Decoupling part geometry from microstructure in directed energy deposition technology : towards reliable 3D printing of metallic components

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DECOUPLING PART GEOMETRY FROM
MICROSTRUCTURE IN DIRECTED ENERGY
DEPOSITION TECHNOLOGY:
TOWARDS RELIABLE 3D PRINTING OF METALLIC
COMPONENTS

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ADDITIVE MICROSTRUCTURE ENGINEERING LABORATORY
SINGAPORE CENTRE FOR 3D PRINTING
SCHOOL OF MECHANICAL AND AEROSPACE ENGINEERING

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TOWARDS RELIABLE 3D PRINTING OF METALLIC
COMPONENTS

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School of Mechanical and Aerospace Engineering

A thesis submitted to the Nanyang Technological University
in partial fulfilment of the requirement for the degree of
Doctor of Philosophy

Statement of Originality

I hereby certify that the work embodied in this thesis is the result of original research, is free of plagiarised materials, and has not been submitted for a higher degree to any other University or Institution.

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YEOH YONG CHEN

Supervisor Declaration Statement

I have reviewed the content and presentation style of this thesis and declare it is free of plagiarism and of sufficient grammatical clarity to be examined. To the best of my knowledge, the research and writing are those of candidate except as acknowledged in the Author Attribution Statement. I confirm that the investigations were conducted in accord with the ethics policies and integrity standards of Nanyang Technological University and that the research data are presented honestly and without prejudice.

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Prof Matteo Seita

Authorship Attribution Statement

The thesis contains materials from one paper published in the following peer-reviewed journal in which I am listed as an author.

Chapter 5 is published on peer review journal *Alloys and compounds* as Yeoh Yong Chen, Guido Macchi, Ekta Jain, Bernard Gaskey, Sudharshan Raman, Grace Tay, Davide Verdi, Alin Patran, Antonio Mattia Grande, Matteo Seita “Multiscale microstructural heterogeneity and mechanical property scatter in Inconel 718 produced by directed energy deposition,”

The contribution of the authors are as follows:

- Prof Matteo Seita and Prof Antonio Mattia Grande provided the initial project direction and edited the manuscript drafts.
- I prepared the manuscript drafts together with Dr Bernard Gaskey and Guido Macchi.
- I co-designed the study with Guido Macchi and performed all the laboratory work at the NTU School of Material Science and School of Mechanical Engineering.
- All microscopy and sample preparation were conducted by Guido Macchi, Ekta Jain and me in NTU School of Material Science.
- All samples production were conducted by me, with the help from Grace Tay, Dr Davide Verdi, and Dr Alin Patran in SC3DP and ARTC
- Sudharshan Raman assisted in validation of experimental data.

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YEOH YONG CHEN

Abstract

Directed energy deposition (DED) is an additive manufacturing technique that enables rapid production and repair of metallic parts with flexible geometry. However, the complex nature of thermal and material transport during DED can yield unwanted microstructure heterogeneity, which causes scatter in parts performance and hinders the adoption of DED technology by the industry. This heterogeneity can be exacerbated by the size and geometry of the build, as well as the deposition strategy employed. In this thesis we aim at unveiling these complex process-structure-property relationships to enable the production of DED parts with controlled microstructure and consistent properties.

The first part (see chapter 4) of this thesis focuses on developing a set of DED process parameters for the deposition of I718. Using a design of experiment approach, we investigate what parameters yield full-density builds and how the build quality changes as a function of the geometry of the printed specimens. We find that the laser power is the most important parameter to control the size of bead, while the powder feeding rate is critical for bead height. Using this knowledge, we produce a series of specimens that we use in the second part of the thesis to investigate the resulting microstructure.

In the second part (see chapter 5 and 6), we investigate microstructure variations at different length scales in the produced specimens. We quantify spatial trends in grain structure, texture, composition, and solidification structure within parts and correlate them with variations in hardness, yield strength, and Young's Modulus to highlight the effect of the thermal environment during solidification. We find that the high energy input employed when using high deposition rates is conducive to significant microstructure heterogeneity along both the build and transversal directions, which stems from the asymmetric cooling rates generated by the deposition strategy used.

Using these findings, in the third part (see chapter 7) of the thesis we demonstrate the ability to produce the desired microstructures through careful selection of process parameters. High thermal input results in strong $\langle 100 \rangle$ texture along building direction and enhances micro-segregation during solidification, which results in formation of different phases across the build. Conversely, processes with low heat input are conducive to a reduced thermal built up and a more homogeneous microstructure. To further demonstrate the effect of thermal history on printed part microstructure, we study the effect of sample size using different set of process parameters. We find variation in dendrite spacing, grain size and grain orientation on parts with different geometry, despite the same process parameter employed. We also find that as-printed elemental segregation and distribution of secondary phase will directly influence heat-treated microstructure.

In summary, the complex process-structure-property relationships in DED processes result in microstructure heterogeneity of the printed parts. This thesis provides insights into the root causes for the observed heterogeneity as well as possible approaches how to control it to achieve consistent builds.

As such, these results have important implications and can serve as a useful reference for the development of process parameters with optimum build rate and desired microstructure.

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1 General Introduction

1.1 Background

Additive manufacturing (AM) entails a variety of joining processes to produce objects or parts from 3D model data, usually in layer-by-layer fashion [1]. In that, AM is distinctly different from subtractive manufacturing methodologies, which instead are used to selectively remove material from an ingot. Owing to the layer-wise nature of the process, AM enables the production of near-net-shape parts with complex geometry, which are typically not achievable via conventional subtractive technologies. In general, AM can be classified into seven different categories based on ASTM F27920-12a standard, namely binder jetting, directed energy deposition (DED), material extrusion process, material jetting process, powder bed fusion (PBF), sheet lamination and vat photo polymerization [2].

This thesis focuses on DED processes, which are often employed to fabricate large metal parts and exhibit significantly higher throughput compared to other AM techniques. In addition, because DED closely resembles welding, it is most suited for re-manufacturing of parts (e.g., repair and maintenance). This technology has one of the highest deposition rates among different AM processes, making it suitable for building bulky parts for marine and aerospace applications.

However, in order to realize the advantages offered by this technology, there are a few challenges that need to be overcome. One of those is the variability in mechanical properties due to the microstructure heterogeneity found on DED parts. This heterogeneity is due to the variability in the thermal history the material undergoes during processing, which dictates the resulting microstructure in the final part. The thermal history governs the solidification rate and the cooling rate of the material, which are functions of the processing conditions used as well as the geometry of the part produced. Because of the challenges associated with predicting such a microstructure heterogeneity, DED parts exhibit a large scatter in mechanical properties which casts uncertainty over part performance and lifetime. Because of this uncertainty, the adoption of DED processes in the industry is currently limited.

1.2 The microstructure of parts produced by DED is geometry-dependent

The microstructure of metals—namely the ensemble of crystal grains composing a polycrystalline material, their size, shape, crystallographic orientation, as well as the defects contained—is the key influencing factor to the material's mechanical properties [3], including strength, toughness, ductility, hardness, corrosion resistance, high/low temperature behavior or wear resistance [4]. Porosity refers to the level of solidity achieved in fabricated metal part, which has a significance effect on the part's fatigue performance [5]. The lack of fusion defects or porosity is a common AM defect formed due to un-optimized process parameter [5, 6]. While looking at the grain morphology, there is an inverse relationship between grain size and delta yield strength as per Hall-Petch relationship [9]. Material properties such as strength and stress corrosion cracking resistance can be highly dependent on material's crystallographic texture—namely the grain orientation distribution [3]. On the other hand, phase arrangement is responsible for the control of mechanical and thermo-physical properties [10].

Microstructure in AM

A common microstructure feature observed in metal AM parts is the epitaxial columnar grain morphology. The orientation-dependence of certain mechanical properties in AM materials has been attributed to such columnar grains that are aligned parallel to the build direction [11], [12]. Given the steep thermal gradient along build direction, AM fabricated parts generally exhibit a preferential $\langle 100 \rangle$ crystallographic texture [14]. The reason is that grains with $\langle 100 \rangle$ orientation have highest solidification velocity compared to other crystallographic orientations and follow the vertical heat flow direction.

Metal AM parts usually undergo complex cyclic thermal history consist of directional heat extraction, repeated melting and rapid solidification, which yield microstructures that intrinsically different from those found in metallic parts produced via conventional manufacturing methods [11]. Similar to other type of manufacturing technology, AM has a list of processing condition/parameter to be fine-tuned and controlled in accordance to the process requirement. Example of key processing parameters in DED processes are scanning speed, laser power, hatching distance, mass flow rate and standoff distance [1], [12]. Specifically, the layer height in DED process, or the thickness of each layer, is usually empirically determined based on key process parameter such as laser power and powder flow rate. There are on-going

research efforts to understand the effect of combination and respective parameter towards the final part microstructure formation. The details on respective effects of these parameters on the microstructure of printed parts are discussed in section 2.1 accordingly. It is reported that there is a difference in microstructure across component with different geometry, even though the process conditions are kept constant [14], [15]. Most of the starting parameters developed by OEM are optimized to achieve full dense parts with little or no porosity. A common approach is to have a fixed ratio of power to scanning speed to ensure constant amount of energy input during the printing process. In other words, the main criterion in process parameters selection has been to achieve a certain energy density, which is known to yield low porosity builds. However, the microstructure of parts (dendrite spacing, texture, phases etc.) fabricated by DED is not a direct function of the energy density. Rather, it depends on the thermal history of the material, such as the cooling and solidification rates.

While the benefit of AM has been widely discussed in many studies, the main hurdle for wide adoption of this technology is due to microstructure heterogeneity [4],[5],[11]. The term heterogeneity is defined as non-uniformity in a part microstructural feature distribution. In conventional practice, superior and consistent mechanical properties of metal AM parts are prerequisites for engineering application. The microstructure heterogeneity will result in uncertainty of material properties during servicing life of application, which limits the application of AM technology. As an attempt to resolve the issue, the study of microstructure heterogeneities in DED process has been conducted. Several studies are working on measuring the local temperature to compute localized cooling rate and solidification rate. However, the in-situ measuring process is challenging as the high processing temperature of alloy has impose a challenge in getting real time temperature profile.

In summary, the current challenges in DED technology are listed as following:

- (i) There is a lack of understanding in how the solidification microstructure evolves during DED processes, and how it may be controlled by changing processing conditions.
- (ii) Limited study in the effect of part geometry on microstructure formation. As a result, the knowledge of attainable microstructure on component with different geometry is restricted.

1.3 Research objectives and methods

Given the challenges described in the forgoing, the main research objective is to **propose a characterization methodology to quantify the thermal history of a DED printed part, and the resulting microstructure. To begin with, we try to understand the root causes of the measured microstructure heterogeneity, in particularly the effect of the deposition rate and part geometry. We then work on estimating the effects of thermal energy and thermal history as a function of processing conditions. This work opens the possibility to produce parts with uniform microstructure and thus consistent mechanical properties, which will be the main contribution of this research.**

As an attempt to accomplish the mentioned objectives, the research approach includes:

- i. Investigating the processing parameter space for DED of Inconel 718 (I718). A matrix of samples printed under different conditions will be characterized to assess their microstructure. The results will help understand the range of I718 microstructures that is attainable by DED process, while keeping the geometry constant.
- ii. Proposing a characterization methodology to quantify the thermal history of printed part, based on the data collected from part (i). The methodology is developed based on process-structure relationship to predict final microstructure formation.
- iii. Estimating the microstructure formation of sample with different geometry but constant processing condition.

In this work, the quality and properties of the DED printed parts will be investigated and validated by a number of characterization techniques, including scanning electron microscopy (SEM) and electron backscattered diffraction (EBSD). These techniques will yield information about structure, crystal orientation, phase or strain of the material [16], [17].

2 Literature Review

2.1 Directed Energy Deposition

Directed Energy Deposition (DED) is a 3D printing method, which uses a focused energy source, such as plasma arc, laser, or electron beam to melt a feedstock material. As compared to other 3D printing method, DED system can be used to deposit material on existing component, making it suitable for repair application and functionalizing pre-existing part.

2.1.1 Equipment

A DED machine consists of a nozzle mounted on a multi-axis arm, which delivers raw material—in the form of powder or wire—onto a substrate (or build platform) where it is melted by a high-energy source (either laser or electron beam). The working principle is illustrated in Figure 1. Wire-based DED processes provide a lower resolution compared to powder-based processes but have higher deposition rates and thus facilitate the production of larger structures. This technology has one of the highest deposition rates among different AM processes, making it suitable for building of bulky parts for marine and aerospace application. There are several suppliers of DED systems, including Trumpf, BeAM, Optomec, DM3d Technology, and DMG Mori.

Figure 1: Illustration of DED process. The deposition head defines thermal energy source: Laser Beam, Electron Beam, or electric arc. The raw material can be a wire or powder. (Jose Luis Davila, 2020)

Nozzle

The two-common types of nozzle found in DED machines are coaxial and off-axis, as shown in Figure 2. The coaxial nozzle offers the advantages of being directionally independent and there is a symmetry between powder and laser. In systems mounting an off-axis nozzle, the powder is fed from the side, which leads to a variable deposition efficiency as a function of deposition direction. A study by Marcel Dias on the effect of nozzle configuration highlighted that a better catchment efficiency is observed in co-axial axis configurations [4].

Figure 2: Illustration of coaxial nozzle (left) and off axis nozzle (right). (Marcel Dias, 2012)

The coaxial nozzle configuration supplies powder stream in annular shape or utilized multiple outlets to create a powder focus. In present work, we utilised two DED machines (see Chapter 3, Table 4 for detail configuration) to produce different samples. Both machines used for samples production have a coaxial nozzle configuration with two different sizes (10Vx and 24Vx) as shown in Figure 3. The nozzle comprises three cones, which are concentrically mounted inside each other with at a precise offset distance. One of the inner slits created by this offset will direct the powder stream to a few millimetres below the nozzle, which coincides with the focus point of laser source. At the same time, the other inner

Figure 3: (a) Coaxial nozzle configuration (b) 10Vx Nozzle (c) 24Vx Nozzle (BeAM, 2019)

slit will supply secondary gas flow to focus the powder stream. This constant offset, also known as standoff distance, needs to be maintained throughout the process for a stable deposition process. The common materials used to make the nozzle are brass and copper, which have a high reflectivity and thus are less likely to absorb scattered laser light. However, the low melting points of copper and brass result in deterioration of the nozzle tip due to excessive heating from repelling of partially melted powder particles and spatter.

Off-axis nozzles use a tube to feed the powder into the melt pool from one direction, similar to a welding process. The powder delivered by this nozzle configuration has a higher scatter rate, which results in lesser powder captured by melt pool and make it less efficient. This leads to process inconsistencies such as uneven layer thickness and a rough surface finish. Therefore, the application of these types of nozzles is limited to single direction deposition. Conversely, this configuration does provide a better accessibility in some cases such as depositing in a corner or grooves.

Powder feeder system

The powder feeder system functions to feed raw material during the deposition process in a consistent and reliable way. A powder delivery system consists of a few main components, namely a powder tank, a powder feeder unit, and a gas supply as shown in the Figure 4a. It is noteworthy that there is an offset between feeding groove and powder outlet, as shown in Figure 4c. This design is to eliminate downward force due to weight of powder disrupting the powder flow rate. The turntable then brings the powder to outlet and controls the powder feeding rate by turntable speed and flow of carrier gas. The actual powder-feeding rate will be a function of rotating disc speed, type of powder used and rate of gas supply, which need to be empirically determined prior to the deposition process.

Figure 4: (a) Powder feeder system (b) Powder feeder unit (c) Detail view of powder feeder unit

Laser

In DED processes, laser is one of the most common energy sources. There are different types of lasers such as atom laser, molecule laser, ion laser, excimer lasers, liquid state lasers, solid-state lasers and fiber lasers. In this project, the machines used are equipped with a fiber laser, in which the active medium is an optical fiber doped with rare-earth elements such as Ytterbium. A Diode-laser pump source is then used to excite this rare earth atom via diode electroluminescence process and emit photons at 1070nm, which is the wavelength of the laser.

As we are dealing with laser-assisted manufacturing, understanding the fundamental absorption mechanisms of light in metals is important. Light impinging on the surface of a metal can be reflected, transmitted, or absorbed. Absorptivity refers to the ratio between absorbed energy and incident energy. An extremely low absorptivity in laser-assisted manufacturing is dangerous because the reflected light-rays may damage the equipment or working personnel. Figure 5 provides a schematic illustration on absorption rates by different metals during laser processing. The fiber laser with 1070nm wavelength used in this project works well with most alloy metal powder such as nickel, titanium, and ferrous metal. However, it cannot be used to process materials with high reflectivity like copper, silver, or aluminium.

Figure 5: Laser absorption rate on Metal (Keyence)

Gas supply

The purpose of the primary gas (or carrier gas) is to transfer powder from the feeder to the melt pool. On the other hand, the secondary gas (which is usually known as forming gas) serves two purposes: i) shielding the melt pool and ii) streamlining the flow of powder. Shielding of the melt pool is meant to protect heated deposit from reacting with atmospheric environment. This is because most metals are highly reactive with oxygen and nitrogen for formation of oxides and nitrides. These constituents are often having deteriorating impact on the part properties and therefore are considered undesirable. As shown in Figure 6, the forming gas helps to streamline and focus the flow of powder during deposition. This will help in maintaining a stable melt during the process.

A study conducted by Shah et al. [10] reported that an increase in carrier flow gas rate allows the powder particles to travel at a higher speed and leads to a shorter and deeper melt pool. On the other hand, a high forming gas flow will lead to less dense powder focus which would decrease the efficiency of the process. It was shown that porosity could form with un-optimised flow rate of forming gas [19]. At the same time, these gases also help in protecting laser optics component from repelling of powder particles or spattering during deposition process.

Figure 6: (a) Illustration of gas flow in nozzle (b) powder stream without forming gas (c) powder stream with forming gas

2.1.2 Process parameters

Similar to other AM technologies, DED has its specific processing variables that have direct influence towards the as-printed microstructure. The quality of a printed component is highly dependent on these processing conditions such as process parameters used, feedstock characteristics, and substrate quality, as illustrated in Figure 7. These process parameters are material-dependent and vary with machines (nozzle design, powder feeder configuration) and operating environment.

Figure 7: Quality of printed component and affecting variable

This part of thesis will focus on the process parameter and their effect on the quality of the printed components. Table 1 lists some of these parameters and their respective description.

Table 1: DED main process parameter

Parameter	Unit	Description
Laser power	W	The power of laser beam which melt and fuses the powder.
Laser spot size	mm	The diameter of laser beam.
Laser beam profile	N.A.	The shape of laser beam.
Standoff distance	mm	The distance between deposition head and focus point.
Layer height	mm	The thickness of each deposited layer.
Scanning speed	mm/min	The traverse speed of deposition head with computer numerical control (CNC).
Powder feeding rate	g/min	The amount of powder fed into melt pool per unit time.
Hatch spacing	mm	The distance between centreline of two adjacent bead.
Shield gas	L/min	The flow rate of inert gas shielding melt pool.
Secondary gas	L/min	The flow rate of inert gas to focus powder stream.
Carrier gas	L/min	The flow rate of inert gas to carry powder from powder hopper to melt pool.
Dwell time	s	The interlayer idle time.

