Electron beam melting of Alloy 718 - Influence of process parameters on the microstructure

Paria Karimi
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To my beloved husband- Esmaeil
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Additiv tillverkning (AM) är namnet för att illustrera teknologier som genom att bygga lager för lager används till att tillverka 3D-komponenter av exempelvis metalliska, polymera och keramiska material. En AM-komponent är vanligtvis konstruerad med hjälp av en dator och med ett så kallat CAD-program (Computer Aided Design). När CAD-ritningen är klar så skickas den till AM-maskinen som i sin tur bygger upp komponenten lager för lager tills den slutliga 3D-geometrin uppnås. Det finns flera olika typer av AM-metoder där just den s.k. elektronstrålesmältningsmetoden (EBM), en metod som är klassificerad som en pulverbäddsmetod, har använts i forskningen som här presenteras. Med EBM så tillverkas komponenter genom att metalliskt pulver smälts med hjälp av en energität elektronstråle. EBM erbjuder ökade möjligheter i jämförelse med konventionella tillverkningsmetoder till att skräddarsy specifika komponenter gällande exempelvis flyg-, rymd- och medicinteknisk industri. EBM ger också möjligheten att tillverka betydligt mer geometriskt komplicerade komponenter än vad som är ekonomiskt gynnsamt om ens möjligt med konventionella tillverkningsmetoder.

Huvudfokus för den forskning som presenteras här har delats in i tre olika fokusområden och börjar med ett bredare perspektiv för att sedan snävas av. i) Första fokusområdet handlar om att finna samband mellan lägesrelaterade parametrar (avstånd mellan provkroppar, höjd från byggplattan och avstånd mellan provkroppar) och vilken inverkan de har på materialets mikrostruktur. Det visade sig att avståndet mellan provkropparna samt att höjden från byggplattan kan ha en signifikant effekt med avseende defektpopulation samt andelen Nb-rika strukturbeståndsdelar. ii) I det andra fokusområdet, utfördes experiment för att titta närmare på vad geometriskt relaterade parametrar hade för inverkan på legering 718 genom att producera enkelsträngar bredvid varandra samt även genom att bygga enkelvägar. Det primära syftet med studien var således att öka kunskapen till inverkan av successivt upprepade termiska cyklar på materialets mikrostruktur och dess utveckling. iii) I sista fokusområdet så undersöktes sambanden mellan maskinrelaterade parametrar (strålhastighet, strömsgård och fokus-offset) och dess inverkan på geometri (smältbredd, smälthöjd, omsmältningstjocka samt kontaktvinkel) samt mikrostruktur (som exempelvis kornstruktur och primärarmsavstånd) gällande enkelsträngar av legering 718. Forskningen påvisade att strålhastigheten och strömstyrkan var de parametrar som hade störst signifikans gällande geometri och mikrostruktur för s.k. enkelsträngarna.
Additive manufacturing (AM) is the name given to the technology of building 3D parts by adding layer-by-layer of materials, including metals, plastics, concrete, etc. Of the different types of AM techniques, electron beam melting (EBM), as a powder bed fusion technology, has been used in this study. EBM is used to build parts by melting metallic powders by using a highly intense electron beam as the energy source. Compared to a conventional process, EBM offers enhanced efficiency for the production of customized and specific parts in aerospace, space, and medical fields. In addition, the EBM process is used to produce complex parts for which other technologies would be either expensive or difficult to apply.

This thesis has been divided into three sections, starting from a wider window and proceeding to a smaller one. The first section reveals how the position-related parameters (distance between samples, height from build plate, and sample location on build plate) can affect the microstructural characteristics. It has been found that the gap between the samples and the height from the build plate can have significant effects on the defect content and niobium-rich phase fraction. In the second section, through a deeper investigation, the behavior of Alloy 718 during the EBM process as a function of different geometry-related parameters is examined by building single tracks adjacent to each other (track-by-track) and single-wall samples (single tracks on top of each other). In this section, the main focus is to understand the effect of successive thermal cycling on microstructural evolution. In the final section, the correlations between the main machine-related parameters (scanning speed, beam current, and focus offset) and the geometrical (melt pool width, track height, re-melted depth, and contact angle) and microstructural (grain structure, niobium-rich phase fraction, and primary dendrite arm spacing) characteristics of a single track of Alloy 718 have been investigated. It has been found that the most influential machine-related parameters are scanning speed and beam current, which have significant effects on the geometry and the microstructure of the single-melted tracks.
Appended Publications

This thesis is written based on the following appended publication;

Paper A.  
Influence of build layout and orientation on microstructural characteristics of electron beam melted Alloy 718  
Paria Karimi, Esmaeil Sadeghi, Dunyong Deng, Hans Gruber, Joel Andersson, Per Nylén  

Paper B.  
Microstructure development in track-by-track melting of EBM-manufactured Alloy 718  
Paria Karimi, Dunyong Deng, Esmaeil Sadeghi, Jonas Olsson, Joakim Ålgårdh, Joel Andersson  
Proceedings of the 9th International Symposium on Superalloy 718 & Derivatives, 3-6 June 2018; Pittsburgh, Pennsylvania, USA

Paper C.  
Influence of successive thermal cycling on microstructure evolution of EBM-manufactured Alloy 718 in track-by-track and layer-by-layer design  
Paria Karimi, Esmaeil Sadeghi, Pia Åkerfeldt, Joakim Ålgårdh, Joel Andersson  

Paper D.  
EBM-manufactured single tracks of Alloy 718: Influence of energy input and focus offset on geometrical and microstructural characteristics  
Paria Karimi, Esmaeil Sadeghi, Joakim Ålgårdh, Joel Andersson  
Submitted to Journal of Materials Characterization, Sep. 2018

As the main Author, Paria Karimi has performed all the experimental characterization, analyzed all the results, designed the structure of all articles, and had the main responsibility in writing the articles. Co-authors contributed in formulating concepts and ideas, planning the project, manufacturing and article editing.
Related work

The following paper is not appended but relevant to the work presented in this thesis.

- **Influence of laser exposure time and point distance on 75-μm-thick layer of selective laser melted Alloy 718**
  Paria Karimi, Tahira Raza, Joel Andersson, Lars-Erik Svensson
  Journal of Advanced manufacturing technology, pp. 1–9, Sep. 2017; doi.org/10.1007/s00170-017-1019-1

- **Effect of powder recycling on the fracture behavior of electron beam melted Alloy 718**
  Hans Gruber, Paria Karimi, Eduard Hryha, Lars Nyborg

- **Effect of heat treatment and hot isostatic pressing on oxidation behavior of EBM-manufactured Alloy 718**
  Esmaeil Sadeghi, Paria Karimi, Mohsen Seifi, Joel Andersson,
  EBAM 2018, 11-13 April 2018; Nurnberg, Germany

- **Isothermal oxidation behavior of EBM-additive manufactured Alloy 718**
  Esmaeil Sadeghi, Paria Karimi, Pimin Zhang, Ru Peng, Joel Andersson, Lars Pejryd, Shrikant Joshi
  Proceedings of the 9th International Symposium on Superalloy 718 & Derivatives, 3-6 June 2018; Pittsburgh, Pennsylvania, USA

- **Influence of thermal post treatments on microstructure and oxidation behavior of EBM-manufactured Alloy 718**
  Esmaeil Sadeghi, Paria Karimi, Soroush Momeni, Mohsen Seifi, Anders Eklund, Joel Andersson
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Abbreviations and nomenclature

3D: Three-Dimensional
AM: Additive Manufacturing
APB: Anti Phase Boundary
BSE: Backscatter detector
BCT: Body Centered Tetragonal
CAD: Computer Aided Design
EBM: Electron Beam Melting
EBSD: Electron Backscattered Diffraction
EDS: Energy Dispersive Spectroscopy
EDM: Electrical Discharge Machining
FCC: Face Centered Cubic
GBs: Grain Boundaries
L: Layer
LOM: Light Optical Microscopy
PRS: Powder Recovery System
PBF: Powder Bed Fusion
SEM: Scanning Electron Microscopy
SLM: Selective Laser Melting
STC: Successive Thermal Cycling
T: Track
TCP: Topologically Close Packed
TEM: Transmission Electron Microscopy
γ: Gamma
\(\gamma'\): Gamma prime
\(\gamma''\): Gamma double prime
\(\delta\): Delta
\(G\): Thermal gradient
\(R\): Solidification rate
\(\bar{T}\): Cooling rate
1 Introduction

Additive manufacturing (AM) technology has excited the aerospace world owing to its capabilities compared to the traditional manufacturing processes [1]. This manufacturing approach is favorable for the aerospace industry because of reduced raw material usage, which leads to lower buy-to-fly ratios that are important for commercial air traffic [2]. The potential for lower fuel consumption owing to lighter components that are manufactured through AM technologies results in the valuable benefit of reduced CO₂ emissions for the aerospace industry.

Electron beam melting (EBM), as a powder bed fusion (PBF) technique, in AM is a unique manufacturing process that has noticeable potential to reduce material waste through near net shape production and increase the value of the manufactured part [3], [4]. The increased value of EBM-manufactured parts originates from their increased structural strengths, the integration of parts to reduce the number of components, and the lowered weights of the products [5], [6]. Additionally, the EBM process has the potential to be used in repair applications in sensitive aerospace parts [7]–[9]. Moreover, the EBM process is ideal for the direct manufacturing of complex parts in low volumes such as fuel nozzles in aerospace industry. The process facilitates customization of parts from computer aided design (CAD) to a 3D metal part in complex geometries, which is difficult to achieve in other manufacturing technologies, thus providing better performances of the parts and adding value to the customer [5]. The future of EBM is certainly promising, but to fully exploit its advantages and disadvantages, the process parameters-microstructure-properties relationship needs to be more closely investigated.

1.1 Objectives and research questions

An increased understanding of how the material characteristics are influenced by the process parameters is of great importance, as this helps in retaining the consistency and repeatability of the EBM process. This work specifically aims to advance the understanding of how Alloy 718 behaves during the EBM process and how the material characteristics (e.g., defects and microstructure) are affected by the position, geometry, and machine-related parameters. The knowledge obtained can be used to tailor the material characteristics depending on the target application.
The objectives of this research are to establish the relationship between: i) the position-related parameters (e.g., part location (X-Y plane), the gap between parts, and height from the build plate (Z-axis)) and the as-built microstructures of parts, ii) explore the influences of successive thermal cycling (the so-called “STC”) by means of the geometry-related parameters of the as-built materials, and, iii) investigate the effects of machine-related parameters on the as-built microstructure, see Figure 1-1. Based on the knowledge gained, the overall goal is to propose a process window to build parts with less defects and having the desired microstructures.

The objectives presented in Figure 1-1 could be achieved by addressing the research questions (RQs) listed below:

- **RQ1**: How can the position-related parameters affect the amount of defects and the microstructure of EBM-manufactured Alloy 718?
- **RQ2**: What is the influence of the geometry-related parameters on the microstructure of EBM-manufactured Alloy 718?
- **RQ3**: How can the machine-related parameters influence the geometrical and microstructural characteristics of EBM-manufactured Alloy 718?

