The background of the cover is a high-magnification scanning electron micrograph (SEM) showing the microstructure of IN718. It displays a complex, layered, and fibrous texture, likely representing the grain structure and precipitates of the alloy after additive manufacturing. The image is in grayscale, emphasizing the topographical features of the surface.

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On the Microstructures and Anisotropic Mechanical Behaviours of Additively Manufactured IN718

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Cover image: A fracture surface of EBM IN718 after fatigue test at room temperature.

During the course of research underlying this thesis, Donyong Deng was enrolled in Agora Materiae, a multidisciplinary doctoral program at Linköping University, Sweden.

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Abstract

Additive manufacturing (AM), also known as 3D printing, offers great design flexibility for manufacturing components with complex geometries, and has attracted significant interest in the aero and energy industries in the past decades. Among the commercial AM processes, selective laser melting (SLM) and electron beam melting (EBM) are the two most widely used ones for metallic materials. Inconel 718 (IN718) is a nickel-base superalloy and has impressive combination of good mechanical properties, weldability and low cost. Due to its excellent weldability, IN718 has been intensively applied in the AM field, to gain more understanding of the AM processes and fully realize AM's potentials.

The study objects in the present thesis include both EBM and SLM IN718. The solidification conditions in EBM and SLM are very different and are different to that of conventional cast, leading to unique microstructures mechanical properties. Therefore, this thesis aims to gain better understanding of the microstructures and anisotropic mechanical behaviours of both EBM and SLM IN718, by detailed characterizations and by comparisons with the forged counterpart.

The as-built microstructure of EBM IN718 is spatially dependent: the periphery (contour) region has a mixture of equiaxed and columnar grains, while the bulk (hatch) region has columnar grains elongated along the building direction; the last solidified region close to the top sample surface shows segregation and Laves phases, otherwise the rest of the whole sample is well homogenized. Differently, the as-built microstructure of SLM IN718 is spatially homogeneous: the grains is rather equiaxed and with subgrain cell structures. These microstructures also respond differently to the standard heat treatment routines for the conventional counterparts.

Anisotropic mechanical properties are evident in the room temperature tensile tests and high temperature dwell-fatigue tests. The anisotropic tensile properties of EBM IN718 at room temperature are more likely due to the directional alignment of porosities along the building direction rather than the strong crys-

tallographic texture of $\langle 100 \rangle$ // building direction. While for SLM IN718, the anisotropy is more likely attributed to the different extents of ‘work-hardening’ or dislocations accumulated between the horizontally and vertically built specimens. The anisotropy mechanisms in dwell-fatigue crack propagations at 550 °C for EBM and SLM IN718 are identical: higher effective stress intensity factor when intergranular cracking path is perpendicular to the loading direction, but lower effective stress intensity factor when intergranular cracking path is parallel to or slightly deviated from the loading direction.

The 2160s dwell-fatigue cracking behaviours at 550 °C are of significant interest for AM IN718, of which test condition is similar to that of real service for IN718 disk in turbine engine. Generally, after conventional or short-term heat treatments, EBM IN718 shows better dwell-fatigue cracking resistance than SLM IN718. The damage mechanism is different for EBM and SLM IN718: the intergranular cracking in EBM IN718 is due to environmentally assisted grain boundary attack, while creep damage is active for SLM IN718. The considerably ‘deformed’ microstructure, specifically the subgrain cell structures in SLM IN718 resulted from the manufacturing process, is believed to activate creep damage even at a low temperature of 550 °C. And for SLM IN718, heat treatment routine must be carefully established to alter the ‘deformed’ microstructure for better time-dependent cracking resistance at elevated temperature.

Populärvetenskaplig sammanfattning

Additiv tillverkning (AM), även kallad 3D-printning, erbjuder stor designflexibilitet för tillverkning av komponenter med komplexa geometrier och har väckt ett stort intresse inom flyg- och energibranschen under de senaste decennierna. Bland de kommersiella AM-teknikerna är selektiv lasersmältning (SLM) och elektronstrålesmältning (EBM) de två mest använda metoderna för metalliska material. Inconel 718 (IN718) är en nickelbaserad superlegering som uppvisar en imponerande kombination av goda mekaniska egenskaper, god svetsbarhet och relativt låg kostnad. Tack vare legeringens utmärkta svetsbarhet har IN718 rönt stort intresse för AM tillämpningar.

Detta arbete inkluderar studier av både EBM och SLM IN718. Stelningsbetingelserna för EBM och SLM är mycket olika och skiljer sig även från det man ser för konventionella tekniker som t.ex. gjutning. Detta leder till unika mikrostrukturer och därför uppvisar AM materialen också andra mekaniska egenskaper. Syftet med denna avhandling är därför uppnå en bättre förståelse för mikrostrukturer och anisotropiska mekaniska egenskaper hos både EBM och SLM tillverkat IN718. Detta uppnås genom en detaljerade karaktäriseringar av materialen och genom jämförelser med konventionellt tillverkade varianter av IN718.

Kornstrukturen hos EBM IN718 är inhomogen och varierar typiskt från ytan och in mot centrum av materialet. Regionen nära ytterytan (konturen) har en blandning av runda likaxliga korn och smala enaxliga kolumnära korn, medan bulkregionen (hatch området) enbart har smala enaxliga kolumnära korn utsträckta längs byggriktningen. Det sista stelnade området nära toppytan av materialet uppvisar även tydliga segregeringar och innehåller Laves-fas, men i övrigt är resten av materialet väl homogeniserat. Mikrostrukturen hos SLM IN718 är dock annorlunda och är mer homogen med mer likformiga korn men med en tydlig cell- eller subkornstruktur. Dessa mikrostrukturer reagerar dessutom annorlunda på värmebehandlingar än vad man typiskt ser för de konventionella motsvarigheterna.

Både EBM och SLM materialen uppvisar tydliga anisotropa mekaniska egenskaper både vid statiska dragprov i rumstemperatur och vid utmattningsprovning

vid förhöjd temperatur. De anisotropa statiska egenskaperna hos EBM IN718 vid rumstemperatur beror mer troligen på porer i materialet som ofta linjerar upp sig längs byggriktningen, i stället för den starka kristallografiska texturen med $\langle 100 \rangle$ riktningen parallell med byggriktningen. För SLM IN718 kan däremot anisotropin troligen tillskrivas skillnaderna i restspänningar och deformationsstrukturer som uppstår mellan horisontellt och vertikalt byggda prover. Det generella anisotropa beteendet vid hålltidsutmattnings vid 550 °C är likartat för både EBM och SLM IN718 då man typiskt ser en intergranulär sprickväxt och har en högre effektiv spänningsintensitetsfaktor när kornen är utdragna vinkelrätt mot belastningsriktningen, men tvärt om får man en lägre effektiv spänningsintensitetsfaktor och sprickor som vikar av från huvudriktningen när kornen är utdragna parallellt med byggriktningen.

Hålltidsutmattnings vid 550 °C är av stort intresse för AM IN718, då det representerar ett vanligt driftsfall för många högttemperaturkomponenter. I allmänhet, efter konventionella eller kortvariga värmebehandlingar, uppvisar EBM IN718 betydligt bättre motstånd mot hålltidsutmattnings än SLM IN718. Skademekanismen skiljer sig dock åt för EBM och SLM IN718 då den intergranulära sprickväxten i EBM IN718 beror på en miljöinducerad försvagning av korngränserna, medan SLM IN718 uppvisar mer klassiska krypskador. Den höga dislokationstätheten och subkornstrukturen som uppstår i tillverkningsprocessen för SLM IN718, verkar aktivera krypskador även vid en så låg temperatur som 550 °C. Av samma anledning måste värmebehandlingsprocessen för SLM IN718 modifieras jämfört med konventionella processer om man vill förbättra motståndet mot tidsberoende sprickväxt vid förhöjda temperaturer.

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和丕敏同门四年，失落迷茫中互相勉励共同成长，感情深一口闷自然不必多说，“自命百炼钢，前程尽愿望”与你共勉。感谢承漠，晴昊还有爽姐，让我除了导师以外还有其他人可以讨论变形机制，砥砺切磋。感谢万军经常带我玩。我也要感谢在林雪平的某微信群的各位小伙伴，你们都身怀绝技，给我的生活增添了太多的欢乐。

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Dunyong Deng

邓敦勇

Oct 2019 Linköping

List of papers

The following papers have been appended in this thesis:

- I **D. Deng**, J. Moverare, R. L. Peng, H. Söderberg (2017). Microstructure and anisotropic mechanical properties of EBM manufactured Inconel 718 and effects of post heat treatments. *Materials Science and Engineering: A*, 693, 151-163.
- II **D. Deng**, J. Moverare, R. L. Peng, H. Söderberg (2018). On the formation of microstructural gradients in a nickel-base superalloy during electron beam melting. *Materials & Design*, 160, pp.251-261.
- III **D. Deng**, R. L. Peng, H. Brodin, J. Moverare (2018). Microstructure and mechanical properties of Inconel 718 produced by selective laser melting: Sample orientation dependence and effects of post heat treatments. *Materials Science and Engineering: A*, 713, 294-306.
- IV **D. Deng**, R. L. Peng, J. Moverare (2019). On the dwell-fatigue crack propagation behavior of a high strength superalloy manufactured by electron beam melting. *Materials Science and Engineering: A*, 760, pp.448-457
- V **D. Deng**, R. Eriksson, R. L. Peng, J. Moverare (2019). On the dwell-fatigue crack propagation behavior of a high strength Ni-base superalloy manufactured by selective laser melting. *Accepted @ Metallurgical and Materials Transactions A*
- VI **D. Deng**, R. L. Peng, J. Moverare (2019). A comparison study of the dwell-fatigue behaviours of additive and conventional IN718. *In manuscript*

In these papers I have been the main contributor, performing all the experimental work, characterization, analysis and manuscript writing, under the supervision of Johan Moverare and Ru Lin Peng.

