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**HIGH CYCLE FATIGUE CHARACTERISATION AND
MODELLING OF 316L STAINLESS STEEL
PROCESSED BY LASER POWDER BED FUSION**

ZHANG MENG

**SCHOOL OF MECHANICAL AND AEROSPACE
ENGINEERING**

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**SCHOOL OF MECHANICAL AND AEROSPACE
ENGINEERING**

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

2019

Statement of Originality

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

... 24 July 2019 ...

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Supervisor Declaration Statement

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

24 July 2019

Date

A/Prof Li Hua

Authorship Attribution Statement

This thesis contains material from 6 paper(s) published in the following peer-reviewed journal(s) / from papers accepted at conferences in which I am listed as an author.

Chapters 4.1 – 4.3 are published as M. Zhang, C.-N. Sun, X. Zhang, P.C. Goh, J. Wei, D. Hardacre, H. Li, Fatigue and fracture behaviour of laser powder bed fusion stainless steel 316L: Influence of processing parameters, *Mater. Sci. Eng., A* 703 (2017) 251-261, 10.1016/j.msea.2017.07.071.

The contributions of the co-authors are as follows:

- I designed the study and performed the laboratory work at the School of Mechanical and Aerospace Engineering of the Nanyang Technological University. I also analysed the data and prepared the manuscript drafts.
- Sample preparation was done with the assistance of Dr Sun Chen-Nan.
- Dr Sun Chen-Nan, Prof Zhang Xiang, Dr Goh Phoi Chin, Dr Wei Jun, Mr David Hardacre and A/Prof Li Hua reviewed and edited the manuscript drafts.

Some of the results reported in Chapters 4.1 – 4.3 are also published in M. Zhang, C.-N. Sun, X. Zhang, P.C. Goh, J. Wei, D. Hardacre, H. Li, Competing influence of porosity and microstructure on the fatigue property of laser powder bed fusion stainless steel 316L, Solid freeform fabrication symposium, University of Texas Austin, Texas, 2017.

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Zhang Meng

ABSTRACT

Laser powder bed fusion (L-PBF) is an additive manufacturing (AM) process that uses high power lasers to selectively melt metal powders in a layerwise manner. It introduces unique process-structure-property relationships, understanding of which is essential for producing structural parts with consistent acceptable properties. In view of the ongoing efforts towards the development of standardised approaches for the determination of suitable processing conditions and part quality for L-PBF-processed parts, this PhD research aims to clarify links of the process-structure-property triad and develop methods for material property assessment.

Particular attention was paid to the high cycle fatigue properties because of the strong influence of manufacturing defects on fatigue crack initiation and the high cycle fatigue resistance. Using stainless steel 316L as the model material, the critical crack-initiating defects under controlled changes in processing parameters were examined. The influence of processing on defect formation and the mechanisms of fracture were elucidated. Several guidelines for assessing the criticality of defects and the fatigue properties were highlighted.

The effects of fatigue loading condition were considered for analysing the process-structure-property relationships. In the presence of tensile mean stress, variations in the plastic deformation properties, as demonstrated by the effects of build orientation and post-processing treatments (annealing or hot isostatic pressing), were found to induce cyclic plasticity, which led to strong fatigue-ratcheting interactions in the high cycle fatigue regime. The simultaneous actions of ratcheting and fatigue generate complex nonlinear relationships between the stress amplitude and mean stress parameters, such that the fatigue properties could not be effectively predicted using traditional stress-based models. A modification to the Goodman relation was proposed to account for the added effects of cyclic plasticity.

Understanding of the fatigue and fracture behaviours served as the physical basis for the development and validation of predictive models via the ‘process-property’ and ‘structure-property’ routes. Statistical models, based on the response surface method,

were developed for approximating the process-property relationship, while the structure-property relationship was modelled by considering the fatigue failure modes. The latter approach allowed the specification of a reduction factor on fatigue life to account for the intrinsic effects of microstructure inhomogeneity, as well as the construction of a modified Basquin equation that incorporates the defect fraction parameter for predicting the effects of lack of fusion defects on the S-N properties.

Lastly, considering the numerous influencing factors arising from the process and the associated failure behaviours, a neuro-fuzzy-based machine learning method was applied to provide an effective unifying approach for high cycle fatigue life prediction. A dataset, consisting of most of the experimental fatigue data obtained in this study, was constructed for simulating a complex nonlinear input-output environment. Two models, by employing the processing/post-processing parameters and the static tensile properties respectively as the inputs, were developed from the training data. Despite the diverse fatigue and fracture properties, the models produced good prediction accuracy when checked against the test data. Moreover, the method demonstrated good generalisation capability on applying to literature data. The strong predictive power exemplifies the use of data science as a convenient and effective alternative to the traditional fatigue modelling approaches.

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Journal papers:

1. M. Zhang, C.-N. Sun, X. Zhang, P.C. Goh, J. Wei, D. Hardacre, H. Li, High cycle fatigue life prediction of laser additive manufactured stainless steel: A machine learning approach, *Int. J. Fatigue* 128 (2019) 105194.
2. M. Zhang, C.-N. Sun, X. Zhang, J. Wei, D. Hardacre, H. Li, High cycle fatigue and ratcheting interaction of laser powder bed fusion stainless steel 316L: Fracture behaviour and stress-based modelling, *Int. J. Fatigue* 121 (2019) 252-264.
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2. M. Zhang, C.-N. Sun, X. Zhang, J. Wei, D. Hardacre, H. Li. Review of fatigue fracture behaviours of materials made by laser powder bed fusion, 3rd International Conference on Progress in Additive Manufacturing, Singapore, 2018.

3. M. Zhang, C.-N. Sun, X. Zhang, P.C. Goh, J. Wei, D. Hardacre, H. Li, Effect of heat treatment on fatigue crack initiation of laser powder bed fusion stainless steel 316L, MATEC Web of Conferences, EDP Sciences, p. 22006, for 12th International Fatigue Congress, Poitiers, France, 2018.
4. M. Zhang, C.-N. Sun, X. Zhang, P.C. Goh, J. Wei, D. Hardacre, H. Li, Competing influence of porosity and microstructure on the fatigue property of laser powder bed fusion stainless steel 316L, Solid Freeform Fabrication Symposium, University of Texas Austin, USA, 2017.
5. M. Zhang, H. Li, X. Zhang, D. Hardacre, Review of the fatigue performance of stainless steel 316L parts manufactured by selective laser melting, 2nd International Conference on Progress in Additive Manufacturing, Singapore, 2016.

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LIST OF SYMBOLS

a	radius of equivalent spherical defect/crack length
a_0	critical defect size in the El Haddad equation
A_s	melt pool surface area
b	fatigue strength exponent
c	fatigue ductility exponent
c_0, c_1, c_2	parameters of the modified Goodman equation
e	elongation to failure
E_d	volumetric energy density
f	linear consequent function of the fuzzy model
F	geometric factor
HT	post-processing treatment
h	hatch spacing
k	friction stress of dislocation along slip band
K	parameter of the modified Goodman equation
L	melt pool length
N_f	cycles to failure
N_i	crack initiation life
O_i^j	output of the i th node of the j th layer of the ANFIS
P	laser power
P_0	standard laser power setting
p	area fraction of defect
q, r, s, t	consequent parameters of the fuzzy model
R	load ratio
T	layer thickness
T_0	standard layer thickness setting
v	laser scan speed
v_0	standard scan speed setting
W_G	specific fracture energy per unit area of grain boundary
W_I	specific fracture energy per unit area of inclusion

W_s	specific fracture energy per unit area along slip band
α	mean of the Gaussian distribution
β	standard deviation of the Gaussian distribution
δ	Gumbel scale parameter
ε_{ap}	plastic strain amplitude
ε_{at}	total strain amplitude
ε_f'	fatigue ductility coefficient
η	exponent of σ_{max}/σ_b for the modified Goodman equation
η'	exponent of σ_{max}/σ_b for the modified Goodman equation considering load ratio effect
γ	Euler-Mascheroni constant
λ	Gumbel location parameter
σ_a	stress amplitude
σ_a^{eq}	equivalent stress amplitude at fully-reversed condition
σ_b	ultimate tensile strength
σ_e	fatigue limit
σ_{e0}	fatigue limit of defect free component
σ_f'	fatigue strength coefficient
σ_m	mean stress
σ_{max}	maximum stress
σ_{open}	crack opening stress
$\sigma_{0.2}$	yield strength
$\Delta\varepsilon_p$	plastic stain range
ΔK	stress intensity factor range
ΔK_{eff}	effective stress intensity factor range due to crack closure
ΔK_{th}	threshold stress intensity factor range
Δm_i	amount of alloy i vaporised during L-PBF for
ΔT	difference between peak melt pool and surrounding temperatures
$\Delta\gamma_p$	plastic shear strain range
$\Delta\sigma$	stress range
$\Delta\tau$	shear stress range
ε_{max}	maximum strain of a cycle

$\varepsilon_{\min}$	minimum strain of a cycle
θ	volumetric thermal expansion coefficient
μ or μ'	shear modulus
μ_a	membership function for P
μ_b	membership function for ν
μ_c	membership function for T
μ_d	membership function for HT
μ_A	membership function for σ_b
μ_B	membership function for e
μ_C/μ_e	membership function for $\sigma_{\max}$
ϕ	laser absorption coefficient
ω_i	firing strength of the i th rule
ϖ_i	normalised firing strength of the i th rule
χ	thermal diffusivity

CHAPTER 1 INTRODUCTION

Laser powder bed fusion (L-PBF) is an additive manufacturing (AM) process that operates by the layerwise consolidation of metal powders using high power lasers. Computer-aided design (CAD) data serves as the input for controlling the movement of the lasers, enabling the realisation of ‘complexity for free’ and is driving the transition of the L-PBF process from rapid prototyping to the production of actual functional parts. This chapter provides an introduction to the technology and the motivation for this work. The objectives, scope and the outline of the thesis are also presented.

1.1 Background

A typical L-PBF system, consisting of the laser source, powder delivery unit, build platform/base plate and gas flow controller, is shown in Figure 1-1. During L-PBF processing, regions of the pre-deposited layer of powder are melted according to controlled laser movements; upon completion of a layer, the build platform lowers and a new layer of powder is deposited for printing of the subsequent layer. This process is done in an inert environment with properly maintained temperature and pressure to prevent contamination. Nd:YAG and Yb:YAG fibre lasers are used as the power source by most current systems due to the good absorptivity of metal at the operating wavelengths of these lasers and the better laser penetration depth [1].

The design flexibility enables the application of the technology in high value-added industries requiring products with complex geometry or customisation. The medical sector is one of the successful adopters of AM, where customised implants and orthodontic products meeting the required surface quality and mechanical properties had been made [2, 3]. The process is also suited for the production of high compressibility lattice structures, where the significant reduction of component weight is desired [4, 5]. In terms of the compatibility of traditional materials with the process, various industrial materials such as aluminium alloys and stainless steels have been successfully fabricated by metal AM processes such as the L-PBF; parts

made of superalloys [6] and functionally graded materials have also been produced [7-9] for meeting material requirement in harsh environments.

Figure 1-1 Schematic illustration of a typical L-PBF system setup. Image taken from the work of Osakada et al. [10].

However, as the industry moves towards the wider adoption of AM, the qualification of AM parts is met with many challenges rooted in the complex material-process-property relationship. As the forming principle of L-PBF is fundamentally different from the conventional subtractive approaches, one of the primary concerns lies in the influence of the process-induced microstructure and defects on the structural integrity and performance. Microstructure features such as laser tracks, melt pools and rapid-cooling-induced particles are potential weaknesses in the material that could promote cracking and premature failure. Defects, in the form of voids, are typically found in L-PBF parts can could act as stress-risers which compromise the properties of the material. In comparison with wrought materials which generally contain less than 0.1% porosity, defect content in L-PBF can be much higher [11]. In some cases, regions with defect content nearly one order higher than the global value had been detected by x-ray computed tomography (XCT) [11]; voids on the order of several hundreds of microns had been observed in near fully dense parts due to process instabilities [12]. Besides, such defects can also be formed under sub-optimal processing conditions, where under- or over-melting had been reported to cause the formation of lack of fusion defects, balling defects [13, 14], keyholes [15] and spatter particles

[16]. Small spherical gas pores are nearly unavoidable as they are pre-existing in the powder feedstock because of processing by gas atomisation. In addition, the layer-by-layer process is known to be associated with strong anisotropy, where interlayer defects are oriented in the direction of the build plane [17, 18].

The microstructure and defects had been demonstrated to affect the mechanical properties, in particular the fatigue properties, of L-PBF-processed parts. Results from existing studies showed that defects, such as lack of fusion defects and poor surface finish, are the major contributing factors for the premature failure of L-PBF parts by causing fatigue crack initiation [19-26]. As current L-PBF systems favour the fabrication of small components, for which the initiation of fatigue crack occupies a significant portion of the total life, the presence of such processing defects is detrimental to the fatigue resistance. Considering that existing non-destructive inspection techniques are still in a stage of maturation and are unable to effectively detect L-PBF defects [27], the associated uncertainties in the mechanical performance could hinder the adoption of the technology for structural applications.

From the industry's perspective, it is of utmost importance to be able to quantify the criticality of the defects for the purpose of component certification and quality assurance. Knowledge of the failure-critical defects allows precautions to be made at the upstream processing stage, as downstream remediation is often difficult and expensive. Such understanding will also aid the implementation of suitable non-destructive examination techniques and in-situ process monitoring systems without incurring unnecessary costs. The effects of remediation measures, such as heat treatment and hot isostatic pressing, for ensuring the consistency of performance also need to be understood for completing the process chain.

1.2 Objectives and scope

This PhD research studies the high cycle fatigue properties, i.e. the $10^4 - 10^6$ cycles region, and fracture behaviours of L-PBF-processed parts in relation to the process-structure-property relationship. The model material for this research is stainless steel 316L, a marine-grade austenitic stainless steel with many fatigue-related applications,

e.g. in medical implants [28], heat exchangers [29, 30], furnace parts and jet engine parts. The main challenge of this research lies in addressing the complex effects of L-PBF processing on the fatigue properties. In view of this, the objectives of the PhD research have been constructed as follows:

1) Establishment of the high cycle fatigue properties and major modes of fatigue fracture

The complex process-structure-property triad is decoupled by examining separately the process-structure and structure-property links. Understanding of the process-structure relationship under controlled changes in build parameter settings is aided by literature studies, while the structure-property relationship is analysed in terms of the crack initiation mechanism. For the latter, critical process-induced defects are characterised and the failure modes corresponding to the critical defects are identified. This allows the critical processing conditions in relation to fatigue failure to be recognised.

2) Investigation of the influence of fatigue loading, i.e. mean stress effects, on the high cycle fatigue properties and fracture behaviours

Depending on the loading condition, processing/post-processing factors that introduce variations in the plastic deformation properties could impact the fatigue response by incurring the cyclic plastic deformation behaviour. The mechanisms of fatigue-ratcheting interaction are investigated for parts made with different build orientation (horizontal and vertical) and subjected to post-processing treatments (annealing or hot isostatic pressing). The results allow evaluation of the applicability of traditional stress-based fatigue models and the development of modified models for describing the S-N properties of L-PBF variables that induce the cyclic plastic deformation response.

3) Development of predictive models for high cycle fatigue property assessment

Models for fatigue prediction are constructed based on the ‘process-property’ and ‘structure-property’ approaches, employing the traditional response surface method and the stress-based fatigue modelling approach respectively. The models

are evaluated in terms of the generalisation capability, leading to the exploration of a data science technique as an alternative to the traditional modelling approaches for tackling the complexity and variability associated with the L-PBF process.

Figure 1-2 summaries the scope of the PhD research in relation to the ‘process-structure-property’ framework. Samples made by varying the L-PBF process parameters and post-processing treatment conditions were used for experimental investigation of the structure characteristics and mechanical properties. The influence of process-induced defects and microstructure on the fatigue fracture mechanism and S-N properties, together with suitable approaches for high cycle fatigue modelling, are discussed. The novelty of the research is in the application of the proposed methodologies for assessing the high cycle fatigue properties of L-PBF materials. The results could enhance understanding of the mechanical properties, thereby facilitating the adoption of the technology for structural applications.

Figure 1-2 Overview of the research scope under the ‘process-structure-property’ framework.

It is to be highlighted that in this research, examination of the fracture mechanism concerns fatigue phenomena at the microscopic and sub-microscopic levels, i.e. features that can be observed with an optical microscope or scanning electron microscope; sub-nanometre features such as slip bands and dislocation structures are not part of the scope of this study. This is because with existing understanding of the fatigue phenomena, studies at the scale of dislocation structures tend to be qualitative in nature and are unable to provide solutions to practical engineering problems [31]. As this research aims at evaluating the capability of L-PBF for processing materials intended for fatigue-related applications, fracture mechanisms at the dislocation level are not of immediate concern. In addition, it is to be noted that the propagation of small cracks is different from the propagation of large macroscopic cracks, such as cracks induced at component design features like holes and sharp corners. This thesis focuses only on the effect of process-induced defects which are typically on the order of one to several hundred micrometres.

1.3 Thesis outline

This thesis consists of seven chapters. Following a brief introduction of the background, objectives and scope in this chapter, a review of metal fatigue fracture and the influence of L-PBF processing on the high cycle fatigue properties and crack initiation of stainless steel 316L, is presented in Chapter 2. Chapter 3 describes the procedures for sample fabrication, microstructure and defect characterisation and mechanical testing for conducting the experimental studies in this research. The influence of processing defects and microstructure on the fatigue properties and fracture behaviours of as-built L-PBF stainless steel 316L is presented in Chapter 4. Chapter 5 investigates the roles of cyclic plasticity in fatigue and fracture from the perspective of fatigue-ratcheting interactions. Chapter 6 discusses predictive models for the high cycle fatigue property assessment. Key findings of the PhD research and recommendations for future work are summarised in Chapter 7.

CHAPTER 2 LITERATURE REVIEW

In this chapter, the mechanisms of fatigue failure and methods for fatigue life/strength estimation, focussing on the roles of microstructure and defects on fatigue crack initiation, are first introduced. A comparison between the high cycle fatigue properties of L-PBF stainless steel 316L and the conventionally-manufactured form is then made, followed by a review of the fatigue properties of the L-PBF form in relation to the process-structure-property relationship. Based on the review, research gaps, aiming at enabling the L-PBF technology for processing materials intended for fatigue-related applications, are identified.

2.1 Metal fatigue: A brief introduction

Fatigue is the localised and progressive damage experienced by a material subjected to cyclic loading. It is estimated that fatigue accounts for nearly 90% of mechanical service breakdowns, where catastrophic failure events had resulted in severe damages to properties, reduced productivity and loss of lives [32, 33]. Advances in the knowledge and tools for fatigue analysis have been driven for enabling the use of materials for safety-critical applications, as well as the ongoing demand for high performance materials, such as in the aerospace, nuclear power and automotive industries.

The mechanisms associated with fatigue were not understood until the remarkable studies conducted by Ewing and Humfrey in 1902 [34], which demonstrated the connection between the generation of slip bands and microcrack formation for a material subjected to rotating-bending fatigue. As shown in Figure 2-1, under cyclic loading, the stages leading to failure start with the movement of dislocations induced by the local plastic strain. The emergence of the dislocations at the external interface of the material results in the formation of slip steps, which, under repeated loading cycles, accumulate to form surface intrusions and extrusions. Such surface features create high stress concentration areas which promote the initiation of microcracks. Besides, dislocation pileup could occur in the presence of obstacles, e.g. inclusions and second-phase particles, where the instability associated with the higher strain

energy can lead to microcracking. The growth of small cracks (Stage I crack growth) ensues, and the continuous growth and coalescence of the microcracks produce macrocracks, which propagate (Stage II crack growth) and cause the eventual failure of the material.

Figure 2-1 Stages of fatigue failure process, adapted from ASM Metal Handbook [35].

The total fatigue life of a part is equal to the summation of the amount of time spent by the material in each of the failure stages. The lifetime associated with the individual failure processes is affected by the loading condition and the material properties.

The influence of applied stress/strain on fatigue life for the different failure stages is shown in Figure 2-2. It can be seen that at lower stress/strain ranges, a large proportion of the fatigue life is occupied by strain localisation and the initiation of

fatigue cracks, while the effect of crack propagation is nearly negligible. In fact, experimental results had shown that for small laboratory specimens tested near the fatigue limit, up to 90% of the total life can be spent on crack initiation [36]. As stress/strain increases, crack initiates more readily and a greater proportion of a material's total life is spent on crack growth. Typically, fatigue analysis can be categorised into the high cycle and low cycle regions, where the line of demarcation between the regions is accepted to be on the order of $10^3 - 10^5$ cycles [37, 38]. The high cycle fatigue region is generally understood to be dominated by crack initiation, while the low cycle fatigue is dominated by crack propagation.

Figure 2-2 Stages of fatigue failure in ductile metal shown on stress-life/strain-life plots. Image taken from the work of Mughrabi [39].

Material-wise, a part can be considered to be with or without defects. Defects, such as inclusions and porosities, are local stress concentration areas which promote microcracking and reduce the crack initiation life. Moreover, when crack or crack-like flaws are present, the crack initiation phase can be bypassed, leading to significantly reduced fatigue life. In fact, defects play important roles in the fatigue properties of engineering materials because of their inevitable formation during

manufacture, e.g. during welding, casting and powder metallurgy. They can also be formed during service, e.g. scratches [40]. In general, the effect of defects on fatigue failure becomes more pronounced as stress reduces, i.e. in the high cycle fatigue and fatigue limit regions, because of their role in reducing the crack initiation life [36].

The log-log relationship between the stress range $\Delta\sigma$ and the fatigue life in terms of the cycles to failure N_f , as presented on the S-N curve in Figure 2-2, can be described by the well-known equation proposed by Basquin in 1910 [41], i.e.:

$$\frac{\Delta\sigma}{2} = \sigma'_f (2N_f)^b \quad (2.1)$$

where σ'_f and b are the fatigue strength coefficient and exponent respectively. Such relationship can also be expressed in terms of the plastic strain range $\Delta\epsilon_p$, where the equation, formulated independently by Coffin [42] and Manson [43] in 1954, is of the form:

$$\frac{\Delta\epsilon_p}{2} = \epsilon'_f (2N_f)^c \quad (2.2)$$

where ϵ'_f and c are the fatigue ductility coefficient and exponent respectively.

These two relationships serve as the basis for the stress-life approach and strain-life approach of fatigue design. The stress-life approach is often adopted for high cycle fatigue analysis because of the elastic-stress-dominated mode of fracture at lower stresses, while the strain-life approach is more appropriate for low cycle fatigue analysis because of the activation of cyclic plasticity at higher stresses. In fact, in view of the permanent deformation in the local region of the crack tip, plastic strain is often considered as the governing factor for fatigue damage. Various plastic-strain-based and energy-based damage parameters had been proposed for life prediction [44-46], though expressing fatigue life in terms of the applied stress is favoured from the engineering standpoint as it allows the nominal stress to be described explicitly.

2.2 Fatigue mechanisms

Microstructure and defects play very important roles in the fatigue properties of polycrystalline materials, especially via the crack initiation mechanism [47, 48]. In many cases, these two terms are used interchangeably, e.g. grain boundaries are referred to as planar defects and volumetric features such as precipitates, inclusions and porosities are referred to as bulk defects [49], while in other literatures, they are considered as part of the microstructural features [50]. In the present review, manufacturing flaws, such as inclusions, porosities and lack of fusion defects are collectively referred to as defects, while other microstructural features, such as grain and grain boundaries, are considered as part of the microstructure. The roles of microstructure and defects in fatigue fracture are reviewed by focussing on the early stage of fatigue failure, i.e. crack initiation and the growth of short fatigue cracks within the Stage I crack growth region.

2.2.1 Role of microstructure

The mechanism for the formation of persistent slip band, surface intrusions and extrusions and microcracks in ductile crystalline metals are well established, and had been discussed extensively in several reviews [39, 51, 52]. The process responsible for the generation of persistent slip bands is related to bulk irreversibility and occurs when thresholds of the plastic strain and the stress amplitude are exceeded. As shown in Figure 2-3a, the dislocation pattern in cyclically deformed material can be represented by pairs of dislocation layers between the persistent slip band (PSB) and material matrix (M) interface. Cyclic loads cause the glide-out motion of the dislocations at the interface, and slip steps are formed as the dislocations emerge at the matrix surface, leaving behind surface extrusion patterns, as illustrated in Figure 2-3b. Continued cyclic deformation leads to the generation of a roughened surface profile, as depicted in Figure 2-3c, and triggers the onset of fatigue damage by the nucleation of microcracks at the PBS-matrix interfaces (sites A and A'). Figure 2-3d shows the SEM image of extrusion features in a fatigued copper single crystal.

Figure 2-3 (a)-(c) Schematic illustration of the formation of extrusions; (d) surface extrusions formed in fatigued copper single crystal. Images taken from the works of Mughrabi [39, 53].

Pile-up of the interfacial dislocations occurs when they encounter obstacles such as grain boundaries, as shown in Figure 2-4a. The normal stresses exerted by the pile-up at the grain boundaries promote brittle debonding at the sites marked as A and A' [54]. Figure 2-4b shows the SEM image of such fracture mode in a copper polycrystal. A mixture of transgranular and intergranular features can be observed at the fracture surface.