These parameters, together with derived process parameter such as deposition rate (cm^3/hr), volumetric energy density, VED (J/mm^3) and linear heat input, LHI (J/mm) are going to directly influence printed part microstructural, mechanical, and dimensional characteristics. The effect of respective process parameters is described in the following sections.

Laser power

The laser power is one the most important parameters in the DED process. Higher laser power may result in significant effects on the i) dimensional accuracy (width of the deposit), ii) dilution iii) surface finish iv) grain orientation [20]. A high laser power promotes growth of columnar grains and increases dilution [21]. However, extremely high energy density may lead to keyhole melting which is undesired as this type of deposition have a high melt-pool flow velocity and a plasma plume that would cause extreme disturbance in power flow [22].

Laser spot size

The laser spot size in DED is at least 10X or larger compared to that found in powder bed fusion (LBPF) processes and usually in the range of mm. This results in a larger size melt pool size and a much faster deposition rate. The machine used in this project mounts two interchangeable nozzles with different laser spot size in diameter (10Vx: 0.8mm. 24Vx: 2.25mm), shown in Figure 3 (b) and (c). Size of deposition bead is kept the same as laser spot diameter to enable a consistent deposition process. In general, deposition process using a larger laser spot size offers higher deposition rate at a cost of reduction in dimensional accuracy due to the size of melt pool. At the same time, a larger laser spot size will also result in a coarser microstructure on the printed parts [23].

Laser Beam Profile

Lasers in DED can come in different intensity profile such as circular, longitudinal elliptical, transverse elliptical, Gaussian, and Top Hat profile [24]. The two most common types of laser beam profile used in DED process are Gaussian and Top Hat due to their symmetrical intensity. As shown in Figure 8, a Gaussian laser beam at the same power will have a peak intensity twice as large as Top Hat profile. During a deposition process, the higher peak intensity results in higher laser penetration depth of melt pool. Different studies showed that the higher intensity by a Gaussian laser beam will result in higher temperature and higher thermal gradients in the melt pool [25], [26]. As a result, the higher cooling rate produces a finer microstructure in final parts. On the other hand, the laser profile will also affect element segregation and phase formation in the printed part [25].

Figure 8: Illustration of laser profile with Top-Hat (left) and Gaussian (right) distribution

Stand-off distance

The laser stand-off distance is defined as distance between nozzle tip and surface of deposition. Under ideal conditions, the laser stand-off distance is the meeting point between focus point of laser and powder feeding stream. It is a key process variable and has to be kept constant during the process to deposit a consistent and stable layer. The laser stand-off distance depends on the type of nozzle (10Vx:3.5mm, 24Vx:13mm).

The laser stand-off distance needs to be calibrated prior to the deposition and monitored closely during the process. This is because any deviation of actual layer height from the programmed layer height will shift the laser standoff distance and cause laser defocus or interference with the nozzle tip.

Layer Height

The layer height is the thickness of each deposited layer. Unlike in PBF processes, where layer height is a pre-fix value, the layer height in DED process is a function of material, powder flow rate, power, and scanning speed. Hence, this value is usually empirically determined based on the initial run and set as Z-increment value, which is the fixed distance the nozzle will move after every layer deposition. Furthermore, the layer height should be kept within the range of 1/3 to 1/5 of the deposition bead width [13].

However, the process using a same energy level will cause the gradual increase in the layer height in subsequent layer. This is because the energy supplied in the first layer is more than sufficient for the other layer, due to the heating up of previous layer [14]. On the other hand, variability in powder flow rate will also result in deviation between Z-increment and actual layer height. Due to the layer-by-layer nature of the process, there will be stacking effect of the errors, resulting in the dimensional discrepancy of final part.

One of the attempts to improve the consistency of the process is via closed loop control, to monitor and adjust the Z-increment on real time basis [14, 15].

Scanning Speed

The scanning speed is a process parameter that has the same significant effect as the laser power on the deposition process and part quality. Decreasing the scanning speed while keeping other process parameter constant results in higher energy input and lower cooling rate, which makes the microstructure of printed part coarser [16]. A study by Gaumann [31] shows that lower scanning speed promote the formation of columnar grains. The scanning speed also have the effects on solidification direction and velocity which will dictate the final part microstructure.

Powder Feeding rate

A higher powder feeding rate means that more powder is supplied to the melt pool at a unit time and this will effectively increase the deposition rate. However, the right combination with other process parameter such as power, scanning speed and standoff distance need to be carefully selected to achieve a desired deposit. It has been shown that powder feed rate has a large effect on the layer thickness [32], laser attenuation, and powder particle temperature [33], [34]. Since the attenuation is increased with higher powder feeding rate, it will inversely effect laser penetration depth.

Hatch spacing

Hatch spacing refers to the spacing between centre lines of two adjacent deposits and it was shown to have an effect on the level of porosity, grain structure, residual stress distribution, and surface finish [35]. As a rule of thumb, the hatch spacing selected should be able to introduce overlap regions of >50% to avoid porosity or lack of fusion in between beads. In Figure 9, lack of fusion is formed in between the bead due to insufficient overlap region. On top of that, a lower overlap distance can promote the growth of columnar grain structure with higher thermal gradient induced between melt pool and substrate. Additionally, built with lower overlap tends to improve the surface finish [22]. On the other hand, higher residual stress was observed in the overlap regions compared to centre regions of the bead [35].

Figure 9: Lack of fusion formation in between beads due to insufficient overlap

Dwell time

The inter-layer dwell time is the finite time elapsed between successive layers of deposit with a range of 0-1000s [37], [38], [39]. The longer the dwell time, the longer the deposit is allowed to cool via

radiation and convection [38][39]. Short dwell time can cause re-melting of previous layers and can lead to warping of parts due to higher thermal build up [40]. Costa has numerically demonstrated the lower dwell time tends to promote heat build up in the part [41].

Dwell time control is a simple approach for tailoring solid-state phase transformation within DED parts. However, in many cases it is sought to be minimized to increase the speed of manufacturing process [42].

2.1.3 Materials

A super alloy is a metallic alloy which can be used at high temperatures, often exceeding 0.7 of absolute melting temperature [46]. Nickel-based superalloy with high thermal stability, excellent creep strength, oxidation resistance, corrosion resistance and fracture toughness make it an excellent choice of material for application under extreme condition. These alloys are used in aircraft engines (50% of the parts), steam turbine power plants (blades, stack gas reheater), reciprocating engines (turbo charger and exhaust valve), metal processing (hot work tool and dies), space vehicles and others.

Inconel 718

The material selected for this project is I718, which is a nickel-based precipitation-hardening alloy designed for very high yield strength, tensile strength, and creep resistance properties. Its ability to retain excellent mechanical properties under extreme high temperature ensures the functional integrity of the engineering application. Several applications of Inconel in aerospace include:

- (i) The Space Shuttle used several Inconel studs to secure the solid rocket boosters to the launch platform., eight total studs supported the entire weight of the ready to fly shuttle system.
- (ii) SpaceX uses I718 in the engine manifold of their Merlin engine which powers the Falcon 9 launch vehicle.
- (iii) SpaceX uses I718 to additively manufacture SuperDraco rocket engine that provides launch escape system for the Dragon V2 crew-carrying space capsule.
- (iv) Rocketdyne used Inconel X-750 for the thrust chamber of the F-1 rocket engine used in the first stage of the Saturn booster.

Composition

Table 2: Chemical composition of the I718 alloy

Element	Ni	Cr	Mo	Nb(+Ta)	Fe	Mn	Al	Ti
Weight (%)	58	20	10	5.5	5	0.5	0.5	0.5

Nickel based superalloys consist of about 10-15 elements, beyond nickel, which all contribute to the performance of materials. In order to understand why these elements are necessary, functions of few primary elements are explained in the list below.

Nickel (Ni): Ni is the base materials with highest percentage. It has high thermal stability and good corrosion resistance [47].

Iron (Fe): Fe improves the malleability of the alloy but it reduces the oxidation resistance and promotes TCP phase, the intermetallic phases that can be detrimental to the mechanical properties of superalloy.

Aluminium (Al): Al in combination with Cr, increases the corrosion resistance. Al contributes to strengthening via solid-solution strengthening and promotes precipitation of γ' phase.

Chromium (Cr): Cr improves strength at elevated temperatures. Additionally, Cr increases resistance to oxidation below temperature of 950C, pitting corrosions, sulfidation, carburization, and corrosion caused by oil ash and molten glass.

Niobium (Nb): Nb helps in creep resistance and increases the short-term creep strength. Nb also increases the solid solution strengthening, promotes γ' , γ'' and NbC formation, and governs γ and δ Ni_3Nb precipitation. The exact roles of these phases are described in section 2.1.3.3.2

Molybdenum (Mo): Mo has a strong correlation with increase in solid solution strengthening and increase the amount of γ' phase in the matrix. It also increases the density of the alloy.

Titanium (Ti): Besides solid solution strengthening, Ti promotes precipitation of γ' and TiC phases.

Microstructure and strengthening mechanism

The essential solutes in a nickel-based superalloy are aluminium and/or titanium, with a total concentration <10%. This generates a two-phase equilibrium microstructure, consisting of gamma (γ) and gamma prime, γ' . Gamma, γ is the face-centred-cubic nickel matrix (Ni) and gamma prime, γ' is face-centred-cubic Ni_3Al or Ni_3Ti precipitate. The high coherency of crystal structure between γ' precipitate with γ matrix, results in low interfacial energy, and a stable microstructure especially in an elevated temperature condition.

In contrast, the most widely used nickel based super alloy, I718 is reinforced by tetragonal Ni_3Nb , the tetragonal gamma double prime, γ'' precipitates. The additional strengthening conveyed by γ'' over γ' is a function of the coherency of precipitate with matrix and distortion caused by C-axis of the tetragonal γ'' . On prolonged exposure at intermediate temperatures, the γ'' transform to δ phase. At relatively lower temperature, δ phase nucleates at austenitic grains boundaries as well as coherent and incoherent twin boundaries, while at higher temperature they precipitate intergranularly [49]. Though the δ phase is incoherent and its precipitation is known to reduce strength, controlled precipitation of δ phase can improve stress rupture properties and promote grain stabilization.

Precipitation strengthening

Precipitation hardening, also known as age hardening or particle hardening, is a heat treatment technique used to increase the yield strength of malleable materials [48]. In the case of Nickel alloy, it is achieved by precipitation of gamma, γ and gamma prime, γ' phases, as described in the earlier section. The addition of elements with limited solubility such as Niobium (Nb), Titanium (Ti) and Aluminium (Al) promote the formation of these phases. The strengthening effect is being achieved by coherency strain between the strengthening precipitates and the matrix phase and, antiphase boundary (APB) energy resisting dislocation movement between ordered strengthening phases in the matrix. Both coherency strain and ordered precipitates force dislocations to cut or climb through the precipitates which significantly increases the strength of the material. The precipitation strengthening effect becomes more effective with increased precipitate size. However, in the event precipitate is too large, the dislocation starts to climb the precipitate rather than cut through it, which results in a lowering of material strength. This is known as Orowan mechanism.

Solid solution strengthening

Opposed to the precipitation hardening, the effect of solid solution strengthening is attained through elements that are in a solution in the gamma, γ matrix. The local non-uniformity in the lattice due to these elements makes plastic deformation more difficult by impeding dislocation motion through stress field [50]. At elevated temperature, creep resistance depends on the diffusion of solute elements hence elements with high melting points and low diffusivity provide better performance.

Lowering the stacking fault energy is another effect of solid solution strengthening. A lowering of the stacking fault energy leads to a higher cross slip strength which prevents dislocations from changing direction into a new slip plane when it encounter a barrier such as carbides, gamma prime, γ' or gamma double prime, γ'' precipitate.

Atomic clustering or short range ordering refers to clustering of atoms attracted to each other by their electron orbitals. This mechanism is commonly observed in solid solution strengthened alloys which further inhibits the movement of dislocations. Some of the common example of elements are molybdenum (Mo), tungsten (W), Aluminium (Al), Chromium (Cr) and rhenium (Re). At a lower temperature, i.e. 60% of melting temperature, the increased diffusion significantly reduced the effect [51].

Oxide dispersion strengthening

Oxide dispersion strengthening is based on incoherency of the oxide particles within the lattice of the material. The oxide particles decrease movement of dislocations within the material and in turn prevent

creep. Since the oxide particles are incoherent, dislocation can only overcome the particles by climb. This is similar to the mechanism in precipitation hardening [52].

State of the art

I718 is one of the most well-developed alloys for metal additive manufacturing. The overview of current research status is summarised as follows:

- The rapid solidification and high cooling rates during the AM process result the microstructure of the as-built AM Inconel consists of a supersaturated γ matrix, Laves phase and a limited amount strengthening phases such as γ' and γ'' . Use of dedicated heat treatment process is needed to dissolve the Lave phase and subsequently maximise the precipitation of γ'' and γ' particles. A commonly used heat treatment process is solution heat treatment at 1000-1080°C followed by double aging treatment (720°C for 8 hours and 620°C for 8 hours).
- The employed AM technique, process parameters, heat treatment conditions, build geometry and loading direction will greatly influence the obtain strength of AM I718. The tensile strength and hardness for the AM I718 is typically between those produced with casting and wrought alloy.
- The fatigue performance of AM I718 is inferior to that of wrought alloy due to presence of AM-induced defects and poor surface quality. However, it has been shown that employment of optimised parameters for the AM process and the subsequent heat treatment process in combination with surface machining enhances the fatigue strength of AM I718.
- The anisotropy in the mechanical response of AM I718 is due to the significant different in microstructure and texture of AM I718. A higher elastic modulus and tensile strength, longer constant stress-amplitude fatigue endurance, shorter constant-strain-amplitude fatigue endurance, and shorter creep rupture time were typically observed for AM I718 loaded perpendicular to the build direction.
- AM-induced defects such as porosity and existence of high thermal residual stresses often suppress the mechanical integrity of AM I718. Employment of optimised AM process parameters and the application of post heat treatment processes are beneficial for decreasing the AM-induced defects and level of residual stress.

2.1.4 Substrate

The size, thermal capacitance and initial temperature of the substrate will have significant impact of thermal history and eventually the microstructure [42]. During deposition, a substrate as a heat sink introduces high temperature gradient with melt pool. Heat loss from the melt pool is then achieved via conduction. A study by Coasta et al. focused on the heat sinking effect brought about by the substrate showed that a larger substrate had greater heat extraction capability while a smaller substrate allowed the heat build-up in the part [41]. The higher cooling rate during the first deposition layer resulted in a finer microstructure in the substrate vicinity [43]. Preheating of substrate is often performed to reduce the residual stress and cracking and/or thermal distortion.

2.1.5 Powder Characteristics

Powder quality plays an important role in dictating the DED printed part quality. There are different powder production processes [44], including gas atomization (GA), plasma rotation electrode process (PREP), Induction Melted Bar Atomisation (EIGA), and Plasma Atomised Wire (PAW). The advantages and disadvantages of the major process are shown in Table 3.

Table 3: Comparison of different powder production process

Process	Advantages	Disadvantages
Gas Atomisation (GA)	<ul style="list-style-type: none">• Excellent metallurgical quality• High powder flow rates• Wide selection of alloys• New and modified alloys can be made easily• Scalable technology: High volume production possible• Large supply base• Relatively low cost	<ul style="list-style-type: none">• Variability in quality across supplies• Reactive and high melting point alloys not wide available. Ex. Titanium
Electrode Induction Melted Bar Atomisation (EIGA)	<ul style="list-style-type: none">• Excellent metallurgical quality• High powder flow rates• Reactive and high melting point alloys available. Ex. Titanium• High production rates	<ul style="list-style-type: none">• Limited but growing supply base• Raw material need to be in bar form• Relatively high cost

Plasma Atomised Wire Process (PAW)	<ul style="list-style-type: none"> • Excellent metallurgical quality • High powder flow rates – perfect sphere • Reactive and high melting point alloys available. Ex. Titanium 	<ul style="list-style-type: none"> • Limited but growing supply base • Raw material needs to be in wire form $\phi < 5\text{mm}$ • Relatively high cost
Plasma Rotating Electrode Process (PREP)		<ul style="list-style-type: none"> • Limited but growing supply base • Raw material need to be in bar form $\phi > 5\text{mm}$ • Relatively high cost
Water Atomisation (WA)	<ul style="list-style-type: none"> • Scalable atomising technology • Low cost 	<ul style="list-style-type: none"> • Metallurgical quality not as good as GA produced powder • Powder not natively spherical

Some of the most important and commonly referred characteristics of powder include chemical composition, particle size distribution (PSD), and hall flow rate. The chemical composition is paramount in a DED processes since it defines the alloy. In an alloy, there are major, minor and trace elements. Major elements determine the alloy properties and minor element can affect an alloy phases and even cause negative effects on weld ability and ability to sinter in AM. As such, the element content needs to be controlled within the prescribed upper and lower limit. The PSD is usually described in percentile: D10, D50 and D90. D10 indicates 10% of powder weight is finer than this micron size and D90 shows 10% of powder by weight is coarser than specified value. Research shows that larger powder particles can distort and melt pool and eventually affect part quality [45]. Hall flow can be assessed using a Hall Flow meter, which determines the time that 50g of powder takes to flow through a 2.5mm orifice. The changes in the powder flowability can indicate detreating in powder properties which may negatively impact printed part quality.

2.2 Solidification in DED

During powder-based DED processes, metal powders are melted “on the fly” by a focused laser beam as they are blown onto the surface of the build platform. The solidification of the melt occurs in lapse of time of milliseconds [53]. In this rapid solidification process (cooling rate $> A = 10^4\text{K/s}$), significant changes in the microstructure such as grain refinement can be obtained. The solidification patterns such as planar, cellular, columnar dendritic or equiaxed dendritic, depend on the local temperature field near the solid-liquid growth interface and the grain orientation of the substrate [54]. Previous work has shown that columnar grains grow epitaxially on single crystalline substrate. AM processing parameter has been reported to have a significant impact on melt pool and thermal condition such as the local

temperature gradient (G) solidification growth rates (R) and the cooling rate $\left[\frac{\partial T}{\partial t}\right]$. In the following, we report in details how these quantities may be calculated [55].

2.2.1 Melt Pool

The melt pool is the region of superheated molten material in proximity to the laser/material interface; typically in the form of spherical shaped droplet that moves at the traverse speed. Figure 10 illustrates a typical melt pool in DED processes, with heat affecting zone underneath. The heat transfer between surrounding and liquid/solid interactions makes melt pool thermodynamically unstable. Since the melt pool is the initiation of the solid part, its morphology, temperature, and wetting behaviour are extremely important in quality control. Dimensional tolerances, microstructural feature and existence of residual stress of the produced part, are highly dependent on the melt pool behaviour and shape during the process. It was reported by Griffith that a smaller particle size (~325 mesh) results in a more stable melt pool for Inconel 625 during DED process [45]. Larger particles with wider range of sizes were found to provide for an unstable melt pool that was highly agitated.

