baseline for evaluation of mechanical properties and changes with heat treatment. Marchese et al. [15] analyzed direct ageing of samples, collecting data points at 2, 8, and 24 hours of exposure for 600°C, 700°C, 800°C, and 900°C with subsequent water quench, and identified an overall increase in hardness, particularly for the 700°C and 800°C samples, with no significant changes in the grain morphology. This relative increase in the hardness can be attributed to formation of precipitates, nanometric γ” in the 600°C and 700°C samples versus δ and Laves phases formed in the 800°C and 900°C samples [15]. It is worth noting that γ” strengthening requires careful consideration for component application since metastable γ” eventually transforms to equilibrium δ-phase, which can significantly reduce IN625 fracture toughness and ductility if developed along the grain boundaries [11].

Alloy IN625 was designed to be solid-solution strengthened and can contain some inherent MC, M₆C, and M₂₃C₆ carbides [16]. For conventionally solidified IN625, formation of γ” requires more than 6 hours of exposure at 650°C while δ and Laves phases take roughly 20 hours at 870°C, even longer at lower temperatures [17]. Zhang et al. [11] performed an in-situ XRD experiment at 870°C to investigate the impact of δ-phase formation, observing that it causes a decrease of the
FCC matrix lattice parameter as the heavy elements, such as Nb and Mo, are depleted from the solid solution that normally strengthens the IN625 alloy. This is particularly problematic since formation of the detrimental δ-phase occurs much faster in as-built AM samples. In the 800°C to 870°C temperature range, the incubation time necessary for nucleation of δ-phase in AM samples was less than 5 minutes; indicating a low nucleation barrier that is possibly due to the high dislocation density observed in as-built parts providing heterogeneous nucleation sites [11]. Nevertheless, it appears that a certain crystallographic orientation of δ-phase with the FCC matrix is required since the long axes of the precipitates always align with the close-packed directions of the matrix [11]. Taking these factors into consideration, it is preferable to relieve residual stresses with a heat treatment temperature lower than 800°C if a sufficiently low stress level for the application can be achieved.

An alternate route is to perform solutioning heat treatment, thus homogenizing the microstructure in an attempt to obtain the same alloy behavior as for conventionally-processed IN625. Marchese et al. [15] evaluated solution treatment of samples at 1000°C and 1150°C for 2 hours followed by water quenching, results indicated a recrystallization of the microstructure with low dislocation density, elimination of the fine dendritic structures, and development of equiaxed grains of sizes ranging from 10 µm up to 90 µm with numerous twin boundaries. The treatment at 1150°C also resulted in formation of fine sub-micrometric Nb,Ti-rich carbides, both primary and high-temperature secondary carbides, which would require higher solutioning temperature to be dissolved [15]. These changes in microstructure translated to reduced hardness, more so for the 1150°C sample than for the 1000°C one, and increased ductility accompanied by lower yield stress (YS) and ultimate tensile stress (UTS) compared to the as-built sample properties. Despite this reduction, the YS and UTS values for the samples were above the minimum values, 276 MPa and
690 MPa respectively, stipulated for wrought IN625 per ASTM B443 (grade 2 solution annealed at least at 1093°C) [15]. The study went one step further, taking samples solutioned at 1150°C for 2 hours and ageing them at 600°C, 700°C, 800°C, and 900°C for up to 24 hours to investigate the alloy behavior. The ageing treatment after solutioning had no effect on the grain size and the intragranular Nb,Ti-rich carbides observed in the solutioned samples remained, but Cr-rich M_{23}C_{6} carbides ranging in size from 100 nm up to microns appeared at the grain boundaries [15]. Intragranular ellipsoidal γ’’ phase between 10 and 30nm formed in the samples aged at 600°C and 700°C, different from the homogeneous appearance of γ’’ in the directly aged samples, while δ and Laves phases still appeared in the samples aged at 800°C and 900°C [15]. The 700°C ageing process proved most effective at increasing hardness for all time periods analyzed by Marchese et al. [15], followed by the 800°C and 600°C ageing temperatures, while hardness for the 900°C aged samples remained at the same level as the solutioned ones. Comparing results of as-built samples with those directly aged at 700°C for 24 hours and solutioned at 1150°C for 2 hours prior to the same ageing treatment, direct ageing resulted in the highest YS and UTS values with a loss in ductility from the as-built condition while solutioning prior to ageing resulted in roughly 60 MPa lower YS, about 75 MPa higher UTS, and same ductility of as-built condition [15].

