Additive Manufacturing (AM), commonly known as 3D Printing, is a modern manufacturing process, in which parts are manufactured in a layer-wise fashion. It is a disruptive technology and is even referred to as the third industrial revolution. Though AM processes offer several advantages, the suitability of these processes to replace conventional manufacturing processes must be studied in detail. Therefore, understanding the process–post-treatment–microstructure–property relationship is crucial, to enable manufacturing of high-performance components. The aim of this work is, therefore, to understand how the fatigue properties of Alloy 718 processed by Powder Bed Fusion (PBF) additive manufacturing is affected by the microstructure and the as-built surface roughness.

Defects are detrimental to fatigue life; however, numerous factors such as the defect type, size, shape, location, distribution and nature determine the effect of defects. Hot Isostatic Pressing (HIP) improves fatigue life as it leads to closure of defects. Presence of oxides in the defects, however, hinders complete closure by HIP. Machining the as-built surface improves fatigue life; however, the extent of improvement can be dependent on the amount of material removed. The rough as-built surface has numerous crack initiation sites, leading to a lower scatter in fatigue life. In PBF processed Alloy 718, fatigue crack propagation is transgranular; crack propagation is affected by grain size and texture of the material.
Fatigue Properties of Additively Manufactured Alloy 718

Arun Ramanathan Balachandramurthi
To my family
“If I have seen further it is by standing on the shoulders of giants.” — Sir Isaac Newton

“... I seem to have been only like a boy playing on the seashore, and diverting myself in now and then finding a smoother pebble or a prettier shell than ordinary, whilst the great ocean of truth lay all undiscovered before me.” — Sir Isaac Newton
Acknowledgements

The research work for this thesis was carried out at the research environment Production Technology West (PTW) of University West in Trollhättan, Sweden. The project has been funded by KK Foundation through the grant SUMAN Next. Financial support by The Swedish Agency for Economic and Regional Growth through the 3DPrintPlus grant is also greatly acknowledged.

It has been a privilege to have Prof. Per Nylén, Prof. Robert Pederson and Prof. Johan Moverare as my supervisors for the doctoral studies. Thank you for all your guidance and support so far! You trusted me, provided me with the scientific freedom I hoped for and offered an excellent environment for me to grow as a professional. I look forward to continuing working with you for the remaining years of the doctoral studies. I am thankful to Dr. Joakim Ålgård, Assoc. Prof. Joel Andersson and Prof. Shrikant Joshi for their valuable inputs during the work.

I would like to thank research engineers Jonas Olsson, Mats Högström, Kenneth Andersson who have been very helpful in co-ordinating various tasks of the project, working in the lab, sharing valuable experience and most of all – asking all the right questions and pushing my boundaries. I would like to express my gratitude to fellow PhD students for all the meaningful brainstorming sessions throughout the last couple of years, particularly Chamara Kumara who has always been positive and open minded for collaborative effort. To the ever-helpful administrative colleagues Eva Bränneby and Victoria Sjöstedt – thanks for all the help; without your support, things might not have been as easy! A collective acknowledgement goes to all the colleagues at PTW for the positive working atmosphere and sharing your research wisdom, I have enjoyed two years of research and look forward to the future years! I also want to thank PhD student Dunyong Deng for all the help while carrying out the microscopy and texture analysis work at Linköping University. It has been a pleasure to supervise Nikhil Dixit during his master’s thesis, who has been very helpful with the material characterization work.

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The industrial collaboration with Arcam, Element (formerly Exova Materials Technology), GKN Aerospace Engine Systems, Sandvik Materials Technology, Siemens
Industrial Turbomachinery and Quintus Technologies made this project, in this exciting field of metal additive manufacturing, even more fascinating. I sincerely thank the representatives from the companies for the material support and technical services, critical feedback, inspiring discussions and sharing valuable knowledge throughout.

To all my friends, here in Sweden, back in India and spread across the rest of the world, thank you for all the love, care and advice all along. Without you people, this journey in Sweden for the past six years would not have been the same!

Last but not the least, I express my deepest gratitude to my parents, Nirmala and Balachandramurthi and my wife, Priya. It is your love, care, constant encouragement and tolerance throughout that made all the academic accomplishments possible!

Arun Ramanathan Balachandramurthi

December 2018
Populärvetenskaplig Sammanfattning

Nyckelord: Utmattning; Additiv tillverkning; Pulverbädd; Superlegering; Mikrostruktur; Ytfinhet

Additiv tillverkning (AM), allmänt känt som 3D printing, är en moderna tillverkningsprocess i vilken material byggs lager på lager. Även om AM-processer erbjuder många fördelar, måste lämpligheten av dessa processer studeras i detalj innan de fullt ut kan komma att ersätta konventionella tillverkningsprocesser. Därför är kunskap och förståelse för sambanden mellan AM-process – efterbehandling – mikrostruktur - egenskaper avgörande för att möjliggöra tillverkning av högpresterande komponenter. Målet med detta arbete är därför att förstå hur utmattningsegenskaperna hos pulverbäddtillverkad legering 718 material påverkas av mikrostrukturen och av ytfinheten hos material med såväl råytor som bearbetade ytor.

Defekter kan ha en dramatisk negativ inverkan på utmattningshållfastheten. Beroende på olika faktorer, så som defekters art, storlek, geometri, position och fördelning, så varierar deras påverkan. Varm Isostatisk Pressning (HIP) förbättrar utmattningshållfastheten eftersom det leder till att de flesta defekterna stängs. Närvaron av oxider i defekterna hindrar emellertid fullständig stängning med hjälp av HIP. Bearbetning av den råa ytan hos färdigbyggt material förbättrar utmattningshållfastheten betydligt. Graden av förbättring av utmattningshållfastheten beror emellertid på hur mycket material som avlägsnas ifrån ytan. Den grova råa ytan i färdigbyggt tillstånd har många sprickinitieringsställen, vilket leder till en mindre spridning i utmattningshållfasthet. Vid utmattningsprovning av legering 718 material tillverkad med pulverbäddstekniker sker sprickpropageringen transgranulärt; sprickpropagering påverkas av kornstorlek och textur hos materialet.
Abstract

Title: Fatigue Properties of Additively Manufactured Alloy 718

Keywords: Fatigue; Additive Manufacturing; Powder Bed Fusion; Superalloy; Microstructure; Surface roughness

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  978-91-88847-12-6 (Electronic)

Additive Manufacturing (AM), commonly known as 3D Printing, is a disruptive modern manufacturing process, in which parts are manufactured in a layer-wise fashion. Among the metal AM processes, Powder Bed Fusion (PBF) technology has opened up a design space that was not formerly accessible with conventional manufacturing processes. It is, now, possible to manufacture complex geometries, such as topology-optimized structures, lattice structures and intricate internal channels, with relative ease. PBF is comprised of Electron Beam Melting (EBM) and Selective Laser Melting (SLM) processes.

Though AM processes offer several advantages, the suitability of these processes to replace conventional manufacturing processes must be studied in detail; for instance, the capability to produce components of consistent quality. Therefore, understanding the relationship between the AM process together with the post-treatment used and the resulting microstructure and its influence on the mechanical properties is crucial, to enable manufacturing of high-performance components. In this regard, for AM built Alloy 718, only a limited amount of work has been performed compared to conventional processes such as casting and forging. The aim of this work, therefore, is to understand how the fatigue properties of EBM and SLM built Alloy 718, subjected to different thermal post-treatments, is affected by the microstructure. In addition, the effect of as-built surface roughness is also studied.

Defects can have a detrimental effect on fatigue life. Numerous factors such as the defect type, size, shape, location, distribution and nature determine the effect of defects on properties. Hot Isostatic Pressing (HIP) improves fatigue life as it leads to closure of most defects. Presence of oxides in the defects, however, hinders complete closure by HIP. Machining the as-built surface improves fatigue life; however, for EBM manufactured material, the extent of improvement is dependent on the amount of material removed. The as-built surface roughness, which has numerous crack initiation sites, leads to lower scatter in fatigue life. In both SLM and EBM manufactured material, fatigue crack propagation is transgranular. Crack propagation is affected by grain size and texture of the material.
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# Acronyms

<table>
<thead>
<tr>
<th>Acronym</th>
<th>Description</th>
</tr>
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<tbody>
<tr>
<td>2D</td>
<td>Two dimensional</td>
</tr>
<tr>
<td>3D</td>
<td>Three dimensional</td>
</tr>
<tr>
<td>AM</td>
<td>Additive Manufacturing</td>
</tr>
<tr>
<td>AMS</td>
<td>Aerospace Material Specification</td>
</tr>
<tr>
<td>ASTM</td>
<td>American Society for Testing and Materials</td>
</tr>
<tr>
<td>ATP</td>
<td>Advanced Turboprop</td>
</tr>
<tr>
<td>BCT</td>
<td>Body Centered Tetragon</td>
</tr>
<tr>
<td>CAD</td>
<td>Computer Aided Design</td>
</tr>
<tr>
<td>EBM</td>
<td>Electron Beam Melting</td>
</tr>
<tr>
<td>EBSD</td>
<td>Electron Backscattering Diffraction</td>
</tr>
<tr>
<td>EDS</td>
<td>Energy Dispersive X-ray Spectroscopy</td>
</tr>
<tr>
<td>FCC</td>
<td>Face Centered Cubic</td>
</tr>
<tr>
<td>FCGR</td>
<td>Fatigue Crack Growth Rate</td>
</tr>
<tr>
<td>GE</td>
<td>General Electric</td>
</tr>
<tr>
<td>HCF</td>
<td>High Cycle Fatigue</td>
</tr>
<tr>
<td>HCP</td>
<td>Hexagonal Close Packed</td>
</tr>
<tr>
<td>HIP</td>
<td>Hot Isostatic Pressing</td>
</tr>
<tr>
<td>LCF</td>
<td>Low Cycle Fatigue</td>
</tr>
<tr>
<td>LEAP</td>
<td>Leading Edge Aviation Propulsion</td>
</tr>
<tr>
<td>LoF</td>
<td>Lack of fusion</td>
</tr>
<tr>
<td>PBF</td>
<td>Powder Bed Fusion</td>
</tr>
<tr>
<td>ppm</td>
<td>parts per million</td>
</tr>
<tr>
<td>Acronym</td>
<td>Description</td>
</tr>
<tr>
<td>---------</td>
<td>-------------</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning Electron Microscope</td>
</tr>
<tr>
<td>SGT</td>
<td>Siemens Gas Turbine</td>
</tr>
<tr>
<td>SLM</td>
<td>Selective Laser Melting</td>
</tr>
<tr>
<td>STA</td>
<td>Solution Treatment and Ageing</td>
</tr>
<tr>
<td>URQ</td>
<td>Uniform Rapid Quenching</td>
</tr>
<tr>
<td>µCT</td>
<td>micro-Computed Tomography</td>
</tr>
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Author’s contributions

**Paper A.** Influence of defects and as-built surface roughness on fatigue properties of additively manufactured Alloy 718.

Published in *Material Science and Engineering A*, Volume 735, Pages 463-474, September 2018 – Authors: Arun Ramanathan Balachandramurthi, Johan Moverare, Nikhil Dixit and Robert Pederson.

*Author contribution:* Principal author and idea originator. Planned the experiments and material characterization methods. Performed major parts of fractography and characterization of defects. Analysed data and complied results.

**Paper B.** Microstructural influence on fatigue crack propagation during high cycle fatigue testing of additively manufactured Alloy 718.

Under review in *Materials Characterization* – Authors: Arun Ramanathan Balachandramurthi, Johan Moverare, Nikhil Dixit, Dunyong Deng and Robert Pederson.

*Author contribution:* Principal author and idea originator. Planned the experiments and material characterization methods. Performed major parts of fractography and material characterization. Analysed data and complied results.


Under review in *Materials* – Authors: Arun Ramanathan Balachandramurthi, Johan Moverare, Satyapal Mahade and Robert Pederson.

*Author contribution:* Principal author and idea originator. Planned the experiments and material characterization methods. Performed major parts of fractography and metallography, analysed data and complied results.
1 Introduction

Firstly, this chapter provides the necessary background information to situate the research topic of this thesis. It prepares the readers to appreciate the relevance of the chosen research problems to the manufacturing industry. Secondly, it summarizes the identified research gap and the scope of this thesis together with the limitations. In addition, it presents the research questions addressed during this thesis along with the adopted research approach. Finally, it also presents the outline of the thesis.

1.1 Background

Additive Manufacturing (AM) or 3D printing has been disrupting the manufacturing industry in such a way that it is being compared to the industrial revolution [1]. As the name suggests, it is a manufacturing method that is “additive” in nature whereby components are built progressively by adding layers of material in a step-by-step fashion. In 3D printing, a computer aided design (CAD) file, corresponding to the part to be manufactured, is first sliced digitally and then each slice is printed one upon the other in a 3D printer. This method is different from the conventional “subtractive” manufacturing in which components are manufactured by progressive material removal.

Powder Bed Fusion (PBF) processes for metals, a sub-class of AM technology, are bringing about a variety of changes in the manufacturing industry e.g. a new design thinking and manufacturing difficult to process materials. In metal PBF, parts are built in a layer-upon-layer fashion with pre-alloyed metal powder as a precursor by selectively melting each layer of powder according to the required geometry while ensuring fusion to the previously built layer. Depending on the heat source for melting, the process could be classified as Selective Laser Melting (SLM), which utilizes a laser beam, or Electron Beam Melting (EBM), which utilizes an electron beam [2].

AM processes are capable of manufacturing complex geometries in near-net-shape; particularly, the powder bed processes have opened up an entirely new design space, which previously has not been accessible with the conventional manufacturing processes. Commercial examples in the aerospace and gas turbine industry that have capitalized on this design space include the fuel nozzle manufactured for the CFM LEAP engines, burners for the SGT-800 turbine, parts for Advanced Turboprop (ATP) engine, parts for CT7-2E1 helicopter engine and the
injection head of the Ariane rocket engine. New redesigned fuel nozzle for the LEAP engine by GE, while sporting an intricate system of cooling channels, is a single part in contrast to the older one that was fabricated by welding and brazing of 18 separate parts [3]. The new burners made by Siemens for the SGT-800 is also a single part instead of a 13-part assembly. It is redesigned, with internal fuel and air pipes, to reduce the risk of damages. In addition, the burner front has an integrated lattice structure that can only be produced by PBF processes [4]. Both these parts are manufactured by the SLM process and are 25% lighter than the older designs. Similar, even more striking, examples of part integration are found in the ATP engine that has 12 AM parts replacing 855 conventionally made parts [5], in the CT7-2E1 engine that has 16 AM parts in place of more than 900 subtractive manufactured parts [5] and the all-in-one injection head of Ariane 6 propulsion system, which replaces 248 individual parts while being 50% lower in overall costs [6]. This extent of part integration does not only make these parts lighter but also cost efficient. The use of AM in some of these cases has expedited the design to product phase as well. At GE aviation, the EBM process has become the first choice process to manufacture low pressure turbine blades in titanium aluminide (TiAl), a relatively difficult to process material, instead of lost-wax casting and spin casting [7]. TiAl blades are used in the low pressure turbine in GEnx [8] and GE9X [9] engines. Siemens has demonstrated that new turbine blades built by AM using nickel based superalloys with complex cooling channels leads to an improvement in overall engine efficiency [10].