### 1.2 Structure of the thesis

This thesis consists of eight chapters, including this introduction that briefly describes the framework of this research and the structure of the thesis. Chapter
INTRODUCTION

2 provides a background of metal AM for a better understanding of the main motivation behind this study and why this work is relevant to the scientific society, specifically to the AM community. A literature review of metal AM and the EBM process is presented in this chapter. Chapter 3 explains the main characteristics of Alloy 718 only, and its microstructural characteristics during the EBM process. Chapter 4 describes the experimental procedure and the characterization techniques utilized for the material investigation. Details of the process conditions and the design of experiments are given as well. In Chapter 5, the results are discussed. Chapter 6 summarizes the articles published in relation to this work, which are appended to the thesis. Finally, Chapters 7 and 8 present the conclusions and the recommendations for future work, respectively.
2 Metal additive manufacturing

The definition of additive manufacturing (AM), according to the American Society for Testing and Materials (ASTM) International standard, is as follows:

“The process of joining materials to make objects from 3D-model data, usually layer upon layer, as opposed to subtractive manufacturing methodologies, such as traditional machining [3], [10]”.

In general, the goal of metal AM technologies is to produce lighter and stronger metal parts and reduce or eliminate post processing steps, which decrease the overall costs of unique exclusive parts in the i.e. aerospace or automotive industry [11]. Even though metal AM is beneficial in terms of complex designs and costs, there is still a long way to go to understand the complex thermo-physical phenomena such as the beam-powder-substrate interactions, heat transfer mechanisms, thermal stresses, and phase transformations. All these phenomena significantly affect the quality and the properties of the final part. Moreover, owing to the localized, fast, and transient nature of the metal AM process, quantitative experimental measurements are extremely difficult [12], [13].

2.1 Benefits and limitations of metal AM

One of the major benefits of metal AM is its great potential for the design of complex shapes and geometries without using dies and molds or employing a tooling process like milling, which is a challenge in conventional manufacturing techniques such as injection molding [14], [15]. In addition, the metal AM technologies are considered to be highly favorable for low-volume productions, e.g., the production of single parts [16], [17]. Depending on the demand, the elimination or reduction of a mechanical post processing step like machining leads to significant decreases in the production ramp-up time, cost, and waste. Another advantage of the metal AM techniques is the possibility of rapidly changing a design. For the specified parts, shorter lead times, lower inventories, and the integration of multiple parts can be achieved through metal AM. Moreover, metal AM offers the possibility of simultaneously manufacturing multiple individual parts [18], [19]. These benefits make metal AM an interesting alternative in many industries, e.g., aerospace and space [20].

On the other hand, the as-built material properties of a part manufactured by metal AM may be influenced by its geometry [21], [22]. Therefore, there is no optimal process setting that works in all instances. Part orientations, heights, and
angles can influence the different localized heat concentrations, leading to various types of defects, phase transformations, or residual stresses [23], [24].

The main disadvantages of the parts manufactured by metal AM are poor surface finish and poor dimensional accuracy. By adding layer upon layer in metal AM, the layer resolution produces a staircase appearance on the surface, which is inherent in all the metal AM techniques. This induces problems like the poor surface finish, and, in combination with other factors, is responsible for the poor dimensional accuracy [25]. Nowadays, some parameters like layer thickness or the temperature profile of the melt pool are controlled to improve the surface finish [26], [27].

Another general drawback with metal AM that is of great importance is that the parts can contain defects, e.g., pores, lack-of-fusion defects, cracks, and inclusions, which are difficult to monitor and quantify during manufacturing [20]. Thus, in situ process control is challenging in many of the metal AM techniques, and is nowadays extensively investigated for all types of metal AM techniques because the material properties are highly dependent on the build history [10], [17], [28], [29].

2.2 Classification of metal AM technologies

Generally, metal AM techniques can be categorized into three main groups based on the type of material input: i) solid-based systems, ii) liquid-based systems, and c) powder-based systems [3]. The first two groups are out of the scope of this study and only powder-based systems will be discussed. Figure 2-1 briefly illustrates a schematic of the different AM techniques.

![Figure 2-1: Process overview of metal-based AM technologies, the highlighted color shows the EBM process, modified from [1].](image-url)
2.3 Powder bed fusion technologies

As the name implies, powder is the feedstock, which is selectively melted by using either a laser or an electron beam as the heat source. Additionally, by melting a layer, the underlying layers are re-melted to achieve sufficient adherence to the newly added layer and to the rest of the part [4], [30]. One of the powder bed fusion (PBF) techniques is the electron beam melting (EBM) process, which uses a high-power scanning electron beam and offers a higher build rate than the other PBF techniques [5].

2.4 EBM

The EBM process was commercialized by a Swedish company called Arcam AB in 1997 [3], [5]. Generally, in this process, powder as the feedstock is sintered and melted according to a 3D CAD file by using a highly intense electron beam energy source. Based on the literature, it has been found that this process can be used for multiple types of conductive metallic materials. The most extensive work has been performed for titanium alloys (mainly Ti-6Al-4V) [6], [31], [32], however, other materials such as nickel-based superalloys (Alloy 718, Alloy 625, Rene 142) [5], [6], [33]–[37], copper [38], CoCr alloys [33], and H13 steel [39] have also been investigated for this process.

2.4.1 Process description

As shown in Figure 2-2, on top of the main chamber of the EBM equipment, three different lenses are located, including magnetic lenses to correct the astigmatism and to produce a circular beam with a Gaussian energy distribution, a focal lens to focus the beam onto a spot of appropriate size, as well as focus/defocus the electron beam during the preheating/sintering/melting step, and a deflection lens to control the beam scan across the build plate.

Inside the build chamber, two powder hoppers containing the powder stock material are located. Below the powder hoppers, there is a raking system that fetches the powder from the hoppers and spreads it over the build plate. The build plate, which is placed under the heat shield, is lowered along the Z-axis during the building process.

During the EBM process, the build chamber and electron gun column are kept under vacuum in order to

- Avoid the interaction of electrons with gaseous atoms
- Prevent the chemical reactions of the elements present in sensitive materials, like titanium, with atmospheric gases, such as oxygen
- Act as an insulator to maintain an elevated process temperature

During the EBM process, the entire powder bed must be heated and maintained at relatively high temperatures, depending on the material (over 1000 °C for Alloy 718 [5], [37]). The heating is accomplished by rapidly scanning a defocused beam over the top powder surface. The elevated temperature will help in reducing the residual stress in the as-built parts. Moreover, when an improper beam setting is applied, smoking phenomenon can occur due to the electrostatic charges in the powder, which causes uncontrolled repulsion/blowing of the powders. This may lead to process instabilities [3], [5], [40]. The smoking phenomenon occurs when the negative repulsive forces between the powder particles are too high in magnitude, higher than the gravitational and friction forces that hold them in place. Thus, the powder particles produce a powder cloud inside the chamber. Another challenge with the negative charge buildup in the powder bed is the risk of beam deflection, which results in a lower beam spot accuracy.

![Figure 2-2: Schematic of the EBM process, including the basic components with the labels.](image)

A typical EBM process follows the sequence below.

1. **Vacuum pumping**: providing vacuum in the build chamber up to approximately \(10^{-5}\) mbar. In addition, a small amount of inert helium at a partial pressure of \(\sim 2 \times 10^{-3}\) mbar is also injected into the vacuum
chamber to prevent electrical powder charging and to provide thermal stability to the process [5], [41], [42].

2- **Build plate heating**: heating of the stainless steel build plate. The heating temperature is highly dependent on the material type. The temperature for Ni-Fe-based superalloys, which is the main material investigated in this research, is ~1020 °C.

3- **Lowering the build plate**: the build plate usually moves down by a distance of between 50 and 100 µm that is dependent on the layer thickness proposed by the operator.

4- **Raking**: the rake spreads a layer of powder over the build plate.

5- **Preheating/Sintering**: heating the distributed powder layer on the build plate up to the desired sintering temperature. This temperature is ~80% of the melting temperature, and a defocused beam scans over the build plate to slightly sinter the powder. The sintering step helps to enhance the conductivity between the individual powder particles and prevent the movement of powder during melting with a focused beam [3], [5], [43].

6- **Support/wafer melting (optional)**: this step can be carried out in any order relative to the preheating and post heating steps. Supports are used for mainly two purposes, i) mechanical integrity and ii) for thermal support. The mechanical support prevents overhangs from the deformation induced by gravity or growth stresses. The thermal supports conduct the applied energy from the melt surface to the build plate.

7- **Contour melting**: as shown in Figure 2-3, the border/frame of a part is named contour and is melted by using a multisport strategy to yield the desired surfaces of the final part. The multibeam strategy means that the beam scans fast enough to simultaneously maintain several melt pools. The advantages of contour melting are optimized surface finish and precision [44]. In fact, typically two contours (inner and outer) are run as a frame for the bulk region; see Figure 2-3. However, the number of inner contours can be more. There is an overlap between the inner and outer contours and also between the inner contour and the bulk region. The optimization of the contour parameters, e.g., beam current, spot melt order, overlaps, and the number of contours, is important for determining the surface finish of the as-built parts.

8- **Hatch/bulk melting**: hatch/bulk is the inner region of the part that is scanned. Typically, the hatch scan strategy is called “snaking,” which involves linear and continuous melting where the beam moves back and forth; see Figure 2-3.
9- **Post-heating:** this step is normally performed after melting a single layer to maintain a constant build temperature.

10- **Repetition until the final finished part:** the steps 3 to 9 are repeated several times in the newly added layers to complete the whole part [36].

11- **Cooling:** as the building job is finished, the chamber is cooled from the elevated temperature to 100 °C minimum.

12- **Powder recovery system (PRS):** the sintered particles are removed from the final part by grit/compressed air blasting. Typically, the powder blasted off from the part is recycled into the process once again.

### 2.4.2 Position and geometry-related parameters

The position-related parameters are linked to the type of stacking or the orientations of the parts on the build plate. These parameters consist of the distance between the parts on the build plate, the height of the part from the build plate, the location of the part on the build plate (the exterior or interior locations of the build plate), and the angle of the part on the build plate. There is a lack of literature in this research topic, however each of those parameters can have a significant effect on the thermal history of a part, which is the main factor influencing microstructure evolution.

- **Height from the build plate:** by elevating the parts from the build plate, the powder below the parts affects the thermal conductivity of the parts to the build plate, which subsequently may affect the microstructural characteristics. For instance, by increasing the height from the build plate, the cooling rate is reduced, which is due to the accumulation of powder with a low thermal conductivity beneath the parts [23].

- **Location on the build plate:** another position-related parameter that could affect the microstructural features is the location of the part on the build plate. The hypothesis that heat is distributed differently depending
on the location of the build plate is considered. In this hypothesis, it is assumed that the interior areas of the build plate would be the warmest and that the relative temperature would decrease outward towards the exterior areas of the plate. Thus, it is expected that the exterior parts would have finer microstructures and better mechanical properties compared to the parts in the interior areas [24].

- **Distance between the parts on the build plate**: the distance between individual parts can be used to define two different terms “open design” and “close design.” This parameter has not been investigated yet and is one of the objectives of the present work. Since in a close design, the parts are fairly close to each other and the distance between them is 2 mm minimum (the distance recommended by the EBM manufacturer), greater heat accumulation is expected. Thus, in a close design, the higher heat accumulation can lead to a lower cooling rate. However, in an open design, with a large distance between the parts, heat dissipation is assumed to be faster and accordingly the cooling rate is higher.

- **Angle of the part on the build plate**: the angle/orientation of a part on the build plate indicates whether the part is built horizontally, vertically, or at a certain angle to the build plate. Since the number of successive thermal cycles can influence the microstructure at different heights of a part, the proper orientation must be taken into account [24].