Differences in the observed hardness, strength, and elongation for as-built and heat-treated samples are also apparent in the fracture mechanisms of tensile specimens. The as-built microstructure resulted in a ductile fracture mode with micro-void coalescence coupled with some brittle fractures while in the directly aged specimens the number of brittle fractures increased [15]. Solutioning of the specimens resulted in a ductile fracture mode with larger micro-voids than the as-built case, but after ageing of the solutioned sample the fracture became mixed ductile and brittle with presence of secondary cracks [15]. The root cause of the brittleness observed in these
fractures was not investigated in detail, but it is proposed that Nb-rich MC carbides, intergranular M\(_{23}C_6\) carbides, and \(\gamma''\) phase play a role [15]. All the observations up to this point, however, are based on room temperature testing of an alloy which is commonly utilized for high temperature applications. Kreitcberg et al. [12] evaluated tensile properties both at room temperature and at 760°C for as-built, stress relieved, and solution treated IN625 samples produced with laser powder bed fusion, observing that the elevated temperature UTS and ductility of the as-built specimens are lower than those of the stress relieved (directly aged) and solution treated ones. The as-built and stress relieved specimens displayed yield strength peaks, not observable in room temperature tests, and the stress relieved condition undergoes continuous work-softening beyond the peak [12]. Examination of the 760°C fracture surface of stress relieved samples showed brittle fracture characterized by intergranular crack propagation along high-angle grain boundaries containing \(\delta\)-phase with triangularly shaped serrations and globular M\(_6\)C carbides [12]. Furthermore, the AM specimens displayed opposite behavior to annealed wrought IN625 alloy (ASTM B443) when comparing ductility at room temperature and 760°C; both as-built and heat treated specimens display a significant reduction in achievable % elongation from room to high temperature while the wrought alloy increases elongation by nearly 15% at 760°C [12]. Considering these observations, further investigation of the high temperature behavior of IN625 processed through AM is recommended for such applications.
CHAPTER 3: EXPERIMENTAL METHODS

3.1 Sample Fabrication

Metallic alloy powders are the starting point in fabricating components through SLM. For this study the powder utilized was gas-atomized IN625 supplied by SLM Solutions Group AG. The size distribution was evaluated using LS 13 320 laser diffraction particle size analyzer (Beckman Coulter). The powders were mounted in epoxy resin and mechanically polished down to 0.25 μm with diamond paste, then etched for microstructural analysis. The etchant utilized was a mixture of hydrochloric acid (HCl), acetic acid (CH₃COOH), and nitric acids (HNO₃) at a volumetric ratio of 3:2:1, respectively. Chemical composition of the alloy powders was verified with a field emission scanning electron microscope (FE-SEM, Zeiss Ultra-55TM) equipped with X-ray energy dispersive spectroscopy (XEDS) capability. As shown by the orange rectangles in Figure 1, individual powders as well as an aggregate group were evaluated for composition.

![Image](image1.png)

Figure 1. IN625 Powder XEDS compositional analysis of (left) aggregate and (right) individual powder particles.

Thirty-three cubic samples with dimensions 10 × 10 × 10 mm were fabricated using laser powder bed fusion system, SLM® 125HL from SLM Solutions Group AG, pictured in Figure 2.
This system has a maximum build volume of 125 mm$^3$, offers a build rate of up to 25 cm$^3$/hr, and is equipped with a single Ytterbium IPG fiber laser (400 W) with 1070 nm ±10 nm wavelength, 100 μm beam focus diameter, and 70 μm spot size.

![Selective laser melting machine model 125HL by SLM Solutions Group AG.](image)

**Figure 2.** Selective laser melting machine model 125HL by SLM Solutions Group AG.

The SLM Solutions recommended parameters for manufacturing IN625 parts are 200 W laser power, 900 mm/s scan speed, 0.12 mm hatch spacing, 0.03 mm layer thickness, and 16-degree scan rotation. Of those parameters, the laser power and scan speed were varied as listed in Table 1 for each cubic sample in order to evaluate impact on the resulting microstructure. To enable evaluation of the melt pool dimensions, samples were built such that the laser path for the final print layer of each sample lied parallel to one set of cube edges. All samples were printed with 100°C base plate heating, stripe laser pattern, and Argon atmosphere (oxygen concentration at or below 0.1%). The inert gas is particularly important for IN625 since it has a stronger tendency
than most Nickel-based alloys to retain Nitrogen due to the relatively high levels of Cr and Mo in the alloy [3].

Table 1. Print parameters utilized to generate the samples in this study

<table>
<thead>
<tr>
<th>Laser Power (W)</th>
<th>Scan Speed (mm/s)</th>
<th>Hatch Spacing (µm)</th>
<th>Slice Thickness (µm)</th>
<th>Scan Rotation (°)</th>
<th>Energy Density (J/mm³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>125</td>
<td>200 – 800</td>
<td>120</td>
<td>30</td>
<td>16</td>
<td>43.4 – 173.6</td>
</tr>
<tr>
<td>200</td>
<td>200 – 1800</td>
<td>120</td>
<td>30</td>
<td>16</td>
<td>30.9 – 277.8</td>
</tr>
<tr>
<td>275</td>
<td>400 – 2000</td>
<td>120</td>
<td>30</td>
<td>16</td>
<td>38.2 – 191.0</td>
</tr>
<tr>
<td>350</td>
<td>600 – 2200</td>
<td>120</td>
<td>30</td>
<td>16</td>
<td>44.2 – 162.0</td>
</tr>
</tbody>
</table>

Samples were cut off from the build plate without any stress-relieving heat treatment so as to retain the as-built microstructure and properties. In preparation for density measurements, all sample surfaces were smoothed with silicon carbide (SiC) paper to reduce formation of surface bubbles upon immersion. Relative density was afterwards determined via Archimedes’ method in accordance with ASTM B962-17 standard. To reveal microstructural features of interest, the samples were then sectioned using a low-speed diamond saw and labeled as shown in Figure 3. Section XZ lies parallel to the build direction while section XY is parallel to the build plate and perpendicular to the build direction. The sectioned samples were mounted in epoxy resin and mechanically polished to a final 0.25 µm with diamond paste, then etched for 30 to 60 seconds with the above-mentioned acid mixture.
3.2 Microstructural Analysis