From an industrial standpoint, near-net-shaping, part integration, and flexibility in design, translates to lowering the manufacturing costs. So, the appropriate use of different AM processes using different materials has been explored within a variety of industries including aerospace, automotive and medicine. At the current level of technical maturity, AM is better suited for industries with low-volume-high-customization production environments. Though AM processes offer flexibility in design, the suitability of these processes to replace conventional manufacturing processes must be studied in detail; for instance, the capability to produce components of consistent quality. Therefore, understanding the relationship between the AM process together with the post-treatment used and the resulting microstructure and its influence on the mechanical properties is crucial, to enable manufacturing of high-performance components.

1.2 Research gap

In EBM processing of Alloy 718, the average bed temperature is around 1000 °C. This, in combination with layer-by-layer melting sets up a thermal gradient from the molten/solidifying layer towards the base plate, on which the parts are built. Due to the directional thermal gradient, epitaxial grain growth is favoured which
INTRODUCTION

leads to a strongly textured columnar microstructure. Such a textured microstructure gives rise to anisotropic properties, particularly ductility and yield strength, in directions parallel and perpendicular to the grain orientation [11]–[13]. It is, however, possible to print Alloy 718 parts with equiaxed microstructure by carefully controlling the EBM process variables and melting strategy [14]–[16]. The mechanical properties associated with this equiaxed microstructure are different from that of the columnar microstructure [17]. Similar approach of modifying the process parameters to tailor the microstructure have been attempted in SLM and the resulting mechanical properties have been investigated [18], [19].

Different melting strategies, however, give rise to different defect distribution patterns within a printed part [20]. Hot Isostatic Pressing (HIP) is routinely employed as a post-process step to get rid of porosity and other process induced defects for superalloy parts made by casting and powder metallurgy [21]. HIP is a crucial post-process/thermal post-treatment step for AM parts which aims at getting rid of porosity and lack of fusion defects; however, it is important to also consider the effects of HIP on different phase constituents at the same time. The thermal signature of the PBF processes are different from that of the casting and forging, which could mean that for standalone PBF parts, new solution treatment and ageing (STA) routines have to be designed to ensure that a desired microstructure, with appropriate mechanical properties, is achieved. However, if PBF parts are to be used in tandem with conventionally manufactured parts – as welded assemblies, conventional thermal post-treatment protocols would have to be used. Therefore, understanding the effect of such thermal post-treatments on the mechanical properties together with properties such as weldability, and how the resulting microstructural features are related to these properties, is crucial.

One of the key strengths of PBF AM technology is the possibility to build complex shapes that are difficult, or even impossible, to manufacture with conventional processes. The as-built surface roughness, however, could be a limiting factor for mechanical performance, especially in fatigue critical components. Post-build machining or other methods of surface finishing of such complex shapes could be difficult and expensive or sometimes impossible; but scaling down the complexity to ease the post-processing would undermine the geometric capabilities of the technology.

Only a limited amount of work has been carried out towards understanding the process–microstructure–mechanical property relationship on AM built Alloy 718 parts compared to conventional processes such as casting and forging. Considering different post-processing steps such as HIP, STA, surface treatments etc., that could be applied, extensive research must be carried out to achieve the same ex-
tent of understanding and knowledge on process–microstructure–property relationship as for cast or forged Alloy 718. The effect of texture control, combining columnar and equiaxed microstructures in different configurations within the same part, remains to be investigated in detail to adopt such design strategies for manufacturing of actual components using AM. Such a texture control, if employed, has to be a design consideration. In addition, it is important to determine the knockdown on properties due to the roughness of the as-built surface and then to explore ways of reducing this knockdown to further improve the overall performance.

1.3 Scope of research

EBM and SLM processes for Alloy 718, a superalloy that is widely used within the aerospace and gas turbine industry, is explored within this work. Apart from the static properties, which include yield strength, Young’s modulus and ductility, the dynamic properties such as fatigue properties are of importance in such applications. Understanding how the mechanical properties are affected by the microstructure i.e., the amount and distribution of different phase constituents and defects obtained by manipulating the process parameters in combination with different post-build treatments is the focus of this research. In addition, the effect of texture in the microstructure and the as-built surface topography, are also investigated. Though both static and dynamic properties are evaluated in this thesis work, emphasis is placed on understanding the factors affecting the fatigue properties.

1.4 Limitations

All the specimens manufactured by the EBM process were built using the Arcam A2X machine R1235, available at University West, Trollhättan, Sweden. The powder utilized is supplied by Arcam. Only one batch of 100 kg powder has been utilized with in the project. EBM control software v4.2 was used to run the A2X R21235 machine for building the specimens. All the specimens in this study were built with Arcam standard theme for building with Alloy 718.

All the specimens manufactured by the SLM process were built using EOS M290 machines at Siemens Industrial Turbomachinery AB, Finspång, Sweden. EOS standard theme for building with Alloy 718 has been utilized along with the powder supplied by EOS.

All the tests have been performed on specimens that were extracted from separate prismatic bars, which were built to dimensions encapsulating the test specimen geometry.
1.5 Research Questions

The following research questions are formulated to address the identified research gaps and form the basis of the work presented in this thesis.

1) How does the microstructure of EBM and SLM built Alloy 718 material, in as-built and/or post-treated condition, affect the mechanical properties?
   i) How do the different defects affect the mechanical properties?
   ii) How do the different phase constituents affect the mechanical properties?
   iii) How does the texture affect the mechanical properties?

2) How does the as-built surface roughness of the EBM and SLM parts affect the fatigue properties?

1.6 Research Approach

An experimental approach is utilized in this project. First, required specimens are built using appropriate melting strategies and process parameters based on literature study and knowledge developed from carefully designed screening experiments. Second, these specimens are subjected to a combination of thermal post-treatments such as HIP and/or STA treatment and surface treatments, keeping in mind the different research questions that are addressed within each of the experiments. Third, suitable mechanical testing for both static and dynamic properties is carried out in as-built and/or post-treated conditions. Last, material characterization work involving fractography and metallography, is performed to understand and map how the microstructure affects the obtained properties. Analysis techniques such as Scanning Electron Microscopy (SEM), Electron Back Scattering Diffraction (EBSD) analysis, among other advanced techniques, have been used to this end whenever appropriate.

1.7 Thesis Outline

The thesis is outlined as follows. Following this introductory chapter are Chapters 2 and 3 that provide a background to Alloy 718 and AM processing of the alloy. These chapters also include a general background to Nickel based superalloys and metal AM, respectively. In addition, these chapters also include a review of fatigue properties of the alloy that is made by conventional manufacturing and AM, respectively. Chapter 4 presents the details pertaining to the materials and the experimental methods that were used in the present work. Chapter 5 presents a summary of the appended papers, which is followed by conclusions and future
work in Chapter 6. At the end, publications out of this research thesis (Paper A, Paper B and Paper C) are appended.
2 Alloy 718

This chapter gives a brief introduction to superalloys and Alloy 718. The chemical composition, phases that constitute the microstructure and the commonly performed heat treatments are presented. Finally, previous research related to the effect of microstructural characteristics on the fatigue properties of Alloy 718 is discussed.

2.1 Nickel-based superalloys

Superalloys, as a class of materials, are difficult to define exactly. Superalloys are alloys based on group VIIIA elements that are developed for high temperature applications, which encounter severe mechanical loading and require high surface stability [21]. Superalloys are capable of operating at a homologous temperature, i.e., ratio of the operating temperature to the melting temperature of an alloy, greater than 0.6 [22]. At such high temperatures, superalloys retain their properties such as yield strength, tensile strength, fracture toughness, fatigue strength etc., without degradation for extended times. Further, these alloys have high resistance to creep deformation and corrosion.

The development of superalloys has been concurrent with the development of gas turbine engines. In addition to gas turbine engines, their excellent properties have made superalloys suitable for application in petrochemical industries, nuclear reactors, power plants, space vehicles, cryogenic tanks etc.

Superalloys are classified as nickel-based, cobalt-based and iron-based superalloys. Another major subclass is nickel-iron based superalloys that have higher iron content but similar characteristics to nickel-based superalloys [21]. A remarkable feature of nickel, responsible for its success as a base material for superalloys, is its high tolerance for alloying without any phase instability. Therefore, Nickel-based superalloys, with addition of more than 10 elements, have a complex chemistry. Each of the alloying element has a specific role towards the microstructure and in turn the properties. Some of the alloying elements and their intended purposes are listed in Table 1. Some other elements such as rhenium, hafnium and zirconium are added as well. Apart from these elements for which the composition is carefully designed and controlled, “tramp” elements such as sulphur, phosphorous, silicon, oxygen and nitrogen are limited by appropriate means.
Table 1. Common elements in superalloys [21], [23].

<table>
<thead>
<tr>
<th>Intended alloying purpose</th>
<th>Ni</th>
<th>Fe</th>
<th>Cr</th>
<th>Co</th>
<th>Mo</th>
<th>W</th>
<th>Al</th>
<th>Ti</th>
<th>Nb</th>
<th>Ta</th>
<th>C</th>
<th>N</th>
<th>B</th>
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<tbody>
<tr>
<td>γ matrix</td>
<td>x</td>
<td></td>
<td></td>
<td></td>
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<td></td>
<td></td>
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<tr>
<td>Solid solution strengthening of γ</td>
<td></td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td></td>
<td>x</td>
<td></td>
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<td></td>
<td>x</td>
</tr>
<tr>
<td>γ' former</td>
<td></td>
<td></td>
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<td>x</td>
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<tr>
<td>γ&quot; former</td>
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<td></td>
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<td></td>
<td></td>
<td>x</td>
</tr>
<tr>
<td>Solid solution strengthening of γ'</td>
<td></td>
<td>x</td>
<td></td>
<td></td>
<td></td>
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<td></td>
<td></td>
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<td></td>
<td>x</td>
<td></td>
</tr>
<tr>
<td>MC and M(C,N) former</td>
<td></td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td></td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td></td>
<td></td>
<td>x</td>
</tr>
<tr>
<td>M_{23}C_6 former</td>
<td></td>
<td>x</td>
<td></td>
<td>x</td>
<td>x</td>
<td></td>
<td></td>
<td></td>
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<td></td>
<td></td>
<td>x</td>
<td></td>
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<tr>
<td>M_6C former</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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<td></td>
<td></td>
<td></td>
<td>x</td>
<td></td>
<td>x</td>
</tr>
<tr>
<td>M_3B_2</td>
<td></td>
<td>x</td>
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<td></td>
<td></td>
<td></td>
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<td></td>
<td></td>
<td>x</td>
</tr>
<tr>
<td>Surface oxide former</td>
<td></td>
<td>x</td>
<td></td>
<td></td>
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<td></td>
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<td></td>
<td>x</td>
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</tr>
</tbody>
</table>
2.1.1 Microstructure

The microstructure of superalloys is complex and dynamic due to the heavy alloying, but a good understanding of the multiple phases that form and various ways of achieving them has been developed over the years. In general, following are the common phases present in nickel-based superalloys, based on specific composition, as summarized by Sims et al [21] and DuPont et al [23]:

1. **γ matrix**: The continuous matrix is an austenitic phase with FCC structure called Gamma. Various alloying elements such as chromium, cobalt, iron, molybdenum, tungsten are present at high levels in solid solution. The elements differ in atomic size by up to 13% and distort the gamma lattice to locally strain it, therefore impede dislocation movement, and provide solid solution strengthening.

2. **γ’ phase**: It is an ordered intermetallic phase of composition Ni$_3$(Al,Ti) and L1$_2$ structure. It has a good crystallographic matching with the FCC γ matrix and hence has a relatively higher stability but is a metastable phase. This phase often has cubic or spherical morphology as precipitates and confers strength by precipitation hardening mechanism. γ’ is unique in a way that its strength increases with increasing temperature up to 800 °C. In some alloys, γ’ is intended to form in film morphology at the grain boundaries to improve rupture properties. In alloys with sufficiently high titanium content, Ni$_3$Ti η phase that has HCP structure can form on long term high temperature exposure, by transformation of γ’, with corresponding loss of strength.

3. **γ” phase**: It forms in alloys that have appreciable amount of niobium. It is an ordered intermetallic of Ni$_3$Nb composition and BCT D0$_{22}$ crystal structure. It often has a disc shaped morphology with considerably higher lattice misfit than γ’ and is a metastable precipitate. The lattice constant of γ” along c$_0$ direction is twice that of γ’. γ’” confers strength by precipitation hardening mechanism.

4. **δ phase**: It also has the composition Ni$_3$Nb but has orthorhombic D0$_a$ structure and can form on long term high temperature exposure by transformation of γ”. It can also form directly on slow cooling from temperatures above 1010 °C. Small quantities of δ phase at the grain boundaries is beneficial for rupture strength, but excessive amounts especially in intragranular form is detrimental for ductility.

5. **Carbides**: The most common carbides found are MC, M$_{23}$C$_6$ and M$_6$C type. MC carbides are usually FCC structured and form at the end of solidification by eutectic type reaction, hence are called primary carbides.
These are distributed both as intra-granular and grain boundary precipitates with random cubic or script morphology. Often MC carbides are formed by reactive elements such as niobium, titanium, tantalum, molybdenum and tungsten. M₃C₆ and M₆C type carbides form by decomposition of MC carbides. M₂₃C₆ carbides are common in alloys with higher chromium and prefer grain boundaries. These form in temperature range of 760–980 °C. M₆C carbides form in alloys rich in molybdenum and/or tungsten in temperature range of 815–980 °C. Carbides present at grain boundaries as discrete particles are beneficial for creep strength and retard grain growth, as well while the film morphology has detrimental effects on ductility.

6. **Borides**: Boron is added to superalloys to improve grain boundary strength under high temperature deformation. It forms M₃B₂ type hard borides at grain boundaries on reacting with metal elements such as molybdenum, titanium, chromium and cobalt, which could be detrimental for weldability.