Figure 2-4 (a) Dislocation model illustrating action of persistent slip band on the grain boundaries; (b) SEM image of intergranular and transgranular cracks in fatigued copper polycrystal. Images taken from the works of Mughrabi et al. [39, 54].

A number of factors affect the initiation of fatigue cracks at grain boundaries. It is generally well understood that fatigue crack initiation occurs more readily in coarse-grained materials, as they are more prone to slip band cracking [55]. The study by Szczepanski et al. showed that for a titanium-based alloy with a duplex microstructure, microcracking tend to take place in primary α grains with sizes on the higher end of the grain size distribution [56]. The reduction in fatigue strength with increase in grain size had been verified by means of S-N curves [57, 58]. Texture is another influencing factor on fatigue cracking as grains oriented for easy slip facilitates crack initiation. When grain colonies with similar orientations are present, continuous slip across several grains can take place such that the effective slip distance is larger than the size of individual grains [59, 60]. The longer slip distance associated with such grain clusters, known as supergrains, promote crack initiation and reduced fatigue resistance [61].

Analytical life prediction models are usually formulated based on the stress- or strain-based approach. For instance, Cheng and Laird [62] proposed the use of plastic shear strain range $\Delta\gamma_p$ as the damage parameter for predicting the initiation life N_i induced by cracking at persistent slip band:

$$\frac{\Delta\gamma_p}{2} N_i^{\alpha'} = C' \quad (2.3)$$

where α' and C' are empirical constants. Microstructure-based crack initiation models were developed to allow explicit representation of microstructural parameters. For example, Tanaka and Mura incorporated grain size d as one of the parameters for modelling the crack initiation life caused by cracking along slip band [63]:

$$(\Delta\tau - 2k)N_i^{0.5} = \left(\frac{8\mu W_S}{\pi d}\right)^{0.5} \quad (2.4)$$

where $\Delta\tau$ is the shear stress range, k is the friction stress of dislocation, μ is the shear modulus and W_S is the specific fracture energy per unit area along the slip band. For crack initiation at grain boundaries, W_S in Equation (2.4) is substituted by the specific fracture energy per unit area of the grain boundary W_G . These constitutive relations had been successfully adopted and extended for life prediction of various alloy systems [64-66].

2.2.2 Role of defects

While 90% of service failures for engineering structures can be attributed to fatigue, post-failure investigations indicate that nearly 100% of these accidents can be traced to areas of high stress concentrations at discontinuities such as defects and scratches. The coexistence of defects and microstructure-based features leads to their competing influence on the initiation of primary fatigue cracks. Size is often used as the parameter for quantifying the criticality of defects in such cases. For example, when defects are small in comparison to the local microstructure such as grain size, they may be perceived as benign. Non-metallic inclusions less than 5 μm in size are usually harmless for low purity parts under fatigue loading [67]. Precise determination of the critical defect size is difficult, however, because of the random arrangement of local microstructure. The non-propagating crack size of a 0.13% C steel had been found to be on the order of 100 μm , even though the average grain size is only 34 μm [31].

Empirical results had shown that defects such as pores and inclusions can be treated as cracks. Thus, the influence of defects on the fatigue strength of metals had been

extensively studied by employing linear elastic fracture mechanics (LEFM) theory [31, 68-70], where the stress intensity factor range ΔK is expressed as:

$$\Delta K = F\Delta\sigma\sqrt{\pi a} \quad (2.5)$$

where F is a geometric factor, $\Delta\sigma$ is the applied stress range and a is the crack size. As the fatigue limit is a result of non-propagating cracks [31], the threshold stress intensity factor range ΔK_{th} is used for predicting the fatigue limit σ_e for a given crack size:

$$\Delta\sigma_e = \frac{\Delta K_{th}}{F\sqrt{\pi a}} \quad (2.6)$$

It is worth noting that the mechanics relating to the growth of small/short and long fatigue cracks are different. Results from experimental studies indicated that small cracks generally grow faster and at lower threshold stress intensity than long cracks. The differentiation of short and long cracks can be considered from the mechanical (relative to plastic zone size), microstructural (relative to grain size), physical and chemical perspectives [71]. As linear elastic fracture mechanics theories pertain to the analysis of long cracks, modifications need to be made in applying these theories to small cracks.

The Kitagawa-Takahashi (K-T) concept is one of the fundamental theories for establishing the influence of defect size on fatigue strength across a range of crack size [48, 72]. As shown by the K-T diagram in Figure 2-5, fatigue strength is unaffected by crack size for short cracks, but it decreases with crack size as cracks transit to the long crack region. The El Haddad formulation attempts to simulate such a relationship through the insertion of the parameter a_0 to Equation (2.6), leading to the expression [73]:

$$\Delta\sigma_e = \frac{\Delta K_{th}}{F\sqrt{\pi(a + a_0)}} \quad (2.7)$$

For a defect-free part with fatigue limit σ_{e0} , a_0 is a material constant described by:

$$a_0 = \left(\frac{\Delta K_{th}}{F \Delta \sigma_{e0}} \right)^2 \frac{1}{\pi} \quad (2.8)$$

By applying Equations (2.7) and (2.8), the K-T diagram can be constructed for an estimation of the critical defect size behind the competing effects of microstructure and defects on crack initiation [74]. Besides LEFM concepts, probabilistic models considering the probabilities of failure for defect- and microstructure-driven modes of crack initiation had been developed for fatigue analysis when both failure mechanisms coexist in a material [48, 75].

Figure 2-5 Schematic representation of the Kitagawa-Takahashi diagram. Image taken from the work of Pessard et al. [74].

For materials where defects are the dominant cause of failure, deterministic fatigue predictions can be made. The semi-analytical and semi-empirical relationship developed by Murakami and Endo correlates the size of defect with the fatigue limit via the following expression [68]:

$$\sigma_e = \frac{c'(Hv + 120)}{(\sqrt{area})^{1/6}} \quad (2.9)$$

where $\sqrt{A_{\text{area}}}$ is the square root of the projected area of a defect; the influence of defect location is account for by the parameter c' , where $c' = 1.43, 1.41$ and 1.56 for surface inclusions, inclusion in contact with surface and interior inclusions respectively.

Stress- or strain-based models resembling the forms of the microstructure-driven crack initiation models had also been developed for simulating the effects of defects on life. For example, Equation (2.4) was extended for modelling inclusion-induced cracking via the formula [76]:

$$(\Delta\tau - 2k)N_i^{0.5} = \left(\frac{4\mu(\mu + \mu')W_l}{\pi\mu'\zeta}\right)^{0.5} \quad (2.10)$$

where μ' is the shear modulus of the inclusion, ζ is the inclusion size and W_l is the specific fracture energy per unit area of inclusion.

2.2.3 Fatigue scatter

Scatter in fatigue data is an essential aspect of fatigue analysis. Significant scatter, exceeding the magnitude of experimental error, had been observed on the stress- or strain-life curves of various metal materials. This can be attributed to the randomness associated with fatigue fracture, especially the crack initiation process [77].

Various sources contribute to this scatter. As described in the preceding sections, the competing influence of microstructure and defects on crack initiation can lead to variability in the fatigue life for identical specimens subjected to the same cyclic loads. For example, in cast aluminium alloy, crack initiation had been observed at both shrinkage pores and silicon-rich intermetallic particles [75]. The randomness in fatigue data is increased by microstructural and defect heterogeneities, where the size, location, shape and orientation of defects are factors that contribute to differences in defect characteristics and fatigue properties across specimens of the same material.

Extreme value analysis is a widely adopted method for estimating the size of the most critical defect in a part. Proposed by Murakami et al. [69, 78] for assessing the rating of inclusions in steels, the statistics of extreme had demonstrated strong correlations between defects and the fatigue strength of components. Various sampling strategies

had been developed for approximating the extreme defect size, e.g. by measuring defect sizes on polished metallurgical samples [79] or by collecting the sizes of crack-initiating defects at fracture origins [80]. Recommended procedures for performing extreme value analysis of nonmetallic inclusions in steel and other microstructural features are also specified in the ASTM E2283, where the process involves acquiring the largest defect size measurements in n specimens and use them as the input for fitting the extreme value distribution using the moments or maximum likelihood methods. The defect size distribution can then be used as the key variable for fatigue analysis and prediction using various analytical, statistical and computational tools. For instance, by obtaining the maximum defect size for a set of specimens and assuming it exists at below the specimen surface, Equation (2.8) can be used for predicting the lower bound fatigue strength [69].

2.2.4 Influence of loading

Mean stress

Asymmetrical cycling, i.e. non-zero mean stress, is a typical load case for many engineering components. The effect of superimposing a tensile stress on an alternating stress in stress-controlled fatigue tests can lead to the accumulation of damage, a phenomenon commonly referred to as cyclic creep or ratcheting. Various damage parameters, such as the stress-strain parameter $\sigma_{\max}\epsilon_{\text{at}}$ [81], where $\sigma_{\max}$ is the maximum stress and ϵ_{at} the total strain amplitude, and the alternative form $\sigma_{\max}\epsilon_{\text{ap}}$ [82], where ϵ_{ap} is the plastic strain amplitude, had been developed for life prediction under the influence of cyclic plastic deformation.

Stress-based methods, such as the Goodman, Gerber and Walker equations, are commonly adopted in engineering designs for assessing the relationship between the mean stress σ_m and the stress amplitude σ_a . Graphically, they are represented by constant life lines where an increase in the mean stress is compensated by a reduction in the stress amplitude for maintaining the same life. The equivalent stress amplitude σ_a^{eq} after adjusting for the effect of mean stress using the Goodman [83], Gerber [84] and Walker [85] equations are expressed respectively as:

$$\text{Goodman equation: } \sigma_a^{eq} = \sigma_a / (1 - \frac{\sigma_m}{\sigma_b}) \quad (2.11)$$

$$\text{Gerber equation: } \sigma_a^{eq} = \sigma_a / [1 - (\frac{\sigma_m}{\sigma_b})^2] \quad (2.12)$$

$$\text{Walker equation: } \sigma_a^{eq} = \sigma_a \left(\frac{2}{1-R} \right)^{1-\gamma} \quad (2.13)$$

where σ_b is the ultimate tensile strength and the Walker parameter γ is a material constant.

Load ratio

Besides mean stress, load ratio dependent cyclic properties had been studied in relation to the mechanism of crack closure. First observed by Elber in 1970 [86], his experimental investigations indicated that crack remained closed even at positive stress level during unloading. Upon subsequent loading, the crack grew only when the crack tip had opened. This implies that only part of the applied stress range contributed to crack growth, causing the maximum stress σ_{max} to be offset by the crack opening stress σ_{open} . The effective stress intensity factor range ΔK_{eff} is therefore lower than the applied value, i.e. [87]

$$\Delta K_{eff} = F(\sigma_{max} - \sigma_{open})\sqrt{\pi a} \quad (2.14)$$

Elber postulated that such closure event is caused by the development of a plastic zone envelope in the areas surrounding an advancing crack, as shown in Figure 2-6. Elastic materials in the crack wake exert compressive stresses, retarding the growth of the crack. Under positive load ratios, the required stress intensity factor range decreases with increasing load ratios as the plasticity-induced closure effect diminishes under large crack tip opening displacements [87-89].

While this concept is applicable to the propagation of long cracks, i.e. the plane stress condition, the growth of short cracks under plane strain is often explained by the mechanism of roughness-induced crack closure. As illustrated in Figure 2-6, the microstructurally-sensitive growth of small cracks generates a faceted fracture

morphology. Collision between the physically rough fracture surfaces interfere with the opening of the crack during subsequent loading, thereby reducing the effective stress [90]. The effect of closure on the near-threshold stress intensity factor range affect the propagation condition of cracks and thereby the fatigue endurance limit.

Figure 2-6 Schematic illustration of plasticity-induced and roughness-induced fatigue crack closure. Image taken from the work of Suresh and Ritchie [91].

2.3 High cycle fatigue and fracture of L-PBF stainless steel 316L

Processing of metal by the novel L-PBF technique introduces different microstructure and defect features, which affect the fatigue properties and fracture behaviour. This section starts with a comparison of the high cycle fatigue properties of L-PBF stainless steel 316L with the conventionally form, followed by a deep dive of the process-structure-property relationship in relation to the effect of L-PBF processing on high cycle fatigue properties and crack initiation behaviour.

2.3.1 Comparison of S-N data with conventional material

The S-N data of stainless steel 316L made by L-PBF, collected from open literature [92-94], are compared with the mean-data curve and the ASME Code fatigue design curve in Figure 2-7. Specifically,

- 1) The mean-data curve for conventional austenitic stainless steel is plotted based on the Argonne National Laboratory (ANL) fatigue life model [95], which was

derived from experimental strain-controlled fatigue tests performed on polished samples of wrought and cast austenitic stainless steel under fully reversed loading ($R = -1$) at ambient conditions. The fatigue strain-life relationship is expressed as:

$$\ln(N_f) = 6.891 - 1.92 \ln(\epsilon_{at} - 0.112) \quad (2.15)$$

The stress amplitudes used for plotting the S-N curve in Figure 2-7 were obtained by multiplying the strain amplitude with the Young's modulus E , which adopts a value of 179.264 GPa for austenitic stainless steel.

- 2) The ASME Code fatigue design curve [96], which defines the allowable number of cycles for structural materials as a function of the applied stress. It was obtained by applying reduction factors to the mean-data curve to account for factors such as data scatter, surface finish, material variability, etc. The fatigue design curve is expressed via the following equations [96]:

$$N_f = 10^x \quad (2.16)$$

where x is expressed as:

$$x = \frac{c_1 + c_3Y + c_5Y^2 + c_7Y^3 + c_9Y^4 + c_{11}Y^5}{1 + c_2Y + c_4Y^2 + c_6Y^3 + c_8Y^4 + c_{10}Y^5} \quad (2.17)$$

and Y is expressed as:

$$Y = \frac{\sigma_a}{c_{us}} \frac{E_{fc}}{E} \quad (2.18)$$

The values of the parameters c_i , c_{us} and E_f were taken from Division 2 of the ASME Boiler and Pressure Vessel Code VIII [96].

The Walker equation was applied to the test data, where appropriate, for plotting the results on the same graph (see Chapter 5 for the rationale for using the Walker equation). A reduction factor of 0.85 was applied to the data of Mower and Long [94] to adjust bending to axial fatigue results [97]. By comparing the S-N data points with the mean-data curve, it can be seen that L-PBF stainless steel 316L in general shows

inferior fatigue resistance to the conventional counterpart, though it is to be noted that some studies were able to achieve fatigue strength higher than the conventional materials. The ASME fatigue design curve does not provide a conservative estimate for some of the data points, indicating that it needs to be modified in order to provide safe design guidelines for the L-PBF form.

However, no conclusions can be drawn with regards to the cause for the fatigue properties of L-PBF stainless steel 316L shown in Figure 2-7 as comprehensive information of the processing condition, defects, microstructure and mechanical properties of the samples used was not provided to allow comparisons to be made across these studies.

Figure 2-7 Comparison of S-N data for laser powder bed fusion stainless steel 316L with conventional austenitic stainless steel at $R = -1$. Results from Spierings et al. [92] were conducted at $R = 0.1$ and had been converted to $R = -1$ using the Walker equation; results from Mower and Long were conducted in bending and were converted to axial using a reduction factor of 0.85 on strength [94].

2.3.2 Process-structure-property relationship

Printability of stainless steel 316L

There are printability issues specific to stainless steel 316L that could affect its fatigue performance. Mukherjee et al. [98] compared the printability of various L-PBF alloys

in terms of the dimensional accuracy, formation of lack of fusion defects and compositional changes. As poor dimensional accuracy stems from the generation of significant thermal strains and material shrinkage, it is indicative of a material's tendency for thermal cracking [99]. Both micro-cracks and lack of fusion defects are potential crack initiation sites and therefore impinge directly on the fatigue properties. In terms of the compositional change, the evaporation of alloying elements, e.g. during welding, had been reported to be associated with the degradation of mechanical properties [100]. The use of high-power laser for L-PBF could promote such compositional change and result in reduced fatigue resistance.

The maximum thermal strain ϵ_T plot in Figure 2-8a indicates that stainless steel 316L has comparable thermal distortion propensity as Ti6Al4V but is more prone to thermal distortion than Inconel 625. The maximum thermal strain is calculated using the equation:

$$\epsilon_T = \frac{0.9081\theta\Delta T}{EI} \frac{t}{F\sqrt{\rho}} H^{3/2} + 0.0009 \quad (2.19)$$

where θ is the volumetric thermal expansion coefficient, ΔT is the temperature difference between the peak and surrounding temperature, $H (= \phi P/v$, where ϕ , P and v are the laser absorption coefficient, laser power and scan speed respectively) is the heat input per unit length, EI is the flexural rigidity of the baseplate, $F (= \chi/vw$, where χ and w are the thermal diffusivity and melt pool length respectively) is the Fourier number for the transient heat transfer process.

Equation (2.19) indicates that for the same processing condition, a larger thermal expansion and laser absorptivity, as well as a smaller thermal diffusivity can give rise to greater thermal strain. This explains the results in Figure 2-8a, where the thermal properties of stainless steel 316L are associated with higher propensity for thermal distortion. Nonetheless, Equation (2.19) suggests that L-PBF process parameters can be adjusted to maintain the overall thermal strain at a low level. Specifically, the settings for process parameters such as laser power and scan speed can be selected to produce smaller energy input and reduce the likelihood of distortion.

Figure 2-8 Comparison of a) dimensional accuracy in terms of the thermal strain (1st, 2nd and 3rd refer to three different processing conditions), b) tendency for formation of lack of fusion defects, and c) percentage evaporation of the heaviest element for laser powder bed fusion alloys. Images taken from the work of Mukherjee et al. [98].

The susceptibility to lack of fusion defect formation is characterised by the lack of fusion index LF, which is the ratio between laser penetration depth and layer thickness. Figure 2-8b shows that amongst the materials, stainless steel 316L is associated with the lowest LF. This suggests that for the same processing condition, its thermal properties, i.e. thermal conductivity and laser absorptivity, produce the shallowest melt pool depth. Poor laser absorptivity, as in the case of copper, could be a contributing factor to low LF as it determines the amount of laser energy available for melting the powder bed. However, this might not be applicable for stainless steel 316L as its laser absorption coefficient is considerably higher than that of copper [101] and is comparable with that of Ti6Al4V and pure aluminium [102]. Instead, the high thermal conductivity (of 17 W/mK, as compared to 7.3 W/mK for Ti6Al4V), could have led to faster cooling resulted in insufficient melting of the parts [18]. Despite

the unfavourable thermal properties, the LF index can be improved by adjusting the L-PBF process parameters, e.g. by increasing the input energy and reducing the layer thickness.

For an alloying element i , the amount of material vaporised Δm_i during L-PBF is calculated by the equation:

$$\Delta m_i = \frac{LA_s J_i}{v} \quad (2.20)$$

where L is the track length, A_s is the surface area of the melt pool and J_i is the vaporisation flux. Figure 2-8c indicates that stainless steel 316L is most susceptible to the loss of the volatile element manganese. A lower percentage composition of manganese had been reported to cause worsened mechanical strength for this alloy [98, 103, 104], and could be indicative of reduced resistance of the material to fatigue cracking. Equation (2.20) suggests that this problem can be ameliorated by employing lower energy inputs, e.g. higher scan speed, which correspond to smaller melt pools.

L-PBF processing

Discussions in the previous section highlight the importance of appropriate process parameter selection for improving the printability of L-PBF stainless steel 316L. The L-PBF process is known to be associated with over a hundred process parameters [105]. According to the Pareto principle, for most events, about 20% of the inputs are responsible for 80% of the outcomes. For L-PBF, Elsen shortlisted 12 significant and independent factors, i.e. oxygen level, beam quality, dynamic viscosity, surface tension of the melt pool, melt enthalpy, laser power and spot size, hatch spacing, scan speed, layer thickness, material diffusivity and bulk density [106]. Existing studies on L-PBF tend to focus on the adjustable parameters, including laser power P [13, 107-109], scan speed v [13, 28, 108, 109], hatch spacing h [94, 110, 111] and layer thickness T [13], as illustrated in Figure 2-9. Design of experiment analysis results indicated that they are the statistically significant process factors on key response indicators such as track characteristics, part density and mechanical properties [112-116].

Figure 2-9 Schematic illustration of laser powder bed fusion process parameters. Image taken from the work of Yap et al. [117].

Several unifying parameters have been used for categorising the L-PBF process window. The P - v relation is applicable for single track synthesis [118, 119], since the P/v ratio is representative of the heat energy from the laser source, as in the conventional welding process. Figure 2-10 shows the P - v diagram being classified into regions, including smooth track, balling/irregular track, over-melted track and un-melted track [14, 120], where the generation of smooth tracks is associated with the best print quality. However, depending on the settings of process parameters other than P and v , this approach can lead to the formation of many local process zones. For example, as can be seen from Figure 2-10, the use of different layer thicknesses (250 μm for the upper block and 100 μm for the lower block) caused two distinct regions to be located on the P - v diagram. This parameter is therefore only suitable for preliminary assessment of the track quality and is not a good indicator of the quality of three-dimensional parts.

Figure 2-10 *P-v* diagram reconstructed based on compiled literature results [14, 120]. The upper block is for processing at a layer thickness of 250 μm and the lower block 100 μm .

By taking into account factors including h and T , the volumetric energy density $E_d = P/vhT$ is often used for quantifying the required energy input for consolidating three-dimensional parts. Figure 2-11 shows the relative density against E_d plot, constructed based on a total of 134 samples manufactured by five different commercial L-PBF machines [121-128]. Despite the wide choice of systems and process parameters, the data show good convergence. By considering $E_d = 100 \text{ J/mm}^3$ as the optimum build setting, where parts with least porosity, good surface finish and hardness had been produced [124], the process window can be divided into regions including:

- 1) $0.7E_{d0} < E_d < 1.3E_{d0}$: Optimum processing region with high relative density close to 99%.
- 2) $0.5E_{d0} < E_d < 0.7E_{d0}$: Region of inconsistent density due to under-melting.
- 3) $E_d < 0.5E_{d0}$: Region of drastic reduction in density due to under-melting.
- 4) $E_d > \sim 1.3E_{d0}$: Region of reduction in density due to over-melting.

Figure 2-11 Relative density against input volumetric energy density E_d data collected from the literature [121-128] for the processing of stainless steel 316L by laser powder bed fusion.

It can be seen that stainless steel 316L is associated with a reasonably wide process window, i.e. $\pm 30\%$ of the optimum processing condition. This provides flexibility for the fabrication of high-quality parts despite the material's inherent propensity for thermal distortion and the formation of lack of fusion defects, as noted in the previous section.

Nonetheless, careful examination of the influencing fatigue factors at the microscopic level is required for characterising the effect of processing on the fatigue properties. For instance, the considerable fluctuation in density from -50% to -30% of the optimum process condition can be associated with random defect distributions, which can lead to poor predictability of the fatigue properties. The nature of the defects due to over-melting and under-melting also needs to be characterised for evaluating their influence on the fracture behaviours. Literature results on the effect of processing on the formation of microstructure and defects, as well as the implication of these features on the fatigue properties are presented in the following sections.

Microstructure and defects

For L-PBF stainless steel 316L, unique hierarchical structures exist at the macro, micro and nano scales [129]. At the macro-level, melt pools on the order of 100 μm form in relation to the laser track, as shown in Figure 2-12a. The exact dimension of the melt pool is dependent on the process conditions, e.g. laser spot size, laser power, etc. Layer-by-layer scanning of the part leads to the formation of periodic melt pool structures. In addition, columnar grains are visible in Figure 2-12a, as shown by the clear contrast upon etching. They are generated as a result of directional solidification and could extend several layers of melt pools under strong re-melting. The anisotropic microstructure is associated with orientation dependent tensile properties, where vertically-build stainless steel 316L had been reported to possess lower strength and higher elongation to failure relative to the horizontal direction because of favourable grain orientations for twinning [130, 131]. Correlations between the melt pool size and grain texture had also been reported, where a long and shallow melt pool was found to promote a strong $\langle 001 \rangle$ crystallographic texture along the build direction, whilst a short and deep melt pool promotes the $\langle 011 \rangle$ texture [132]. The $\langle 011 \rangle$ texture had been demonstrated to produce significantly enhanced ductility by favouring the mechanism of nano-twinning [132].

At the micro-level, intragranular cellular sub-grains on the order of 1-2 μm are present, as shown in Figure 2-12b. In comparison with cast parts, where the dendritic microstructure and second phase particles are on the order of tens of microns [133, 134], the length scale of the cellular grains are much smaller. Significant undercooling on the order of 10^5 - 10^7 K/s gives rise to the high temperature gradient and cooling rate, and subsequently the formation of the ultrafine-grained structure. The cellular grains can adopt equiaxed or bar-like shapes, depending on the growth direction of the macro-scale grains. The ultrafine-grained microstructure had been shown by various studies to be critical for the superior tensile strength of L-PBF materials in comparison to the conventional form [129, 131, 135].

Figure 2-12 (a) Etched vertical plane of laser powder bed fusion stainless steel 316L showing melt pool and columnar grains and (b) intragranular cellular sub-grains; (c) elemental composition analysis of nano-inclusion, where Spectrum 1 covers area with inclusion and material matrix and Spectrum 2 covers only the material matrix. Image taken from the work of Zhong et al. [129].