For determination of relative density through image analysis, a Nikon Metaphot optical microscope was utilized to capture micrographs from the sample cross-sections prior to etching. These were then processed through ImageJ (National Institutes of Health) software to quantify porosity as illustrated in Figure 4. Data from 10 optical micrographs per sample, taken at 5 random locations on each of the two sections, was utilized to approximate relative density of the samples. The porosity area percentage values for the 10 micrographs were averaged to obtain a single value per sample, then subtracted from 100% to determine the relative density.
After the samples were etched, additional optical micrographs of the XZ cross-section were captured at random locations along the edge corresponding to the final layer of the SLM build. These images allowed measurement, with ImageJ software, of the melt pool depth and width as illustrated in Figure 5. There is a noticeable superposition on the melt pool widths resulting from overlap of the melt pools generated by each adjacent laser scan. Assuming symmetry of the individual melt pools, the width for each melt pool was approximated by measuring from the identifiable edge to the center and multiplying by a factor of two. The average depth and width values were determined from the measurements of 14 melt pools per sample in optical micrographs from different locations on each sample.

![Figure 5. Schematic of melt pool measurement method and overlay on representative optical micrograph.](image)

The etched samples were also observed by secondary and backscatter electron (SE and BSE) micrographs from the XY cross-sections. These images serve to quantify the cellular microstructure within the melt pools through the linear intercept method as stipulated in ASTM E112 – 13. ImageJ was utilized to superimpose horizontal gridlines at random locations on the BSE micrographs. The intersections with cellular boundaries on each gridline were counted, as shown in Figure 6, and averaged over 5 gridlines for each micrograph. Cell size was then estimated
using Equation 2, averaging cell size values from 6 micrographs to obtain approximate cell size per sample. This data was then utilized to calculate an estimated cooling rate with Equation 3 [13] by applying cell size in place of secondary dendrite arm space \((d)\). The remaining variables are \(\varepsilon\) for cooling rate and constants determined by the material; for nickel-based alloys, \(a \approx 50 \, \mu m\) and \(b = 1/3\) [13].

\[
\text{Cell Size} = \frac{\text{Total Length of Line}}{\text{Total Number of Intercepts}} \quad (2)
\]

\[
d = a \varepsilon^{-b} \quad (3)
\]

Figure 6. Representative SEM BSE image with overlay of lines utilized to count intersections between cellular gridlines as indicated by the red points.

To evaluate phase constituents and texture in the grains, X-Ray diffraction (XRD) was performed on the samples using PANalytical Empyrean Basic diffraction system with 1.8 kW Cu X-ray tube operating at 45 keV voltage and 40 mA current. A Cu-\(\alpha\) radiation source with a wavelength of 1.54 Å was used. XRD pattern was collected with Bragg-Brentano diffractometer
geometry in the 2θ range of 30° – 120° with a step size 0.033° within 2θ range of 30° to 120° and dwell time resulting in a minimum of 10,000 counts at the highest intensity peak.
CHAPTER 4: RESULTS

4.1 Powder Feedstock Analysis

The acquired IN625 powders were analyzed in the as-received condition prior to printing the samples for study. Determining size distribution was the first step in characterizing the powder feedstock, measurement results are presented in Table 2 and graphically shown in Figure 7. The 33.24 μm mean particle size, with 90% of the distribution below 46.42 μm, is within the typical 10 – 60 μm particle size range [1] for laser PBF technology.

Table 2. IN625 powder size data.

<table>
<thead>
<tr>
<th>Mean Particle Size (µm)</th>
<th>Standard Deviation of Particle Size (µm)</th>
<th>D10 (µm)</th>
<th>D90 (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>33.24</td>
<td>9.817</td>
<td>21.12</td>
<td>46.42</td>
</tr>
</tbody>
</table>

Figure 7. Plot of IN625 powder size distribution.
Chemical composition was afterwards evaluated with XEDS from cross-sectioned samples, for both individual powder particles and an aggregate group. Results of measured composition are listed in Table 3 next to the supplier certificate data for the powder and ASTM F3056 – 14 acceptable compositional ranges. It is likely that the differences in measured composition with respect to the material certificate are due to the measurement technique, inductively coupled plasma mass spectrometry (ICP-MS) utilized by the supplier, and interference from mounting material in XEDS. Overall, elemental composition percentages obtained with XEDS are comparable to those of the supplier certificate and also lie within or near the ASTM-defined ranges.

Table 3. IN625 powder particle composition data.