Figure 10: Melt pool with heat affected zone (HAZ) in a typical DED process

During DED process, the natural limitation of thermal radiation (e.g., no true blackbody) together with interference of blown powder results in only a portion of the incident laser to be absorbed by the melt pool. The laser heat flux can be estimated as follows [56]:

$$q_{laser} = \frac{2\alpha_m P}{\pi R^2} \exp\left(-\frac{2r^2}{r_{b,i}^2}\right) \quad (1)$$

P: Laser Power, W

R: Laser spot radius, mm

$r_{b,i}^2$: radius of the laser beam at the melt pool interface, mm

r : radial distance from the center of the laser beam, mm

α_m : absorptivity of the melt pool

At the junction of melt pool/vapor gas interface, the local heat loss is a result of convection with adjoining shielding gas, net radiation with surrounding and heat loss due to vaporization of melt pool, which can be estimated as

$$q_{loss} = h_x(T(x) - T_{\infty}) + \varepsilon\sigma(T^4(x) - T_{\infty}^4) + \rho\left|\frac{\Delta x}{\Delta t}\right| h_{LV} \quad (2)$$

h_x : Local heat transfer coefficient

$T(x)$: Local surface temperature

T_{∞} : Temperature of shielding gas

ε : melt pool emissivity

σ : Stefan-Boltzmann's constant

ρ : density of the liquid

$\left|\frac{\Delta x}{\Delta t}\right|$: rate of melt pool deformation due to evaporation

h_{LV} : latent heat of vaporization

The effective heat flux which contributes to melt pool formation, and the sensible heating of melt pool and part, is then found as

$$q = q_{laser} - q_{loss} \quad (3)$$

2.2.2 Solidification Parameter

In a DED process, high cooling rate ($>100\text{K/s}$) and rapid growth of solidification front ($>10\text{mm/s}$) directly implies a rapid solidification process. The few key solidification parameter such as temperature gradient (G), growth rate (R), and cooling rate ($\frac{\partial T}{\partial t}$) will directly dictate the as-printed part microstructure, as shown in Figure 11. Here, we report the definition and effects of respective solidification parameter as follows:

Temperature Gradient (G)

The heat flow direction at any point on the solidification surface is normal to the surface. The predominant heat flow direction and temperature gradient (G) can be presented as following:

$$\nabla T = \frac{\partial T}{\partial x}i + \frac{\partial T}{\partial y}j + \frac{\partial T}{\partial z}k \quad (4)$$

$$G = ||\nabla T|| = \sqrt{\left(\frac{\partial T}{\partial x}\right)^2 + \left(\frac{\partial T}{\partial y}\right)^2 + \left(\frac{\partial T}{\partial z}\right)^2} \quad (5)$$

T: Temperature

i: Scanning direction (x)

j: Width direction (y)

z: Vertical direction (z)

G: Temperature gradient

In order to relate temperature gradient G with process condition in a DED process, Li et al. proposed a simplified qualitative model as follows.

$$G = \frac{2\pi K(T - T_0)^2}{\eta P} \quad (6)$$

T: Liquidus temperature of alloy

T_0 : Temperature

η : Alloy absorption coefficient

P: Laser power

K: Alloy thermal conductivity

Figure 11: The effect of G & R on the solidification morphology and size

Growth Rate (R)

Growth rate or solidification rate is the rate of advance of the solid/liquid interface into the liquid. The rate of movement of solidification front determines solute redistribution during solidification, scale of solidification structure and the growth undercooling. Research has shown that growth rate in a laser processing can be determined using the method describe at below [57]. During a consistent laser process with constant power and speed, it created a steady state melt-pool, as shown in Figure 12. Taking a longitudinal cross-section through the centreline along the melt-pool (plane XZ), the growth rate can be related to the laser beam speed with following formula.

$$R = V \cos \theta \quad (7)$$

R: Solidification velocity

V: Laser scanning speed

θ : Angle between solidification front and scanning direction

The growth rate varies markedly over the pool depth, from zero at its interface with the substrate, to a maximum on its surface.

Figure 12: (a) Melt-pool with reference to beam axis (b) Longitudinal cross section of melt pool along XZ plane (Gremaud, 1990)

Cooling rate

The cooling rate is the temperature change of the melt pool with respect to time and varies along the volume of melt pool; being the highest at the solid-liquid interface and decreasing with distance from the centre of the melt pool. It will depend on the heat loss and conduction heat transfer through the part.

The cooling rate, $\frac{\partial T}{\partial t}$, at a given location near the melt pool, is presented as:

$$\frac{\partial T}{\partial t} = G \cdot R \quad (8)$$

The cooling rate can be empirically determined with correlation with dendrite arm spacing, using the following formula

$$\lambda = K \left(\frac{\partial T}{\partial t} \right)^{-n} \quad (9)$$

λ : Dendrite arm spacing

K: Material constant

n: Material constant

The material constant K and n for I718 was reported to be 130 and 0.33, respectively [58]. The two critical derived solidification parameters are ratio G/R , which affects the solid liquid interface, and cooling rate, $\frac{\partial T}{\partial t}$, which affects the microstructure dimensions. Three major structure morphologies because of different G and R values are i) columnar (elongated grain morphology), ii) columnar and equiaxed, and iii) equiaxed (isotropic grain morphology). In general, increase in solidification rate induces the transition from columnar to equiaxed morphology and at the same time, increases the

cooling rate, $\frac{\partial T}{\partial t}$. The respective effects [59] of both solidification parameters and their relation to process parameter are summarised as follows:

- 1) Higher cooling rate results in finer microstructure. This can be achieved by higher scanning speed and lower laser power, which introduce lower thermal gradient during the process by allowing the part to cool down.
- 2) Lower cooling rate results in coarser microstructure. This can be achieved by lower scanning speed and higher laser power, which introduce steeper thermal gradient during the process by effectively heating up the part.
- 3) Higher G/R ratio promotes the growth of columnar grains. This can be achieved by lower laser power (reducing the heating effect on part for higher G) or higher scanning speed (increasing the solidification rate, R)
- 4) Lower G/R ratio promotes the growth of equiaxed grains. This can be achieved by higher laser power (increasing the heating effect on part for lower G) or lower scanning speed (decreasing the solidification rate, R)

2.3 Microstructure & mechanical heterogeneity in DED

Given that one of the main advantages of DED is to directly manufacture and repair the component, understanding the heterogeneity that exists in the metal AM part is important for reliability control. Microstructural heterogeneity of metal AM parts can arise from difference in morphology, size, orientation and chemical composition of phases and grains. Such differences are mainly attributed to the complex and varying thermal conditions (e.g. thermal gradient, cooling rate, processing temperature) during the process.

2.3.1 Microstructure heterogeneity

Grain Size

In a study conducted by T. Wang on grain morphology evolution in DED process [60], the two dominant solidification mechanism reported are heterogeneous nucleation on partially melted powders for equiaxed grains and the epitaxial growth from the pool-bottom for columnar grains, as shown in Figure 13. A large melting penetration to the underlying layer and high temperature gradient in front of the solidification interface making the bottom epitaxial growth mechanism prevail and promoting the formation of large full columnar grains structure. Conversely, on the top of deposited bead, heterogeneous grain nucleation with partially melted powder forms the equiaxed grain zone.

Figure 13: (a) Cross section of single bead showing typical as solidified grain morphologies, the depth of the equiaxed grain area, d_{EG} and depth of the columnar grain area d_{PM} (b) schematic diagram of two dominant solidification mechanism. (T.Wang, 2015)

Precipitate

Other than the difference in grain morphology, study shows that microstructure heterogeneities can be a result of solidification segregation. A study in DED-built I718 alloys attributed the formation of precipitates caused heterogeneity in hardness across the build [61]. The precipitate evolution is illustrated in Figure 14. In a separate study by Abdullah on controlling microstructure by different process parameters, they demonstrated that higher thermal gradient due to lower heat input tended to form equiaxed grains and more Nb-segregation, whilst a higher heat input developed columnar grains with low Nb-segregation [6].

Figure 14: Schematic diagram for microstructural evolution. (a) Primary solidification with the formation of γ phase and eutectic products occurs after the first laser scan. (b) Remelting of eutectic products and diffusion of Nb into the dendrite core occurs during the second laser layer. (c) Precipitation of γ'' close to the eutectic products due to high Nb concentration during subsequent cycles. (d) Precipitation of γ'' within the γ phase close to the dendrite boundaries with subsequent thermal cycles. (e) Growth of existing precipitates and next wave of precipitation within the dendrite core after more thermal cycles. (f) Onset of nanoscale precipitation within the dendrite core after many thermal cycles (Yuan Tien, 2014)

Grain Orientation

In general solidification theory, the fastest growing direction for a face-centered cubic (fcc) material is $\langle 100 \rangle$ and that occurs by nucleation or growth of crystal [49]. Depending on the solidification condition, either nucleation or growth of crystal may be predominant. For example, epitaxial growth will be favoured if the $\langle 100 \rangle$ direction of substrate is oriented parallel to the maximum heat flow. Conversely, nucleation of new grains will be more likely if the $\langle 100 \rangle$ direction is not aligned to the maximum heat flow direction.

This concept is well illustrated in the work by Dinda et al about texture control during laser deposition [62]. In this paper, the effect of laser beam scanning pattern on dendrite growth morphology was investigated by EBSD. When the scanning strategy had kept constant, there would be constant heat flow direction throughout the build and the microstructure would consist of highly textured columnar grains formed with only one $\langle 100 \rangle$ crystallographic orientation aligned towards the maximum heat flow direction. This phenomenon was the result of grain competition, only those grains had a $\langle 100 \rangle$ direction nearly parallel to the heat flow would grow rapidly where growth of other grains would cease.

Site Specific Grain Orientation Control

Some researchers have shown the site specific grain orientation can be controlled by manipulating G and R during the AM process, shown in Figure 15. In the experiment conducted by R.R. Dehoff [63], they demonstrated the change in scan strategy between point and line heat source mode could promote steady state and/or transient thermal gradient and liquid/solid interface velocity. Karl A. Sofinowski showed that varying the laser scanning angle was able to produce samples with controlled crystallographic texture [64].

Figure 15: Crystallographic orientation map of site-specific orientation control in Electron Beam Melting (EBM) build (Dehoff,2018) (b) Crystallographic orientation map of site-specific orientation control in Powder Bed Fusion (PBF) build (Karl. A,2020)

2.3.2 Mechanical heterogeneity

In general, mechanical properties in DED parts are similar or higher than wrought or cast materials. This is due to the relatively high cooling rates experienced by parts during the process, which leads to formation of finer microstructure and results in higher tensile strength or hardness. However, common DED defects such as high porosity, lack of fusion or residual stress may impose detrimental effects on the mechanical properties. The effect of dissimilar thermal histories across the parts had been widely reported to be responsible for the mechanical heterogeneity.

Tensile Properties

The tensile test is one of the most common mechanical testing for measuring material strength. In a study conducted by Thale R. Smith [65], they found that yield strength was a function of distance from the base plate as illustrated in Figure 16. They attributed the variation to the difference in the dislocation density and compositional micro segregation. Similar study on Ti64 using other AM process also reported higher tensile properties in lower half of the block as compared to the upper half, which attributed to oxygen strengthening and finer microstructure [66]. Furthermore, a study on EBM-built Ti64 also reported increase in strain hardening effect with increasing build height due to lesser annealing [67].

Figure 16: Yield strength as a function of distance from build plate

Hardness properties

Hardness testing is an effective method to characterize the localized mechanical strength of metal AM part. Researchers have been reported that for steels, the micro-hardness values of subsequent deposited layers decreased from the first deposited layer and then increased towards top layer [72], [38], [40]. This variation in thermal history experienced by the part can be used to explain this phenomenon. The time variable cooling rate during the process results in higher hardness value at top and bottom (higher cooling rate) as compared to middle region (lower cooling rate). In addition to that, it was shown that in other AM processes, the increasing cross sectional area results in decreasing of hardness [73], [75], [76]. The rationale behind is microstructural coarsening due to greater thermal input and slow cooling rates experienced by part with thicker cross-sectional area. Many researchers then concluded that hardness heterogeneity is dependent on the thermal history at respective location. A large cross-sectional area would result in higher thermal input as compared to smaller cross-sectional area. Separately, dissimilar cooling rate across the sample height, will also result in different microstructure. Future improvement on varying process parameter with regards to cross sectional area and height could help alleviate such heterogeneity in hardness.

2.3.3 Residual Stress

Residual stress is defined as stress in a body which is at rest and in equilibrium and at uniform temperature in the absence of external mass force [68]. The high cooling rate and thermal gradient in DED process, together with rapid local heat transfer rate, are the main reason of residual stress in DED parts. Study has also shown that raster direction might have some effect on residual stress of deposit [18]. It was reported that in vertical direction, there exists the largest amount of compressive residual stress in DED. Material properties, such as thermal conductivity, elastic modulus and yield stress, together with part geometry, and process parameter all influence the magnitude and pattern of the residual stress [69][70].

Presence of residual stress is undesirable because it reduces the strength or life of mechanical parts, and induces dimensional inaccuracies due to warpage [70], [71].

3 Characterization Methods

3.1 Sample preparation

We use two DED machines for samples production. They are i) Trumpf Trulaser Cell 7040 ii) BeAM Magic 800 (see Figure 17). Both machines are using DED technology with their similarities and difference highlighted in Table 4. We use BeAM Magic 800 to conduct parameter development and production of X-series and R-series samples (see chapter 4, 6 and 7). Separately, we use Trumpf machine with industrial specific propriety parameter to produce H, M, L and R samples (see chapter 5).

Figure 17 (a) Trumpf Trulaser Cell 7040 (b) BeAM Magic 800

Table 4: Specification comparison between Trumpf and BeAM machine

Machine	Trumpf Trulaser Cell 7040	BeAM Modulo 800
Technology	Directed energy deposition (DED)	
Motion	5-axis CNC control	
Max Power	5000 W	2000 W
Laser	Solid state (disk laser)	Solid state (fiber laser)
Laser wavelength	1030nm	1070nm
Laser spot size (Diameter)	Single nozzle with variable spot sizes: 1,2 and 3 mm	Dual nozzle with fixed spot sizes: 2.25mm (24Vx), 0.8mm (10Vx)
Laser profile	Gaussian distribution	Top Hat distribution
Carrier Gas	Yes	
Shielding Gas	Yes	

All the sample were printed on SS316L substrate, with a thickness of 6mm. The substrate is clamped on the CNC machine table for deposition. We heat treated (unless otherwise stated) all samples following the method described by AMS 5663, which consists of a solution treatment at 954°C for 1 hour, followed by two age hardening steps at 718°C for 8 hours and 621°C for an additional 8 hours. After heat treatment, we cut the samples into multiple slices by means of wire electrical discharge machining (EDM) for microstructure analysis.

3.2 Mechanical characterization

3.2.1 Hardness test

We measured hardness across the polished surface of sample slices using a Nano Indenter G200 (Agilent Technologies) with standard XP indentation head in compliance with the ISO 14577 standard [76]. During nanoindentation, we set the depth limit to 1000 nm, the strain rate to 0.05 s⁻¹, the harmonic displacement to 2 nm, the frequency to 45 Hz, and the Poisson ratio to 0.29 [78]. To calculate the hardness and elastic modulus, we defined the lower and upper depth limits at 850 nm and 950 nm, respectively, where the hardness and modulus have approached their asymptotic values. We used a Micro Measurements FM-300e to perform microhardness measurements with a load of 300 grams and a dwell time of 10 seconds.

3.2.2 Compression and tensile test

We performed compression (ASTM E9-19) and tensile (ASTM E8) test using a displacement-controlled Shimadzu AGS-X 50kN. Prior to compression testing, we lathed the sample to obtain symmetrically flat surfaces. We adopted a strain rate of 0.003 mm/s and assumed quasi-static mechanical testing. We applied the same setting for tensile test. During compression test, we lubricated the specimen's surface to reduce barrelling of the material under compression.

Figure 18: (a) Tensile coupon and (b) compression coupon

3.3 Microstructural characterization

We prepared the sample with standard metallographic procedure, which is grinding with 320, 800, 1200, 2000, 4000 SiC sand paper and then polished by 3 μm diamond solution and a 0.25 μm colloidal silica suspension. The samples were then etched for 7-9 minutes in a bath of waterless Kalling's solution (5 grams CuCl_2 , 100 milliliters HCl , 100 milliliters Ethanol) [65]. We characterized the samples microstructure using a Nordlys 2S EBSD detector (Oxford Instruments, UK) mounted to a JEOL JSM 7600F field emission scanning electron microscope (FE-SEM). We used the same FE-SEM for metallographic observation and for compositional analysis of the sample surface by means of EDS.

3.4 Density & Porosity characterization

We conducted density measurement of the sample using a XS Analytical Balance with sensitivity of 0.001 g and repeatability of 0.0001 g (XS 204, Mettler Toledo), based on the Archimedes Principle. Archimedes measurements are based on a combination of dry weighing and weighing in deionized water. The absolute density of each specimen was calculated using the equation:

$$\rho_{abs} = \frac{m_{air}}{m_{air} - m_{water}} \times \rho_{water} \quad (10)$$

ρ_{abs} : Absolute density of the specimen

m_{air} : Mass of specimen in dry weighing

m_{water} : Mass of specimen fully submerged in deionized water

ρ_{water} : Density of deionized water

We then calculate the relative density using following equation

$$\rho_{relative} = \frac{\rho_{abs}}{\rho_{material}} \times 100\% \quad (11)$$

$\rho_{material}$: Density of material

To characterize the lack of fusion or gas porosity dimensions, we used Image J to measure the maximum pore size observed on the sample.

4 Formation of I718 alloy with DED machine

4.1 Parameter development

The initial part of the work involves conducting a parameter development study to obtain machine specific I718 printing parameter yielding high quality and high density builds. As highlighted in section 2.1.2, there are numbers of process parameter directly affecting the printed part geometry and quality. To simplify the parameter development process, we focused on only two key processing parameters, namely powder feeding rate and laser power while keeping other process parameter constant. This approach would allow us to quickly obtain the processing window for near full dense to perform microstructure characterization and analysis.

Powder feeding rate

Powder from the powder tank is carried by carrier gas to nozzle passing through feeder system. Both carrier gas flow rate and speed of turntable inside the feeder system will determine the powder flow rate. Usually, the carrier gas flow rate is kept constant to avoid turbulent flow of gas in the feeding process due to high carrier gas flow rate. The speed of turntable is based on revolution per minute (RPM) and controlled in terms of percentage, ranging from 0 - 100%. While the speed of turntable is constant at 3RPM, the actual powder feeding rate at certain speed depends on type of material, condition of powder and environmental condition such as temperature and humidity. Due to the reason highlighted above, the powder feeding rate needs to be determined empirically prior to every deposition. We first set the turn table to different speed to collect the respective powder feeding rate. The data was then used to extrapolate from 0 - 100% turn table speed using linear relationship between the two. Figure 18 shows the typical flow rate acquired prior to the deposition. The process required powder feeding rate can be determine from Figure 18a and input in the program. Conversely, we can observe an inconsistency of powder feeding rate in Figure 18b, from a separate experiment conducted across different days. This is possibly due to the powder degradation or change in environmental conditions.