7. **σ, μ and Laves**: In some alloys other phases such as σ, μ and Laves phases form, under specific conditions for an alloy dependent on its chemistry. They form either at the end of solidification due to elemental segregation, or during thermal processing and/or long-term exposure. In general, these phases have complex structures and are detrimental to mechanical properties. They often have plate like morphology and are brittle in nature due to which they act as crack initiation sites; they also consume, and deprive the matrix of, important elements that are responsible for strengthening mechanisms.

### 2.2 Microstructure of Alloy 718

Alloy 718 was introduced by Huntington Alloys Division of International Nickel Company in 1959 under the name *Inconel 718* [24]. In the following years, it was quickly adapted by aerospace companies – first, for military engine components and later, for several components of space shuttle engines and commercial aircraft engines. The major reasons for wide spread adaptation were its high strength, its processability in cast and wrought forms, its weldability and lower cost [24], [25]. Later, the potential of Alloy 718 was realized in oil and gas industry [26] due to its aqueous corrosion resistance. Since then it has become one of the widely used superalloys and is used as generic alloy in nuclear and cryogenic applications [27].

Alloy 718, like other superalloys, has a complex chemistry; the chemical composition of aerospace grade Alloy 718 is presented in *Table 2*. The common phases found in the alloy are γ, γ', γ”, δ, (Nb,Ti)C, TiN and Laves; other phases such as
σ and M₆C are not observed anymore due to better cleanliness of the alloy [28]. M₂₃C₆ commonly found in other superalloys is not present in Alloy 718. α-Cr forms in small quantities only on long term exposure in the temperature range 593 °C to 732 °C [28], [29].

Table 2. Chemical composition of Alloy 718 as per AMS 5662 [30]

<table>
<thead>
<tr>
<th>Element</th>
<th>Min</th>
<th>Max</th>
<th>Element</th>
<th>Min</th>
<th>Max</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni</td>
<td>50.00</td>
<td>55.00</td>
<td>Mn</td>
<td>-</td>
<td>0.35</td>
</tr>
<tr>
<td>Cr</td>
<td>17.00</td>
<td>21.00</td>
<td>Si</td>
<td>-</td>
<td>0.35</td>
</tr>
<tr>
<td>Fe</td>
<td>-</td>
<td>Remainder</td>
<td>P</td>
<td>-</td>
<td>0.015</td>
</tr>
<tr>
<td>Nb</td>
<td>4.75</td>
<td>5.50</td>
<td>S</td>
<td>-</td>
<td>0.015</td>
</tr>
<tr>
<td>Ta</td>
<td>-</td>
<td>0.05</td>
<td>Cu</td>
<td>-</td>
<td>0.3</td>
</tr>
<tr>
<td>Al</td>
<td>0.20</td>
<td>0.80</td>
<td>B</td>
<td>-</td>
<td>0.006</td>
</tr>
<tr>
<td>Ti</td>
<td>0.65</td>
<td>1.15</td>
<td>Pb</td>
<td>-</td>
<td>5 ppm</td>
</tr>
<tr>
<td>Mo</td>
<td>2.80</td>
<td>3.30</td>
<td>Bi</td>
<td>-</td>
<td>0.3 ppm</td>
</tr>
<tr>
<td>C</td>
<td>-</td>
<td>0.08</td>
<td>Se</td>
<td>-</td>
<td>3 ppm</td>
</tr>
<tr>
<td>Co</td>
<td>-</td>
<td>1.00</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

2.2.1 Solidification of Alloy 718

The partitioning behavior of the alloying elements results in the formation of a segregated microstructure. During the solidification process, the larger atoms such as Nb, Mo and Ti are rejected from the γ matrix due to which NbC, TiN and Laves phase form [28]. A solidification diagram, for Alloy718, based on pseudo-binary system of Ni-Nb was proposed by Knorovsky et al [31] while studying the non-equilibrium solidification conditions that occur during welding. According to the diagram, the γ matrix starts to form from the liquid phase, which is followed by the formation of the primary carbide, NbC. The formation of carbides is driven primarily by the partitioning of elements leading to the enrichment of the liquid phase. Following the NbC precipitation, the composition shifts back to solidification of only γ phase from the liquid. The terminal solidification happens with formation of γ/Laves eutectic. While studying the effect of cooling rate on solidification of Alloy 718 [32], in the multicomponent system, it was shown that the precipitation of TiN preceded NbC formation. Further, the study also showed that as the cooling rate increased, the dendritic solidification structure of Alloy 718 became finer which then changed to a cellular morphology. At very high cooling rates, diffusionless transformation occurred; diffusionless transformation, however, resulted only in formation of nucleation sites for cellular solidification.
While $\gamma$, NbC, TiN and Laves form during the solidification, other phases such as $\gamma'$, $\gamma''$ and $\delta$ form due to solid state transformation.

### 2.2.2 $\gamma'$ and $\gamma''$ phases

The strengthening phases in Alloy 718 are $\gamma'$ and $\gamma''$, which precipitate coherently in the matrix. They confer strength by three main mechanisms, namely – modulus hardening, coherency hardening and order hardening [21]. In peak aged condition of Alloy 718, the total volume fraction of $\gamma'$ and $\gamma''$ is $\sim 20\%$, with $\gamma'$ to $\gamma''$ volume fraction ratio of $\sim 3:1$ [33], [34]. The lattice mismatch of $\gamma''$ is 2.86% while that of $\gamma'$ is only 0.407% due to which $\gamma''$ confers a higher coherency strain in the matrix. It has been shown that, in fact, this coherency hardening contributes more to the strength of Alloy 718 [33], [35]. In addition, the hardening effect of $\gamma''$ is directly proportional to its size [36]. So, Alloy 718 mainly derives its strength from $\gamma''$ due to both its higher volume fraction and higher coherency hardening. The morphology of $\gamma'$ and $\gamma''$, their relative amounts, their respective phase stabilities can be modified by altering the content of Al+Ti, Al/Ti ratio and (Al+Ti)/Nb ratio [37]–[39].

### 2.2.3 $\delta$ phase

The $\delta$ phase is an equilibrium phase and is incoherent with the matrix. It does not contribute to the strength and is generally associated with loss of ductility when present in excess amounts [21], [22]. When present in small quantities along the grain boundaries, it is considered beneficial but excessive amounts in intragranular form is detrimental to the properties. The use of $\delta$ phase precipitation to pin the grain boundaries during forging is well known [28], [40]; it also has beneficial effect on notch rupture strength [41], [42]. Analyzing the $\delta$ phase morphology by extraction technique has shown that the needle like or globular morphology observed on metallographic sections are both plate like [43]. In general, $\delta$ phase forms by direct precipitation from the supersaturated matrix at temperatures above $\sim 900 \, ^\circ C$ and by transformation of $\gamma''$ in the temperature range $\sim 700 \, ^\circ C$ to $\sim 900 \, ^\circ C$. The $\delta$ phase particles are surrounded by a $\gamma''$ denuded zone, but $\gamma'$ phase is present [44].

### 2.2.4 Laves phase

Laves phase is a brittle, low melting phase that forms as a result of segregation, although it can also form in the solid state. Owing to the brittle nature, it acts as a crack initiation site and provides an easy crack propagation path. Due to the low melting nature, it affects weldability due to heat affected zone liquation cracking [23]. It is rich in Nb, Mo and Si relative to the matrix and is generally accepted to
be of in the chemical form \((\text{Ni,Fe,Cr})_2(\text{Nb,Mo,Ti})\) [45]. Due to the high Nb requirement for its formation, Laves phases usually occurs in the heavily segregated interdendritic regions and could be observed as islands in cast Alloy 718 [28]. It can be solutioned by a suitable homogenization heat treatment, but complete homogenization of Nb is impractical. If the homogenization treatment is not properly carried out, Laves phase can be carried over to wrought products as well [28], [45]. The cooling rate during solidification affects the segregation of Nb, along with it, the formation of Laves phase. With increasing cooling rates, segregation of Nb decreased and a finer distribution of Laves phase occurs. At very high cooling rates, such as those obtained in levitation casting, the segregation was so low that Laves phase does not form [32].

### 2.2.5 NbC

The only carbide phase present in Alloy 718 is the primary NbC. The carbide often has Ti substituting for Nb due to which it is denoted \((\text{Nb,Ti})\)C; it has a lattice parameter between NbC and TiC, but closer to NbC [46]. NbC particles are often heterogeneously nucleated on the TiN particles due to which they are called as carbonitrides [47], [48]; the TiN particles themselves often have an alumina core on which they have nucleated [48]. NbC, being a primary carbide, forms directly from the melt and is characterized by non-uniform distribution both at intragranular sites and at grain boundaries. Below 1265 °C, very few new carbides nucleate and instead growth of existing particles proceeds [32].

### 2.3 Heat treatments for Alloy 718

Heat treatments, in general, rely on solid-state transformations. Heat treatments are routinely carried out for Alloy 718 to achieve suitable mechanical properties; these treatments involve multiple steps, each of which serve a different purpose. Homogenisation treatment resets the compositional segregation incurred during the solidification. It is primarily carried out to dissolve the Laves phase in castings. Solution treatment dissolves the precipitates from the former processing steps and supersaturates the \(\gamma\) matrix. It is usually carried out at a temperature higher than the \(\gamma', \gamma''\) solvus; the temperature may be chosen to be higher than \(\delta\) solvus to dissolve the \(\delta\) phase completely or below it to precipitate \(\delta\) phase only at grain boundaries. Ageing treatment, usually a two-step process for Alloy 718, is carried out to achieve uniform precipitation of \(\gamma', \gamma''\) phases from the supersaturated \(\gamma\) matrix. HIP is carried out to eliminate porosity. It is usually performed before the homogenization treatment for castings; wrought material, which is usually pore-free, does not require a HIP treatment. Depending on whether the material is in cast or wrought form the heat treatment protocols vary and the ones most commonly used in aerospace applications are listed in Table 3.
Table 3. Commonly used standard heat treatment protocols for Alloy 718

<table>
<thead>
<tr>
<th>AMS Specification</th>
<th>Homogenisation treatment</th>
<th>Solution treatment</th>
<th>Ageing treatment</th>
</tr>
</thead>
<tbody>
<tr>
<td>AMS 5383 [49] (Cast material)</td>
<td>1093±14 °C for 1∼2h, followed by air cooling or faster cooling</td>
<td>954∼982±14 °C for not less than 1h, followed by air cooling or faster cooling</td>
<td>718±8 °C for 8±0.5h, furnace cool to 621±8 °C, hold at 621 °C for total precipitation time of 18h, followed by air cooling or faster cooling</td>
</tr>
<tr>
<td>AMS 5662 [30], AMS 5663 [50] (Wrought material)</td>
<td>-</td>
<td>941∼1010±14 °C for 1h (except not exceeding 1016 °C), followed by air cooling or faster cooling</td>
<td>718∼760±8 °C for 8h, furnace cool to 621±8 °C, hold at 621∼649±8 °C for total precipitation time of 18h, followed by air cooling or faster cooling</td>
</tr>
<tr>
<td>AMS 5664 [51] (Wrought material)</td>
<td>-</td>
<td>1066±14 °C (except not below 1038 °C) for 1∼2h, followed by air cooling or faster cooling</td>
<td>760±8 °C for 10±0.5h, furnace cool to 649±8 °C, hold at 649 °C for total precipitation time of 20h, followed by air cooling or faster cooling</td>
</tr>
</tbody>
</table>
2.4 Fatigue properties of Alloy 718

The components of an aerospace or a land turbine engine made with Alloy 718 are typically subjected to high temperatures and cyclic loading. Under such operating conditions, together with the static properties, fatigue properties are important. Low Cycle Fatigue (LCF), High Cycle Fatigue (HCF) and Fatigue Crack Growth Rate (FCGR) testing are some of the most common tests to evaluate the fatigue properties. Various microstructural features such as the grain size, defects, carbides and other non-metallic inclusions, the strengthening precipitates etc., affect these properties.

2.4.1 Effect of δ phase

In forgings that have complex geometric shapes, unlike simple shapes such as sheets and bars, have a forging flow pattern associated with them. Secondary phases such as carbides and δ phase in Alloy 718 follow the flow pattern and may be retained following a direct ageing treatment. Such a preferential arrangement, known as flow induced arrangement, of δ phase results in the δ phase particles arrangement lines. The crack growth resistance is the highest when the crack front is, locally, perpendicular to the flow lines that have the δ phase particles [52]. The aspect ratio of the crack has also been shown to be affected by the same mechanism [53]. The δ phase distribution, when controlled by a suitable heat treatment like the “modified Merrick” treatment, to form serrated grain boundaries improves fatigue crack growth resistance especially at 650 °C [54]. Grain boundary serration suppresses grain boundary sliding and at the same time makes the crack growth path more torturous by deflecting the crack. However, contradicting results about the effect of δ phase distribution have also been reported [55]. For the same grain size, δ phase content variation from 0 to 29 % did not have any observable effect on the fatigue life at 650 °C for notched specimens.

2.4.2 Effect of carbides

The presence of carbides at the grain boundaries as discrete particles, instead of a carbide film, prevents grain boundary sliding at elevated temperatures. This has a beneficial effect on fatigue at the temperature domain (~650 °C) where there is significant fatigue-creep interaction. In-phase thermomechanical fatigue properties, also, show improvement with presence of a good distribution of grain boundary carbides [56], [57]. Carbides, sometimes, also have deleterious effects on fatigue properties. Such an effect is dependent on maximum carbide size, the frequency and location of large carbides and their tendency to cluster together. All
these factors are dependent on the melting practice. Typically, the larger carbide particles are of primary carbides – NbC in Alloy 718. NbCs of size greater than 15 µm when present at sub-surface locations (up to 20 µm below the surface) act as crack initiation sites [58], [59]. In addition, the tendency of carbides to crack and to initiate cracks is higher at low temperatures [21]. The reduced severity of carbides at high temperatures is related to change of hardness and ductility with increase in temperature. When carbide particles are present at the surface, at high temperatures in the presence of oxygen, there is a considerable oxidation of these carbides. Due to oxidation induced volume expansion or oxidation induced cracking, these carbides act as crack initiation sites [17]. Such an oxidation phenomenon counteracts the effect of surface finish. A good surface finish, in general, is beneficial for fatigue performance.