<table>
<thead>
<tr>
<th>Element</th>
<th>ASTM F3056 – 14</th>
<th>Supplier Certificate</th>
<th>XEDS Powder Units N ≥ 5</th>
<th>XEDS Powder Aggregate</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni (wt%)</td>
<td>remainder</td>
<td>remainder</td>
<td>58.91 ± 0.36</td>
<td>55.97</td>
</tr>
<tr>
<td>Cr (wt%)</td>
<td>20.0 – 23.0</td>
<td>20 – 23</td>
<td>21.28 ± 0.14</td>
<td>20.79</td>
</tr>
<tr>
<td>Mo (wt%)</td>
<td>8.0 – 10.0</td>
<td>8 – 10</td>
<td>7.77 ± 0.16</td>
<td>9.01</td>
</tr>
<tr>
<td>Nb (wt%)</td>
<td>3.15 – 4.15</td>
<td>3.15 – 4.15</td>
<td>7.57 ± 0.12</td>
<td>5.97</td>
</tr>
<tr>
<td>Fe (wt%)</td>
<td>0 – 5.00</td>
<td>5.0</td>
<td>4.13 ± 0.22</td>
<td>4.01</td>
</tr>
<tr>
<td>Co (wt%)</td>
<td>0 – 1.00</td>
<td>1.0</td>
<td>0.05 ± 0.05</td>
<td>0</td>
</tr>
<tr>
<td>Si (wt%)</td>
<td>0 – 0.50</td>
<td>0.50</td>
<td>0.12 ± 0.03</td>
<td>0.96</td>
</tr>
<tr>
<td>Mn (wt%)</td>
<td>0 – 0.50</td>
<td>0.50</td>
<td>0.002 ± 0.004</td>
<td>0</td>
</tr>
<tr>
<td>Ti (wt%)</td>
<td>0 – 0.40</td>
<td>0.40</td>
<td>0.02 ± 0.02</td>
<td>0.02</td>
</tr>
<tr>
<td>Al (wt%)</td>
<td>0 – 0.40</td>
<td>0.40</td>
<td>0.04 ± 0.04</td>
<td>0.58</td>
</tr>
<tr>
<td>C (wt%)</td>
<td>0 – 0.10</td>
<td>0.10</td>
<td>not included</td>
<td>not included</td>
</tr>
<tr>
<td>S (wt%)</td>
<td>0 – 0.015</td>
<td>0.015</td>
<td>0</td>
<td>1.99</td>
</tr>
<tr>
<td>P (wt%)</td>
<td>0 – 0.015</td>
<td>0.015</td>
<td>0.16 ± 0.04</td>
<td>0.69</td>
</tr>
</tbody>
</table>

4.2 Evaluation of Defects

Common defects with adverse effects in mechanical properties of AM-produced parts are porosity and lack of fusion flaws [1]. Laser power and scan speed, as the main determinants of
energy transferred to the powder, were varied systematically in the fabrication of the IN625 samples to evaluate the isolated impact of the change on print outcome. The manufacturer-recommended 0.12 mm hatch spacing, 0.03 mm layer thickness, and 16-degree scan rotation were deemed acceptable for the evaluated power and scan speeds, thus kept constant for all samples.

Optical micrographs from the XY and XZ plane cross-sections of the samples were taken to visually analyze porosity with respect to changing SLM parameters; this variation can be observed through a compilation of the micrographs in Figure 8. Samples printed at lower scan speeds, hence greater energy densities, display circular porosity which is generally attributed to entrapped gases. On the other end of the spectrum, at higher scan speeds and lower energy densities, the observed flaws can be attributed to insufficient melting due to its more irregular shape. Between the two extremes there is a region of highly dense samples observable for all four of the laser powers. In terms of porosity, there is a smaller range of scan speeds for optimal processing of IN625 at 125W laser power than for the higher laser powers investigated.
Figure 8. Compilation of optical micrographs from XY and XZ planes for all samples organized with respect to laser power and scan speed. Optimal processing window indicates most dense samples for every laser power investigated (constant 120 µm hatch spacing, 30 µm layer thickness, and 16-degree scan rotation).
Relative density was also quantitatively evaluated through image analysis of the porosity percentage in the micrographs and Archimedes density measurements. As shown by the graphs in Figure 9, there is consistency in the average sample density values obtained with both methods. The trend visually identified with the micrographs is also present in the density vs scan speed plots, greater density is obtained with the intermediate range of scan speeds evaluated. It can also be observed that the impact of scan speed on part density percentage is less for the higher laser powers evaluated, showing least variation for 275W samples.

![Density vs Scan Speed Graphs](image)

Figure 9. Density plotted as a function of scan speed for various laser powers employed.

Plotting density as a function of energy density as exhibited in Figure 10 allowed identification of normalized trends. For all evaluated laser powers, there is one point of inflection
in the data which corresponds to the highest part density. Peak density (>99.6%) for the samples in this study corresponds to energy density values in the range of 55 – 69 J/mm$^3$ with the exception of one outlying 99.9% density sample at 97 J/mm$^3$ for 350W laser power.

**Archimedes’ Method Relative Density**

![Graph showing Archimedes' Method Relative Density vs Energy Density](image)

Figure 10. Average density values determined from Archimedes’ method plotted as function of energy density for various laser powers employed.