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PART PART I:

BACKGROUND & THEORY

CHAPTER 1

Introduction

1.1 Background

Additive Manufacturing (AM) is a manufacturing concept that builds net shape components via an additive layer-by-layer or drop-by-drop manner. It offers great design freedom for complex-geometry and topologically optimized components, and attracts significant interest in the aero and energy industries. The ultimate aim of adapting AM would be to obtain ready-to-use components instead of just rapid prototyping. To additively manufacture metallic components, electron beam melting (EBM) and selective laser melting (SLM) are the two most widely used processes. The main parameters of these two processes are relatively different, for example energy source (electron for EBM and laser for SLM), chamber atmosphere (vacuum for EBM and protective gas for SLM) and powder bed temperature (relatively high for EBM but low for SLM, depending on the materials and machines). These factors result in unique solidification conditions that are very different from the conventional cast process, and therefore different microstructures and mechanical properties. For the confident application of EBM and SLM processes for the high-end critical components, it is important to systematically characterize the microstructure and understand how the microstructure corresponds to post heat treatment and correlates to mechanical properties.

1.2 Research aims and questions

Inconel 718 (IN718) is a nickel-base superalloy and is being intensively used as disk material in gas turbine engines. Due to its excellent weldability, IN718 has been popular within the metallic AM field. As mentioned, the AM processes give very

different solidification conditions compare to conventional cast process, and therefore lead to different microstructures. How the AM microstructures behave during high temperature applications is of great interest and importance, but yet not well studied at the moment, comparing to the conventional counterparts. On the other hand, the in-depth knowledge of AM IN718 might also provide fundamentals for successful application of AM processes to non-weldable Ni-base superalloys.

Generally, this thesis aims to characterize the microstructures of EBM and SLM IN718 and understand how the microstructures correspond to the anisotropic mechanical behaviour at room temperature and elevated temperature. Specifically, this thesis addresses the following questions regarding EBM and SLM IN718:

For EBM IN718,

1. Is the as-built microstructure homogeneous in EBM IN718?
 - Given the relatively high powder bed temperature, the previously processed layers/parts inevitably experience longer ‘in-situ’ annealing than the subsequently deposited layers/parts. Would this lead to the microstructural gradient?
 - Do the ‘contour’ and ‘hatch’ melting parameters applied to the frame and the core of builds, respectively, lead to different microstructures in the corresponding regions.
2. What is the reason for the anisotropic tensile properties at room temperature?
3. What are the damage mechanism and cracking behaviours under dwell-fatigue condition at elevated temperature?

For SLM IN718,

1. What is the as-built microstructure in SLM IN718? Given the relatively low power bed temperature and rapid cooling rate, would that cause segregation and residual stress in the as-built SLM IN718?
2. Is the as-built SLM 718 textured? How to rationalize the anisotropic mechanical properties with building orientations and textures?
3. How would the applied heat treatments affect the microstructure evolution and anisotropic mechanical properties?
4. What are the damage mechanism and cracking behaviours under dwell-fatigue condition at elevated temperature?

1.3 Outline of the thesis

This thesis is divided into two parts: Part I *Background & Theory* and Part II *Appended papers*. The first part *Background & Theory* is partly based on the

author's Licentiate thesis *Additively Manufactured Inconel 718: Microstructure and Mechanical Properties* [1], which was presented in February 2018. However, some contents have been modified and updated in the present thesis. The secondary part *Appended papers* appends six papers aiming to address the research questioned previously mentioned.

In Part I after Introduction chapter, chapters regarding IN718, fatigue crack propagation and dwell fatigue are given to introduce the microstructure and test methods of focus in the present thesis. After that, the literature on EBM and SLM IN718 in the past years is reviewed, to gain a better understanding of microstructure, heat treatment, fatigue and creep properties. The experimental details are summarized in Chapter 6. A summary and discussion of the appended papers are present in Chapter 7, based on which a conclusion and outlook is suggested in Chapter 8.

CHAPTER 2

Inconel 718

Inconel 718 is a nickel-base superalloy. It was developed by International Nickel Company in 1959 [2]. The excellent combination of high strength, good weldability and fabricability and low cost has earned IN718 great success in a wide range of applications for decades. This chapter will give a general introduction to the alloying elements and phases of IN718. Heat treatments established for conventional IN718 are also given in the following, and might provide some hints to establish heat treatments for AM counterparts.

2.1 Elements and Phases in IN718

The composition ranges of alloying elements in IN718 are as shown in Table 2.1. Note that IN718 contains significant amount (~ 20 wt.%) of Fe, due to which IN718 is occasionally classified as iron-nickel-base superalloy, and the manufacturing cost is considerably lowered. Another noticeable feature is the relatively high content of Nb, which makes the IN718 unique with the strengthening phase of γ'' . With these alloying elements, several phases can be formed in IN718 under certain heat treatment conditions. The crystal structures and chemical formulas of the common phases in IN718 are summarized in Table 2.2, of which γ'/γ'' , δ and Laves will be specifically introduced since these phases intensively involve in the following appended research papers.

2.1.1 γ' and γ''

IN718 is a precipitate-strengthened Ni-base superalloy, and is strengthened principally by γ'' and marginally by γ' . γ' and γ'' precipitate from the γ matrix during ageing, and both of them are coherent with the γ matrix. The lattice mismatch at

Table 2.1. IN718 composition per Aerospace Material Specifications (AMS) 5383

Element	Ni (plus Co)	Cr	Fe	Nb (plus Ta)	Mo
wt.%	50.00 - 55.00	17.00 - 21.00	Bal.	4.75 - 5.50	2.80 - 3.30
Element	Ti	Al	Co	C	Mg
wt.%	0.65 - 1.15	0.20 - 0.80	1.00 max	0.08 max	0.35 max
Element	Si	P	S	B	Cu
wt.%	0.35 max	0.015 max	0.015 max	0.006 max	0.30 max

Table 2.2. Phases commonly observed in IN718

Phase	Crystal structure	Chemical formula
γ	fcc	Ni
γ''	bct (ordered $D0_{22}$)	Ni_3Nb
γ'	fcc (ordered $L1_2$)	$\text{Ni}_3(\text{Al}, \text{Ti})$
δ	orthorhombic (ordered $D0_a$)	Ni_3Nb
MC	cubic B_1	$(\text{Nb}, \text{Ti})\text{C}$
Laves	hexagonal C_{14}	$(\text{Ni}, \text{Fe}, \text{Cr})_2(\text{Nb}, \text{Mo}, \text{Ti})$

the coherent γ'/γ and γ''/γ interfaces can introduce coherency strain hardening, which confers principally strength to this alloy at the peak aged condition [3, 4].

Of the commercial and standardly aged IN718, γ' precipitates in a spherical morphology, while the presence of γ'' is in a disk morphology. In the $D0_{22}$ crystal structure of γ'' , the tetragonal lattice distortion is $c/a = 2.04$, which results in considerable coherency strain at c -axis [5]. The lattice mismatch between γ'' and γ matrix is reported as 2.86 % [4], while between γ' and γ matrix the mismatch is less than 0.5 % [6]. The volume fraction of γ'' in the standardly aged condition varies largely with the measurement technique. The transmission electron microscopy (TEM) [5, 7] and atom probe tomography (APT) [8] measurements similarly give a range of about 10~15 %. However, the latest quantification by small-angle neutron scattering (SANS) [9] and neutron diffraction [10] suggest lower volume fractions of γ'' : 3.8 % and 7.9 %, respectively.

It should be noted that γ'' is metastable, and can transform into the incoherent thermodynamically stable δ phase over 650 °C with loss of strength. The thermal stability of γ'' limits the main applications of IN718 under 650 °C [11, 12]. In the aged conventional IN718, the morphologies of γ' and γ'' can be in the form of γ' or γ'' monoliths, γ'/γ'' duplets, sandwich-like $\gamma''/\gamma'/\gamma''$ or $\gamma'/\gamma''/\gamma'$ [13]. By increasing the (Al + Ti)/Nb atomic ratio beyond 0.9 (which is 0.69 for conventional IN718), a compact morphology with cube-shaped γ' particles coated with a γ'' shell over their six faces, has been proved to associate with better thermal stability than the conventional IN718 [14]. Han et al. [15] suggested that the coherent interfaces of γ''/γ -matrix and γ''/γ' in the compact-morphology precipitate can act as a strong diffusion barrier for exchange of Al, Ti and Nb, which contributes to the better thermal stability in the modified IN718. On the other hand, by increasing

the Al/Ti ratio along with (Al + Ti)/Nb atomic ratio in IN718, a more thermally and mechanically stable co-precipitate γ''/γ' microstructure can be also achieved, which could be due to a higher volume fraction of γ' or the smaller γ'' precipitate sizes [16]. Such a slow coarsening effect of co-precipitation of γ''/γ' also provides a promising solution to avoid the overageing of γ' -strengthened disk alloys during processing [17, 18]. There is possibly another way to minimize the coarsening of γ'' by tailoring its variants. γ'' has three variants with its c-axis along the $\langle 001 \rangle$, $\langle 010 \rangle$ and $\langle 100 \rangle$ of γ matrix. The preferential coarsening of these variants during ageing has been observed with applied stress or strain within the specimens [19–21].