2.4.3 Effect of Boron

Addition of boron in the order of a few ppm to superalloys is known to have beneficial effect on high temperature properties. Unlike carbon, that forms carbides, beneficial effect of boron comes from its atomic state. Solubility limit of boron in the γ matrix in nickel superalloys is quite low and hence it segregates to the grain boundaries. Boron forms a co-segregated layer with nickel that facilitates transmission of slip from one grain to the other. This leads to an improvement in thermomechanical fatigue properties [57]. Diffusion of oxygen through the grain boundaries has been known to cause intergranular cracking [60]. However, boron ties up the vacancies at grain boundaries that reduces the number of diffusion paths available for foreign elements. This effect of boron, at high temperatures, hinders the diffusion of oxygen through the grain boundaries and thereby increases the cohesion strength of the grain boundaries. As a result, the cracks grow by transgranular mode, which has a lower crack growth rate compared to intergranular cracking mode [61]. At high concentrations (~100 ppm), boron forms complex molybdenum and chromium rich boride stringers and clusters. The continuous stringers act as preferred crack initiation sites and lead to reduced thermomechanical fatigue life [56]. Therefore, boron concentration is usually limited to 60 ppm for retaining its beneficial effects, while avoiding problems such as liquation and formation of borides.

2.4.4 Effect of grain size

Fatigue crack growth resistance is higher for coarse grain material while resistance for crack initiation is higher in fine-grained material [21], [62], [63]. However, if large grains are present at random, they serve as crack initiation sites as well as affect crack growth rates negatively [64]. It has been observed that irrespective of the size of the strengthening precipitates, whether in under-aged or over-aged condition, crack growth rate is higher in fine-grained material compared to a
coarse-grained one [65]. Crack initiation at twin boundaries is also common; especially within grains at the high end of the size distribution. Stress concentration at such twin boundaries is high due to the specific crystal symmetry across these boundaries [66]. The elastic incompatibility leads to stress concentration and the twin boundaries owing to their lower slip transition ability are more prone to crack initiation compared to grain boundaries.

The fatigue crack growth in metallic materials occur in two stages – stage I where the fracture surface is faceted and stage II where the fracture surface is relatively flat with formation of striations in many alloys [67]. When the crack tip plasticity is smaller than the characteristic microstructural dimension, for example, grain size, then the crack grows by single shear mode in the direction of the primary slip system called stage I growth. Such an effect of the microstructural size scale is observed in a number of alloy systems and has been summarized by Suresh [67]. In stage II growth, the crack grows in a plane perpendicular to the loading direction, by duplex slip when the crack tip plasticity encompasses a few grains. Stage I crack growth mode is associated with crack propagation in the near-threshold regime while, stage II crack growth mode is associated with the Paris regime of crack propagation. In the near-threshold domain of crack growth for Alloy 718, transgranular crystallographic fracture mechanism has been observed to have a faceted macroscopic appearance of the fracture surface [68], [69].
3 Additive Manufacturing of Alloy 718

This chapter presents a review of additive manufacturing of Alloy 718. A general background to additive manufacturing is given, including the two AM processes investigated in this thesis – EBM and SLM. In addition, manufacturing Alloy 718 using these two processes, the common microstructural features and the associated properties are presented.

3.1 Additive Manufacturing

Additive manufacturing, commonly referred to as 3D printing, has transformed significantly in the last 3 decades, since the development of the technology for printing polymers [70]. The most significant change, and the reason for the hype in the recent years, is that AM has moved from being used for just prototyping to being used for commercial production of parts. Since the 1980s, numerous new AM processes have been developed for manufacturing with different material classes; there are equally unique number of names that were given for these new processes. The ASTM F42 Committee on AM issued standard process terminology and categorized the processes [71]. Of the seven categories, four pertain to metal AM – Powder bed fusing, Direct Energy Deposition, Binder jetting and Sheet lamination. In this thesis, PBF processes are utilized and further details regarding the same are given in later sections.

3.1.1 AM process steps

In general, all the additive manufacturing processes involve building a specific geometry layer-by-layer. The most common steps in AM processes are listed below, which is a simplified form described by Gibson [2].

1. Generation of a Computer Aided Design (CAD) file to describe the geometry of the part to be built, either using a software program or by laser/optical scanning.
2. This CAD file is converted to a .stl file, which almost all AM machines accept. This file is then sliced into a stack of 2D profiles, process parameters are defined, support structures are generated and a build file is created using the manufacturer’s specific program.
3. The build file is transferred to the AM machine and the machine is setup for the build process. The building process takes place by addition of
material corresponding to the geometry of each of the slices i.e., the 2D profile, on top of one another until the entire part is built.

4. The built part is removed and post-processed. The method and the extent of post-processing vary depending on the AM process that is used. Common post-processing steps for metal AM include removal of support structures, surface treatment, HIP and heat treatment.

### 3.1.2 Benefits of AM

AM offers unique capabilities such as hierarchical complexity, shape complexity, material complexity and functional complexity, all of which require a new design thinking [2]. For example, the constraints involved in conventional manufacturing processes do not apply to AM; instead, new constraints such as mode and resolution of material delivery, required post-processing, heat dissipation direction etc. are important [72].

AM can produce complex shapes in near-net-shape, without the use of dies or molds. This leads to shorter lead-time, less material waste and economic production of one-off or low-volume parts. It is possible to achieve design changes and customizations in a shorter duration. Design and manufacture of shapes such as freeform structures, lattice structures and topology-optimized structures for lightweight design are relatively easy. It is possible to integrate parts and reduce the number of assemblies; build functionally complex parts, for instance, with complex cooling channels etc. AM processes also offer possibilities for repair and remanufacture of end-of-life parts.

### 3.1.3 Limitations of AM

One of the most common limitations of AM, particularly PBF, is the low deposition rate. PBF processes are limited by the small build volume (typically less than 1 m$^3$), however, a new generation of machines with larger build volumes, multiple energy sources are being introduced.

Another common problem is the poor surface finish and dimensional accuracy. In addition, defects such as porosities, lack of fusion and inclusions that affect the mechanical properties of the structures are a major concern.

Other challenges with AM is the repeatability and reliability in the production process. The lack of an efficient feedback control to detect anomalies in-process and make corrective measures, in many AM systems, needs to be addressed to enable standardization. Equipment manufacturers have started introducing monitoring systems for process verification and validation that are available commercially, but efficient feedback systems need to be developed.
3.2 Powder bed fusion process

Powder bed fusion is a sub-class of metal AM, which is constituted by SLM and EBM processes. In PBF processes, metal parts are built layer-wise, by selective melting of a layer of pre-alloyed powder. In general, the sequence of operations in PBF include spreading a layer of powder on top of the build plate/previousl solidified layer, selectively melting the geometry in the newly spread powder layer, lowering the build platform by one layer thickness. This sequence is iterated until the complete part is built. During this melting process, re-melting of previously solidified layers occurs, which ensures proper fusion between all the layers [70].

In both the processes, selective melting of the desired geometry, in each layer, is performed by sequentially melting the contour and the hatch. The order of melting of the contour and the hatch regions, the number of contours and the associated parameters is often a user choice. Figure 1 is a schematic representation of the contour and the hatch regions, in each layer, for the two processes used in this thesis.

![Figure 1. Schematic representation of contour and hatch regions in every layer for EBM and SLM standard themes used in this thesis. Note that the sketch is not to scale.](image)

The principle of part generation is similar in both the processes; however, there is a significant difference in the hardware setup. The focused energy beam that melts the powders is an electron beam in the EBM process, while it is a laser beam in the SLM process. The electron beam in EBM is deflected with magnetic coils, so the beam velocity is much higher than in SLM, in which physical mirrors control the laser path. EBM operates in controlled vacuum, whereas SLM requires inert atmosphere. Other major differences between the two processes include size fraction of the powders used – larger powders are used in EBM than in SLM, pre-heating temperature used – higher temperature is used in EBM than in SLM so that the powders are sintered before melting, powder recovery methods adapted – utilized powders are in sintered cake form in EBM process but exist as loose particles in SLM. Numerous other differences such as type of powder dispensing system, differences in melting themes/scan strategies, requirements on support
structures and the possibility of stacking, requirement of a stress relief treatment, smallest feature size that can be built, differences in build rate etc., exist as well.

### 3.2.1 Microstructure and surface roughness

The resulting microstructure, residual stress state in the as-manufactured/as-built condition from the PBF processes is dependent on the scan strategy and the melting parameters used [16]–[18], [73]–[75]. Further information about the microstructure of PBF manufactured Alloy 718 is presented in the later sections.

Apart from the microstructure, which includes the phases and defects, the as-built surface roughness resulting from the PBF processes is of interest. The as-built surface roughness could be a limiting factor for mechanical performance, especially in fatigue critical components. For characterization of the roughness, numerous techniques such as metallography, µCT, contact and non-contact roughness measurement methods have been investigated; in general, it is observed that the areal (non-contact) methods are better suited for the purpose [76], [77]. The prospect of using µCT is of particular interest because it can be used to characterize the roughness of internal features, while performing the non-destructive testing (NDT) to detect defects. However, a lot of research is required in the area before a standard operating procedure can be reached, since parameters such as magnification, filament life, algorithm for feature extraction have shown to influence the measured surface roughness [78]–[80].

In general, the surface roughness of PBF manufactured parts are dependent on the layer thickness, part orientation/inclination of the surface, size fraction of the powder used, spot size, etc. Therefore, the SLM manufactured material generally has smoother surface compared to the EBM manufactured surface [70], [77], [81]. The effect of using finer powders and thinner layers on surface roughness have been investigated for EBM built Ti-64 [77], and it was found that the roughness decreases for both cases; but the reduction in roughness was significantly larger when using finer powders for the same layer thickness than it was when using thinner layers for the same powder size.

### 3.3 EBM of Alloy 718

In the Arcam A2X machine, numerous process parameters can be varied, such as scanning strategy, beam current, beam velocity, focus offset, line offset etc. In addition to these parameters, there are multiple compensation functions such as thickness function, turning point function which are coupled to the heat model algorithm to ensure a good build characteristic [82]. Such a complex interaction between all the direct process parameter settings and alterations by the compensation functions makes it difficult to define the local solidification conditions such
as the thermal gradient and the solidification velocity or even the melt pool shape precisely.

The typical microstructure in the hatch region, with the *raster scan* strategy of melting, is long columnar γ grains with strong <100> texture along the building direction [13], [83]–[85]. Further, due to the *spot melting* strategy, the microstructure in the contour region is different from that of the hatch region. The contour region has a mix of curved columnar grains, fine equiaxed grains and some large grains [13]. In addition, it has also been demonstrated that it is possible to alter the morphology of grains, such as to vary the primary dendrite arm spacing and to attain equiaxed grain structure, by carefully modifying the scanning strategy for melting [14], [16], [17], [85], [86]. A gradient in the microstructure, due to the thermal history, yields different properties at different heights of a specimen in the as-built condition; this gradient disappears after thermal post-treatment [12]. The distribution of δ phase in an as-built part is dependent not just on the processing temperature, but is affected by other parts that are co-built during the manufacturing process [87], [88].

Anisotropic tensile properties have been reported for EBM built Alloy 718 material, in terms of strength, modulus and ductility [11]–[13], [84], [89]. In general, yield strength, ultimate tensile strength and ductility were found to be higher along the build direction; whereas, Young’s modulus was higher perpendicular to the build direction [13], [89]. Further, the equiaxed microstructure has tensile strength that is between the two normal directions [17]. The equiaxed microstructure in EBM built Alloy 718 shows isotropic tensile behaviour at both 20 °C and 650 °C, but it shows anisotropic behaviour under creep loading. The time to rupture is shorter perpendicular to the build direction than along the build direction. The columnar microstructure has better creep properties than the equiaxed microstructure; it is anisotropic with the longest time to rupture along the build direction [90].

The common defects found in EBM built Alloy 718 are gas pores, shrinkage pores and lack of fusions (LoF) [11], [91]. Disruption of epitaxial growth of the columnar grains structure was observed above the LoF sites. There were equiaxed grain clusters associated with such disruptions, but only up to a few micrometers, after which the equiaxed grains serve for epitaxial growth [92]. Another, less reported issue is in-process formation of aluminium oxide during AM. Helmer [89] reported this phenomenon of formation of a film of aluminium oxide during solidification and attributed it to high oxygen content of the powder as well as change in partial pressure of the build chamber due to desorption of gases from the powder particles during manufacturing. He observed these oxides to act as crack initiation sites in high cycle fatigue tested samples.
3.4 SLM of Alloy 718

The SLM process has numerous process parameters, similar to the EBM process, and these often vary between the different equipment manufacturers. The machine parameters such as scanning strategy, beam velocity, beam power, stripe distance, layer thickness etc., control the size of the weld pool and the intrinsic solidification parameters, such as local thermal gradient and solidification velocity, which determine the type of microstructure that forms. Despite the variety of settings across different equipment manufacturers, the common as-built microstructure is reported to have weakly textured, elongated grains with cellular sub-grain morphology [93]–[99].

The sub-grains generally have a columnar or mosaic pattern based on how they are aligned within a single grain, but the grain itself has a single orientation with respect to the build direction [95]. It is, also, possible to generate a microstructure with a strong <100> texture by changing the laser power, instead of the commonly reported weakly textured microstructure [18], [19]. The solidification cellular sub-structures is also associated with dislocation networks at the cell boundaries [95], [97], [98], [100]. The precipitation of Laves phase in these intercellular regions in the as-built condition, due to microsegregation of Nb, is also a commonly observed phenomenon.

In general, the SLM built Alloy 718 material shows anisotropic mechanical properties – particularly, the tensile strength is higher perpendicular to the building direction than along the building direction and the opposite trend is observed for elongation at break [93], [95], [99], [101]–[103]. The material in as-built condition is softer and attains peak strength after suitable thermal post-treatments [95], [97], [100], [101], [104], [105]. In addition, different microstructures, obtained by using different process parameters, have different properties in both as-built and thermally post-treated conditions [19]. The presence of the cellular substructure is favourable for creep properties [94].

3.5 Fatigue properties of PBF AM Alloy 718

Only a limited number of investigations on fatigue properties of PFB AM Alloy 718 are available [96], [106]–[109]. The equiaxed microstructure of EBM built Alloy 718 has fatigue properties that lies between the two directions of the columnar microstructure, under low cycle fatigue loading conditions at 650 °C. It, however, has higher cyclic plastic strain accumulation when loaded parallel to the build direction than when load is perpendicular to the building direction [106].

In case of the SLM manufactured material, the dislocation substructures are dissolved due to HIP treatment; annealing twins also form after HIP. At low cyclic strain amplitudes, the non-HIPed material that retains the dislocation
substructure has better fatigue performance. This is because the dislocation substructures interact with the dislocations that form during cyclic deformation. At high strain amplitudes, however, the poorer fatigue performance of HIPed material is related to presence of annealing twins, which act as preferred crack initiation site [96].