### 4.3 Dimensional Analysis of Melt Pool

Morphology of the melt pool provides insights into the mode of melting achieved by the different printing parameters. Optical micrographs of the etched longitudinal (XZ) plane were utilized to analyze melt pool depth and width for all samples. Representative images of melt pool development with respect to scan speed are exhibited in Figure 11. The deep melt pools in Figure
11a are indicative of keyhole mode and some characteristic nearly spherical pores of this melting mode are observable. A depth much greater than the half-width of the melt pool is also noticeable in keyhole mode cases. Micrographs in Figure 11b, Figure 11c, and Figure 11d display conduction mode of melting while Figure 11d also shows initial stages of lack of fusion flaws. Plots of melt pool depth and width dimensions as function of scan speed are reported in Figure 12. The general trend is that, for the same laser power, both depth and width dimensions of the melt pool reduce with increasing scan speeds. It is also noticeable that the rate of change in melt pool size is greater for slower scan speeds, corresponding to an energy density of approximately 80 J/mm³ and higher within the evaluated ranges.

Figure 11. Optical micrographs of melt pools for samples built with 200W laser power and (a) 400 mm/s, (b) 800 mm/s, (c) 1200 mm/s, (d) 1600 mm/s.
To estimate the melt pool sizes with respect to printing parameters analytically, the three-dimensional Rosenthal’s equation [18] for heat flow during welding was utilized:

\[
\frac{2\pi(T-T_0)kR}{Q} = \exp\left[\frac{-\nu(R-x)}{2\alpha}\right]
\]  

(4)

where \( T \) is final temperature, \( k \) is thermal conductivity, \( \nu \) is laser scan speed, and \( \alpha \) is thermal diffusivity. \( T_0 \) is workpiece temperature and for this application the 100 °C base plate heating temperature was utilized. \( Q \) is heat transferred from the heat source, determined by the laser power.
multiplied by the absorptivity of the material. The absorptivity for IN625 was taken as a constant 0.57 per the effective absorptivity determined in Montgomery’s work [19]. Variable \( R \) represents radial distance from the origin and is given by \( (x^2 + y^2 + z^2)^{1/2} \) where, for this purpose, \( x \) is length of the melt pool along laser travel direction, \( y \) is melt pool width, and \( z \) is melt pool depth. In addition to the assumptions intrinsic to Rosenthal’s equation, the width was taken to be twice the depth (\( y = 2z \)). Temperature gradient graphs were generated in Matlab and the depth was taken at the maximum boundary to the solidus temperature (1513 K). Thermal diffusivity was calculated with a specific heat value of 650 J/kgK corresponding to the solidus condition. Analytical results obtained are plotted in Figure 12 along with the experimental data to display correlation. Despite the simplifying assumptions taken for the analytic model, obtained values follow the data trend and approach the measured sample melt pools; particularly for the faster scan speeds.

4.4 Cellular Spacing and Cooling Rate Calculation

BSE micrographs of the transverse (XY) plane allow quantification of cell size due to segregation of elements. IN625 dendrite cores appear darker than the inter-dendritic regions which are enriched in Nb and Mo, elements with relatively higher atomic number. Change in cell size with respect to scanning speed can be perceived in the micrographs on Figure 13, all corresponding to the same laser power. It is worth noting on Figure 13c that, even within a sample, there is variability in the cell size. This is presumably due to changes in cooling rate over the depth of the melt pool in combination with the selected laser pattern affecting cooling rate across the sample. Plots of average cell size determined from the samples against scan speed are shown in Figure 14, including trendlines. There is an overall tendency for smaller cell size with increasing scanning
speed, however, variations within the samples result in broader size ranges that lessen the correlation.

Figure 13. SEM images of cellular structure for samples built with 125W laser power at (a) 200 mm/s, (b) 400 mm/s, (c) 600 mm/s, (d) 800 mm/s.
Figure 14. Cell size plotted as function of laser scan speed for various laser powers employed.

Since the cell size is dependent on cooling rate, it serves as basis from which to estimate how quickly the AM samples dissipate heat with respect to changes in print parameters. Sample cooling rates were calculated with Equation 3 utilizing the measured cell sizes in place of secondary dendrite arm spacing (SDAS). In addition, cooling rate along the build direction ($x$) was estimated on the basis of Equation 4 with the simplifying assumption that melt pool width ($y$) and depth ($z$) equal zero. The resulting expression for cooling rate calculation is [18]:

$$
\left( \frac{\partial T}{\partial t} \right)_x = \left( \frac{\partial T}{\partial x} \right)_y \left( \frac{\partial x}{\partial t} \right)_z = -2\pi k v \frac{(T - T_0)^2}{Q} \tag{5}
$$
where T and corresponding k values are listed in Table 4. Cooling rates were calculated for both the solidus and liquidus states, utilizing ambient condition thermal conductivity for the solidus temperature boundary. Results of SDAS and Rosenthal cooling rate estimations are listed in Table 5 and plotted with respect to scan speed in Figure 15. Overall trend observed is that cooling rate increases with the faster scan speeds corresponding to shallower, conduction mode, melt pools. Rosenthal calculation with solidus parameters approximated more accurately the cooling rates obtained from cellular spacing in the samples (SDAS).

Table 4. IN625 properties used in cooling rate calculation [20].

<table>
<thead>
<tr>
<th>Condition</th>
<th>Temperature, T</th>
<th>Thermal conductivity, k</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ambient</td>
<td>298 K</td>
<td>11 W/mK</td>
</tr>
<tr>
<td>Solidus</td>
<td>1513 K</td>
<td>30 W/mK</td>
</tr>
<tr>
<td>Liquidus</td>
<td>1607 K</td>
<td>30 W/mK</td>
</tr>
</tbody>
</table>

Table 5. Results of cooling rate calculations for each sample.