Second phase particles in the form of oxide nano-inclusions, as shown in Figure 2-12c, exemplify the nano-scale structures [129]. The identity of the nano-inclusions, as indicated by the elemental composition analysis result, suggests silicon-rich oxides. The formation of such oxide inclusions is promoted by the reaction between the silicon in stainless steel 316L and the residual oxygen in the build chamber, where the high process temperature provides the activation energy required for the reaction to take place.

Recent studies reported the presence of a pre-existing dislocation network at the cellular grain boundaries [136], as shown in Figure 2-13a. The elemental distribution maps (Figure 2-13b-f) suggests that the dislocation network is characterised by a mild segregation of chromium, molybdenum and manganese. The formation of such

dislocation and segregation structures is promoted by the rapid-cooling nature of L-PBF [137], where the slightly misoriented cells give rise to the dislocation walls, while the rejection of alloying elements at the solidification front is responsible for their observation at the cell boundaries [138].

Figure 2-13 (a) Bright field STEM image of dislocation network in laser powder bed fusion stainless steel 316L; (b) – (f) elemental distribution maps of the selected region in (a). Image taken from the work of Liu et al. [136].

Defects form a major part of the L-PBF structure. Small spherical pores on the order of several microns, as shown in Figure 2-15b, are nearly unavoidable even under optimum processing. This is because of the entrapment of gas bubbles originated from the evaporation of low melting point elements or gases, such as nitrogen or argon, that are pre-existing in the powder feedstock due to powder fabrication by gas atomisation [139] (Figure 2-15a). Also, depending on the packing density of the powder, spaces between powder particles can dissolve and be retained in the melt pool.

Figure 2-14 (a) Gas pores in gas atomised powder particles and (b) small spherical pores in a laser powder bed fusion part. Images taken from the works of Choo et al. [140] and Thijs et al. [141].

During processing, under-melting occurs because of many reasons, e.g. bad choice of build settings, dirty laser window, poor recoating, etc. The lower-than-optimum energy input gives rise to insufficient penetration of laser energy into the substrate and prevents the formation of contact zones that stabilise the melt pool [142]. Besides, the high liquid viscosity at low temperature is associated with poorer wetting capability, which can lead to the generation of coarsened balls [14]. Large lack of fusion defects on the order of hundreds of microns can be formed under these conditions, as shown in Figure 2-15. Further increase in defect size can cause the joining of neighbouring defects, leading to the formation of a network of interconnected defects, such as that shown in Figure 2-15b.

Figure 2-15 Large irregular lack of fusion defects. Larger defects are formed at higher layer thickness setting (b). Image taken from the work of Qiu et al. [143].

Defects can also be generated under excessive energy input, as shown by the reduction in density at higher input energy levels in Figure 2-11. With an increase in the laser energy and size of the melt pool, larger volume of powder participates in track synthesis. The complex flow dynamics are associated with the generation of vortices, denudation zones [144], keyholes [15] and spatter particles [16, 145], triggering the formation of defects.

Fatigue crack initiation and fracture behaviour

The microstructural features and defects highlighted in the previous section are potential weaknesses in the material matrix and can contribute to premature fatigue crack initiation. In particular, the process-induced defects had been reported to be responsible for the poor high cycle fatigue resistance of L-PBF materials [24, 94]. As shown by the fractograph in Figure 2-16, crack initiation occurred at a near-surface lack of fusion defect on the order of hundreds of microns. Under cyclic loading, such pre-existing defects generate strong notch effect, where the high local stress concentration promotes the nucleation of cracks. Moreover, as the irregularly-shaped lack of fusion defects are characterised by sharp edges, they can be perceived as cracks. This greatly shortens the crack incubation period, leading to reduced fatigue resistance.

Figure 2-16 SEM image showing the fatigue fracture surface of a horizontally-build stainless steel 316L sample produced by laser powder bed fusion; the enlarged view indicates crack initiation from a near-surface lack of fusion defect. Image taken from the work of Shrestha et al. [24].

Unlike conventional materials where slip band and microstructural weakness compete with defects in initiating cracks, the prevalence of critical lack of fusion defects makes defect-induced crack initiation the dominant mode of failure for L-PBF materials [22]. Factors such as location, size and shape of the defects had been shown to be responsible for the large scatter in the high cycle fatigue data of L-PBF materials [146-148]. Recall the compilation of S-N data for L-PBF stainless steel 316L in Figure 2-7, variability in defect characteristics could be a key reason behind the wide distribution of the S-N results. Systematic evaluation of the defects and fracture behaviour is necessary to verify this claim as such information is not provided in the studies.

As cyclic stress reduces and approaches the fatigue limit, small defects and microstructural features may participate in crack initiation and short crack growth because of the reduced plastic zone sizes. Small defects on the order of short range microstructure can be activated [149] and microstructure-induced crack initiation may occur. Based on understanding of the fracture behaviour of parts produced by equal-channel angular pressing, crack initiation tend to be intergranular in nature for ultrafine-grained materials, whereas for the coarse-grained form, fracture takes place via the transgranular path because of the high strain concentration inside slip bands [150]. Thus, the intergranular form of crack initiation may be favoured for L-PBF materials because of the cellular sub-grain microstructure. In addition, the rapid-cooling-induced ultrafine cellular grains and particles could potentially delay fatigue crack initiation by retarding dislocation movements [151, 152]. For L-PBF AlSi10Mg, silicon-based particles at the melt pool boundaries had been shown to improve fatigue strength by acting as obstacle to crack propagation in very high cycle fatigue regime [153].

For additive manufactured stainless steel 316L, the generation of strain-induced martensite at the crack tip had been observed for samples made by a direct metal deposition system [154]. This serves as a strengthening mechanism as the martensitic phase could promote the reduction of fatigue crack growth rate. Besides, the effect of build orientation on crack growth had been studied, as illustrated in Figure 2-17. The anisotropic grain arrangement, where the epitaxial grains are aligned along the build

direction, could lead to better crack growth resistance for vertically-built samples as the smaller intergranular spacing can contribute to a more tortuous crack path and slower crack growth rate [155]. However, it is to be noted that influencing factors on crack growth may be less relevant to high cycle fatigue properties because of the dominating effects of crack initiation.

Figure 2-17 Effect of grain orientation on crack growth for (a) vertical and (b) horizontal laser powder bed fusion samples. Image taken from the work of Leuders et al. [155].

In addition, as the technology matures and users' familiarisation with the process increases, better defect control can be achieved such that fatigue assessments should consider the competing influence of defects and microstructure on crack initiation. The sensitivity of L-PBF materials towards defects had been studied by comparing the fatigue properties of as-built parts and parts subjected to hot isostatic pressing (HIP). The results showed that both Ti6Al4V [94] and CoCr [156] alloys possessed strong defect-oriented fatigue behaviours, where the fatigue strengths were markedly improved after HIP, while stainless steel 316L is highly defect-tolerant as HIP was unable to improve its fatigue strength [93, 155]. Being a single phase austenitic structure that is devoid of the brittle martensitic phase, the defect-tolerant behaviour of stainless steel 316L could be attributed to its high ductility, which serve to reduce

the notch sensitivity [93, 155]. However, fracture analysis was not performed in these studies for examining the crack initiation mechanisms of the samples.

2.3.3 Influence of loading

As mentioned, the influence of defects and microstructure on the high cycle fatigue properties diminishes at higher stresses because of the shortened crack initiation phase. In addition, cyclic plasticity could be incurred, resulting in elastic-plastic deformation behaviour. In particular, the lower strength of vertically-build L-PBF stainless steel 316L relative to the horizontal direction could subject vertically-build parts to cyclic plastic deformation. Ratcheting, associated with the activation and build-up of plastic strain under stress-controlled cyclic loading and superimposed tensile mean stress, could take place and exhaust the ductility of the material. The high ductility stainless steel 316L had been reported to survive high cycle fatigue tests in spite of the plastic damage [157]. This is implicative of the relevance of ratcheting activities on the high cycle fatigue properties of the material. As the stress level decreases and failure becomes increasingly dominated by the initiation and propagation of fatigue cracks [82, 158], the combined effects of fatigue and ratcheting could impact the overall life.

2.3.4 Post-processing treatment

As a common industrial practice, heat treatment is almost always performed for standardising material properties and preventing issues related to corrosion. For AM-processed parts, various post-processing operations, including those for annealing and hot isostatic pressing, have been specified in standards such as the ASTM F3184. This makes understanding the effects of these treatments an important aspect for assessing the properties of L-PBF materials.

Conventional stainless steels, including the austenitic type, are not hardenable by heat treatment, which instead is performed to remove phases that are detrimental to corrosion resistance or fracture toughness. High temperature annealing followed by quenching is recommended by international standards such as the AMS 2759/4 for

recrystallization and the elimination of chromium carbides, i.e. $M_{23}C_6$, to prevent sensitisation and intergranular corrosion. The extra-low carbon content in stainless steel 316L serves to reduce the formation of carbides. According to the carbide precipitation kinetics curve [159], carbide precipitation for stainless steel 316L occurs most rapidly at 800 – 900 °C; at temperatures above 950 °C, they dissolve in the austenite matrix, whereas at temperatures below 500 °C, the thermal energy is insufficient for their formation. Besides, stress relieving is applied to weld structures, and annealing is performed on cold worked parts for restoring ductility [160].

Various post-processing treatments, including stress relieving, solution annealing and HIP, had been studied for L-PBF stainless steel 316L, as summarised in Table 2.1. Coarsening of the cellular sub-grains occurs at 800 °C, with their complete dissolution happening at temperatures above 900 °C. Such microstructural changes are associated with lower hardness and static strength, but higher ductility and impact energy, consistent with understanding of the effect of annealing on the strength-ductility trade-off of metal alloys [161].

In relation to the fatigue properties, stress relieving had been demonstrated to be an ineffective strategy for improving the fatigue resistance of L-PBF stainless steel 316L [93, 155]. The high ductility of the material could have obscured the influence of residual stress, probably via stress relaxation by plastic deformation. Besides, a large part of the residual stress could have been released due to cutting and machining test samples to the final size as fatigue specimens are usually small. The effects of HIP could vary depending on the defect condition of the sample as well as the loading condition. For instance, the fatigue resistance of parts containing critical crack-initiating defects should improve after HIP because of the closure of the defects [162]. However, results by Leuders et al. [93] showed that at higher cyclic stresses, the fatigue strength of the HIPed parts was significantly lowered, despite the removal of processing defects, because of the reduced strength and lower sensitivity to defects at this loading condition. Studies on the influence of high temperature annealing on the fatigue properties are not available at the time of writing this thesis. The lower tensile strength after high temperature heat treatment is expected to affect the fatigue

properties via cyclic plastic deformation, as has been explained for the effects of build orientation in Chapter 2.3.3.

Table 2.1 Relevant post-processing treatment conditions for laser powder bed fusion stainless steel 316L.

Treatment	Condition	Effects	Ref.
Stress relieving	450 °C, 2hrs	• No change in hardness, not an effective stress relief condition.	[163]
	600 °C, 2 hrs	• Melt pool and cellular grain structures were retained.	[164]
	650 °C, 2 hrs	• Slightly reduced yield and fatigue strengths.	[93]
	650 °C, 2 hrs	• Small increase in fatigue limit.	[155]
Annealing	800°C, 6 mins	• Coarsened sub-grains (3–5 µm). • Fewer dislocations observed at cellular grain boundaries.	[165]
	900°C, 6 mins	• Cellular sub-grain structure disappeared.	[165]
	950 °C, 2hrs	• Reduced hardness and stress corrosion cracking.	[163]
	950 °C, 2 hrs	• Dissolved melt pool boundaries and sub-grains. • Small pits observed. • Lower strength and higher impact energy.	[164]
	1100°C or 1400°C, 6 mins	• Duplex austenite-ferrite phase observed. • Grains grew proportionally with annealing time (200 µm when treated for 1 hr). • Significant grain growth and twinning at 1400 °C.	[165]
Hot isostatic pressing	1150 °C, 100 MPa, 4 hrs	• Grain growth and loss of grain orientation. • Lower fatigue strength at high stress.	[93]
	1150 °C, 100 MPa, 4 hrs	• Increased fatigue stress intensity threshold.	[155]
	1190 °C, 145 MPa, 4 hrs	• Improved high cycle fatigue properties.	[94]

2.3.5 Predictive models

As highlighted by Nicoletto [166], the complex processing environment make fatigue failure analysis and property assessment of L-PBF parts inherently challenging. This is worsened by the part-to-part and machine-to-machine variability, where the inconsistency in part quality compromises the predictability of fatigue models. This section provides a brief review of the approaches for fatigue modelling of L-PBF materials.

The use of unifying parameters such as E_d is a simple and straightforward means for process-property mapping. Data convergence had been demonstrated for the density of parts made across different studies, as shown by Figure 2-11. However, noting the large variance in the data, especially in the under-melting region, using such parameter for fatigue prediction could cause the S-N scatter band to be amplified because of the strong sensitivity of fatigue failure towards defects and microstructure variations. Being thermodynamic quantities, energy density parameters such as E_d are unable to capture hydrodynamic phenomena and recoil pressure [167]. The inability to represent the melt pool geometry makes accurate defect prediction impossible, thereby restricting the use such parameters for high-accuracy fatigue predictions.

Traditional phenomenological models, such as the $\sqrt{\text{area}}$ model by Murakami and the Kitagawa-Takahashi diagram with El Haddad formulation, as discussed in Chapter 2.2, had been adapted for correlating L-PBF defects with fatigue strength [22, 168]. However, they demonstrated limited applicability that is contingent on the processing and post-processing strategies or defect conditions. For instance, it was found to be too conservative for predicting the fatigue strength of L-PBF TiAl6V4 in the long crack region [22]. Also, it is unable to predict the fatigue strength of the material after HIP treatment [168]. In addition, as these models do not contain L-PBF-related factors, they cannot be directly applied for mapping the process-property relationships. The use of separate models for treating the process-structure and structure-property relationships [169] is associated with increased cost and various sources of model uncertainties [170].

Finite element models had been constructed for studying the fatigue damage process [144, 171]. However, the interaction between defects and microstructure on the fatigue fracture behaviour make the implementation of mechanistic fatigue model for L-PBF materials challenging. Specifically, defect-based constitutive models often assume negligible effects of microstructure or homogenous microstructure arrangement [172]. But such assumptions are not valid because of the highly anisotropic and heterogeneous microstructures [173]. Microstructure-based models [63, 66] can be employed, but the characterisation of microstructural features, e.g. grain and inclusion sizes, is not easy because of significant part-to-part and built-to-built variabilities [174] and the influence of processing on microstructure and defect formation.

Statistical methods may be used for mapping the process-property relationship. However, they are subjected to the same limitations of the other traditional models because of the complexity the L-PBF processing environment. For example, for a system with two input factors, nine design points are necessary for constructing a response surface. This amounts to a large number of fatigue tests, especially for high-dimensional systems, and is therefore impractical considering the costs associated with fatigue testing. Advanced data science methods may be leveraged because of the powerful pattern recognition capability. Such methods had been used for parameter selection and the development of new processing domains using existing processing and material knowledge [175-177]. For instance, a Bayesian-based model coupled with machine learning techniques were used for identifying the relevant micromechanical and microstructural variables on fatigue crack propagation [178]. However, while the model enhances qualitative understanding of the fatigue fracture mechanism, the complex procedures, involving characterising the crack geometry and the surrounding microstructure combined with simulation for obtaining the associated micromechanical field, for obtaining the input data (650 data points) make the model unsuitable for fatigue assessment from the engineering standpoint.

2.4 Summary and research gap

Literature results show that the high cycle fatigue properties of L-PBF parts could be inferior to those of conventional materials because of premature fatigue crack initiation at process-induced defects. For L-PBF stainless steel 316L, the high thermal conductivity could subject to the formation of lack of fusion defects and thermal distortion, but this can be remediated with process optimisation. The single-phase austenitic microstructure is believed to contribute to defect-tolerant behaviour, as evidenced in the ineffectiveness of HIP as the means for improving its fatigue strength.

While knowledge and method for traditional fatigue analysis can be applied to materials made by the L-PBF route, there are challenges that remain to be addressed before this technology can be adopted for the fabrication of engineering parts. This section lists the research gaps identified based on the literature review in this chapter.

2.4.1 Fracture behaviour

While the detrimental influence of process-induced defects on the fatigue properties of L-PBF materials is widely accepted and supported by experimental results, the roles of microstructure and defects, and their interactions on the high cycle fatigue properties have not been widely explored. This is because failure-critical defects are nearly always present in the samples reported in literature studies. Considering the wide process region and associated material properties, systematic examination of the fracture behaviour in relation to the different processing regions could provide a more holistic understanding of the failure mechanisms. Such information will allow fatigue results to be compared in the context of processing, such that fair judgement can be made of the processing capability.

2.4.2 Baseline fatigue curve

Fatigue design requires reference to the load versus life data. Fatigue test database and guidelines are well established for traditionally-manufactured steels, but they cannot be applied directly for L-PBF materials because of the different material

properties. Baseline fatigue properties, in the form of stress-life or strain-life plots, and the extent of fatigue scatter should be properly defined for L-PBF materials. This will ensure the implementation of safety factor and increase the confidence of employing L-PBF for fatigue-related applications.

2.4.3 Complexity of L-PBF processing

The major challenge for fatigue analysis of L-PBF materials lies in addressing the complex processing environment. The large number of process variables and process-induced structures are associated with a high-dimensional nonlinear input-output space. This is further complicated by the nonnegligible machine-machine and build-build variability, which makes establishing the fracture behaviour and fatigue properties difficult. The transferability of traditional models for fatigue analysis of L-PBF materials is limited as they do not contain L-PBF-related factors. In fact, application of these models to L-PBF materials has not achieve much success because of the unique L-PBF microstructure and defect properties. Furthermore, existing experimental studies lack consistent reporting of the processing conditions, microstructure characteristics and failure behaviours, such that results from the published domain cannot be easily adapted for analysing the process-structure-property relationship.

CHAPTER 3 MATERIALS AND EXPERIMENTAL METHOD

3.1 Sample preparation

3.1.1 Stainless steel 316L powder

Stainless steel 316L powder with a predominantly spherical shape (Figure 3-3a) was purchased from Höganäs AB and used as the feedstock for fabricating the samples. The powder was produced by gas atomisation with typical particle size of 20-53 μm and an inner pore level of less than 0.6%. Chemical compositions of the powder, as shown in Table 3.1, comply with the typical alloy composition of type 316L stainless steel [179].

Figure 3-1 SEM image of stainless steel 316L powder used for fabricating the samples in this research.

Table 3.1 Elemental compositions in weight percentage of stainless steel 316L powder feedstock.

C	Si	Cr	Mn	Ni	Mo	Fe
0.02	0.8	17	1.1	12	2.5	Balance

3.1.2 L-PBF process parameter

All parts were prepared on an EOS M290 L-PBF system installed with an Yb-fibre laser. The standard build parameter settings, as recommended by the machine manufacturer for part fabrication on a preheated platform, were adopted for producing near fully dense parts. Samples made using this processing condition are denoted as S_0 , which corresponds to a laser power P_0 of 195 W, scan speed v_0 of 1083 mm/s, layer thickness T_0 of 20 μm and hatch spacing h_0 of 90 μm . Other process parameters such as laser spot size, hatch pattern and preheat temperature, etc. are not provided in this thesis as full information of the standard parameter setting of EOS is not to be disclosed.

In Chapter 4, the effects of process parameters are examined by varying the laser power P and layer thickness T from the standard condition one factor at a time, as shown in Table 3.2, while keeping the other process parameters at their standard conditions.

Table 3.2 Process parameters for the one-factor-at-a-time analysis samples in Chapter 4.

Samples	P (W)	T (μm)
S_0	195	20
$1.5P_0$	293	20
$1.3P_0$	254	20
$0.7P_0$	137	20
$0.5P_0$	98	20
$2T_0$	195	40
$3T_0$	195	60
$4T_0$	195	80

In Chapter 6, laser power and scan speed were varied from the standard condition according to the central composite design (CCD) shown in Figure 3-2, while keeping the other process parameters at their standard conditions. Some of the results from

Chapter 4 were reused and denoted accordingly. The parameter settings are listed in Table 3.3. Scan speed was used as one of the process variables rather than layer thickness, as varying the layer thickness would require a large number of build jobs for producing the CCD samples.

Figure 3-2 Sample design points $S_0 - S_8$ for the central composite design. Unfilled dots indicate the validation runs.

Table 3.3 Process parameters for the central composite design samples in Chapter 6.

Samples	P (W)	v (mm/s)
S_0	195	1083
S_1	126	1313
S_2	264	1313
S_3	264	853
S_4	126	853
$S_5/0.5P_0$	98	1083
S_6	195	1408
$S_7/1.5P_0$	293	1083
S_8	195	758

3.1.3 Test specimens

Samples were first built as rectangular blocks and subsequently machined by wire cut electric discharge machining (EDM) into standard fatigue test sample geometries. Orientations and dimensions of the rectangular blocks are as shown in Figure 3-3a. The blocks were positioned at a small angle from the recoating direction to reduce uneven powder deposition due to interaction between the build parts and the recoater [180]. Samples were manually grinded before mechanical testing, leading to a minimum material removal of 20 μm on each surface. This served to remove any near-surface microstructure and residual stress generated by the wire cutting process, which could be detrimental to the fatigue resistance [181].

Note that rectangular blocks and test samples with two different dimensions were prepared for each orientation, i.e. standard ASTM E466 flat fatigue test specimens with tangentially blended fillets between the uniform gauge sections and the ends, and sub-sized specimens with the same geometry. The respective dimensions are as indicated in Figure 3-3, where the standard specimens were made with thickness 3 mm, gauge length 14 mm, width 7 mm, and overall length 119 mm, and the sub-sized samples with thickness 3 mm, gauge length 12 mm, width 5 mm, and overall length 101 mm. All mechanical tests were conducted using the standard specimens, except for the specimens subjected to HIP, for which sub-sized samples were used because of the availability of test parts. Such difference in sample size had been reported to produce insignificant effects on the high cycle fatigue properties [182], though the use of sub-sized samples could lead to underestimated ductility values [183].

For the horizontal samples, a maximum of eight test samples were cut from each rectangular block, resulting in the samples being positioned at different heights from the baseplate. To examine the effects of build height on the quality of the parts, two-dimensional defect area fraction as well as hardness measurement were done at six different heights (of approximately 5 mm intervals) on the vertical planes of the samples and evaluated by Analysis of Variance (ANOVA). The results indicated no significant change of defect and hardness properties with build height at 95%

confidence level. Thus, fatigue samples sliced from the same blocks were assumed to be identical despite being fabricated at different heights from the build plate.

For the horizontal S_0 samples, test samples were obtained from four different rectangular blocks fabricated in a single build job but positioned at different locations of the build platform. To ensure the reproducibility of defects across the parts, dimensional defect area fractions were obtained for each of the four test blocks, and the results were analysed by ANOVA. The results suggested insignificant change in the defect densities across the build parts, such that the test blocks were assumed to be identical.

Figure 3-3 (a) Orientation of sample blocks on the build platform with dimensions (in mm) of the blocks for both standard and sub-sized specimens indicated in table and (b) geometry and dimensions (in mm) for both standard and sub-sized (indicated in brackets) fatigue samples. The xy plane is referred to as the horizontal plane and plane parallel to the build direction is referred to as the vertical plane in the main text.

3.2 Post-processing treatment

The effects of post-processing on high cycle fatigue are examined in Chapter 5. Some of the S_0 samples were subjected to annealing or hot isostatic pressing treatments, collectively referred to as heat treatments in this thesis. Solution annealing were carried out according to specifications of the AMS 2759/4 at two different holding temperatures, i.e. $982 (\pm 14) ^\circ\text{C}$ and $1093 (\pm 6) ^\circ\text{C}$, for 25 minutes each in a vacuum furnace and gas quenched, as shown in Figure 3-4. Both annealing temperatures are expected to cause the dissolution of the sub-grain microstructure, based on the results shown in Table 2.1. Low temperature stress relieving was not examined as it is associated with negligible effect on the fatigue properties. HIP was performed at $1190 ^\circ\text{C}$ and 145 MPa for four hours, in accordance with the conditions used in literature studies, as listed in Table 2.1. Final grinding step was done for removing surface defects and contaminants generated from the thermal treatments.

Figure 3-4 Setpoint temperature versus time profiles for the solution annealing treatments.

3.3 Defect and microstructure characterisation

Characterisation of microstructural features was done on the small test blocks built adjacent to the sample blocks shown in Figure 3-3a. Samples were grinded to remove end of track defects before making any measurements. Area fractions of defects, including pores and lack of fusion defects, were measured by image analysis of metallographic cross sections. Etching of as-built samples was achieved using a solution of 5g CuCl₂, 100 mL HCl and 100 mL ethanol.