Figure 19: (a) Consistent powder feeding rate (b) Inconsistent powder feeding rate

Calibration or proper cleaning of powder feeding mechanism, i.e., powder hopper, turntable, feeding tube etc. might be required. Because of this, it is recommended to perform powder feeding rate check before any deposition / experiment.

4.1.1 Methodology

The sequential methodology of parameter development involves i) single bead printing ii) wall printing iii) cube printing. The purposes are to i) determine the process window for power, scanning speed and powder feeding rate, ii) determine layer height iii) determine the parameter sets to yield full dense cube, respectively.

Single bead printing

We started the experiment by developing parameter to print a continuous and undistorted bead, with the bead width equivalent to nozzle size. The purpose was to identify the suitable processing window. Three levels (high, medium and low) and two factors (A: laser power and B: powder feeding rate) full factorial design of experiment (DoE) had been conducted to understand the effects of respective processing parameter on bead width and bead height.

We conducted the development on 10Vx nozzle with range of laser power (200W, 400W, and 600W) and powder feeding rate (3g, 6g, and 9g). We measured the width and height of each single bead and record them in Table 5 (bead width) and Table 6 (bead height). The results are illustrated in Figure 20 (bead width) and Figure 21 (bead height).

Table 5: Recorded bead width according to DoE matrix with varying powder feeding rate and laser power.

Powder Feeding Rate (g/min)	B, Power(W)					
	600		400		200	
9	893	836	799	783	494	506
6	718	600	734	748	428	278
3	705	612	787	774	347	424

Figure 20 Effect of Laser Power and Powder Feeding Rate on Bead Width

Table 6: Recorded bead height according to DoE matrix with varying powder feeding rate and laser power.

Powder Feeding Rate (g/min)	B, Power(W)					
	600		400		200	
9	245	260	247	232	220	216
6	220	218	195	211	200	198
3	195	194	170	182	132	160

Figure 21 Effect of Laser Power and Powder Feeding Rate on Bead Height

We then carry out Analysis of Variance (ANOVA) to understand the respective effect of input parameter (laser power and powder feeding rate) on the output parameter (bead width and bead height), as shown in Table 7 and Table 8. The F-distribution value obtained from different confidence interval (90%, 95% and 97.5%) are $F_{0.1,2,9} = 3.006$, $F_{0.05,2,9} = 4.2565$, $F_{0.025,2,9} = 5.7147$. From the ANOVA results, we then concluded that

- i) laser power has the effect on bead width with 95% confidence,
- ii) powder feeding rate mainly affects the bead height with 90% confidence.

Table 7: ANOVA results for bead width

Source of variation	SS	DOF	Mean Square	F0
A	27151	2	13576	0.27
B	457933	2	228967	4.50
AB	541008	4	135252	2.66
Error	457576	9	50842	
Total	568516	17		

Table 8: ANOVA results for bead height

Source of variation	SS	DOF	Mean Square	F0
A	12507	2	6254	3.70
B	3543	2	1772	1.05
AB	16566	4	4142	2.45
Error	15221	9	1691	
Total	17396	17		

Using the results, we then carried out the respective parameter development for both 24Vx and 10Vx nozzle. We selected the suitable combination of parameter set is based on evaluation of bead quality, as shown in Figure 22.

Figure 22: (a) Bead with excessive powder feeding and insufficient powder feeding, (b) bead with excessive power causes the burnt mark around the bead, and (c) continuous bead dimension with optimum parameter. (d) Cross section of bead with excessive dilution, (e) cross section of bead with optimum dimension and dilution, and (f) cross section of bead with insufficient dilution.

The target printed bead width is the same as laser spot diameter, for 24Vx (diameter: 2.25mm) and 10Vx (diameter:0.8mm) nozzles, respectively. The printed bead should be straight and continuous without any unfused powder due to insufficient energy and burnt mark due to excessive energy. Separately, a minimum level of metallurgical bonding is required between subsequent layers, which can be quantified using a dimensionless parameter called dilution[80].

$$D = \frac{d}{h + d} \quad (12)$$

d: depth of the melt pool below substrate level (see Figure 22e)

h: height of material above substrate level (see Figure 22e)

A high energy input and low powder feed rate result in a high value of d. Conversely, low energy input and high powder flow rate result in high value of h. The optimum level of dilution should be kept around 10-30% for good metallurgical bonding. This is because a high level of dilution will result in keyholing, and low level of dilution may result in lack of fusion across layers. Both phenomena will inversely affect the mechanical properties of the printed component.

Wall printing

Upon obtaining the optimum power and mass flow rate of high-quality bead, we proceed to determine the actual layer height in the printing process via printed wall specimen, as shown in Figure 23. It is noteworthy that due to difference in thermal environment of bulk printing, the actual layer height may be different from h dimension we acquired earlier, despite the same process parameter is used. To begin with, we estimate the number of layers based on h dimension to construct a wall. We carefully monitor the deposition process to avoid any possible collision due to printed layer height > estimated layer height. This is because when actual wall height is more than estimated wall height, the stacking error will reduce the laser standoff distance layer by layer, resulting in interference between nozzle and printed part eventually. After a few iterations, the actual layer height can be obtained using the following formula:

$$\text{Layer height} = \frac{\text{Actual wall height}}{\text{Number of layers}} \quad (13)$$

Figure 23: Wall specimen used in layer height determination

Cube printing

A cube printing is made possible after obtaining all the material specific key process parameter through the earlier iteration. We then used the Autodesk software to generate tool path for printing. The tool path is serpentine raster which rotates clockwise 90° each layer, as shown in Figure 24a. We kept constant the other critical process parameter such as overlap ratio, flow rate of central gas and secondary gas. The powder feeding rate was adjusted accordingly to achieve the required layer height. Figure 24 b, c, d and e show cubes with 20mm x 20mm x 20mm dimension produced from two machines separately. We observed the improvement in dimension accuracy when a smaller bead width is used.

Figure 24: (a) Schematic illustrating the deposition strategy adopted (black arrow), red arrow (deposition direction) and the cross-sectional plan (X-Z) used to analyse samples microstructure. (b)(c) Sample produced using Trump machine, with bead width 3mm and 2mm respectively. (d)(e) Sample produced using BeAM machine, with bead width 2.25mm and 0.8mm respectively..

4.2 Effect of linear heat input and dwell time

We produced R-series samples to investigate effect of linear heat input and dwell time on the microstructure formation and mechanical properties. A total of 6 samples namely R1-R6 were produced. We set the dwell time as 5 minutes after every 4mm height of materials deposited. During dwell time, both laser and powder feeding rate were put on hold to allow parts to cool down. Separately, linear heat input is defined using following equation.

$$\text{Linear heat input, J/mm} = \frac{\text{Power, W}}{\text{Scanning Speed, mm/s}} \quad (14)$$

We varied linear heat input by changing scanning speed while keeping the power constant at 1600W. Since the mass flow rate was kept constant at 15 g/min, the layer height was expected to vary accordingly. Parameter sets with lower scanning speed are expected to yield thicker layer thickness due to more powder supplied per unit time. We then generated the tool path based on actual layer height obtained with wall specimen. Table 9 lists the parameter details for all 6 samples. The dwell time was introduced after every 4 layers being printed. All the R-series samples were characterized in as-built and non-heat-treated conditions.

Table 9: R-Series sample process parameter

Sample	Geometry	Power	Scanning Speed	Layer Height	Mass Flow Rate	Dwell Time	Linear Heat Input
Unit	mm	W	mm/min	mm	g/min	Yes / No	J/mm

R1	Cube specimen 20 x 20 x 20	1600	430	4.0	15	No	223
R2			430	4.0		Yes	223
R3			610	2.5		No	157
R4			610	2.5		Yes	157
R5			1070	2.0		No	90
R6			1070	2.0		Yes	90

Results

Figure 25 shows the relative density of R-series samples compared to the energy supplied during deposition. We plotted the results according to (a) linear heat input and (b) volumetric energy density supplied during deposition to understand the relationship if any. We found that higher volumetric energy density improves the relative density by reducing the maximum pore size (see Figure 26).

Figure 25: Relative density value in respect to (a) linear heat input and (b) volumetric energy

Figure 26 a to d compare R1-R6 sample microstructure as measured by EBSD. These maps show grain orientation along Z (the building direction) and reveal a non-uniform grain size and orientation distribution within and across all samples. We observed mainly $\langle 100 \rangle$ grains oriented along Z, except sample R5 and R6. Among group of samples without dwell time (Figure 26a, b and c), we observed a gradual refining of grain size and transition of $\langle 100 \rangle$ texture to random grain orientation along Z. In R1 sample (Figure 26a), we notice $\langle 100 \rangle$ columnar grains growing from bottom to top of melt pool in every layer. Furthermore, we observe randomly orientated equiaxed grains in between every deposited layer. This is consistent with findings from other researchers [21]. Compared with R1 produced using highest linear heat input, sample with lower linear heat input - R3 and R5 show a relatively smaller density of $\langle 100 \rangle$ grains along Z. We then performed texture strength analysis in terms of M.U.D. (Multiple of Uniform Distribution) for all samples. As shown in Figure 27, we observed a clear transition of grain orientation from $\langle 100 \rangle$ to $\langle 110 \rangle$ along Z for the sample with lower heat input.

Figure 26: EBSD grain orientation map for sample without dwell time (a) R1, (b) R3, (c) R5 and with dwell time (d) R2 (e) R4 (f) R5

There is no significant difference in microstructure, when comparing the set of samples processed with and without dwell time, as shown in Figure 26. There is only a marginal decrease in density of $\langle 100 \rangle$ grains along Z for the sample printed with dwell time, as shown in Figure 27. We noticed the same transition trend of grain orientation from $\langle 100 \rangle$ to $\langle 110 \rangle$ in samples processed with dwell time, from Figure 26 d, e, f and Figure 27 b, d, f.

Figure 27: Inverse pole figure (IPF) map along X, Y and Z direction for sample (a) R1 - LHI = 223J/mm (b) R2 - LHI = 223J/mm with dwell time (c) R3 - LHI = 157J/mm (d) R4 - LHI = 157J/mm with dwell time (e) R5 - LHI = 60J/mm (f) R6 - LHI = 60J/mm with dwell time.

To qualitatively compare grain orientation along different directions, we then plot the texture strength (MUD) of $\langle 100 \rangle$ oriented grains along all 3-specimen direction, namely Z (building direction), X (transversal direction) and Y (out-of-plane direction) in Figure 28. We found texture strength of $\langle 100 \rangle$ oriented grains along Z to reduce from 1.1 (R1 sample) to 0.4 (R5 sample), when linear heat input decreased by 50% as scanning speed nearly doubled. Conversely, texture strength of $\langle 100 \rangle$ grains along Y doubled from 0.4 to 1. There was no significant difference in M.U.D. of $\langle 100 \rangle$ grains, between sample produced with and without dwell time. The texture strength of $\langle 100 \rangle$ grains along X direction was nearly constant for all samples.

Figure 28: $\langle 100 \rangle$ Texture strength in different specimen direction X,Y and Z

To understand the change in grain sizes across all samples, we computed average area of grains on all samples from the EBSD data we acquired earlier. We then plot the computed value in Figure 29. We

Figure 29: (a) Grain size for R-series sample. (b) Grain size along Z in R1, R3 and R5 sample

found that grain size decreased $\sim 40\%$ from $25 \times 10^4 \mu\text{m}$ to $15 \times 10^4 \mu\text{m}$ with reduction of linear heat input across samples. Separately, to understand the grain changing behaviours within the sample, we plot the grain size along Z direction for R1, R3 and R5 sample in Figure 29. We notice the grain sizes fluctuate along Z, with a periodic pattern of every $\sim 2\text{mm}$ in R5 sample, $\sim 2.5\text{mm}$ in R3 sample and $\sim 4\text{mm}$ in R1 sample. In couple with grain orientation map in Figure 26, the area with smaller, randomly oriented grains are mainly located near the layer boundaries of all samples with different layer thickness (R1 & R2 – 4mm, R3 & R4 – 2.5mm, R5 & R6 – 2mm).

To understand the effect of measured microstructural difference on change in mechanical properties, we measured the hardness value of all sample by taking six micro-hardness measurements on every sample. Figure 30 shows that hardness is higher in the sample produced with lesser linear heat input, despite the large variation in every measurement. Besides that, we also observed sample processed with dwell time showed marginally higher hardness as compared to sample processed without dwell time.

Figure 30: Micro hardness across R-series sample

Discussion

In our experiment, we characterized the effect of linear heat input and dwell time on the formation of as printed microstructure. We found that decreasing in heat input via increasing scanning speed yield thinner layer thickness, when powder-feeding rate is kept constant. At the same time, it helped to produce a finer and relatively more homogenous microstructure. The reduction in layer thickness is due to lesser material supplied per unit time and it reduces the melt pool depth. The microstructure formation occurs in a layer-by-layer manner, during solidification of melt pool. Hence, finer microstructure (Figure 26) is observed in the sample with thinner layer thickness and smaller melt pool. In the sample with highest heat input (R1), the 100 grains mainly oriented along Z direction within the melt pool. This is because the alignment of grain growing axis with the $\langle 100 \rangle$ direction is often found in directionally

solidified FCC alloys, and controlled by the anisotropic crystal growth rate [54]. It is common to observe that grain orientations are selected by competitive grain growth in one of the six $\langle 100 \rangle$ preferred growth directions parallel or nearly parallel to the local heat flow direction for cubic materials [62]. However, the $\langle 100 \rangle$ grains in the R1 sample did not grow across the layer and were interrupted by fine, randomly oriented equiaxed grains. This interruption of grain growth behavior can be observed in other samples as shown in grain orientation map (Figure 26) and analysis performed (Figure 29). The formation of these equiaxed grains along layer boundaries is mainly due to insufficient re-melting of previously deposited layer. The partially melted solid powders on top of each layer then became the favorite heterogeneous nucleation sites for the formation of fine equiaxed grains [60]. Despite the high power (1600W) employed, the excessive amount of material supplied results in insufficient energy to fully melt the entire deposited layer. This can be further confirmed by lack of fusion defect observed near the layer boundaries in all samples (Figure 26).

The grain refinement trend observed across the sample is due to i) lesser linear heat input, ii) smaller layer thickness. Lesser linear heat input in processing reduces the heat built-up within layers due to lesser energy supplied to the melt pool. This improves the inter-layer cooling rate in the process, which eventually refines the microstructure formation within the layers. The introduction of dwell time allows more time for bulk part to cool down and improves the overall cooling rate. However, we do not see the effects (microstructure refinement) of such approach in our experiment results. This is due to the dominating factor for microstructure formation in our samples is cooling rate within each layer, instead of bulk component. We can observe the microstructure formation in the sample occurs within layers, and there is no significant variation along Z in all samples.

Layer thickness will determine the size of melt pool produced during deposition, which act as limiting factor of microstructural feature size. Since maintaining powder feeding rate will still result in different melt-pool sizes, followed by different layer thickness and microstructure formation while using separate set of process parameter, it makes more sense to maintain the layer thickness across sample via varying the powder flow rate. In fact, the current industrial practice is already making use of in-situ monitoring system, to vary the process parameter in keeping the layer thickness consistent across the process [15].

5 Mechanical and microstructural heterogeneity of I718 alloys

One of the benefits DED has as compared to other AM process is its high deposition rate. DED process can yield deposition rate as high as 2.2kg/h [81] as compared to PBF process 0.12kg/h [82]. However, higher deposition tends to yield the stronger microstructure heterogeneity in the sample [83]. The resultant heterogeneity in mechanical properties might have implication in the application using DED-made components. To better understand the effects of deposition rate on the resulting microstructure and mechanical performance, we produced samples in different sizes by varying the deposition rate (Figure 31).

Figure 31: Photograph of I718 (a) H sample (b) M sample (c) L sample and (d) R sample printed on a stainless steel 316L substrate

We built the samples directly on a 6 mm thick stainless steel 316L plate firmly clamped on the CNC rotating table of Trumpf TruLaser Cell 7040 machine with a three-jet nozzle and a 3 kW rating disk laser. We employed the same serpentine scanning pattern with the scan lines parallel to the sample diagonal, which we rotated by 90° each layer to alternate the scanning direction (Figure 24a). The produced cubic samples have identical geometry (Figure 31 a-c) but using different deposition rate, which is high deposition rate (H), medium deposition rate (M) and low deposition rate (L). We used industrial proprietary settings to produce these two samples, which led to “high” (H) and “low” (L) deposition rates of $11.4 \text{ mm}^3\text{s}^{-1}$ and $3.3 \text{ mm}^3\text{s}^{-1}$. To achieve such deposition rates and ensure full density samples, we optimized the DED parameters and used an energy density, E , of 50 J mm^{-2} and 35 J mm^{-2} for sample L and H, respectively. We define the energy density by:

$$E = \frac{\text{Laser power}}{\text{Scanning speed} \times \text{Laser spot diameter}} \quad (14)$$

We produced the rectangular (R) sample (Figure 31d) using the same process parameters employed for the H sample. We optimized the deposition parameters to achieve full density. The respective process parameters information is listed in Table 10. We heat treated H, M, L and R samples according to AMS 5663 prior to microstructure characterizations.

Table 10: Parameter information for H, M, L and R sample. The parameters provided here are only estimation based on energy density due to industrial proprietary issue.

Sample	Deposition rate, g/hr	Energy density, J/mm	Laser Power, W	Laser Spot Size, mm	Scanning speed, mm/min	Sample size, mm	Layer height, mm	Overlap, %
H	1200	48	1900	3	800	20x20x20	1.35	60
M	360	38	1000	2	800		0.75	
L	120	38	500	1	800		0.30	
R	1200	48	1900	3	800	40x30x30	1.35	

5.1 Grain structure and texture heterogeneity

Figure 32 compares the H, M and L sample microstructures as measured by EBSD. These maps—which show grain orientation along Z and Y—evinced a non-uniform grain size and orientation distribution in the H sample compared to the more homogeneous grain structure and texture of M and L sample. The H sample exhibits progressively coarser grains with stronger <100> texture along Z (Figure 32a). We also note the presence of “bands” of equiaxed grains interrupting the prevailing columnar, <100>-oriented grains with a periodicity that reflects the layer-by-layer deposition process due to the scanning strategy in use. Conversely, there’s no presence of ‘bands’ in both M and L sample and their microstructure appear to be relatively uniform. These qualitative trends are confirmed by our quantitative texture and grain size analysis in all 3 samples H, M and L along Z

Figure 32: EBSD grain orientation map along Z for (a) the H sample, (b) the M sample (c) the L sample. EBSD grain orientation map along Y for (d) the H sample, (e) the M sample and (f) the L sample.