<table>
<thead>
<tr>
<th>Laser Power (W)</th>
<th>Scan Speed (mm/s)</th>
<th>SDAS (x10^5 K/s)</th>
<th>Rosenthal Solidus (x10^5 K/s)</th>
<th>Rosenthal Liquidus (x10^5 K/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>125</td>
<td>200</td>
<td>3.676 ± 1.542</td>
<td>2.521</td>
<td>8.057</td>
</tr>
<tr>
<td></td>
<td>400</td>
<td>3.196 ± 1.793</td>
<td>5.043</td>
<td>16.114</td>
</tr>
<tr>
<td></td>
<td>600</td>
<td>2.627 ± 0.648</td>
<td>7.564</td>
<td>24.171</td>
</tr>
<tr>
<td></td>
<td>800</td>
<td>5.659 ± 1.76</td>
<td>10.085</td>
<td>32.228</td>
</tr>
<tr>
<td>200</td>
<td>200</td>
<td>3.408 ± 2.434</td>
<td>1.576</td>
<td>50.357</td>
</tr>
<tr>
<td></td>
<td>400</td>
<td>3.696 ± 2.399</td>
<td>3.152</td>
<td>10.071</td>
</tr>
<tr>
<td></td>
<td>600</td>
<td>6.311 ± 3.351</td>
<td>4.727</td>
<td>15.107</td>
</tr>
<tr>
<td></td>
<td>800</td>
<td>4.361 ± 2.132</td>
<td>6.303</td>
<td>20.143</td>
</tr>
<tr>
<td></td>
<td>1000</td>
<td>7.522 ± 3.806</td>
<td>7.879</td>
<td>25.178</td>
</tr>
<tr>
<td></td>
<td>1200</td>
<td>10.519 ± 3.966</td>
<td>9.455</td>
<td>30.214</td>
</tr>
<tr>
<td></td>
<td>1400</td>
<td>11.877 ± 4.793</td>
<td>11.031</td>
<td>35.250</td>
</tr>
<tr>
<td></td>
<td>1600</td>
<td>8.318 ± 4.178</td>
<td>12.607</td>
<td>40.285</td>
</tr>
<tr>
<td></td>
<td>1800</td>
<td>6.258 ± 2.733</td>
<td>14.182</td>
<td>45.321</td>
</tr>
<tr>
<td>Laser Power (W)</td>
<td>Scan Speed (mm/s)</td>
<td>SDAS (x10^5 K/s)</td>
<td>Rosenthal Solidus (x10^5 K/s)</td>
<td>Rosenthal Liquidus (x10^5 K/s)</td>
</tr>
<tr>
<td>----------------</td>
<td>-------------------</td>
<td>------------------</td>
<td>-------------------------------</td>
<td>-------------------------------</td>
</tr>
<tr>
<td>275</td>
<td>400</td>
<td>1.681 ± 0.898</td>
<td>2.292</td>
<td>7.325</td>
</tr>
<tr>
<td></td>
<td>600</td>
<td>2.966 ± 1.258</td>
<td>3.438</td>
<td>10.987</td>
</tr>
<tr>
<td></td>
<td>800</td>
<td>2.364 ± 1.393</td>
<td>4.584</td>
<td>14.649</td>
</tr>
<tr>
<td></td>
<td>1000</td>
<td>6.344 ± 2.597</td>
<td>5.730</td>
<td>18.311</td>
</tr>
<tr>
<td></td>
<td>1200</td>
<td>6.651 ± 3.182</td>
<td>6.876</td>
<td>21.974</td>
</tr>
<tr>
<td></td>
<td>1400</td>
<td>7.037 ± 2.938</td>
<td>8.022</td>
<td>25.636</td>
</tr>
<tr>
<td></td>
<td>1600</td>
<td>5.391 ± 2.308</td>
<td>9.168</td>
<td>29.298</td>
</tr>
<tr>
<td></td>
<td>1800</td>
<td>6.828 ± 2.363</td>
<td>10.314</td>
<td>32.961</td>
</tr>
<tr>
<td></td>
<td>2000</td>
<td>5.342 ± 3.038</td>
<td>11.461</td>
<td>36.623</td>
</tr>
<tr>
<td>350</td>
<td>600</td>
<td>3.047 ± 1.396</td>
<td>2.701</td>
<td>8.633</td>
</tr>
<tr>
<td></td>
<td>800</td>
<td>6.175 ± 2.248</td>
<td>3.602</td>
<td>11.510</td>
</tr>
<tr>
<td></td>
<td>1000</td>
<td>6.282 ± 0.474</td>
<td>4.502</td>
<td>14.388</td>
</tr>
<tr>
<td></td>
<td>1200</td>
<td>5.138 ± 0.859</td>
<td>5.403</td>
<td>17.265</td>
</tr>
<tr>
<td></td>
<td>1400</td>
<td>6.222 ± 1.374</td>
<td>6.303</td>
<td>20.143</td>
</tr>
<tr>
<td></td>
<td>1600</td>
<td>6.095 ± 0.907</td>
<td>7.204</td>
<td>23.020</td>
</tr>
<tr>
<td></td>
<td>1800</td>
<td>3.409 ± 0.713</td>
<td>8.104</td>
<td>25.898</td>
</tr>
<tr>
<td></td>
<td>2000</td>
<td>4.829 ± 1.489</td>
<td>9.005</td>
<td>28.775</td>
</tr>
<tr>
<td></td>
<td>2200</td>
<td>4.763 ± 3.367</td>
<td>9.905</td>
<td>31.653</td>
</tr>
</tbody>
</table>
Figure 15. Cooling rate plotted as function of laser scan speed for various laser powers employed.
CHAPTER 5: DISCUSSION