Defect size was described by the Gumbel extreme value distribution, where the parameter values were obtained in accordance with ASTM E2283 for the evaluation of indigenous inclusions. 30 random images, each of area $A_0 = 0.31 \text{ mm}^2$, were taken on the vertical plane of each sample using optical microscope. The equivalent spherical diameter of the largest defect in each image was used as the input for estimating the Gumbel distribution parameters based on the maximum likelihood method. The Gumbel cumulative distribution is described by:

$$F(x) = \exp \left(- \exp \left(- \frac{x - \lambda}{\delta} \right) \right) \quad (3.1)$$

where x is the equivalent spherical diameter of defects, λ and δ are the location and scale parameters of the distribution respectively. For the Gumbel distribution, λ is equal to the mode of the distribution. The mean and standard deviation of the Gumbel distribution can be calculated using standard formulae:

$$\text{Average defect size } 2a = \lambda - \gamma\delta \quad (3.2)$$

$$\text{Standard deviation} = \frac{1}{\sqrt{6}}\pi\delta \quad (3.3)$$

where a is the average equivalent spherical radius of the defect and γ is the Euler-Mascheroni constant. It is to be noted that the use of equivalent spherical diameter for quantifying defect size assumes circular defects, as based on the works of Murakami [68], the maximum stress intensity factor at the crack front of an irregularly shaped defect had been shown to be proportional to the square root of the

defect area projected on to a plane normal to the applied stress. The influence of defect location was not considered, as fractography inspection indicated that the primary fatigue crack origins were always close to the sample surface.

3.4 Mechanical tests

Load-controlled fatigue tests were performed under sinusoidal loading on a servo hydraulic testing system (MTS 810) at a frequency of 5 Hz and ambient conditions. Specimens are fixed by the upper grip on one end, while the lower grip, which is connected to the actuator, move during fatigue testing for exerting the loads. The clamp force for securing the specimens was set to 20 Bars.

Figure 3-5 MTS 810 fatigue testing system.

Samples that did not fail after 10^6 cycles were considered as run-outs. All tests were conducted at a load ratio of $R = 0.1$, except for the some of the tests presented in Chapter 5 where R was varied for examining the mean stress effects. Strain values were calculated based on the machine's actuator outputs, where the actuator displacements were divided by the sample gauge length for approximating the strains.

Therefore, the strain results are only valid for qualitative comparison and should not be taken as the actual values. Tensile tests were done on a universal testing machine using the same sample as the fatigue tests. They were tested in displacement control with a crosshead speed of 0.2 mm/min.

CHAPTER 4 FATIGUE AND FRACTURE BEHAVIOUR OF AS-BUILT PARTS

This chapter establishes the general fatigue and fracture behaviour of L-PBF stainless steel 316L in relation to the processing conditions. Processing variables, including laser power and layer thickness, were varied one factor at a time for fabricating the samples. The critical crack-initiating defects and their effects on crack initiation and the high cycle fatigue properties are presented. Sections of the work reported in this chapter had been published [157, 184, 185].

4.1 Defect characterisation

Relative densities of the samples are shown in Figure 4-1. The effects of E_d on the relative density are consistent with literature data. The processing conditions resulted in five near fully dense samples (S_0 , $0.7P_0$, $1.3P_0$, $1.5P_0$ and $2T_0$), with relative densities above 99.6%, and three porous samples ($3T_0$, $4T_0$ and $0.5P_0$), with relative densities below 99%, being fabricated. Noting the large difference in the densities of $0.5P_0$ and $2T_0$ despite the same input energy densities, metallographic examinations were conducted for evaluating the nature of the defects.

Average sizes of the maximum-occurring defects were obtained from analysis of the Gumbel distribution parameters, as listed in Table 4.1. Average equivalent circular defect sizes for S_0 , $1.3P_0$ and $2T_0$ are smaller than 10 μm ; such defects are indicative of gas pores which are typical of the L-PBF process due to the entrapment of gas pre-existing in the raw stainless steel 316L powder due to manufacturing by gas atomisation, ambient gas from the build chamber [186] or spaces between adjacent powder particles in the powder bed [187]. As rapid cooling and solidification prevented the gas pores from escaping the melting pool, they were retained in the as-fabricated parts.

Figure 4-1 Comparison of relative density against input volumetric energy density of the samples with literature data [121-128].

Table 4.1 Equivalent circular diameters of the maximum-occurring defects as described by the Gumbel Extreme Value Distribution parameters and the corresponding average diameters of the defects.

Samples	Gumbel distribution parameter		Average diameter, $2a$ (μm)
	Location parameter, λ (μm)	Scale parameter, δ (μm)	
S_0	6.6	4.3	9.1 ± 5.5
$1.3P_0$	6.7	2.7	8.3 ± 3.4
$1.5P_0$	8.7	4.8	11.5 ± 6.2
$0.5P_0$	41.8	9.6	47.3 ± 12.4
$0.7P_0$	17.3	6.6	21.1 ± 8.5
$2T_0$	7.1	4.3	9.6 ± 5.6
$3T_0$	43.7	30.7	61.4 ± 39.4
$4T_0$	146.6	51.7	370.9 ± 178.0

Increasing the input energy led to a marginal increase in defect size to about $11.5 \mu\text{m}$, as demonstrated by $1.5P_0$. Besides pores, metallographic examination revealed the

presence of large interconnected and randomly distributed cracks, as shown in Figure 4-2. As larger heat input is known to decrease the threshold stress for cracking during welding [188], the higher laser power used for fabricating $1.5P_0$ could have increased the cracking susceptibility and promoted the formation of thermal cracks.

Figure 4-2 Large interconnected cracks in $1.5P_0$ (horizontal plane).

Defects exceeding $20\text{ }\mu\text{m}$ were present in $0.7P_0$, $0.5P_0$, $3T_0$ and $4T_0$. Figure 4-3 shows representative defects on the vertical planes of the samples. Average defect size in $0.7P_0$ is about $21\text{ }\mu\text{m}$, while those in the other samples are on the order of 50 to hundreds of micrometres. The irregular shapes of the dominating defects are indicative of lack of fusion defects (Figure 4-3a-c), which were formed as result of poor melting and remelting of adjacent tracks and layers under the low input energies. Figure 4-3d shows $0.7P_0$ after etching, where the presence of un-melted powder, as suggested by the coarse dendritic microstructure, further confirms the identity of the defects as lack of fusion defects.

Figure 4-3 Optical micrographs of (a) $3T_0$, (b) $4T_0$, (c) $0.5P_0$ and (d) $0.7P_0$ (vertical planes). Black regions indicate defects.

4.2 S-N curves

S-N curves of the samples are displayed in Figure 4-4. Using S_0 as the reference, the nearly identical S-N curves for $0.7P_0$, $1.3P_0$, $1.5P_0$ and $2T_0$ indicate that the high cycle fatigue properties of were not significantly affected by the changes in processing conditions. Considerable reductions in fatigue resistance are applicable to $0.5P_0$, $3T_0$ and $4T_0$, where the fatigue lives decrease by approximately 92%, 58% and 99% respectively from S_0 . This suggests that the high cycle fatigue resistance is not sensitive to small defects such as those in $0.7P_0$, $1.3P_0$, $1.5P_0$ and $2T_0$, but are strongly affected by larger defects as exemplified by $0.5P_0$, $3T_0$ and $4T_0$.

Figure 4-4 S-N curves of samples made with varying (a) laser power and (b) layer thickness.

4.3 Fatigue crack initiation mechanisms

As crack initiation constitutes a significant portion of the high cycle fatigue life, fractographs were examined for samples tested at loads corresponding to the longer fatigue life region ($>10^5$ cycles) where crack initiation is strongly affected by defect and microstructure because of the small size of the plastic zones. Results indicate three types of failure-critical defects, i.e. grain boundaries, lack of fusion defects and over-heating defects. The following section discusses the fatigue fracture mechanisms corresponding to these defects.

4.3.1 Type I – Microstructure-driven crack initiation

Type I microstructure-driven crack initiation was observed in $2T_0$, $0.7P_0$, S_0 and $1.3P_0$. In Figure 4-5, the boundaries between crack initiation and stable propagation of these samples are marked by dash lines. The crack propagation regions are characterised by the radial marks, which are the distinct lines that radiate outwards from the crack origins and were formed as a result of the intersection of brittle cleavage fractures at different levels of the material [189]. It can be seen that the size of the crack initiation region is the largest for $2T_0$, and decreases from $0.7P_0$ to S_0 , followed by $1.3P_0$. A mix of transgranular ('A') and intergranular ('B') features are observable from the enlarged views of the crack origins. Large areas of intergranular fracture are present in $2T_0$ and $0.7P_0$, whereas for S_0 and $1.3P_0$, the intergranular regions are much smaller.

Figure 4-6 compares the solidification sub-grains at the crack origins of $0.7P_0$ and $1.3P_0$. The image for $0.7P_0$ was taken for a sample loaded at $\sigma_a = 197$ MPa, whereas for $1.3P_0$, sample tested at $\sigma_a = 230$ MPa was examined as at the lower applied stress, intergranular fracture was not as distinct. It can be seen that at the interface between the differently-oriented grains, $0.7P_0$ clearly failed by the intergranular route, revealing the surfaces of the cellular sub-grains; for $1.3P_0$, only part of the sub-grains is discernible at the grain boundary as cracking could have taken place along the inter-cellular boundaries, giving rise to the transgranular mode of fracture.

The above results allude to the significance of processing condition on the fracture of Type I samples. L-PBF processing produces finer grains and particles at lower laser power or higher layer thickness, as the lower energy densities, being associated with smaller melt pools with shorter lifetimes, facilitate the dissipation of heat via faster cooling [190-193]. As the sub-grain boundaries of L-PBF stainless steel 316L are concentrated in dislocations because of the presence of misplaced molybdenum elements and oxide silicate nano-inclusions [135], they are discontinuities in the material matrix and serve to create high local stress concentration, which promote cracking under cyclic loading.

Figure 4-5 SEM images of fatigue crack initiation regions for (a) $2T_0$, (c) $0.7P_0$, (e) S_0 and (g) $1.3P_0$. (b), (d), (f) and (h) are enlarged views of (a), (c), (e), (g) respectively showing transgranular ('A') and intergranular ('B') failure modes.

Figure 4-6 SEM images of cellular sub-grains in (a) $0.7P_0$ and (b) $1.3P_0$ showing transgranular ('A') and intergranular ('B') fracture modes.

Previous experimental studies had shown that for cast materials characterised by the solidification microstructures, fatigue resistance is affected by the size of the dendritic cells and second phase particles [194, 195]. For $2T_0$ and $0.7P_0$, the cellular sub-grains are smaller because of the higher cooling rate. Particles tend to accumulate at grain boundaries rather than intercellular boundaries, such that the grain boundaries are heavily strained, as illustrated in Figure 4-7a. Consequently, particle cracking and the joining of micro-cracks tend to occur at grain boundaries, leading to intergranular fracture. At higher laser power or smaller layer thickness, i.e. S_0 and $1.3P_0$, the coarser cellular sub-grain structures can accommodate more particles, such that fatigue cracks initiated at the intercellular boundaries, promoting transgranular fracture, as shown in Figure 4-7b.

Figure 4-7 Schematic illustration of the crack linkage process (bold lines) for the (a) intergranular and (b) transgranular fracture modes.

4.3.2 Type II – Lack-of-fusion-defect-driven crack initiation

Type II failure involves fatigue crack initiation from surface or sub-surface lack of fusion defects. Both Type I and Type II failures were observed for $0.7P_0$ (of the three samples tested at $\sigma_a = 197$ MPa, one failed by Type I mode, while the rest failed by Type II mode), while for $0.5P_0$, $3T_0$ and $4T_0$, only Type II failures were observed. As shown in Figure 4-8, for $3T_0$ and $0.7P_0$, the primary fatigue crack origins could be traced back to single isolated defects at the sample surfaces, whereas for $4T_0$ and $0.5P_0$, multiple crack initiations took place at large, closely-spaced defects.

Figure 4-8 SEM images of fatigue initiation regions of (a) $3T_0$, (b) $4T_0$ (c) $0.7P_0$ and (d) $0.5P_0$.

The influence of lack of fusion defects on fatigue strength was analysed in terms of the K-T diagram. A planar near surface circular defect was modelled and the fatigue limit as a function of defect size was predicted based on the El Haddad formulation, i.e. Equations (2.7) and (2.8). Experimentally determined fatigue crack growth data were used for estimating ΔK_{th} [155] and F were calculated according to specifications

of the BS7910 [196]. The fatigue limit of S_0 was used for estimating the fatigue limit of a defect-free part $\Delta\sigma_{e0}$. Half crack sizes a of the samples were estimated based on the average defect sizes obtained from the Gumbel distributions in Table 4.1 and the fatigue limits were estimated from the extrapolated S-N curves at 10^6 cycles in Figure 4-4.

The K-T diagram in Figure 4-9 shows that defects in S_0 are in the short crack region where the fatigue limit is not influenced by marginal changes in crack size; defects in $0.7P_0$, $0.5P_0$ and $3T_0$ are in the transition region between short and long fatigue cracks, while those in $4T_0$ are approaching the long crack size. The model estimated effectively the fatigue limits of $0.7P_0$, $0.5P_0$ and $3T_0$, but overestimated that of $4T_0$ by approximately 60 MPa.

As discussed by Pessard et al. [74], cracks in the transition region between microstructurally short and long cracks are in a state of competition between the microstructure- and defect-dominated damage mechanisms. For L-PBF stainless steel 316L, the microstructure-dominated and defect-dominated mechanisms are exemplified by the Type I and Type II failure modes respectively. Based on this understanding, the following comments could be made from results of the K-T and fractography analyses:

Figure 4-9 Kitagawa-Takahashi diagram comparing the El Haddad prediction with the experimental data from this work.

a) *Critical defect size* – As there exists a critical defect size that triggers the transition to Type II failure, and noting that both Type I and Type II failure modes were observed for $0.7P_0$, defects in $0.7P_0$ could be used as non-conservative estimates of the critical defect conditions, e.g. the fractograph in Figure 4-8c indicates critical defect on the order of $50\ \mu\text{m}$. The large size of the critical defect alludes to the material's tolerance towards processing defects, such as porosity and small lack of fusion defect. Leuders et al. [93] attributed such defect-tolerance to the good ductility of stainless steel 316L, which reduces the notch sensitivity. The present result shows that such defect-tolerance is because of the presence of grain boundary defects which act as the weakest links in the material.

b) *Interacting defects* – For parts approaching the long crack region, the overestimated fatigue limit for $4T_0$ is likely to be caused by the interaction between closely-spaced defects. Repeated laser scanning during L-PBF is known to generate periodic defects. The uniformly distributed defects can be characterised by unit cells [197] of length d and interpore spacing λ , as illustrated in Figure 4-10. Sections of the BS 7910 for assessing the acceptability of defects state that when $\lambda \leq 2a$, neighbouring flaws could be considered as interacting. Under such condition, the size of the interacting flaws should be replaced by an effective dimension that is equal to the sum of the individual flaw sizes and the interpore spacing. Referring to the optical micrographs in Figure 4-3, and supported by the fractographs in Figure 4-8, maximum defects in $4T_0$ are clearly interacting. The use of individual defect size for characterising parts containing interacting defects would therefore lead to overestimated fatigue strength. For $0.5P_0$, the defects are further apart relative to the defect size such that the fatigue strength was only slightly overestimated.

Figure 4-10 Schematic illustration of two adjacent unit cells containing circular defects.

4.3.3 Type III – Over-melting-induced crack initiation

Type III failure, corresponding to the over-melting-induced crack initiation mode, occurred in $1.5P_0$. As shown in Figure 4-11a, four major crack origins could be identified and are labelled as O1-O4. The size of the defects at O1 to O3 are about 40 to 50 μm . Figure 4-11b is an enlarged view of the crack origin at O2. A rugged morphology was formed because of the linkage of the primary defect (O2) with the series of small defects on the order of 10 μm on both sides of it. The revelation of these small defects on the fracture surface indicates that they are large enough to trigger fatigue crack initiation. Figure 4-11c shows that the crack origin at O4 could be a surface pore about 10 μm . Intergranular cracking that characterises the Type I samples was not observed.

Figure 4-11 SEM images of fatigue crack initiation regions for $1.5P_0$. (a) Crack initiation sites labelled as O1-O4; (b) and (c) are enlarged views of O2 and O4 respectively.

Since defects on the order of 10 μm were benign for the Type I samples, the present results indicate that cracks initiate more readily from smaller defects at higher laser

energy inputs. This could be attributed to factors such as: (1) high-density thermal cracks, as shown in Figure 4-2, that facilitated cracking; (2) material degradation, due to factors such as grain coarsening and the vaporisation of volatile alloying elements such as manganese [98, 103, 104], which could have led to reduced resistance to crack initiation and growth (tensile strength of $1.5P_0$ is 20 MPa lower than S_0); (3) dislocation annihilation under slower cooling rate, where the reduced grain boundary defects could have rendered the small gas pores the weakest links in the material.

4.4 Comparison with conventional materials

The different fatigue properties between conventional and L-PBF stainless steel 316L had been demonstrated by the S-N curves in Figure 2-7. Fracture studies in this chapter provide the physical basis behind the possible underlying effects of L-PBF processing on the fatigue properties. Knowing that fatigue crack initiation of conventional coarse-grained stainless steel 316L is often attributed to ductile cyclic slip localisation [198], the intergranular and defect-driven crack initiation behaviour of the L-PBF form could be responsible for the different fatigue properties. In this section, the defect characteristics and high cycle fatigue properties of the conventionally-manufactured material are compared with the L-PBF processed form, using the data reported in the previous sections. Reasons behind the variation in the S-N properties between the materials processed by the different manufacturing routes and the significant scatter for the L-PBF form are explained.

4.4.1 Defects and S-N curves

Figure 4-12 shows the Gumbel distribution parameters of the L-PBF samples presented in this chapter and conventional alloy steel [68, 199] collected by Beretta et al. [78]. For the Type I and Type III L-PBF samples, the defect sizes are in general comparable with conventional alloy steel, as exemplified by the similar values for the Gumbel location parameter λ ; for these samples, the L-PBF material shows a slightly wider size distribution, as demonstrated by the larger scale parameter δ . In comparison, defects in the Type II samples are significantly larger with more random distributions as indicated by the larger λ and δ values.

Figure 4-12 Comparison of the Gumbel Extreme Value Distribution parameters of laser powder bed fusion stainless steel 316L and conventional alloy steel. Data for the conventional material were collected by Beretta et al. [78] from the literature [68, 199].

Figure 4-13 compares the high cycle fatigue data obtained from this study with the mean-data curve for conventional austenitic stainless steel [67] and the ASME Code fatigue design curve [68]. It can be seen that the S-N data of the Type I and Type III samples fall around the mean-data curve for the conventional material, indicating comparable fatigue properties. For these samples, the ASME fatigue design curve can be used for providing conservative estimates of the fatigue life. However, the S-N data for the Type II samples are significantly lower than the mean-data curve, with some of them falling below the design curve. As defect sizes for these samples are far beyond the range of the conventional alloy steel, as shown by Figure 4-12, the poor fatigue strength can be attributed to the presence of critical lack of fusion defects which had led to defect-driven failures.

This result sheds light on the S-N properties shown by the literature data for L-PBF stainless steel 316L in Figure 2-7. Specifically, for the data by Spierings et al. [92] and some of the data by Mower and Long [94], the inferior fatigue resistance vis-à-vis the conventional material could have resulted from different extents of crack-initiating lack of fusion defects. For tests where the S-N data are close to the mean-

data curve, crack initiation could have been microstructure-driven or occurred at small processing defects. This suggests that the optimum fatigue properties of the L-PBF material is comparable with the conventional form. Moreover, with better process and defect control, the variability in the S-N data reported by different studies could be reduced.

Figure 4-13 Comparison of S-N data of laser powder bed fusion stainless steel 316L obtained in this research with conventional austenitic stainless steel.

4.4.2 Static-fatigue properties relation

The brittle intergranular and defect-driven crack initiation modes for the L-PBF form could affect not only the fatigue properties, but also the static-fatigue properties relationship. Specifically, the fatigue limit σ_e and tensile strength σ_b of conventional ferrous metals is often described by the following expression [200]:

$$\sigma_e = (0.4 \sim 0.5) \sigma_b \quad (4.1)$$

The S-N curves in Figure 4-4 were replotted after normalising the cyclic stress amplitude by σ_b and applying the Goodman relation (as Equation (4.1) is applicable to the zero mean stress condition). From Figure 4-14, it can be seen that data

normalised by σ_b did not show convergence, and the $\sigma_e/\sigma_b = 0.4\sim 0.5$ relation for conventional ferrous steels no longer produces conservative prediction of the fatigue limit for L-PBF material. In fact, the highest σ_e/σ_b obtained is only about 0.35, indicating that the fatigue strength of L-PBF parts does not match the increased static strength. Based on understanding of the fracture mechanisms for L-PBF stainless steel 316L, intrinsic microstructural defects that trigger the low-energy brittle fracture could have contributed to the general reduction in fatigue resistance of the L-PBF material. Moreover, it is worth noting that the values of σ_e/σ_b is defect-dependent, with $\sigma_e/\sigma_b = 0.25$ for $0.5P_0$ and $\sigma_e/\sigma_b = 0.15$ for $4T_0$.

Figure 4-14 S-N curves of samples normalised by ultimate tensile strength.

The nonlinear relationship between the tensile and fatigue strength had also been reported by Pang et al. [201]. In attempting to apply Equation (4.1) for fatigue prediction of high strength metals, the authors found that for the lower strength materials, fatigue failure occurs by the nucleation of cracks at slips bands, whereas for the high strength materials, the tendency for defect-induced failure increases, leading to the reduced fatigue resistance relative to the tensile strength for the high strength materials. However, this situation is not applicable for the L-PBF samples, where the reduced tensile strength is caused by the presence of lack of fusion defects and the slip-band-induced crack nucleation is not possible. The unique microstructure and defect characteristics of L-PBF materials prevent the direct application of

analysis/solutions obtained based on materials made by conventional manufacturing methods.

Both tensile strength and ductility are key parameters that characterise the static tensile properties of metal alloys. Experimental and simulation results of Hardin et al. showed that defects produce much stronger effects on ductility than on tensile strength [202]. This is supported by results of the present samples (Figure 4-15), where the elongation to failure shows larger percentage reduction with defect area fraction than tensile strength. This implies that the defect-induced failure behaviour could potentially be better represented by the ductility parameter than tensile strength.

Figure 4-15 Ultimate tensile strength and elongation to failure against the relative density of the samples.

Based on the above analysis, the applicability of ductility as the normalising factor for the fatigue limit of L-PBF stainless steel 316L was evaluated. As shown in Figure 4-16, by using the elongation to failure e as the normalisation factor, better convergence was obtained, leading to the relationship:

$$\frac{\sigma_e}{e} = (0.5 \sim 0.6) \text{ (GPa)} \quad (4.2)$$

Figure 4-16 S-N curves of samples normalised by elongation to failure.

Normalising the fatigue limit by the ductility parameter generated significant upward shift of the S-N curves for samples with high defect contents, such that the data nearly converged in the longer life region. Based on this, the linear relationship between fatigue limit and static tensile property can be more appropriately described by employing elongation to failure as the correlation parameter.

4.5 Summary and conclusions

This chapter studies the influence of laser power and layer thickness on the S-N properties and fatigue crack initiation mechanisms of L-PBF stainless steel 316L. The following conclusions can be drawn from the results obtained:

- 1) Regardless of the processing parameter, the fatigue properties and fracture mechanisms are strongly related to the process-induced microstructure and defects: optimum processing produces parts devoid of critical defects and incurs the microstructure-driven crack initiation mode; over-melting leads to reduced defect-tolerance, where the critical defect size for fatigue crack initiation is reduced; lack of fusion defects have the most significant influence on the S-N properties, where the fatigue strength reduces with increasing defect fraction.

- 2) For the microstructure-driven crack initiation, fatigue crack initiation from slip planes in dendrite cells or grain boundary particles leads to defect-tolerant behaviour, such that gas pores on the order of 10 μm are benign to fatigue crack initiation.
- 3) For the lack-of-fusion-defect-driven crack initiation mode, fatigue strength decreases with defect size. The strong interaction among closely-spaced defects leads to rapidly reduced fatigue resistance as compared to the case of isolated defects with equivalent defect size. The effects of interacting defects therefore need to be considered for high cycle fatigue modelling.
- 4) Fatigue strength varies directly with both tensile strength and ductility. However, it shows a stronger linear correlation with ductility as both parameters are sensitive towards defects via the mechanism of crack nucleation. The conventional linear relation between tensile strength and fatigue limit is therefore not suitable for assessing the fatigue properties of L-PBF materials due to the influence of crack-initiating defects and microstructures.

CHAPTER 5 ROLES OF CYCLIC PLASTICITY IN FATIGUE AND FRACTURE

Besides defects, variability in L-PBF processing could induce subtle microstructural variations, e.g. grain size, evaporation of alloying elements, anisotropic microstructural arrangements, etc., which affect the material properties, as well as the local and macroscopic deformation behaviour. These factors do not only impinge on the high cycle fatigue properties via the fatigue crack initiation mechanism under nominal elastic loading, in the presence of large tensile mean stress, the differences in the elastic-plastic properties could also impact the fatigue properties by the activation and accumulation of intense plastic strains, i.e. ratcheting. In this chapter, the roles of cyclic plasticity and fatigue-ratcheting interactions in the high cycle fatigue properties and fracture mechanisms of L-PBF stainless steel 316L are examined for S_0 samples built at different build orientations and subjected to post-processing treatments (annealing or hot isostatic pressing). Based on the empirical results, a modification to the traditional Goodman equation is proposed to account for the added effects of cyclic plasticity. Sections of the work reported in this chapter had been published [203].

