Characterization of Laser Deposited Alloy 718

PENGCHENG CAO
Abstract

Additive Manufacturing (AM) is a method of producing three-dimensional objects using additive processes. It allows great flexibility in the processes and reduces the design-to-production time. Laser Metal Deposition (LMD) is one of AM methods under development and is based on the deposition technology. LMD has advantages in grain growth control, material functional grading, lower material storage requirement and more spatial freedom. Considering the outstanding features, it is important to study the characteristics of LMD products, which in this study is Alloy 718 for aerospace applications.

Single-wall Laser LMD samples are built with varied process parameters using gas-atomized Alloy 718 powders. Two experiments were carried out with focuses on 1) evaluations and comparisons of the microstructural characteristics, porosity and hardness of the samples are performed; 2) The effect of heat treatments including solution treatment and aging on the microstructure as well as the hardness.

The results of the experiments revealed directional solidification features and typical phases such as γ matrix, Laves phase and carbide. 0.06% average porosity and a majority of < 20 µm size are measured from the LMD samples. Only spherical gaseous pores are found while no lack-of-fusion pore is found. A hardness Vickers of 246 in average hardness is measured from the LMD samples. In the heat treated samples, δ phases were found; By direct-aging at 750 °C for 10 to 15 hours, the samples reach a maximum hardness of around 382 HV. The same hardness was reached by 1 hour of solution treatment at 950 °C followed by 5 hours aging at 750 °C.

The effects of processing parameters on the characteristics of LMD processed Alloy 718 are compared and discussed. A 2-dimentional map of porosity distribution along the length of the sample is made and the patterns are investigated along both the length and the height of the sample. It is found in the sample that the starting part of the deposit is higher in number of pores while the finishing part is larger in pore size. It is also found that the top layer of the deposit has the highest porosity level, pore number and pore size. Moreover, the hardness gradient along the build-up direction is evaluated and discussed. No significant hardness gradient was found. The precipitation hardening effect of LMD process and possible improvements are also discussed.

Key words: Additive, Deposition, Alloy 718
# Table of contents

1. Introduction ........................................................................................................................................ 3
   1.1 Challenges in conventional metal-processing ................................................................................. 3
   1.2 Development of Additive Manufacturing ....................................................................................... 3
   1.3 Aim of the thesis .............................................................................................................................. 4
2. Background ....................................................................................................................................... 4
   2.1 Additive Manufacturing .................................................................................................................. 4
   2.2 Laser Metal Deposition ................................................................................................................... 5
   2.3 Alloy 718 ........................................................................................................................................ 7
      2.3.1 Microstructures ......................................................................................................................... 8
      2.3.2 Precipitation Hardening ........................................................................................................... 10
   2.4 Porosity .......................................................................................................................................... 11
   2.5 Directional Solidification ............................................................................................................... 12
3. Experiment ....................................................................................................................................... 14
   3.1 Objectives ..................................................................................................................................... 14
   3.2 Experiment designs and processes ................................................................................................. 14
      3.2.1 Experiment 1 ............................................................................................................................. 14
      3.2.2 Experiment 2 ............................................................................................................................. 15
      3.2.3 Follow-up experiment: porosity distribution ........................................................................... 16
      3.2.4 Follow-up experiment: hardness gradient ............................................................................... 16
   3.3 Set-up and sample preparation ...................................................................................................... 16
   3.4 Experiment method ....................................................................................................................... 18
      3.4.1 Metallographic study ............................................................................................................... 18
3.4.2 Porosity evaluation .................................................................................. 18
3.4.3 Hardness test .......................................................................................... 19

4. Results ............................................................................................................ 20

4.1 Experiment 1 .................................................................................................. 20
4.1.1 Microstructure ......................................................................................... 20
4.1.2 Porosity .................................................................................................... 22
4.1.3 Hardness .................................................................................................. 23
4.2 Experiment 2 .................................................................................................. 24
4.2.1 Microstructure ......................................................................................... 24
4.2.2 Hardness .................................................................................................. 26
4.3 Follow-up experiment: porosity distribution .................................................. 27
4.3.1 Porosity distribution along the length ...................................................... 28
4.3.2 Porosity distribution along the height ...................................................... 28
4.4 Follow-up experiment: hardness gradient .................................................... 29

5. Discussion ....................................................................................................... 31

5.1 Dendritic growth .......................................................................................... 31
5.2 Effect of processing parameters on porosity .................................................. 31
5.3 Porosity distribution ...................................................................................... 35
5.3.1 Porosity distribution along the length ...................................................... 35
5.3.2 Porosity distribution along the height ...................................................... 35
5.4 Hardness gradient ........................................................................................ 36
5.5 Hardening effect of LMD process in Alloy718 ............................................. 36

6. Conclusion ....................................................................................................... 37

6.1 Microstructure .............................................................................................. 37
6.2 Porosity ..................................................................................................................37
6.3 Hardness ..................................................................................................................37
6.4 Heat treatment ........................................................................................................38
7. Acknowledgements ....................................................................................................39
8. References ..................................................................................................................40
1. Introduction

1.1 Challenges in conventional metal-processing

In the past few centuries, the processing of metallic materials was dominantly conducted via casting, deformation and metal cutting, etc. These methods normally introduce a large number of costs based on the consumption of energy, material, cutting tools, and moulds, etc. For example, metal-cutting, which is the most essential processing method in the modern manufacturing industries, produces a high amount of material wastage, and consumes a large number of expensive cutting tools at the same time, which also requires extra energy for transportation, re-melting and re-making. Meanwhile, the conventional methods normally rely on the capability of machines, and have limitations on the complicity of geometry and material type.

Especially in the area of aerospace, the conventional metal processing faces great difficulties, as the products are highly complex and use special materials in order to work efficiently in extreme environments. The geometry of the products usually consists of curved surfaces and mechanical interferences, and the materials used are normally special alloys such as Alloy 718 with a high hardness [1]. To manufacture such products, the manufacturers then have to design special process strategies and purchase special cutting tools. The designing of the process strategies is time consuming thus a long design-to-produce time, and the cutting of the materials uses expensive but short-lasting cutting tools thus a high cost.

To summarize the challenges in conventional metal-processing, the overall challenges are the high costs of various consumptions and the limitations on product complicity and material. Specifically, to overcome the latter challenges of limitations, longer design-to-produce time and higher costs would have to be considered.

1.2 Development of Additive Manufacturing

Different from the conventional ways of metal-processing, Additive Manufacturing (AM) is a method of producing three-dimensional objects in an additive manner which means adding materials on processed parts. It allows great flexibility in terms of geometry and material, and remarkably reduces the material wastage. In the past 20 years, a number of AM methods were developed based on various applied technologies, and the potential of application is
being evaluated in almost every modern industry such as automotive, nuclear and aerospace.

Laser Metal Deposition (LMD) is one of AM methods that are based on the deposition technology. LMD has advantages in controlling the grain growth, controlling the grade of functional materials, and allowing spatial freedom for the movement of both product and raw material. With its unique competence, LMD is believed to have a high potential in manufacturing aerospace components such as turbine blades.

1.3 Aim of the thesis

Considering the competence of LMD and its potential in aerospace application, studies have been done focusing on its processing, microstructure, hardness, heat treatment, etc. However, there is a lack of studies characterizing the common features of LMD processed materials in the aerospace area. Thus, in this thesis study, the aim is to characterize the LMD processed aerospace material, which in this study is Alloy 718.

2. Background

2.1 Additive Manufacturing

Additive manufacturing, also known as 3-D printing, is a method based on the concept of producing three-dimensional objects using additive processes. The typical process of AM follows a sequence of model designing, manufacturing followed by further treatments. Products are manufactured with successive layers following the design from a CAD file, which enables a variety of specifications in terms of geometries, materials and mechanical properties [2].