The surface roughness of as-built SLM manufactured material is different depending on the inclination of the surface with respect to the building direction. This variation in roughness has an influence on the fatigue performance as the rough surface provides sites for crack initiation. When the surface roughness is higher, the fatigue life is lower [108]. Similarly, the SLM manufactured material with machined surfaces has better notched fatigue performance than the ones with as-built surface; the machined SLM manufactured material has similar performance to that of wrought material [109]. The fatigue crack growth threshold, however, is lower for SLM manufactured material compared to wrought material [107].
4 Materials and Methods

In this chapter, the materials and the experimental methods used in this research thesis are presented. It includes details pertaining to the manufacturing of the specimens, surface and thermal post-treatments, mechanical testing and material characterisation. For detailed information pertaining to the content of this chapter, the readers are advised to refer to the appended papers – *Paper A, Paper B* and *Paper C*.

4.1 Materials

In the present work, material manufactured using the two different powder bed fusion AM processes is investigated.

4.1.1 EBM Alloy 718

The specimens were manufactured using an Arcam A2X EBM machine with EBM control software. Arcam supplied the powder that had nominal composition of Alloy 718. It was plasma atomized powder with size ranging from 40-105 µm. The Arcam standard theme for Alloy 718 was used to build the specimens. The layer thickness utilized was 75 µm and the preheat temperature was 1000 °C. After the build completion and the recommended powder recovery process, the specimens were sectioned off from the base plate.

4.1.2 SLM Alloy 718

EOS M290 machine together with EOSPRINT software was used to manufacture specimens by the SLM process. Gas atomized powder with an average size of 40 µm and nominal size less than 65 µm was used to build the specimens. A layer thickness of 40 µm together with the EOS standard theme for Alloy 718 were used. The specimens were cut off from the base plate after the recommended powder recovery process. Note that the specimens were cut off in the as-built condition without any stress relief treatment.

4.1.3 Thermal post-treatment

One batch of the specimens, manufactured by both EBM and SLM, were HIP:ed in a Quintus QIH21 HIP furnace at 1200 °C and 120 MPa for 4 hours followed by uniform rapid quenching (URQ). All the specimens, including the ones that
were not HIP:ed, were heat treated in accordance to AMS 5664 in a vacuum furnace. It involved a solution treatment at 1066 °C for 1 hour followed by argon quenching to 65 °C, first ageing treatment at 760 °C for 10 hours followed by furnace cooling to 649 °C at 55 °C/hour and second ageing treatment at 649 °C for 8 hours and then air cooling to room temperature.

4.2 Mechanical testing

To address the different Research Questions formulated, mechanical tests for evaluating hardness, tensile properties and fatigue properties were performed. Though different mechanical properties were evaluated, emphasis was on fatigue properties as stated earlier in the Scope of research.

4.2.1 Hardness test

Vickers hardness (HV$_1$) was measured using HMV Microhardness Shimadzu machine. The tests were performed on cross sections perpendicular to the build direction, in as-polished samples, by following the ASTM E 384 standard [110]. For each sample, no less than 15 indents were performed.

4.2.2 Tensile test

Tensile tests were conducted on machined specimens (the “raw” as-built surface was removed completely) along the building direction, both in as-built condition and after thermal post-treatments. The tests were conducted in ambient conditions at a strain rate of $10^{-3}$/s. For additional information about specimen geometry and testing conditions, please refer to the appended Paper C. No less than 3 specimens were tested for each test condition.

4.2.3 Fatigue test

Specimens for fatigue testing were prepared by machining and low stress grinding after the thermal post-treatments. One group of specimens were machined on all four sides, while another group retained the “raw” as-built surface on one of the sides. A summary of the specimen conditions is presented in Table 4.

Four-point bending fatigue tests were performed in load controlled constant amplitude mode on a servo-hydraulic test machine. The tests were performed at room temperature, stress ratio of R=0.1 and 20 Hz frequency. The EBM specimens were tested at four stress ranges while the SLM specimens were tested at two stress ranges. For further details regarding the specimens and testing conditions, please refer to the appended papers – Paper A and Paper C.
Table 4. Specimen types for fatigue testing.

<table>
<thead>
<tr>
<th>AM process</th>
<th>Thermal post-treatment</th>
<th>Surface post-treatment</th>
<th>Cross-section (mm²)</th>
<th>No. of specimens</th>
</tr>
</thead>
<tbody>
<tr>
<td>EBM</td>
<td>HIP+STA</td>
<td>As-built</td>
<td>10 x 10</td>
<td>9</td>
</tr>
<tr>
<td>EBM</td>
<td>HIP+STA</td>
<td>Machined</td>
<td>10 x 10</td>
<td>9</td>
</tr>
<tr>
<td>EBM</td>
<td>HIP+STA</td>
<td>Machined</td>
<td>6 x 6</td>
<td>6</td>
</tr>
<tr>
<td>EBM</td>
<td>STA</td>
<td>As-built</td>
<td>10 x 10</td>
<td>9</td>
</tr>
<tr>
<td>EBM</td>
<td>STA</td>
<td>Machined</td>
<td>10 x 10</td>
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<td>EBM</td>
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<td>Machined</td>
<td>6 x 6</td>
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<tr>
<td>SLM</td>
<td>HIP+STA</td>
<td>As-built</td>
<td>10 x 10</td>
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<tr>
<td>SLM</td>
<td>HIP+STA</td>
<td>Machined</td>
<td>10 x 10</td>
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<tr>
<td>SLM</td>
<td>STA</td>
<td>As-built</td>
<td>10 x 10</td>
<td>6</td>
</tr>
<tr>
<td>SLM</td>
<td>STA</td>
<td>Machined</td>
<td>10 x 10</td>
<td>6</td>
</tr>
</tbody>
</table>

4.3 Characterization methods

The surface roughness and the microstructure of the material utilized in the thesis were characterized using the following methods.

4.3.1 Surface roughness measurement

Areal surface roughness parameters $S_a$ (average mean deviation in the sampled area), $S_v$ (maximum valley depth in the sampled area) and $S_z$ (maximum total deviation in the sampled area) were measured by focus variation microscopy technique. An Alicona InfiniteFocusSL microscope fitted with a 20x objective was utilized for the purpose. An area of 2.5 mm by 2.5 mm was imaged for each measurement and a total of 6 measurements were obtained for each surface condition from different specimens.

4.3.2 Metallography

4.3.2.1 Sample preparation

Samples for metallographic analysis were extracted, from selected specimens, from the top, bottom and the midsection. During the machining of the thermally post-treated specimens, the identifiers for the top and bottom of the specimens were not transferred by mistake; so, these are referred to as “end 1” and “end 2” henceforth. Samples were cut using a Struers Secotom 10 precision cutting machine and were mounted in conductive epoxy using a Buehler SimpliMet 3000 automatic mounting press. Samples were ground and polished by standard metallographic procedures using a Buehler EcoMet 300 Pro semi-automatic machine.
fitted with AutoMet 300 power head. Etching was performed either by immersion in Beraha III reagent for ~20 seconds or electrolytically with oxalic acid at 3V for ~5 seconds.

4.3.2.2 Microscopy

The microstructural evaluation and characterization were performed using optical microscopes – either Olympus BX60M or Zeiss AX10. The types of defects present, and their amount and distribution were analysed in both as-polished and etched conditions at suitable magnifications. Image analysis technique using Fiji software was utilized to determine the volume fraction of defects present in the specimens [111]. The ASTM E 1245 standard was followed for image analysis to choose an appropriate magnification, thresholding technique and number of image fields [112]. Further, high magnification analysis of the microstructure was performed using scanning electron microscope (SEM) – either with Zeiss EVO 50 SEM or with Hitachi SU70 FEG SEM; both SEMs had energy dispersive X-ray spectroscopy (EDS) detector from Oxford Instruments together with INCA software for chemical analysis. Texture analysis was performed in Hitachi SEM which had electron backscatter detector (EBSD) from Oxford Instruments together with HKL Channel 5 software. A scanning step size of 1-2 µm was used during the analysis depending on the grain size.

4.3.3 Fractography

After the mechanical testing (tensile and fatigue), the fracture surfaces of the ruptured specimens were analysed using an Olympus SZX9 stereomicroscope. Further fractographic analysis was performed in a Zeiss EVO 50 SEM using secondary electron (SE) imaging. Prior to the SEM investigation, the specimens were immersed in ethanol and cleaned using an ultrasonic cleaner. Initially, one of the two fractured surfaces was investigated and if deemed necessary, the other one was evaluated. Further, in case of fatigue tested specimens, crack path investigation was performed on metallographic sections extracted perpendicular to the fracture surface.
5 Summary of Papers

This chapter provides a summary of the three appended papers. This summary and the papers present the major findings from the conducted research.

Paper A

Influence of defects and as-built surface roughness on fatigue properties of additively manufactured Alloy 718

This paper presents various observations regarding how the fatigue properties of PBF AM Alloy 718 material is affected by the defects – the different types of defects and their distribution. In addition, the influence of the as-built surface roughness on fatigue properties is also discussed. This paper addresses the Research Questions 1(i) and 2.

In the metallographic investigations, three major types of defects were identified, namely – shrinkage porosity, lack of fusion (LoF) and spherical porosity (pores). All three types of defects were present in the EBM manufactured material, while no shrinkage porosity was found in the SLM manufactured material. In the SLM manufactured material, the defects were randomly distributed; whereas, in the EBM manufactured material, the distribution was not random and a correlation to the processing parameters existed. For example, shrinkage porosity was present mainly in the hatch region, LoF defects were concentrated in the contour region and the hatch-contour interface.

In both the EBM and the SLM manufactured material, some of the LoFs and pores were filled with aluminium oxide. In addition, some titanium nitride particles were found precipitated on these oxides in the EBM manufactured material. The presence of oxides prevented complete closure and healing of defects during HIP treatment. These LoF defects adversely affected the fatigue properties in the EBM manufactured material. These defects, in the contour region, were not completely machined off; therefore, machining improved the fatigue life of EBM manufactured material only marginally compared to when the as-built surface was retained. The SLM manufactured material had approximately an order of magnitude higher fatigue life compared to the EBM manufactured material. This is related to the difference in the volume fraction of defects and their random distribution.
The as-built surface roughness, in both the processes, resulted in a lower scatter in fatigue life because of multiple crack initiations from the valley-like features of the as-built surface. However, the fatigue life of specimens in the as-built surface condition was lower than the ones in the machined condition, which had fewer crack initiation sites (at least an order of magnitude fewer). HIP+STA treated specimens, in both the surface conditions, showed an improvement fatigue life due to closure/partial closure of defects.

**Paper B**

*Microstructural influence on fatigue crack propagation during high cycle fatigue testing of additively manufactured Alloy 718*

This paper summarizes the observed microstructural differences between the EBM and the SLM manufactured Alloy 718. The effect of thermal post-treatments on the microstructure and in turn how it influences the fatigue crack propagation behaviour are discussed. This paper answers the Research Questions 1(ii) and 1(iii).

The microstructure, in terms of morphology and texture of the grains, after STA treatment is similar to the typical as-built microstructure. There is a significant difference in the microstructure between the hatch and the contour regions in the EBM manufactured Alloy 718; however, there is no such, significant difference for the SLM manufactured material. The EBM manufactured material has columnar, <100> textured microstructure in the hatch region. In the contour region, there are curved fine columnar grains, fine equiaxed grains and some large grains. The SLM manufactured material has elongated grains, shorter and less textured compared to the EBM manufactured material, with cellular substructures. After HIP+STA treatment, the grains are coarsened in the contour region of EBM manufactured material, while in the hatch region there is no significant grain growth. The grain growth is uniform throughout in the SLM manufactured material, along with dissolution of the cellular substructures. The microstructure is similar at different heights within the specimens, for both EBM and SLM, in both thermal post-treated conditions.

The fatigue crack propagation is transgranular in EBM and SLM manufactured material, both in STA condition and in HIP+STA condition. The fracture surface has a faceted appearance corresponding to the regions with coarse grains present in HIP+STA condition. This is related to the single shear near-threshold crack growth behaviour in these coarse-grained regions.
Paper C

Additive Manufacturing of Alloy 718 via Electron Beam Melting: Effect of Post-treatment on the Microstructure and Mechanical Properties

This paper presents a summary of microstructure evolution of EBM manufactured Alloy 718 during thermal post-processing and the associated mechanical properties. In addition, it also presents the bulk fatigue properties evaluated by removing the contour region completely. Research Questions 1(i), 1(ii) and 1(iii) are addressed in this paper.

In the as-built condition, the grains in the hatch region are <100> textured and columnar, which is typical in EBM manufacturing. Strings of vertically aligned carbides in the inter-dendritic regions are observed. There are occasionally few grain boundaries that have needle like δ phase precipitates. There are rows of shrinkage porosities along the horizontal direction (in the plane of the layers). The contour region is composed of curved columnar grains, fine equiaxed grains and some random large grains in the overlap between the three contours. Due to these different types of grains, the contour is not as textured as the hatch region. In the transition (hatch-contour interface) region between the contour and the hatch, the grains are curved at a small angle towards the building direction. The contour region and the hatch-contour interface have a lot of LoF defects, which often have partially melted particles and oxides.

In STA condition, several microstructural aspects are similar to that of the as-built condition – the morphology of the grains, carbides and defects are unaffected by STA. The δ phase is present in a smaller size fraction, possibly because the solution treatment temperature is not high enough to dissolve it completely. In HIP+STA condition, the grains in the contour region are coarsened, but the grains in the hatch are not affected. In addition, the shrinkage porosities and most LoFs are all completely healed. However, the oxide filled defects are not healed which is apparent from partially healed LoF defects.

Tensile ductility is adversely affected by both shrinkage porosity and LoF defects. The HIP+STA treated material exhibited the best tensile properties. The strong <100> texture of EBM manufactured material leads to a lower yield strength along the building direction. Machining off the contour and the hatch-contour interface region completely (which also removes most of the LoF defects) improved fatigue properties by more than an order of magnitude. The best fatigue properties were observed for HIP+STA treated material without the contour and interface region. In addition, the fatigue fracture surfaces of specimens without contour and interface region in HIP+STA condition did not show any faceted behaviour. In the specimens with the as-built surface (with the whole contour and
interface region present) and machined specimens with some portion of contour and interface region, faceting behaviour is observed. This indicates that in addition to grain size, texture also influences the crack propagation behaviour.
6 Conclusions and Future work

The research conducted for this licentiate thesis is on EBM and SLM manufactured Alloy 718. The core objective is to understand how the various defects, phases and texture present in the microstructure affect the mechanical properties. The observations from the research have been reported as journal articles that are appended to this thesis; a summary of the articles is presented in the previous chapter. This final chapter presents a summary of the answers to the Research Questions formulated in the Introduction chapter and discusses the future work.

6.1 Conclusions

Defects:

The effect that the defects can have on the mechanical properties is related to the Research Question 1(i) and is addressed in Paper A and Paper C.