To assess texture variations, we divide the EBSD data set from all 3 samples into 0.3 mm thick bins along Z. We select this bin size because it is smaller than the layer thickness in all 3 samples. In this way, we avoid averaging texture information across different layers and can study the occurrence of local differences in cooling rate as a function of the deposition strategy used. We compute texture strength in each bin by taking the highest Multiple of Uniform Density (M.U.D.) value in Z and Y direction for grain orientation along the $\langle 100 \rangle$, $\langle 110 \rangle$, and $\langle 111 \rangle$ direction. The higher the M.U.D value, the higher the spatial concentration of a specific grain orientation.

Figure 33: Z texture strength for (a) H, (b) M, (c) L samples and Y texture strength for (d) H, (e) M, and (f) L sample

The analysis in Figure 33a, b and c show that grains in H and M sample are mainly $\langle 100 \rangle$ oriented along Z. Conversely, grains are mainly in $\langle 110 \rangle$ orientation along Z for L sample. We also observed that the $\langle 100 \rangle$ texture strength along Z doubles from bottom to top of the H sample (Figure 33 a), while it does not vary significantly in the M and L sample (Figure 33 b, c). Figure 33 a also clearly highlights the texture “banding” which we observe in Figure 2a. Interestingly, there seems to be an inverse correlation between the $\langle 100 \rangle$ and the $\langle 110 \rangle$ texture. In other words, the microstructure “bands” of smaller grains are characterized by a relatively strong $\langle 110 \rangle$ texture. We observed no such periodicity in both M and L sample.

Figure 33d, e and f show grain orientation of three samples along Y(out-of-plane) direction. In H sample, $\langle 110 \rangle$ orientation has the highest M.U.D as compared to the other two orientations. By comparing Figure 33 a and d, we observed a correlation of $\langle 100 \rangle$ and $\langle 110 \rangle$ texture grains along Z with $\langle 100 \rangle$ and $\langle 111 \rangle$ texture grains along Y. Though M sample exhibits a more consistent texture strength for all three orientations, we observed grains in L sample are mainly composed of $\langle 100 \rangle$ texture.

Figure 34: Grain diameter of (a) H sample, (b) M sample, (c) L sample along Z. Grain diameter of (d) R sample along Z across different X location.

To assess grain size variations along Z in all samples, we computed the equivalent circle diameter (ECD) of grains and plot the values in Figure 34. We noticed grain coarsening in samples R (from $\sim 94 \mu\text{m}$ at the bottom to $\sim 116 \mu\text{m}$ at the top of the build) and H (from $\sim 100 \mu\text{m}$ at the bottom to $\sim 115 \mu\text{m}$ at the top of the build), but no significant difference in sample M (from $\sim 58 \mu\text{m}$ at the bottom to $\sim 60 \mu\text{m}$ at the top of the build) and sample L (from $\sim 60 \mu\text{m}$ at the bottom to $\sim 65 \mu\text{m}$ at the top of the build). The analysis also shows that the average grain size in samples H and R is comparable and $\sim 60\%$ larger than that in sample M and L. Indeed, when comparing the grain coarsening trends in samples H and R over same length (i.e., up 20 mm), we compute the same increase of 15% in the two samples. This result is

rationalized using of same process parameters to produce both H and R samples, which led to higher deposition rates (and thus higher energy input) compared to those used for the M and L sample. Hence, H and R samples experienced similar thermal history during processing. Moreover, we notice that grain size variations in sample H reflect the presence of the microstructure “bands” seen in Figure 32 a and b, confirming the smaller average grain size in those regions. The “banding” is not visible in Figure 34d because of the sparse data points we collected from upper circular surface of cylinder extracted from sample R (Figure 35).

Figure 35: (a) CAD Drawing showing compression cylinders distribution in R sample. (b) EBSD grain orientation map along Z for the upper circular surface (parallel to the XY plane) of the compression cylinders.

The corresponding EBSD data set for this sample is shown in Figure 35. Here, we acquired EBSD scans from the top surface of each individual compression cylinder, taken from different locations across the R sample (Figure 35a). Qualitatively, the grain structure in these maps matches the trend shown in Figure 34d. We also find a consistently smaller average grain size along X—with the right-hand side of the sample in the cross-section exhibiting the smallest value—and a change in texture from $\langle 100 \rangle$ (at approximately $X = 10\text{mm}$) to random (at approximately $X = 20\text{mm}$).

Our texture and grain size results are overall consistent with the findings on DED I718 by Parimi et al. [21] who reported complex microstructures consisting of regions of fine, randomly oriented grains in the lower part of the build—closer to the cold substrate—and regions of coarse, columnar grains with strong $\langle 100 \rangle$ texture parallel to Z in the higher part of the build. The alignment of grain growing axis with the $\langle 100 \rangle$ direction is often found in directionally solidified FCC alloys, and controlled by the anisotropic crystal growth rate [54].

5.2 Compositional heterogeneity

Besides the differences in grain structure and texture, we also investigated variations in the spatial distribution of the intragranular phases and precipitates that are commonly found in I718 [27]. The as-printed inter-dendritic structure is dominated by a Nb-rich phase referred to as Laves phase, which forms upon coalescence of γ -matrix dendrites during solidification of the melt pool. Upon heat treatment, the Laves phase decomposes and a fraction of Nb diffuses into the dendrites to form γ'' precipitates, which is the well-known strengthening precipitate [84]. The Nb fraction that remains in-between dendrites leads to the formation of small clusters of aligned platelets of δ - Ni_3Nb . This phase inherits improved corrosion resistance brought about by Nb, which makes it more resistant to chemical attack compared to the γ -matrix. For this reason, δ platelets are readily visible protruding from the inter-dendritic area across the sample surface after chemical etching (as shown in Figure 36).

Figure 36: Scanning electron microscopy images showing the local H (a, b, c), M (d, e, f), and L (g, h, i) sample surface along Z from bottom of the build (a, d, g), middle (b, e, h) and top (c, f, i). Sample schema (j) showing the microstructure at different regions.

Using this preparation technique, we qualitatively observe the precipitate geometry and distribution in sample H, M and L by scanning electron microscopy (SEM). We find a progressive increase in density and size of δ platelets along Z on the H sample. δ platelets are hardly observed at the bottom of the build but clearly visible at the top of the build, as shown in Figure 36a, b, c. Conversely, the M and L samples show a uniform distribution of δ platelets along Z (Figure 36d-i). To investigate the origin of this change in precipitate structure in the H sample, we take EDS line-scan measurements along individual dendrites (see inset in Figure 37) to assess the composition of the γ -matrix across the longitudinal and transverse direction in all samples. We take a matrix of such measurements uniformly spaced in X and Z every 4 mm and 2 mm, respectively. We compute the average Nb concentration along X and Z and plot them along both directions (Figure 37). While we observe no obvious difference of Nb concentration along X direction, we note that Nb concentration follows different trends along Z direction in all samples. We correlate these trends with spatial variations in the density and size of δ platelets and use them to estimate the amount of Nb in solid solution as well as that which leads to the formation of γ'' precipitates in the γ -matrix, which are not resolvable by means of SEM. Because the mean Nb content—which includes the Nb contained in δ platelets, as well as that within the dendrites—is constant throughout the builds and equals the nominal concentration in I718 (Table 2), the fraction of inter-dendritic δ phase must be inversely proportional to the Nb concentration within dendrites and, in turn, to the fraction of nano-sized precipitates that form during the aging treatment. In other words, regions with higher (respectively lower) Nb in solid solution exhibit sparser (respectively denser) distribution of smaller (respectively larger) δ platelets (Figure 36a, b, c). In the H sample, we measure more than 4 wt% of retained Nb in the γ -matrix near the bottom of the build and less than 3 wt% at the

Figure 37: Nb content change in the γ -matrix (a) along Z and along (b) X as measured along dendrites (as indicated by the white arrow in the inset map) by EDS in both the H, M and L sample. The inset shows a representative EDS Nb map on the H sample surface approximately 16 mm from the base plate. Nb is mainly concentrated in the inter-dendritic regions.

top of the build, as shown in Figure 37. By contrast, M and L sample exhibits a more consistent mean Nb concentration along Z.

We attribute the compositional gradient and microstructure differences in samples H, M and L to changes in cooling rate (CR) brought about by the high deposition rates, which amplify the thermal build-up throughout the sample and drive higher Nb segregation in the inter-dendritic region as the build progresses. Since we produced samples H and R using the same process parameters, we expect the latter to exhibit similar trends in CR (Figure 39), microstructure (Figure 35), and composition. However, the sparse data set and large size of the R sample prevented us from carrying out a detailed analysis as we did for the other two samples.

Figure 38: (a) Secondary electron micrograph showing PDAS structure in $\langle 100 \rangle$ grains near the bottom of H sample. The red arrows indicate locations for measurement of PDAS. Positions for PDAS measurements in grains $\langle 100 \rangle$ oriented in (b) Z-orientation map, and $\langle 110 \rangle$ oriented in (c) Y-orientation map in H sample.

To evaluate the relative changes in CR quantitatively, we measure the primary dendrite arm spacing (PDAS) by means of SEM, in H and R sample (Figure 34a). For H sample, we perform a total of 34 sets of PDAS measurements on different grains with similar orientation (Figure 38b, c). Specifically, we select grains with $\langle 100 \rangle$ orientation along Z and $\langle 110 \rangle$ along Y, such that the dendrite spacing can be measured directly without correcting for perspective error. This is achieved by extracting the XY coordinate of these grains from the EBSD raw data, follow by navigating to respective position for PDAS measurement using SEM. In R sample, we perform the same PDAS measurement on top of the compression sample. Figure 39 shows that PDAS increases along Z, indicating a progressive decrease in CR as more material is deposited. To quantify the CR from PDAS, we use the empirical relationship [28–30]:

$$\text{PDAS} = K(\text{CR})^{-n} \quad (15)$$

where K and n are material dependent constants, which reported to be 130 and 0.33, respectively [31]. The resulting change in CR along Z in the H sample is plotted in. In H sample, we compute a difference in CR from $\sim 2800\text{K/s}$ at the start of the build to $\sim 100\text{K/s}$ at the end of it. For R sample, the difference ranges from $\sim 3800\text{K/s}$ to $\sim 600\text{K/s}$. This result corroborates the hypothesis that the local thermal environment changes more abruptly at the beginning of the build because of the high heat extraction rate of the substrate and the high heat input employed during high deposition rate DED builds. Assessing PDAS in grains with similar orientation and computing CR for the M and L sample is difficult because of the finer grains as compared to the H sample. However, random measurements across the surface of sample M and L suggest that PDAS is more consistent across the build and of the order of $5\text{ }\mu\text{m}$. These differences in CR within and across the samples correlate well with the spatial heterogeneity in microstructure and composition reported in Figure 32, 30, 31.

Figure 39: Variation of PDAS along Z in (a) H-sample (c) R-sample and corresponding CR for (b) H-sample and (d) R-sample.

5.3 Mechanical heterogeneity

To quantify the effects of the measured microstructural and compositional heterogeneity on the alloy mechanical properties, we probe variations in the local hardness on all 4 samples along Z using micro- and nanoindentation (Figure 40 and Figure 41). In the H sample, the measurements indicate that hardness is highest near the start of the build—close to the base plate—but decreases progressively moving away from it. Conversely, the dispersion of the measured hardness increases along Z (Figure 40a). The relative change in local hardness of the H sample is striking when comparing histograms of nanoindentations taken near the base plate with those near the top of the sample (Figure 41). The average hardness varies by 3.5% and 9% in micro- and nanoindentation measurements, respectively. In both measurements, the standard deviation of the dataset more than doubles from bottom to top, indicating a general increase in the dispersion of the local hardness. We observe the exact same trends when probing the micro-hardness in the R sample (Figure 40), with the stark difference that the hardness value plateaus towards the top region of the build. This plateau suggests the establishment of a CR steady-state, as shown in Figure 39d. From the bottom of the sample to when the hardness value reaches a plateau (at around $Z = 20$ mm), we measure an average micro-hardness variation of $\sim 8.8\%$.

Figure 40: Plot of microhardness in (a) H-sample (b) M-sample (c) L-sample (d) R-sample as a function of distance from build plate.

As expected, the more uniform microstructure and composition in both M and L sample yields no significant variation in hardness (Figure 40b, c). The measured hardness values lie within the same margin of error throughout the whole build. Moreover, the dispersion of individual measurements is also relatively constant. The smaller variation between measurements in this sample suggests more consistent hardness along Z. This greater uniformity has implications on the macroscopic mechanical performance of the entire part.

Figure 41: Nanoindentation hardness histograms in H-sample

Grain size and crystallographic texture are known to affect the yield strength and elastic/plastic anisotropy of metal alloys [85]-[86][87]. To investigate how large-scale variations in texture and grain size affect the bulk mechanical properties in DED I718 we plot and analyze the engineering yielding point and elastic modulus of the R sample along Z and X (Figure 42a, b respectively). Owing to its large size, the R sample is ideal to perform series of compression tests and assess these properties at a larger scale compared to what is achievable by indentation tests. We computed the average value of Young's modulus from 4x loading and unloading cycle. We took 0.2% yielding value from the first loading only because subsequent loading and unloading cycle had strengthen the test cylinders and resulting in a higher value. We found both strength and modulus of elasticity to decrease along Z, but to increase along the X-axis. In contrast to hardness, we record no appreciable plateau in either properties along Z. The decrease in strength along Z matches the grain size variation seen in Figure 34d according to the well-known Hall-Petch relationship (discussed in detail in the following section) [81]. At the bottom of the build, we observe no difference in strength along X, which reaches a value of ~980 MPa (the highest measured in our experiments). In this region, the high CR resulting from the large heat sink effect associated with the base plate drives the formation of fine grains of similar size (Figure 34d) with high concentration of Nb in solid solution (assuming the measurements in Figure 37 can be extended to sample R). However, differences in yield strength along X become more prominent as the distance increases from the base plate (Figure 42a). In general, we find the yield strength value at the right-hand

side of the sample cross-section ($X = 20$ mm) the highest. This result is in line with the average grain size measurements at different X -coordinates along Z (Figure 34d).

We attributed the decrease in Young's modulus along Z and X (Figure 42b)—which we compute from the unloading of compression cylinders—to the variation of texture across the sample. Along Z , we observed 19%, 15%, and 4% decrease in Young's modulus for $X = 10$ mm, $X = 15$ mm, and $X = 20$ mm, respectively. The large difference in Young's modulus found at $X = 10$ mm (i.e., the left-hand side of the R sample cross-section) was probably due to the combined effect of the texture transition from random to $\langle 100 \rangle$ and the grain structure transition from equiaxed to columnar (Figure 35). Along X , the Young's modulus computed from measurements at $X = 20$ mm is the highest and most consistent. We attribute this trend to the random grain orientation and equiaxed grain structure in this region of the R sample.

Figure 42: (a) Engineering yielding of the compressed pillars along the build direction Z (b) Young modulus along Z , averaged from the four loading unloading cycles within each stress strain curve. (c) Stress strain curve for 4x loading and unloading cycle.

5.4 Discussion

5.4.1 Heterogeneity in the building directions

In DED processes, parts naturally heat up during production. Conduction to the substrate contributes a significant fraction of the total heat removal, and the path to the substrate grows together with the build while the heat input remains constant [42]. When I718 is deposited at higher deposition rates, parts can be glowing red or orange upon completion, roughly corresponding to temperatures exceeding 700°C. This temperature increase has direct effects on the microstructure. First, it acts as a cyclic heat-treatment on the early parts of the build, coarsening their microstructure as subsequent material is added, which is clearly observed in both H and R sample produced with same deposition strategy. A second consequence is the change in thermal environment around the melt pool, which affects the CR. As the solid region around the melt heats up, the CR diminishes resulting in the formation of larger grains with stronger crystallographic texture [58], [88]. Conversely, the more fine-grained areas are associated with regions of high CR. These regions are more prevalent towards the lower part of the build—which is closer to the cold substrate—or in certain layers, where the area had the longest time to cool down.

These phenomena can be readily observed in the H and R sample, which exhibits progressively stronger $\langle 100 \rangle$ texture and coarser grains along Z (Figures 2a). Our microstructure analysis of the H sample (Figure 3a and Figure 4a) confirms a higher density of $\langle 100 \rangle$ textured grains with larger equivalent circular size near the top. This similar trend is observed in R sample too (Figure 34d and Figure 35b). The higher heat accumulation rate towards the top of sample H—which leads to lower CR—promotes re-melting of the previous layers, which in turn leads to epitaxial growth of columnar grains with their fastest growth direction parallel to Z. These effects become less pronounced in large-scale parts because of the faster heat dissipation, which comes from the thicker cross section in X-Y plane providing more effective heat conduction towards build plate. Indeed, the smaller PDAS measured in R sample indicate higher cooling rate (Figure 39) compared to H. We still observe the decreasing trend of cooling rate, but it is more moderate as compared to H sample. Conversely, the M and L sample exhibits smaller and more consistent PDAS along Z, indicating higher but more uniform cooling rate and hence lesser heat accumulation.

Beside the microstructure coarsening and strengthening of the $\langle 100 \rangle$ texture along Z, using high deposition rates in DED I718 also leads to peculiar microstructure “bands” consisting of fine grains with different texture (in this case $\langle 110 \rangle$), which interrupt the columnar growth with a periodicity of about two layers. We attribute the origin of the “bands” to serpentine scanning strategy used during DED. With reference to Figure 24a, and calling A, B, C, and D the four corners of each layer, when a first layer is deposited the serpentine raster starts at point A and ends at point C. Upon completion of the layer, this raster strategy leads to the establishment of a transverse thermal gradient, with point A (the colder) having cooled for longer than point C (the hotter). When the second layer is deposited the

serpentine raster rotates by 90° and moves from point B to point D. In this second layer, points A and C lie along the same scan line and thus are printed almost simultaneously, halfway through the layer onto areas with different temperatures (i.e., points A and C in the first layer). Conversely, B and D are the first and last point printed in the second layer, but they were printed nearly simultaneously in the first layer. In this example, A and D would have cooled for one and a half layers of printing time while B and C were covered by the next layer after only half a layer was printed. That results in a factor 3 difference in cooling time within a single layer. As additional layers are added, each corner will have two long cooling times followed by two much shorter cooling times, which would result in possible microstructure heterogeneity with a repeat unit of four build layers. However, the thickness and extent of the finer, randomly textured “bands” decreases as the sample grows because of the prevailing thermal build-up along Z. Similar “banded microstructures” were observed by Parimi *et al.* [21] in DED I718 thin walls, and were attributed to the inter-layer idle time difference when using unidirectional and bidirectional scanning strategies. In our samples, the “banded” grain structure and texture are the result of sample geometry (a cube rather than a thin wall) and the high thermal input employed, which enhances differences in thermal build-ups across different layers even though the scanning strategy is kept constant throughout the build. We observe no such large-scale periodicity in the M & L sample, where the lower laser power minimizes the effect of thermal build-up. The “banding” in sample H is not to be confused with that which arises from compositional, or phase changes found in the heat affected zone in DED samples [51]. The optical micrograph in Figure 71 (Appendix 1) shows the individual melt pool boundaries across sample H. This micrograph indicates no presence of “bands” that could be attributed to the heat affected zone. Moreover, if the “bands” we find in this sample were associated with the heat affected zone, they should appear at the interface between each deposited layer and not with a periodicity of two layers.