- **Part size**: the thickness of the parts can have a significant effect on the microstructures (e.g., texture and grain structure) of EBM-manufactured materials [45]. It is expected that larger parts would have higher heat accumulations due to longer scan lines, which result in lower cooling rates and coarser microstructures [23].

### 2.4.3 Machine-related parameters

There are more than a hundred parameters in the EBM process that can be directly/indirectly altered [44]. It suggests that some parameters are changed according to the setting and some are changed automatically as part of the different process settings. Some of the main parameters are beam scanning speed, layer thickness, beam current, line offset, focus offset, scanning strategy, and powder type [40]. The brief description of certain important parameters, based on the literature related to the scope of this research, is as follows:

- **Beam scanning speed**: how fast a beam moves is mainly controlled by deflection coils. According to Eq. 1 [46], the beam scanning speed has an inverse effect on the energy input per volume of a material, thus, it can have a significant effect on the microstructural characteristics. The levels of the scanning speed at the contour and the hatch regions are different.
owing to the difference in the scanning strategy between those two regions. In addition, the beam scanning speed slightly changes along the scan line (X- and Y- axes) and build direction (Z-axis) to maintain energy balance during the EBM process [5], [37], [40], [44], [47]. In fact, the energy balance is ensured by a complex set of process parameters (of which the scanning speed is one) that allow the operator to control the behavior of the beam, which affects the melt pool locally and across the powder bed. Typically, the maximum scanning speed of an EBM A2X machine can reach 8000 mm/s [5].

\[
\text{Energy input (J/mm}^3) = \frac{\text{Power = voltage (kV) \times current (mA)}}{\text{scanning speed (mm/s) \times line offset (\mu m) \times layer thickness (\mu m)}}
\]  

(1)

- **Beam current**: this parameter has a direct correlation with the energy input (see Eq. 1), and can reach a maximum of 50 mA. Indeed, the relationship between beam current and scanning speed is highly important in EBM owing to its great effect on the elimination of process-induced defects and the determination of the grain structure [37], [48], [48].

- **Focus offset**: the focus offset is the value of the current used by the focusing coils located in the electron gun column to concentrate the beam, and significantly affects the geometry of the melted tracks [5], [40]. The value of the focus offset is given in milliamperes, and it has an impact on the beam spot size; see Figure 2-4. Changes in the focus offset affect the beam spot size, which is affected by the applied beam current. Therefore, increasing the focus offset does not always imply that the spot size is increased. As shown in Figure 2-4, the beam spot size is affected by the position of the focal point which can be above the build plate (a), on the build plate (b), or below the build plate (c), and is mainly affected by the applied focus offset and the applied beam current.

**Figure 2-4**: Schematic illustration of the focus offset: a) focal point above the build plate, b) focal point on the build plate, and c) focal point below the build plate.
- **Line offset**: it is the distance between two adjacent beam scanning passes; see Figure 2-5a. One approach to reducing the amount of pores is to optimize this parameter. By decreasing the value of line offset to some extent, the overlap between two passes that are adjacent to each other can be increased, along with the heat input, therefore, there is less risk of the presence of unmelted powders. On the contrary, for a higher line offset, the risk of unmelted powders being present at the bottom of the overlapping area can be high, see Figure 2-5(b-c) [30], [49].

![Figure 2-5: Schematic illustration of a) the line offset, b) the overlapping area with a lower line offset, and c) the overlapping area with a higher line offset.](image)

- **Scanning strategy**: the electron beam path employed during the process is termed “scanning strategy,” which can be used as a tool for tailoring the microstructure [50]. The scanning strategy is typically different in the hatch and contour regions. Commonly, spot melting is used in the contour region; on the other hand, different types of scan strategies can be applied in the hatch region, which include unidirectional, bidirectional/snaking, and spot melting; see Figure 2-6. In the case of unidirectional or bidirectional/snaking strategy, the rotation of the hatch direction is performed at a predefined angle between each layer. In addition, for a specific scanning strategy, the beam scanning speed and current must be optimized [5], [48].

![Figure 2-6: The different scanning strategies used in the EBM process in the XY plane: a) unidirectional, b) bidirectional/snaking, and c) spot melting.](image)
3 Alloy 718

The development of Alloy 718 as a Ni-Fe-based superalloy in 1959 was a great achievement owing to its unique properties, the most important of which is the maintenance of its strength/mechanical properties at elevated temperatures (up to 650 °C) under high stresses. It also has good weldability, is relatively insensitive to strain age cracking, and exhibits good resistance to oxidation and corrosion [51], [52]. Based on these properties that reflect the advantage of Alloy 718 compared to other materials, it has been a material in high demand for many high-temperature applications such as gas turbines and rocket engine components, as well as for low-temperature applications like reactor internals for nuclear power plants [52]–[55].

3.1 Strengthening mechanisms

There are different strengthening mechanisms in Alloy 718, three of which include the following: I) solid solution strengthening, II) precipitation strengthening, and III) grain size refinement, which are described here [56].

I) Solid solution strengthening: One of the primary strengthening mechanisms in metallic materials is where an element (solute) is dissolved into the lattice of a host metal (here, the γ matrix phase) to form a solid solution strengthened alloy, whose strength is greater than that of the host metal. The main strengthening mechanism is attributable to the difference in atomic radius, which causes distortions in the face centered cubic (FCC) γ lattice structure. These distortions hinder dislocation movements in the matrix, thereby strengthening the alloy [28], [51], [56].

II) Precipitation strengthening: The main strengthening mechanism in Alloy 718 is the precipitation of γ' and γ'' phases. In order to obtain these precipitates, certain elements with low solubilities in the γ matrix, such as niobium, titanium, and aluminum, must be added. Principally, the strengthening effect is obtained by the coherency strain developed between the strengthening precipitates and the matrix phase. In addition, antiphase boundary (APB) energy is developed that resists dislocation movement between the ordered strengthening phases in the matrix. Furthermore, by increasing the size of the precipitates to a certain level, the precipitation strengthening mechanism becomes more dominant. The precipitates, which can be coherent or semicoherent intermetallic compounds such as γ'-Ni₃(Ti, Al) or γ''-Ni₃Nb, respectively, prohibit the movement of
dislocations. The movement of a dislocation in the matrix containing the precipitates can only take place by cutting through or bypassing the particles [56].

In addition to the main strengthening precipitates in Alloy 718, elements such as niobium, titanium, and molybdenum, which have limited solubility, are able to form carbides such as NbC and TiC to provide high-temperature strengthening. In general, carbides are preferentially precipitated in the intergranular regions and subsequently in the intragranular regions. By designing the distribution of carbides in the microstructure, the mechanical properties of a material can be enhanced. The carbides formed at the boundaries prevent grain boundary slip and transfer the deformations to the interior grain regions, where creep diffusion is slower [57], [58].

III) *Grain size refinement:* The reduction in grain sizes inside a material leads to the formation of more grain boundaries, which prevents the movement of dislocations, thus delaying failure and improving the strength of a material. A fine-grained microstructure of Alloy 718 is often desirable. It is well recognized that a fine-grained microstructure displays good tensile properties and toughness, as well as improved high-cycle fatigue performance [59].

### 3.2 Common phases in EBM-manufactured Alloy 718

The microstructure of Alloy 718 is highly complex, with a large number of dispersed intermetallics and other phases in the γ matrix phase that modify the alloy properties through their composition, morphology, and location. A summary of the common phases observed in EBM-manufactured Alloy 718 depending on the thermodynamics of the process is given below; see Table 3-1.

**Table 3-1: Common phases observed in EBM-manufactured Alloy 718. Table based on [5], [51], [56]**

<table>
<thead>
<tr>
<th>Phase</th>
<th>Crystal structure</th>
<th>Formula</th>
<th>Solvus temperature (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>γ</td>
<td>FCC</td>
<td>-</td>
<td>1260-1364</td>
</tr>
<tr>
<td>γ'</td>
<td>FCC</td>
<td>Ni₃(Al, Ti)</td>
<td>850-910</td>
</tr>
<tr>
<td>γ&quot;</td>
<td>BCT</td>
<td>Ni₃Nb</td>
<td>910-940</td>
</tr>
<tr>
<td>δ</td>
<td>Orthorhombic</td>
<td>Ni₃Nb</td>
<td>1020</td>
</tr>
<tr>
<td>MC</td>
<td>Cubic</td>
<td>TiC, NbC</td>
<td>1260-1305</td>
</tr>
<tr>
<td>Laves</td>
<td>Hexagonal</td>
<td>Fe₂Nb, Fe₂Ti, Fe₂Mo, Fe₂Ta, Fe₂Ti</td>
<td>1163</td>
</tr>
</tbody>
</table>

- **γ phase:** an austenitic solid solution phase that can maintain its structure up to its melting temperature. This phase has a high capacity for solid solution strengthening by elements such as iron, chromium, and molybdenum. These
elements have 1 to 13\% larger atomic diameters than nickel and their strengthening effect is related to the atomic oversize [56].

- **γ’ phase**: an intermetallic strengthening phase that is coherent with the γ matrix. Typically, the morphology and size of γ’ (from spherical to cuboidal) varies with temperature and exposure time. Apart from the thermodynamic effect, the chemical variations like molybdenum content or aluminum/titanium ratio can change the morphology of γ’. In general, by increasing the lattice mismatch between γ and γ’, the morphology changes in the following order: spherical, globular, blocky, and cuboidal. The aluminum/titanium ratio should be higher than 1.5 for promoting stable γ’ precipitation [51], [56], [58].

- **γ” phase**: the main intermetallic strengthening phase, which is semicoherent with the γ matrix. The γ” phase often forms together with the γ’ phase in Alloy 718, and its precipitation is strongly dependent on the local niobium concentration. The γ” precipitates are usually disk-shaped. In Alloy 718, the lattice mismatch between γ” and γ (2.86\%) is more than that between γ’ and γ (<0.5\%) [60], [61]. Further, the volume fraction of γ” (γ”/γ’ is around 3.0 [61]) is higher. Therefore, γ” is the principal strengthening phase, and γ’ contributes to the strength to some extent. One of the main drawbacks of the γ” phase in comparison to γ’ is its transformation to the δ phase at very high temperatures for very long exposure times [58].

- **δ phase**: the formation of different morphologies of the δ phase is highly dependent on the time-temperatures profile of the material [56]. The δ phase is incoherent with the γ matrix, and, since the precipitation of the δ phase would occur at the expense of niobium, which is associated with the loss of γ” and therefore strength, it is a potential reason for the reduction in strength. At low temperatures (650–700 °C), cellular morphology of the δ phase can be observed at the grain boundaries [62]. By increasing the temperature to 750–800 °C, in addition to the cellular precipitation of the δ phase, the transformation γ”→ δ + γ’ occurs, which leads to the formation of γ”-free regions surrounding the particles of the δ phase. The main reason for this transformation is the growth of the δ phase, which results in significant amounts of dislocations that may in turn produce more stacking faults in γ”, and therefore, may promote δ phase formation [62], [63]. An additional reason is the metastable nature of the γ” phase in Alloy 718, which can convert to the stable δ phase with a needle or plate-like morphology under thermal exposure [56]. Through a continuation of the aging process, intragranular δ-needles and/or δ-needles formed from the γ” transformation may grow from the grain boundaries into the matrix. At 900 °C, the γ” phase is completely replaced by the δ phase. At higher temperatures,
around 960 °C, there is direct precipitation of the δ phase from the supersaturated matrix. The highest growth rate of the δ phase has been observed in 960–980 °C for short-time aging of a material or a solutionized material. It is pertinent to mention that the δ phase has some benefits on the microstructure and mechanical properties in certain situations. The δ phase formed at the grain boundaries can efficiently limit grain growth during the service life, which is an important aspect of current Alloy 718 production [64].