5.1 Microstructural Analysis

Evaluation of changes in scan speed for different laser powers, holding other SLM print variables constant, indicates a significant impact on outcomes. Dense samples were obtained over a range of laser powers and scan speeds, indicated by the optimal processing window in Figure 8. Hence, there is some flexibility in the selection of these parameters with respect to the common AM defect that is porosity; provided hatch spacing and layer thickness are selected appropriately to allow for subsequent melt pools to overlap [5]. Energy density, a value more heavily impacted by laser power and scan speed (see Equation 1), could serve as a guide for parameter selection given the correlation observed with sample density measurements. Principal phenomena driving this correlation are the melting mode and dynamics occurring upon laser-material interaction. High energy density results in keyhole melt mode; keyholes can become unstable and collapse, thus trapping vapor in the structure [1]. Too low energy density led to voids due to insufficient melting while an intermediate energy density, associated with conduction melt mode, yielded the best results. Furthermore, laser beam interaction with the surface material causes rapid vaporization of the melt surface which results in recoil pressure together with Marangoni and other hydrodynamic effects [21]. High-power and high-speed laser conditions provoke a larger volume of both displaced solid particles and ejected liquid metal, thus increasing the likelihood of morphological defects in the build [7]. These melt pool dynamics can cause solid particles to fly back into the melt pool and promote solidification, resulting in irregularities due to melt pool break-up through impingement of the liquid flow [7].
Un-melted powder particles were observed in several of the samples. This is commonly the case for fabrication with high speeds, i.e. low energy density, wherein there is often partial melting. Figure 16a depicts this phenomenon in which un-melted powder particles are found in an area with irregular porosity associated to lack of fusion flaws. On the other hand, the un-melted powder particle in Figure 16b was found in a sample fabricated with a relatively high energy density. Although the print parameters for that particular sample resulted in a significant amount of gas trap porosity, the un-melted powder does not lie adjacent to a void. A possible explanation for the finding in Figure 16b is for the solid powder particle to have flown backwards into the melt pool. The velocity field of the gas flow generated from laser-material interaction is stronger in the backward direction, thus smaller and lighter particles are generally ejected forwards while most of the material is pushed towards the tail of the melt pool [7]. The small cells immediately surrounding the un-melted powder in Figure 16b suggest that it served as nucleation site and heat sink, thus promoting faster solidification. It is worth noting that the un-melted powder in Figure 16b has a diameter roughly twice that of the powder particles visible in Figure 16a.

Figure 16. SEM images of un-melted powder particles observed in samples built with (a) 350W laser power at 2000 mm/s, and (b) 200W laser power at 200 mm/s.
It has been widely observed that the thermal gradient associated with layer-by-layer material deposition leads to grain elongation, however, Kreitberg et al. [12] suggested this also leads to anisotropy in mechanical properties for IN625. XRD was utilized to evaluate presence of a preferred grain growth orientation that could lead to macroscopic anisotropy, obtained data is presented in Figure 17 along with Nickel powder diffraction file for comparison. The minimal texture observed for both transverse (XY) and longitudinal (XZ) planes indicate isotropic mechanical properties can be expected for that sample.

Figure 17. XRD patterns for (left) IN625 sample printed with 275W laser power, 1600mm/s scan speed, 0.12 mm hatch spacing, 0.03 mm layer thickness, and 16° scan rotation, (right) pure Nickel.

5.2 Theoretical Approximations

Given the similar nature with SLM process, concepts and models from welding metallurgy were utilized to approximate cooling rates. Although there is more thermal cycling occurring in a SLM build, localized heat flow into the material and out of the melt pool compares with that in welding. Morphology and size of the solidification microstructure are determined by the relationships between temperature gradient ($G$) and speed of solid–liquid boundary progression, referred to as growth rate ($R$) [18]. The solidification pattern observed through microstructural
analysis of the samples was consistently cellular in nature, but displaying noticeable variability in size as depicted by the error bars in Figure 14. Ghosh et al. [21] identified a correlation between melt pool dimensions and variability of $G$ and $R$ values, also indicating that differences in microstructural morphology can be expected within a melt pool. The growth rate ($\varepsilon$) determined per the following relation [18]:

$$\varepsilon = GR$$

provides indication of the relative size, specifically coarseness, of the solidification structure [18]. The model presented in Equation 3 is based on SDAS, however, the cooling rate associated with SLM does not yield such solidification structure and therefore the cell size was utilized as closest approximation. Despite this assumption, calculated cooling rates in the range $1.7 \times 10^5 - 1.2 \times 10^6$ K/s are in accordance with the high cooling rates (in the order of $10^4 - 10^6$) associated with SLM manufacturing [2]. Also, elemental segregation encountered in the samples is consistent with constitutional supercooling in a system pushed far from equilibrium behavior.