The fact that the “bands” exhibit a $\langle 110 \rangle$ texture is also an interesting observation. A possible reason for this texture transition may be that these regions have cooled down for longer compared to the inter-band regions of the sample. As a result, the local thermal gradients within the melt pool may be more sensitive to the melt pool geometry than to the prevailing heat flux along Z. Several researchers have shown that in these cases the local thermal gradients may grow at an angle with respect to the build direction and lead to the establishment of a $\langle 110 \rangle$ texture along Z [64][89]. The reason why these bands in H sample appear to have a more equiaxed grain structure compared to the $\langle 100 \rangle$ -textured regions could also be attributed to the growth direction of these grains. Following the tilted thermal gradients, grains in these “bands” may grow out of the sample (X-Z) plane at an angle and thus be imaged along their cross-section. Similarly, we also observed the smaller, equiaxed grains in L sample exhibit the same $\langle 110 \rangle$ texture along Z with a $\langle 100 \rangle$ texture along Y. Using the same principle for grain growth behaviour, the grains in L sample may grow perpendicular out of the sample (X-Z) plane. This is due

to the change in overall heat flux from along Z to other directions as a result of different processing parameter set.

The change in CR along Z described in Figure 39 contributes not only to large-scale changes in grain structure and crystallographic texture; it also affects the local elemental distribution in the alloy. A lower CR gives more time for partitioning of alloying elements into dendrites (as seen in the inset in Figure 37) decreasing solute content in the inter-dendritic regions [80]. This effect should be most pronounced in high melting point alloying elements such as Nb (melting point 2477°C), which have the greatest partition coefficient at the solid-liquid interface [90]. Although the overall composition of I718 is consistent throughout the part (as confirmed by EDS analysis), the local Nb content in the γ -matrix varies measurably along Z in the H sample (Figure 37) and results in hardness variation along Z. We also measure the concentration of other solute elements (such as molybdenum, aluminum, and titanium) along Z using EDS, but find no significant variation (see Figure 72 in Appendix 1). We measure the same hardness variation in the R sample too. However, in this sample the hardness value plateaus beyond a height of 15 to 20 mm. This result suggests that the thermal build up does not increase indefinitely throughout the entire deposition, but eventually reaches a steady state beyond 20mm (Figure 39b, d).

Figure 43: (a) The relationship between micro hardness and grain diameter (b) The relationship between micro hardness and texture (c) The relationship between micro hardness and local Nb wt% in the γ -matrix. (d) The relationship between micro hardness and PDAS.

To investigate the combined effect of this multiscale microstructure heterogeneity on the alloy mechanical properties, we plot hardness versus grain size, texture, PDAS and Nb content in the H

sample in Figure 43a, b, c and d, respectively. These plots indicate no direct correlation between variations in grain size and texture (taken along the direction parallel to the loading axis) with hardness. By contrast, we find strong correlation between the hardness trend and variations in Nb concentration and PDAS. To estimate the relative contribution of these microstructural features on the alloy strength, we consider the strengthening mechanisms associated with the grain boundary fraction (σ_{GB}), solute concentration in solid solution (σ_{SS}), precipitates ($\sigma_{\gamma'}$ & $\sigma_{\gamma''}$), and dendrite spacing (σ_{PDAS}). To this end, we use the yield strength model established by Shuya Zhang [96] and infer hardness across our sample. We provide details on the model in the Appendix 1 and report the main results in Table 11. Among the different microstructural features, we find the precipitate content and Nb concentration in solid solution to contribute the most to the variations in hardness along Z. While a progressively larger grain size is conducive to a decrease in hardness (following the Hall-Petch relationship), our calculations predict that grain size variations accounts for only 11% difference in hardness from bottom to top of sample H. This result confirms the lack of correlation seen in Figure 43: (a) The relationship between micro hardness and grain diameter (b) The relationship between micro hardness and texture (c) The relationship between micro hardness and local Nb wt% in the γ -matrix. (d) The relationship between micro hardness and PDAS.. Moreover, we estimate the contribution stemming from the increasing size of dendrites to account for 22% of the hardness variation. It is well known that the solidification structure—and specifically the size of cells or dendrites—plays an important role on strength in materials produced by fusion-based AM processes [97][98]. The strengthening effect is thought to arise from dislocation pile ups and solute-enriched chemical boundaries found at cells/dendrites boundaries, which hinder plasticity. In general, the dislocation density at these sub-grain structures can be estimated from the cell or dendrite spacing and the corresponding strengthening contribution can be computed using the Bailey-Hirsh relation [96]. In our sample H, the relatively large dendrite size ($>10\mu\text{m}$) is probably the reason why the contribution from σ_{PDAS} to the hardness variation is not a major one. By contrast, we find the strengthening mechanisms linked to Nb concentration in the γ matrix to be the dominant ones, contributing to 68% of the overall hardness variation. Nb acts to strengthen the Ni-base alloy either when in solid solution [105], or when it forms secondary-phases—namely the γ'' and δ precipitates—after heat treatment. The high σ_{SS} value is intuitive when considering that Nb has close to the largest lattice mismatch with Ni of any element that disperses into a substitutional solid solution (+18 %). In addition, $\sigma_{\gamma''}$ is widely reported as the main strengthening mechanism at play in I718. As the CR decreases, we measure a reduction in the Nb concentration within dendrites (by EDS) and, in turn, we speculate a lower volume fraction of γ'' . Therefore, we ascribe the progressively lower hardness along Z to the combined decrease in σ_{SS} and $\sigma_{\gamma''}$; which are directly linked to the variations in Nb concentration arising from the change in CR along Z. We also believe that the progressive increase in δ precipitates along Z has negligible effects on the sample hardness, as previously reported by other researchers [87, 88].

Table 11: Hardness contribution in H sample by intrinsic strength σ_0 , grain boundary strengthening σ_{GB} , solid solution strengthening σ_{SS} , dendrite spacing strengthening σ_{DAS} , γ'' strengthening $\sigma_{\gamma''}$, and γ' strengthening $\sigma_{\gamma'}$.

Hardness		$\Delta \text{Hardness}_{\text{Top-Bottom}}$	
H sample	Contribution	Δ GPa	Contribution
σ_0	5%	0.00	0%
σ_{GB}	8%	0.02	11%
σ_{SS}	31%	0.03	15%
σ_{PDAS}	1%	0.04	22%
$\sigma_{\gamma''}$	41%	0.11	53%
$\sigma_{\gamma'}$	15%	0.00	0%
σ_{hardness}	100%	0.20	100%

5.4.2 Heterogeneity in transversal directions

Besides these complex microstructure changes along Z, we measure substantial differences in texture and grain size along the transversal direction too (corresponding to the X-axis in Figure 35a). These changes are best appreciated in the R sample owing to its larger size. We believe that this transversal microstructure gradients stem from a variable thermal build-up along the X axis, which results from the off-cantered position of the build onto the substrate, couple with the asymmetric ambient conditions on both side (an exhaust was installed right next to deposition regions for fume extraction). Indeed, as shown in Figure 35a, material deposition started at 12 mm and 34 mm from the substrate corner along X and Y, respectively. Since the thermal mass of the substrate is asymmetrically distributed around this build, it may induce an equally asymmetric thermal flux—and thus CR—along the transversal direction. As a result, we find larger, columnar grains with stronger $\langle 100 \rangle$ texture at left-hand side of the R sample (from where we took the compression samples color-coded in purple in Figure 35a) compared to the right-hand side column (color-coded in blue in Figure 35a).

To investigate the effects of these transversal gradients (both in texture and grain size) on the mechanical behaviour of the build, we correlate the grain size with yield strength and estimate the Hall-Petch hardening effect expected from the grain size reduction, shown in Figure 44. Interestingly, we observe that a linear Hall-Petch relationship cannot fit all data points at once. Instead, we need use three separate linear fits on the three sets of data points coming from the left, central, and right column of compression cylinders taken from the R sample. Noteworthy is that the Hall-Petch slope decreases from the left-hand side to the right-hand side of the sample. We attribute this result to texture variations along the transversal direction, which have shown to affect the relationship between yield strength and grain size. Godon *et al.*[93] for instance, showed that a randomly textured microstructures yield lower Hall-Petch slope compared to textured ones due the fundamentally different deformation mechanisms at play at grain boundaries. Random textures comprise larger number fraction of high-angle grain boundaries, which promote the formation of larger dislocation pile-ups and strain gradients. By contrast, they attributed a higher Hall-Petch slope in textured microstructures to a lower density of dislocation

sources—because of the lower fraction of high-angle grain boundaries—as well as to limited grain boundary shearing of the copious low-angle grain boundaries. Another possible interpretation of the mechanical anisotropy found along X is the presence of microstructure “bands” in sample R (similarly to those found in sample H), which are more prominent on the right-hand side of the build (Figure 31a). Kong *et al.*[98] suggested that localized microstructure heterogeneities such as these “bands” may induce stress buildups in AM component. This additional strengthening mechanism—which is less prominent within the strongly texture and more uniform microstructure found at the left-hand side of the sample—could also contribute to lowering the Hall-Petch slope along the X-axis. We are investigating the localized deformation of these microstructure more in detail and plan to present the results in a follow up study.

Figure 44: Hall-Petch law computed by correlation of the yielding data and average grain diameter on R sample

6 Effect of sample size on cooling rate

We carried out the deposition using BeAM Modulo machine and selected 2 sets of different process parameters (X1 and B) to study the effect of sample geometry (cube, cuboid and block) on the resulting microstructure. All the samples were deposited on a 6mm thick base plate except X1-block sample, which we deposited on a 20mm thick plate to avoid possible warpage due to the high amount of total heat input in the process and its size (see Figure 45).

Figure 45: Illustration of (a) cube and (b) cuboid sample printed using B process parameter, together with (c) cube, (d) cuboid, and (e) block sample printed using X1 process parameter.

Table 12 illustrates detailed process parameters employed for sample production. We used 24Vx nozzle for parameter set A and 10Vx nozzle for parameter set B. We employed vertical serpentine strategy, with deposition direction parallel to the side of the sample and rotate by 90 degrees every layer. We removed all samples from the base plate and cut them into slices using EDM. We then characterised all samples in as-printed conditions.

Table 12: Process parameter set for sample with different geometry.

Sample	Power, W	Scanning Speed, mm/min	Layer height, mm	Overlap distance, mm, overlap, %	Nozzle	Sample Geometry, mm		
						H	W	L
X1- Cube	1600	430	1	0.75, 33	24Vx	20	20	20
X1- Cuboid						80	30	30
X1- Block						20	60	100
B - Cube	400	1500	0.33	0.48, 60	10Vx	20	20	20
B - Cuboid						80	30	30

6.1 Effect of cooling rate on grain orientation

Figure 45 illustrates the cube, cuboid and block samples microstructure as measured by EBSD. Despite the same process parameter employed for sample production, we found differences in grain size and orientation distribution in X1-series samples (Figure 45 a, b and i), as compared to B-series samples. The X1-cube sample shows strongest $\langle 100 \rangle$ texture, whereas the X1-cuboid and X1-block samples show relatively weaker $\langle 100 \rangle$ texture strength along building direction. The $\langle 100 \rangle$ textured grains in X1-cube are spanning across the entire sample and parallel to the building directions. Conversely, grains in X1-cuboid are more random in orientation and we observed grains in X1-block sample aligning in a zig-zag pattern along building directions, which reflects the layer-by-layer deposition process due to scanning strategy in used. It is interesting to note the existence of fine, equiaxed like “bands” near the top of block sample. The observed “bands” are much thinner than layer thickness ($<1\text{mm}$) used for sample production. While analysing the grain orientation along Y directions (Figure 45 e, f and j), we found strong $\langle 110 \rangle$ texture grains along building direction in X1-block sample, and relatively random texture in both X1-cuboid and X1-block. On the other hand, we observe a rather similar grains orientation distribution in both B-cube and B-cuboid samples. Along building directions, both samples are characterised by strongly $\langle 110 \rangle$ grains across entire sample whereas grain along Y directions are mainly $\langle 111 \rangle$ textured.

We computed grain area in μm^2 to assess the grain size variation across all samples. The grain size analysis shows that X1-cube has the largest average grain area ($36.3 \times 10^3 \mu\text{m}^2$), whereas the average grain area of both X1-cuboid and X1-block sample are similar ($\sim 21.5 \times 10^3 \mu\text{m}^2$). We found no significant grain size difference ($\sim 3.8 \times 10^3 \mu\text{m}^2$) in B-cube and B-cuboid samples.

Figure 46: EBSD grain orientation amp along Z for (a) X1 (b) X1-cuboid, (c) B, and (d) B-cuboid and (i) X1-block. EBSD grain orientation amp along Y for (e) X1 (f) X1-cuboid, (g) B, and (h) B-cuboid and (j) X1-block.

To investigate the origins of the observed microstructure differences, we measured average primary dendrite arm spacing (PDAS) in all samples. We performed a total of 10 sets PDAS measurement in each sample and use the measurements to estimate the cooling rate using Equation 15. We found X1-cube sample has the largest average PDAS ($\sim 16 \mu\text{m}$) with lowest cooling rate ($\sim 600\text{K/s}$). The measurement shows the X1-block sample has an average PDAS of $9 \mu\text{m}$ with higher cooling rate ($\sim 4000\text{K/s}$). While comparing the PDAS measurement across different location in X1 block sample, we find centre position has the finest microstructure, as compared to both ends of the sample. The estimated cooling rate from PDAS measurement is lower in both ends ($\sim 3000\text{K/s}$) and higher in centre regions ($\sim 6000\text{K/s}$). Conversely, PDAS in both B-cube and B-cuboid sample are rather similar in the range of $5 \mu\text{m}$ with much higher cooling rate.

Figure 47 PDAS of X1 block sample along Z direction.

The difference in cooling rate across different positions in X1-block sample is due to the combined factor of scanning strategy and sample geometry. The serpentine scanning strategy employed is 90 degrees across alternating layers. As illustrated in Figure 48, during printing of layers where depositing directions is parallel to the X-axis of block sample, it introduces a difference in time interval for a specific point to be reheated by moving heat source again. As a result, cooling interval at different locations are non-invariant. In this example, the time interval at point A (near the centre of block) is much longer than point B (near the edge of sample) due to longer travel distance by moving heat sources. The longer time interval between subsequent reheating by moving heat source allow more time for the part to cool down. Then, the lower temperature substrate (in this case the previous deposited layer) results steeper thermal gradient between the melt pool and increase the cooling rate.

Figure 48: Illustration of location dependent cooling along X-axis

The higher cooling rate in X1-block sample is a result of sample geometry. Firstly, due to the bigger cross-sectional area (X-Y plane), it provides a bigger conduction path towards the build plate for more effective heat loss. As a result, thermal build-up in the sample is lesser and the block temperature is lower. During the deposition of subsequent layers, the higher thermal gradient results in a higher cooling rate and promotes the formation of a finer microstructure. Secondly, the time interval between moving heat source reaches the same point is longer in X1-block sample due to huge cross-sectional area (100mm x 60mm). As compared to X1-cube sample, where the small cross-sectional area (20mm x 20mm) greatly reduces the time interval for moving heat source return to same position, X1-block sample has longer cooling interval before subsequent deposition, which increases the cooling rate as well. It is noteworthy that this effect is less pronounced in the process with high cooling rate (sample printed using B process parameters) as we do not observe microstructural changes across B-cube and B-cuboid sample. The cooling rate are known to affect the precipitation behaviours of secondary phases, we then selected X1-cuboid and B-cuboid to study the cooling rate dependent segregation rate and their effect on mechanical properties.

6.2 Cooling rate dependent segregation

As-printed microstructure

The microstructure of X1-cuboid and B-cuboid samples are shown in Figure 49. We found irregular white particles segregated along inter-dendritic regions in both samples. These white particles are Laves phase formed as a result of non-equilibrium solidification conditions during the DED process. Laves phase is a brittle intermetallic phase with topologically close-packed (TCP) hexagonal structure. The presence of Nb- rich Laves phase is known to inversely affect the mechanical properties of part, as it depletes the necessary constituent for $\gamma''(Ni_3Nb)$ strengthening phase in precipitate-hardened I718 [99]. While comparing the Laves phase morphology in both samples, we found Laves phase in sample A is much bigger (~micron size) than those in sample B (~nano size). The Laves phase in A sample also tends to connect with each other to form long striped Laves phase. Conversely, Laves phase in sample B are distributed more uniformly. We measured dendrite spacing in both samples and estimated the cooling rate using formula $PDAS = K(CR)^{-n}[100][101][102]$, where PDAS is primary dendrite arm spacing, K and n are material constant. We found the cooling rate of X1-cuboid ($10^3 K/s$) is much lower than B-cuboid ($10^4 K/s$). The grain morphologies are shown in Figure 49c and d. In both samples, columnar grains growing epitaxially along building directions can be seen, with average grain size of A ($22000 \mu m^2$) is much bigger than B ($6800 \mu m^2$). Additionally, the XRD results shows the presence of MC carbides in X1-cuboid sample, where M is either Al, Nb or Ti.

Figure 49: The SEM micrographs of sample (a) X1-cuboid and (b) B-cuboid showing micro and nano size Laves phase. The EBSD grain orientation map showing grain morphology of sample (c) X1-cuboid and (d) B-cuboid along building directions. (e) XRD results of as-printed X1-cuboid and B-cuboid sample

The chemical formula of Laves phase is known as $(Ni, Fe, Cr)_2(Mo, Nb, Ti)$. To understand the segregation level of these constituent elements in both samples, we performed EDS analysis to plot distribution of Niobium, Aluminium, Nickel, Titanium and Carbon in both samples (Figure 50). In X1-cuboid, we observed depletion of Ni in those regions with white particles, together with strong segregation of Niobium and Molybdenum along the inter-dendritic regions, which confirms those white particles are Laves phase. Meanwhile, we also observed segregation of Aluminium and Titanium in these regions, which are the necessary constituent for metal carbide formation. In B-cuboid, we found

that the level of segregation of these intermetallic elements are less severe as compared to X1-cuboid. These results are further confirmed by elemental EDS wt% analysis shown in Table 13, which we observed the average Nb content of Lave phase in sample A (30.7%) is much higher than sample B (14.1%)

Figure 50: The elemental distribution showing Niobium (Nb), Aluminium (Al), Titanium (Ti), Nickel (Ni) and Carbon (C) in as-printed A (a-e) and B (f-j) sample.

Table 13: Chemical composition (wt%) along inter and intra-dendritic regions.