5.1 Determination of fatigue loading condition

Appropriate fatigue loading conditions for evaluating cyclic plasticity effects in the $10^4 - 10^6$ cycles region were determined by considering the tensile properties, as shown in Table 5.1. Noting the small standard deviations for conditions subjected to repeated tests, single test results were used for conditions with limited samples, i.e. the vertical as-built and annealed samples.

For the as-built parts, anisotropic tensile properties are exemplified by the horizontal orientation having higher tensile strength but lower ductility than the vertical direction, in agreement with results reported in the literature [130, 131]. The different plastic properties are suggestive of orientation-dependent fatigue responses, especially under high stress conditions involving cyclic plasticity. To examine this

effect, both horizontal and vertical as-built samples were subjected to fatigue tests at $R = -1, 0.1$ and 0.7 , where at the highest load ratio, the maximum stress $\sigma_{\max}$ exceed the yield strength of the materials.

Orientation effects were examined for the HIP samples as well. As shown in Table 5.1, the yield strengths after HIP are nearly half of the as-built condition for both build orientations. In fact, the tensile strength properties of the HIPed samples are very close to those of the wrought form, where based on the experimental results of Carlton et al. [11], the ultimate tensile strength of wrought stainless steel 316L is 545 MPa and the yield strength is 245MPa. Similar extent of reduction in yield strength had been reported in the literature for L-PBF stainless steel 316L subjected to HIP [204], where the authors explained the results in terms of the homogenisation of the microstructure upon HIP treatment and indicated that both the HIPed and wrought materials showed similar dimple fracture when subjected to tensile testing. Consequently, tests were done at $R = 0.1$ only, as $\sigma_{\max}$ at this load ratio is above the materials' yield strengths and allows the cyclic plasticity effects to be examined. Note that tensile anisotropy was reduced by the HIP treatment, where the difference in ultimate tensile strength was reduced from 100 MPa for the as-built samples to 40 MPa after HIP, while the difference in elongation to failure was reduced from 25% to 14%. The yield strengths for the differently-oriented samples are nearly homogeneous after HIP.

Similarly, both annealing treatments led to strength reduction and ductility improvement. For these materials, $\sigma_{\max}$ at $R = 0.1$ exceeds the yield strengths such that the cyclic plasticity effects could be examined by fatigue testing at $R = 0.1$ only.

Table 5.1 Ultimate tensile strength, 0.2% offset yield strength and percentage elongation to failure of the samples. Conditions without standard deviations are single test results.

Sample	Ultimate tensile strength (MPa)	Yield strength (MPa)	Elongation to failure (%)
As-built (horizontal)	723 ± 7	586 ± 29	42.7 ± 2.2
As-built (vertical)	620	500	67.7

Annealed at 982 °C (horizontal)	673	440	51.9
Annealed at 1093 °C (horizontal)	665	420	56.1
Hot isostatically pressed (horizontal)	626 ± 3	283 ± 6	53.6 ± 1.4
Hot isostatically pressed (vertical)	588 ± 4	286 ± 4	63.4 ± 2.8

5.2 S-N curves

5.2.1 Effects of build orientation

S-N curves for the differently-oriented as-built and HIPed samples are displayed in Figure 5-1. For the as-built parts (Figure 5-1a), orientation effects can only be observed at $R = 0.7$. The lower fatigue strength of the vertical direction at this loading condition is implicative of its poorer resistance to plastic deformation, consistent with its lower static strength. This suggests that the tensile anisotropy had translated to fatigue anisotropy because of the plasticity-dominated failure. As the load ratio reduces, fatigue loading is associated with lower mean stress σ_m and stress amplitude σ_a for the tested life region. The effect of cyclic plasticity diminishes with the lower mean stress, such that plasticity-induced fatigue anisotropy reduces and the S-N data for the differently-oriented parts nearly overlap. Instead, the increased stress amplitude could have led to greater contribution of fatigue activities on deformation [82], such that as stress reduces, the smaller plastic zones rendered the development of fatigue crack more sensitive to microstructural heterogeneities at the crack tip [149]. This could be responsible for the considerable scatter in the S-N data at smaller stresses, e.g. $\sigma_a = 197$ MPa and $R = 0.1$. It is to be noted that as single tests were conducted at different loads at $R = 0.7$, the validity of the trend shown for this loading condition should be confirmed by further experimental studies.

For parts subjected to hot isostatic pressing (Figure 5-1b), no obvious fatigue anisotropy can be detected. This suggests that the extent of tensile anisotropy is too

small to inflict appreciable effect on the fatigue properties. Nonetheless, at the smallest tested load, i.e. $\sigma_a = 162$ MPa, the large variations in fatigue life, as in the case of the as-built parts, may again be attributed to the transition from plasticity- to fatigue-dominated failure as cyclic stress reduces.

Figure 5-1 S-N curves of horizontal and vertical (a) as-built samples tested at different load ratios and (b) hot isostatically pressed samples tested at $R = 0.1$.

5.2.2 Effects of heat treatment

The effects of annealing or HIP on the S-N properties are shown in Figure 5-2a. The different tensile properties after the post-processing treatments are clearly reflected in the finite life fatigue properties. Fatigue strength reduces with annealing intensity, where the fatigue life decreased by about 42% and 47% for the lower and higher annealing temperatures respectively at $\sigma_a = 200$ MPa. This is consistent with the reductions in tensile strength after annealing. In spite of the considerable reduction in yield strength, S-N data for the HIP parts are generally within the data range of the annealed parts. Normalising the maximum cyclic stress by the respective yield strengths of the samples shows that the fatigue to yield strength ratios are greater than unity after heat treating (Figure 5-2b). This alludes to the significance of elastic-plastic deformation on the fatigue properties. In particular, the HIP samples are exemplified by much better cyclic plastic resistance than both the as-built and annealed parts despite the reduced fatigue resistance.

Figure 5-2 (a) S-N curves of as-built and heat treated horizontal samples tested at $R = 0.1$; (b) replotted S-N curves by normalising the maximum stress by the yield strength.

Fatigue limits did not deteriorate as much as the fatigue strength in the finite life region, as runouts were obtained at similar loading conditions for the annealed and as-built parts. For these samples, nominal elastic deformation was the driving failure mode. Fatigue limit for the HIP samples is lower. This is because the samples experienced plastic damage within the entire tested life region.

5.3 Fatigue fracture behaviours

Results from Chapter 5.2 indicate that build direction and the post-processing treatment affect the high cycle fatigue properties primarily via the combined actions of ductile and fatigue fractures. Specifically, difference in the static tensile properties is reflected in the S-N properties when the global plastic deformation mode is activated, e.g. the as-built parts at $R = 0.7$ and the finite-life heat treated parts; fatigue activities involving the initiation of fatigue cracks become increasingly pertinent as stress decreases and proceeds to nominal elastic loading. The fatigue fracture mechanisms associated with cyclic plasticity and fatigue crack initiation are discussed in this section.

5.3.1 Ratcheting

Cyclic plasticity was examined in terms of the ratcheting mechanism, where the ratcheting strain ε_r was calculated according to the formula:

$$\varepsilon_r = 0.5(\varepsilon_{min} + \varepsilon_{max}) \quad (5.1)$$

where ε_{min} and ε_{max} are the minimum and maximum axial strains respectively of a particular cycle. The ratcheting strain against load cycle plots of the samples are shown in Figure 5-3. The curves show resemblance to a material's deformation behaviour under creep, consisting of the primary, secondary and tertiary deformation phases. The primary stage is associated with the generation of a large initial strain, which is suppressed from further increase by cyclic hardening; the secondary stage involves a balance between geometric softening and cyclic hardening, which determines the ratcheting rate; the tertiary stage is exemplified by significant necking and rapid rupture of the part.

For the horizontal as-built parts (Figure 5-3a), the nearly flat curves for the secondary deformation stage at $R = 0.1$ are indicative of close-to-zero ratcheting strain accumulation rates. At the higher load ratio of $R = 0.7$, however, considerable plastic deformation was generated, as shown by the higher ratcheting strain and strain rate. Noteworthy is the longer life at $R = 0.7$ than $R = 0.1$ despite the greater ratcheting

damage. This is because while the higher mean stress at $R = 0.7$ served to exhaust the ductility of the material, the smaller stress amplitude, being the key driver for the extent of cyclic slip movement, contributed to less severe fatigue damage. Besides, further strain reduction, as a result of more intense strain hardening at higher mean stresses [205], could have alleviated the damage caused by ratcheting, leading to the steady state ratcheting strain rates. The simultaneous effects of ductility exhaustion, strain-induced hardening and fatigue competed and led to the overall improved fatigue life at $R = 0.7$, in spite of the more significant ratcheting. This result is suggestive of the material's low sensitivity to load ratio or mean stress relative to the stress amplitude. Similar 'mean stress insensitivity' had been reported for conventionally-produced stainless steel 316L [206, 207], indicating that the general cyclic plastic behaviour is not affected by the L-PBF fabrication route.

Figure 5-3b shows that the vertical orientation is associated with larger ratcheting strains, as reflected by the higher ratcheting strain and strain rate in comparison with the horizontal samples. As the applied stress at $R = 0.7$ and $\sigma_m = 558$ MPa surpassed the tensile strength of the material, the unconstrained increase in true stress due to localised deformation and necking resulted in failure within 10^4 cycles. This explains the inferior S-N properties of the vertical samples at $R = 0.7$, confirming that the fatigue anisotropy was consequent of the different plastic properties. Again, despite the higher mean stress, the sample tested at $R = 0.7$ and $\sigma_m = 527$ MPa survived longer life cycles than those tested at the lower load ratio because of the lower applied stress amplitude. This indicates that mean stress insensitivity is relevant regardless of the build direction.

Figure 5-3 Ratcheting strain against load cycle of (a) horizontal and (b) vertical as-built samples, and (c) heat treated horizontal samples. Unfilled symbols in (c) represent data for runouts tested at $\sigma_m = 221$ MPa and 201 MPa for annealing at 982 °C and 1093 °C respectively.

Figure 5-3c shows the ratcheting strain curves of the annealed and HIP parts. For the runouts, insignificant amount of ratcheting strains was generated and they did not accumulate with the progression of cycles. This confirms that the longer life fatigue properties pertain to the global elastic deformation mode. For the finite life samples, larger initial strains and longer primary deformation phases ($> 10^2$ cycles) than the as-built samples can be observed. This is because unlike the as-built samples where the dislocations rearranged into a new configuration upon straining, gradual generation of dislocations occurred in the absence of the pre-existing dislocation network after heat treatment [136]. The latter microstructure served to produce more sustained resistance to deformation and better cyclic hardening capacity [208], such that stable ratcheting resulted despite the large initial strain. Together with the better improved exhaustion capacity, this explains the better cyclic plastic properties after heat treatment shown in Figure 5-2b.

Despite the substantial strain induced after hot isostatic pressing, its fatigue life is comparable with the annealed parts. The higher applied stress relative to the yield strength could have led to stronger cyclic hardening such that the strain accumulation rate during the secondary deformation phase was not significantly aggravated. This explains the similar finite life properties of the HIP and annealed parts in Figure 5-2a, as well as the better cyclic plastic properties of the HIP parts in Figure 5-2b.

5.3.2 Crack initiation

Optical micrographs indicating defects in the samples are shown in Figure 5-4. As mentioned in the earlier chapters, the as-built part contained small sporadic pores (Figure 5-4a). Such defect structure was not altered at the lower annealing temperature (Figure 5-4b). Annealing at 1093 °C, however, incurred considerable increase in the defect population (Figure 5-4c), with the size of the defects being comparable with the as-built condition (at 5-10 μm). For the HIP sample (Figure 5-4d), submicron voids with average sizes on the order of 0.5 μm constituted the prevailing defects.

Figure 5-4 Optical micrographs of (a) as-built, (b) annealed (982 °C), (c) annealed (1093 °C) and (d) hot isostatically pressed samples. Dark pixels indicate defects.

The increase in defect density at the higher annealing temperature is likely to be attributable to the nucleation and coalescence of cracks at the pre-existing defects. As the samples were fabricated in an argon environment, the low solubility of argon in iron could have prevented the dissolution of argon gas into the material matrix [209]. Remnant gas pores could have expanded and joined to form larger pores upon heating because of the internal pressure [210]. In addition to gas pores and intercellular spaces, pre-existing or thermally-induced micro-cracks could have served as additional nucleation sites for gas diffusion and contributed to the higher defect density. Specifically, strong oxide formers, e.g. Mn, Cr and Si in stainless steel 316L, could have led to the formation of oxide layers on the powder surface during gas atomisation [211] and prevented effective wetting of the melt pool, causing discontinuities in the as-built parts. Thermal cracks could be generated via reheat cracking [212, 213], where stress relaxation as temperature ramps up during annealing could have caused the accumulation of intense plastic strains at weak sites, such as grain boundaries, and triggered intergranular cracking. This process is

especially relevant to L-PBF parts, considering the significant residual stress resulted from rapid cooling [214, 215]. As the higher annealing temperature and the longer time taken to reach the temperature promoted the expansion and mobility of gas pores, as well as the formation of reheat cracks, such thermally-induced defects were observed in the higher-temperature-annealed parts only.

Hot isostatic pressing led to consolidation of the pre-existing defects but generated submicron spherical voids, as shown on the optical micrograph in Figure 5-4d. Upon examination of the rapid rupture region of fatigue samples (Figure 5-5), it appears that the voids were occupied by second phase particles which could have been dislodged during metallographic sample preparation. Chemical compositions of the site 'A' were examined by EDX analysis. As listed in Table 5.2, the particle contained higher concentrations of oxygen, silicon and manganese than the material matrix. Such silicon- and manganese-rich oxide particles existed on the nanoscale in as-built stainless steel 316L [127, 129], where they were formed because of remnant oxide on the powder surface reacting with the alloying elements. The high temperature and long processing time of HIP could have promoted coarsening of the nano-inclusions to submicron size [216, 217], which became critical in causing micro-crack nucleation and was responsible for the poor yield strength.

Figure 5-5 SEM fatigue fracture image of hot isostatically pressed sample showing micro-crack nucleation at spherical inclusions in the rapid fracture region.

Table 5.2 Elemental compositions in weight percentage of region ‘A’ in Figure 5-5 and region ‘B’ in Figure 5-10e, and the material matrix of hot isostatically pressed samples.

Region	O	Si	Cr	Mn	Fe	Ni	Mo
‘A’	17.4	6.9	14.2	4.7	45.7	7.2	4.0
‘B’	64.4	5.8	13.0	15.7	1.04	0.0	0.0
Matrix	0.0	1.87	18.3	1.1	63.3	10.8	4.6

Fractography analysis were done for parts tested at $R = 0.1$ in order to examine the influence of the defects on fatigue crack initiation. Comparing the fatigue fractographs for the horizontal and vertical as-built parts at $\sigma_a = 197$ MPa in Figure 5-6, anisotropic microstructural arrangements could have affected the fracture mechanisms by operating at three length scales:

- 1) Macro-grain level: Different macro-grain arrangements caused the size and shape of the crack origins to differ between the samples (Figure 5-6a-b).
- 2) Sub-grain colony level: Individual sub-grain colonies, being sheet-like (as indicated in Figure 5-6c-d), could have affected the lengths of the local crack propagation paths, e.g. crack path for the vertical sample is apparently longer because of propagation across the long edge of the sub-grain colony.
- 3) Cellular sub-grain level: Cellular sub-grains within the colonies are rod-like, which could have resulted in anisotropic crystallographic arrangements with respect to the loading direction and affected the local transgranular fracture mode, e.g. the vertical sample adopts a faceted surface because of stronger cleavage-type fracture.

As the microstructure features affect the local stress concentration and mode of fracture, as well as the crack propagation path, they could impinge on the crack initiation and propagation lives and led to the observed scatter of the high cycle fatigue data.

Figure 5-6 SEM fatigue crack initiation regions of as-built (a) horizontal and (b) vertical samples; (c) and (d) are enlarged views of the boxed regions in (a) and (b) respectively.

Figure 5-7a shows that the sample annealed at 982 °C preserved the intergranular crack initiation mode. Nonetheless, the ultrafine cellular sub-grain structures are not visible and the grain boundaries are characterised by smoother surfaces. This is because the heat treatment had led to grain growth at the sub-grain level with the macro-scale grains remained unaffected, consistent with reported results on the effects of annealing at this temperature (Table 2.1). As the defect structure was not significantly altered, as indicated by the metallography results in Figure 5-4, the macro-scale grain boundaries remained the weakest links under high cycle fatigue loading.

The higher annealing temperature caused further consolidation of the sub-grains, as large grains on the order of tens of microns are traceable from the faint lines on the surface of the defect at the crack origin (Figure 5-7b). Considering the significant increase in defect, micro-cracks could have initiated at the edges of the thermally-induced defects, as illustrated in Figure 5-8. This process could have been facilitated

by the more intensive annihilation of grain boundary dislocations, which were responsible for the intergranular fracturing mode of the as-built parts.

Figure 5-7 SEM fracture images of samples (a) annealed at 982 °C, showing intergranular crack initiation, and (b) annealed at 1093 °C, showing crack initiation from thermally-induced defect.

Figure 5-8 Schematic illustration of the crack linkage process for the (a) as-built and (b) high-temperature-annealed parts.

However, the crack origin, as observed on the fracture surface, is considerably bigger than the defects in Figure 5-4c. While crack initiation seeks the maximum-occurring defects, large defects could have been missed because of the limited inspection area of metallography examination. In fact, smaller defects comparable in size with those in Figure 5-4c were observed along the crack propagation path, as shown in Figure

5-9. Judging by compressed shape of the thermal defects, and noting that measurements on two-dimensional metallographic sections are susceptible to stereological errors, smaller defects could have been on the optical micrographs, resulting in the mismatch size of the defects [69]. Therefore, the present results suggest that high-temperature-annealing promotes the transition from intergranular- to defect-driven crack initiation. However, as the fatigue limits of the as-built and annealed samples are similar based on runouts obtained at 10^6 cycles, understanding the influence of the defects on the S-N properties would require further investigation in the longer life region.

Figure 5-9 Possible secondary crack initiation at thermally-induced defect in sample annealed at 1093 °C.

For the HIP samples, defect annihilation led to crack initiation by cyclic slip, as shown in Figure 5-10a. However, significant crack branching can be observed as the primary crack propagates because of secondary cracking at closely-spaced spherical pits (Figure 5-10b-c). This indicates that other than lowering the yield strength, the coarsened inclusions, being highly clustered, also generated sufficient local stress concentration that promoted cracking under cyclic loading.

Figure 5-10 SEM fracture images of hot isostatically pressed samples. (a) Crack initiation and early crack propagation regions; (b)-(c) enlarged views of the boxed regions in (a) showing crack paths being directed by highly-clustered oxide inclusions; (d)-(e) crack initiations from unfused region and oxygen-contaminated particles respectively.

Furthermore, fractography analysis confirmed the fatigue life scatter at $\sigma_a = 162$ MPa to be caused by different crack origins. Figure 5-10d shows a vertical sample with fatigue life of 7.6×10^5 cycles failed from an unfused region on the order of $10 \mu\text{m}$. The low-solubility argon gas could have prevented complete elimination of gas pores and thermal cracks by HIP, leaving behind defects with contacting surfaces [218]. In comparison, crack initiation from a large agglomeration of oxygen-rich particles

(refer to EDX analysis of region 'B' in Table 5.2) led to significantly reduced fatigue life of 3.5×10^5 cycles (Figure 5-10e). Contaminated particles, generated during powder recycling, could be among the factors that led to the presence of such oxygen-rich compounds. Therefore, the results suggest that although HIP reduces the scale of critical defects from macro-scale grain boundaries or lack of fusion defects to submicron inclusions, parts become more sensitive to defects which could not be removed by HIP and are often less detectable, e.g. unfused regions or contaminants. This is responsible for the significant scatter in the S-N data under fatigue-dominated failure, rendering the fatigue properties less predictable.

5.4 Stress-based modelling

Build orientation and post-processing treatments had been demonstrated to affect the high cycle fatigue properties of L-PBF stainless steel 316L primarily via the mechanism of fatigue-ratcheting interaction; critical defects and microstructure heterogeneities are generated, but they are relevant to the longer life region where failure is dominated by fatigue activities. As the accumulative nature of ratcheting can lead to unexpected failure of engineering structures e.g. thinning and ovalisation of pipes [219], it becomes imperative to incorporate the plastic damage mechanism for fatigue modelling.

The effects of ratcheting could be modelled using the stress [220, 221], strain or energy [222-224] approaches. From the engineering standpoint, the stress-based approach is favoured as it allows the nominal stress associated with high cycle fatigue and ratcheting to be described explicitly. The applicability of traditional parameter-based stress models for predicting the S-N properties of L-PBF stainless steel 316L are examined in this section. Additional fatigue tests were conducted at $R = -0.8, -0.5, -0.2$ and 0.5 using horizontal as-built samples and a few post-processed samples for further experimental evaluation of the stress-dependent effects of fatigue-ratcheting interaction. Based on the experimental results, a modified Goodman model was proposed for fatigue prediction in the $10^4 - 10^6$ cycles life region.

5.4.1 Traditional models

Recall Equation (2.1), for fully reversed loading, the straight-line S-N relations on log-log plots is described by the form of the Basquin equation [41]:

$$\sigma_a = \sigma'_f (2N_f)^b \quad (5.2)$$

where σ'_f and b are the fatigue strength coefficient and exponent respectively. The effect of mean stress can be estimated by adjusting σ_a to the equivalent stress amplitude σ_a^{eq} according to the Goodman [83] (Equation 2.11), Gerber [84] (Equation 2.12) or Walker [85] (Equation 2.13) equation. For the Walker equation, the Walker parameter γ adopts a value of 0.527 for stainless steel 316L [225].

Figure 5-11 shows σ_a^{eq} obtained from applying the above-mentioned equations and compared with the Basquin curve for the horizontal as-built condition. To present the data on the same plot, σ_a^{eq} was normalised by the respective tensile strengths, except for the vertical as-built samples, for which the data were normalised by the tensile strength of the horizontal direction, as both have similar Basquin curves (Figure 5-1a).

Figure 5-11a shows that the Goodman relation achieved reasonable predictions for the longer life samples at small load ratios only. As life decreases or R increases, the predicted σ_a^{eq} are above the Basquin curve, indicating that the parts performed better than the Goodman relation. The Gerber equation, in comparison, shifted the data points downwards such that it predicted the shorter life conditions effectively, but underestimated σ_a^{eq} as life increases (Figure 5-11b). The Walker equation (Figure 5-11c) produced the most accurate predictions, except for the heat-treated samples at higher stresses and the vertical sample at $R = 0.7$.

Figure 5-11 Normalised equivalent stress amplitude against fatigue life for the (a) Goodman, (b) Gerber and (c) Walker models. Solid lines are the Basquin equations for the as-built condition and dotted lines indicate factor of two on life. Meanings of symbol notations: AB – as-built; HT – heat treated; H – horizontal sample; V – vertical sample; R – load ratio, with values in brackets.

The above results indicate that the Goodman and Walker equations do not predict well for cases of significant ratcheting. This can be interpreted by noting that the Goodman equation was formulated for high cycle fatigue where the global deformation is elastic. Since cyclic plasticity improves the fatigue properties of stainless steel 316L via the hardening mechanism, the conservative predictions of the Goodman model are reasonable. Moreover, as the linear relationship between the stress amplitude and mean stress for a specified number of cycles to failure, as assumed by the Goodman model, is not demanded by any physical mechanism [226], it becomes inadequate when a wide range of stress, covering both elastic and plastic deformation phenomena, is considered [206, 221, 225, 227]. The same reasoning applies for the parabolic relation of the Gerber equation, resulting in the models being effective for fatigue prediction over limited stress ranges only. The Walker model produced the best results, as it was correlated with the stress fields at crack tips [85], which characterise the plastic deformation state more effectively. Nonetheless, the result shows that it does not predict well when the extent of ratcheting strain accumulation is severe.

5.4.2 Modification to traditional models

As the data points fall within the Goodman and Gerber predictions, and noting that these models differ in terms of the degree of the σ_m/σ_b term, i.e. 1 and 2 respectively, the nonlinear relations between the stress amplitude and mean stress can be modelled by incorporating a stress-dependent function η to the degree of σ_m/σ_b , i.e.

$$\sigma_a^{eq} = \sigma_a / [1 - (\frac{\sigma_m}{\sigma_b})^\eta] \quad (5.3)$$

Ideally, $\eta = 1$ for pure elastic loading, i.e. the Goodman relation. The amount of deviation of η from unity serves to provide a quantitative measure of the nature and extent of cyclic plasticity. Specifically, as a material's fatigue performance under fatigue-ratcheting interaction is influenced by the combined effect of geometric softening, cyclic hardening/softening and fatigue activities, stronger cyclic hardening will give rise to larger η : in particular, $\eta > 1$ to reflect the conservative nature of the Goodman relation. In comparison, η for a cyclic softening material is lower as the

greater accumulated strains are associated with more significant deformation and damage [228, 229], which render the Goodman relation less conservative. (See, for example, the Goodman predictions for cyclic hardening materials such as stainless steels 304 [221] and 316L [207], and cyclic softening materials such as Inconel 718 [221, 230] and a copper alloy [231]; for the cyclic softening materials, η could be smaller than 1 at low-to-moderate mean stresses.)