More recently, AM has received increasing amount of attention due to its greater allowance for flexibility in the design process and less design-to-production time. The most important advantage is that AM is capable of producing integrated and complex components with a single and close-to-net shape structure without using dies [3]. It is also considered to be efficient and economic with reduced design-to-produce time as well as reduced cost of material and energy since it minimizes the wastage by adding up materials instead of cutting it off. Moreover, together with planned post-treatments, it is possible to manipulate the
microstructure and mechanical properties of the component during AM. These outstanding advantages of AM are for the main reason driving current developments.

Research for the application of Additive Manufacturing is currently being carried out in the industries of aerospace, automobile, nuclear energy, medicine as well as art, etc. For instance, research in aerospace industries mainly focuses on the engine parts which consist of complex components and work in extreme environments such as high temperature and high pressure. It is reported that in 2015 NASA (National Aeronautics and Space Administration) in USA and Aerojet Rocketdyne successfully completed tests for a 3-D printed rocket engine chamber part which demonstrated a significant increase in the performance compared to traditional chambers [4]. In the same year, Rolls-Royce constructed a 3-D printed Front Bearing Housing for its aero-engine XWB-97 and will install it on the Airbus A380 jumbo-jet, which would probably be the first airborne 3-D printed engine component [5].

Due to over 20 years of development, AM now composes a variety of manufacturing methods. These methods can be classified in terms of applied technologies such as laminated object manufacturing (LOM), powder bed technology and deposition technology as listed in Table 1 [6]. The materials used in the manufacturing can be in the form of sheets, powders or wires and can be processed by cutting, sintering or fusion. The applied energy sources can be water jet, electron beam, electric arc or laser, etc.

<table>
<thead>
<tr>
<th>Applied technology</th>
<th>Example</th>
<th>Material feedstock</th>
<th>Processing form</th>
<th>Energy source</th>
</tr>
</thead>
<tbody>
<tr>
<td>Laminated manufacturing</td>
<td>LOM</td>
<td>Sheets</td>
<td>Cutting</td>
<td>Jetting / electron beam / laser</td>
</tr>
<tr>
<td>Powder bed technology</td>
<td>SLM</td>
<td>Powder</td>
<td>Sintering / fusion</td>
<td>Electron beam / laser</td>
</tr>
<tr>
<td>Deposition technology</td>
<td>LMD</td>
<td>Powder / wire</td>
<td>Fusion</td>
<td>Arc / electron beam / laser</td>
</tr>
</tbody>
</table>

The method employed for this study is that of Laser Metal Deposition (LMD), also referred to as Direct Metal Deposition (DMD).

### 2.2 Laser Metal Deposition

LMD is based on the deposition technology, where materials are melted and then directly deposited on a working piece rendering it similar to the process of welding. Figure 1 shows a
schematic set-up of an LMD nozzle while building a single-wall object [6]. As illustrated, the nozzle consists of three tunnels: the centre tunnel of the nozzle is a pathway for a laser, the one adjacent is a pathway for blowing in powders, and the outside tunnel is a pathway for shield gas. While the nozzle is travelling along the working direction, the metal powders are injected onto the surface and the laser is applied to melt the powder, creating a melt pool. This, in turn, eventually produces a deposited bead on the working piece. The shield gas is injected around the powder path in order to protect the melt pool from oxidation.

Figure 1. Demonstration of LMD nozzle

The characteristics and the performance of the LMD products are highly related to and can be controlled by the process parameters. The main process parameters of LMD are: laser power (W), nozzle traverse speed (mm/s), laser spot size (mm), powder feed rate (g/min), shield gas flow rate, and carrier gas flow rates (L/min), laser standoff distance (mm) and powder standoff distance (mm). The laser standoff distance and powder standoff distance respectively refer to the distance between the laser focus and the working surface, and the distance between the powder focus and the working surface. In addition, specific energy and line mass are also essential index for manufacturing, and respectively account for the energy and mass input of the process. The specific energy is the theoretical energy input. These parameters can be calculated using Equation (1) and (2):

\[
Specific \ energy = \frac{P_L}{D_L \times V} \quad (1)
\]

\[
Line \ mass = \frac{Q_p}{V} \quad (2)
\]
where $P_i$ is the laser power (W), $D_i$ is the diameter (mm) of the laser spot, $V$ is the nozzle traverse speed (mm/s) and $Q_p$ is the powder flow rate (g/min).

Meanwhile, the characteristics of the powder used in the process also have an influence on the outcome. The properties of the powder are commonly described as size distribution, surface morphology, porosity, inclusions and satellite content. These will affect the melting as well as the quality in terms of porosity, inclusion and surface roughness [6]. When producing the powders, Gas Atomization (GA) and Plasma Rotation Electrode Process (PREP) are currently believed to be preferable for AM. A previous study has shown that PREP powder has three times less porosity in comparison to GA powder, and PREP powder deposits show better surface roughness, lower interlayer porosity and a higher deposition rate [7].

The LMD is believed to have advantages in grain growth control and material functional grading [6][8]. The grain growth control is based on the directional solidification in the deposited material (see section 1.5). The functional graded material can be theoretically achieved by either switching nozzles carrying different powders or building extra powder tunnels in the nozzle. Moreover, in comparison to the powder bed technology, such as Selective Laser Melting (SLM), LMD has its own advantages in lower material storage requirement and more spatial freedom. The SLM works as pre-placing a layer of powder and selectively melting the designed parts, while the LMD injects the powder only to the designed parts which requires a dramatic lower amount of material. Also the SLM works only with a horizontal surface since the powder bed is placed horizontally, while the LMD is able to create more spatial freedom by applying angles to either the nozzle or the working piece.

In this study, the material that is being processed by LMD is Alloy 718 which is widely applied in aerospace industries.

2.3 Alloy 718

Alloy 718 is a nickel-iron-based superalloy, whose composition is given in Table 2 [9]:

<table>
<thead>
<tr>
<th>Table 2. Composition of Alloy 718</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni</td>
</tr>
<tr>
<td>-----</td>
</tr>
<tr>
<td>Bal.</td>
</tr>
</tbody>
</table>
Alloy 718 is well-known for its high strength achieved through the gamma-double-prime strengthening phase (γ'''), as well as its good weldability especially in terms of post-weld cracking resistance [9][10]. It also possesses good corrosion and oxidation resistance and good creep properties. The high strength of Alloy 718 is obtained from the precipitation hardening by the nanometric Gamma-prime (γ’) and Gamma-double-prime (γ’’) precipitates [11]. For a discussion on precipitation hardening, please see section 1.3.2.

Due to the excellent performance and relatively low cost, 35% of the world’s total wrought superalloy production is contributed by Alloy 718 [12]. Alloy 718 is commonly used in aerospace and nuclear industries since the applied working environment consists of high temperature and high pressure, for instance turbine blades, rotors and combustors. However, when manufacturing products made of Alloy 718, conventional machining methods often face difficulties such as worn out tool as well as poor work piece surface integrity due to its high hardness and low thermal conductivity. Thus, it would be preferable to manufacture parts of Alloy 718 by using powder metallurgical methods.

2.3.1 Microstructures

Alloy 718 has a face-centered-cubic (fcc) γ-phase matrix containing a number of secondary phases. The instinctive secondary phase in Alloy 718 is the ordered body-centered-tetragonal (bct) γ''-phase. Other secondary phases include the ordered fcc γ’-phase, the hexagonal Laves phase, delta phase and several carbides. The descriptions of the phases are as follows [12]:

- γ-phase: The fcc austenitic matrix phase is composed of base metal elements (Fe, Ni), as well as a high percentage of solid solution elements (Nb, Co, Cr, Mo, Ti, etc.). The solution elements provide a solid-solution-hardening to the matrix. The γ phase is found ideal for high-temperature structural alloys because of its high modulus, multiple slip system, low diffusivity of alloying elements and broad solubility for secondary elements.