	Inter-dendritic regions (wt%)							Intra-dendritic regions (wt%)						
	Ni	Nb	Mo	Al	Ti	Cr	Fe	Ni	Nb	Mo	Al	Ti	Cr	Fe
As printed A	32.1	30.7	6.4	0.5	3.0	11.3	12.1	51.9	3.0	2.8	0.4	0.5	19.2	20.4
As printed B	47.1	14.1	3.8	0.3	1.5	16.6	16.8	52.2	3.0	2.6	0.5	0.8	19.5	21.3
Heat treated A	48.5	19.7	4.8	0.3	1.7	12.1	12.7	52.3	3.2	2.7	0.4	0.7	19.6	21.0
Heat treated B	52.4	11.7	3.1	0.4	1.3	15.0	15.8	51.0	4.3	3.1	0.5	0.8	19.4	20.8

6.3 Effect of heat treatment

Heat-treated microstructure

The heat-treated microstructure for both A and B samples are shown in Figure 51. The homogenization heat treatment dissolves Laves phase in both samples and releases the segregated Nb, Al, and Ti to matrix for formation of strengthening phase (γ'' , γ' and δ) in the double aging treatment. Indeed, we find plat-like δ phase presence in both samples. The δ phase in X1-cuboid are bigger in size ($\sim 10 \mu m$) and mainly cluster around the regions rich in Niobium, or regions rich in Lave phases in as-printed condition. Conversely, delta phases in B-cuboid are much smaller in size ($\sim 1 \mu m$) and distributed evenly across the sample. The EBSD grain orientation map shows similar grain morphology and grain size for both X1-cuboid ($21000 \mu m^2$) and B-cuboid ($7100 \mu m^2$) sample, as compared to their as-printed condition, respectively. Additionally, the XRD results shows the presence of δ phase in both samples, where MC carbide still retain in X1-cuboid.

Figure 51: The SEM micrographs of (a) X1-cuboid and (b) B-cuboid showing micro and nano size δ phase. The EBSD grain orientation map showing grain morphology of (c) X1-cuboid and (d) B-cuboid sample along building directions (e) XRD results of heat-treated X1-cuboid and B-cuboid sample

In both samples, we can still find undissolved Lave phase with high Nb content, as shown in Figure 52. The undissolved Laves phase in B-cuboid sample are smaller and distributed more evenly. Conversely, we find that delta phase in X1-cuboid concentrated around regions rich in Nb content (Laves phase in as-printed conditions) and distributed more evenly in B-cuboid. Using EDS analysis, we find that Nb content retained in the inter-dendritic regions is much higher in A sample (19.7%) than B sample (11.7%). Furthermore, we still observe segregation of Ti and Al in A sample, which indicate the homogenization temperature is not high enough to dissolve the carbides.

Figure 52: The elemental distribution showing Niobium (Nb), Aluminium (Al), Titanium (Ti), Nickel (Ni) and Carbon (C) in heat-treated X1-cuboid (a-e) and B-cuboid (f-j) sample.

Mechanical properties

Figure 53 shows the room temperature tensile properties of all samples. It is interesting to note that X1-cuboid has a higher ultimate tensile strength (UTS: 910 MPa) and lower ductility (e: 22%) in as-printed condition. Conversely, B-cuboid has a lower ultimate tensile strength (UTS: 891 MPa) and higher ductility (e: 38%). However, after heat treatment, B-cuboid exhibits better tensile properties in both ultimate tensile strength (UTS: 1315 MPa) and ductility (e: 21%).

Figure 53: Tensile properties of (a) as-printed and (b) heat treated conditions for both samples.

Figure 54 shows the longitudinal sections of fractures for all samples. During deformation process of as printed X1-cuboid, long-strip Laves phase break up and become sites for micro-voids formation. This phenomenon can be clearly observed in Figure 6a, where we observed cracks formation within long stripped Laves. These micro-voids have significantly reduced ductility of X1-cuboid, as compared to more ductile B-cuboid, where we do not observe micro-voids across the sample. We observed similar distribution of micro-voids in heat treated samples, where these micro-voids are segregated in the Nb-rich regions in X1-cuboid, Conversely, formation of micro-voids are evenly distributed in heat-treated B-cuboid. These results showed B-cuboid are more ductile compared to X1-cuboid in both as-printed and heat-treated conditions.

Figure 54: The SEM micrographs showing longitudinal sections of fracture in as printed (a) X1-cuboid and (b) B-cuboid, together with heat treated (c) X1-cuboid, (d) B-cuboid, with white arrow indicate micro-voids form during deformation process. (e) Schematic illustration of SEM micrographs location on tested tensile coupon.

6.4 Discussion

As-printed condition

Figure 55a shows phase diagram of I718 of idealized situation for an interstitial free alloy [103]. Points A and B are the average solidus and liquidus temperature plotted at average bulk Niobium concentrations (4.8%). However, the actual solidification sequence is much more complicated due to non-equilibrium solidification. Figure 55b shows schematically the sequence of actual solidification path of I718 in DED process. The solidification path begins with formation of dendrite (γ) and the liquid enriches in Nb, Mo, Al and Ti elements. The dendrite will continue to grow until the interfacial composition satisfied the condition for formation of non-invariant eutectic of γ/MC . The formation of γ/MC eutectic depletes the remaining liquid of Nb, Mo, Al and Ti and bring back the local liquid composition towards γ compositions. The solidification then continues and terminates with formation of non-invariant eutectic reaction of γ and Laves phase, X1-cuboid with lower cooling rate (CR) results in longer interval across each solidification stage and induce higher segregation rate, as compared to B-cuboid with higher cooling rate.

Figure 55: The (a) phase diagrams of I718 and (b) solidification path for X1-cuboid and B-cuboid sample

Heat-treated conditions

The significant strength improvement (see Figure 49) for both samples can be attributed to the formation of primary strengthening phase after heat treatment, known as γ'' precipitates. These coherent γ'' precipitates strengthen the alloy via coherency strain hardening mechanism. The AMS 5663 heat treatment used in this experiment is a well-established strategy for the formation of γ'' [20] in I718. In particular, the superalloy strength is proportional to the amount of γ'' precipitated through heat treatment at temperature higher than 620°C.

As for the formation of δ phase after heat treatment, it can be achieved via i) precipitation along the boundaries, i.e., grain boundary, twinning boundaries or phase boundaries and ii) transformation from γ'' to δ phase. It was also reported that nucleation and growth of δ phase needed a certain amount of Nb [104], and it usually depleted the amount of γ'' precipitates. The formation of δ phase in both of our samples were likely a result of transformation from γ'' to δ phase. In X1-cuboid where the areas with higher Nb content provided a more favourable conditions for the precipitation of δ phase, we observed a bigger in size and highly segregated δ phases, as shown in Figure 51. It is widely believed that δ phase had an adverse effect on ductility and toughness. In our samples, the X1-cuboid with a slightly inferior tensile properties after heat treatment could be due to this reason. We also found additional evidence from the SEM micrographs on the tensile coupons after testing. There are micro-voids formation concentrated in these δ -rich regions during the deformation process (see Figure 54).

Effect of sample size

In DED processes, parts naturally heat up during production. The as-printed microstructure is highly dependent on the cooling rate during solidification process. However, the parts will not heat up indefinitely. In the smaller samples with a height of 20mm and 40mm (see Figure 39), we observed a gradual decrease in cooling rate and the value plateau when the build reaches a certain height, which suggests the establishment of a CR steady state. This similar observation is found across sample X1 (see Figure 67) and sample X1-block (see Figure 47) with only the variation in magnitude of change. Hence, it is reasonable to assume that large scale sample will also achieved CR steady state at some point of time during deposition. Generally speaking, a higher cooling rate results in smaller grain size, higher hardness and heterogeneous texture whereas lower cooling rate results in bigger grain size, reduction in hardness and highly textured microstructure.

7 Microstructure control

The R-series samples in section 4.2 exhibit lack of fusion defects along layer interface. We characterize the defects using optical microscope (OM) and find that the defects size is in the range of mm scale, as shown in Figure 56a. Other than that, we observed the columnar grain growth within the layer itself and equiaxed grains form a thin “band” as a separation between layers. These observations suggest the process is not fully optimized due to excessive layer thickness results in insufficient bonding between layers.

To resolve this issue, we decrease powder feeding rate while keeping other process parameter constant, to reduce the layer thickness from 4mm to 1mm, and achieve better re-melting and proper bonding with previous layer. The sample with optimised parameter, named X1, shows little to no porosity based on OM analysis, as shown in Figure 56b. Other than that, we observed the $\langle 100 \rangle$ textured grains are growing across layers from bottom to top of the build. Other than that, the grain growth is no longer interrupted by the equiaxed grains within the layer. We also observe highly $\langle 100 \rangle$ textured grains are mainly concentrated in the centre of the cube for X1 sample, instead of growing within the layers in R1 sample (Figure 56 c, d). These observations are additional evidence of better inter-layers bonding with optimised parameter.

Figure 56 (a) Optical microscope image of R1 sample (b) Optical microscope image of X1 sample, upon optimised with layer thickness. Grain orientation map along Z of (c) R1 sample and (d) X1 sample

7.1 24Vx nozzle

We then use X1 parameter as a baseline to design an experiment aimed at understanding the effects of power, scanning speed, overlap distance, powder feeding rate and dwell time on the grain orientation of printed part. We carry out these experiments using the same 24Vx nozzle with 2.25mm nozzle diameter. First, we produce X-series sample by keeping the scanning speed constant and varying the power and dwell time for X1, X2 and XX samples. Thereafter, we produced X1-series samples by keeping the power constant and vary the scanning speed, layer height and dwell time accordingly. The details parameters of respective sample are listed in Table 14.

Table 14: Parameter table for X-series and X1-series sample

Sample	Power, W	Scanning Speed, mm/min	Layer height, mm	Overlap distance, mm and overlap percentage, %	Powder feeding rate, g/min	Dwell time, Yes/No
X-series						
X1	1600	430	1	0.75, 33	5	No
X2	1400	430	1	0.75, 33	5	No
XX	1600	430	1	1.25, 56	3.8	No
X1-series						
X1-a	1600	800	1	0.75, 33	10.5	No
X1-b	1600	610	1	0.75, 33	8.5	No
X1-c	1600	430	1.35	0.75, 33	7.2	No
X1-d	1600	430	1	0.75, 33	5.5	Yes

We characterise the X-Series sample by EBSD and produce grain orientation map along Z (building direction), Y (out-of-plane), and X (in-plane) as shown in Figure 57. We notice pronounced grain coarsening from bottom to the top of build, in which most of the grains are columnar in nature at top of the build, due to high power employed in producing the X1 sample. These grains are mainly $\langle 100 \rangle$ textured along Z, the building directions, and $\langle 110 \rangle$ texture in Y, the out of plane direction, similar to the H sample produced using Trumpf machine (Figure 32). However, in X1 sample, we do not observe banding interrupting columnar growth like what we observed in H sample.

Separately in sample X2, even though the power only reduces by 200W (12.5%) as compared to X1, we observe quite a different microstructure. The EBSD map of X2 along Z shows much smaller amount of $\langle 100 \rangle$ texture grains along Z and grain size as compared to X1 sample. We still notice the grains in X2 sample growing across layers, however, the extent of columnar grain growth is not as pronounced as what we observed in X1 sample.

We produce sample XX with same power of 1600W as X1 but increase the overlap level from 33% to 56%. It is noteworthy that to maintain the same layer thickness of 1mm, it is necessary to compensate the material supplied by reducing powder flow rate. We observed a reduction in amount of $\langle 100 \rangle$

textured grains along Z compared to X1 sample, but higher amount as compared to sample X2. In addition to that, these grains are also bigger in size and spanning across more layers (Figure 57 d-f).

While analysing grain orientation map along Y, out-of-plane and X, in-plane directions, the grain orientations are quite different as compared to Z, but similar between X and Y for all samples. We observed minimal amount of $\langle 100 \rangle$ orientated grains along both X and Y directions, as compared to Z. Qualitatively, the $\langle 100 \rangle$ grains along Z are mainly $\langle 110 \rangle$ and $\langle 111 \rangle$ textured along Y and X directions.

In terms of grain morphology, we found that increasing the overlap or reduction of laser power are effective in breaking down the long columnar grains that spanning across the entire build, like what we observed in X1 sample.

Figure 57: EBSD grain orientation map along Z(BD), Y(out-of-plane) and X(in-plane) directions for (a, b, c) sample X1 (d, e, f) sample X2 and sample XX (g, h, i).

We then carried out texture analysis to further confirm our qualitative observation on the samples. Indeed, we found the M.U.D. strength of $\langle 100 \rangle$ texture grains in X1 along Z is almost 100% and 30% higher when compared to X2 and XX sample. At the same time, $\langle 110 \rangle$ and $\langle 111 \rangle$ textured grains along

Z are lower in density for all three samples. Along Y, X1 sample has the highest density of $\langle 110 \rangle$ grains and X2 sample has the highest density for $\langle 111 \rangle$ grains. We observed a rather consistent density distribution for $\langle 100 \rangle$, $\langle 110 \rangle$ and $\langle 111 \rangle$ grains along X directions in all 3 samples.

Figure 58: Texture analysis using multiple of uniform density (M.U.D.) on all 3 samples along (a) Z (building direction) (b) Y (out-of-plane direction) (c) X (in-plane direction)

To better understand the effect of different process parameters on the resultant grain orientation, we produced additional samples using X1 parameter as the baseline, followed by varying scanning speed, layer thickness, and introduced the dwell time (Table 14). Similarly, we then characterized all samples using EBSD and the grain orientation map is presented in Figure 59.

We first compared the grain orientation map along Z for all samples, with the baseline we established earlier – sample X1. We observe qualitatively that in all samples, the density of $\langle 100 \rangle$ texture grains are reduced as compared to sample X1. However, sample X1-c (Figure 59g) shows the most similar grain morphology as sample X1, with long and bulky $\langle 100 \rangle$ textured grains concentrated near the top-centre portion of the build, along Z. In fact, sample X1-d (Figure 59j) also has a very similar morphology of $\langle 100 \rangle$ columnar grains with a slightly lower density. It is noteworthy that both sample X1-c and sample X1-d were produced using same power and scanning speed as sample X1, in which X1-c uses a higher layer thickness and X1-d introduce dwell time after every 4 layers of depositions. Furthermore, we noticed that most $\langle 100 \rangle$ texture grains along Z are $\langle 110 \rangle$ texture along X and Y directions.

While comparing the grain morphology and grain sizes across the samples, we found that introduction of thicker layer thickness (sample X1-c) and dwell time (sample X1-d) has the minimum effects in breaking down the long and bulky $\langle 100 \rangle$ texture grains in X1 sample into the smaller grains. On the other hand, the higher scanning speed employed in sample X1-a and X1-b is more effective in breaking down the big and bulky $\langle 100 \rangle$ grains along Z directions into smaller and randomly textured grains.

Figure 59: EBSD grain orientation map along Z(BD), Y(out-of-plane) and X(in-plane) directions for (a, b, c) sample X1-a, (d, e, f) sample X1-b, (g, h, i) sample X1-c, and (j, k, l) sample X1-d.

To visualize texture differences across samples, we then performed again the texture analysis as shown in Figure 60. Indeed we found that sample X1-b has the lowest density of $\langle 100 \rangle$ textured grains along Z. It is also interesting to note that sample X1-b with the lowest density of $\langle 100 \rangle$ texture grain has the highest amount of $\langle 110 \rangle$ grains along the Z. In X1-c and X1-d samples, we found similar distribution of large $\langle 100 \rangle$ textured grains near the top-center of the build with lesser density, as what we observed in X1 sample. The texture analysis also shows that $\langle 110 \rangle$ texture grains are highest in density across all samples along Y directions. Furthermore, highest density of $\langle 110 \rangle$ grains are found in the X1 and X1-c samples with the minimum presence of $\langle 100 \rangle$ texture grains across all samples. Meanwhile, we found

a relatively consistent density for the grains of all orientation along X direction across all samples. In general, the analysis shows that reducing speed, increasing layer thickness and introduction of dwell time are effective in reducing <100> textured grains along Z and <110> texture grains along Y.

Figure 60: Texture analysis using multiple of uniform density (M.U.D.) on all 4 samples (X1, X1-a, X1-b, X1-c, X1-d) along (a) Z (building direction) (b) Y (out-of-plane direction) (c) X (in-plane direction).

7.2 10Vx nozzle

The BeAM machine is equipped with nozzles of different sizes; 24Vx – 2.25mm nozzle diameter and 10Vx – 0.8mm nozzle diameter. In order to understand the microstructure formation mechanism on parts printed with 10Vx nozzle, we printed two samples with different process parameter as shown in Table 15. These parameters are chosen, as they are able to yield near full dense samples with minimum porosity.

Table 15: Parameter table for A and B sample

Sample	Power, W	Scanning Speed, mm/min	Layer height, mm	Overlap distance, mm and overlap percentage, %	Powder feeding rate, g/min	Dwell time, Yes/No
A	450	2000	0.4	0.48, 60	4.1	No
B	400	1500	0.33	0.48, 60	2.7	No

We then characterized both samples using EBSD and plot the grain orientation map as shown in Figure 61. As compared to the sample produced using 24Vx nozzle (X-series), we find a much smaller grain size in sample A and B. The grain morphology are mainly consist of columnar grains growing across the layers, Conversely, we find <110> grains density along Z is the highest instead of <100> grains we observed from the X-series samples. The grain orientation along Y and X directions are similar for respective samples, with A sample consisting mainly <100> texture grains and B sample consists mainly <111> textured along both directions.

In terms of grain morphology, sample B consists of columnar grains growing across layers from bottom to top, with a zig-zag patterns. This is in line with other researcher findings [62], which suggest the difference in grain growing direction due to scanning strategy employed.

Figure 61: EBSD grain orientation map along Z(BD), Y(out-of-plane) and X(in-plane) directions for (a, b, c) sample A and (d, e, f) sample B.

To better visualize the change in texture across samples, we plotted the $\langle 100 \rangle$, $\langle 110 \rangle$, and $\langle 111 \rangle$ texture strength along X, Y, Z directions for both samples, as shown in Figure 62. The quantitative analysis shows that $\langle 110 \rangle$ texture strength along Y direction is almost 4x of what we observed in A sample. This is also significantly different as compared to the X and X1-series samples produced using the big nozzle. Conversely, the texture strength of $\langle 100 \rangle$ and $\langle 110 \rangle$ along Z are the same in X sample.

Figure 62 Texture analysis using multiple of uniform density (M.U.D.) on samples A and B along (a) Z (building direction) (b) Y (out-of-plane direction) (c) X (in-plane direction).

7.3 Microstructure control

The BeAM machine with dual nozzle configuration makes the dual nozzle deposition in a single process possible. In other words, we can perform the deposition using different nozzle production of a single part with a single set up. This technique is useful for creation of functionally graded materials (FGM). As an attempt to achieve texture control in the deposition, we employed two sets of parameters from different nozzles (Table 16), on the construction of a single deposit. The idea is to create a part with alternating regions, which highly textured in $\langle 110 \rangle$ and $\langle 100 \rangle$ respectively.