1. Defects were randomly distributed in the SLM manufactured material, whereas, in the EBM manufactured material, the distribution was dependent on the process parameters.
2. Defects have a detrimental effect on the mechanical properties – reduce the fatigue life and tensile ductility. However, there are number of influencing factors such as the type of defect, size, shape, location, distribution and nature of defect.
   a. LoF defects, in general, act as crack initiation site and reduce the fatigue life drastically under bending load conditions.
   b. Shrinkage porosity has detrimental effect on the crack propagation part of fatigue life.
3. HIP treatment improves fatigue life and tensile ductility. However, oxides present in the LoF defects prevents complete closure/healing after HIP. Among the EBM manufactured specimens, the ones with such partially healed sub-surface defects perform better in fatigue than the ones with surface breaching unhealed defects.
4. The improvement in tensile ductility of the EBM built material subjected to HIP treatment is related to closure of the shrinkage porosity and healing/partial healing of LoF defects.
5. Upon machining off the regions where the LoF defects are concentrated, particularly the contour region as well as the hatch-contour interface of
the EBM manufactured material, the fatigue life improved by more than an order of magnitude.

6. Defects in the SLM manufactured material affect the fatigue crack initiation under bending load condition only when present in surface breaching form. In addition, it was not possible to conclusively determine the effect of defects in the crack propagation phase.

Phases and Texture:

The effect of different phases and texture on the mechanical properties is related to the Research Questions 1(ii) and 1(iii). These are answered in Paper B and Paper C.

1. The strong <100> texture of EBM manufactured material leads to a lower Young’s modulus ~140 GPa along the building direction.
2. The hardness of the SLM and the EBM manufactured material in thermal post-treated condition is equivalent at different locations within the specimens. This correlates well with the microstructural homogeneity observed in microscopy.
3. The grain size of the γ matrix is dependent on thermal post-treatment and influences crack propagation. Grain coarsening is observed in HIP+STA condition in both SLM and EBM manufactured material; however, in EBM manufactured material, only the grains in the contour region are coarsened.
4. In the coarse grained regions, the fracture surfaces have a faceted appearance. This is related to the single shear mode fatigue crack growth in the near-threshold domain.
5. The fracture surfaces of the specimens which have only <100> textured grains do not show any faceting behaviour, indicating that texture also influences crack propagation.
6. The fatigue crack growth is transgranular and is unaffected by the precipitate phases such as NbC, TiN or δ phase.

Surface roughness:

The effect that the as-built surface roughness has on the fatigue properties is related to the Research Question 2 and is addressed in Paper A.

1. The surface roughness in as-built condition is three times higher for the EBM manufactured material compared to the SLM manufactured material. The higher surface roughness is a result of the relatively thicker layers and the coarser powder used in the EBM process.
2. The valley-like geometrical features in the as-built surface creates stress concentrations, which are crack initiating sites. The high frequency of crack
initiating features reduces the randomness in fatigue failure, leading to a lower scatter in fatigue life in the as-built condition compared to the machined condition.

3. The effect of surface roughness on fatigue life dominates the effect of defects i.e., when the as-built surface roughness is retained, post-treatments such as HIP do not improve fatigue properties significantly, even if the defects are healed.

6.2 Future work

Metal additive manufacturing is not just a hype anymore – it is already being successfully used to manufacture commercial parts. However, a lot of research is still needed, not just to mature the technology further but also to capitalize on its full potential.

In this research project, focus is on understanding how the mechanical properties of PBF AM Alloy 718 are affected by the microstructure. This licentiate thesis is a summary and reflection of the research conducted so far and serves as a basis for continued research towards the PhD degree. Fatigue properties of PBF Alloy 718 will remain as the primary research area in this future work – strain controlled low cycle fatigue tests both at room temperature and at elevated temperature will be conducted. In addition, the effect of texture will be evaluated in relation to both fatigue properties and tensile properties, and the influence of different thermal post-treatments on the mechanical properties will be investigated.

Further, to evaluate the 3D residual stresses both in the as-built condition and after thermal post-treatments, experiments using a neutron synchrotron have been planned. In the as-built condition, the focus will be on how the different process parameters affect the resulting residual stresses, if any, in the manufactured part. In the post-treated condition, the emphasis will be to understand how the residual stresses, if present, affects the recovery, recrystallization and grain growth behaviours.


2016.


Appended papers
Influence of defects and as-built surface roughness on fatigue properties of additively manufactured Alloy 718

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Influence of defects and as-built surface roughness on fatigue properties of additively manufactured Alloy 718

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\textbf{A R T I C L E   I N F O}

\textbf{Keywords:}
Additive manufacturing
Alloy 718
Fatigue
Surface roughness
Hot isostatic pressing

\textbf{A B S T R A C T}

Electron beam melting (EBM) and Selective Laser Melting (SLM) are powder bed based additive manufacturing (AM) processes. These, relatively new, processes offer advantages such as near net shaping, manufacturing complex geometries with a design space that was previously not accessible with conventional manufacturing processes, part consolidation to reduce number of assemblies, shorter time to market etc. The aerospace and gas turbine industries have shown interest in the EBM and the SLM processes to enable topology-optimized designs, parts with lattice structures and part consolidation. However, to realize such advantages, factors affecting the mechanical properties must be well understood – especially the fatigue properties. In the context of fatigue performance, apart from the effect of different phases in the material, the effect of defects in terms of both the amount and distribution and the effect of “rough” as-built surface must be studied in detail. Fatigue properties of Alloy 718, a Ni-Fe based superalloy widely used in the aerospace engines is investigated in this study. Four point bending fatigue tests have been performed at 20 Hz in room temperature at different stress ranges to compare the performance of the EBM and the SLM material to the wrought material. The experiment aims to assess the differences in fatigue properties between the two powder bed AM processes as well as assess the effect of two post-treatment methods namely – machining and hot isostatic pressing (HIP). Fractography and metallography have been performed to explain the observed properties. Both HIPing and machining improve the fatigue performance; however, a large scatter is observed for machined specimens. Fatigue properties of SLM material approach that of wrought material while in EBM material defects severely affect the fatigue life.

1. Introduction

Additive manufacturing (AM), which is a relatively new manufacturing technology, offers the possibility of manufacturing complex geometries, part integration and has even relatively eased the processing of some difficult to process materials. Some commercial products, particularly aerospace engine components, have already capitalized these possibilities offered by AM such as the fuel nozzle in the LEAP engine (complex design) \cite{1}, injection head of Ariane propulsion module (part integration) \cite{2} and titanium aluminate (TiAl) low-pressure turbine (LPT) blades (difficult to process material) \cite{3}. Aerospace industry is, however, known to be conservative and even though AM has gained high interest, extensive research into characterization of material properties, non-destructive inspection, process control etc. would be necessary to mature the AM technology to produce critical high-value components \cite{4}.

In powder bed AM, parts are built in a layer-by-layer fashion based on digitally sliced CAD data. The sequence of build operations are as follows:

1. spreading of powder, which has a certain size distribution, as a layer of predefined thickness,
2. melting the geometry on the powder layer corresponding to the slice data,
3. lowering the build table corresponding to the layer thickness and repetition of the sequence until the entire part height is built.

Though the basic idea of building is similar, the two processes have some differences as well. The heat source in the EBM process is an electron beam while it is a laser beam in the SLM process. The EBM process is carried out in a controlled vacuum environment at a high bed temperature and the powder layers are pre-sintered before the melting begins; in SLM, however, the powder bed is maintained at a lower temperature and the build chamber is filled with argon gas. The powder...
utilized in the SLM process is finer compared to the EBM process. While the EBM process produces parts with less residual stress in the as-built surface, the SLM process has relatively better roughness on the as-built surface.

One of the major advantages offered by powder bed AM processes is the complexity of geometries that can be produced, which could be expensive or sometimes impossible to manufacture by conventional methods. However, the as-built surface is rough and could negatively affect the fatigue properties. Machining or other methods of surface finishing of complex shapes could be difficult, expensive or impossible; but scaling down the complexity to ease such post-processing would undermine the geometric capabilities of the technology. Therefore, it is important to investigate the effect of the as-built rough surface on the mechanical properties. For characterization of the roughness, numerous techniques such as metallography, μCT, contact and non-contact roughness measurement methods have been investigated; in general, it is observed that the stylus profilometry is not suitable and that the areal methods are better suited for the purpose [5,6].

There are numerous process parameters that control the energy input for melting the powder such as beam power, spot size, distance between adjacent melt lines, beam path etc. A variety of strategies to control beam path during melting of a layer are available; these, so called, scan strategies also influence the microstructure [7], residual stress distribution [8] and the defect distribution [9] in the built parts. Hot isostatic pressing (HIP) has been evaluated to minimize defects from the AM process; however, remnant pores after HIP [10] and regrowth of pores during subsequent heat treatment of that were formerly closed by HIP [11,12] have been reported.

The effect of as-built surfaces and defects on fatigue properties have been investigated by a number of investigators for the AM built aerospace alloy Ti-64 [13-17]; effect of surface roughness on fatigue properties [15], effect of the surface roughness in presence of geometric notches [13] and effect of surface finishing methods on fatigue [14,17] have been investigated. An approach to use micro computed tomography (μCT) to characterize the as-built surface and use stress concentration factors to quantify the effect of surface roughness has also been attempted [16]. Such studies for Alloy 718, however, are quite limited [18]; in fact, there are only a few investigations related to fatigue of Alloy 718 produced by powder bed AM processes [19-21]. Fatigue properties of SLM built material has been investigated by conducting low cycle fatigue tests at room temperature [20] and by high cycle fatigue tests at 650 °C [21]. Similarly, low cycle fatigue testing at 550 °C has been performed on EBM built material [20].

Fatigue has contributed to failure of 55% of components in aerospace and is expected to remain a major concern for metallic parts [4]. In the context of application of metal AM parts in aerospace, understanding the effect of defect distribution and the as-built surface roughness on fatigue properties is crucial. The aim of this work, therefore, is to study the effect of defects and the as-built surface on high cycle fatigue properties of EBM and SLM built Alloy 718 material at room temperature. For the sake of brevity, the effect of microstructural features will be reported separately and the discussion in this paper is limited to the defects and the as-built surface.

2. Materials and methods

2.1. Test specimens

Test specimens were built in Alloy 718 using both EBM and SLM methods. EBM specimens were built in an Arcam A2X machine with powder having a size range of 40-105 µm. The standard Arcam parameters for building Alloy 718 and 75 µm layer thickness were utilized. SLM specimens were built in an EOS M290 machine with powder, which had an average size 40 µm, and nominal size less than 65 µm; standard EOS parameters for Alloy 718 and 40 µm layer thickness were used. The respective equipment manufacturer supplied the powder for each of the processes.

Following the build completion and powder recovery by recommended protocols, specimens were cut off from the base plates. No stress relief treatment was performed before cutting off the specimens. One batch of specimens from both EBM and SLM were HIPed in a Quintus QIH21 HIP furnace at 1200 °C and 1200 bar for 4 h followed by uniform rapid quenching (URQ). All the specimens, including the ones that were not HIPed, were heat treated in accordance to AMS 5664 in a vacuum furnace: solutioning treatment at 1066 °C for 1 h followed by a two-step ageing at 760 °C for 10 h, cooling to 649 °C at 55 °C/hour and held at 649 °C for 8 h.

The specimens were machined to a dimension of 10 mm by 10 mm square cross section and 80 mm length and the radii rounded off to 0.75 mm. The as-built specimens had 40 mm, in the mid-section, left without machining on one of the four sides. The as-built surfaces were perpendicular to the building direction for both EBM and SLM. All the machined surfaces were finished to $R_a$ of 0.2 µm by low stress grinding. Specimens with different surface conditions are shown in Fig. 1. The types of specimens w.r.t. processes, heat treatments and surface conditions utilized in this project are summarized in Table 1. Due to constraints in building all the SLM specimens in a single build, 20 SLM specimens were taken from one build and 4 specimens from another build; all EBM specimens were from a single build.

![Image](image-url)

**Table 1** Specimens in different combination of test factors utilized in the project.

<table>
<thead>
<tr>
<th>Process</th>
<th>Heat treatment</th>
<th>Surface</th>
<th>No of specimens</th>
</tr>
</thead>
<tbody>
<tr>
<td>EBM</td>
<td>HIP + HT</td>
<td>Machined</td>
<td>9</td>
</tr>
<tr>
<td>EBM</td>
<td>HIP + HT</td>
<td>As-built</td>
<td>9</td>
</tr>
<tr>
<td>EBM</td>
<td>HT</td>
<td>Machined</td>
<td>9</td>
</tr>
<tr>
<td>EBM</td>
<td>HT</td>
<td>As-built</td>
<td>9</td>
</tr>
<tr>
<td>SLM</td>
<td>HIP + HT</td>
<td>Machined</td>
<td>6</td>
</tr>
<tr>
<td>SLM</td>
<td>HIP + HT</td>
<td>As-built</td>
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<td>SLM</td>
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<tr>
<td>SLM</td>
<td>HT</td>
<td>As-built</td>
<td>6</td>
</tr>
</tbody>
</table>
2.2. Surface roughness measurement

The surface roughness of the specimens was investigated by focus variation microscopy technique. An Alicona Infinite Focus SL microscope fitted with a 20× objective was utilized for the purpose. An area of 2.5 mm by 2.5 mm was imaged for each measurement and a total of six measurements were obtained for each surface condition from different specimens. The area roughness parameters $S_a$ (average mean deviation in the sampled area), $S_v$ (maximum valley depth in the sampled area), and $S_z$ (maximum total deviation in the sampled area) were calculated.

2.3. Defects

To analyze the defects, 5 specimens were chosen randomly for each process and heat treatment combination. The types of defects present, and their distribution were analyzed in both as-polished and etched condition at various magnifications. Samples were extracted from the chosen specimens perpendicular to the build direction, mounted, ground and polished by standard metallographic procedure; etching was performed electrolytically with oxalic acid at 3 V for 5 s.

Image analysis technique using Fiji software was utilized to determine the volume fraction of defects present in the specimens. 16 images were acquired with an Olympus BX60M light optical microscope at 50× magnification on each sample in as-polished condition. The ASTM E 1245 standard was followed for image analysis to choose an appropriate magnification, thresholding technique and number of image fields. Chemical analysis of the defects was carried out in a Zeiss EVO 50 scanning electron microscope (SEM) operating at 20 kV with an Oxford XMax® 20 mm² energy dispersive x-ray spectroscopy (EDS) detector. It is evident from the figure that the surface is free from valleys and peaks such as in Fig. 3a and only a slight waviness is present.

The roughness parameters measured for the as-built surfaces and the machined surfaces are presented in Fig. 5. The EBM surface is approximately three times rougher than the SLM surface. The as-built surfaces from both the processes are approximately an order of magnitude rougher than the machined surfaces.