- γ’-phase: The fcc Ni₅ (Ti, Al) precipitate is the most important strengthening phase in superalloys. The precipitation can be promoted by adding Al and Ti. Generally the γ-phase precipitates and disperses coherently within the γ-matrix and the morphology varies from spherical to cuboidal which can be controlled by the Mo content and Al/Ti ratio. When exposed to high temperature, film-like γ’ forms along the grain boundaries and improves the creep resistance. However, a prolonged exposure will cause undesired γ’-to-η (hcp Ni₅Ti) or γ’-to-δ (orthorhombic Ni₅Nb) transformations.
- γ''-phase: The bct Ni₃Nb precipitate with coherent disk-shape morphology is the principle hardening phase in Alloy 718. When subjected to over-aging, the γ''-to-δ transformation will occur. In comparison to γ', the major disadvantage of γ'' is that it has a lower solvus temperature which will result in a remarkable decrease in the strength of Alloy 718 upon the exposure to temperature higher than 650 °C by the γ''-to-δ transformation.

- Laves phase: The Topologically-close-packed (tcp) hexagonal A₂B (A=Fe, Ni, Co; B=Nb, Ti, Mo, Cr, etc.) is an undesirable phase in Alloy 718 applications. Laves has a much higher hardness comparing to the matrix due to a ductile-brittle transition [13], which causes grain-boundary embrittlement and reduces rupture strength and ductility. It also limits the effect of solution hardening since the Laves phase has a low interatomic distance which attracts the solute elements to come out of the γ matrix and form Laves phase.

- Carbides: There are several types of carbides in Alloy 718, for example the primary carbide MC and secondary carbide can be M₂₃C₆, where M is composed of molybdenum, niobium, tungsten, chromium and/or titanium. The primary carbides are normally formed as discrete particles located at both intragranular and intergranular positions and often between dendrites while the secondary carbides are formed during heat treatments and locates at grain boundaries. The primary carbide mainly provides the carbon source for the formation of the secondary while the secondary carbides mainly provide hardening effect. However, carbides with certain morphology or location can be detrimental to the strength by initiating or propagating cracks.

On a broader scale, when manufacturing Alloy 718 with LMD, a directional solidification microstructure can form which contains dominantly columnar structures similar to single crystals. This structure is anisotropic and is favourable for components working under high temperature conditions. The reason is that a single crystal structure gives a better creep resistance and thermal-mechanical fatigue behaviour due to the absence of grain boundaries which is normally an origin of dislocation [14].

However, a Columnar-to-Equiaxed transition may occur during solidification which destroys the columnar structure and should thus be avoided in productions requiring anisotropic performance. In Gauman’s model [15], it is suggested that a high temperature gradient and a low solidification velocity are the most favourable and important coefficients for developing the columnar structure. To achieve that, it is suggested that a low substrate temperature and
a reduced laser power can ensure a high thermal gradient. Furthermore, if the substrate also has a single crystal structure, then a small laser diameter with highly focused energy can ensure a sufficient remelting and initiate the columnar growth from the substrate \[15\].

Thus, with optimized processes and post-treatments, it is possible to manipulate the microstructure and obtain good anisotropic properties. On the contrary, when isotropic properties are required for the LMD product, it is also possible to manipulate the microstructure by changing the scanning paths during the process. Experiments show that by scanning in a cross-direction manner, as illustrated in Figure 2 \[16\], the continuous directional growth of columnar grains can be interrupted and more homogenous grains are produced. The cross-direction scanning shows a similar ultimate tensile property to single-direction scanning while it is proven to have higher ductility which is believed to be a result of the grain-size homogeneity \[16\].

![Figure 2. LMD laser scanning paths: (a) Single-direction scanning, (b) Cross-direction scanning](image)

### 2.3.2 Precipitation Hardening

The main strengthening mechanism of Alloy 718 is precipitation hardening which is mainly achieved by the precipitation of $\gamma'$ and $\gamma''$ intermetallic phases. The precipitation hardening consists of two major processes: solution treatment and precipitation treatment (aging). As illustrated in Figure 3, the alloy is first solution treated by being heated up to a high temperature ($T_0$) and kept for a certain time to allow complete dissolution of solute elements, followed by a rapid cooling to a low temperature ($T_1$), which by the end becomes a supersaturated non-equilibrium matrix phase. Then, the super-saturated matrix is heated up again to an intermediate temperature ($T_2$) and kept for a certain time in order to precipitate very fine particles, i.e. $\gamma'$ and $\gamma''$. The finely dispersed $\gamma'$ and $\gamma''$ phases create lattice strain in the interface which stops dislocation movements during plastic deformation and thus improves the strength and creep resistance \[17\][18].


However, the time for the aging should be carefully limited since a prolonged aging (over-aging) causes coarsening of the precipitates. A coarsened precipitate is larger in size and can result in a reduction in the strength since the dislocation movements can loop around the particle easier than for small precipitates\textsuperscript{[12]}. The addition of Al and Ti is proven to control the $\gamma'$ precipitation while Nb is controlling the $\gamma''$ phase. Furthermore, the precipitation of carbides also contributes to the strength as well as high temperature creep resistance.

It is believed that LMD promotes the precipitation hardening by creating the related conditions. During the LMD process, the material is melted by a laser with very high energy and immediately cooled when the laser is removed since the material is exposed to the atmosphere with a high specific surface. This process acts as the solution heat treatment where a single supersaturated $\gamma$ matrix phase is formed.

Subsequently when more layers are deposited above the solidified layers, the $\gamma$ phase should be constantly reheated and remained at an intermediate temperature which acts as the precipitation treatment (aging) and $\gamma'$ and $\gamma''$ phases are precipitated.

### 2.4 Porosity

Due to the characteristics of AM, porosity is commonly experienced. There are generally two types of pores existing in AM products, i.e. gas pores and shrinkage pores. The gas pores are introduced by the powders with entrapped gas as well as the mixture of powders and shielding gas during injection. The lack-of-fusion pores are produced when the material has an insufficient melting or two adjacent melt pools have an insufficient overlapping or too much shrinkage. The gas pores are spherical and normally exist occasionally with a rather
random distribution while the shrinkage pore is angular and appear periodically along the intersection between adjacent beads.

It is suggested that a Marangoni flow is driving the motion of the pores in the melt pool in AM \textsuperscript{[18]}. Figure 4 is a schematic illustration of the Marangoni flow, which shows how the flow goes along the edge of the pool from the centre to the bottom and rises due to the accumulation of mass. While the mass comes to the accumulation in the bottom centre of the melt pool, the bubbles are pushed together and have a chance to coalesce and form larger bubbles.

![Figure 4. Marangoni flow\textsuperscript{[17]}](image)

However, the significance of the Marangoni flow should be further verified since the effect of the density-driven convection might be stronger than the effect of Marangoni convection. The density-driven convection works as a descending flow of the mass and an ascending flow of the bubbles due to the density difference. In this study, the motion of the bubbles will be considered as following a procedure of nucleate-coalesce-ascend-escape.

The occurrence of pores is definitely detrimental to the mechanical property of AM products since it decreases the strength of the material and can be an initiation of cracks and consequently fractures. The lack-of-fusion pores can be manipulated and greatly minimized by optimized processing parameters while the gas pores can still exist even with optimized setting. In cases with extreme requirement for a low porosity, optimizations such as using pore-free powders, no shield gas and stable melt pools can be considered \textsuperscript{[18]}.

2.5 Directional Solidification

One important feature of AM is the occurrence of directional solidification (DS) \textsuperscript{[6,8,15]}. DS refers to the phenomenon that the melt solidifies in a single direction, resulting in a highly anisotropic structure in the solid. Especially in AM Alloy 718, due to the ultra-high cooling rate, the primary dendrite arms of the y phase predominantly grows towards the build-up direction which forms columnar structures while the secondary and subsequent dendrite
arms are minimized. The formation of the dendrites during the solidification process is caused by the undercooled liquid metal. The undercooled liquid initially causes the formation of solid nuclei in the melt which keeps growing during the solidification. At some point, the anisotropy in the surface of the solid-liquid interface leads to a preferred growth due to the attempt of the solid to minimize the surface energy. The minimization normally takes place at the tips of the dendrites with the highest specific surface energy, i.e. the lowest surface energy, which subsequently leads to the growth of the dendrite structures. The dendrites can have secondary dendrites growing out from the primary dendrites, and then even tertiary dendrites from the secondary. However, if a rather high cooling rate is applied, the primary dendrites will grow and solidify dominantly before the secondary and subsequent dendrites form. This, as discussed above, can be ensured by a high temperature gradient and a low solidification velocity.
3. Experiment

3.1 Objectives
Based on the aims of the thesis as well as the background study, the following objectives should be listed for this experiment:

1. To evaluate the characteristics of Alloy 718 produced by Laser Metal Deposition using powders (LMD-p) in terms of microstructure, porosity and hardness.
2. To understand the influence of processing parameters on the products’ characteristics.
3. To understand the hardening effect of LMD.