The η versus cyclic stress plots are shown in Figure 5-12. For a fixed positive load ratio, e.g. $R = 0.1$, η increases from 1 as mean stress (Figure 5-12a) or maximum stress (Figure 5-12b) increases because of the greater cyclic hardening at higher stresses. The normalisation term σ_b allowed the data for the differently-oriented and heat treated samples to converge, as it quantifies the extent of plasticity effect. However, two observations could be made from the plots and require further elaborations:

- 1) At positive load ratios, R dependency applies for the η vs. σ_m/σ_b plot, but not for the η vs. σ_{max}/σ_b plot.
- 2) At negative load ratios, as cyclic plasticity is not relevant because of the nominal elastic loading, the mechanism that invoked the changes in η needs to be investigated.

To explain the first observation, it is to be reminded that besides cyclic hardening, mean stress insensitivity at higher load ratios is also attributable to the lower stress amplitudes, which incur less fatigue damage. Therefore, σ_m accounts only for the mean stress effects, but not the reduced fatigue damage in relation to the lower stress amplitudes. This resulted in the smaller η at higher load ratios on the η vs. σ_m/σ_b plot. Instead, as $\sigma_{max} = \sigma_m + \sigma_a$, employing the maximum stress as the correlation term summed up the effects of all operating mechanisms and unified the data.

Thus, using σ_{max} as the correlation term, the effects of fatigue-ratcheting interaction on η can be expressed using an exponential function:

$$\eta = K \exp \left[c_0 \left(\frac{\sigma_{max}}{\sigma_b} \right) \right] \quad (5.4)$$

where K and c_0 are material constants and their values are as shown in Figure 5-12b. A positive c_0 , such as that for stainless steel 316L, is reflective of the direct relationship between η and $\sigma_{\max}$ due to hardening, while cyclic softening suppresses η at higher stresses and gives rise to less positive c_0 ; K dictates the order of magnitude of η , which, as mentioned, quantifies the deviation of actual material properties from the Goodman relation due to cyclic plasticity.

Figure 5-12 Variation of η with (a) σ_m and (b) $\sigma_{\max}$ at different load ratios. Meanings of symbol notations: AB – as-built; HT – heat treated; H – horizontal sample; V – vertical sample; R – load ratio, with values in brackets.

For the negative load ratios, as the corresponding loading conditions are in the elastic domain, the changes in η are not related to cyclic plasticity. Denoting by η' to differentiate the different operating mechanism, an attempt at explaining the

observations is made here. A mechanism that often explains fatigue properties at small load ratios is roughness-induced crack closure. For L-PBF parts, closure effects could be more pronounced because of the ultrafine-grained microstructure, as illustrated in Figure 5-13. In comparison with the conventional coarse-grained form, where the propagation of short fatigue cracks is confined to the local microstructure such that roughness-induced crack closure cannot be activated [91], the cellular sub-grains and differently-oriented sub-grain clusters for L-PBF materials promotes closure by reducing the effective grain size and generating asymmetric deformation at the crack tip [232, 233]. This reduces the crack growth rate and improves the fatigue resistance [90], which demand larger η' . This mechanism could further explain the R independency for the heat treated sample, e.g. at $R = -0.5$, as the coarsened grains after heat treatment created larger effective grain sizes and less significant closure. In addition, since closure is not effective under large crack tip opening displacements [87-89], the R independency at the positive load ratios is consistent with this concept.

For the negative load ratio cases, while the fracture mechanisms leading to the results in Figure 5-12 require further investigation, the prediction accuracy can be improved by accounting for η' , which can be modelled by incorporating the load ratio into Equation (5.7), i.e.:

$$\eta' = K \exp \left[(c_1 - c_2 R) \left(\frac{\sigma_{max}}{\sigma_b} \right) \right] \quad (5.5)$$

where c_1 and c_2 are empirical constants for which the values are as shown in Figure 5-12b. It is to be emphasised that Equation (5.5) was derived purely from the empirical data and further study of the fracture mechanisms are necessary to support its validity.

Figure 5-13 Schematic illustration of roughness-induced crack closure in coarse-grained material and ultrafine-grained laser powder bed fusion material with solidification microstructure.

5.4.3 Model validation

Significant improvements in the prediction accuracy were achieved by applying the proposed modifications, as shown in Figure 5-14, where most of the predicted fatigue lives are within a factor of two of the experimental values. The constant fatigue life diagrams in Figure 5-15 show that the σ_a vs. σ_m relations are life-dependent, unlike those assumed by the traditional models. At 10^4 cycles, the material is more fatigue resistant than that predicted by the Goodman equation because of cyclic plasticity at positive load ratios and the possible effects of roughness-induced crack closure at negative load ratios. At 10^6 cycles, difference between the predictions of the proposed model and the Goodman model diminishes, as failure is increasingly dominated by fatigue activities with elastic dominance and requires smaller adjustments for η . The slightly worse performance at the lower load ratios could be due to the brittle crack initiation mode caused by the process-induced defects. In addition, as accounting for

the load ratio effect, i.e. Equation (5.5), produced less conservative results, load ratio independency may be assumed for fatigue design purposes.

Figure 5-14 Normalised equivalent stress amplitude against fatigue life obtained from applying the proposed model. Solid line is the Basquin line and dotted lines indicate factor of two on life. Meanings of symbol notations: AB – as-built; HT – heat treated; H – horizontal sample; V – vertical sample; R – load ratio, with values in brackets.

Figure 5-15 Comparison of the constant life diagrams for the Goodman and the proposed models.

The proposed modification was further cross-validated against (1) L-PBF stainless steel 316L test data reported by Spierings et al. [92], (2) rolled stainless steel 316L [182] and (3) other L-PBF alloys, including AlSi10Mg [234-236] and Inconel 718 [237, 238]. These materials were selected as the reported maximum applied cyclic stresses exceed the yield strengths in the high cycle fatigue region, making ratcheting the relevant failure mechanism. Table 5.3 shows that all materials, with the exception of Inconel 718, exhibit direct relationships between η and $\sigma_{\max}$, as indicated by the positive c_0 . In fact, c_0 adopts a nearly constant value of 2.13 for these materials.

Table 5.3 Material constants and sources of data for validating the proposed model.

Materials	K	c_0	η	Sources of data
L-PBF stainless steel 316L	0.261	2.131	0.9 – 2.2	Basquin equation: this work Positive R data: this work Validation data: Spierings et al. [92]
Rolled stainless steel 316L	0.434	2.130	2.2 – 3.1	All data adapted from Huang et al. [182]
L-PBF AlSi10Mg	0.135	2.133	0.2 – 0.6	Basquin equation: Uzan et al. [236] Positive R data: Aboulkhair et al. [235] Validation data: Maskery et al. [234] Basquin equation: Yadollahi et al. [238]
L-PBF Inconel 718	1.486	-0.249	1.0 – 1.4	Positive R data: Konečná et al. [237] Validation data: Konečná et al. [237]

The large positive c_0 for stainless steel 316L and AlSi10Mg suggests strong cyclic hardening, where conventionally manufactured stainless steel 316L cyclic hardens by strain-induced martensitic transformation [239, 240], while that for L-PBF AlSi10Mg pertains to precipitations-induced pinning effect [241]. The relatively constant c_0 values across the materials confirm that σ_b provided effective scaling of the plasticity effects. The significance of adjusting for the stress-dependent effects of cyclic plasticity via the $c_0(\sigma_{\max}/\sigma_b)$ term is demonstrated by the more comparable slopes of the predicted results with the gradients of the Basquin lines as compared to the

Goodman results, as shown in Figure 5-16a-c. For Inconel 718, the close-to-zero c_0 is attributable to its cyclic softening properties [242], consistent with the discussions that c_0 reduces with cyclic softening. In fact, this had led to the lower η such that the fatigue properties were well predicted by the Goodman equation, as shown in Figure 5-16d.

Figure 5-16 Validating the proposed model against (a) L-PBF stainless steel 316L, (b) rolled stainless steel 316L, (c) L-PBF AlSi10Mg and (d) L-PBF Inconel 718. Solid lines are the Basquin equations obtained from the references specified in Table 5.3 and dotted lines indicate factor of two on life.

The general improvements across the predictions of the proposed model over the Goodman model are due to calibrating η with the experimental data via the parameter K . Stainless steel 316L in the rolled form exhibits stronger hardening than the L-PBF form, as shown by the larger η , while the close-to-unity η for Inconel 718 can be attributed the cyclic softening properties. However, η for AlSi10Mg does not agree with its plastic properties, i.e. $\eta < 1$ even though it is cyclic hardening. This is because of the use of independent studies for deriving the material parameters and validating the models, where the different experimental setups could affect the accuracy of both

the Goodman and the proposed models. In particular, different defect characteristics across the samples could be a key factor that created the variations in the fatigue properties. This applies to the case of L-PBF stainless steel 316L, where possible defect-driven failure, as pointed out by Spierings et al. [92], could have given rise to the worse fatigue properties as compared to the microstructure-driven failure pertaining to samples used in this study.

5.5 Summary and conclusions

In this chapter, the influence of build orientation and post-processing treatments on the high cycle fatigue properties and fracture behaviours of L-PBF stainless steel 316L was examined. The following conclusions can be made from the results obtained:

- 1) Build orientation and post-processing affect the high cycle fatigue properties primarily by the mechanism of fatigue-ratcheting interaction. The activation and accumulation of ratcheting strains are responsible for the fatigue anisotropy and degraded fatigue resistance after heat treatment. Mean stress insensitivity at higher load ratios occurs as a result of cyclic hardening and the reduced stress amplitudes.
- 2) Defects and microstructural heterogeneities affect fatigue properties in the longer life region where the bulk deformation mode is elastic. After high temperature annealing, crack initiation occurs at thermally-induced defects as the microstructural defects are removed by recrystallisation and grain regrowth. HIP causes previously benign defects to be exposed, resulting in larger scatter of the S-N data.
- 3) Traditional stress-based models such as the Goodman produced poor predictions when cyclic plasticity is significant. A modified Goodman equation was proposed by introducing an exponential function $\eta = f(\sigma_{\max}/\sigma_b)$ to account for the material- and the stress-dependent effects of fatigue-ratcheting interaction.
- 4) While the proposed model was developed based on fracture behaviours observed for changes in build orientation and heat treatment conditions, it is expected to describe the S-N properties of other L-PBF variables that incur the cyclic plastic

deformation response. However, for factors contributing to the brittle defect-driven fatigue failure, the effects need to be considered separately.

CHAPTER 6 PREDICTIVE MODELS FOR FATIGUE PROPERTY

Methods for assessing the high cycle fatigue properties of L-PBF stainless steel 316L are examined in this chapter. Experimental S-N data were obtained for samples subjected to controlled changes in laser power and scan speed. Using these data as the input, models for predicting the S-N properties via the ‘process-property’ and ‘structure-property’ routes respectively were proposed. For the ‘process-property’ relationship, regressions models expressing the fatigue life as a function of the process parameters were derived by employing the statistical response surface method. The stress-based approach was adopted for mapping the ‘structure-property’ relationship, where the influence of defect on the S-N properties were characterised via modified forms of the Basquin equation. Limitations of the models are discussed, leading to the implementation of a machine learning technique as the unifying approach to tackling the complexity of the L-PBF process and the associated failure behaviours. Test results from the previous chapters were used where appropriate for constructing and validating the models. Sections of the work reported in this chapter had been published [243, 244].

6.1 Process-property approach

6.1.1 Fatigue data prepared via central composite design

Horizontal samples were prepared according to the central composite design (CCD) in Figure 3-2 and fatigue tested at $R = 0.1$. The CCD was selected as it is an effective design architecture for modelling second-order systems [245], which had been reported to represent well the relationships between processing parameters and defect [246, 247] or mechanical properties [116] of L-PBF materials.

Experimental fatigue life data were obtained for two different cyclic loading conditions, i.e. $\sigma_a = 296$ MPa and 197 MPa, to allow examination of the *high stress, low cycle* ($N_f < 10^5$ cycles) and *low stress, high cycle* ($N_f > 10^5$ cycles) conditions

respectively (though it is to be noted that the fatigue lives of some of the samples did not fit exactly according to the 10^5 cycles criterion). A minimum of two samples were tested at each loading condition for all CCD design points. Statistical analysis and model derivation were performed using the commercial Design-Expert® software, where the significant model factors were identified by ANOVA. The upper and lower limits of the process variables were coded as 1.414 and -1.414 respectively.

6.1.2 Regression models

Results from the statistical analysis allowed the variables to be represented by second-order polynomial equations:

$$N_{f(\text{low stress})} = 5873000 - 23626P - 11127v + 56.113Pv - 20.726P^2 + 4.6835v^2 - 0.023610Pv^2 \quad (6.1)$$

$$N_{f(\text{high stress})} = 760850 - 3349.9P - 1445.3v + 8.6571Pv - 4.4993P^2 + 0.56325v^2 - 0.0035038Pv^2 \quad (6.2)$$

Table 6.1 shows the p-values of the regression models. Terms with p-values less than 0.05 are significant factors and were used for constructing the regression equations. Insignificant terms, such as v and v^2 , were also included in order to preserve the model hierarchy, i.e. to allow conversion between the coded and natural variables.

Table 6.1 List of p-values for the model terms and lack of fit for the regression models.

	Model	P	v	Pv	P^2	v^2	Pv^2	Lack-of-fit
$N_{f(\text{low stress})}$	<0.001	<0.001	0.863	0.004	<0.001	0.856	0.019	0.070
$N_{f(\text{high stress})}$	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	0.898

The low stress model exhibits more significant lack-of-fit, in agreement with the fitting results in Table 6.2. However, adding additional term, i.e. P^2v , to the low stress model would only marginally improve the adjusted R^2 , a parameter that takes into

consideration the effect of additional terms on model fitting, from 0.733 to 0.765. Moreover, the predicted R^2 , which evaluates the capability of the model for predicting new observations, would also improve only slightly, from 0.586 to 0.595, after adding the new term. Thus, to prevent overfitting, additional terms were not included in the low stress model. With the reasonable R^2 values, the small difference between the predicted and adjusted R^2 , as well as the large adequate precisions, i.e. more than 4, the models can be considered to have good signal-to-noise ratio and predictive capability for navigating the variable space.

Table 6.2 Fit statistics of the regression models.

	R^2	Adjusted R^2	Predicted R^2	Adequate precision
$N_{f(\text{low stress})}$	0.809	0.733	0.586	8.832
$N_{f(\text{high stress})}$	0.985	0.975	0.954	30.071

Figure 6-1 shows the normal probability plots of the residuals. The data points fit well to the straight lines such that the normality assumption is valid.

Figure 6-1 Normal probability plots of the fatigue life residuals for the (a) low stress and (b) high stress regression models.

6.1.3 Model validation

Figure 6-2 shows the fatigue life contour plots obtained from the regression equations. It can be seen that the optimum processing region occurs in the diagonal direction,

while the combinations of high P and low v as well as low P and high v result in lower fatigue lives. This is in agreement with understanding of the failure mechanisms, where over-melting and under-melting had been shown to trigger the Type III and Type II crack initiation mechanisms respectively, and were responsible for the reduced fatigue lives. Detailed fractography analysis of the samples can be found in Appendix A.

The more significant prediction error for the low stress condition, as indicated by the wider 95% confidence region of the centre point, can be attributed to the more complex fracture behaviour as the fatigue limit is approached [36]. This, based on the analysis in Chapter 4, pertains to the intricate effects of cooling rate on the generation of the intergranular/transgranular mode of crack initiation and is associated with more significant fatigue scatter. As crack initiation constitutes a greater portion of the total life as stress reduces, the influence is more pronounced for the low stress, high cycle model. Worth noting also is that both models predict longer fatigue lives at small P and v values, such that the predicted optimum processing regions are beyond the current design space.

Figure 6-2 Contour plots of (a) low stress fatigue life and (b) high stress fatigue life in terms of laser power and scan speed. Dots indicate the sample design points S_0 - S_8 ; dotted lines are the lower 95% confidence region of S_0 .

The regression models were validated for samples processed at conditions marked by the unfilled dots in Figure 3-2. As shown in Table 6.3, the models effectively predicted the fatigue lives for $1.3P_0$, with errors that are within $\pm 10\%$. However, fatigue lives for $0.7P_0$ were significantly underestimated, with an error exceeding 50% for the low stress condition. The underestimation could have stemmed from the lack of design points for representing the increase in fatigue resistance close to the processing region of $0.7P_0$, where the substantial error for $N_{\text{low stress}}$ can be attributed to the greater impact of crack initiation on fatigue life at lower stresses. Furthermore, the poor prediction accuracy for $0.7P_0$ implies that the prediction of optimum fatigue properties at small P and v is likely to be misleading. This result indicates that a larger number of design points are needed to provide an effective representation of the input-output space, especially in regions close to the optimum fatigue properties, where the fracture mechanism has a more intricate relationship with the underlying microstructural features.

Table 6.3 Actual and predicted fatigue lives of the test samples.

Sample	$N_{\text{f(low stress)}} (\times 10^5 \text{ cycles})$			$N_{\text{f(high stress)}} (\times 10^4 \text{ cycles})$		
	Actual	Predicted	% error	Actual	Predicted	% error
$0.7P_0$	5.90 ± 0.43	2.19	-62.9	4.29 ± 0.60	3.39	-21.0
$1.3P_0$	4.10 ± 0.11	3.80	-7.32	4.83 ± 0.84	5.28	9.32

The models were further cross-validated using data from the literature, as listed in Table 6.4. For comparing the fatigue test data, the Walker equation was applied to the test data by Mower and Long as they were obtained at $R = -1$. Also, a modifying factor of 0.85 on fatigue strength was applied to the same data to adjust for the bending load [97]. Besides laser power and scan speed, layer thickness is among the variables that was not kept constant across the studies, i.e. higher layer thicknesses of 30 μm [24, 92] and 40 μm [94] were used. However, since the P - v combination for fabricating S_0 produces sufficient laser penetration at $T = 40 \mu\text{m}$, and the fatigue properties at 20 μm and 40 μm are similar, some tolerance for variation in layer

thickness setting could be allowed. Specifically, though the samples used by Mower and Long were fabricated at 40 μm , the P - v values are close to S_0 such that no significant shift in the processing region is expected. For the other two works, the deviation in layer thickness is even smaller and could be assumed to produce negligible effect.

Table 6.4 Input information from literature for validating the regression models.

Sources	Laser power, P (W)	Scan speed, v (mm/s)
Spierings et al. [92]	103	425
Shrestha et al. [24]	400	1000
Mower and Long [94]	195	750

Fatigue test results from Shrestha et al. were only used for qualitative analysis as the tests were done in strain-controlled mode. The predicted fatigue life exceeds the test results of Spierings et al. by more than an order of magnitude, as shown in Figure 6-3. As mentioned, the overestimations at low P and v could have been caused by the lack of data points for representing the fatigue properties in that region. Also, as the build settings used in this reference are significantly beyond the CCD window, the poor predictions suggest that the validity of the regression equations is constrained by the experimental test space. Negative fatigue lives were predicted for the processing parameters used by Shrestha et al. It is interesting that while the processing conditions are closer to the over-melting region, lack of fusion defects were reported by the authors. This alludes to the non-transferability of processing information across L-PBF machines. In comparison, as machines from the same manufacturer and similar processing parameters were used by Mower and Long and this study, the predicted S-N curve produced more reasonable fit to the reported test data.

Figure 6-3 Comparison of fatigue test data from literature with predicted results of the regression equations. Plots were obtained for $R = 0.1$.

6.2 Structure-property approach

6.2.1 Model intuition

The structure-property relationships of the CCD samples were examined by analysing the mechanisms of fatigue fracture. Results from fractography analysis allowed the processing regions corresponding to the different crack initiation modes (Type I, II and III) to be identified: as shown in Figure 6-4, the optimum processing region is associated with Type I failure, while the low- P -high- v and high- P -low- v regions correspond to Type II and Type III failures respectively. As the fundamental fracture behaviours are explained in Chapter 4.3, they are not repeated here. Refer to Appendix A for the detailed fractography examination of the CCD samples.

Figure 6-4 Processing regions in terms of the fatigue crack initiation modes. LOF stands for lack of fusion.

Based on this understanding, the relations between the parameters of the Basquin equation (σ'_f and b) and the failure regions were evaluated, as shown in Figure 6-5. Since σ'_f is related to the static tensile strength, the values of σ_b were first analysed. From Figure 6-5a, it can be seen that within the optimum processing region, tensile strength shows a steady reduction with input energy, as the slower cooling rate is associated with increased primary dendrite spacing and lower strength [248]. Beyond this region, both under- and over- melting produced significantly deteriorated strength. The relations between tensile strength and the *equivalent* fatigue strength parameters (i.e. at $R = 0.1$) are shown in Figure 6-5b. While σ'_f varies directly with σ_b , the resulting fatigue limit σ_e (fatigue strength at 10^6 cycles) converges to a constant value with certain scattering for samples showing Type I failure. This, as shown in Figure 6-5b, is due to the opposing effect of b on the fatigue resistance. As b characterises the slope of the S-N plot, it strongly affects long life performance: as b increases, the S-N slope becomes gentler, indicating that the material is more

damage-tolerant. For the Type I samples where b decreases with increasing tensile strength, the steeper S-N slopes retarded the increase in fatigue strength at long lives, such that the fatigue limits stabilised despite the increase in σ_f' .

Figure 6-5 (a) Tensile strength of the samples; (b) equivalent fatigue strength parameters/fatigue limits against tensile strength at $R = 0.1$.

The opposing trend between b and σ_f' for ultrafine-grained alloys had been studied by Zhang et al. and was found to be related to grain size [249]. For the Type I samples, even though σ_f' increases with tensile strength because of grain refinement, b

decreases as a result of the higher grain boundary density and microstructural instability. This explains the intergranular cracking in L-PBF stainless steel 316L in a more simplistic manner without accounting for the intercellular particle effect and the transition to transgranular crack initiation. The scatter in fatigue limit values is therefore a reflection of the subtle changes in microstructure caused by the variation in L-PBF processing conditions.

In real-life production, since the optimum processing region produces high strength parts devoid of critical defects, any processing parameter combination in this region could be identified as the optimum condition. Though the corresponding variations in microstructure and tensile strength are not easily detectable, the risk associated with finite life fatigue loading is not trivial and should be taken into consideration for safe life structural designs. This could be achieved applying an appropriate reduction factor on fatigue life or strength.

On the basis of the above relations between the failure mechanisms and Basquin parameters, the S-N properties can be modelled by considering:

- 1) Type I failure – by deriving the reference S-N curve for representing the optimum behaviour of L-PBF stainless steel 316L. The scatter due to intrinsic microstructural changes is accounted for by the probability of failure Pr .
- 2) Type II failure – by correlating defect with the fatigue strength parameters to account for the effect of both interacting and non-interacting defects.

Type III failure is not discussed as the samples tested in this study does not provide sufficient coverage of over-melting phenomena for modelling of the failure mode.

6.2.2 Modelling of Type I failure

Figure 6-6a shows the Pr - N curves fitted to the experimental data of the Type I samples for both the high and low stress conditions according to the form of the lognormal distribution. This allows the mean-data curve to be modelled by the Basquin equation based on the results at 50% failure probability.

Figure 6-6 (a) Family of Pr - N curves plotted in terms of the lognormal distribution at applied stress amplitudes of 296 MPa and 197 MPa for Type I samples; (b) Type I sample test data and S-N curves corresponding to 5%, 50% and 95% failure probabilities at 95% confidence level. The plots were obtained for $R = 0.1$.

Consequently, the equivalent values of σ'_f and b for the mean-data curve were derived, as shown in Figure 6-6b, to be 2560 MPa and -0.189 respectively. To cover the 5% failure probability curve, a reduction factor of about 1.9 on fatigue life needs to be applied to the mean-data curve. Noting that the current ASME code fatigue design

curves adopts a reduction factor of 20 on life to estimate fatigue life for 95% of the population of austenitic stainless steel [250] (Figure 2-7), an additional reduction factor on the order of 1.9 could be applied to account for the microstructural variations due to processing for L-PBF parts.

6.2.3 Modelling of Type II failure

Fatigue life is plotted against the defect area fraction p and equivalent spherical diameter $2a$ in Figure 6-7 for samples failed via Type II. Defect conditions for S_6 were used as conservative estimates of the critical defect condition, which serves to indicate the transition from Type I to Type II failure. They adopt values of $p = 0.05 \pm 0.03\%$, and equivalent circular defect diameter $2a = 18.9 \pm 8.4 \mu\text{m}$ based on results from the extreme value analysis. Comparing the relative effects of the area fraction and size of the defects, it can be seen that the latter parameter corresponds to a more rapid reduction in fatigue life from S_1 to S_5 . Approximating the N_f - p relations by power-law equations, as indicated in Figure 6-7, the exponent of the equation was estimated at -0.44 for the low stress condition, whereas under high stress, a smaller exponent of -1.39 was obtained.