- **Laves phase**: depending on the thermal history of the material during the EBM process, Laves phases can form. The formation of a Laves phase was reported in relatively bigger parts mainly at the top layers of the material [36]. In fact, segregation of elements like niobium and molybdenum during solidification creates interdendritic regions that are niobium-rich, which if not properly homogenized can form a Laves phase [5]. As a result of niobium segregation, the matrix is depleted of this element for the principal strengthening phase γ”, therefore, a reduction in the strength results. The Laves phase, as a TCP phase, is an intermetallic phase that is brittle in nature and detrimental to the mechanical properties. The morphology of the Laves phase is typically in the form of irregular-shaped globules, which are often elongated, or platelets, after extended high-temperature exposure [58], [65], [66].

- **MC-type carbides**: the most common carbide that forms in Alloy 718 during the EBM process is the MC-type carbide, with a predominantly globular/blocky morphology. Here, “M” is mainly niobium, owing to its high content in Alloy 718, however, it can also be titanium or tantalum. As already discussed in the strengthening mechanisms, the MC-type carbides can be beneficial or detrimental to the mechanical properties, depending on the size and the distribution regions (intragranular or intergranular regions). The intragranular MC-type carbides have a lower strengthening effect (comparing with the strengthening effect of the γ” phase) that is produced by hindering the movement of dislocations in the matrix of Alloy 718 [56], [67].

### 3.3 Microstructure evolution during EBM process

Currently, several studies have been performed on the EBM of Alloy 718 [5], [34], [36], [37], [48], [68]–[70]. The microstructure of Alloy 718 manufactured by EBM is a function of the thermal history of the process and the successive thermal cycling effects, which produce inhomogeneities in the microstructure. A microstructure gradient along the build direction during the EBM of Alloy 718 was reported from several studies [36], [71], [72]. These reports state that there is a height dependency on the thermal exposure experienced by different regions of a part [71]. Typically, in the uppermost layers of a part, a dendritic structure is
observed, and the $\gamma''$ phase is precipitated within the dendrite cores, while MC-type carbides and Laves phases are precipitated in the interdendritic areas. The cooling rate was estimated to be $\sim$1000 °C/s, based on the primary dendrite arm spacing in the top layers. Lower, in the middle of the part, an intermediate transition zone is found which is characterized by a diffuse dendritic structure that is largely lacking in secondary dendrite arms and the interdendritic Laves phase. In the bottom layers, the lack of dendritic segregation is reported [36].

Strondl et al. [34] reported that the as-built microstructure of Alloy 718 in a standard EBM process setting consists of elongated grains along the building direction, with $<100>$ crystal orientation. Most of the grains were several millimeters in length and up to 1 mm in diameter, although smaller grains also existed. The microstructure contained strings of MC-type carbides, as well as fine $\gamma''$ precipitates (5–10 nm), along with some coarse $\gamma''$ precipitates (50–100 nm). A few $\gamma'$ precipitates 2–5 nm in size were also observed, however, no Laves phase was found [69]. Several efforts have shown that by altering the machine-related parameters and consequently changing the thermal history, a higher density, a tailored grain structure, and crystallographic texture can be achieved [37], [48], [70], [73]–[76]. The thermal history is totally dependent on three groups of parameters, including a) position-related parameters, b) geometry-related parameters, and c) machine-related parameters; see sections 2.4.2 and 2.4.3. The effects of the position- and geometry-related parameters on the microstructure of EBM-manufactured Alloy 718 have not yet been completely investigated. The influences of some machine-related parameters on the microstructure evolution of Alloy 718 in EBM have been reported and are described as follows.

Screening the machine-related parameters is vital to achieving a fully dense desirable microstructure, depending on the final application of the parts. By tuning the machine-related parameters, the microstructural features such as defects, the type of grains (columnar or equiaxed), and phases can be controlled. Some of these features are discussed below.

**Defects:** One goal of investigating the influence of machine-related parameters is to limit the formation of defects in the material. The presence of pores is detrimental to the mechanical properties, especially for crack initiation and propagation. Therefore, either tuning the machine-related parameters or improving the feedstock quality can decrease the level of pores during the process [5], [77]. There are typically three types of defects in an as-built material that can negatively affect the mechanical properties:

- **Round-shaped pores:** the main driving force for this type of pores is the raw material (powder). The origin of the pores in powder particles is the gas trapped inside the particles during the powder production process, and
these pores can transfer directly to the as-built parts. In the EBM process, owing to the rapid solidification of the material, the trapped gas within the gas-atomized powders does not have sufficient time to escape from the melt pool, even though the vacuum level is high. Thus, these pores are typically round-shaped; see Figure 3-1a [5].

Figure 3-1: Scanning electron microscopy (SEM) and light optical microscopy (LOM) images of defects.

- **Lack-of-fusion defects**: the major concern in the EBM of Alloy 718 is to avoid lack-of-fusion defects, which are dominated by the machine-related parameters. It implies that the machine-related parameters must be properly set/optimized to prevent mechanisms that can generate these defects. The size of these defects can range from a few micrometers to a few millimeters and the morphology is mainly irregular. A potential reason for the formation of these pores, among others, is the lack of sufficient applied energy input to completely melt the particles, as a result of which some unmelted powder particles can be seen within these pores. Alternatively, a high amount of applied energy density can cause the spattering of the melted material away from the melt pool, leading to formation of the lack-of-fusion defects; see Figure 3-1b [5], [29], [40].

- **Shrinkage pores**: it is another common type of defect, and is a collection of round-shaped pores, which form a band at certain regions, mainly the interdendritic areas, along the build direction, which is typically the last area to solidify [69]. The dominant mechanism that induces such shrinkage pores is the residual thermal stresses generated during the solidification. Generally, during the solidification of the top molten layer,
the material shrinks and its volume reduces; this shrinkage is hindered by the underlying layers that have been previously processed; see Figure 3-1c [5].

Grain structure: Körner et al. [70] reported that the grain structure of Alloy 718 manufactured by EBM can be influenced by the scanning strategy during melting. This result showed that local tailoring of the grain structure of a part was possible by varying the scanning strategy. Helmer et al. [48] also confirmed that the grain structure of Alloy 718 can be changed during EBM by altering the scanning strategy and thus the local solidification conditions. The microstructural adaption was achieved by changing the orientation of the thermal gradient during solidification. The columnar grain structures grew if the thermal gradient remained in alignment with the building direction. Equiaxed grains resulted from the sharp change in the orientation from one layer to the other. Under these circumstances, succeeding layers did not share a preferred growth direction. Thus, the columnar grain growth was hampered and new nuclei began to grow. This finding makes it possible to alter the solidification grain structure locally within a single part [37], [48]. In their other research on the influence of machine-related parameters (beam power, scanning speed, and beam spot size) on pore content and grain structure, it was found that porosity is a result of insufficient melting [37]. A more focused beam was found to increase the formation of new grains and prevent the development of the columnar grain structure, which is desired for favorable high-temperature mechanical properties such as creep resistance [37], [70]. The stronger motion of the melt resulted from the more focused beam led to dendrite fragmentation and enhanced grain nucleation [48].
4 Material and experimental setup

The goal of this research is to gain knowledge of the influences of position-, geometry-, and machine-related parameters on the microstructural characteristics of parts, more specifically on the microstructural evolution of Alloy 718 during the electron beam melting (EBM) process. In order to realize this goal, cubic and fine-scale sized samples have been manufactured by using EBM. All the techniques used to investigate the powder and the characterization approaches are described in this chapter.

4.1 Feedstock powder

The powder utilized in the EBM process was plasma atomized (PA) Alloy 718 powder supplied by Arcam AB (Sweden). The chemical composition provided by Arcam AB is given in Table 4-1. The powder used during this research was always a mixture of virgin and re-used powders.

<table>
<thead>
<tr>
<th>Element</th>
<th>Ni</th>
<th>Co</th>
<th>Cr</th>
<th>Mo</th>
<th>Ti</th>
<th>Mn</th>
<th>Nb</th>
<th>P</th>
<th>Ta</th>
<th>Al</th>
<th>Fe</th>
<th>Si</th>
<th>S</th>
<th>C</th>
</tr>
</thead>
<tbody>
<tr>
<td>wt%</td>
<td>54.11</td>
<td>0.04</td>
<td>19</td>
<td>2.99</td>
<td>1.02</td>
<td>0.12</td>
<td>4.97</td>
<td>0.004</td>
<td>&lt;0.01</td>
<td>0.52</td>
<td>Bal.</td>
<td>0.06</td>
<td>&lt;0.001</td>
<td>0.03</td>
</tr>
</tbody>
</table>

The virgin powder had a spherical morphology with some small satellite particles attached onto the surface. However, after recycling the powder, agglomerated particles were present; see Figure 4-1. The content of internal pores in the powder was low and only small internal pores were observed within the cross section of the powder particles. The size distribution of the virgin powder particles was approximately in the range 45–105 μm (diameter). It was observed that by recycling the powder many times (e.g., 20 times), the size distribution measured through an optical light scanning and imaging analysis technique widened (~40%), which was attributed to the presence of partially sintered powder particles. Scanning electron microscopy (SEM) images of the cross section of the mixed powder particles show some particles containing a fine secondary phase; see Figure 4-1. Energy dispersive spectroscopy (EDS) revealed that the secondary
phase was rich in niobium, as shown in Figure 4-1, and was most likely a mixture of the $\delta$ phase and niobium carbides.

**Figure 4-1**: SEM (BSE mode) images of the morphology of powder particles and the cross section of the particles: a) virgin powder, b) re-used powder, c) cross section of the virgin powder, d) cross section of the re-used powder, and e) size distribution of the re-used powder.

<table>
<thead>
<tr>
<th>Elements (wt%)</th>
<th>Ni</th>
<th>Fe</th>
<th>Mo</th>
<th>Al</th>
<th>Cr</th>
<th>Nb</th>
<th>Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td>Matrix ($\gamma$-phase (1))</td>
<td>Bal.</td>
<td>19.8</td>
<td>2.51</td>
<td>1.04</td>
<td>17.50</td>
<td>5.10</td>
<td>1.30</td>
</tr>
<tr>
<td>$\delta$-phase (2)</td>
<td>Bal.</td>
<td>5.40</td>
<td>2.32</td>
<td>0.80</td>
<td>3.50</td>
<td>21.40</td>
<td>3.00</td>
</tr>
</tbody>
</table>

![Particle size distribution](image)
MATERIAL AND EXPERIMENTAL SETUP

4.2 Sample manufacturing setup

All manufacturing in this work was performed by using an Arcam A2X machine. The key machine-related parameters used are briefly presented in Table 4-2. More details of the sample design in each appended paper are presented as follows.