2.1.2 δ

As mentioned, γ'' can convert to δ , with loss of strength, under thermal exposure. The formation of δ is believed as a result of the excessive coherent mismatch between γ'' and γ matrix, and can be retarded by increasing the Al/Ti ratio and/or the Al + Ti content in IN718 [16]. With higher Al/Ti ratio and/or Al + Ti content, the size of γ'' is reduced as well as the lattice mismatch between the γ'' and γ matrix, therefore decreasing the driving force to form δ .

Besides the undesired convert from γ'' , δ can also form by applying a solution heat treatment in an approximate temperature range of $871 \sim 1010$ °C [22]. The precipitation of δ might also depend on the homogeneity of Nb distribution within the material. If very little segregation of Nb is assumed, grain boundary can be a preferential site for precipitation of δ , and an appropriate amount of grain boundary δ is believed to be beneficial for mechanical properties under certain circumstances. For example, over 4% of δ phase at grain boundaries can efficiently inhibit the grain growth during heat treatment and hot working, which is an important aspect of current high-strength IN718 production [23, 24]. And under creep or stress rupture conditions at elevated temperature, grain boundary δ phase might retard grain boundary sliding and increase the creep resistance [25, 26], if grain boundary sliding mechanism is operative for the intergranular fracture.

2.1.3 Laves

Laves is a brittle intermetallic topologically close packed (TCP) phase, and is commonly observed in the as-cast components. The formation of Laves phase is a result of Nb, Si and Mo segregation during solidification, where these alloying elements are rejected from the dendrites into the interdendrites [22, 27]. Chang et al. [28] suggested that the relatively high contents of Cr and Fe in IN718 are necessary for forming Laves phase, aside from the segregation of Nb. The chemical composition of Laves might differ with solidification condition, but is generally referred as $(\text{Ni,Fe,Cr})_2(\text{Nb,Mo,Ti})$. In addition to its brittle nature, Laves depletes Nb in the γ matrix and reduces the amount of principal strengthening γ'' phase. Therefore, high temperature homogenization treatment must be applied to dissolve the Laves phase, if it exists, and to homogenize Nb distribution. The solvus temperature of Laves phase might depend on the actual composition and might

differ from one case to another. Care must be taken to avoid rapid heating up to 1162 °C (normal solvus temperature of Laves) to prevent incipient melting at the grain boundaries [22].

In addition, though the IN718 is reputed for good weldability in the context of strain age cracking resistance, the heat affected zone (HAZ) liquation cracking/microfissuring is still a major concern for IN718 during welding. During the heating cycles of welding, the low-melting-point Laves phase at grain boundaries can be liquated, forming grain boundary liquid. With the development of thermal stress during the cooling cycles of welding, the liquated grain boundaries are easily torn apart, leading to the hot cracks/microfissures [29, 30].

2.2 Heat treatments

Applying post heat treatments to IN718 is generally to remove the compositional segregation, and alter the presences and distributions of phases to obtain a homogeneous and appropriately strengthened microstructure. Specifically, homogenization is to dissolve the Laves phase, if there is any, and homogenize the compositional segregation; solution treatment is to homogenize the less-segregated microstructure and to precipitate small amount of δ ; ageing is to precipitate the strengthening phases γ' and γ'' . Note that the establishment of heat treatments is based on the microstructure inherited from the manufacturing process, application as well as desired properties.

2.2.1 Wrought IN718

IN718 is being predominantly used in the wrought form. Wrought is a process that mechanically works a cast billet or ingot several times at high temperature to get the final product. Therefore, the wrought microstructure is generally more homogeneous and has finer grains than cast microstructure. The common heat treatment details and applicable AMS specifications are summarized in Table 2.3. STD1 is the standard heat treatment for aerospace applications, gas turbine disks for instance, producing high rupture properties, room-temperature tensile strength and fatigue strength [24]. For the tensile-limited applications, STD2 is preferred since it produces the best transverse ductility in heavy sections, impact strength and low-temperature notch tensile strength. Note that, all the grain boundary δ phases, pinning the grain boundaries and providing the notch ductility, would be dissolved by STD2, which would result in notch brittleness in stress rupture capability [24]. If a high-quality billet is used as the starting material and then forging is done below the δ solvus, direct ageing (DA) heat treatment is recommended for obtaining the highest tensile properties though a slight loss in stress rupture capability [24].

2.2.2 Cast IN718

Castings are intrinsically stronger than forgings at elevated temperature since the coarser grains in castings favour high temperature strength [24]. Though IN718

Table 2.3. Heat treatments for wrought IN718

	Specifications	Solution heat treatment	Ageing heat treatment
STD1	AMS 5589	927~1010°C for 1~2h, followed by rapid cooling, usually in water	719°C for 8h,
	AMS 5596		furnace cool to 621°C,
	AMS 5562		hold at 621°C for a total
	AMS 5662		ageing time of 18h, followed by air cooling
STD2	AMS 5590	1038~1066°C for 1~2h, followed by rapid cooling, usually in water	760°C for 10h,
	AMS 5597		furnace cool to 649°C,
	AMS 5564		hold at 649°C for a total
	AMS 5663		ageing time of 20h, followed by air cooling
DA	-	-	719°C for 8h, furnace cool to 621°C, hold at 621 °C for a total ageing time of 18h, followed by air cooling

is being used in the wrought form as turbine disk material as mentioned, cast IN718 has still gained applications in aircraft engines for compressor and turbine frames, combustor cases, fuel nozzle rings and other hot engine structures [31]. Severe interdendritic segregation and presence of Laves phases, with/without cast porosity, make the cast microstructure considerably different from that of wrought form. Cast porosity can be closed by applying a hot isostatic pressing (HIP) cycle; minimizing segregation can be achieved by the HIP cycle or by a separate homogenization heat treatment [22, 31, 32]. The homogenization temperature and duration are largely dependent on the size of Laves phase: the bigger Laves phases, the higher homogenization temperature and longer homogenization duration [33]. Higher homogenization temperature can surely accelerate the homogenization process, but attention must be paid to incipient melting at grain boundaries caused by the rapid heating over the Laves solvus temperature [22]. Note that the grain boundary δ is dissolved during the homogenization treatment, which would cause unfavourable notch brittleness. Therefore, a solution treatment following the homogenization treatment is applied to re-precipitate δ phases. Ageing treatment for precipitating γ'/γ'' is basically the same as that for wrought IN718. Standard heat treatment for cast IN718 per AMS 5383 is listed in Table 2.4.

Table 2.4. Standard heat treatment for cast IN718 per AMS 5383

Homogenization treatment	Solution treatment	Ageing treatment
1093±14°C for 1~2h, followed by air cooling or faster cooling	954~982°C for more than 1h, followed by air cooling or faster cooling	718°C for 8h, furnace cool to 621°C at 55°C/h , hold at 621°C for 8h, followed by air cooling

2.2.3 Powder metallurgy IN718

Powder metallurgy (PM) approach has been proposed to produce integral IN718 turbine rotors for space vehicles with short life but subjected to high temperature and high stress [34]. The PM route is able to produce finer grains, more uniform properties and near-net-shape components. By applying the STD1 heat treatment (see Table 2.3), the PM IN718 yields comparable tensile strengths to those of wrought IN718 but inferior ductility, which is attributed to the intergranular fracture induced by the decorations of brittle oxides (Al_2O_3 , TiO_2) and MC type carbides at prior particle boundaries (PPBs) [34]. Further study showed that the oxygen pick up during the inert gas atomization of the master alloy would favour the formation of oxides and MC PPB network, drastically decreasing the ductility and stress rupture property at elevated temperature [35]. The standard heat treatment per AMS5662 is not suitable for PM IN718. Instead, a solution treatment at 1270 °C and a HIP at 1100 °C/130 MPa/3 h were suggested prior to the standard heat treatment per AMS5662 [36], in order to break the PPB networks.

CHAPTER 3

Fatigue crack propagation

Fatigue, by definition in ASTM E 1150, is a process of progressive localized permanent structural change occurring in a material subjected to conditions that produce *fluctuating stresses and strains* at some point or points and that may culminate in cracks or complete fracture after *a sufficient number of fluctuations*. The emphasised terms *fluctuating stresses and strains* and *a sufficient number of fluctuations* differentiate fatigue failure from the monotonic overload tensile/compressive failure. In this chapter, a brief review on the common approaches for estimating fatigue resistance will be given firstly. Then the focus will be put on the Paris-regime of fatigue crack propagation behaviour. A general discussion on cycle or time dependent fatigue crack propagation will also be given.

3.1 Estimating fatigue resistance

Lifing approach and damage-tolerant approach are commonly applied to estimate a material's fatigue resistance. The essential difference between these two approaches is that if defects or flaws are assumed in the materials. If the material is assumed as free of defects or flaws, the stress-life or strain-life approach is applicable. Fatigue test is conducted under a series of stress σ or strain ε amplitude, and correspondingly the life cycles to failure will be obtained. The stress-life approach is suitable for long life (usually infinite life) situations where the applied stress always remains elastic compared to the material's yield strength. Differently, the strain-life approach considers plastic strain under loading and the cyclic strengthening/softening behaviours.