3.2 Experiment designs and processes
To achieve the objectives, two sets of experiments are designed respectively focusing on characteristics and hardening effect. Additionally, two more follow-up experiments are designed to study the distribution of porosity and the hardness gradient along the build-up direction.

3.2.1 Experiment 1
As presented in Figure 5, a group of samples with different processing parameters are prepared. The microstructure, porosity and hardness of the samples are evaluated in order to study the characteristics. The porosity and the hardness of the samples are then compared together with the processing parameters in order to understand the influence of the parameters.

<table>
<thead>
<tr>
<th>Sample preparation</th>
<th>Porosity evaluation</th>
<th>Metallographic study</th>
<th>Hardness evaluation</th>
</tr>
</thead>
</table>

Figure 5. Process of experiment 1

The processing parameters of the samples are selected based on literature surveys [19-24] with reported good performance (Table 3). The parameters are basically set to three levels as low, medium and high, generating different levels of heat input and line mass. Specially, Sample 1, 6 and 11 are made with exactly the same parameters with intermediate levels (700 W, 17.5 mm/s, 6 g/min, etc.) as a reference to verify the consistency of the results as well as to investigate the relevant performance.
Table 3. Process parameters for Experiment 1

<table>
<thead>
<tr>
<th>Laser power (W)</th>
<th>Scanning speed (mm/s)</th>
<th>Powder Feed rate (g/min)</th>
<th>Shield gas flow (L/min)</th>
<th>Powder standoff (mm)</th>
<th>Laser standoff (mm)</th>
<th>layer number</th>
<th>Exp No</th>
</tr>
</thead>
<tbody>
<tr>
<td>400</td>
<td>5</td>
<td>2</td>
<td>8</td>
<td>-1</td>
<td>5</td>
<td>15</td>
<td>4</td>
</tr>
<tr>
<td>400</td>
<td>5</td>
<td>10</td>
<td>8</td>
<td>1</td>
<td>10</td>
<td>5</td>
<td>5</td>
</tr>
<tr>
<td>400</td>
<td>30</td>
<td>2</td>
<td>15</td>
<td>1</td>
<td>10</td>
<td>15</td>
<td>7</td>
</tr>
<tr>
<td>400</td>
<td>30</td>
<td>10</td>
<td>15</td>
<td>-1</td>
<td>5</td>
<td>15</td>
<td>10</td>
</tr>
<tr>
<td>700</td>
<td>17.5</td>
<td>6</td>
<td>11.5</td>
<td>0</td>
<td>7.5</td>
<td>5</td>
<td>1</td>
</tr>
<tr>
<td>700</td>
<td>17.5</td>
<td>6</td>
<td>11.5</td>
<td>0</td>
<td>7.5</td>
<td>5</td>
<td>11</td>
</tr>
<tr>
<td>1000</td>
<td>5</td>
<td>2</td>
<td>15</td>
<td>-1</td>
<td>10</td>
<td>15</td>
<td>9</td>
</tr>
<tr>
<td>1000</td>
<td>5</td>
<td>10</td>
<td>15</td>
<td>1</td>
<td>10</td>
<td>5</td>
<td>15</td>
</tr>
<tr>
<td>1000</td>
<td>30</td>
<td>2</td>
<td>8</td>
<td>1</td>
<td>5</td>
<td>5</td>
<td>8</td>
</tr>
<tr>
<td>1000</td>
<td>30</td>
<td>10</td>
<td>8</td>
<td>-1</td>
<td>10</td>
<td>15</td>
<td>2</td>
</tr>
</tbody>
</table>

3.2.2 Experiment 2

As presented in Figure 6, a group of samples with the same processing parameters are prepared. The samples are heat treated for varying time and temperatures. One none-heat-treated sample is also spared as an as-deposited sample for reference. The microstructure and the hardness of the samples are evaluated and compared in order to understand the hardening effect of LMD process.

<table>
<thead>
<tr>
<th>Sample preparation</th>
<th>Heat treatment</th>
<th>Metallographic study</th>
<th>Hardness evaluation</th>
</tr>
</thead>
</table>

Figure 6. Process of experiment 2

The heat treatments are carried out in a small and fast responsive lab furnace in air environment. The samples are made with the highest heat input (Table 4). One sample is kept in the as-deposited condition for reference purpose. Another sample is solution heat treated at 950 °C for 1 hour followed by aging at 750 °C for 5 hours. The rest of the samples are direct-aged at 750 °C for 1, 5, 12 and 24 hours individually (Table 5).

Table 4. Process parameters for Experiment 2

<table>
<thead>
<tr>
<th>Laser power (W)</th>
<th>Scanning speed (mm/s)</th>
<th>Powder Feed rate (g/min)</th>
<th>Shield gas flow (L/min)</th>
<th>Powder standoff (mm)</th>
<th>Laser standoff (mm)</th>
<th>layer number</th>
</tr>
</thead>
<tbody>
<tr>
<td>1000</td>
<td>5</td>
<td>10</td>
<td>15</td>
<td>1</td>
<td>10</td>
<td>15</td>
</tr>
</tbody>
</table>
Table 5. Heat treatments of LMD samples made of Alloy 718

<table>
<thead>
<tr>
<th>Sample</th>
<th>Temperature</th>
<th>Time</th>
<th>Comment</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>As-deposited</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>1) 950 °C, 2) 750 °C</td>
<td>1)1h, 2)5h</td>
<td>Step 1) is air-cooled to room temperature before step 2)</td>
</tr>
<tr>
<td>3</td>
<td>750 °C</td>
<td>1h</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>750 °C</td>
<td>5h</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>750 °C</td>
<td>15h</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>750 °C</td>
<td>24h</td>
<td></td>
</tr>
</tbody>
</table>

3.2.3 Follow-up experiment: porosity distribution

Considering the probable existence of Marangoni flow\(^{[18]}\), there could be a pattern for the distribution of porosity. To verify this pattern, another 3 samples with the same processing parameters as Sample 5 (400 W, 5 mm/s, and 10 g/min) are made to investigate the pore distribution on the wall-side face which is perpendicular to the width of the single wall sample.

3.2.4 Follow-up experiment: hardness gradient

Considering the decreasing heating duration along the build-up direction in the samples, there could be a gradient of hardness due to precipitation hardening effect. In order to verify this gradient, a single wall sample with the highest heat input as well as a low porosity level is built with sufficient layers (parameters see Table 6). The sample is then cut across the longitudinal section and tested on the hardness along the build-up direction on each layer where three tests are carried out individually. The hardness of each layers are carefully taken 3 times from the left to the right side using a force of 2.94 N.

Table 6. Process parameters for the hardness gradient study

<table>
<thead>
<tr>
<th>Laser power (W)</th>
<th>Scanning speed (mm/s)</th>
<th>Powder Feed rate (g/min)</th>
<th>Shield gas flow (L/min)</th>
<th>Powder standoff (mm)</th>
<th>Laser standoff (mm)</th>
<th>layer number</th>
</tr>
</thead>
<tbody>
<tr>
<td>1000</td>
<td>5</td>
<td>10</td>
<td>15</td>
<td>1</td>
<td>10</td>
<td>20</td>
</tr>
</tbody>
</table>

3.3 Set-up and sample preparation

The set-up of the processing system consisted of a powder feeding module with a copper alloy nozzle, a laser module (IPG 6 kW YAG fibre laser) and a Robot arm controlling the movements (Figure 7). During the production, gas atomized Alloy 718 powders with a size of ~45 - ~75 μm were blown through the nozzle, and melted by the laser beam in order to be deposited on the under-laying material.
Figure 7. Set-up of the experiment, including a powder feeding module with a copper alloy nozzle, a laser module and a Robot arm.