Rosenthal’s model for heat flow in welding (Equation 5) served as another approximation to determine cooling rate of the samples. Calculation with solidus parameters yielded a $1.6 \times 10^5 - 1.4 \times 10^6$ K/s range, overall closer to the SDAS cooling rates than the $5.0 \times 10^5 - 4.5 \times 10^6$ K/s obtained with liquidus parameters. Furthermore, Rosenthal models using the 0.57 average effective absorptivity calculated by Montgomery et al. [19] approached calculated SDAS cooling rates better than the 0.5 utilized in the Ghosh et al. [21] simulation. Even the consistently higher liquidus cooling rates, however, are generally lower than the $3 \times 10^6 - 3 \times 10^7$ range obtained by Ghosh et al. [21] through finite element analysis. All models are limited in some form due to the underlying assumptions that enable calculation, but laser absorptivity is typically mentioned as a key factor impacting results [7, 19-21]. For example, Ridolfi et al. [20] used effective thermal conductivity
and laser absorptivity as fitting parameters for the model, ultimately proposing a variable absorptivity based on laser specific energy (J/mm). The laser specific energy is determined dividing laser power by scan speed. Proposed IN625 variable absorptivity values were tested in the Rosenthal solidus and liquidus calculations and graphically compared to results obtained with constant absorptivity; the plots in Figure 18 reveal that 0.57 constant absorptivity yielded an overall better approximation to the cooling rates obtained from SDAS. This discrepancy could be attributed to the constant thermal conductivity values utilized in the Rosenthal model compared to the variable effective thermal conductivity employed by Ridolfi et al. [20] as fitting factor, suggesting interplay between the two parameters. Incorporating dependence of thermal conductivity on temperature along with variable absorptivity in the Rosenthal model calculations could yield a better approximation.

![Figure 18](image)

**Figure 18.** Plots of calculated cooling rate for samples printed with 200W laser power. SDAS results compared to Rosenthal model with (left) constant 0.57 absorptivity, (right) absorptivity between 0.36 and 0.96 increasing linearly between 0.2 and 0.5 laser specific energy (J/mm) [20].

Melt pool depth and width were also calculated from the Rosenthal equation to evaluate its accuracy in predicting dimensions. As can be seen in Figure 12, the model yielded results closest to the experimentally measured melt pools for the higher scan speeds, i.e. lower energy density
condition. Considering the difference in aspect ratio observed for melt pools exhibiting keyhole melt mode compared to conduction mode, the width to depth assumption in the model \( y = 2z \) is likely a significant factor responsible for the deviation at low scan speeds. Finite element analysis simulations by Ghosh et al. [21] also had a general under-prediction of melt pool depth and width, accompanied by incorrect melt pool shape prediction for keyholing condition. The authors attributed this deviation to an increase in complexity of melt pool dynamics since the associated surface tension and recoil pressure were not considered in the simulation [21].
CHAPTER 6: CONCLUSIONS

A range of laser power and scan speeds were utilized to fabricate cubic IN625 samples to evaluate the isolated impact of these parameter variations on SLM-built components. A certain laser power may be preferred, depending on the application or available resources, and results indicate that the scan speed can be adjusted accordingly to achieve satisfactory print outcomes. In the evaluated 125W to 300W range, at least one sample with relative density above 99.7% was obtained for each laser power. However, the optimal processing window for dense parts is wider for laser power equal to or greater than 200W. Additionally, highest density samples were correlated to energy densities in the 55 – 69 J/mm³ range.

Microstructural analysis revealed a cellular solidification pattern and trend of reduction in size with increasing laser scan speed, i.e. decreasing energy density. Melt pool geometry followed the same trend and allowed visualization of transition from keyhole melt mode to conduction mode with decreasing energy density. The deeper melt pools associated with keyhole mode displayed significant spherical porosity due to vapor entrapment. Conduction mode resulted in higher density samples and melt pools were characterized by a depth approximately equal to half the width. For excessively high scan speeds, however, insufficient melting led to formation of irregular voids between the conduction mode melt pools.

Melt pool dimensions and cooling rates were calculated utilizing the Rosenthal models from welding metallurgy to approximate experimental data. The baseline for cooling rates was calculated from measured sample cell size applied in a SDAS model considering material constants. Both approximation methods yielded consistent cooling rate values in the order of $10^5 – 10^6$ K/s which are in agreement with published IN625 studies. These analytical models produce
more realistic results for samples displaying conduction melt mode, but do not properly predict behavior under keyhole mode. More complex models considering melt pool dynamics such as surface tension and recoil pressure are recommended for more accurate approximations.
CHAPTER 7: FUTURE WORK

This thesis served to identify optimal processing parameters with regards to part density, however, mechanical properties remain to be validated. Hardness and tensile strength associated with the high-density samples is required to validate properties and mechanical integrity of printed components. In addition, testing for isotropy of these properties to verify agreement with texture analysis. Furthermore, evaluation of correlation between microstructure and mechanical properties would provide valuable insight regarding dependence on printing parameters. Lastly, heat treatment cycles warrant investigation since high residual stresses and localized compositional variations are associated with IN625 processed through SLM.
REFERENCES