The damage-tolerant approach assumes the presence of defects or flaws in the initial material or component, and during operation such defects or flaws are tolerant until they grow to a critical size. When damage-tolerant approach is employed,

crack growth with fatigue cycles is of interest to quantify the fatigue resistance. With the regular inspection and evaluation, it is possible to track the damage development after operation cycles and predict the potential behaviour. Compared to lifing approach, damage-tolerant approach is more suitable for safety-critical situations.

3.2 Fatigue crack propagation

Fatigue crack propagation (FCP) data is typically plotted as da/dN versus ΔK on a log-log scale, as shown in Fig. 3.1. da/dN is the crack propagation rate, which is determined by derivative of the crack length a to the number of cycles N . ΔK is a function of crack length a , load amplitude $\Delta F (=F_{max} - F_{min})$ and dimensionless geometric factor depending on the crack and component geometries. For the specific ΔK solution please refer to some stress intensity factor handbooks.

Figure 3.1. Schematic plot of fatigue crack propagation rate da/dN versus stress intensity range ΔK on a log-log scale.

The fatigue crack propagation behaviour can be divided into three distinct regimes in the da/dN versus ΔK plot, as shown in Fig. 3.1. The crack growth rate da/dN increases rapidly with ΔK in both regime I and III. Crack does not propagate when ΔK is below the threshold ΔK_{th} , failure happens when ΔK is approaching the fracture toughness K_c . In the regime II crack propagates in a steady state, within which regime the Paris' law provides an adequate engineering description of da/dN versus ΔK [37]. The regime II is of significant engineering interest, since in the regime I crack propagates relatively slowly, while stepping

into regime III it is practically regarded as failed. In the following discussion the fatigue crack propagation is limited in steady state.

It should be noted that the correlation of stress intensity factor K to crack propagation is based on the linear elastic fracture mechanics, in which the mathematical description of the stress at the very tip of a crack becomes infinite. However, it is not realistic since the crack tip material cannot withstand infinite stress. It is more reasonable to expect a plastic deformation zone ahead the crack tip due to stress concentration. The cyclic plastic zone size R_p depends on both ΔK and yield strength σ_{ys} :

$$R_p = \begin{cases} \frac{1}{3\pi} \left(\frac{\Delta K}{2\sigma_{ys}} \right)^2 & \text{plane - strain} \\ \frac{1}{\pi} \left(\frac{\Delta K}{2\sigma_{ys}} \right)^2 & \text{plane - stress} \end{cases}$$

R_p has to be much smaller than the crack size and the un-cracked ligament, to validate the application of stress intensity factor K . This is small-scale yielding condition. In the regime II where ΔK is much smaller than K_c , both linear elastic fracture mechanics and small-scale yielding condition are usually well-satisfied, and the Paris' law is able to give an adequate engineering description of fatigue crack propagation behaviours.

3.3 Cycle-dependent and Time-dependent FCP

The mechanical loading variables (stress ratio R , frequency, waveform, load interactions), environment variables (temperature and atmosphere) and microstructure variables (grain size, precipitate, crystallographic texture, etc.) in the fatigue service condition can significantly affect the fatigue crack propagation behaviours [38]. In general, cycle-dependent and time-dependent damages can simultaneously happen and compete with each other within every fatigue cycle, depending on the aforementioned factors. The cycle-dependent crack propagation rate can be expressed as $\frac{da}{dN} = C\Delta K^n$, indicating that the damage is driven by ΔK and is from the pure-mechanical fatigue (ramping up and down) part during each fatigue cycle. The time-dependent crack propagation damage is usually in the form of creep or environmental attack [39], which usually but not necessarily happen during the hold period superimposed on the fatigue cycles. Such a time-dependent term can be written in the form of $\frac{da}{dt} = CK_{hold}^n$.

How can one identify that if a fatigue crack propagation behaviour is cycle-dependent or time-dependent? It simultaneously depends on all the mechanical loading, environment and microstructure variables as mentioned, and it is very difficult to find a universal criterion that above which it is cycle-dependent and below which it is time-dependent. Typically, cycle-dependent crack propagation is in a ductile transgranular manner (see Fig. 3.2a), and fatigue striation can be present on the fracture surface and indicate the local fatigue crack propagation

rate. Differently, time-dependent fracture is more likely in a brittle intergranular manner, and on the fracture surface the grain facets are visible but no fatigue striations (see Fig. 3.2b). It is also possible that the fatigue crack propagation is in a mixed mode, and both transgranular and intergranular features are visible on the fracture surface.

Figure 3.2. (a) Cycle-dependent transgranular fracture surface with ductile striations in IN718 fatigued at 20 Hz at 650 °C; (b) time-dependent brittle intergranular fracture surface in IN718 fatigued with 10-300-10 waveform at 650 °C [40].

Generally, by increasing frequency, lowering temperature, shortening hold time at peak load and changing to a less aggressive atmosphere, it is more likely that cycle-dependent crack propagation gradually dominates. However, for Ni-base superalloys, the service conditions of high temperature and aggressive oxidizing atmosphere can accelerate the time-dependent intergranular damage (creep or environmental attack), which might result in a significant reduction of service life and catastrophic failure of engine components. And with prolonging the hold time at the peak load during each fatigue or service cycle at high temperature would increase the creep or environmental damage. Therefore, it is of great importance and interest to understand the role of time-dependent creep or environmental attack to the crack propagation behaviours. This hold time (dwell) effect will be specifically discussed in the next chapter.

CHAPTER 4

Dwell fatigue

A service cycle of turbine components typically includes starting-up, dwell (hold or sustained load) and shutting down. The duration of dwell part might vary from one application to another, but it is usually at high temperature and under peak load, accelerating crack propagation in a brittle intergranular manner. This might place a critical challenge to the materials used in hot sections of turbine engines. In this chapter, the dwell effects, and dwell damage mechanisms regarding environmental-assisted grain boundary attack and creep will be reviewed.

4.1 Accelerated crack propagation

It is widely reported that the addition of a dwell period at maximum load at high temperature can significantly increase the crack growth rate per fatigue cycle in Ni-base superalloys [40–52]. Among these literature, Gustafsson et al. [51, 53–55] has reported systematic results on the crack propagation behaviours with different dwell periods at different temperatures of a forged IN718. As shown in Fig. 4.1a, fatigue crack propagation rate per cycle increases significantly with prolonging the dwell period at the peak load. If the crack propagation rate is plotted on da/dt , one can see that the 2160s-dwell, 21600s-dwell and sustained(creep)-load data collapse into one single line when K_{max} is above $20 \text{ MPa}\sqrt{m}$, but not the 90s-dwell data. That indicates with prolonging the dwell time, the crack propagation behaviour would transit from fully cycle-dependent to fully time-dependent. On the other hand, though dwell effect is usually detrimental, the susceptibility to dwell effects is largely dependent on the microstructure, which means by understanding the dwell damage mechanisms and optimize the microstructure it is possible to improve the dwell-fatigue resistance of a material.

Figure 4.1. (a) da/dN versus ΔK and (b) da/dt versus K_{max} of forged IN718 with different dwell periods at 550 °C. Data are adapted from Gustafsson et al.[51, 54]

4.2 Environmental-assisted damage

The detrimental effects of the oxidising atmosphere at elevated temperature and under mechanical load have been widely recognized when comparing the dwell-fatigue crack propagation tests conducted under air, low oxygen partial pressure and vacuum conditions, where under vacuum fracture it is usually ductile trans-granular while under air it is brittle intergranular [50, 56–60]. Two contending theories, namely dynamical embrittlement (DE) and stress assisted grain boundary oxidation (SAGBO), have been proposed to interpret the environmental-assisted grain boundary damage.

4.2.1 DE

The time-dependent intergranular cracking of IN718 at elevated temperature has been explained with the theory of DE in [61–66]. The essence of such a dynamic embrittlement process is that, a short-range diffusion of embrittling element oxygen under tensile load at elevated temperature lowers the grain boundary cohesion, and cause debonding of the embrittled grain boundary ahead the crack. This process is similar to hydrogen and sulphur embrittlement. Note that no oxidation involves in DE, advocated by the observation of smooth fractured grain boundary facets and very high crack propagation rate [62]. Since the diffusion of the embrittling element along the grain boundary is critical in the DE process, it is possible to improve the resistance to DE cracking by reducing the diffusivity. It shows that the special coincident site lattice (CSL) grain boundary and low angle grain boundary might have lower oxygen diffusivity than random high angle grain boundaries. Therefore, better resistance to DE can be achieved by increasing the CSL fraction, specifically the $\Sigma 3$ (twin) boundary [63, 67, 68].

4.2.2 SAGBO

Different to DE, SAGBO involves firstly the formation of brittle oxides at grain boundary due to the long-range of oxygen diffusion and subsequently fracture of the grain boundary oxides. The high resolution characterizations on the propagating crack tip at elevated temperature do confirm the presence of oxide intrusion ahead the crack tip [57, 69–74]. The detailed SAGBO model proposed in [75, 76] suggested that tensile stress state ahead the crack tip can promote the outward diffusion defects (oxygen vacancy for Cr and metal cation for Ni), correspondingly increase the oxidation rate. The layered oxide intrusion might suggest different SAGBO enhancement factors for different elements, since the stress effects on the diffusion of oxidising defects largely depend on the element and its activity.