The samples were built layer-by-layer into 15-layer single walls with a uniform length of 35 mm on a stainless steel substrate (Figure 8). Sample 5 and Sample 8 were built into only 5 layers since Sample 5 had poor penetration to the substrate which broke off from the substrate, whereas sample 8 had low build-up rate which produced no significant height.

When the samples are made, both the starting part and the finishing part (Figure 8), are cut off approximately at a distance of 8 mm from their respective edge and mounted. The cross-sections of both end-parts perpendicular to the scanning direction are ground and polished.

Figure 8. LMD single-wall sample
3.4 Experiment method

3.4.1 Metallographic study
The samples are first analysed using Light Optical Microscopy (LOM) in terms of porosity without etching and later etched electrolytically using oxalic acid using a voltage of 3 V. The etched samples subsequently go through microstructure observation by LOM (in this case Olympus BX60M) and an electron scanning microscope (SEM, HITACHI TM3000).

3.4.2 Porosity evaluation
The measurement of porosity level is based on the area percentage of the pores in the entire cross-section. Both the areas of the pores and the cross-sections are measured in the program ‘ImageJ’ by applying thresholds. For instance, a merged picture representing the entire cross-section (Figure 9.a) was first produced in PhotoShop with LOM photos taken throughout the cross-section. Then, a mask of highlighted pores (Figure 9.b) was produced in ImageJ and the areas were calculated in the program as well. It should be mentioned that the pores are carefully filtered by manual identification. Several shadowed areas are not counted as pores since no gradient of height was observed under higher magnification.

Figure 9. Demonstration of porosity measurement
3.4.3 Hardness test

HV Micro-hardness tests in Experiment 1 and 2 are carried out after the microstructure analysis using Shimadzu HMV micro hardness tester (0.98 N load, 15 s duration). The indents are taken at approximately every layer from the bottom to the top, i.e. 15 indents per sample, except from Sample 8 which was only 5 indents due to the small size.
4. Results

In this chapter, the results of designed experiments: experiment 1, experiment 2, follow-up experiment 1 and follow-up experiment 2 will be individually presented.

4.1 Experiment 1

4.1.1 Microstructure

The overview of a cross-section under LOM is presented in Figure 10.

Figure 10. (a) Dendrites in the bottom part of the sample; (b) Dendrites in the middle part of the sample; (c) Dendrites in the top part the sample; (d) Overview of the sample
The LOM observation for LMD samples discloses epitaxial growths of columnar dendrites throughout the whole sample. No micro-crack was found. The dendrites grow along the direction parallel to the build-up direction or with a small angle. SEM observation also sees the phases described in previous studies [8][11]: matrix of Gamma phase (γ) in the dendrite bodies, the precipitated Laves phase lying in the interdendritic region, as well as several Carbides (Figure 11). The spacing of primary dendrite arms is measured in Sample 7 (400 W, 30 mm/s, 2 g/min) and shows an increase from the bottom to the top (Table 7, Figure 12).

![Figure 11. SEM photo (X10000) of Sample 7 with matrix of Gamma phase (γ) in the dendrite bodies, the precipitated Laves phase lying in the interdendritic region, as well as several Carbides](image)

Table 7. Dendrite arm spacing distribution of Sample 7

<table>
<thead>
<tr>
<th>Sample</th>
<th>Location</th>
<th>Number of measurements</th>
<th>Spacing (μm)</th>
<th>Standard deviation (S.D.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>7</td>
<td>Bottom</td>
<td>3</td>
<td>5.92</td>
<td>0.35</td>
</tr>
<tr>
<td>7</td>
<td>Middle</td>
<td>3</td>
<td>9.61</td>
<td>0.96</td>
</tr>
<tr>
<td>7</td>
<td>Top</td>
<td>3</td>
<td>12.88</td>
<td>1.25</td>
</tr>
</tbody>
</table>

![Figure 12. Dendrite arm spacing distribution of Sample 7](image)
It is also found that some dendrites grow out in a radiation fashion from a centre located at the surface of the top (Figure 13), which indicated that some particles were partially melted and acted as a nucleation site.

![Figure 13. LOM photo of partially melted powder](image)

**4.1.2 Porosity**

The results of the porosity evaluations are listed in Table 8. From the results, the average level of porosity measured from the samples is ~0.06%. The results of Sample 1, 6 and 11 show a good consistency as produced with the same processing parameters.

The pores that were found in the overall samples all appeared spherical which indicates gaseous pores (Figure 14.a). No lack-of-fusion was found in the samples which thus prove that the LMD material was well fused when building a layer-by-layer single wall using powders. However, it does not mean that no lack-of-fusion would occur when building objects horizontally with adjacent beads.

The size distribution of the pores is shown in Figure 14.b. It shows that the size of the pores ranges from ~1 µm to 91 µm and the majority of the pores have sizes less than 20 µm. Although SEM observation disclosed smaller spherical pores with sizes of less than 1 µm entrapped in Laves phase, it might be a type of Laves mushy zone and is not considered in this study.
Table 8. Results of the porosity

<table>
<thead>
<tr>
<th>Sample name</th>
<th>Shield gas flow (L/min)</th>
<th>Laser power (W)</th>
<th>Scanning speed (mm/s)</th>
<th>Powder feed rate (g/min)</th>
<th>Porosity (area percent)</th>
<th>S.D.</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>8</td>
<td>400</td>
<td>5</td>
<td>2</td>
<td>0.037%</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>15</td>
<td>400</td>
<td>30</td>
<td>2</td>
<td>0.148%</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>8</td>
<td>400</td>
<td>5</td>
<td>10</td>
<td>0.209%</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>15</td>
<td>400</td>
<td>30</td>
<td>10</td>
<td>0.043%</td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>11.5</td>
<td>700</td>
<td>17.5</td>
<td>6</td>
<td>0.028%</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>11.5</td>
<td>700</td>
<td>17.5</td>
<td>6</td>
<td>0.048%</td>
<td>0.01%</td>
</tr>
<tr>
<td>11</td>
<td>11.5</td>
<td>700</td>
<td>17.5</td>
<td>6</td>
<td>0.055%</td>
<td></td>
</tr>
<tr>
<td>9</td>
<td>15</td>
<td>1000</td>
<td>5</td>
<td>2</td>
<td>0.004%</td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>8</td>
<td>1000</td>
<td>30</td>
<td>2</td>
<td>0</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>15</td>
<td>1000</td>
<td>5</td>
<td>10</td>
<td>0.040%</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>8</td>
<td>1000</td>
<td>30</td>
<td>10</td>
<td>0.067%</td>
<td></td>
</tr>
</tbody>
</table>

Figure 14. (a) Gaseous porosity; (b) Size distribution of pores

4.1.3 Hardness

The results of hardness tests for the general samples are listed in Table 9.

Table 9. Hardness of the samples in Experiment 1

<table>
<thead>
<tr>
<th>Exp No</th>
<th>4</th>
<th>5</th>
<th>7</th>
<th>10</th>
<th>1</th>
<th>6</th>
<th>11</th>
<th>2</th>
<th>3</th>
<th>8</th>
<th>9</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hardness (HV)</td>
<td>242</td>
<td>238</td>
<td>264</td>
<td>265</td>
<td>241</td>
<td>246</td>
<td>255</td>
<td>251</td>
<td>242</td>
<td>222</td>
<td>239</td>
</tr>
<tr>
<td>S.D.</td>
<td>9.4</td>
<td>13.7</td>
<td>16.0</td>
<td>10.5</td>
<td>8.4</td>
<td>10.9</td>
<td>13.8</td>
<td>8.2</td>
<td>11.6</td>
<td>9.8</td>
<td>8.6</td>
</tr>
</tbody>
</table>
The results of Sample 1, 6 and 11 show a good consistency as produced with the same processing parameters. No significant difference is found between the hardness of the starting part and the finishing part. It can be seen that the average HV hardness of the samples varies from 221 to 263. The lowest micro-hardness was found to be 200 while the highest value was found to be 284. It should be understood that the hardness is much higher when the indents hit a complete Laves phase or Carbide which are hard, whereas the hardness would be much lower when the indents hit a site right above an existing pore since the pore gives no support to the up-laying material and deforms when the indent is being taken. This might be one reason why the average hardness is lower for Sample 5 which has the highest porosity level.