Table 4-2: Brief illustration of the key machine-related parameters used for the EBM-based manufacturing of samples in different papers

<table>
<thead>
<tr>
<th>Machine-related parameters</th>
<th>Paper A</th>
<th>Paper B</th>
<th>Paper C</th>
<th>Paper D</th>
</tr>
</thead>
<tbody>
<tr>
<td>Powder Re-used</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Speed function</td>
<td>On-mode</td>
<td>Off-mode</td>
<td>Off-mode</td>
<td>Off-mode</td>
</tr>
<tr>
<td>Layer thickness (µm)</td>
<td>75</td>
<td>75</td>
<td>75</td>
<td>75</td>
</tr>
<tr>
<td>Beam current (mA)</td>
<td>7</td>
<td>7</td>
<td>7</td>
<td>7-15</td>
</tr>
<tr>
<td>Focus offset (mA)</td>
<td>15</td>
<td>15</td>
<td>15</td>
<td>0-10</td>
</tr>
<tr>
<td>Line offset (µm)</td>
<td>125</td>
<td>125</td>
<td>125</td>
<td></td>
</tr>
<tr>
<td>Scanning speed (mm/s)</td>
<td>1250</td>
<td>1250</td>
<td>1250</td>
<td>300-1000</td>
</tr>
<tr>
<td>Voltage (kV)</td>
<td>60</td>
<td>60</td>
<td>60</td>
<td>60</td>
</tr>
<tr>
<td>Scan strategy</td>
<td>Rotation 60°</td>
<td>Uni-direction</td>
<td>Uni-direction</td>
<td>Uni-direction</td>
</tr>
</tbody>
</table>

- In the first study, cubic samples in different groups (A, B, C, D, E, and F) were built on a build plate at 45° angle; see Figure 4-2c. The machine-related parameters recommended by Arcam were used to investigate the effect of position-related parameters, such as the distance between samples, the height from the build plate, and the location of sample on the build plate; see Figure 4-2.
In the second study, different geometry-related parameters were used in order to understand the microstructural evolution during the EBM process. In this study, both track(T)-by-track and layer(L)-by-layer (single wall) samples were manufactured by using the machine-related parameters for the hatch region of a cubic sample of size $20 \times 20 \times 20$ mm$^3$. For this study, all the samples were produced on an additional substrate, which is called “stand” and is made of the same material as the samples (Alloy 718), by using the Arcam recommended parameters, see Figure 4-3.

The track-by-track samples started from a single track and comprised up to 10 tracks that were next to each other, e.g., Txl1 (x: 1, 2, 3, …, 10). The layer-by-layer samples started from 1 and consisted of up to 50 layers (T1L1, T1L2, … etc.). The length of the tracks was kept constant at 20 mm. Moreover, in order to determine the effect of wall thickness, samples with 5 tracks and different numbers of layers were produced as well.
The aim of the last study was to evaluate the effect of machine-related parameters on the geometrical (track height \(d_1\), melt pool depth \(d_2\), melt pool width \(w\), and average of contact angle \(\Theta = (\Theta_1 + \Theta_2)/2\); see Figure 4-4) and microstructural features of the single-track samples. In the first step, a design of experiments (DoE) approach was implemented over a specific range for certain machine-related parameters; see Table 4-3. In the next step, another DoE was implemented in the appropriate range obtained from the results of the first DoE.

**DoE (1):** A full factorial design scheme was used for the three main machine-related parameters: beam current, scanning speed, and focus offset. Two levels for each parameter were selected based on the literature and initial tests were performed to determine the endpoints. A total of 11 parameter sets were used, as summarized in Table 4-3.

**Table 4-3: The machine-related parameter settings used for the full factorial two-level DoE**

<table>
<thead>
<tr>
<th>Trial #</th>
<th>Scanning speed (mm/s)</th>
<th>Beam current (mA)</th>
<th>Focus offset (mA)</th>
<th>Linear energy input = Power/Scanning speed (J/mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>300</td>
<td>2.0</td>
<td>0.0</td>
<td>0.40</td>
</tr>
<tr>
<td>2</td>
<td>300</td>
<td>2.0</td>
<td>5.0</td>
<td>0.40</td>
</tr>
<tr>
<td>3</td>
<td>300</td>
<td>5.0</td>
<td>0.0</td>
<td>1.00</td>
</tr>
<tr>
<td>4</td>
<td>300</td>
<td>5.0</td>
<td>5.0</td>
<td>1.00</td>
</tr>
<tr>
<td>5</td>
<td>650</td>
<td>3.5</td>
<td>2.5</td>
<td>0.32</td>
</tr>
<tr>
<td>6</td>
<td>650</td>
<td>3.5</td>
<td>2.5</td>
<td>0.32</td>
</tr>
<tr>
<td>7</td>
<td>650</td>
<td>3.5</td>
<td>2.5</td>
<td>0.32</td>
</tr>
<tr>
<td>8</td>
<td>1000</td>
<td>2.0</td>
<td>0.0</td>
<td>0.12</td>
</tr>
<tr>
<td>9</td>
<td>1000</td>
<td>2.0</td>
<td>5.0</td>
<td>0.12</td>
</tr>
<tr>
<td>10</td>
<td>1000</td>
<td>5.0</td>
<td>0.0</td>
<td>0.30</td>
</tr>
<tr>
<td>11</td>
<td>1000</td>
<td>5.0</td>
<td>5.0</td>
<td>0.30</td>
</tr>
</tbody>
</table>
The second design was developed in order to determine the appropriate process window for the three parameters. In this design, a full factorial screening scheme across three levels was used. Each machine-related parameter set was repeated four times and for each sample, two cross sections were evaluated in order to increase the statistical significance of the experiment; see Figure 4-4. The parameter settings of the design are shown in Table 4-4.

Table 4-4: The machine-related parameter settings used for the full factorial three-level DoE screening

<table>
<thead>
<tr>
<th>Trial #</th>
<th>Scanning Speed (mm/s)</th>
<th>Beam current (mA)</th>
<th>Focus offset (mA)</th>
<th>Linear energy input: Power/Scanning speed (J/mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>300</td>
<td>15</td>
<td>0</td>
<td>3.00</td>
</tr>
<tr>
<td>2</td>
<td>300</td>
<td>15</td>
<td>5</td>
<td>3.00</td>
</tr>
<tr>
<td>3</td>
<td>300</td>
<td>15</td>
<td>10</td>
<td>3.00</td>
</tr>
<tr>
<td>4</td>
<td>300</td>
<td>11</td>
<td>0</td>
<td>2.20</td>
</tr>
<tr>
<td>5</td>
<td>300</td>
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4.3 Metallographic preparation

For characterization by LOM and SEM, all the cubic samples were cut from the middle by using an abrasive precision cutter. For the same characterizations, the fine-scale samples of track-by-track, layer-by-layer/single walls, and single tracks from the DoE were cut at two different points that are approximately 7 mm from the start and end points; see Figure 4-4a. All the samples were then hot mounted in a non-conductive Bakelite holder. An example of a mounted sample is shown in Figure 4-5. The mounted samples were gradually ground using SiC papers up to 1200 grit. The ground samples were then polished down using a 0.05 μm SiC suspension. Moreover, in order to reveal the microstructural features such as cellular-dendrites, the grain structure, and various phases, the polished samples were etched by using oxalic acid diluted with water in 1:10 ratio. The electro-etching was carried out by using 3–4 V of potential for 5–10 s.

Figure 4-4: a) Light optical microscopy (LOM) image of the top of sample # 7, b) a schematic of the cross section of a single track with the geometrical features.

Figure 4-5: Examples of the prepared samples: a) cross section of the cubic samples, b) cross section of the layer-by-layer samples.
4.4 Characterization techniques and measurement

4.4.1 LOM

LOM (OLYMPUS-BX60M, Tokyo, Japan) was utilized to observe the type of defects and quantitatively measure them, as well as to observe the characteristics of the melt pool geometry, e.g., the melt pool depth, melt pool width, contact angle, track height, and type of defects.

4.4.2 SEM

A Hitachi (TM3000, Tokyo, Japan) SEM equipped with an EDS system was used to characterize the different phase constituents in terms of chemical composition. The SEM was typically operated at 15–20 kV in the backscatter electron mode (BSE). By means of this system, the microstructural features like the primary dendrite arm spacing (PDAS) and the different phases such as the niobium-rich phase NbC (MC-type carbide) were determined and identified. In order to further investigate through high-accuracy images, other types of SEMs (ZEISS EVO 50, Cambridge, United Kingdom, and GAIA3-Tescan, Cambridge, UK) were also used to examine the solidification mode and the corresponding phase constituents such as the δ phase (in case of its presence). The EDS analysis was used to detect the different phases, to determine the chemical composition, and for elemental mapping.

4.4.3 Electron backscattered diffraction

A Philips FIB-SEM (TESCAN-GAIA3, Cambridge, United Kingdom) coupled with an electron backscattered diffraction (EBSD) detector was used for the EBSD analysis in order to investigate the grain orientation.

4.4.4 Hardness test

Hardness measurements were carried out by using a Shimadzu HMV-2 micro Vickers hardness tester. The load was set to 500 g and the dwell time 15 s.

4.4.5 Porosity measurement

For each cubic sample, 16 LOM images were taken in the normal reference plane (parallel to the build direction) in order to examine the defects such as porosity and lack-of-fusion defects. The porosity measurement was performed by using the ASTM-E562-08 standard point counting technique [78] on optical
micrographs with a horizontal field width of 700 μm. In the point counting method, the images were first gridded with a predefined grid size (1540 cross section), and then, the areas with black spots that overlapped with the cross sections were counted.

### 4.4.6 Quantification of the niobium-rich carbide

The amount of the niobium-rich carbide was measured by using the point counting approach for all the samples. 5–10 SEM images with the horizontal field width of 60 μm were used to quantify this phase. Figure 4-6 shows the grid pattern on a SEM image. In this method, the white particles were confirmed by EDS to be rich in niobium, as heavier elements absorb higher energies during SEM observation.

![Figure 4-6: SEM (BSE mode) image with gridding for point counting of the niobium-rich carbide.](image)

### 4.4.7 Measurement of primary dendrite arm spacing

Figure 4-7 illustrates the approach used to measure the PDAS by using Eq. 2 [79] and SEM images with the horizontal field width of 45 μm. However, depending on the surface area of the cross sections, the number of SEM images varied in the range 5-10.

\[ PDAS = \frac{L}{n} \]  

(2)

Where L is the length from the middle of one arm to the middle of another one, and n is the number of arms.
Figure 4-7: a) SEM (BSE mode) image of the dendritic structure, and b) a schematic view of the dendrites.
5 Results and discussion

The general microstructural features, e.g., the observed phases, grain structure, and type and amount of defects, in electron beam melting (EBM)-manufactured Alloy 718 are highly dependent on the thermal history of the samples. Mainly, three categories of parameters can affect the microstructural characteristics: position-related, geometry-related, and machine-related parameters. In this research, the effect of the position-related parameters on various features of the microstructure has been primarily investigated to understand the positioning effects on a material due to the different thermal mass/heat accumulations on the build plate. Secondly, both track-by-track and layer-by-layer samples with different geometries were manufactured to gain knowledge of the microstructural evolution of EBM-manufactured Alloy 718. Finally, to acquire a proper track with the desired geometrical and microstructural features, the three main machine-related parameters and their interactions have been analyzed.

5.1 Solidification mode and variation in phases

The solidification mode/morphology of EBM-manufactured samples is highly dependent on their thermal history, which can differ along the build direction. In the cubic geometry with 10 mm height, the solidification mode was found to be predominantly dendritic-cellular with no/fine secondary arms; see Figure 5-1. It was noted that in the cubic samples with different position-related parameters, on the build plate, the columnar dendrites/cells grew epitaxially from the underlying layers and were generally orientated along the \( <100> \) crystallographic direction, which is parallel to the build direction.