4.2.3 DE or SAGBO?

It is still under debate that which theory exactly governs the intergranular embrittlement during dwell cracking. The evidence of presence of oxide intrusion ahead the crack tip does advocate the SAGBO theory, but by the diffusion analysis in the SAGBO model in [75, 77] it indicates that a normal stress of $\sim$ GPa is required to have such an enhanced oxidation effect at the intrusion tip, which stress might be possible for strong alloys such as RR1000 but unreasonable for weaker alloys at elevated temperature. Pfaendtner and McMahon et al.[66, 78] suggested that DE-induced intergranular cracking velocity is on the same order of intergranular oxygen diffusion coefficient, which is more physically realistic than the long-range diffusion of SAGBO process. A correlation between the local cracking velocity and diffusion process might be an attractive manner to identify the governing mechanism. Jiang et al.[71, 79] suggested that the intergranular embrittlement mechanism is SAGBO at low ΔK level but switches to DE at relatively high ΔK level, evidenced by the gradual reduction of oxides presence along the dwell crack. Further study is still needed to clarify this issue.

4.3 Creep damage

It is also possible that creep damage outweighs environmental-assisted damage during dwell-fatigue cracking, depending on the material, temperature and loading waveform. The presence and morphology of the secondary crack in the vicinity of the main crack are indicative to differentiate the creep-induced damage from the environmental-assisted damage: the secondary crack resulted from the environmental-assisted damage is connected to the main open crack, while it is not necessarily originated from the main open crack in creep-induced cracking. This is due to that oxygen supply from the main open crack is indispensable in the environmental-assisted damage process, but creep is a thermal activation process that does not require oxygen supply.

For the Ni-base disk superalloys at the intermediate service temperature, creep crack propagation is usually intergranular. The time-dependent steady creep crack propagation behaviour can be expressed with the stress intensity factor K in a

Paris' law manner $\frac{da}{dt} = A \cdot K^n$ for Ni-base superalloy [80–82]. The stress intensity exponent n is similar but not identical to the power-law creep stress exponent obtained from the normal creep test under fixed stress and temperature [83]. Grain boundary sliding (GBS) might play an important role in the intergranular creep cracking cases.

Now one might consider under what dwell-fatigue condition can creep damage dominate the crack propagation? It significantly depends on the relative resistance of creep to environmental embrittlement at the testing condition, since these two damage processes might be simultaneously competing during the dwell period. Long dwell-fatigue (e.g. 90s dwell for LSHR at 704 °C [84] and even at 760 °C [85]) and even sustained load (e.g. for IN718 at 550, 600 and 650 °C will be discussed in the appended Paper V) at elevated temperature does not necessarily guarantee a dominant creep damage.

4.4 Dwell and AM

From the discussion above and from our experience on dwell-fatigue test, specifically with long-hold dwell at elevated temperature, the cracking resistance and behaviour are very sensitive to the microstructure. Therefore, by doing dwell fatigue crack propagation tests on AM components, how the AM microstructure correlates to the time-dependent deformation behaviour at elevated temperature can be revealed, which is less well-understood at the moment. That motivates the author to investigate the dwell-fatigue cracking behaviours of AM materials in the present thesis work.

CHAPTER 5

EBM & SLM IN718

The materials involved in this thesis include both Electron Beam Melting (EBM) IN718 and Selective Laser Melting (SLM) IN718. Generally, these two AM processes produce microstructures significantly different from those of conventional counterparts. For each of these AM processes, microstructures can also be dramatically different with different process parameters and with different post-process heat treatments. In this chapter, EBM and SLM processes will be briefly introduced, and then the typical microstructures and some general mechanical properties of EBM and SLM IN718 will be reviewed, as well as a general discussion for the post heat treatment.

5.1 General introduction to EBM & SLM

EBM and SLM are two of the most widely used powder bed AM processes for building metallic components. The essence of these two processes is basically the same: using an energy source to selectively melt the powders in a layer-by-layer manner (see Fig. 5.1). EBM process uses an electron beam as the energy source, which offers specific advantages. For example, to enable the electron beam to work appropriately, high vacuum is maintained throughout the process, making EBM particularly suitable for manufacturing the chemical-sensitive materials, e.g., titanium. With the electromagnetic lenses, the electron beam can be focused or defocused to adjust the energy density for heating or melting purposes. In addition, the state-of-the-art *MultiBeam* enables the extremely rapid movement of the electron beam within the building area, allowing melting at multiple points simultaneously and achieving high melting capacity and high productivity [86]. Further, it is worth mentioning that with the rapid movement and defocus of the electron beam, the entire powder bed is heated and maintained at a relatively high

temperature throughout the process, producing the components almost free from residual stresses. Differently, SLM uses a laser beam as the energy sources. Usually, the laser beam is in comparatively lower power and smaller size than electron beam, due to which there is not such a top down laser beam preheating step during SLM process (see Fig. 5.1), and the SLM powder bed can not be maintained at a relatively high temperature. Some other general differences between EBM and SLM processes are summarized in Table 5.1.

It is of interest to go further into the melting stage of EBM and SLM cycles. Normally, during the melting stage, two scanning strategies, namely contour and hatch, are typically applied in both EBM and SLM cycles: contour is to ‘draw’ the frame of the build, while hatch is to ‘fill in’ the interior of the build. The contour is generally associated with lower power and lower scanning speed to improve the geometry accuracy and roughness, while the hatch is adjusted to a higher power and higher scanning speed to increase the productivity [87, 88]. Specifically, in the EBM process, contour strategy uses the *MultiBeam* technology that splits the electron beam into multiple spots and rapidly ‘draw’ the frame, enabling optimization of surface finish, precision and build speed simultaneously [86, 89]. While, hatch strategy scans continuously the beam in a forwards-and-backwards pattern at each layer, and the scanning direction is rotated by a certain angel between each layer. Depending on the specific settings, the contour and hatch strategies usually result in more significantly different microstructures between the corresponding regions in an EBM build than in a SLM build.

Figure 5.1. Schematic building cycle of EBM and SLM processes. Illustration is adapted from [90].

Table 5.1. General differences between EBM and SLM [91]

	Energy	Atmosphere	Powder size	Layer thickness	Base plate temperature
EBM	Electron	Vacuum	40~140 μm	50~150 μm	>700 $^{\circ}\text{C}$
SLM	Laser	Ar/N ₂	15~45 μm	20~40 μm	~RT

5.2 EBM IN718

Typically, in a standard Arcam EBM process, contour (spot) and hatch (line) scanings are typically applied to the frame/surface and bulk part of a build, resulting in very different microstructures in these two regions [92]. For the hatch bulk region, the typical as-built EBM IN718 has the columnar grains elongated along the building direction and strong $\langle 100 \rangle$ // building direction texture [92–95]. Due to the elevated chamber temperature and slow cooling down after processing, the strengthening precipitates γ''/γ' are present in the as-built condition but are not in the peak-aged condition [92, 94, 95]. Some small (Ti,Nb)(C,N,B) precipitates can be found to string and align parallel to the building direction [93]. The crystallographic texture might play an important role on the anisotropic tensile properties that are commonly observed and reported. At both room temperature and elevated temperature of transgranular fracture, tensile properties of the as-built specimens are anisotropic: higher strength and longer elongation along the building direction than perpendicular to the building direction [92, 96, 97]. However, it has been shown that the directional agglomerate of porosities is more likely to be responsible for the experimentally observed anisotropy, rather than the intrinsic crystallographic properties [96]. A similar indication can also be given by comparison made between the tensile properties of before and after HIP specimens [95], where after HIP the anisotropy is almost ignorable while the columnar feature is still maintained, and HIP is believed to reduce the porosities inherited from manufacturing process [95, 98]. However, at elevated temperature when intergranular fracture becomes predominant, anisotropy might also be expected due to the relative orientation between the elongated columnar grain boundary and loading direction. One can simply imagine that the columnar grain boundary aligning perpendicular to the loading axis might have less resistance than aligning parallel to the loading axis.

The relatively elevated powder bed temperature can have an *in-situ* ‘heat-treatment’ effect on the built-and-solidified material during processing. That means, the as-built microstructure is spatially dependent, since the early solidified part of a build inevitably experiences longer *in-situ* ‘heat-treatment’ than the subsequently solidified part. Therefore, a microstructure gradient is observed in the as-built EBM IN718 component [94, 99–102]. However, such a segregation or Laves/ δ -phases gradient [94, 95, 101] might be unfavourable for the mechanical properties, and it is also largely dependent on the process parameters. It is possible to do controlled *in-situ* heat treatment within the build chamber by heating

the top surface to get the build peak-aged [103], which might open the other possibility to control the thermal history to *in-situ* eliminate the undesired segregation gradient.

Tailoring the process parameters to customize the microstructure is always a popular topic within this field. For example, it is possible to change the typical columnar and textured grain microstructure to equiaxed by varying the scan strategy and solidification condition [104–110]. By aligning the sample longitudinal axis deviating from the building direction, the crystallographic texture along the sample longitudinal direction can transit from $\langle 100 \rangle$ to $\langle 110 \rangle$ and $\langle 111 \rangle$, as reported in [111, 112]. The most recent work by Balachandramurthi et al. [110] has demonstrated that with tailoring the hatch strategies, it is possible to get columnar, bimodal and equiaxed microstructures with sharp microstructural transition interfaces within one single build. All these results show the great potentials to customize the microstructures for optimum properties.