To interpret this result, the unit-cell model illustrated in Figure 4-10 was used for representing the defects. By definition, p is related to a by the equation:

$$p = \frac{\pi a^2}{d^2} \quad (6.3)$$

The interpore spacing λ is related to p and a as:

$$\lambda = d - 2a = \left(\sqrt{\frac{\pi}{p}} - 2 \right) a \approx \sqrt{\frac{\pi}{p}} a \quad (6.4)$$

Figure 6-7 Fatigue life against defect area fraction and average extreme equivalent spherical defect diameter for the (a) low stress and (b) high stress conditions.

In a study by Holmes et al. [251], the form of λ/a was referred to as the damage ratio, as it represents the space where stress can accumulate relative to the size of the stress concentrator. Thus, the area fraction of defect does not only quantify the effect of an isolated flaw, but also its interaction with neighbouring flaws. Optical micrographs

in Figure 6-8 show that while the maximum defects in S_1 and S_5 are comparable in size, those in S_5 are clearly interacting. As a result, for S_5 , defect size determined from optical micrograph is smaller than the effective dimension of the interacting defects, such that fatigue life reduces more rapidly with defect size for S_5 . In addition, fatigue life decreases more rapidly at the higher stress as the material was significantly overloaded and catastrophic failure occurred after a small number of cycles.

Figure 6-8 Optical micrographs showing (a) isolated defects in S_1 and (b) interacting defects in S_5 .

Approximating the exponent of the power-law equation in Figure 6-7a as -0.5, and replacing p by λ/a according to Equation (6.4), it can be seen that fatigue life is linearly related to the damage ratio λ/a . This provides a physical explanation for the observed inverse-square-root relation between defect fraction and fatigue life. However, as the relation estimates relatively high fatigue life as p approaches the fully porous condition (i.e. $p \rightarrow 1$), it is expected that such relation is only valid within a certain range of defect fraction p_c . Beyond this, fatigue life will decrease rapidly

due to significant reduction in load-bearing area and stronger interacting effect among the defects.

Based on this, for $p_{\text{ref}} < p < p_c$, the relation between defect and high cycle fatigue life can be approximated by a power-law equation:

$$2N_f = mp^n \quad (6.5)$$

where m and n are stress-dependent parameters; under low stress and high cycle fatigue, $n \approx -0.5$. Considering parts with defect fractions p_1 and p_2 , the Basquin equation leads to:

$$\sigma_a = \sigma'_{f1}(2N_{f1})^{b1} = \sigma'_{f2}(2N_{f2})^{b2} \quad (6.6)$$

As shown in Figure 6-5b, b increases sharply from the Type I to Type II failure modes and remains nearly constant (approx. -0.144 to -0.153) with further reduction in strength. Thus, b was assumed to adopt a constant value for failures driven by lack of fusion defects. Here, an equivalent value of -0.15 was assigned.

Combining Equations (6.5)-(6.6), the fatigue strength coefficient can be expressed as a function of the defect fraction:

$$\sigma'_{f2} = \left(\frac{p_1}{p_2}\right)^{nb} \sigma'_{f1} \approx \left(\frac{p_1}{p_2}\right)^{0.075} \sigma'_{f1} \quad (6.7)$$

This expression predicts that for a part with increasing defect fraction, there will be a parallel downward shift of the S-N curve that is proportional to the log of the ratio between the defect fractions. Approximating p_1 as 0.05% and σ'_{f1} as 2560 MPa (i.e. the mean stress curve), the Basquin equation can be modified as a function of the defect fraction, i.e.

$$\sigma_a = 1447.6p^{-0.075}(2N_f)^{-0.15} \quad (6.8)$$

As the exponent 0.075 was calculated for the low stress case (where $n = -0.5$), the proposed equations are expected to be more suited to low stress, long life fatigue

prediction. In addition, it is to be emphasised that the power-law relation was approximated from a limited number of empirical results. The applicability of this relation for fatigue prediction is examined in the next section.

6.2.4 Model validation

The model was validated against the test samples listed in Table 6.5. For $1.3P_0$ and $2T_0$, applying the defect parameters of S_6 for approximating the critical defect condition correctly predicted the failure modes as Type I. For $0.7P_0$, uncertainties exist as the defects had been demonstrated to produce both Type I and Type II failures. Figure 6-9a shows that the S-N data of the samples are well covered by the 5% failure probability line, such that applying the 1.9 reduction factor on life is conservative. Thus, the Pr -S-N curves are appropriate for modelling the fatigue properties of $0.7P_0$, for which the defect-oriented approach produced significant error.

Table 6.5 Defect fraction, equivalent spherical defect size, crack initiation mechanism and the actual and predicted equivalent σ'_f values of the test samples.

Test sample	Defect fraction, p ($\times 10^{-2}$)	Defect size, $2a$ (μm)	Failure mechanism	Equivalent σ'_f at $R = 0.1$		
				Actual	Predicted	% error
S_1	0.41 ± 0.26	42.1 ± 16.7	Type II	1286	1293	0.54
S_5	3.38 ± 1.42	47.3 ± 12.4	Type II	1210	1104	-8.76
$0.7P_0$	0.16 ± 0.05	21.1 ± 8.5	Type I/II	1763	1388	-21.27
$1.3P_0$	0.04 ± 0.04	8.3 ± 3.4	Type I	1210	-	-
$2T_0$	0.04 ± 0.05	9.6 ± 5.6	Type I	1822	-	-
$3T_0$	0.70 ± 0.81	61.4 ± 39.4	Type II	1323	1243	-6.05
$4T_0$	10.85 ± 4.23	370.9 ± 178.0	Type II	897	1011	12.71

The rest of the samples contained critical defects and therefore failed via the Type II mode. As listed in Table 6.5, the predicted σ'_f values are in good agreement with the actual values, with less than 10% error. This is with the exception of $4T_0$, where the

model overestimated σ_f' by 13%. Figure 6-9b compares the predicted and experimental fatigue lives. It can be seen that except for $4T_0$, the predictions are generally within a factor of two of the experimental results (dash lines). For $4T_0$, the fatigue lives were overestimated by about an order of magnitude, indicating that the defect fraction of $4T_0$ is larger than p_c , such that the proposed equation is not valid.

Figure 6-9 (a) S-N data of Type I samples are within the 5% failure probability band; (b) predicted versus test measured fatigue life data for Type II samples.

The models were further cross-validated with the literature data in Table 6.4. In Figure 6-10, lines of constant defect fraction based on Equation (6.7) were plotted and compared with the literature data. Agreeing with results from the regression model, test data by Mower and Long are comparable with those of the optimum samples in this work, i.e. most of the data points are close to and above the $p = 0.05\%$ line. Results by Spierings et al. are mostly covered by the $p = 0.1\%$ and $p = 1\%$ lines. This suggests Type II failure, testifying to the speculation of the authors that internal defect could be responsible for the similar fatigue properties between the samples with different surface finishes. The significant scatter of the test data could indicate inconsistency of the build process, which had led to nonhomogeneous defect distribution of the samples.

Figure 6-10 Comparison of fatigue test data from literature with predicted results of the structure-property model. The plots were obtained for $R = 0.1$.

In summary, predictions obtained from applying the ‘structure-property’ model to the data obtained in this study as well as literature data are satisfactory. In particular, considering the results obtained for Spierings et al., the structure-property approach appears to be associated with better transferability than the process-property approach. This is because the defect fraction is associated with more fundamental representation

of the fatigue properties via the mechanisms of fracture. Nonetheless, the validity of the power-law relation between fatigue life and defect fraction remains to be further investigated because of the small amount of data points used in its derivation. Also, noting that since fatigue strength is related to tensile strength, the fatigue strength parameters obtained in this research applies to parts with comparable tensile strength, and for parts belonging to different strength regimes, e.g. due to processing at higher layer thickness where grain coarsening could cause a general reduction of material strength [252], adjustment factors may need to be applied.

6.3 Data science approach

Models proposed in the previous sections have a number of limitations which restrict the applicability for fatigue assessment of L-PBF-processed materials. Firstly, the regression models developed based on the design of experiment technique are unable to characterise effectively the machine-machine variability and the influence of the wide L-PBF processing regions on the fatigue properties. Second-order polynomial equations were used for modelling the two-input system, however, the use of the statistical approach will become unrealistic as the complex processing/post-processing conditions amount to a large number of input variables. The modified Basquin equations characterise the S-N properties of parts associated with a limited defect range only, and are unable to predict the effects of cyclic plasticity and other possible failure modes of L-PBF materials. The presented models thereby lack the flexibility for mapping the high dimensional, nonlinear input-output space as a function of the complex L-PBF processing environment.

In comparison, data science methods are time- and cost-effective means for solving engineering problems as they are capable of recognizing patterns in complex data [253]. Advanced data science algorithms such as the artificial neural network had been used for mapping the process-property relationships of various manufacturing processes, e.g. classification of welding defects for a three-input system [254] and tensile strength prediction of rolled steel plates for a six-input system [255]. The strong versatility may be leveraged for addressing the intricacies of L-PBF. Therefore, the applicability of machine learning is examined in this section as a unifying

approach for assessing the fatigue and failure properties of the L-PBF stainless steel 316L under the influence of various processing, post-processing and loading conditions.

6.3.1 Fatigue data

In this thesis, a total of 167 experimental S-N data, for samples fabricated with varying L-PBF processing parameter settings, herein laser power, scan speed, layer thickness and build orientation, and subjected to different post-processing treatment (annealing or HIP) and stress conditions, had been reported. The wide processing regions and failure properties are associated with a high-dimensional and nonlinear input-output environment, which could be used as the dataset for constructing the machine learning model. Data for the vertical as-built samples were excluded, however, as they constitute only 5% of the dataset and are distributed over a wide range of load ratios (Figure 5-1a). Considering the scatter associated with fatigue data, the limited data points for each parameter setting can lead to a noisy dataset and impinge on the model performance. Similarly, data obtained at load ratios other than 0.1 were not considered as some of the parameter combinations consist of single-test data only. By excluding these data, a dataset consisting of 139 data points was established for building the machine learning model. The processing conditions used for fabricating the samples are shown in Figure 6-11. It can be seen that besides the CCD samples obtained from varying the laser power and scan speed settings, the dataset also contains samples processed at various layer thicknesses. In addition, the post-processed samples (produced at S_0) had also been included in the dataset.

In terms of the failure modes, Type I samples, which correspond to the microstructure-driven crack initiation mode, constitute about 55% of the dataset, while Type II samples showing defect-driven crack initiation constitute about 21%. (Note: For convenience of presentation, data points for Type III samples are shown using the same symbols as Type I samples because of the limited data and the comparable fatigue properties.) The post-processed samples, which exhibited ratcheting-dominated deformation, make up the remaining 24% of the dataset.

Figure 6-11 Laser powder bed fusion processing conditions for samples used for constructing the machine learning model.

6.3.2 Model selection

From the perspective of big-data analytics, one of the major difficulties for fatigue assessment of L-PBF parts lie in the lack of experimental data from the literature and public domain that could be used for training the models. In addition, data science methods often adopt attributes of the ‘black box’, where the complex computational algorithms cannot be easily comprehended. This is not desirable for modelling novel processes like the L-PBF, where understanding of the underlying material science is still lacking. The inability to relate the quantitative results with physical understanding of the failure mechanisms compromises the reliability of the fatigue assessment.

The adaptive neuro-fuzzy inference system (ANFIS) is a neural-network-based technique that could potentially address the problems. By integrating fuzzy reasoning into the network structure [256], the system can be interpreted in terms of linguistic fuzzy rules. For instance, key fatigue assessment indicators could be related to the fatigue stress-life relations via a set of ‘if-then’ rules of the form:

Rule 1: If tensile strength is high and ductility is high and stress is low, then fatigue life is long.

Rule 2: If tensile strength is low and ductility is low and stress is high, then fatigue life is short.

The use of the linguistic terms provides the desired transparency to the system and makes the results comprehensible for the human operator. This could simplify the design and validation process, thereby allowing better error convergence for sparse data [257]. Besides, fuzzy logic, in contrast to the traditional Boolean logic, is suited to fatigue modelling because of the ability to characterise intermediate conditions via the use of membership functions [258, 259]. This allows meaningful representations of the uncertainties in fatigue studies [260], e.g. fatigue scatter due to the probabilistic nature of factors such as defect and microstructure on crack initiation [261, 262]. In cases where the input and output variables are ill-defined, imprecise but meaningful representations of the system behaviour can be achieved by fuzzy logic. Successful applications of the ANFIS for fatigue modelling had been reported, such as in predicting the S-N properties of composite laminates [263, 264] and the fatigue threshold of a superalloy [257]. Based on the above considerations, the ANFIS method was chosen for predicting the fatigue life of L-PBF stainless steel 316L in this study.

6.3.3 Input variables

The fatigue properties of L-PBF stainless steel 316L can be correlated to the processing/post-processing strategies. Besides, results from Chapter 4.4 indicate strong relations between the static tensile and fatigue properties because of the influence of microstructure and defects on the fracture mechanisms. Figure 6-12 shows the tensile properties of the dataset. It can be seen that samples with the various failure modes demonstrate different trends in terms of the tensile strength and ductility properties. Type I/III samples are associated with the optimum tensile strength, while the Type II samples are characterised by concurrent reductions in strength and ductility with increasing defect fraction. For the post-processed parts,

the increase in post-processing intensity is characterised by lower strength but higher ductility, in compliance with the strength-ductility trade-off that is typical of structural metals [161].

Based on the above observations, two models, employing different variables as the inputs, were implemented, i.e.

- 1) ‘Process-based’ model, where the processing and post-processing parameters, i.e. P , v , T and HT served as the inputs; HT was assigned with levels 1, 2, 3 and 4 for representing the as-built, low-temperature annealing, high-temperature annealing and HIP conditions respectively.
- 2) ‘Property-based’ model, where the ultimate tensile strength σ_b and elongation to failure e served as the inputs.

In addition, as the fatigue tests were conducted at a constant load ratio, the maximum applied cyclic stress was used for characterising the loading condition.

Figure 6-12 Ultimate tensile strength against elongation to failure of samples exhibiting the different fatigue fracture behaviours.

6.3.4 Architecture of the adaptive neuro-fuzzy inference system

A typical feedforward neural network consists of three layers: an input layer, a set of hidden layers, and an output layer [265]. Each of the layers contains a number of nodes, known as neurons; signals are transmitted from the first layer to the last layer via the series of interconnected neurons. For neuro-fuzzy-based networks, the fuzzy inference system provides the rule base that forms the structure of the neural network, while the neural network algorithm manages the learning aspect by adapting model parameters according to the behaviour of the data [256].

70% of the 139 data points was used for training the model; 15% was used for validation, where, by comparing the training and validation errors, model parameters were selected to prevent overfitting; the remaining 15% was used for testing the model performance. Considering the small dataset relative to the number of variables for the models, training data were not chosen at random, but were selected to provide sufficient coverage of the design space. Figure 6-13 and Figure 6-14 show the architectures of the ‘process-based’ and ‘property-based’ ANFIS models respectively. As the ‘property-based’ model is associated with a simpler structure, it is used for explaining the ANFIS framework here.

As mentioned, the neural network structure of an ANFIS is formulated based on a set of ‘if-then’ rules, such as ‘if x is A , then y is B ’ [266]. The *if* part of the rule is known as the antecedent or premise, and the *then* part is the consequent; the variables x and y are expressed in terms of A and B , which are linguistic labels that characterise concepts such as *very good*, *good*, *bad* and *very bad*. The ‘property-based’ model is made up of four rules, with the input parameters σ_b , e and $\sigma_{\max}$ being partitioned into classes denoted by A_j , B_k , and C_l respectively. Neurons in Layer 1 are connected by arrows to neurons in Layer 2, forming the rules. For example, the first and second rules for the ‘property-based’ model in Figure 6-14 can be expressed respectively as:

Rule 1: If σ_b is A_1 , δ is B_1 and $\sigma_{\max}$ is C_1 , then

$$f_1 = q_1 \sigma_b + r_1 e + s_1 \sigma_{\max} + t_1 \quad (6.9)$$

Rule 2: If σ_b is A_2 , δ is B_2 and $\sigma_{\max}$ is C_2 , then

$$f_2 = q_2 \sigma_b + r_2 e + s_2 \sigma_{max} + t_2 \quad (6.10)$$

where f_i is the linear consequent function of the i th fuzzy rule and q_i , r_i , s_i and t_i are the consequent parameters of the rule.

Figure 6-13 Structure of the adaptive neuro-fuzzy inference system for the ‘process-based’ model.

Figure 6-14 Structure of the adaptive neuro-fuzzy inference system for the ‘property-based’ model.

Steps of fuzzy reasoning include [256]:

- 1) Deriving membership functions for the linguistic variables via fuzzification;
- 2) Combining membership functions on the antecedent part of the rule using T-norm operators to produce the firing strength of each rule;
- 3) Obtaining the consequents of the rules based on the firing strength
- 4) Producing a crisp output by aggregating the consequents via the process of defuzzification.

Nodal operations associated with each of the steps are described below:

Layer 1

Nodes in the first layer specify the degrees to which the given input parameters belong to each of the linguistic labels via membership functions μ :

$$O_j^1 = \mu_{Aj}(\sigma_b), \quad j = 1, 2, 3, 4 \quad (6.11)$$

$$O_k^1 = \mu_{Bk}(e), \quad k = 1, 2, 3, 4 \quad (6.12)$$

$$O_l^1 = \mu_{Cl}(\sigma_{max}), \quad l = 1, 2, 3, 4 \quad (6.13)$$

Membership functions can adopt the forms of the triangular, trapezoidal, Gaussian or bell-shaped functions [267]. The latter two are often used for representing fuzzy sets because of the smooth distribution curves [268]. The Gaussian distribution was used in this study as it contains one parameter less than the bell-shaped distribution. Good modelling accuracy had been reported for ANFIS models that utilised this membership function for fatigue life prediction [263, 264]. For example, the membership function μ_{Aj} for the variable σ_b is expressed as:

$$\mu_{Aj}(\sigma_b) = \exp\left[\frac{-(\sigma_b - \alpha_j)^2}{2\beta_j^2}\right], \quad j = 1, 2, 3, 4 \quad (6.14)$$

where α_j and β_j are parameters of the Gaussian distribution.

Layer 2

By multiplying the corresponding membership values, the firing strength ω_i , or the ‘degree of fulfilment’, of a particular rule is obtained in the second layer:

$$O_i^2 = \omega_i = \mu_{Aj}(\sigma_b) \times \mu_{Bk}(e) \times \mu_{Cl}(\sigma_{max}), \quad i = 1, 2, 3, 4 \quad (6.15)$$

Layer 3

Output of a node in the third layer is equal to the product of the normalized firing strength, which is the ratio of the i th rule’s firing strength to the summation of the firing strengths of all the rules, and the consequent function f of a rule, i.e.

$$O_i^3 = \bar{\omega}_i f_i, \quad i = 1, 2, 3, 4 \quad (6.16)$$

Layer 4

This layer constitutes the summation of all the incoming signals and is equal to the predicted fatigue life:

$$O_1^4 = \log(N_f) = \sum_i \bar{\omega}_i f_i \quad (6.17)$$

The logarithmic values of N_f were used for modelling to avoid extreme differences in fatigue lives, which are within the range of $10^3 - 10^6$ cycles for the dataset. The square nodes, i.e. Layer 1 and Layer 3, are adaptive nodes where the parameters are optimized by the adaptive neural network. For supervised learning, models learn by direct comparison of the predicted outputs with the actual values in order to minimise the error function, in this case, the root mean squared (RMS) error. Gradient descent-based method can be used for tuning the parameters of the ANFIS [269], but because of its tendency to be trapped in local minima, the hybrid learning rule, which combines the gradient method and least square estimation, is often preferred [270] and was used here. All computations and model developments were performed using the Neuro-Fuzzy Designer app in Matlab.

6.3.5 Extraction of fuzzy rules by data clustering

For large multivariate datasets, clustering is often performed to identify natural groupings in the data so as to allow a concise representation of the system behaviour. For ANFIS, clustering is used as a rule extraction algorithm where the cluster centres provide the basic premises of the rule base [271, 272]. Specifically, it partitions the input variables into classes, as demonstrated by the neurons in Layer 1 of the ANFIS model; neurons belonging to the same cluster centre are connected in Layer 2, leading to the respective rules. This technique serves to initialise the ANFIS structure, where the model parameters are subsequently tuned by the neural network during training to fit the data. Values of α for the Gaussian membership functions characterise the cluster centres after parameter tuning.

Subtractive clustering was performed on the training datasets using the Clustering tool in Matlab. The algorithm carried out the following steps for extracting the cluster centres [273, 274]:

- 1) Point with the highest potential, based on calculation of the Euclidian distances with respect to all other data points, was chosen as the first cluster centre;
- 2) Data points in the neighbouring regions of the first cluster centre, based on a predefined range of influence (ROI), were deleted and the remaining point with the highest potential was selected as the next cluster centre;
- 3) These steps were repeated until all data points are in the range of a cluster centre.

The ROI, a scalar quantity that adopts value between [0, 1], is a key parameter in subtractive clustering as it affects the range of the data points around a cluster centre that are removed: a larger ROI yields bigger and less cluster centres [274], and vice versa. As the ROI is directly related to the number of fuzzy rules, suitable ROI value can be determined by evaluating the performance of the corresponding ANFIS model. Prediction error generally decreases with increase in model complexity as the larger number of model parameter allows more explicit representation of the system behaviour [275]. However, overfitting occurs when the increase in model complexity does not only lead to better fitting of the trends in the data, but also the noises. This impinges on the model performance when applying the model to a fresh set of data, in this case, the validation set. Figure 6-15 shows the training and validation errors as a function of the number of fuzzy rules, obtained by varying the ROI. For both the ‘process-based’ and ‘property-based’ models, the training errors decrease with the number of fuzzy rules, as expected. The validation errors, however, increase following the initial reductions. This is suggestive of overfitting as the model is unable to generalise to the unseen data despite the increase in model complexity. Therefore, to prevent overfitting, the numbers of fuzzy rules were selected for the conditions with the smallest validation errors before they start diverging. This led to nine rules for the ‘process-based’ model (ROI = 0.65) and four rules for the ‘property-based’ model (ROI = 0.5), as highlighted in Figure 6-15.

Figure 6-15 Training and validation errors as a function of the number of fuzzy rules for the (a) ‘process-based’ and (b) ‘property-based’ ANFIS models. Selected conditions for model construction are highlighted.

6.3.6 Interpretation of the fuzzy rules

For the ‘process-based’ model, the input variables P , v , T , HT and $\sigma_{\max}$ were partitioned into a set of 5-1-3-3-5 Gaussian-type membership functions, which are schematically shown in Figure 6-16. By carefully examining the membership functions constituting the antecedents of the fuzzy rules, five out of the nine rules were found to characterise the effects of P and v at $T \approx 20 \mu\text{m}$ and $HT \approx 1$, two rules for the effects of T , with P and v at the standard processing conditions and $HT \approx 1$, and two rules for the effects of HT , with P , v , and T at the standard processing conditions. This is in agreement with the distribution of the input data, indicating that the rules were derived according to the different sample classes.

Figure 6-16 Membership functions for the 'process-based' model: (a) laser power, (b) scan speed, (c) layer thickness, (d) post-processing intensity and (e) maximum cyclic stress.

Figure 6-17 shows the fatigue life contour plot as a function of P and v for the as-built samples fabricated at $T = 20\ \mu\text{m}$ and fatigue tested at $\sigma_a = 296\ \text{MPa}$ (replotted based on Figure 6-2b). Values of α of the membership functions for the five rules characterising the effects of P and v are indicated by the black dots. They are conveniently referred to as the ‘cluster centres’, though it is to be noted that their values differ slightly from the initial cluster centres obtained from subtractive clustering due to parameter tuning by hybrid learning. It can be seen that the locations of the ‘cluster centres’ fall into some of the major fatigue life regions. In fact, they correspond to samples exemplifying the different fracture modes, i.e. S_5 , $0.7P_0$, S_0 , $1.3P_0$ and S_7 , as defined in Figure 3-2.

Figure 6-17 Locations of the ‘cluster centres’ for the ‘process-based’ model represented on the fatigue life contour plot as a function of laser power and scan speed for as-built samples fabricated at layer thickness of $20\ \mu\text{m}$ and fatigue tested at $\sigma_a = 296\ \text{MPa}$.

As mentioned, one of the advantages of the ANFIS over the conventional neural network is ability to interpret the system via the linguistic fuzzy rules. Based on the above observations, linguistic labels such as *Very Low*, *Low*, *Optimum*, *High* and *Extreme* can be assigned to the membership functions for P , as shown in Figure 6-16a. Specifically, the *Optimum* level ($P \approx 195\ \text{W}$) refers to processing conditions that produce the optimum fatigue properties; the *Low* ($P \approx 137\ \text{W}$) and *Very Low* ($P \approx 98$

W) levels pertain to crack initiation from single and multiple interacting defects respectively, while the *High* ($P \approx 254$ W) and *Extreme* ($P \approx 293$ W) levels are associated with the transition to transgranular-dominated crack initiation and the generation of critical over-heating-induced defects respectively. Note that the use of membership functions, in contrast to the Boolean logic, for describing the input variables is appropriate, as the change from one fracture mode to another is not triggered by abrupt changes in the input settings, but by a gradual transition.