![Figure 5-1: Scanning electron microscopy (SEM) (backscatter electron mode; BSE mode) images of the dendritic-cellular structure: a) top area of sample B5, and b) representative image of the middle and bottom areas of sample C6.](image)
By closer inspection of the microstructural evolution of the track-by-track and layer-by-layer samples, it was found that in a single track, the microstructure consisted of the dendritic mode with fine secondary arms in the melt pool; see Figure 5-2. Through the addition of tracks adjacent to each other, the dendritic growth direction in the overlap zone changed, which can be attributed to a severe change in the direction of the thermal gradient during the solidification. The dendritic growth direction in the overlap zones changed about 90° towards either the next track or the scanning direction, as shown at a higher magnification in Figure 5-2(c-d).

![Figure 5-2](image)

As the solidification proceeded, the heavy elements like niobium, molybdenum, and titanium were depleted from the matrix to the liquid and the subsequent formation of common carbide phases such as NbC or TiC was observed mainly in the interdendritic regions; see Figure 5-3 [34]. Among all the elements, regardless of the minor alloying elements, niobium is one of the most important, having a segregation coefficient (defined as the composition of the interdendritic regions divided by that of the dendrite cores) as high as 4.30 [71], [80]. The dendritic core contained mainly the $\gamma$-matrix phase, and in the interdendritic regions, the most typical phase was identified as MC-type carbides by SEM-energy dispersive spectroscopy (EDS) analysis.
In fact, the niobium content in the interdendritic areas depended on both the cooling rate (liquid-to-solid), which was very high during the EBM process, and the dwell time inside the chamber (solid state). A higher cooling rate resulted in lower niobium segregation, since niobium had no time for segregation during the rapid cooling from the liquid to the solid state. Generally, by increasing the dwell time at high temperatures and due to the back diffusion of niobium from the interdendritic areas to the dendrite cores, the niobium segregation decreases, however, it should be considered that during the dwell time, the diffusion distance also increases, which can offset the amount of niobium back diffusion [80].

- **Effect of position-related parameters**

The niobium-rich phase fraction can be affected by manipulating the sample design, owing to the changes in the cooling rate. Upon increasing the height of the samples from the build plate, the cooling rate is lowered, due to the low thermal conductivity of the powder beneath the samples. The reduction in the cooling rate can lead to a high amount of niobium segregation and produce more NbC particles or result in the formation of the δ phase, as shown in Figure 5-3.

Decreasing the gap between the samples leads to high thermal mass/heat accumulations, thus lowering the cooling rate, which promotes the formation of a higher amount of the niobium-rich phase [81]. However, no results were obtained that confirmed this hypothesis, since there were negligible differences between the groups B (10 mm sample gap; 0 mm height from the build plate) and C (2 mm sample gap; 0 mm height from the build plate) or between the groups A (10 mm sample gap; 2 mm height from the build plate) and D (2 mm sample gap; 2 mm height from the build plate). Furthermore, the locations of the samples, either the interior (E1) or the exterior regions (E2, and E3) of the build plate had no significant impact on the niobium-rich phase fraction. However, to clearly explain the effect of sample location on the microstructural characteristics, more investigations are needed.
- **Effect of geometry-related parameters**

The niobium-rich area fraction did not significantly change along the direction of track addition in the track-by-track samples. Moreover, the niobium segregation did not show any clear trend at the top and bottom of the layer-by-layer samples for both the thicknesses of one and five tracks. It was found that by increasing the thickness of the walls (T5L1 to T5L50), the niobium segregation slightly increased, which can be due to the lower cooling rate of the thicker walls.

- **Effect of machine-related parameters**

The niobium-rich phase fraction was measured for the different process conditions considered through design of experiments (DoE) (2) for the EBM-manufactured single tracks. As shown in Figure 5-4, which is a 4D (3 machine-related parameters plus 1 response, which is the niobium-rich phase fraction) contour response graph extracted from the DoE, upon increasing the linear energy input values, the niobium-rich phase fraction also increased. One potential reason for this observation could be the lower cooling rate in a bigger melt pool. It was also observed that at low scanning speeds (up to ~400 mm/s), a higher focus offset was more favorable for obtaining more of the niobium-rich phase fraction; see Figure 5-4. It could be attributed to the geometrical features of the melt pool, which provided the deepest melt pool for the highest focus offset (10 mA), as shown in the section 5.6 (Geometrical features of the melt pool). This led to a lower cooling rate and, subsequently, higher niobium segregation. On the other hand, at higher scanning speeds, a lower focus offset resulted in a greater niobium-rich phase fraction, the reason for which is not fully clear. In general, based on the DoE, the focus offset is an insignificant machine-related parameter with regard to the niobium-rich phase fraction.

![Figure 5-4: 4D contour response illustrating the effect of machine-related parameters on niobium-rich phase fraction.](image)
It was noted that as a result of higher niobium segregation in a bigger melt pool at higher linear energy input values (sample # 1-3, with the highest linear energy input value of 3.00 J/mm), the presence of a few plate-like δ phase particles was observed mainly at the grain boundaries (GBs); see Figure 5-5. This could be due to the lower cooling rate as a result of the higher linear energy input value.

![Image showing δ phase at GBs](image_url)

**Figure 5-5:** SEM (BSE mode) images showing the δ phase at the grain boundaries: a) sample # 1, and b) sample # 3.

### 5.2 Defects

As discussed in chapter 3, different types of defects, e.g., round-shaped pores, lack-of-fusion, and solidification shrinkage pores, can be observed in EBM-manufactured Alloy 718 samples. The locations of the round-shaped pores did not reveal a regular pattern, and were most likely induced by the feedstock powder. On the other hand, the lack-of-fusion defects could have multiple formation mechanisms. One such mechanism can be the lower energy input, which can lead to a poor bonding between the layers in such a rapid solidification process.

One of the most common defects reported from several studies [5], [68], [82] is strings of solidification shrinkage pores that are observed in the interdendritic areas. It is noticeable from this study that this type of pore formed as a band of shrinkage pores that are arranged along the melt pool boundaries, which was not reported in the literature; see Figure 5-6. The potential reason can be the inadequate liquid metal available for compensating the shrinkage occurring during the solidification [83], [84]. The dominant mechanism that induced such shrinkage pores was the cooling of the top molten layers, which started to shrink, but, the underlying layers opposed that shrinkage, finally resulting in shrinkage pores. Moreover, the strings of the shrinkage pores, shown in Figure 5-6, were located in a region perpendicular to the layers and mainly in the interdendritic areas. The type and amount of defects is highly dependent on the position-, geometry-, and machine-related parameters, as will be discussed in the following sections.
Effect of position-related parameters

The amount of defects/pore fraction is one of the critical features that can be sensitive to the position-related parameters. It was observed that by increasing the distance between the samples from 2 mm (the minimum value recommended by the EBM equipment manufacturer) to 10 mm, the pore content increased by ~94%. One reason was the lower thermal mass/heat accumulation for a higher sample gap, which produced a higher amount of unmelted powder particles for pore formation. Therefore, how close the samples can be placed on the build plate is an important consideration.

Elevating the samples relative to the build plate, which acts as a heat sink, is assumed to affect the amount of thermal mass/heat accumulated by altering the cooling rate [23]. It was presumed that the elevated samples of group F, with a higher distance from the build plate (45 mm), were less porous than the samples of groups A (2 mm height from the build plate) and B (0 mm height from the build plate). However, the pore fraction was almost similar in all the three groups. To explain this, it should be mentioned that the temperature of the melt pool in the samples is affected by the thermal conductivity of the powder beneath the samples [23], [85]. A high amount of powder beneath the samples of group F with a floated support structure increased the thermal mass/heat accumulation and decreased the cooling rate of the material, both of which can lead to the formation of a high-temperature melt pool with low stability and vigorous motion. This phenomenon probably promoted the formation of more voids and pores during the solidification of group F samples [86], [87].

The final position-related parameter investigated was the location of the samples on the build plate. It was found that one of the two samples located in the exterior regions (sample E2) exhibited a lower amount of pores compared to the sample located in the interior region (sample E1) of the build plate; on the other hand, the other sample (sample E3) showed a higher pore content than sample E1.
Therefore, the exact reason remains unknown, and further investigation is needed.

- **Effect of machine-related parameters**

In order to understand the root causes of the defects in 3D parts, the single tracks were built with different linear energy input parameters (scanning speed and beam current) and focus offsets. It was found that by applying a low amount of linear energy input, a discontinuous single track was produced, in which the bonding between the track and the substrate did not have enough adhesion (sample # 19, with linear energy input of 0.66 J/mm); on the other hand, at a higher linear energy input, the single track became more stable and continuous (sample # 4, with linear energy input of 2.20 J/mm); see Figure 5-7.

By visually inspecting the top surface of the single tracks (see Figure 5-8), it was found that upon increasing the focus offset for each linear energy input value, the stability of the single tracks increased. In order to rationalize this behavior, analysis of the results of the melt pool width and depth shown in Figure 5-17 reveals that a higher focus offset produces a sharper beam. A sharper beam increases the energy density, which increases the energy absorbed, and leads to a reduction in the exposure area. A less focused beam, on the other hand, reduces the energy density, i.e., the energy absorbed, and increases the exposure area, which further results in unmelted powder [88].

![Figure 5-7: LOM images of the top surface and the cross sections of the start and end points of a) sample # 19, with the scan speed of 1000 mm/s, and b) sample # 4, with the scan speed of 300 mm/s; see Table 4-4.](image-url)
5.3 Grain structure

Figure 5-9 shows a typical grain structure of EBM-manufactured Alloy 718. Generally, when adding a new layer upon the underlying layers, the grains of these layers are partially re-melted and oriented along the build direction. The grain orientation is determined by the alignment of the thermal gradient with the build direction, which is characterized by a high degree of texture along the $<100>$ crystallographic orientation, which is perpendicular to the scanning direction. Upon rapid cooling from the melt, the growing grains aligned themselves with the steepest temperature gradients, which resulted in a columnar morphology. The orientation of the grains is highly dependent on the position-, geometry-, and machine-related parameters, as will be discussed in the following sections.
Effect of position-related parameters

Comparing the grain structures of all the EBM-manufactured sample groups (A, B, C, D, E, and F), no significant differences were observed. The only difference was the group F samples, which exhibited a high degree of texture, namely the $<100>$ crystallographic orientation, with an alignment parallel to the build direction; see Figure 5-10. The presence of more fine grains in all the groups compared to group F demonstrated that the nucleation of new grains ahead of the solidification front was accelerated, which resulted in a lower grain width.

![Figure 5-10: SEM (BSE mode) images of grains: a) sample B4, b) sample F4.](image)

Effect of machine-related parameters

As shown in Figure 5-11, at higher linear energy input values, the probability of obtaining a more equiaxed microstructure is high, whereas at relatively lower linear energy input values, a more or less columnar microstructure is observed. During the solidification, the melt pool boundary acts as a nucleation site for the new grains, where they begin growing towards the beam incidence point [89]. A closer look at the melt pool boundary revealed that the grain growth was intensely competitive during the initial stage of solidification. The nucleation of new grains occurred at the bottom of the melt pool and those with the most favorable orientations with respect to the temperature gradient at the liquid-solid interface grew further, whereas the grains with less favorable orientations were terminated after a short period of growth close to the melt pool boundary. As a result of this competitive grain growth, many small grains can be observed near the melt pool boundary; see Figure 5-11. The large temperature gradient inside the melt pool leads to a high growth rate of the elongated grains and, therefore, there is little chance of grains nucleating at new sites in the bulk melt during the solidification [89], [90]. By increasing the linear energy input, the melt pool size was increased, and resulted in high re-melted depth and melt pool width, as indicated in Figure 5-11. In addition, Figure 5-11 shows that at a low linear energy input value, such as 0.66 J/mm or 0.90 J/mm, the penetration depth is the lowest, and mainly
epitaxial growth of the partially re-melted grains is observed from the stand. Increasing the linear energy input to 3.00 J/mm leads to a larger melt pool, where the probability of crystallization and observation of new small grains is high.