5.3 SLM IN718

Grains in as-built SLM IN718 are typically less columnar and less elongated along the building direction than in as-built EBM IN718. Due to the smaller size of the laser beam and the considerably low powder bed temperature, the solidification cooling is so rapid that strengthening phases γ''/γ' are absent in the as-built condition [113–115]. Note that, such a rapid solidification cooling can also introduce large residual stress/strain in the as-built microstructure, as the subgrain cell structures or considerable dislocations are believed as the proof of residual deformation inherited from the SLM process [113, 115, 116]. In comparison to the typical textured as-built EBM counterpart, both strong $\langle 001 \rangle$ // building direction texture [117–119] and relatively isotropic crystallographic orientations [115, 120–122] have been reported. Though the mechanical properties largely depend on the specific process parameters and crystallographic textures, the general anisotropic tensile properties are common to all SLM IN718 builds: the horizontally built samples have slightly higher tensile strength than the vertically built samples [115, 117, 122–125]. Regarding the anisotropy, besides the crystallography, one should also consider that the residual stress inherited might have significant effects on the mechanical properties, specifically in the transgranular fracture cases. By the comparison between as-built and after heat treatment conditions, it seems that removing the residual stress during high temperature heat treatment can largely reduce the anisotropy [115, 126]. However, for the high temperature intergranular fracture cases, the anisotropy can be largely due to the grain boundary orientation to the loading axis, which is similar to the EBM cases.

5.4 Post treatment for AM IN718

As mentioned, typically the as-built AM IN718 is underaged, due to which the as-built strength can be further improved by applying the standard two-step ageing treatment. Besides increasing strength, it is more important to establish post

treatment for optimizing both strength and ductility, considering the possible defects/porosities, residual stress and segregation in the as-built microstructure. On the other hand, the as-built AM microstructure is significantly dependent on the specific process, and there is no well-established heat treatment for the AM counterparts. Standard specification ASTM F3055 provides a guideline for establishing heat treatment for powder-bed-fusion AM IN718, as shown in Table 5.2. Further optimizing the heat treatment should be based on the specific applications.

Table 5.2. Heat treatment recommended for powder-bed AM IN718 per ASTM F3055

Stress relief	Hot isostatic pressing	Solution + Ageing
1065±15°C for 85~105min, performed while the components are attached to the build platform	1120~1185°C at ≥100 MPa inert atmosphere for 240±60min, followed by furnace cool to ≤425°C	AMS 2774

Hot isostatic pressing (HIP) appears as a common practice for AM components to heal the residual defects/porosities, which is very important for applying AM components in critical services. HIP simultaneously applies both high temperature and isostatic inert gas pressure to components, and is effective to close the remained porosities and obtain nearly full densification. Besides healing the internal defects, HIP might also cause significant grain growth, depending on the grain boundary characteristic and mobility. For the EBM cases, the very different grain growth behaviours are observed in contour and hatch region after HIP at 1200 °C/120 MPa/4 hours: noticeable grain growth is present in the contour region but not in the hatch region [127, 128]. In addition, even for the columnar grains of EBM IN718, the coarsening behaviours can be different from one case to another, due to the presence of grain boundary carbide and its impinging effect [98, 129, 130]. Comparatively, as mentioned, the as-built SLM IN718 usually associates with large residual stress than the as-built EBM counterpart, which can provide an additional driving force for grain growth. Therefore, it is common to see more significant grain growth after HIP in the SLM IN718 than in the EBM counterpart [128, 131]. The common HIP condition for EBM IN718 [95, 98] and SLM IN718 [127, 132, 133] reported in literature is at the range of 1180 ~ 1200 °C .

Usually, the as-built SLM builds have much fewer defects or porosities than the EBM builds, and therefore HIP might be not necessary for SLM builds to close the internal porosities, while it is recommended for EBM builds. Since Laves phase is present in the as-built SLM IN718 microstructures, a homogenization step (see Table 2.4) is necessary to dissolve it if HIP is not applied. Mostly the heat treatments reported in the literature [113, 115, 117, 124, 133–137] follow or are similar to the standards that established for the forged, wrought and cast components. And after these heat treatments, the tensile properties or hardnesses of SLM IN718 are comparable to or even slightly better than the conventional counterparts. However, when it comes to high temperature long-term or time-dependent applications (e.g., creep and dwell-fatigue), these heat treatments are obviously not optimum. As mentioned that there are very complicated dislocation sub-structures in the as-built SLM IN718 microstructures, how to ‘recover’ the

dislocation-ed as-built microstructure by heat treatment seems to be an important factor for creep and dwell-fatigue properties. The homogenization temperature ($< 1100\text{ }^{\circ}\text{C}$) and duration of ($1 \sim 2$ hours) established for the cast counterpart might be able to dissolve the Laves phase and homogenize the chemical segregation, but is inefficient to eliminate the residual dislocation sub-structure and to get full recrystallization. For example, Tucho et al. [116] showed that after 7 hours at $1100\text{ }^{\circ}\text{C}$, the residual dislocation sub-structure is still obvious, while $1250\text{ }^{\circ}\text{C}$ could be very efficient to remove the residual dislocation and recrystallize. From the point of view of recovery and recrystallization, applying HIP to SLM component might be more practical to remove the crystallographic defects rather than porosity defects, since the HIP temperature is much higher than the homogenization temperature.

5.5 Fatigue and creep properties of AM IN718

Fatigue and creep properties are of interest to the AM IN718 components. As known, surface roughness is a critical factor for the fatigue performance. Due to partially-melt/sintered powders at the build's periphery, the as-built AM IN718 surface is usually worse than the conventional counterpart, and surface finish is always required to improve the fatigue resistance [138]. And the surface roughness is significantly dependent on the process parameters, but usually EBM materials have rougher surfaces than SLM counterparts, due to the smaller power and thinner power layer used in SLM process [139]. In addition to surface roughness, internal defects such as gas porosity and lack-of-fusion also play a detrimental role on the fatigue strength and statistics, since HIP is not able to close the gas porosities [139, 140] as well as the large lack-of-fusion defects. Based on these considerations, the overall fatigue performance of AM (both EBM and SLM) components seems to be commonly inferior, or at maximum comparable, to the wrought counterpart [126, 139–143]. But, if excluding the effects of surface roughness and defects, similar deformation or fracture behaviours to the wrought counterparts are expected [97, 144]. Specifically, the $\langle 100 \rangle$ crystallographic orientation has instinctively better fatigue resistance than other orientations [145], and therefore, the superiority of the typical $\langle 100 \rangle$ textured columnar EBM microstructure should be taken advantage of. Though it is less crystallographic textured of SLM IN718 material, anisotropic fatigue life is also shown in the as-built condition and in the heat-treated per ASM 5662 condition at room temperature, but such an anisotropy can be migrated by heat treatment [126].

Comparing to fatigue, grain boundary plays a more important role in creep or time-dependent deformation since the intergranular fracture is usually predominant. As mentioned above, for the elongated grain microstructure, better intergranular fracture resistance could be expected when the columnar grain boundary aligning parallel to the loading axis than aligning perpendicular to the loading axis. Such a noticeable anisotropic creep performance is reported for HIP-ed-and-heat treated EBM IN718 under 580 and 600 MPa at $650\text{ }^{\circ}\text{C}$ by Shassere et al. [146], as the columnar longitudinal direction shows lower creep rate than the transverse direction. Generally, the EBM creep properties show spread data but comparable

to the conventional counterpart, if the EBM specimens are without significant deposition defects [146].

Figure 5.2. Creep data of EBM and SLM IN718 with a comparison to a hot rolled AMS 5596 counterpart at 650 °C, showing the comparability of AM creep resistance to the conventional counterpart. The EBM creep data were adapted from [146] under 580 and 600 MPa. The SLM creep data were adapted from [132] under 550 MPa and from [147] under 650 MPa. For the specific data point and the corresponding microstructure, please refer to the references. The hot rolled AMS 5596 creep data is adapted from [148].

Popovich et al. [149] reported that the heat-treated SLM IN718 has shown inferior creep resistance to the wrought counterpart under 690 MPa at 650 °C, which might be attributed to the porosities even though the overall porosity fraction is acceptably low. Further, Kuo et al. [132, 150, 151] systematically studied the effects of post treatment (HIP and heat treatment) on creep properties of SLM IN718 under 550 MPa at 650 °C, suggesting that HIP is more efficient than heat treatment to improve the creep resistance of SLM components. Interestingly, if to carefully compare the steady-state creep rate and creep strain between EBM, SLM and C&W (cast & wrought) IN718 listed in [132, 151], one can find that even after HIP the SLM IN718 still shows relatively inferior creep resistance to the C&W counterpart, and the EBM IN718 is slightly better than the SLM IN718 but similarly inferior to the C&W counterpart. The inferiority of the EBM coun-

terpart can be due to the possibility of internal defects, since they are not HIP-ed. But for the SLM counterpart, such an inferior creep resistance can not be simply attributed to the presence of internal defects if almost porosity-free is assumed after HIP. Such an inferiority of HIP-ed SLM creep resistance is not clearly addressed in these studies, even though Kuo et al. [132, 151] claimed that HIP can introduce serrated boundaries which should be beneficial to the creep performance. Similarly, Witkin et al. [152] reported an anomalous and inferior notch rupture behaviour of HIP and heat treated SLM IN718 at 650 °C in combination stress rupture test, which shows zero elongation and is supposed to not be so if comparing to the wrought IN718. In [152] it is suggested that the relatively small grain boundary δ phase formed after HIP and standard solution treatment can not impart a beneficial resistance to notch rupture. This explanation seems plausible since grain boundary precipitate may inhibit grain boundary sliding and is beneficial for creep resistance. However, in the most recent study [147] it shows that grain boundary δ or Laves phases do promote creep voids and micro cracks and deteriorate creep resistances in SLM IN718 under 650 MPa at 650 °C.