3.2. Defects

3.2.1. Metallography

In the metallographic investigations, three major types of defects were identified, namely – shrinkage porosity, lack of fusion (LoF) and spherical porosity (pores), see Fig. 6. LoFs and pores were present in both the SLM and the EBM material while shrinkage porosities were present only in the EBM material, mainly in the hatch region (which extends between ~ 2 mm from the outer surface and deeper); there was almost no shrinkage porosity in the contour region (which is up to ~ 2 mm deep from the outer surface). Several of the shrinkage porosities were aligned horizontally in a linear string pattern, perpendicular to the build direction. The distance between these strings along the build direction was ~ 350 µm, see Fig. 7. The spherical pores, in contrast, were homogenously distributed.

Two types of LoFs defects were identified – one with a wide separation (> 10 µm) between the two faces of the defect and another with narrow separation (< 10 µm) between the faces. LoF defects with wide separation were present only in the EBM material and were preferentially located within ~ 2 mm from the surface. These defects, sometimes, contained partially melted or completely unmelted powders. LoF defects with narrow separation were present in both the EBM and the SLM material; these were homogenously distributed. LoF defects were oriented perpendicular to the build direction and lay in the plane of the layers.

LoFs and pores occurred in two conditions – oxide-filled and empty. Oxide-filled defects were present in both the EBM and the SLM material. Frequently, the oxide-filled defects in the EBM material had a few particles of a different phase precipitated on the oxide. EDS analysis confirmed that the oxides were composed of aluminium and that the particles were titanium nitride.

Representative images of the types of LoFs and pores aforementioned are presented in a summarized form in Fig. 8. In both the EBM and the SLM material, empty and oxide-filled pores were present in HT as well as HIP + HT condition. However, the frequency of open pores in the EBM material in HIP + HT condition was relatively low. Oxide-filled LoFs were present in both the EBM and the SLM material irrespective of the heat treatment condition; in contrast, empty LoFs were found only in the EBM material in HT condition. Additionally, shrinkage porosities were not present in the EBM material in HIP + HT condition.

3.3. Results

3.3.1. Surface roughness

The surface morphology for the EBM and SLM specimens were different, as shown in Fig. 3. In EBM specimens, the surface had a rippled appearance with embedded particles; the ripples were perpendicular to the build direction. SLM specimens had embedded particles on the surface as well, while the rippling was almost absent, see Fig. 4. It is evident from the figure that the surface is free from valleys and peaks such as in Fig. 3a and only a slight waviness is present.

The roughness parameters measured for the as-built surfaces and the machined surfaces are presented in Fig. 5. The EBM surface is approximately three times rougher than the SLM surface. The as-built surfaces from both the processes are approximately an order of magnitude rougher than the machined surfaces.

Fig. 2. A representative sketch of the 4-point bending fatigue test set up. The open circles indicate the position of loading rollers and the shaded circles indicate the position of support rollers.

Fig. 3. The EBM surface is approximately three times rougher than the SLM surface. The as-built surfaces from both the processes are approximately an order of magnitude rougher than the machined surfaces.
image analysis, is presented in Fig. 9. The EBM and the SLM material are both ~ 99.95% dense in HIP+HT condition; the defects were lesser in HIP+HT condition than HT condition for both the EBM and the SLM material. The difference between the amount of defects between HT and HIP+HT condition is larger for the EBM material than the SLM material. The SLM material in HT condition had less defects than the EBM material in the same condition.

3.2.2. Fractography

In the fractographic investigations, two types of defects were most often identified – LoF and shrinkage porosity. In the EBM material, LoF defects were found at a majority of the fatigue crack initiation sites, while the shrinkage porosities were found in the crack propagation area and the final fracture area, see Fig. 10(a)–(c); the shrinkage porosities, however, were present only in HT condition. In the EBM material, 3 out of 36 specimens had main crack initiation at features other than a defect; only 1 out of these 3 specimens had no defect in the vicinity of the initiation area. In the SLM material, defects were found to initiate the failure for 3 out of the 24 tested specimens.

In the EBM material, the LoF defects were relatively large in both size and number. Some of these defects, as shown in the fractograph in Fig. 10(d), were larger than 750 µm, which was longer than the sizes observed in the metallographic investigations of cross sections. These defects were highly irregular in shape as well. In addition, the LoF defects in the EBM material were concentrated close to the periphery, which is the contour region. Often, a few of such defects occurred adjacent to one another as in Fig. 10(e). In both the HT and the HIP+HT condition, LoF defects with partially melted or unmelted particles were seen as shown in images aforementioned. In the HIP+HT condition, however, partially healed LoF defects were often found, see Fig. 10(e).
3.3. Hardness

The hardness values measured for materials in different heat treatment conditions are presented in Fig. 11. The difference in hardness between the different groups was within 15 HV. Hence, the data is only an indicative result of the effect of different processes and heat treatment combinations, which are explained in Section 4.3. The hardness in HIP + HT condition was higher compared to HT condition for the EBM material; whereas, for the SLM material, the hardness was higher in HT condition. Further, in HIP + HT condition, both the EBM and the SLM material had the same hardness; while in HT condition, SLM material was harder.

3.4. Fatigue

The result from the fatigue testing of both the EBM material and the

Fig. 8. Summary of different types of LoFs and pores. (a)-(d) EBM material. (e) & (f) SLM material. All images have been acquired in etched condition. (a) Empty LoF defect with wide separation between the faces. A partially melted powder particle appears like a hump and is indicated by the arrow. (b) Oxide-filled LoF defect with narrow separation between the faces. TiN particles are present around the oxide. (c) Empty pore. (d) Oxide-filled pore. TiN particles are present around the oxide. (e) Oxide-filled LoF with narrow separation between the faces. (f) Oxide-filled pore.

Fig. 9. Defect volume fraction in EBM and SLM material in different heat treatment conditions.

(1)-(g).
SLM material is plotted as SN curves and presented in Fig. 12 for the machined specimens and in Fig. 13 for the as-built specimens. Fatigue data for wrought material from the MMPDS-09 [22] database is plotted alongside for comparison. To compare the scatter in fatigue data at each load level, the statistical parameter Coefficient of Variance (CoV) has been used. CoV is defined as

\[ CoV = \frac{\sigma}{\mu} \times 100 \]

where, \( \sigma \) is the standard deviation and \( \mu \) is the mean of the fatigue life at each load level. CoV normalizes the standard deviation relative to the mean and aids in comparing data that are different in scale. The CoV for the EBM and the SLM material in different heat treatments and surface conditions is presented in Fig. 14 for various load levels. In the following sections, the fatigue results are presented and compared pairwise for the different group of specimens. In addition, the typical appearance of the fracture surfaces is also described.

3.4.1. SLM vs. EBM

The SLM material had higher life than the EBM, almost an order of magnitude higher, in both the as-built and machined condition. The scatter in fatigue life, assessed by CoV, indicated no clear difference in degree of variation between the two material groups in all combinations of test factors.

3.4.2. As-built vs. machined surface

On average, the specimens with machined surface performed better in fatigue compared to the specimens with as-built surface for both the
EBM and the SLM material regardless of the heat treatment condition. However, CoV for as-built condition is, in general, lower than the machined counterparts when compared between the same process and heat treatment combination. The EBM material in HT condition at 725 MPa, which has the lowest CoV among all the machined specimen groups, is an exception; in this specific case, the CoV was lower than the as-built counterpart.

In the specimens that were in as-built condition, there were multiple fatigue crack initiations from the surface; these were primarily from the valley like features of the roughness profile. This phenomenon was true both in HIP + HT and in HT conditions for the EBM as well as the SLM material. In the specimens that were in machined condition, considering the ones that did not have crack initiation at a defect, there was often only one main crack initiation site. In specimens that failed from defects, there were instances of multiple crack initiations; however, the number of initiations was at least an order of magnitude lesser than in the as-built condition. Representative fractograph images illustrating these effects are presented in Fig. 15.

3.4.3. HIP + HT vs. HT
The material in HIP + HT condition outperformed the material in HT condition for both the EBM and the SLM processed material; the effect was evident for specimens with an as-built surface while, for the machined specimens the effect was relatively diffused in the high scatter of fatigue life. CoV for HIP + HT condition was higher for the machined specimens at all load levels compared to HT condition.

In addition, the crack propagation area created before the final fracture in HIP + HT material was different from that of HT material, at all loading conditions, which could be seen at even low magnifications of a stereomicroscope, see Fig. 16. In HIP + HT condition, there was a strong tendency for the cleavage type fracture while in the HT material it appeared to be a more ductile crack propagation behaviour; this tendency of material in HIP + HT condition to exhibit cleavage type fractures is evident in the SE images shown in Fig. 15. However, in the EBM material this effect is, in general, less pronounced than for the SLM material, see Fig. 16 (note that the specimen in Fig. 15 (a) and Fig. 16 (b) are the same). This effect in EBM material is more evident in the specimens as-built surface than the ones that are machined.

4. Discussion
4.1. Surface roughness
In the current investigation, higher roughness values observed for as-built EBM surfaces compared to SLM surfaces could be related to the relatively larger layer thickness and coarser powders used in the EBM process. In addition, higher average build temperature in EBM could give rise to a relatively higher melt flow, which could contribute to the roughness as well, in the form of ripples. The effect of using finer powders and thinner layers on surface roughness have been investigated for EBM built Ti-64 [16], and it was found that the roughness decreases for both cases; but the reduction in roughness was significantly larger when using finer powders for the same layer thickness than it was when using thinner layers for the same powder size. Similar influence of smaller particles on roughness has been reported by Greitemeier et al. while comparing SLM and EBM built Ti-64 [15]. In their study, EBM and SLM parts were built with 50 µm and 60 µm thick layers.
respectively; however, because of the coarser powder used in the EBM process, the EBM surface was twice as rough as that of the SLM surface even though thicker layers were used for SLM. Kahlin et al. [13] reported that the roughness of EBM produced Ti-64 using 50 µm thick layers was twice as that produced by SLM using 30 µm thick layers. In the present study, 75 µm and 40 µm thick layers were used, for EBM and SLM respectively, which could explain the higher difference in roughness observed than the one reported by Kahlin et al.

Fig. 15. SLM specimens in HIP+HT condition (a) Stereomicroscope image of a fracture surface of a specimen with as-built surface condition. (b) 200× magnification SE image of the area indicated in (a) showing multiple crack initiations from the “notch-like” regions along the entire surface. (c) Stereomicroscope image of a fracture surface of a specimen with machined surface condition. (d) 200× magnification SE image of initiation area indicated in (c). Arrow indicates the single crack initiation site.

Fig. 16. Fractographs from a stereomicroscope. a) SLM specimen in HT condition b) SLM material in HIP+HT condition c) EBM material in HT condition d) EBM material in HIP+HT condition. Difference between crack propagation modes is clear between HT and HIP+HT condition for both materials.
4.2. Defects

With HIP, one has the potential to close defects such as shrinkage porosity, LoF and pores. Since these are not normally healed after a solutioning and ageing heat treatment, it could be inferred that the SLM material in the as-printed condition has lesser defects. This is evident in the defect volume fraction presented in Fig. 9; the SLM material in HT condition has lesser defects than the EBM material. The relative maturity of the SLM process for building Alloy 718 compared to the EBM process could be a possible reason for lesser defects in the SLM material. The standard process themes for building Alloy 718 using EBM are relatively new (only ~3–4 years since launch) and as a result, it could be that the SLM process parameters are relatively better optimized for reduction of defects than EBM.

The presence of strings of shrinkage porosity is indicative of the possibility of too low energy input for melting. The Arcam software adjusts the current and velocity of the electron beam based on the length of the lines that need to be melted, such that the time to melt each line remains the same [23]. For longer hatch lines, both the parameters are increased but, the current has a maximum limit beyond which only velocity is increased leading to lower energy input per unit length. The distance of ~350 μm between the strings is approximately five times the layer thickness. The standard melting theme uses a rotation angle for the hatching region such that every fifth layer has the same hatching direction. A possible interaction between hatch rotation and hatch length leads to a lack of sufficient energy for melting, which in turn could give a non-optimal melt pool and thus forming strings of shrinkage pores.

The reduction in the amount of defects after HIP treatment is higher for EBM material. This could be because of the closure of almost all shrinkage porosity that is present in the material. Almost none of the HIP+HT specimens had shrinkage porosity on the fracture surface; the same is true in case of the investigated cross-sections as well. Hence, the SLM material that has almost no shrinkage porosity to start with appears to have responded less dramatically to the HIP treatment. Another reason could be that the defects in the SLM process, both remnant from the powder and induced by the process, have entrapped argon. Argon does not diffuse out of the lattice easily, which prevents complete healing of the defects during HIP. The remnant spherical pores in the EBM material could be powder induced and the entrapped Argon in the powder particles could be the possible explanation. The remnant pores could also be explained by re-growth of pores during heat treatment, which were formerly closed by the HIP treatment. Such a phenomenon of re-growth of pores, which were closed after HIP, during the subsequent heat treatment has been observed for AM Ti-64 [11,12].

The remnant defects in HIP+HT condition could also be explained by, apart from argon entrapped, the fact that there are oxides in the defects, which would prevent closure and complete healing of the defects during HIP. In the current study, oxide-filled defects are present in both SLM and EBM material, see Fig. 8. The oxides could be formed during the spatter formation (the powder particles or the molten droplets ejected from the melt pool due to high energy density [24]) in AM processing of 718; the high surface temperature of the spatters in combination with the impurities in the chamber environment is a possible cause. From Ellingham diagram [25], the equilibrium partial pressure of oxygen that has to be maintained to prevent oxidation of aluminium and chromium are 10^{-58} bar and 10^{-12} bar respectively at their melting points. The melting point of aluminium and chromium are 660 °C and 1857 °C respectively, which would be lowered further in the vacuum environment of the EBM process. The industrial grade helium for maintaining the controlled vacuum EBM and argon for SLM are, in general, not pure enough to prevent the oxidation completely. The ejected spatters if returned to the powder bed could be trapped as oxides in the printed part. Another plausible source for the oxides is the powder itself, since the oxides are present in the spherical pores as well; the oxides could have been present in the precursors used for atomizing the powder or formed during the atomization process. Helmer [26] has reported presence of aluminium oxides in EBM built Alloy 718. He suggests that the increased partial pressure due to desorption of gases from the powder particles prevents effective degassing of the melt. As a result, when the solubility limit of oxygen is exceeded, the thermodynamically stable aluminium oxide is formed. These oxides do not dissolve during re-melting while adding a new layer on top, because of oxygen enrichment in the melt, and get agglomerated by convection. This effect could explain the formation of oxide-filled LoF defects. In-process oxidation and remnant oxides from precursor material have been investigated and reported as reasons for the presence of oxides in the built parts in a wire-based AM of Alloy 718 with laser heat source [27]. Tang et al. have reported formation of oxides due to spatter ejection during SLM process for Al10SiMg alloy [28]. Zhang et al. have also reported that the oxides can serve as nucleation sites for precipitation of intermetallic phases. Precipitation of titanium nitride particles on the oxide surface in the oxide-filled defects has also been reported for EBM processed Alloy 718 [29]. Similar phenomenon is observed in the present study, see Fig. 8.