Figure 5-11: a) Schematic of the grain structure obtained by increasing the linear energy input values, and SEM (BSE mode) images of the cross sections showing the grain structure; b) sample #1, with a linear energy input value of 3.00 J/mm; c) sample #10, with a linear energy input value of 1.38 J/mm; d) sample #18, with a linear energy input value of 0.90 J/mm; and e) sample #20, with a linear energy input value of 0.66 J/mm.

The effect of focus offset on the orientation of the grains was revealed by EBSD mapping, and the results are shown in Figure 5-12. It seems that for the linear energy input value of 3.00 J/mm and a focus offset of 0 mA, the grain orientations were mainly along the <100> direction, and the misorientation was slightly lower than those for the focus offsets of 5 and 10 mA. This result was also confirmed for another value of linear energy input. At the linear energy input of 3.00 J/mm, the melt pool was slightly wider and shallower for the focus offset of 0 mA, compared to those observed for the focus offsets of 5 and 10 mA; see Figure 5-12. For a low re-melt depth, the degree of the thermal gradient became higher in a direction perpendicular to the layers, which resulted in a lower degree of misorientation of the grains in the microstructure.
RESULTS AND DISSCUSION

**5.4 Hardness**

As with the other microstructural characteristics, the hardness of the samples was also evaluated in different process conditions.

- **Effect of position-related parameters**

The effect of the height from the build plate on the hardness values showed that the hardness of the samples of groups A (2 mm height from the build plate) and B (0 mm height from the build plate) were almost the same, at around 387±13 HV<sub>0.5</sub>, but slightly lower than that of group F (45 mm height from the build plate), which was around 410±18 HV<sub>0.5</sub>. This small difference can be attributed to the prevention of grain boundary movement due to the precipitation of more δ phase particles in the group F samples. This effect was in turn due to the lower cooling rate of group F samples compared to those of groups A and B. The results of the hardness measurements for different sample gaps between A (10 mm) and C (2 mm), or B (10 mm) and D (10 mm), showed no significant differences, and the hardness values were in the range 387–395 HV<sub>0.5</sub>. The reason might be that a larger difference in the sample gap could be required to observe any changes in the hardness. For the final position-related parameter, the location of the samples on the build plate, no significant difference in the hardness was observed for the samples in the interior and exterior areas of the build plate, and the measured values were around 380–396 HV<sub>0.5</sub>.

- **Effect of geometry-related parameters**

The hardness of the track-by-track samples was in the range ~360–400 HV<sub>0.5</sub> and did not show any significant change between the tracks up to 10 tracks. Figure 5-13 shows the hardness profiles of 1-, 5-, 10-, 25-, and 50-layer deposits obtained from the micro Vickers tests. The slight difference in the hardness values measured at different locations can be attributed to the solidification
characteristics and also to the fact that they experienced successive thermal cycling (STC) after the solidification. Moreover, the differences in the hardness at the bottom layers (the first 20 µm measured from the stand) compared to the top region (3200 µm) in the layer-by-layer samples (both thin and thick walls) were low (<50 HV₀.₅). The slightly higher (~11%) hardness observed at the bottom of these samples can be associated with the finer microstructure obtained as a result of a higher cooling rate.

![Figure 5-13: Hardness profiles for a) single wall with one track and b) thicker walls with five tracks.](image)

- **Effect of machine-related parameters**

The hardness measurement was performed at the center of the melt pool for each sample in each cross section. As discussed in the section on grain structure, increasing the linear energy input value led to a larger melt pool and greater crystallization and formation of new grains. In addition, the grain size, grain orientation, and grain boundaries can affect the hardness of the specimens. Most of the important mechanical properties of alloys, such as the yield stress, hardness, and ductility, can be altered by changing these microstructural features [91]. Therefore, by changing the grain shape (size, orientation, and boundaries), some changes could be expected in the final mechanical properties obtained from the EBM process. It was hypothesized that by the nucleation of more grains at high linear energy input values, the hardness could exhibit an increasing trend due to the increase in the grain boundaries, which act as strong barriers to dislocation movement [49], [92], [93]. However, as shown in the 4D contour response graph obtained from the DoE (Figure 5-14), none of these three parameters (scanning speed, beam current, and focus offset) had a significant effect on the hardness values, and the average values of hardness were in the range 385-409 HV₀.₅.
RESULTS AND DISCUSSION

Figure 5-14: Estimated 4D contour response of the effect of machine-related parameters obtained by a DoE on hardness for different focus offsets (0, 5, and 10 mA).

5.5 Variation in primary dendrite arm spacing

The solidified microstructure is characterized by appropriate length scales, such as the primary and secondary cellular/dendrite arm spacings. The size of these microstructural features can be controlled during the EBM process through proper selections of the growth rate and the temperature gradient. Moreover, PDAS is an important characteristic tool for the estimation of the cooling rate corresponding to the liquid $\rightarrow$ solid and solid $\rightarrow$ solid transformations occurring during solidification [94].

In the EBM process, the beam spot size is about several hundreds of microns, which, in comparison to other metal additive manufacturing (AM) processes, such as laser metal deposition, is quite small [37]. Therefore, the melt pool is significantly small, and accordingly, the solidification is very fast, and the dendrite/cell spacing is also an order of magnitude smaller than that observed in other metal AM techniques [95], [96]. The size of the PDAS is highly dependent on different process parameters, as will be discussed in the following sections.

- Effect of position-related parameters

The PDAS was in the range 6–10 $\mu$m for all the sample groups (A, B, C, D, and E) with different position-related parameters, except group F. The mean value of the PDAS in the group F sample (with 45 mm height from the build plate) was around 17 $\mu$m, which suggests that the dendrite arms are coarser than the others; this can be attributed to the lower cooling rate observed in this sample group, as already explained.
- **Effect of geometry-related parameters**

As the EBM process essentially involves the addition of tracks and layers to build a 3D part, the STC doubtlessly has an influence on the microstructure. The average values of the PDAS slightly increase along the track widths in the track-by-track samples having 3, 5, 7, and 10 tracks. For example, in the sample with 10 tracks, the average value of PDAS increases from 1.7 to 2.5 µm as the number of tracks increased. This is most likely due to the decrease in the cooling rate within the tracks from the first to the last melted track of each sample. The slightly increasing trend can be due to the increased amount of material as a result of the addition of tracks in the samples, which leads to slower heat dissipation and a lower cooling rate.

In the layer-by-layer samples (T1Lx; x: 5, 10, 25, and 50 layers), the average PDAS was lower at the bottom layers than at top of the sample (see Figure 5-15a), which indicated that the cooling rate was higher at the bottom of the samples. The bottom layers were in close contact with the build plate, where the temperature was lower compared to the melt pool temperature, which is above the melting temperature of Alloy 718. Thus, a higher cooling rate was obtained for the bottom layers [97]. A similar result was also observed for the wall thickness from the top to the bottom of the sample with five tracks; see Figure 5-15b.

![Figure 5-15: Variation in the PDAS of a) single wall samples and b) thick walls with five tracks.](image)

- **Effect of machine-related parameters**

As illustrated in Figure 5-16, the dendrite-cell spacing increased upon increasing the linear energy input value, owing to a larger melt pool and a lower cooling rate. The maximum and minimum average values of the PDAS were 5.53±0.43 and
2.8±0.5 µm for samples # 3 and 18, respectively. Based on the DoE analysis, the effect of focus offset on the PDAS was determined to be insignificant.

Figure 5-16: PDAS measurements for different values of linear energy input.

5.6 Geometrical features of the melt pool

Typically, the melt pools are long and shallow during the hatch melting process, which indicates that the solidification involves a downward heat flow at the melt pool boundaries that is opposite to the build direction [29]. In addition, in the bottom layers of cubic samples with 10 × 10 × 10 mm³ geometry, the melt pool boundaries are not clearly visible. However, at the top layers, the melt pool boundaries can be seen, which is likely a result of the severe STC occurring during the manufacturing of the part. By following the trace of the melt pool boundaries in the samples with different geometries (both track-by-track and layer-by-layer samples), the ranges of the width and depth of the melt pool were measured as 250–390 and 40–110 µm, respectively. Considering a consolidated layer thickness of ~75 µm, re-melting occurred in a maximum of two underlying layers. Moreover, the overlap between the tracks was measured to be ~30–50% of each track, which implied that a part of the track was re-melted twice or thrice upon the subsequent addition of a track.

- Effect of machine-related parameters

Variations in the linear energy input parameters, including the scanning speed and beam current, as well as the focus offset, produced different geometrical features in the melted single tracks that were quantified in terms of re-melt depth (d2), melt pool width (w), track height (d1), and average of contact angle (θ).

As shown in Figure 5-17, it can be noted that upon increasing the linear energy input value (high beam currents for lower values of the scanning speed), the width
and depth of the melt pool increased for all the three focus offset values. The averages of the melt pool width and depth were in the ranges 444–822 and 91–511 μm, respectively. Based on the DoE analysis, the main parameters influencing the width and depth of the melt pool were the scanning speed and the beam current, while the other parameters and the interactions between them were insignificant. The increase in the melt pool size upon increasing the linear energy input value can be explained by assuming that large volumes of the powder are involved in track formation.

Figure 5-17: Estimated 4D contour response of the machine-related parameters, obtained by DoE, for a) melt pool width (w) and b) re-melt depth (d2).

The other two geometrical features of interest are the track height and the contact angle, which from literature, have been found to be correlated to each other to a certain extent [98]. With the increase in the track height, the contact angle also increases; see Eq. 3.

\[ \theta = 2 \times \tan^{-1}\left(\frac{2 \times d_1}{w}\right) \] (3)
RESULTS AND DISCUSSION

The track height is highly dependent on the powder layer thickness and most likely varies depending on the stand roughness, particularities of powder raking, levelling, and spreading, and geometrical characteristics of the powder [95]. The average track height for stable and continuous tracks was in the range 88–173 μm. The DoE analysis showed that for a focus offset value of 0 mA, upon increasing the linear energy input, the track height increased. However, for a focus offset value of 5 mA, the track height appeared to be almost constant, regardless of the total linear energy input; see Figure 5-18. In contradiction, for the final focus offset value of 10 mA, a lower linear energy input value resulted in a taller track. It was found that the investigated parameters had no effect on the track height in this predefined range.

The contact angle is a function of the wetting phenomenon and reveals the spreadability of the molten powder on the stand or on a previously melted layer.

Figure 5-18: Estimated 4D contour response of the machine-related parameters, obtained by DoE, for a) track height (d1) and b) contact angle (θ).