4.2 Experiment 2
4.2.1 Microstructure

Figure 15 are the SEM photos of the heat treated samples: (a) as-deposited sample; (b) solution treated sample; (c) 1 hour direct-aged sample; (d) 5 hours direct-aged sample. It shows that in the solution treated sample (Figure 15.b), large amount of Laves-δ phase transformation occurred in the interdendritic regions \(^{[12]}\). Some Carbides were also found in the solution treated sample within the Laves phase (Figure 16), which are richer in Niobium as revealed by EDX analysis (Figure 16, Table 10 and Table 11). No significant Laves-δ phase transformation was found in the 1h direct-aged sample (Figure 15.c). However, in the 5h direct-aged sample, some small needle shaped δ phase appear inside the Laves phase, which is believed to be the early stage of Laves-δ phase transformation (Figure 15.d).
Figure 15. SEM images of heat treated samples. (a) Laves phase in the as-deposited sample; (b) 5 phase transformation in the solution treated sample; (c) Laves phase in the 1 hour direct-aged sample; (d) Laves phase in the 5 hours direct-aged sample.

Figure 16. EDX map in the solution treated sample (950 °C * 1h AC to RT + 750 °C * 5h)

Table 10. EDX Spectrum: A

<table>
<thead>
<tr>
<th>Element</th>
<th>Series</th>
<th>unn. C</th>
<th>norm. C</th>
<th>Atom. C</th>
<th>Error (3σ)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al</td>
<td>K-series</td>
<td>0.56</td>
<td>0.61</td>
<td>1.62</td>
<td>0.16</td>
</tr>
<tr>
<td>Ti</td>
<td>K-series</td>
<td>9.07</td>
<td>9.92</td>
<td>14.78</td>
<td>0.87</td>
</tr>
<tr>
<td>Cr</td>
<td>K-series</td>
<td>6.76</td>
<td>7.40</td>
<td>10.14</td>
<td>0.67</td>
</tr>
<tr>
<td>Fe</td>
<td>K-series</td>
<td>6.03</td>
<td>6.60</td>
<td>8.43</td>
<td>0.63</td>
</tr>
<tr>
<td>Ni</td>
<td>K-series</td>
<td>14.68</td>
<td>16.07</td>
<td>19.51</td>
<td>1.45</td>
</tr>
<tr>
<td>Nb</td>
<td>L-series</td>
<td>51.83</td>
<td>56.74</td>
<td>43.54</td>
<td>5.67</td>
</tr>
<tr>
<td>Mo</td>
<td>L-series</td>
<td>2.42</td>
<td>2.65</td>
<td>1.97</td>
<td>0.34</td>
</tr>
<tr>
<td>Total</td>
<td></td>
<td>91.35</td>
<td>100.00</td>
<td>100.0</td>
<td></td>
</tr>
</tbody>
</table>
Table 11. EDX Spectrum: B

<table>
<thead>
<tr>
<th>Element</th>
<th>Series</th>
<th>unn. C</th>
<th>norm. C</th>
<th>Atom. C</th>
<th>Error (3σ)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti</td>
<td>K-series</td>
<td>1.21</td>
<td>1.25</td>
<td>1.71</td>
<td>0.19</td>
</tr>
<tr>
<td>Cr</td>
<td>K-series</td>
<td>11.34</td>
<td>11.70</td>
<td>14.76</td>
<td>1.07</td>
</tr>
<tr>
<td>Fe</td>
<td>K-series</td>
<td>12.59</td>
<td>12.99</td>
<td>15.25</td>
<td>1.20</td>
</tr>
<tr>
<td>Ni</td>
<td>K-series</td>
<td>38.19</td>
<td>39.41</td>
<td>44.02</td>
<td>3.60</td>
</tr>
<tr>
<td>Nb</td>
<td>L-series</td>
<td>24.83</td>
<td>25.61</td>
<td>18.08</td>
<td>2.76</td>
</tr>
<tr>
<td>Mo</td>
<td>L-series</td>
<td>8.76</td>
<td>9.04</td>
<td>6.18</td>
<td>1.00</td>
</tr>
</tbody>
</table>

Total 96.92 100.00 100.0

4.2.2 Hardness

The results of hardness are presented in Table 12 and Figure 17.

Table 12. Hardness of heat treated samples

<table>
<thead>
<tr>
<th>Sample</th>
<th>Temperature</th>
<th>Time</th>
<th>Hardness (HV)</th>
<th>S.D.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>\</td>
<td>\</td>
<td>234</td>
<td>5.7</td>
</tr>
<tr>
<td>2</td>
<td>1) 950 °C, 2) 750 °C</td>
<td>1)1h, 2)5h</td>
<td>382</td>
<td>7.2</td>
</tr>
<tr>
<td>3</td>
<td>750 °C</td>
<td>1h</td>
<td>338</td>
<td>6.2</td>
</tr>
<tr>
<td>4</td>
<td>750 °C</td>
<td>5h</td>
<td>360</td>
<td>5.7</td>
</tr>
<tr>
<td>5</td>
<td>750 °C</td>
<td>15h</td>
<td>382</td>
<td>6.2</td>
</tr>
<tr>
<td>6</td>
<td>750 °C</td>
<td>24h</td>
<td>378</td>
<td>8.0</td>
</tr>
</tbody>
</table>

![Figure 17. Hardness of heat treated samples](image)

It shows that the as-deposited sample has a hardness of 234 which is similar to as-cast 718 [1]. By direct-aging at 750 °C, the hardness of the samples increases and reaches a maximum value around 382 after 10 to 15 hours. It means that direct-aging at 750 °C can
improve the hardness of the laser deposited sample by ~60%. Meanwhile, the same level of hardness can be achieved by 1 hour of solution treatment at 950 °C followed by 5 hours aging at 750 °C.

4.3 Follow-up experiment: porosity distribution

The overviews (longitudinal section) of the porosity distribution in one sample are presented in Figure 18.a and 18.b. The sample was cut in halves: Figure 18.a is the half with the finishing part and Figure 18.b is the half with the starting part. It shows that the sample was made by scanning from the right side of Figure 18.b to the left side of Figure 18.a, and from the bottom layer to the top layer.

Figure 18. Overviews of porosity distribution in the sample (pores marked white). (a) The half with the finishing part; (b) The half with the starting part.
4.3.1 Porosity distribution along the length

To evaluate the porosity along the length, the area is divided into 3 parts: starting part, middle part and finishing part. The results are presented in Table 13. It shows that middle part has the highest porosity level in comparison to the starting part and the finishing part. However the porosity levels of all three sections are too low, thus the focus should be on the number of pores as well as the pore size. As shown in Figure 19, the number of pores decreases from the starting part to the finishing part by 24% from 229 to 174, while the pore size (average area) linearly increases by 15.26% from 308.11 µm² to 261.94 µm².

Table 13. Longitudinal distribution of porosity in the sample

<table>
<thead>
<tr>
<th>Section</th>
<th>Porosity</th>
<th>S.D.</th>
<th>Number</th>
<th>S.D.</th>
<th>Pore size (µm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Starting part</td>
<td>0.082%</td>
<td></td>
<td>229</td>
<td></td>
<td>261.94</td>
</tr>
<tr>
<td>Middle part</td>
<td>0.088%</td>
<td>0.01%</td>
<td>216</td>
<td>2.4</td>
<td>285.90</td>
</tr>
<tr>
<td>Finishing part</td>
<td>0.079%</td>
<td></td>
<td>174</td>
<td></td>
<td>308.11</td>
</tr>
</tbody>
</table>

Note: Standard deviations (S.D.) for Porosity and Number are obtained from Sample 1, 6 and 11 with the same process parameters; S.D. for Pore size (average area) is not necessary since the deviation is too large and the purpose of having pore size calculated is not to obtain an accurate value but to estimate the size distribution.