Some of the aforementioned creep data for both EBM and SLM IN718 are summarized in Fig. 5.2 with a comparison to a hot rolled and standard heat-treated IN718. It clearly shows that 1) the EBM IN718 creep behaviour is more similar and comparable to the conventional counterpart than the SLM IN718, and 2) the minimum creep rate of SLM IN718 seems to not really depend on stress, which is unexpected and illogical. So far, only Pröbstle et al. [120] reported a superior creep strength for SLM IN718 under 900 MPa at 630 °C comparing to a C&W counterpart. For now, probably no a common conclusion or agreement can be reached for the creep properties of AM IN718 with such limited and contradictory data, further studies are of importance to address this issue.

CHAPTER 6

Experimental methods

The experimental methods used in the research of this thesis will be present in this chapter. The EBM IN718 materials were provided by Sandvik, Sweden and the SLM IN718 materials were provided by Siemens AG, Germany. The post heat treatments, metallographic preparations, microstructure characterizations, hardness measurements, tensile tests and dwell-fatigue crack propagation tests were performed at Linköping University.

6.1 Materials

6.1.1 EBM IN718

The EBM IN718 was manufactured with an Arcam A2X EBM machine at Sandvik Machining Solutions AB. The powders used were plasma atomized, with nominal size ranging from 25 to 106 μm . The chemical composition is given in Table 6.1. The process parameters were set as suggested by Arcam AB, and the powder bed temperature (measured under the base plate) was kept at about 1020 °C throughout the process. The manufacturing batch contained 16 identical blocks. Each block is dimensioned as in Fig. 6.1a. Note that all these blocks were provided in the as-built condition, directly removing from the base plate without any treatment. For more information about this process please refer to appended paper I.

6.1.2 SLM IN718

Gas atomized IN718 powders with the nominal size less than 65 μm were used as the raw material for the SLM process. The nominal chemical composition

Figure 6.1. Schematics of as-built (a) EBM and SLM IN718 blocks and (b) SLM IN718 rectangle bars.

Table 6.1. Nominal chemical composition of the Arcam plasma atomized IN718 powder

Element	Ni	Cr	Fe	Nb	Mo	Co	Ti	Al
wt.%	Bal.	19.1	18.5	5.04	2.95	0.07	0.91	0.58
Element	Mn	Si	Cu	C	P	S	N	O
wt.%	0.05	0.13	0.1	0.035	0.004	0.001	0.0128	0.0133

of the gas atomized IN718 powder is given in Table 6.2. All the samples were manufactured with an EOS M290 machine equipped with a maximum 400 W Yb-fiber laser. The process parameters were as recommended by EOS. The SLM IN718 specimens were provided in the geometries of blocks (Fig. 6.1a) and rectangle bars (Fig. 6.1b). The rectangle bars' longitude direction is either parallel to or perpendicular to the base plate, designating as horizontally built and vertically built, respectively. Note that all these blocks and bars were provided in the as-built condition, directly removing from the base plate without any treatment. For more information about this process please refer to appended paper III.

Table 6.2. Nominal chemical composition of the gas atomized IN718 powder

Element	Ni	Cr	Fe	Nb	Mo
wt.%	50~55	17.0~21.0	Bal.	4.75~5.5	2.8~3.3
Element	Co	Ti	Al	Mn	Si
wt.%	<1.0	0.65~1.15	0.20~0.80	<0.35	<0.35
Element	Cu	C	P	S	B
wt.%	<0.3	<0.08	<0.015	<0.0015	<0.006

6.2 Microstructure characterization

6.2.1 Metallographic preparation

For scanning electron microscopy characterization, samples were cut at the interested plane and mounted, then mechanically ground successively from 500 Grit to 4000 Grit, and polished with diamond suspension from 3 μm to 1/4 μm and finally with OP-U colloidal silica suspension. Specifically for the SLM IN718, to reveal the dendritic microstructure, the polished samples were etched for a few seconds using a 10 ml hydrochloric acid + 1.5 ml 30% hydrogen peroxide etchant. For transmission electron microscopy characterization, thin foil samples were prepared by mechanically grinding down to 50 μm . To further thin down to electron transparent, electropolishing was conducted in 10 vol.% perchloric acid + 90 vol.% Ethanol electrolyte at -20 °C with a Struers TenuPol-5 electrolytic machine, or ion milling was performed with a Gatan 691 Precision Ion Polishing system.

6.2.2 Scanning electron microscopy

A Hitachi SU70 FEG scanning electron microscope (SEM) equipped with energy dispersive X-ray spectroscopy (EDS) and electron back scatter diffraction (EBSD) system from Oxford Instrument, was operated at 10~20 kV for microstructure characterization.

6.2.3 Transmission electron microscopy

The transmission electron microscopy (TEM) characterization was performed using a FEI Tecnai G2 TEM, operating at an accelerating voltage of 200 kV. A double tilt holder was used for all the TEM experiments to tilt the sample to desired zone axis for better imaging.

6.3 Mechanical test

6.3.1 Hardness test

A Struers DuraScan G5 hardness tester was used to measure the Vickers microhardness with 300 g load and 15 s dwell-time. For each sample no less than 20 indentations were performed to get good statistics.

6.3.2 Tensile test

Tensile test samples were electrical discharge machined with reference to the building orientation, namely parallel/perpendicular to the building direction, to investigate the sample orientation dependence of tensile properties. For the geometries of tensile test samples please refer to the appended papers. 3~4 samples were tested per test condition to obtain acceptable statistics. Tensile tests were conducted under room temperature and open air, using an Instron 5582 universal test machine

with a 100 kN load cell and at a 0.10 %/s strain rate. Since the test sample was too small to measure strain with extensometer, the strain was measured using a digital image correlation (DIC) system from Image System AB.

6.3.3 Dwell fatigue crack propagation test

Compact tension (CT) geometry was adopted in the present study to investigate the crack propagation behaviours. Two sample orientations were tested to investigate the anisotropic cracking resistance: the N-type has the machined notch normal to the building direction, while the P-type has the machined notch parallel to the building direction, as shown in Fig. 6.2a and b. These CT specimens were first pre-cracked at room temperature under pure-fatigue condition to generate a pre-crack of about 1.5 mm in length from the electrical discharge machined (EDM) notch tip. The pure-fatigue loading was under the load ratio of $R = P_{min}/P_{max} = 0.05$ and the cyclic frequency of 10 Hz.

Dwell fatigue crack propagation tests were conducted in a 100 kN Zwick KAPPA 50 DS system at 550 °C in laboratory air. Each cycle includes 10 s ramping up to the maximum load, 2160 s dwell period at the maximum and 10 s ramping down to the minimum load. The load ratio was $R = P_{min}/P_{max} = 0.05$. During testing, the crack length was measured using a direct current potential drop (DCPD) system with a pulsed 10 A current. The crack length was first derived from the potential drop measurements using the Johnson analytical formula [153] for CT specimen, assuming that a straight through-thickness crack front was maintained during the test. Later, the crack length was corrected by fracture surface inspection: the pre-crack front and the dwell-fatigue crack front were identified, and then the dwell-fatigue crack growth was averaged over about 20 measurements at different locations on the fracture surface. The stress intensity factor range ΔK was also calculated per ASTM E647.

Figure 6.2. (a) and (b) compact tensile (CT) geometry with the notch normal and parallel to the building direction (BD), respectively.

CHAPTER 7

Summary of appended papers

Paper I

Microstructure and anisotropic mechanical properties of EBM manufactured Inconel 718 and effects of post heat treatments

This paper is partly motivated to investigate the microstructures corresponding to contour and hatching regions and the mechanisms behind the anisotropic mechanical properties. Tuning the post heat treatment with comparison to the AMS 5662 specification is also of interest.

The contour microstructure is characterized as heterogeneous grain morphologies and the overall weak texture, while the hatch region is mostly coarse columnar grains elongated along the building direction and has strong $\langle 001 \rangle$ // building direction texture. The anisotropic tensile properties are observed as higher tensile strength but lower elongation along the building direction than normal to the building direction. However, the anisotropy does not seem to mainly result from the $\langle 001 \rangle$ // building direction texture, but instead is possibly attributed to the alignment and distribution of porosities. Since the as-built microstructure has already been strengthened by the strengthening phases γ'/γ'' , heat treatments just slightly increase the strength, and the direct ageing without solution treatment seems to be the optimum post heat treatment.

Paper II

On the formation of microstructural gradients in a nickel-base superalloy during electron beam melting

This paper is a sequel of the first paper, with focus on characterizing the as-built microstructure gradient along the building direction and rationalizing the phase evolution during the complex thermal cycles.

Due to the relatively high powder bed temperature, there is actually an ‘in-situ’ annealing during the EBM process. The previously processed layers experience longer ‘in-situ’ annealing than the subsequently deposited layers within one build, resulting to microstructural gradient shown in the as-built sample: from the top surface towards the bottom, the Laves phase volume fraction increases from about zero, peaks to about 2.3% at 150 μm from the top surface, and decreases gradually to zero again at 1800 μm from the top surface. With this gradient, we sectioned the as-built microstructure into three regions: as-solidified, partly homogenized and fully homogenized regions. With the identified regions, the solidification and ‘in-situ’ annealing conditions during the EBM process can be roughly deduced. The estimated time to fully dissolve the Laves phase of 2.3 % present in the as-solidified microstructure is 40 minutes, for which duration the average or effective thermal condition might be much higher than 1020 °C which is the powder bed temperature measured under the base plate, and specifically at the beginning of ‘in-situ’ annealing the temperature might even go close to 1200 °C. Such a temperature is above the Laves precipitation temperature window predicted with Scheil model. This can significantly affect the solidification thermal condition and contribute to segregation and precipitation of Laves phase in the as-solidified microstructure. The precipitations of γ'/γ'' and δ are during the cooling down after the building process is finished, but the cooling thermal condition can not yield the peak-aged precipitation of γ'/γ'' due to the sluggish precipitation kinetics.