The contact angle is a function of the wetting phenomenon and reveals the spreadability of the molten powder on the stand or on a previously melted layer.
The contact angles measured in the different process conditions were in the range 33–67°. According to Figure 5-18, at all the three focus offset values, upon decreasing the linear energy input value, the contact angle increased. In addition, the DoE showed that the main parameter affecting the contact angle was the scanning speed, while the other parameters and their interactions did not show any significant effect. By increasing the beam current, the temperature of the melt pool increased [95]. High temperatures expand the melt pool, which leads to an enlargement in the curvature of the liquid surface as a result of the Marangoni effect, and consequently, an increase in the contact angle. However, the surface tension of the liquid generally decreases at higher temperatures, where wettability and subsequent reduction of the contact angle are promoted [95], [100]. Thus, the contact angle is a function of the Marangoni effect and the surface tension, both of which vary with temperature.
6 Summary of appended papers

Paper A- Influence of build layout and orientation on microstructural characteristics of electron beam melted Alloy 718

Author contribution: As the main and corresponding author, Paria Karimi performed all the experimental characterizations, analyzed all the results, developed the structure of the paper, and was chiefly responsible for writing it. The coauthors contributed in formulating the concepts and ideas, planning the project, manufacturing, and reviewing articles.

Connection to the research questions: The paper tried to answer how the position-related parameters affect the microstructure of Alloy 718 during the EBM process, which is related to RQ1 (refer to section 1.1).

Summary: In the present study, the effects of certain position-related parameters consisting of; a) height from the build plate (Z-axis), b) distance between samples, and c) location in the build plate (X-Y plane) on porosity, NbC fraction and hardness in EBM-manufactured Alloy 718 were studied. The as-built samples predominantly showed columnar structure with strong <100> crystallographic orientation parallel to the build direction, as well as NbC and δ phase in interdendrites and grain boundaries. Increasing the distance from the build plate led to the formation of predominant columnar microstructure. The fine grains were found less in the samples built with the higher height from the build plate. By increasing the height to 45 mm from the build plate, formation of δ phase was enhanced as well as the NbC fraction increased about 116% and hardness slightly increased around 6%, but the porosity fraction was shown not to vary significantly. By increasing the sample gap (from 2 to 10 mm), no significant effect was found on the NbC fraction and hardness, but in the larger sample gap, the porosity fraction was increased about 94%. The effect of sample location on the porosity was evident in which the former decreased in the exterior sample while the latter increased, however, its effect on NbC fraction and hardness was insignificant.

Paper B- Microstructure development in track-by-track melting of EBM-manufactured Alloy 718

Author contribution: As the main and corresponding author, Paria Karimi performed all the experimental characterizations, analyzed all the results, developed the structure of the paper, and was chiefly responsible for writing it.
The coauthors contributed in formulating concepts and ideas, planning the project, manufacturing, and reviewing articles.

**Connection to the research questions:** This paper tried to find some answers related to the microstructural evolution of Alloy 718 within a single layer during the EBM process, which is a part of RQ2 (refer to section 1.1).

**Summary:** The aim of this study was to gain an increased fundamental understanding of the relationship between the geometry-related parameters and the microstructure formed within a single layer. Hence, different numbers of tracks of equal heights were produced using same machine-related parameters for this purpose, varying from one to ten. The microstructural characteristics (grain structure and phases) were analyzed by using characterization techniques. The deposited tracks became a continuous solid material after adding three tracks next to each other upon using a constant scanning speed and current along the track. The direction of dendrites in the overlap zone changed around 90° from the build direction, towards either the adjacent track or the scanning direction. The cross sections of the EBM-manufactured samples showed epitaxial growth of columnar grains with strong <100> texture from the underlying layers and the nucleation of fine elongated grains in the overlap zones. In addition, small equiaxed grains on the surface of the tracks were observed, which are most likely a result of the heat radiated from the surface. Coarsening of the new grains in the overlap zone relative to the new grains on the surfaces of the tracks was an indication of the slightly lower cooling rate in the overlap zone than in the rest of the track, which is due to double beam exposure and thus slower heat dissipation. Characterization of the primary dendritic arm spacing (PDAS) in different tracks for each sample showed slight increases in the PDAS along a single layer, and subsequently, the cooling rate was estimated to be in the range $0.6 \times 10^5$–$3 \times 10^5$ K/s.

**Paper C- Influence of successive thermal cycling on microstructure evolution of EBM-manufactured Alloy 718 in track-by-track and layer-by-layer design**

**Author contribution:** As the main and corresponding author, Paria Karimi performed all the experimental characterizations, analyzed all the results, developed the structure of the paper, and was chiefly responsible for writing it. The coauthors contributed in formulating concepts and ideas, planning the project, manufacturing, and reviewing articles.

**Connection to the research questions:** The aim of this paper was to answer the question, what is the effect of the successive thermal cycling resulting from the geometry-related parameters on the microstructural characteristics of EBM-manufactured Alloy 718, which is related to RQ2 (refer to section 1.1).

**Summary:** The aim of this study was to determine the effects of STC during the multitrack and multilayer manufacturing of Alloy 718 by using the EBM process.
Therefore, samples composed of a single track to 3D samples with maximum 10 longitudinal tracks and 50 vertical layers were manufactured. The relationship between STC, solidification microstructure, interdendritic segregation, phase precipitation (MC, δ phase), and hardness was investigated. The cooling rates (for the liquid-to-solid and solid-to-solid transformations) were estimated by measuring the PDAS, and showed increased values at the bottom, compared to the top of the multilayer samples. Thus, a microstructural gradient was identified along the build direction. Moreover, extensive formation of solidification micro-constituents, including MC-type carbides, which are formed by microsegregation, was observed in all the samples. The electron backscattered diffraction technique revealed a highly textured structure along the <100> direction, with a few grains misoriented at the surfaces of all the samples. The finer microstructure observed at the bottom of the samples resulted in higher (~11%) hardness values compared to the top of the samples.

Paper D- EBM-manufactured single tracks of Alloy 718: Influence of energy input and focus offset on geometrical and microstructural characteristics

Author contribution: As the main and corresponding author, Paria Karimi performed all the experimental characterizations, analyzed all the results, developed the structure of the paper, and was chiefly responsible for writing it. The coauthors contributed in formulating concepts and ideas, planning the project, manufacturing, and reviewing articles.

Connection to the research questions: This paper focused on answering how the machine-related parameters influence the microstructural characteristics of EBM-manufactured Alloy 718, which is related to RQ3 (refer to section 1.1).

Summary: In this research, the linear energy input parameters beam scanning speed, beam current, as well as focus offset, and their effects on the geometry and microstructure of EBM-manufactured single tracks of Alloy 718 were analyzed. Increased scanning speed led to lower linear energy input values (<0.90 J/mm) in a specific range of focus offsets (0–10 mA), which resulted in instability and discontinuity of the single tracks, as well as a balling effect. Decreasing the scanning speed and increasing the beam current resulted in higher melt pool depth and width. Through statistical evaluation, the most influential parameters on the geometrical features were determined to be primarily the scanning speed, and secondly, the beam current. The PDAS decreased significantly upon increasing the scanning speed for lower beam current values as the linear energy input value decreased. By increasing the linear energy input, the probability of obtaining more equiaxed grains was high; on the other hand, for a lower linear energy input, an increased amount of columnar grains were observed. In addition, a lower focus offset resulted in a more uniform grain structure.
7 Conclusions

- The orientations of the parts on the build plate during the electron beam melting (EBM) process can have significant effects on the microstructural appearance. This could be attributed to the different thermal mass/heat accumulations observed during the melting of the part, which can lead to different solidification parameters and, subsequently, different microstructures. It was found that the amount of defects (mainly lack-of-fusion defects), phase fractions, and grain structure were changed for different position-related parameters associated with the build plate. In addition, for lower sample gaps, the amount of defects decreased. Finally, upon elevating the parts relative to the build plate and accordingly reducing the cooling rate, the probability of niobium segregation was increased, which led to the precipitation of the $\delta$ phase.

- Through a closer inspection of the microstructural evolution for different geometries, the influence of successive thermal cycling (STC) on the microstructural features was investigated. It was found that the PDAS was slightly coarser in the overlap zone, compared to a once-melted area of a track. This indicates a slightly lower cooling rate in the overlap zone than in the rest of the track. Characterization of the PDAS in different tracks for each sample showed slightly higher PDAS widths by increasing the number of tracks, which indicated that the cooling rate was decreased by tracks addition along the same layer. In addition, a slightly higher cooling rate was observed in the bottom than in the top layers. Moreover, by increasing the wall thickness, the cooling rate was reduced. The hardness profile along the walls (both single and thick walls) revealed slightly higher values (~11%) for the bottom layers compared to the more elevated layers, due to the finer microstructure of the bottom layers.

- The effects of the three main machine-related parameters (scanning speed, beam current, and focus offset) and their interactions on the geometrical and microstructural features of single tracks were investigated. At lower linear energy input values ($\leq 0.90$ J/mm), the risk of discontinuous tracks and instability was the highest. The most influential parameter on the geometrical features was the scanning speed, followed by the beam current. Compared to the other two parameters, the focus offset was found to have quantitatively less of an effect on the geometrical features. Moreover, by increasing the linear energy input value, the probability of obtaining more equiaxed grains was high; on the other hand, for a lower linear energy input value, more columnar grains were observed. A lower focus offset resulted in a reduced misorientation within the grains.
8 Future work

More work is needed to determine the effects of position-related parameters, including build location, part size, and part angle, on the microstructural characteristics of EBM-manufactured Alloy 718.

Further evaluation of the influences of successive thermal cycling on parts with higher numbers of tracks and layers is necessary to fully understand the microstructural evolution in a typically sized part. Moreover, it is important to gain knowledge on how to achieve the desired microstructure for the required application. In this research, track-by-track samples, containing up to 10 tracks, and layer-by-layer (single wall) samples, containing up to 50 layers, were used to obtain basic knowledge of the EBM technique. Further efforts are needed with other geometries.

In this research, three machine-related parameters (scanning speed, beam current, and focus offset) were investigated over defined ranges, however, the outer frame of the parameter ranges need to be analyzed for establishing a broad window. In addition, the other parameters like layer thickness need to be investigated to understand the effects of these parameters on the microstructural features such as defects. Since, in this study, the machine-related parameters were investigated on a single-track geometry, the next plan can involve building a typical cubic geometry and investigating in the process window considered in this study. A predictive model for the solidification parameters is needed for a more accurate estimation of the cooling rate in EBM-manufactured Alloy 718.

Transmission electron microscopy could be helpful in characterizing very fine phases like γ' and γ'' and their sizes and fractions in the layer-by-layer samples, which can be affected by successive thermal cycling. It can also provide information on the presence/changes to these phases in samples with different machine-related parameters.
References


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Electron beam melting (EBM) as a powder bed fusion technique is an innovative additive manufacturing (AM) process in which a metallic powder is completely melted by using a high-energy electron beam as the heat source. The EBM process typically takes place in vacuum at high temperatures, resulting in relatively dense parts with low residual stresses and material properties that are better than as-cast materials and comparable to those of wrought materials.

The main focus of this research was on understanding the microstructural evolution of Alloy 718 during the EBM process and correlating three groups of process parameters, including the position-related parameters, geometry-related parameters, and machine-related parameters, with the microstructure of the alloy. The position-related parameters considered were the height from the build plate, the distance between parts on the build plate, and part location. The geometry-related parameters were evaluated for track-by-track and layer-by-layer designs to understand the microstructural evolution of Alloy 718 during the EBM process. The machine-related parameters scanning speed, beam current, and focus offset were investigated. These three groups of process parameters have a significant effect on the thermal history of the material, which, among others, affects the amount of defects, grain structure, phases, and geometrical features of the melt pool. It was found that the orientation and design of a part on the build plate has a noticeable effect on the microstructural characteristics. In addition, the most influential machine-related parameters on the geometrical and microstructural features were the scanning speed and the beam current, which were related to the amount of linear energy input.