Similar analysis applies to layer thickness (Figure 6-16c), where primary fatigue cracks originate from a single defect at $T \approx 60$ μm (*Low* level), and from multiple interacting defects at $T \approx 80$ μm (*Very Low* level). For the post-processing variable *HT*, membership functions were identified for the as-built, high-temperature annealing and hot isostatic pressing conditions (Figure 6-16d), where the increase in post-processing intensity correlates directly with the extent of fatigue strength degradation due to cyclic plastic deformation.

A simpler ANFIS structure was obtained for the ‘property-based’ model (Figure 6-18). Figure 6-19 shows the ‘cluster centres’ in relation to the tensile properties of the dataset. The σ_b - e pairs forming the premises of Rules 1 and 2 correspond to the tensile properties of the Type I samples, while those for Rules 3 and 4 correspond to the post-processed and Type II samples respectively. Note that rules were generated for two different cyclic stresses for the Type I samples but not for the other sample types. This could be attributed to the larger percentage of input data belonging to this group. As the clustering algorithm defines cluster centre by calculating the potential of a data point in terms of its distance to all other data points, high density region with many neighbouring data, such as the Type I samples, are more likely to be chosen as the cluster centre [273].

From the above results, it can be noted that regardless of the input variables, subtractive clustering effectively captured the underlying fracture mechanisms of the dataset. The assignment of fuzzy rules based on the cluster centres ensured mapping of the input-output relationships in accordance to the fracture behaviours of the samples.

Figure 6-18 Membership functions for the ‘property-based’ model: (a) tensile strength, (b) elongation to failure and (c) maximum cyclic stress.

Figure 6-19 Locations of the ‘cluster centres’ for the ‘property-based’ model in relation to the tensile strength and elongation to failure of the samples.

6.3.7 Model validation

Table 6.6 lists the RMS errors obtained from applying the ANFIS models. The overall errors range from about 11% to 16% across the datasets. In a similar study by Vassilopoulos et al. [263], where the ANFIS was used for predicting the high cycle fatigue life of composite laminates, the training and test errors were reported to be 25% and 47% respectively (for a model constructed using the same percentage allocation of training data). The better prediction accuracy achieved here could be attributed to the simpler system behaviour of the dataset (consider that a 15-17-rule model was obtained by Vassilopoulos et al. for 257 data points). An increase in the complexity of the data is likely to impinge on the modelling performance. This is discussed further in the context of applying the model to literature data in the later part of this section.

Table 6.6 Root-mean-squared errors for the ‘process-based’ and ‘property-based’ ANFIS models.

Model/data type		RMS errors (%)			
		Type I/III	Type II	Post-processed	Overall
‘Process-based’	Training data	8.84	14.82	10.80	10.86
	Test data	13.76	20.87	15.24	16.00
‘Property-based’	Training data	11.32	16.44	13.61	13.09
	Test data	14.55	18.58	11.93	14.66

The RMS errors for the individual sample types expand a bigger range, from about 9% to 21%. Worth noting is that the Type II samples are associated with the greatest errors which consistently surpass the overall errors, while errors for the Type I/III samples tend to be the least and are always less than the overall errors. The good prediction accuracy for the Type I/III conditions stems from the large amount of data belonging to this sample group, where the more representative dataset ensured effective training, as demonstrated by the larger number of rules corresponding to the input space of these samples. Between the Type II and the post-processed samples, which have similar data distributions, the worse modelling performance of the former

could have been caused by a number of reasons. For the ‘process-based’ approach, sample fabrication incurred the variables P , v and T , while post-processing is a function of HT only. The synergistic effects of the input variables for the Type II samples could have resulted in greater variance, thereby the larger errors. For the ‘property-based’ model, note that the tensile properties of some of the Type II samples, corresponding to fatigue crack initiation from non-interacting lack of fusion defect, are very close to those of the Type I/III samples (Figure 6-12). Despite the different fatigue properties, the similar input values will lead to similar life predictions. The error distributions for Type I/III and Type II samples with overlapping tensile properties are compared in Figure 6-20. It can be seen that the histogram for the Type II samples shows a heavy right tail, which is indicative of overestimations, while the opposite trend applies for the Type I/III samples. This is reasonable because as the model sought to minimise the sum of squared errors, it arrived at solutions that approximate the average fatigue lives of the two types of samples. The resulting biased predictions, however, are not desirable, especially for the Type II samples as they can lead to unconservative estimates of the fatigue properties.

Figure 6-20 Histograms showing the error distributions for Type I/III and II samples with overlapping tensile properties for the ‘property-based’ ANFIS model.

Figure 6-21 compares the predicted and experimental fatigue lives. The predicted results are generally within a factor of two of the experimental data, even for the Type II samples which are associated with the greatest errors. Figure 6-22 presents the results for selected samples on S-N plots. The predicted S-N curves fit well the experimental data across the different failure modes, except for the Type I and II samples with overlapping tensile properties. It is worth noting that the prediction at each loading is generally within the experimental scatter band, indicating that the models are able to account for the variations in fatigue data due to scatter. For the Type II sample with overlapping tensile properties with the Type I samples (Figure 6-22d), the biased predictions by the ‘property-based’ model amount to a factor of about 1.7 on life (Figure 6-22b). This is acceptable considering the extent of data scatter.

Figure 6-21 Predicted against actual fatigue lives of the (a) training and (b) test data of the ‘process-based’ ANFIS model; predicted against actual fatigue lives of the (c) training and (d) test data of the ‘property-based’ ANFIS model. Dotted lines represent factor of two on life.

Figure 6-22 Comparison of experimental and predicted S-N data for (a)-(b) Type I samples, (c)-(d) Type II samples and (e)-(f) post-processed samples.

In relation to the underlying working mechanism of the ANFIS, the good prediction capability, despite the diverse mix of fatigue properties and fracture behaviours, can be attributed to the decomposition of the modelling task by the fuzzy rules [264]. For a given set of inputs, only rules corresponding to the specific input class are activated for life prediction. By dividing the design space into the sub-regions/clusters, with each being managed by the respective fuzzy rule, the dimensionality of the system is significantly reduced, leading to the good model performance.

The generalisation capability of the ANFIS was further evaluated by applying where appropriate the model to the literature data in Table 6.4, and those of Leuders et al. [93]. Relevant input information is listed in Table 6.7. Only the ‘property-based’ model was examined, as input values for the ‘process-based’ approach are not always provided or comparable.

Table 6.7 Relevant information and input parameters of literature data used for validating the ‘property-based’ ANFIS model.

References	Sample condition	Number of S-N data	Tensile properties	
			σ_b (MPa)	e (%)
Spierings et al. [92]	#1 Vertical as-built, machined	26	760	30
	#2 Vertical as-built, polished	26	760	30
Leuders et al. [93]	#1 Vertical as-built, machined	8	600	55
	#2 Vertical, machined, heat treated at 650 °C	8	618	54
	#3 Machined, HIPed	3	587	64
Mower and Long [94]	#1 Horizontal, stress relieved	11	717	28
	#2 45° inclined, stress relieved	10	680	30

Cross-validation results in Chapter 6.1.3 demonstrated the variability in the reported fatigue properties across the references because of the use of different L-PBF machines and processing regions. This is further confirmed by applying directly the ANFIS model derived based on the training data in this study to the literature data. As shown in Figure 6-23, a range of prediction accuracies was obtained depending on the dataset. Specifically, the model produced reasonable results for the heat treated samples of Leuders et al., but not for the other samples by the same authors, especially at the lower and higher fatigue life regions. Results for Spierings et al. and Mower and Long were significantly overestimated, by as much as over an order of magnitude in the extreme cases. Mismatched material properties reported by the studies are responsible for the varied modelling performance. For example, the tensile strengths of 760 MPa and 717 MPa, reported by Spierings et al. and Mower and Long respectively, are higher than those for samples with equivalent ductility values used in this study. As the higher tensile strength implies better fatigue resistance, the

fatigue lives were overestimated. For the HIP samples used by Leuders et al., as the tensile properties are comparable with this study, the poor predictions are suggestive of dissimilar fatigue properties. Noteworthy also is the considerably worse predictions for the inclined samples than the horizontal samples of Mower and Long despite the similar tensile properties. This is a result of actual difference in the experimentally determined S-N data, which, as noted by the authors, could have been caused by surface defects that promoted premature failure of the inclined samples.

Figure 6-23 Actual and predicted fatigue lives (load cycles) obtained from applying the ‘property-based’ ANFIS model to literature data. Dotted lines represent factor of two on life.

Considering the significant part-part and machine-machine variability observed for the literature data, a new ANFIS model was constructed by incorporating these data into the dataset for training. The load ratio was included as an additional input parameter such that the effects of load ratio need not be adjusted. A 6-rule ANFIS model was obtained from retraining the model. The prediction results for the retrained model are satisfactory, as shown in Figure 6-24, where the data points are generally within the factor of two on life bands. The overall RMS errors are larger than those for the original model, at 17.41% for the training data and 20.25% for the test data, because of the increased data complexity.

Figure 6-24 Actual and predicted fatigue lives (load cycles) for the (a) training and (b) test data obtained from applying the ‘property-based’ ANFIS model to literature data.

The better modelling performance can be appreciated by examining the fuzzy rules. Figure 6-25 shows the locations of the ‘cluster centres’ on the σ_b - e plot. In comparison with the original ‘cluster centres’ in Figure 6-19, three new ‘cluster centres’ were extracted. The locations of the cluster centres correspond to the tensile properties of the literature data, indicating that the clustering algorithm correctly captured the oddities in the data and assigned new rules for prediction. Task decomposition by the rule-based approach reduced the dimensionality of the problem, such that the use of the tensile properties as the inputs is still valid.

Figure 6-25 Locations of the ‘cluster centres’ for the ANFIS model constructed by incorporating the literature data into the dataset, in relation to the tensile strength and elongation to failure of the samples.

In comparison with the traditional modelling approaches, as demonstrated by the ‘process-property’ and ‘structure-property’ models, the machine-learning-based method clearly demonstrates better versatility and adaptability for modelling more complex data with significant variability. Besides being capable of accounting for the variability in properties across the difference data sources, it is also able to provide a uniform representation of the fatigue properties regardless of the fracture behaviour, e.g. recall that the regression models are not able predict effectively the fatigue life in the optimum processing region, while the modified Basquin equation is not able to predict the fatigue properties of the highly porous samples. The present results suggest the possibility of extending the machine learning model to highly complex systems, e.g. one that involves more processing variables, machine models and material types, provided sufficient training data are available.

6.4 Summary and conclusions

In this chapter, methods for predicting the high cycle fatigue properties of L-PBF stainless steel 316L are presented. Validations were done using the same literature

data, where applicable, for demonstrating and comparing the capability of the models. The following conclusions can be made from the results obtained:

- 1) The second-order polynomial equation predicts well the influence of laser power and scan speed on high stress fatigue life, leading to the construction of regression equation for mapping the process-property relationship. For low stress, high cycle fatigue, however, the model produces poorer performance because of the more complex fracture mechanism and a higher-order model is desirable. Cross validating the model with literature results show that the applicability of the regression models is process-window- and machine- dependent.
- 2) In exploring the structure-property relationship, a reduction factor of 1.9 on life was obtained to account for the effects of microstructural heterogeneity on the S-N properties. The area fraction of defects and fatigue life were found to follow a power-law relation, which was incorporated into the Basquin equation for fatigue modelling of parts containing critical lack of fusion defects. The proposed equation accurately predicted the fatigue fracture mechanisms, i.e. defect- or microstructure-driven failure, of literature data. Quantitative confirmation of the proposed defect-fatigue properties relation was not possible due to a lack of defect information.
- 3) Both the traditional design of experiment technique and stress-based fatigue modelling approach, as exemplified by the ‘process-property’ and ‘structure-property’ models respectively, require tremendous efforts for describing the high-dimensional L-PBF system and the associated complex failure mechanisms. A data science method was examined as the means to a unified approach for fatigue assessment. Good prediction accuracy was achieved by the proposed adaptive neuro-fuzzy inference system for a dataset containing samples exhibiting a wide range of fatigue properties. The use of fuzzy logic circumvents the shortcomings of conventional learning algorithms by allowing the system to be interpreted using linguistic rules. This allowed better appreciation of the model, simplifying the model design and validation process.
- 4) Transferability of the machine learning model to different L-PBF machines and processing regions can be easily achieved by incorporating the data for training. This suggests that the method could be used to address the part-part and machine-

machine variability, and the complex process-property relationship of L-PBF materials.

CHAPTER 7 CONCLUSIONS AND FUTURE WORK

This PhD research studied the high cycle fatigue properties and fracture behaviours of L-PBF stainless steel 316L in relation to the process-structure-property framework. The influence of critical processing defects on fatigue crack initiation and S-N properties were analysed; the role of cyclic plasticity as a function of processing and post-processing were examined in terms of the mechanism of fatigue-ratcheting interactions; models for predicting the fatigue properties were proposed and evaluated. This chapter summarises the conclusions made and recommendations for future work.

7.1 Conclusions

Three types of crack initiation modes corresponding to different L-PBF processing regions were observed. Firstly, microstructural defects, i.e. intergranular and intercellular particles and dislocations, are the weakest links in the material under optimum processing and contribute to intergranular cracking. They serve to enhance the material's tolerance to small gas pores and lack of fusion defects. Secondly, under-melting leads to lack-of-fusion-defect-driven crack initiation, where an estimate of the critical defect size was made to be on the order of 20 μm . Defect size and density associated with aggregating effects among the closely-spaced periodic defects were analysed by means of the Kitagawa-Takahashi diagram and the relations with fatigue life. Lastly, over-melting reduces the material's tolerance towards defects such that pores on the order of 10 μm become critical to crack initiation.

Worsened tensile strength as a result of build orientation or post-processing treatments, i.e. annealing or hot isostatic pressing, do not impinge on the fatigue properties under nominal elastic loading. Ratcheting occurs in the presence of high tensile mean stress, where cyclic plasticity is characterised by hardening. A modification to the Goodman relation was proposed to account for the effects of

plasticity on the fatigue properties. The relation is expected to describe the S-N properties of other L-PBF variables that incur the cyclic plastic deformation response. For factors contributing to the brittle defect-driven fatigue failure, the effects need to be treated separately.

The unique process-structure-property relationship of L-PBF demands modifications to conventional fatigue models. This was investigated via the process-property and structure-property routes respectively. For the process-property relation, results from employing the response surface method indicate that the second-order polynomial equation predict well the high stress fatigue life for a system with two input variables, but it produced poorer performance for the low stress, high cycle fatigue condition. For the structure-property relation, the stress-based fatigue modelling approach was used, where the microstructure-driven and lack-of-fusion-defect-driven failure modes and their effects on the S-N properties were modelled based on the premise of the Basquin equation. The latter approach demonstrated better capability for dealing with the machine and processing variabilities, as the use of defect fraction as the correlating term is associated with more fundamental representation of the failure mechanisms.

The general shortcomings of the traditional approaches to fatigue modelling of L-PBF materials lie in the inadequacies for tackling the high dimensionality of the L-PBF system and the complex fatigue failure properties. The fuzzy-based neural network method was demonstrated to be a promising alternative as it successfully predicted the fatigue lives without being restricted by the processing condition, machine, stress level and type of failure. The use of fuzzy logic effectively characterised the smooth transitions between fracture modes as functions of the input parameters and is therefore suited for high cycle fatigue modelling. Besides, the linguistic rules allowed interpretation of the neural network structure in terms of the fracture mechanisms, aiding the design and validation of the models.

The methodologies proposed for predicting the high cycle fatigue properties of L-PBF stainless steel 316L may be generalised to other L-PBF alloys, though some of the model parameters need to be recalibrated because of the different material

properties. For the modified Goodman equation, values of K and c_0 had been derived and validated for several material systems in this study.

Lastly, several design and assessment guidelines for L-PBF stainless steel 316L could be highlighted from the results obtained:

- Defects larger than 20 μm should be avoided during processing to prevent defect-driven crack initiation and ensure consistent structural performance.
- A linear relation was observed between fatigue limit and elongation to failure, which can be adopted for estimating the fatigue strength of porous parts. The conventional linear relation between fatigue limit and tensile strength is not valid because of the influence of defects.
- For parts devoid of critical lack of fusion defects, i.e. failure from microstructural defects, a reduction factor of 1.9 on fatigue life should be applied to account for the effects of microstructural heterogeneities on fatigue data scatter.
- Post-processing treatments such as annealing generate thermally-induced defects and subject the material to ratcheting, thus they are not recommended for L-PBF stainless steel 316L. Hot isostatic pressing should be performed with caution as it exposes previously benign defects, such as contaminants and unfused regions, to crack initiation.

7.2 Recommendations for future work

This PhD research examined the high cycle fatigue properties of L-PBF stainless steel 316L for enabling the use of the technology for structural fatigue applications. However, there are various issues that need to be addressed before the process could be used for the production of safety-critical components. Processing of parts with consistent quality remains one of the biggest challenges of the technology. In this work, small samples of simple geometry were produced under well-controlled processing environment, but in reality, the production of complex, large-sized components, involving the building of overhanging, support and even lattice structures, could lead to the formation of defects of various characteristics and different failure properties. This makes assessing and assuring the quality of L-PBF-

processed parts difficult. Therefore, types of critical defects associated with different processing conditions need to be categorised and the fatigue properties characterised. This could be complemented by design and property studies at the component scale.

Metal fatigue is an interdisciplinary field of research involving the study of materials science and fracture mechanics. This PhD research examined the high cycle fatigue properties, focussing on the crack initiation mechanisms and the S-N curves. Besides this, other fatigue design philosophies, such as those stated in the *British Standard 7608 Code of Practice for Fatigue Design and Assessment of Steel Structures*, deal with aspects such as probabilistic assessment of fatigue failure, crack growth properties for damage tolerant design, as well as the use of safety factors for safe life design. Depending on the specific applications, the effects of environmental factors, e.g. corrosion, and complex loading, e.g. multiaxial and variable loads, are also relevant. The mechanism of crack closure in relation to the L-PBF ultrafine grains, as highlighted in Chapter 5, could also be an interesting area for future research.

In terms of the application of data science for fatigue assessment of L-PBF parts, the experimental dataset should be significantly expanded to include larger design space that is more representative of the different types of fatigue failures that are relevant to the process. Processing information of different L-PBF systems could also be included for training the model. These will lead to a more complex system such that the use of machine learning methods could be better justified.

Stainless steel 316L was used as the model material in this research. Future works could be directed at expanding the knowledge of the process-structure-property relationships for other L-PBF alloys. Methodology proposed in this study can be applied and validated for these material systems. Considering the current trend towards multi-material processing, the fatigue properties of a wide range of L-PBF alloys in the pure or graded forms should also be understood.

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APPENDIX A: FRACTOGRAPHY ANALYSIS AND FRACTURE BEHAVIOURS OF CCD SAMPLES

This appendix provides detailed fractography analysis for the central composite design samples with respect to the processing regions indicated in Figure 6-4. The fracture mechanisms, in terms of the Type I, Type II and Type III failure modes, are discussed in relation to the L-PBF processing condition and process-induced structures.

Optimum region

The Type I failure mode applies for samples within the optimum region marked on the P - v map in Figure 6-4. Figure A-1a to Figure A-1c show the fractographs of S₄. Under high stress, fatigue crack develops with a considerable area of stable crack propagation (Figure A-1a). Intergranular crack initiation took place at solidification dendrite boundaries close to the specimen surface (Figure A-1b), as well as in the interior of the specimen (Figure A-1b), forming secondary crack origins.

As mentioned in Chapter 4.3.1, the competitive grain growth during rapid cooling produces differently-oriented grain clusters and sharp angle areas [248, 276], which are heavily stressed sites that promote intergranular fracture via the mechanism of slip/grain boundary interaction [277, 278]. Second phase particles such as oxide nano-inclusions were found to line the sub-grain boundaries [129] and could create stress concentration sites that promote intergranular cracking. Under the high applied stress, grain boundary defects in the bulk of the material could also develop into cracks such that failure traces back to multiple intergranular crack origins.

Figure A- 1 SEM fracture images of S4 (a-c), S7 (d-e) and S3 (f) under the high applied cyclic stress. (a) Large area of stable crack propagation in S4; (b) enlarged view of the boxed region in (a) showing intergranular crack initiation via de-bonding at cellular sub-grains; (c) secondary intergranular crack initiation site in the bulk material; (d) unstable crack propagation containing cleavage steps; (e) enlarged view of the boxed region in (d) showing transgranular crack initiation and slip bands; (f) featureless fracture profile due to the static ductile fracture mode.

The average equivalent circular defect diameter in these samples, as determined from the extreme value analysis, are on the order of 10 μm . They are mainly spherical gas pores generated by the trapping of chamber gas or impurities within the raw powder [279]. Some small lack of fusion defects, with average size of about 19 μm , were also detected in S_6 , but they did not constitute the primary crack origins. Microstructure defects create stronger stress concentration effect such that lack of fusion pores of this size are not critical to crack initiation.

Over-melt region

Figure A-1d to Figure A-1f show fractographs of the over-melted samples S_3 and S_7 . Comparing Figure A-1d with Figure A-1a, unstable crack propagation occurred in S_7 , whose fracture surface consists of large cleavage steps and a rugged morphology. In contrary to the grain boundary cracking in S_4 , crack initiation occurred by the transgranular mode, as shown in Figure A-1e.

Figure A-1f shows that S_3 failed via the static mode of dimple rupture with no distinct crack origin. Over-melting led to significantly reduced material strength such that the applied load exceeded the yield strength of the material. Nonetheless, under the low stress condition, the same transition in crack initiation mechanism (from intergranular to transgranular cracking) is applicable, as shown in Figure A-2. It can be seen that S_4 contains a large intergranular crack initiation region on the order of 100 μm (Figure A-2a to Figure A-2b), whereas crack initiation in S_3 is transgranular (Figure A-2c to Figure A-2d). Arrows in Figure A-2d trace the grain boundaries, where instead of sliding along the grain boundary as in Figure A-2b, cracking took place through the grains.

Figure A- 2 SEM fracture images, under the low applied cyclic stress, of (a) crack initiation zone in S₄; (b) enlarged view of (a) showing cellular sub-grains revealed due to intergranular cracking; (c) crack initiation zone in S₃; (d) enlarged view of (c) showing transgranular cracking through grains (arrows indicate grain boundaries).

As discussed in Chapter 4.3.3, over-melting impacts fatigue resistance by causing strength degradation. It is known that both low scan speed and high laser power reduce cooling rate [280], which impacts sub-grain size significantly by causing grain coarsening [281]. This, besides affecting the load-bearing capacity, also affects the distribution of second phase particles and the fracture behaviour. Specifically, lower cooling rate in L-PBF processing was found to be related to the transition from intergranular to transgranular fatigue crack initiation [184], as the larger dendritic arm spacing facilitates the trapping of particles between intercellular spacing rather than grain boundaries [133]. As a result, slip occurs more readily across the dendrite cells, causing the observed transgranular cracking. As fracture path is shorter for transgranular cracking, fatigue resistance could be reduced. Simultaneous cracking created the higher crack density and disturbance to the major crack front, where the

linking of individual cracks produced the stepped fracture profile and lower fatigue life.

Under-melt region

Reducing energy input from the optimum condition creates lack of fusion defects that trigger the Type II crack initiation mode. Figure A-3 shows defect-dominated failure in S_1 and S_5 . Under low stress, crack initiation could be traced to lack of fusion defects at the surface of S_1 (Figure A-3a). Interior defects, as indicated by arrows, could have caused localised cracking, but propagation of the major crack front is still transgranular and stable. For S_5 , no distinct major crack origin could be found (Figure A-3c). Brittle rupture occurred via the joining of large and interconnected defects.

Under high stress, even S_1 exhibited catastrophic failure. As shown in Figure A-3b, no stable crack propagation could be found adjacent to the crack origin. Ductile failure involving the coalescence of micro-voids dominates areas between large defects. In this case, overloading occurred since the load-bearing area was significantly reduced by the presence of the lack of fusion defects.

In addition, Figure A-3d shows S_6 under high stress, where crack fronts advanced individually from closely spaced lack of fusion defects and joined up with the main crack front to produce ‘crack jumps’. Even though primary crack initiation in S_6 is intergranular under both the high and low applied stress conditions, the lack of fusion defects were large enough to create sufficient local stress concentration such that secondary cracks were formed under the high applied stress. Defects in S_2 were the second largest for specimens in the optimum processing region, at 14 μm ; they could also have contributed to cracking in the bulk material. This explains the stronger effect of scan speed on high stress fatigue life: even though defects in S_2 and S_6 were benign under the low applied stress condition, they created sufficient stress concentration for cracking under the high applied stress and were responsible for the greater reduction in fatigue life. In fact, this explains the smaller amount of data scatter at high stress: crack initiation took place at uniformly distributed defects throughout the bulk material under high stress, whereas one or a few high stress

concentrating defects are responsible for crack initiation under low stress. In the latter case, attributes of individual defects, such as orientation, size and location, as well as characteristics of neighbouring microstructure, are all relevant factors which could have resulted in the more probabilistic failure properties.

Figure A- 3 SEM fracture image of S_1 (a) showing crack initiation from a subsurface lack of fusion defect (dash line) under the low applied stress; (b) it failed catastrophically under the high applied stress; (c) S_5 failed via the brittle route even under the low applied stress; (d) secondary cracking occurred at lack of fusion defects in S_6 under the high applied stress. Arrows indicate lack of fusion defects.