4.3. Hardness

In the present study, the difference in hardness, for Alloy 718 specimens built with different AM processes and subjected to different heat treatments is rather small. The main reason could be that all the specimens are in a similar aged condition. The main strengthening mechanism in Alloy 718 is precipitation strengthening through γ’ precipitates. The volume fraction of γ’ is dependent on the amount of niobium available in the matrix in the supersaturated condition after the solution heat treatment. The solutioning temperature chosen for the work is higher than the 8 solvus; consequently, almost all the niobium in the matrix would be available for the formation of γ’. After solutioning at 1066 °C, probably there is equivalent volume fraction of γ’ for both EBM and SLM material in HIP+HT and HT condition which leads to equivalent hardness. This is, however, different from AM Ti-64 for which a reduction in hardness and tensile strength are reported following a HIP treatment [13,30]. The main strengthening mechanism in Ti-64 is from the grain structure, which coarsens during HIP leading to the lower hardness of the final AM parts. The small difference in hardness between the different groups could be explained as follows. A slightly higher hardness observed for EBM in HIP+HT could be due to the closure of defects after HIP treatment. In EBM material subjected to a HIP cycle similar to the one in this study and/or a solution treatment above 8 solvus followed by ageing, absence of both laves phase and 8 phase have been established [31,32]; hence, there could be no contribution from these phases. In addition, it has been previously shown that presence of larger defects affects the hardness measurements [33]. Since there are many shrinkage porosities and large LoFs in the EBM material in HT condition, it has relatively lower hardness compared to the HIP+HT condition and relatively larger scatter in the measured hardness. In SLM, however, in HIP+HT condition the material has lower hardness relative to HT condition. This could be due to the dissolution of the cellular sub-structure after HIP treatment. It has been previously shown that the dissolution of dislocation structure, which is present in the as-built condition, leads to softening of SLM built Alloy 718 [34]. Deng et al. have reported that there is remnant laves phase in the SLM material, built with similar process parameters, after treatment at 1080 °C followed by two step ageing; however, the amount of laves is minimal [35]. Zhang et al. have reported complete dissolution of laves phase after the aforementioned heat treatment [36]. So, the amount of niobium available for γ’ precipitation could be considered to be the same for both HIP+HT and HT conditions; consequently, an equivalent volume fraction of γ’ precipitates could be expected in both conditions. Moreover, the dissolution of the sub-structures is more efficient when treated at higher temperatures and for longer times [34]; therefore, the material in HIP
4.4. Fatigue

4.4.1. SLM vs. EBM

The difference in fatigue performance between EBM and SLM material could be explained by the presence of defects that are discussed in Section 3.2 and Section 4.2. Defects found on fracture surfaces are larger than the others found in metallographic cross sections. This could be because the probability of extracting a cross section at a plane having the longest dimension of the defect is smaller compared to the probability of the defect causing crack initiation at its longest dimension being observed on the corresponding fracture surface. This could also be the reason for large scatter observed in defect volume fraction measurement of the EBM material in HT condition.

The larger LoF defects in the EBM material have, being concentrated in the contour melt region and under the bending load condition, a bigger influence on crack initiation than the ones that are in the bulk; clustering of such defects makes the condition even more severe. An explanation for the preferential distribution of such defects is the process parameters for the contour melting. The presence of large lack of fusion defects, often with unmelted or partially melted particles is indicative of the lack of sufficient beam energy for melting. The beam energy could be increased by increasing the beam current, decreasing the velocity, increasing the focus offset or a combination of these [23].

The SLM material with fewer, smaller defects therefore performs relatively better in fatigue. However, the SLM material is inferior to the wrought material in fatigue properties. The difference could be related to the microstructural aspects, for example the wrought material in reference is treated according to AMS 5662 and could have controlled δ phase distribution at the grain boundaries; δ phase could have beneficial effect in retarding crack growth [37]. The SLM material in the current study is treated to AMS 5664, which could prevent δ phase precipitation. A closely related effect is the size and volume fraction of γ’ precipitates which is the primary strengthening phase; both the size and volume fraction in the SLM material could be different from that of the wrought material. Another microstructural aspect is the grain size and texture, which could also be different for the AM material than the wrought material that it is compared to. Additionally, the load case for the wrought data is axial while it is bending in the current study. A relatively higher emphasis on the surface under bending load condition could also have contributed to the difference.

The scatter in fatigue life as presented in Fig. 14 could be explained by the presence of defects, particularly the ones near the surface experiencing the maximum tensile stress and act as a crack initiation site. Tammas et al. [38] have reported that apart from the size of the defect, other factors such as the shape, location and proximity to the surface and other defects affect the fatigue life. Similarly, an attempt to predict the fatigue life of weldments of aerospace alloys has been made by using the information about defects such as size, shape, distribution and location [39]. In the present work, it is observed that the size, shape, number, location and nature of the defects affect the fatigue life. In general, a larger difference in the number of defects among the specimens in a particular test condition would correspond to a larger scatter i.e., larger CoV. For example, among three EBM specimens tested at 875 MPa in HIP+HT condition, one specimen had 2 crack initiating LoFs (least among the 3 specimens) while another had 13 crack initiating LoFs (most among the 3 specimens). The difference in number of crack initiating defects is 11, which is large, and results in a larger scatter. Belan et al. [40] have reported that when there are more crack initiating sites, the fatigue life is shorter. By analogy, a lesser CoV would be expected if the difference in number of defects is smaller. Indeed, the smallest CoV among the machined specimens, at 725 MPa in HT condition of EBM material, could be related to the presence of 11, 12 and 13 crack initiating LoFs respectively in the three specimens of the group.

The size and shape of the defects also affects the scatter. For example, among the EBM material in machined and HT condition at tested at 600 MPa, the specimen that had the lowest fatigue life had a LoF defect that is large (~ 1 mm in dimension along two perpendicular directions and is irregular in shape), see (d). In this case, even though the other two specimens had more number of LoFs, the scatter is not so large; the probable reason is the size of LoFs – i.e., the specimen having a larger but lesser number of defects perform equally worse like specimens which have smaller but more defects. To emphasize the effect of nature of LoFs, one EBM specimen in machined and HIP+HT condition that has a fatigue life of more than 100,000 cycles at 600 MPa can be pointed out. The LoF in this specimen appear to be partially healed due to HIP treatment, see (g). The other two specimens in the group, however, have LoFs connected to the surface that are unaffected by HIP. The unhealed LoFs severely affect the fatigue life while the specimen with partially healed defects had the longest life among the group. Additionally, in the presence of the as-built surface, LoFs alone do not explain the scatter; the surface roughness could have had a bigger influence on reducing the scatter, which is explained in Section 4.4.2 with the observations from Section 3.4.2.

For the SLM specimens, the defects affect the crack initiation in only 3 out of 24 specimens; 2 of which are LoFs and the other one is a pore. Though the specimens with defect-based crack initiation have a lower fatigue life, the shortest life does not always correspond to the specimen having the defects. This is, indeed, the case for material in the HIP+HT condition at 875 MPa; a probable reason could be that there are specimens from two different batches. There were two SLM specimens from the second batch in the machined condition, both in HIP+HT condition, and they had the lowest fatigue life at the respective load conditions, one tested at 875 MPa and other at 725 MPa. This indicates that there could be additional factors affecting the fatigue properties that result from a batch-to-batch variation; such a difference could be microstructure related, the investigation of which is out of the scope of the current paper. Two of the remaining specimens from the second batch are in as-built HT condition and discussed in Section 4.4.2.

4.4.2. As-built vs. machined surface

The machining of as-built surfaces improves fatigue performance; however, some machined specimens have a fatigue life in the range of the average life of as-built specimens. The lower life in some of these machined specimens could be explained by the presence of defects as discussed earlier. The higher number of crack initiations from the as-built surface could be because of all the valley like features in the surface causing stress concentration and acting as micro-notches [13]. The machined surface, relatively, has lower number of crack initiating sites because of the absence of stress raisers to the same extent as the as-built surface. The lower number of crack initiating sites could explain the better fatigue performance of the machined specimens, in general. Crack initiation from the machined surface is most likely due to persistent slip band (PSB) formation mechanism [41] or inclusions or carbides present at the surface [42].

Chan [16] has reported that the rough as-built surface could lead to a reduction of 60 ~ 75% of fatigue strength due to stress concentration. Using the roughness profile extracted from µCT, he approximated the elastic stress concentration due to the roughness for Ti-64 material to be up to ~ 2.3 for SLM and ~ 3.3 for EBM built surfaces. Kahlin et al. have reported that the notch sensitivity is equivalent for EBM and SLM surfaces even though fatigue notch factor is higher for EBM surface [13]. Attempts to relate the surface roughness parameters such as Rr [16] and Ra [15] to the fatigue performance have been made, but, several challenges remain in characterizing the surface roughness of AM surfaces with suitable techniques [5]. The SLM material from the different batches have a similar performance in the as-built condition. This indicates that the surface roughness effect is possibly larger than the microstructural effects, if any.
4.4.3. HIP vs. non-HIP material

The material in HIP + HT treated condition performs better than the HT condition for both the EBM and the SLM process. The increase in fatigue performance, however, is quite small and could be related to decrease in the amount of defects and thereby reducing the crack propagation rate. The facedet appearance of the HIP + HT material could be due to the grain morphology. In fatigue crack growth rate studies conducted on wrought Alloy 718 by Krueger et al. [43], macroscopically rough, faceted appearance of the fracture surfaces were observed to be common for a coarse grained microstructure compared to a fine grained microstructure. Both grain structures had transgranular cracking, but a lower crack growth rate was reported for the coarse grained material, which was related to the longer slip bands that formed. Faceted appearance of the fracture surface have also be attributed to transgranular cracking in high cycle fatigue tests by Ono et al. [44].

In the standard melting strategy for Alloy 718 using Arcam A2X EBM machine, the contour region is melted by spots while the hatch (bulk) region is melted by a line raster. It has been reported by Dehoff et al. [45] and Deng et al. [31] that there are equiaxed grains in the spot-melted contour region and that the raster-melted hatch is composed of columnar grains. It has been shown previously [7] that the equiaxed grains undergo coarsening when subjected to a HIP + HT treatment, similar to the one utilized in the current study, while the columnar grains do not undergo appreciable grain coarsening. Hence, it is possible that in the EBM specimens subjected to the HIP + HT condition, the equiaxed grains in the contour region are coarsened compared to the HT condition. Further studies are needed in order to confirm this hypothesis.

The microstructure of SLM built Alloy 718 material in EOS M290 machine with similar build parameters has been reported by Deng et al. [35]. In the as-built condition, the grains morphology was rather irregular with the longitudinal axes of the grains oriented at random angles to the build direction indicating that the grains grow principally upwards but not necessarily along the build direction. The presence of dendritic structure with the grains comprising of columnar and/or mosaic pattern has been reported for SLM built Alloy 718 by other authors as well [34,46]. It has also been reported in these previous investigations that SLM built Alloy 718 material undergoes recrystallization and grain growth when subjected to a HIP [46] or high temperature (above 1200 °C) solutioning treatment [34].

Therefore, the coarse grains in HIP + HT condition could explain the faceted appearance of the fracture surface. Such coarser grains are possibly present in the contour region of EBM material and in the bulk region of SLM material, which could explain the relatively larger proportion of the fracture surface with faceted appearance in the SLM material. The EBM specimens with the as-built surface have a larger portion of the contour region in tact compared to the machined ones, in which a portion of the contour region is removed while removing. Thus, the as-built specimens are likely to have a larger proportion of the fracture surface with coarser grains compared to the machined ones and hence have a more evident faceted appearance compared to the machined specimens.

5. Conclusions

- The surface roughness in as-built condition is three times higher for EBM compared to SLM. This is due to the relatively thicker layers and the coarser powder used in EBM.
- Machining improves fatigue life, but the scatter is larger compared to as-built condition. The larger scatter could be related to the larger number of crack initiation sites in the as-built condition, which is at least an order of magnitude higher than the machined condition. Such high number of crack initiation sites reduces the randomness in fatigue failure leading to a lower scatter.
- HIPing improves fatigue life for both EBM and SLM materials. The difference in fatigue performance could be due to the reduction in the amount of defects after HIP treatment.
- The crack propagation appears to be more ductile for HT material while it has a more cleavage appearance for the HIP + HT material. This difference in macroscale appearance of the crack propagation region could be due to the recrystallization and grain coarsening caused by the HIP treatment.
- SLM material outperforms the EBM material in high cycle fatigue; this could be related to the cause of fatigue crack initiations. Failures in the EBM material are dominated by large lack of fusion defects in the contour and contour-hatch interface region. Under the bending load condition, these defects are closer to or at the surface and hence experience the maximum or very high tensile stresses; thus have a larger influence than the material in the bulk.
- Oxides present in the material prevent complete healing of LoF defects after HIP. Specimens with partially healed sub-surface defects perform better in fatigue than surface breaching un-healed defects.

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Data availability

The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

References

Fatigue Properties of Additively Manufactured Alloy 718

Additive Manufacturing (AM), commonly known as 3D Printing, is a modern manufacturing process, in which parts are manufactured in a layer-wise fashion. It is a disruptive technology and is even referred to as the third industrial revolution. Though AM processes offer several advantages, the suitability of these processes to replace conventional manufacturing processes must be studied in detail. Therefore, understanding the process–post-treatment–microstructure–property relationship is crucial, to enable manufacturing of high-performance components. The aim of this work is, therefore, to understand how the fatigue properties of Alloy 718 processed by Powder Bed Fusion (PBF) additive manufacturing is affected by the microstructure and the as-built surface roughness.

Defects are detrimental to fatigue life; however, numerous factors such as the defect type, size, shape, location, distribution and nature determine the effect of defects. Hot Isostatic Pressing (HIP) improves fatigue life as it leads to closure of defects. Presence of oxides in the defects, however, hinders complete closure by HIP. Machining the as-built surface improves fatigue life; however, the extent of improvement can be dependent on the amount of material removed. The rough as-built surface has numerous crack initiation sites, leading to a lower scatter in fatigue life. In PBF processed Alloy 718, fatigue crack propagation is transgranular; crack propagation is affected by grain size and texture of the material.