![Figure 19. Porosity distribution along the length](image)

4.3.2 Porosity distribution along the height

The porosity distribution along the height is evaluated by layers from the bottom to the top. The results are presented in Table 14 and Figure 20. It shows that the top layer has the highest porosity level at ~0.1% which is ~1.7 times the amount of the lowest. The porosity
levels remain at around 0.07% in the rest of the layers except from the third layer which is slightly higher at ~0.09%. The pore numbers and the pore size show the same trend whereas the differences that the top layer makes are more significant. The pore number of the top layer is 195 which is as 2 times as the amount of the lowest and the pore size is 302.2 µm² which is 68.5 µm² larger than the lowest.

Table 14. Porosity in each layer of the LMD sample

<table>
<thead>
<tr>
<th>Layers</th>
<th>Porosity</th>
<th>S.D.</th>
<th>Number of pores</th>
<th>S.D.</th>
<th>Pore size (µm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.112%</td>
<td></td>
<td>195</td>
<td></td>
<td>302.24</td>
</tr>
<tr>
<td>2</td>
<td>0.068%</td>
<td></td>
<td>107</td>
<td></td>
<td>250.54</td>
</tr>
<tr>
<td>3</td>
<td>0.088%</td>
<td>0.01%</td>
<td>145</td>
<td>2.4</td>
<td>267.70</td>
</tr>
<tr>
<td>4</td>
<td>0.070%</td>
<td></td>
<td>96</td>
<td></td>
<td>280.89</td>
</tr>
<tr>
<td>5</td>
<td>0.067%</td>
<td></td>
<td>105</td>
<td></td>
<td>233.74</td>
</tr>
</tbody>
</table>

Figure 20. Porosity distribution along the height of the LMD sample

4.4 Follow-up experiment: hardness gradient

The hardness along the height of the sample is presented in Figure 21. No significant hardness gradient was found. Instead, the distribution of the hardness along the build-up direction fluctuates in the same way as in Experiment 1. The micro-hardness interval between the highest and the lowest is about 30.
Figure 21. Hardness distribution from the bottom of the sample to the top (Extreme values are marked as ‘X’ and not included in the average value)
5. Discussion

In this chapter, the results of the experiments will explained and discussed, focusing on the dendritic growth, effect of processing parameters on porosity, porosity distribution, hardness gradient, and the hardening effect of LMD process.

5.1 Dendritic growth

From the LOM result in Experiment 1, it can be seen that the bottom part of the deposit which is close to the substrate presents denser and larger areas of dendrites (Figure 10.d). Under higher magnification (Figure 10.a), it also shows that the dendrites in the bottom part are mainly primary dendrites growing out from the interface of the substrate, together with only a few secondary dendrites. The reason is that the substrate which is at room temperature having a large surface in-relation, acts as a heat sink for heat conducting away from the deposit and rapidly cools down the material. The cooling effect from the substrate then creates a high cooling rate and large temperature gradient from the bottom to the top which is an ideal condition for primary dendrite growth. SEM observation also shows long, thin and continuous morphology of Laves phase precipitated along the interdendritic region (Figure 10.a) which is faced because the secondary dendrites are not well developed in this area and thus the continuous space between primary dendrites is majorly provided for Laves phase precipitation.[25]

More well-developed secondary dendrites appear in the middle part (Figure 10.b) and longer secondary dendrites appear in the top part (Figure 10.c). It can be explained by the decreasing cooling rate as the distance to the substrate has increased. The morphology of the interdendritic Laves phase becomes shorter and discontinuous since the secondary dendrites are developed and cut through the Laves phases (white area).

5.2 Effect of processing parameters on porosity

The effect of processing parameters on porosity is compared in Figure 22. The discussed parameters will be laser power, scanning speed, powder feed rate and shield gas flow, which are believed to influencing the gas entrapment and powder melting.
The results from Table 8 show that the samples generally have low levels of porosity which are less than 0.1% except for Sample 5 and Sample 7. In this study, porosity levels lower than 0.1% will be considered as low, whereas levels above 0.1% will be considered as high. The comparisons and evaluations will be made among individual groups of samples as follows:

- **Sample (1, 6, and 11):**

  Sample 1, 6 and 11 are all made with the same process parameters. It can be seen in Table 15 that the porosity levels of Sample 1 (0.03%), Sample 6 (0.05%) and Sample 11 (0.06%) were close enough indicating that the porosity levels of the samples made by the same processing parameters are consistent and the results from the porosity measurement method is reliable to use for evaluation and comparison. Thus, the standard deviation based on the three samples is 0.01%.

**Table 15. Porosity of Sample 1, 6 and 11**

<table>
<thead>
<tr>
<th>Sample</th>
<th>1</th>
<th>6</th>
<th>11</th>
</tr>
</thead>
<tbody>
<tr>
<td>Porosity</td>
<td>0.03%</td>
<td>0.05%</td>
<td>0.06%</td>
</tr>
<tr>
<td>Standard deviation</td>
<td></td>
<td></td>
<td>0.01%</td>
</tr>
</tbody>
</table>
- **Sample 5:**
  Sample 5 has a high porosity level at 0.2% and had a complete separation from the substrate in the end of the process, which suggested a poor fusion penetration to the surface of the substrate. The reason might be that the processing parameters of Sample 5 provided it with a low laser power (400 W) while blowing in powders with a high feed rate (10 g/min). Thus, the heat input was not high enough to melt all the blown powder, neither to form a good melt pool. It resulted in a high amount of entrapped gas bubbles from the blown powders despite a low shield gas flow (8 L/min), which were not able to coalesce and escape from the deposit but formed pores inside the solidified sample. This indicates that the porosity level might be influenced by the amount of heat input needed for the formation of a good melt pool. The good melt pool here should be described as giving the bubbles enough energy and retention time to coalesce, ascend and escape from the melt surface before it solidifies.

- **Sample 5 vs. Sample 10:**
  Sample 10 with the same laser power (400 W) and feed rate (10 g/min) as Sample 5, however, generated a low porosity level (0.04%). The reason might be that the scanning speed of this sample is much higher (30 mm/s) which means shorter retention time for the powder blowing, thus fewer powders were blown into the melt pool. The amount of powder can be completely melted by the heat input in order to obtain a good melt pool and complete the ‘coalesce – ascend – escape’ process before complete solidification. It indicates that the porosity level is influenced by the powder input and increasing scanning speed can reduce the powder input and thus lower the porosity level. It should also be noticed that when the scanning speed is higher, the retention time of the laser exposure is reduced. Thus, it can be interpreted that the reduced laser exposure time still gives enough heat input for the gas bubble to complete the ‘escaping process’.

- **Sample 5 vs. Sample 4:**
  Sample 4 has a low porosity level at 0.04%, and the difference in the process parameter in comparison to Sample 5 is that it has a lower powder feed rate (2 g/min). It can be understood as, although the laser power is low (400 W) and the scanning speed is low (5 g/min) which means low heat input upon more powders, the reduced powder feed rate is still able to reduce the overall powder input and form a good melt pool. It proves that the
powder input influences the porosity level, and indicates that by reducing powder feed rate it is possible to reduce the powder input and thus lower the porosity level.

- **Sample 7 vs. Sample 4**
  Sample 7 has a relatively high porosity level at 0.2% while Sample 4 with the same laser power (400 W), scanning speed (5 mm/s) and powder feed rate (2 g/min) (i.e. having the same heat input and powder input). The major difference between these two samples is that Sample 7 has a higher shielding gas flow (15 L/min) which can be understood as increasing the chance of gas entrapment. It indicates that the shielding gas flow is also able to increase the porosity in some cases.