Table 16 Process parameter X1 and B

Process parameter	Resultant texture along Z	Power, W	Scanning Speed, mm/min	Layer height, mm	Overlap distance, mm and overlap percentage, %	Powder feeding rate, g/min	Dwell time, Yes/No
X1	$\langle 110 \rangle$	1600	430	1	0.75, 33	5	No
B	$\langle 100 \rangle$	400	1500	0.33	0.48, 60	2.7	No

We printed a block with 80mm in height, alternating between X1 and B parameter using 24Vx nozzle and 10Vx nozzle for every 4mm of deposit. We then extracted a sample of 20mm (H) x 5mm (W) in size from the middle of the block and characterize the sample with EBSD. The results are shown in Figure 63. For the regions printed with X1 parameter (24Vx), we notice a loss of texture and change in grain orientation along Z. While we observed retaining of small portions of $\langle 100 \rangle$ texture grains in the lower part of the sample, the grain orientation along Z on top regions of the sample are random in orientation. On the other side, regions printed with 10Vx nozzle also experience the loss in texture across the build. The strength of $\langle 110 \rangle$ texture that we obtained from the block printing are weakened by random grains in different orientations.

Figure 63 EBSD map of hybrid sample along (a) Z, (b) Y and (c) X directions.

We performed the grain size analysis along Z, to characterize the difference in grain size formation along Z (Figure 64). The grain sizes for respective regions were similar to those printed using only single parameter. Our results were similar to the research that utilize of ultrasound to produce part with alternating layers of microstructure [95].

Figure 64: Grain size calculated using (a) equivalent circle diameter and (b) area along Z, the building direction.

7.4 Discussion

Density & porosity

In DED process, powder is supplied coaxially from nozzle and melted by laser beam on the fly. To achieve production of parts free from defects such as lack of fusion and porosity, sufficient energy needs to be supplied to ensure proper melting of materials. A widely reported metrics of measurement for porosity control in AM is volumetric energy density (VED). The definition of VED involves laser power P , scanning speed s , overlap distance h , and layer thickness t as shown:

$$VED = \frac{P}{h \times s \times t} \quad (10)$$

It is noteworthy that above equation is established based on power bed fusion (PBF) process, where layer thickness is defined as t . The main challenge in applying this equation for DED process is that layer thickness is not an input parameter but an output parameter. Instead, we use powder feed rate [g/min] to control the amount of material supplied and determine the layer thickness in combination with other process parameter. However, as highlighted in the earlier section 2.1.2, it is recommended to achieve a pre-set layer thickness by changing the power feeding rate. In other words, we alter the

powder feeding rate to make layer thickness as the input parameter. After that, we were able to calculate the VED and used it as a metric for process control.

Figure 65: The relationship between VED with (a) maximum pore size and (b) relative density

Since the first step of microstructure control is to achieve full dense part, we then correlated VED of all the printed part produced using different nozzles with the calculated porosity value. We characterised porosity using two different metrics, i) maximum pore size ii) relative density, which was calculated using equation 11 from section 3.4. The results are illustrated in Figure 65. We found those samples produced using lower VED consist of pores in the range of 1 – 2 mm with some of the acicular pores characterized as lack of fusion within layers (Figure 26 and Figure 56). In general, the maximum pore size reduces with increasing VED as higher energy supply ensure full melting of material supply and proper bonding with previous layers [93]. In our experiment, the maximum pore size reduces significantly to the range size of <0.5 mm when VED exceeded 50 J/mm³. A similar trend is observed when we characterise the level of solidity using relative density, with reference to equation 11 from section 3.3. Sample with VED more than 50 J/mm³ achieve relative density of 99% and above. Our results are in good agreement with other researcher who reported that numbers and sizes of pores decreased significantly with increasing of VED in additive manufactured I718. [93][94].

Grain orientation control

In general, there are two common solidification mechanisms, namely nucleation or growth of crystal. It is well established that <100> crystal direction is the preferred and fastest growth orientation for face-centered cubic (FCC) materials during solidification [62]. This means that if we supplied strong laser power and aligned the maximum heat flow direction to Z direction in a DED process, growth of crystal will prevail and the solidification will occur only by the epitaxial growth of <100> grains along Z.

These phenomena can be clearly observed in our experiment. In the process with high laser power employed, we observed strong <100> texture columnar grains align to Z, the building direction (Figure

32 & Figure 57). The high power produced a melt pool with higher temperature and increased thermal gradient between the melt pool and substrate. As a result, the heat dissipation will occur via conduction in the direction along the Z, since the large cross section of the build provides a conduction path for most effective heat dissipation. In this case, it results the alignment of maximum heat flow direction along the Z and promote the <100> growth of crystal in the same directions. Conversely, in the sample with lower heat input, i.e. sample X2, we observed a reduction in density for columnar grains in <100> orientation. This was because the lower laser power, coupled with the scanning strategy, changed the maximum heat flow direction in the process.

In our experiment, we tried to reduce the intensity of <100> texture grains by introducing dwell time and increasing layer thickness or scanning speed, to achieve lesser heat input and heat build-up in the process (Figure 57 and Figure 59). However, the effect of such approach proved to be less effective as compared with lowering the laser power.

Figure 66 Effect of (a) linear heat input, (b) energy density on the <100> texture strength along Z.

In order to better understand the effect of heat input on the formation of <100> grains in the process, we calculated linear heat input and energy density for every sample and correlate them with the M.U.D. of <100> grains in the samples (Figure 66). The results show that increasing in energy supplied promote the growth of <100> grains along building directions.

Another reason for the formation of bulky and strong <100> textured grains is the reduction in cooling rate along Z due to high power employed in the deposition process. The continuous heat input in the process will heat up the part, resulting a smaller thermal gradient between the melt pool and the substrate, and eventually lower the cooling rate as the build progress. Here, we compared the two samples produced using different machines and find that when high power are employed during the build (H: 1900W, X1:1600W), the dendrite arm spacing formed become wider which indicates cooling rate reduced along Z. The lower cooling rate at the top of the build gave more time for the growth of crystal,

which facilitated the formation of bigger grains as compare to bottom regions with higher cooling rate. In other samples produced using smaller power, we observed a relatively consistent PDAS measurement across the build.

Despite the fact that cooling rate we measure in the X1 and H samples are in the similar range, we did not observed the <110> texture banding in H sample on X1 sample, as shown in Figure 32 and Figure 57. This is probably due to the higher effective powder deliver rate in H sample. We calculated the effective powder delivery rate using following equation:

$$\text{Effective powder delivery rate} = \frac{\text{Powder flow rate}}{\text{Scanning speed}}$$

This equation effectively calculates the actual supply of powder per unit length, as g/mm by taking into account relationship between power flow rate and scanning speed. We calculated that 0.025g/mm of effective powder delivery rate is more than double of 0.012g/mm in X1 sample. The high amount of effective powder delivery rate may result in formation of bigger melt pool in those layers with <110> banding, resulting a longer cooling time and the prevailing of local thermal gradient for the grain formations. It is noteworthy that the difference in the laser beam profile may be another contributing factors to the difference. Several researcher have shown that different laser profile may lead to difference in microstructure, even the process parameter was kept constant[24],[25],[26].

Figure 67: Variation of PDAS along Z in (a) H-sample (c) X1-sample and corresponding CR for (b) H-sample and (d) X1-sample.

The fact that samples produced using smaller laser spot size, which are sample B (Figure 61) and sample L (Figure 32) exhibited a strong $\langle 110 \rangle$ texture along Z is also interesting. Coupled with the EBSD map from other orientation, we found that $\langle 100 \rangle$ texture grains are oriented in 135° from the scanning directions in both samples, as shown in Figure 68. In these samples, the measurement shows higher and more consistent cooling rate in Z direction, which evince by consistent DAS spacing measurement across the samples. This results local thermal gradient within the melt pool are higher and local heat flux plays a dominant role. In these samples, the maximum heat flow direction deviates from Z and aligning towards the 135° from scanning direction instead, as compared to other samples. Hence, we can conclude that the bulk heating effect on these samples are lower and the localized maximum heat flow directions is a function of scanning strategy employed, mainly due to localised thermal conditions of melt pool.

Figure 68: Illustration of cross-sectioning plane with maximum heat flow direction for (a) L sample and (b) B sample and crystal orientation for (c) L sample and (d) B sample.

Grain morphology and grain size control

We found that most of our samples consisted unique grain morphology characterised by highly texture large columnar grains along building directions. Additionally, we found that equiaxed grains were frequently observed near the substrate. This is in line with researcher observation [45]. Following the solidification conditions discussed in section 2.2.2, the two main solidification mechanism in our samples are columnar dendritic and equiaxed dendritic structure, as shown in Figure 69.

Figure 69: Schematic representation of the combined effect of thermal gradient and solidification velocity on solidification microstructure.

We grouped our samples into three categories, namely by deposition rate (H, M and L sample, section 0), big nozzle (X-series sample) and small nozzle (A and B sample). We found that samples produced with higher power will increase the temperature gradient, results in the full columnar dendritic solidification mechanism, as shown by H, X1, X1-b, X1-c, X1-d, XX and B sample, regardless the type of nozzle used. Conversely, samples M and X2, which produce with relatively moderate power will decrease the temperature gradient; and sample X1-a with higher scanning speed that increase the interface velocity, result the mixing of columnar and equiaxed dendritic solidification. As for sample produced with combination of low power and high scanning speed such as L and A sample, the microstructure formation tend to be equiaxed grains dominated.

At the same time, to analyse the grain size variation in the sample produced, we calculated the grain sizes in all sample, by using two different metrics, namely area and equivalent circle diameter. The results are illustrated in Figure 70. We find that grains size of samples produced using small nozzle are significantly lower than those samples produced using bigger nozzle. This can be explained by the much smaller melt pool formation while deposition is carried out using smaller nozzle. These smaller melt pool sizes solidified in a smaller time interval, due to higher cooling rate. As a result, nucleation of grains dominated and this promoted the formation of equiaxed grains. Among the X-series and X1-series samples, we could see the largest grain size in the sample X1, X1-c and X1-d. This was because sample produced using a bigger nozzle, in couple with high power will generate larger melt-pool that required longer time to cool down. As such, there are sufficient time for the grain to grow in size, as compared to the former scenario.

Figure 70: Grain size in terms of (a) area (b) equivalent circle diameter for all samples produced using BeAM machine.

In summary, our results showed that in order to achieve homogeneous, equiaxed structure for a DED part, fine calibration of key process parameter by lowering the power, and increasing the scanning speed is necessary. Lowering the laser power and increasing the scanning speed will promote the formation of fine-equiaxed grains and increasing the laser power with lowering scanning speed will promote the formation of bulky-columnar grains structure. On the other hand, approaches like dwell time and increasing layer thickness proved-to-be less effective than the former two key process parameter.

8 Conclusion and future work

The main conclusions of this work are:

- The process parameter in DED has a significant effect on printed part geometry and microstructure. Using ANOVA analysis, we found that laser power has the most significant effect on bead width with 95% confidence and powder feeding rate has the most significant effect on bead height with 90% confidence.
- Layer thickness in DED processes will determine the melt pool size, which acts as an upper limit for the microstructure feature size. Processes with high layer thickness increase deposition rate but require higher thermal input and powder flow rate.
- The high thermal input associated with high build rate DED processes may yield a multiscale structural heterogeneity, which includes large-scale microstructure “bands” of equiaxed grains with possibly dissimilar crystallographic textures with respect to the prevailing columnar growth, as well as local gradients in both composition and second phase precipitate structures. Such a heterogeneity cannot be removed using standard heat treatments.
- We ascribe the microstructure “bands” to layer-wise differences in cooling rate, which can be directly related to the deposition strategy we employed. Conversely, composition gradients and precipitate structure variations stem from a progressively lower cooling rate as the build progresses, which results from the increasingly longer distance of the melt pool to the heat sink (i.e., the base plate).
- The solidification texture of I718 could be controlled by careful selection of process parameter during deposition process. Processes with high energy input (high power and low scanning speed) tends to promote growth of $\langle 100 \rangle$ columnar grains aligned with Z-direction. On the other hand, lower energy input (low power and high scanning speed) tends to promote epitaxial grain growth with not specific orientation.
- We assess the property scatter induced by this multiscale microstructure heterogeneity using a combination of indentation and compression tests. We found that hardness variations correlated well with changes in Nb content in γ matrix and size of the dendrites. By contrast, bulk mechanical properties are strongly affected by changes in texture (from $\langle 100 \rangle$ to random) and grain size. Interestingly, the correlation between the average grain diameter and the mechanical properties did not generate a linear Hall-Petch relationship, whose slope appeared to be affected by the local crystallographic texture.
- Sample geometry or sample size has a direct effect on the cooling rate in the process. Bulky samples with bigger cross-sectional area provide a better conduction path for thermal dissipation, resulting in lower thermal built-up and higher cooling rate, which refined the microstructure.

- Homogeneous microstructure and consistent mechanical properties may be attained by proper control of the cooling rate and specially designed deposition strategies during DED. Through careful selection of the process parameter, it is possible to manipulate the thermal build up and thus the cooling rate to achieve the desired microstructure. In some cases, however, such achievements may come at the cost of a significantly reduced build rate.
- Standard heat treatment following AMS 5663 was unable to fully homogenize the microstructure. Strongly segregated Nb in the printing process induces the formation of undesired Lave phase, in which directly affect precipitation and distribution of strengthening phase (γ'' and δ) after heat treatment. Careful selection of heat treatment parameters, i.e., duration and temperature are necessary to achieve desired heat-treated microstructure.

The following future research directions are recommended:

- In this study, we performed the cooling rate estimation using PDAS measurement and established process-structure-property relationship in DED I718. These experimental data can be useful input for development of thermal simulation, which can simulate the printed microstructure based on processing parameters.
- In this study, we demonstrated the effect of thermal build up on the microstructure of printed part using constant parameter. An in-situ process monitoring in couple with real-time process parameter control can be developed based on the proposed process-structure-property relationship. Such approach will provide possibilities of controlling the thermal history on a real-time basis to achieve homogenous microstructure.
- Development of DED specific heat treatment procedure is proposed for fully homogenization of Laves phase and precipitation of strengthening phase (γ' , γ'' and δ phase). The approach in optimizing the heat treatment duration and temperature based on printed part microstructure will allow the production of DED part with desired microstructure.
- In this study, we presented the mechanical heterogeneity of DED-printed I718 part, which includes hardness, yield strength and Young's modulus. However, for real applications in oil and gas or aerospace sector, ductility and toughness are also important properties to be evaluated. It is recommended that tensile tests and Charpy impact tests are conducted to assess the mechanical performances more comprehensively. It will be also meaningful to study the effects of heterogenous microstructure on these properties.

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Appendix A

This appendix contains supplementary information for Chapter 5.

Figure 71: OM (optical micrograph) analysis for layer thickness and grain orientation. The layers are identified by characterising the melt pool boundaries for every $n+2$ layers, indicated by arrow in white. The alignment of OM and EBSD map revealed that the banding (characterised by $\langle 100 \rangle$ columnar grains and $\langle 110 \rangle$ equiaxed-like grains) repeated with an interval of about 2 layers.

Figure 72: (a) Mn (b) Al (c) Ti content (necessary constituent in formation of γ'' phase) change along Z as measured along dendrite

The strength of I718 can be calculated using[97]:

$$\sigma_{YS} = \sigma_0 + \sigma_{GB} + \sigma_{SS} + \sigma_{PDAS} + \sigma_{\gamma''} + \sigma_{\gamma'}$$

Intrinsic strength calculation

$$\sigma_0 = M\tau_{crss}$$

σ_0 , intrinsic strength

M, Taylor factor: 2.91

τ_{crss} , critical resolve stress: 17.5Mpa

$$\sigma_0 = 51\text{Mpa}$$

Grain boundary strength calculation

$$\sigma_{GB} = \frac{K}{D^{\frac{1}{2}}}$$

σ_{GB} , grain boundary strengthening

K, Hall-Petch coefficient :750MPa $\mu\text{m}^{\frac{1}{2}}$

$D_{nominal}$: 107 μm

D_{top} : 115 μm

D_{btm} : 100 μm

$$\sigma_{GB,nominal} = 72.5\text{MPa}$$

$$\sigma_{GB,Top} = 69.9\text{MPa}$$

$$\sigma_{GB,Btm} = 75.0\text{MPa}$$

Solid solution strength calculation

$$\sigma_{SS} = \left[\sum_i (a_i c_i^{\frac{1}{2}})^2 \right]^{\frac{1}{2}}$$

a_i , strengthening constant of solute atom i

c_i , concentration of solute atom i

Parameter	a_{Al}	a_{Ti}	a_{Cr}	a_{Fe}	a_{Mo}	a_{Nb}^*
Value	225	775	337	153	1015	1183
Parameter	c_{Al}	c_{Ti}	c_{Cr}	c_{Fe}	c_{Mo}	c_{Nb}
Value, % - Nominal	0.7	0.7	19	20	3.1	1.6
Contribution, %	0.42	3.24	16.61	3.61	24.58	51.7
Δ Composition	Top - Bottom					
Value, % - Top	0.7	0.7	19	20	3.1	1.3
Value, % - Bottom	0.7	0.7	19	20	3.1	1.0
$\sigma_{SS} - nominal$	291 MPa					
$\sigma_{SS} - Top$	277 MPa					
$\sigma_{SS} - Bottom$	284 MPa					

* The composition used to estimate the contribution of Nb atom in solid solution is 1/3 of the measure value, where the remaining 2/3 are consumed in formation of γ'' .

Primary dendrite arm spacing, PDAS strength calculation

$$\rho = \frac{200}{\pi d^2}$$

$$\sigma_{PDAS} = \alpha M G b \sqrt{\rho}$$

$$\sigma_{PDAS} = \alpha M G b \sqrt{\frac{200}{\pi}} d^{-1}$$

ρ , dislocation density

α , constant : 0.35

M, Taylor factor: 2.91

G, shear modulus: 80GPa

b, Burges vector: 0.2539nm

$d_{nominal, z=10mm}$: 17.884 μ m

$d_{top, z=20mm}$: 26.724 μ m

$d_{btm, z=1mm}$: 9.928 μ m

$$\sigma_{PDAS, nominal} = 9.2MPa$$

$$\sigma_{PDAS, top} = 6.2MPa$$

$$\sigma_{PDAS, bottom} = 16.6MPa$$

Ultimate tensile strength calculation

$\sigma_{yield, nominal}$: 950MPa

$\sigma_{yield, z=4mm}$: 979MPa

$\sigma_{yield, z=20mm}$: 931MPa

Estimated γ'' and γ' contribution

$\sigma_{\gamma''} : \sigma_{\gamma'} = 1:3$ [107]

Summary of contribution to the Δ Strength**

	Nominal	Contribution	Top	Bottom	Δ YS (Yield Strength)		Δ Hardness
H sample	MPa	Wt%	MPa	MPa	Δ MPa	Wt Δ %	Δ GPa
σ_0	51	5%	51	51	0	0%	0.00
σ_{GB}	72.5	8%	69.9	75	5.1	11%	0.02
σ_{SS}	291	31%	277	284	7	15%	0.03
σ_{DAS}	9.2	1%	6.2	16.6	10.4	22%	0.04
$\sigma_{\gamma''}$	388	41%	389	414	25	53%	0.11
$\sigma_{\gamma'}$	138	15%	138	138	0	0%	0.00
σ_{UTS}	950	100%	931	979	48	100%	0.20

** Δ Hardness is measured value, and the relative contribution from each microstructure feature is inferred from Wt Δ % calculated in Yield Strength.