Paper III

Microstructure and mechanical properties of Inconel 718 produced by selective laser melting: Sample orientation dependence and effects of post heat treatments

Aims of this paper are to characterize the as-built microstructure and optimize the post heat treatment for SLM IN718, regarding to homogenizing the segregation and correctly precipitating the strengthening phases for peak strength. Also this paper is also motivated to uncover the mechanisms for the ‘isotropic crystallographic orientations but anisotropic mechanical properties’ behaviour.

The as-built SLM IN718 has a very fine cellular-dendritic subgrain structure, with numbers of fine Laves phases precipitating in the interdendritic region and relatively weak texture. Though no strengthening phases γ'/γ'' precipitated in the as-built condition, the strength is still higher than that of fully solution treated condition (which is also without γ'/γ'') reported in the literature, due to the ‘work-hardening’ by residual strain/dislocations. The horizontally built samples have higher tensile strength but lower elongation than the vertically built samples. These anisotropic tensile properties are mainly attributed to the different amounts of residual stress accumulated, with increasing the heat treatment temperature or duration the differences of tensile properties are minimized. From the point of

view of homogeneous microstructure and high tensile strength, it is recommended to prolong the homogenization duration at 1080 °C but reducing the holding time of solution at 980 °C.

Paper IV

On the dwell-fatigue crack propagation behavior of a high strength superalloy manufactured by electron beam melting

This paper is motivated to preliminarily characterize the anisotropic intergranular dwell-fatigue cracking behaviours of EBM IN718 under the 2160s-dwell condition at 550 °C, and do comparison with a forged counterpart to show the inferiority or superiority of the typical EBM columnar microstructures.

The dwell-fatigue cracking tests have shown anisotropic cracking resistances between the two orientations: P-type (notch parallel to the building direction/columnar grain boundaries) is inferior to N-type (notch normal to the building direction/columnar grain boundaries). Intergranular cracking is the predominate fracture mode for both orientations. However, in the N-type orientation, the intergranular crack is propagating along the loading direction, which gives lower crack propagation driving force than the P-type orientation with the intergranular cracking perpendicular to the loading direction, and therefore leading to the anisotropy. The cracking mechanism is believed as environmental-assisted grain boundary damage, and is the typical mechanism well-agreed for IN718 under the temperature of 550 °C. With comparison to the forged counterpart, the EBM IN718 shows superior dwell-fatigue cracking resistance, which can be attributed larger grain size, lower fraction of random high angle grain boundary and less grain boundary δ precipitates. The direct ageing and standard solution + ageing heat treatments applied in this study seems to not significantly affect the dwell-fatigue cracking resistance, since the differences of grain size, grain boundary characteristics and grain boundary δ after these two heat treatments are ignorable.

Paper V

On the dwell-fatigue crack propagation behavior of a high strength Ni-base superalloy manufactured by selective laser melting

This paper is initially motivated to do the similar dwell-fatigue cracking behaviour study of heat-treated SLM IN718 as for the EBM IN718. After careful comparison of the SLM IN718 results with the forged counterpart, this paper is further driven to identify the dwell-fatigue cracking mechanism and the possibility of having creep damage at the temperature of 550 °C.

Three heat treatment conditions (SA, HA and HSA) and two sample orientation (P-type and N-type) of SLM IN718 and are included in this study. Intergranular cracking is the predominate fracture mode in both P-type and N-type specimens. Since the intergranular crack path is about 70 ° deviated from the pre-

crack plane in the N-type specimens, the effective stress intensity factor is lower and the dwell-fatigue cracking resistance is better than the P-type specimens. By comparison with the data from literature, the dwell-fatigue cracking resistance of SLM IN718 is inferior to the forged counterpart, and the cracking mechanism is possibly creep damage, which might be unusual at the temperature of 550 °C. A general discussion on the inferior creep resistance of SLM IN718 has been made and suggested that, the largely deformed microstructure that inherited from the SLM process and can not recover by the heat treatments might be the cause, since it might be easier to creep a largely deformed microstructure.

Paper VI

A comparison study of the dwell-fatigue behaviours of conventional and additive Inconel 718

This paper is motivated to systematically compare the different dwell-fatigue behaviours of EBM, SLM and forged IN718 at 550 °C, of which the microstructures are significantly different. The other motivation is to find the detailed microstructure evidence for the creep damage of SLM material at 550 °C, which was raised in Paper V. In addition, by detailed comparison on the microstructures, this paper also aims to find a general conclusion on how the dislocation substructures correlate to the dwell-fatigue damage mechanisms.

Heat treatments and materials included in this study are EBM SA hatch, EBM SA contour+hatch, forged, SLM 48HSA and SLM HSA, which are arranged in a sequence of low to high cracking propagation rate. Crack paths and microstructures are examined and compared to indicate the possible damage mechanisms. It shows that the typical SLM cell substructure is associated with well-order dislocation network at the cell boundary and random dislocations in the cell interior. Such a dislocation substructure is similar to that in the tertiary-creep regime reported in literatures, which can easily result in grain boundary voids and creep fracture. If without such a dislocation cell substructure, the material would have environmental-assisted grain boundary cracking instead of creep damage. In the EBM hatch microstructure, dislocation network is also observed but is possibly in a equilibrium state as low angle grain boundary. Such a low angle grain boundary dislocation network does not affect environmental-assisted cracking propagation, but the random and tangled dislocations do, by affecting the crack tip blunting.

Conclusion & Outlook

Based on the studies aforementioned, the following conclusions can be drawn and answer the research questions raised in Chapter *Introduction*.

For EBM IN718:

- The as-built microstructure is not homogeneous, but instead is location-dependent. The contour region of the build is mixed of columnar and equiaxed grains and shows overall weak texture, while the hatch region has mostly coarse columnar grains elongated along the building direction with strong $\langle 001 \rangle$ texture. By the gradient of Laves fraction shown along the building direction, the build can be divided into three regions from the point of ‘in-situ’ annealing view along the building direction: as-deposited, partially homogenized and fully homogenized.
- The tensile test shows higher tensile strength but lower elongation along the building direction than perpendicular to the building direction. The anisotropy results possibly from the alignment of porosities rather than the crystallographic texture.
- The contour region of the EBM build gives inferior dwell-fatigue cracking resistance to the hatch region at 550 °C under the 2160 s dwell condition, but the overall dwell-fatigue cracking resistance of EBM build is better than that of forged counterpart.

For SLM IN718:

- The as-built SLM IN718 has very fine dendritic microstructure and weak texture, with very fine Laves phase in the interdendrites and without strengthening phases γ'/γ'' in the matrix.

- The different amounts of residual stress accumulated are responsible for the ‘anisotropic’ tensile properties, namely the horizontally built samples have higher strength but lower elongation than the vertically build samples.
- In the present thesis, heat treatments applied and studied are basically the same for the conventional counterpart. A homogenization step at 1080 °C of 1 hour can significantly remove the segregation and Laves phase, but not the subgrain cell structure. No noticeable recrystallization or grain growth happened during these these heat treatment. The subgrain cell structure might be beneficial for tensile strength but probably not for time-dependent mechanical properties at high temperature. The optimization of heat treatment for SLM counterpart still needs further work with the specific consideration for service condition.
- Generally the heat-treated SLM IN718 studied show inferior dwell-fatigue cracking resistance to the forge counterpart at 550 °C under the 2160s dwell condition. The subgrain cell structure inherited from the manufacturing process seems to indicate sever plastic deformation and can easily render the material to tertiary creep at the temperature of 550 °C.

It is doubtless that there is still a lot of work needed to better understand the microstructures and mechanical properties of AM IN718 components for different service conditions, especially for the high temperature and time-dependent application interested. Specifically, for the SLM IN718, the subgrain cell structure inherited from the manufacturing process is not similar to any of conventional IN718 under any deformation processes, to the best of the author’s knowledge. How to correlate such a microstructure to the mechanical behaviours might deserve further work, since it can not be simply or straightforward based on our knowledge on the conventional counterpart. By comparison, the hatch and contour grains of EBM IN718 might be more closer to that of conventionally directional solidification (DS) and forged ones respectively, from which the references on the mechanical behaviours can be more straightforward. So far, all the high temperature mechanical tests in present thesis are at 550 °C, and it is of interest to extend these tests to 650 °C to see the applicability of the AM component at the maximum service temperature for IN718. In addition, optimizing heat treatment routines for AM counterparts is vital for the desired mechanical properties in the future work. It is clear that the heat treatment plays an important role on the recover and recrystallization behaviours on the AM microstructure and on the time-dependent mechanical behaviours, given that there might be considerable amount of dislocations inherited from AM process. Also, considering the different processes parameters of hatch and contour, the microstructures and heat treatment responses in the corresponding regions are different, which might require an optimized heat treatment to take consideration on the duplex microstructures within one build for the favourable mechanical properties. The further understanding on AM IN718 might also pave the way for successful application of AM to non-weldable Ni-base superalloys.

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