- **Sample 7 vs. Sample 10:**
  This part of the comparison reveals a contradictive situation to the previous discussions. As is shown, Sample 10 has a higher powder feed rate, which means more powder input, has a lower porosity level than Sample 7. The reason is not clear. An attempt of the explanation can be as the porosity level of Sample 7 is still within a ‘low’ range which means that the porosity difference between these two samples (0.1%) is not great enough to make a critical difference, in which case the indication from the ‘7 vs. 4’ could be incorrect. Another attempt can be that the actual porosity difference between these two samples could be smaller due to the error of the measurements.

- **Sample 3:**
  Sample 3 is an extreme example of the laser power’s effect on the porosity. It is produced with the highest powder feed rate (10 g/min), lowest scanning speed (5 mm/s) and the highest shield gas flow (15 L/min), which should make it difficult to obtain a good melt pool based on previous discussion. However, with the highest laser power (1000 W), the porosity is surprisingly low as 0.04%. This indicates that when the laser power is high enough, it can dominantly diminish the porosity with a good melt pool. This would also explain the low porosity levels for the samples (2, 3, 8, and 9) produced with 1000 W laser power.

To conclude, the condition for a low porosity level is the formation of a good melt pool. The good melt pool should be able to provide the bubbles enough energy and retention time to coalesce, ascend and escape from the melt surface before it solidifies. The process parameters, based on the present investigation, are assumed to have the following influence on the porosity levels of the samples:
(1) The heat input has a decreasing effect on the porosity. The heat input is a result from the laser power, scanning speed and laser spot size. It can be recommended to refer to the Specific Energy (see 1.2 Laser metal deposition);

(2) The laser power can dominantly diminish the porosity when it exceeds certain criteria;

(3) The powder input has an increasing effect on the porosity. The powder input is a result from the powder flow rate and the scanning speed. It can be recommended to refer to the Line Mass (see 1.2 Laser metal deposition);

5.3 Porosity distribution

5.3.1 Porosity distribution along the length

From the results in Figure 19, it shows that there are more pores in the starting end of the deposit compared to the finishing part while the sizes of pores are larger in the finishing part. The reason might be that the finishing part of each layer went down due to its liquid statues and lack of support from the side, which exposed more surfaces and shortened the height of the layer (Figure 8). It shortened the distance for the bubbles to travel and increased the chance for them to coalesce (resulted in bigger pores in the finishing end) and escape from the surface (resulted in lower number and lower area percentage of pores).

The reason for the increasing size of the pores from the starting end to the finishing end might be that during the deposition of each layer, part of gas bubbles stayed in the solidified area while the rest of the bubbles stayed inside the flow and followed the advancing of the melt pool. While the melt pool was moving forward, more entrapped gas was blown into it and coalesce with the existing bubbles. Thus, the size of bubbles keeps growing along the deposition path.

5.3.2 Porosity distribution along the height

From the results in Figure 20, it shows that the top layer of the deposit has the highest in porosity level, pore number and the average pore area. It can be explained that most of the pores tended to ascend to the top of each layer. When a new layer was being made, the laser partially melt the top of the underneath layer and then the gas entrapped in the underneath layer went up to the new melt pool and repeat the same process. At the same time, the larger the bubble is, the faster it ascends, and thus more large pores will eventually present in the top layer.
5.4 Hardness gradient

The reason of believing that there could be a hardness gradient along the build-up direction of the samples is as followed: when building up subsequent layers, the previously built layers are heated up repeatedly and thus have longer heating time which is related to the aging effect. The fast cooled deposits are supersaturated during the process, and when subjected to aging, fine γ' and γ'' hardening phases start to precipitate, i.e. precipitation hardening. Thus the higher it builds, the longer aging occurs for lower layers, and together with the anisotropy effect of epitaxial columnar dendrites, the deposit is expected to create a decreasing hardness gradient along the build-up direction.

However, from the results in Figure 21, no significant hardness gradient was found along the height of the sample, together showing a limited level of fluctuation (<30). It might indicate that the aging effect of the repeated heating is not great enough to make a significant difference in the degree of precipitation hardening, thus difference in hardness. However, this fact might be an advantage for producing objects requiring isotropic performance.

5.5 Hardening effect of LMD process in Alloy718

The hardness curve of the heat treated samples indicates that the effect of precipitation hardening that LMD can reach is limited in these samples, as the hardness of as-deposited sample is similar to as-casted sample. However, potentials for improving the hardening effect still exists basing on improving both solution effect and aging effect.

The potential in solution effect can be seen from the higher hardness that the solution treated sample was able to reach. It means that if the LMD process can achieve a higher super-saturation level, i.e. higher temperature, longer heating duration and faster cooling, then higher hardness can be reached by subsequent treatments. Meanwhile, the potential in aging effect can be seen from the rapid increase of hardness during the first hour of aging. It means that a longer aging duration in LMD process can easily improve the hardness of as-deposited samples.

Moreover, the presence of the δ phases at the grain boundaries has been reported to be favourable for enhancing the ductility as well as the toughness of the material. The reason is that the thin and long needle shaped morphology provides restrictions to the grain boundary movement such as sliding\textsuperscript{[12]}. 
6. Conclusion

Following the focus areas of the evaluations, the conclusions of this study will be presented in corresponding areas – microstructure, porosity, hardness and heat treatment.

6.1 Microstructure

1) The laser deposition process investigated had a directional solidification microstructure with columnar dendrites along the build-up direction. Typical phases such as austenitic γ phase, eutectic (γ + Laves) phase and carbides were also observed in the deposit.

2) The bottom part of the deposit had mostly primary dendrites, while the middle part had more well-developed secondary dendrites followed by the top part with ternary dendrites.

3) The primary dendrite arm spacing increases from the bottom to the top.

4) The Laves phase area percentage was the highest in the bottom of the deposit while it slightly decreased from the middle to the top.

5) No micro-crack was found in any of the LMD samples.

6.2 Porosity

1) The morphologies of the pores in the produced samples are all spherical gaseous pores. No lack-of-fusion pore was found.

2) The pore sizes range from ~1 µm to 90 µm while the majority of the pores have sizes less than 20 µm.

3) Well fused samples generally have levels of porosity less than 0.1%. However a sample with higher porosity, poorer fusion and poor penetration to the surface of the substrate was found with a laser power of 400 W, powder feed rate of 10 mg/s and scanning speed of 2 mm/s.

4) A higher pore number was found in the starting part of the deposit while larger sizes of pores were found in the finishing part.

5) The top layer of the deposit was the highest in porosity level, pore number and the average pore area.

6.3 Hardness

1) The average HV hardness of the deposited samples varies from ~210 to 270. The lowest micro-hardness was found to be 200 while the highest value was found to be 284.

2) No significant hardness gradient was found.
6.4 Heat treatment

1) In solution heat treated samples, large amount of needle shaped δ phase transformed from Laves phases in the interdendritic regions. Carbides rich in Niobium were also found within the remaining Laves phase. In direct-aged samples, some small needle shaped phase appear at the boundaries of Laves phase, which is believed to be the early stage of Laves-to-δ phase transformation.

2) The as-deposited samples had an average hardness of ~230 which is similar to as-cast 718.

3) By direct-aging the deposited samples at 750 °C, the maximum hardness around ~380 is reached after 10 to 15 hours of aging. The same hardness was achieved by 1 hour of solution treatment at 950 °C followed by 5 hours aging at 750 °C.
7. Acknowledgements

This thesis project was supported by GKN Aerospace Sweden AB. The work was performed at University West (HV) and specifically at the Production Technology Centre (PTC) and Innovatum in Trollhättan, Sweden. Some of the analysis work was also performed in the Material Science lab at the Royal Institute of Technology (KTH). My studies at KTH were supported by the Swedish Institute Study Scholarship.

I would like to thank Dr. Joel Andersson from GKN Aerospace Sweden AB and Prof. Anders Eliasson from KTH for their arrangement, guidance and support to my work throughout the project.

I would like to thank Mr. Andreas Segerstark from University West for his tremendous help with the entire sample production as well as with my daily work.

I would like to thank Mr. Kjell Hurtig and Kenneth Andersson from the PTC at HV for all the technical support.

I give my sincere acknowledgements to the above mentioned institutions and personnel.
8. References


