Investigation of microstructure and mechanical properties of A131 EH36 steel additively manufactured by selective laser melting and direct laser deposition

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INVESTIGATION OF MICROSTRUCTURE AND MECHANICAL
PROPERTIES OF A131 EH36 STEEL ADDITIVELY
MANUFACTURED BY SELECTIVE LASER MELTING AND
DIRECT LASER DEPOSITION

WANG JINGJING

SCHOOL OF MECHANICAL AND AEROSPACE ENGINEERING

2020

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PROPERTIES OF A131 EH36 STEEL ADDITIVELY
MANUFACTURED BY SELECTIVE LASER MELTING AND
DIRECT LASER DEPOSITION

WANG JINGJING

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

2020

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.

A handwritten signature in black ink, reading 'Wang Jingjing'. The script is cursive and fluid, with the first name 'Wang' and last name 'Jingjing' clearly distinguishable.

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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.

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Authorship Attribution Statement

This thesis contains material from 2 papers published in the following peer-reviewed journals in which I am listed as an author.

Chapter 4 is published as J.J. Wang, W.J. Wu, W. Jing, X.P. Tan, G.J. Bi, S.B. Tor, K.F. Leong, C.K. Chua, E.J. Liu, Improvement of densification and microstructure of ASTM A131 EH36 steel samples additively manufactured via selective laser melting with varying laser scanning speed and hatch spacing, Materials Science & Engineering A 746 (2019) 300-313.

The contributions of the co-authors are as follows:

- Associate Professor Liu Erjia, Associate Professor Tor Shu Beng, Associate Professor Leong Kah Fai and Professor Chua Chee Kai provided the initial project direction.
- I prepared the manuscript drafts. The manuscript was revised by Associate Professor Liu Erjia.
- I co-designed the study with Associate Professor Liu Erjia and Dr. Bi Guijun, and performed all laboratory work at the School of Mechanical and Aerospace Engineering and Singapore Centre for 3D Printing. I also analyzed the data.
- All microscopy analyses, mechanical tests and X-ray diffraction, including sample preparation, were conducted by me.
- Dr. Tan Xipeng and Mr. Wu Wen Jin participated in discussions on the material microstructure and properties.
- Mr. Jing Wei participated in sample preparation.

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- I prepared the manuscript drafts and Associated Professor Liu Erjia edited the manuscript drafts.
- I co-designed the study with Associate Professor Liu Erjia and Dr. Bi Guijun, performed all laboratory work at the School of Mechanical and Aerospace Engineering and Singapore Centre for 3D Printing. I also analyzed the data.
- Dr. Tan Xipeng, Ms. Zhang Meng and Associate Professor Li Hua participated in discussions on the microstructures and mechanical properties of the materials.
- All microscopy analyses, mechanical tests, including sample preparation, were conducted by me.

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- Associated Professor Liu Erjia and Associate Professor Tor Shu Beng provided the initial project direction.
- I prepared the manuscript drafts, and Associated Professor Liu Erjia and Dr. Bi Guijun edited the manuscript drafts.
- I co-designed the study with Associate Professor Liu Erjia and Dr. Bi Guijun, performed all laboratory work at the School of Mechanical and Aerospace Engineering and Singapore Centre for 3D Printing. I also analyzed the data.
- Dr. Tan Xipeng, Ms. Zhang Meng, Dr. Wang Bing, Mr. Liu Yang and Associate Professor Li Hua participated in discussions on the microstructures and mechanical properties of the materials as well as the methodologies.
- Dr. Liu Tong performed CT scan at Singapore Institute of Manufacturing Technology (SIMTech).
- All microscopy analyses and mechanical tests, including sample preparation, were conducted by me.

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- Mr. Wu Wen Jin, Ms. Sharine Tan Ying Jia, Mr. Lee Yuan Hao and Dr. Aziz Merchant participated in discussions on the materials, AM processes, and testing methods.
- I prepared the manuscript drafts. The manuscript was revised by Associated Professor Liu Erjia and Dr. Bi Guijun.
- I co-designed the study with Associate Professor Liu Erjia, Dr. Bi Guijun, Associate Prof. Tor Shu Beng and Dr. Tan Xipeng, and performed all the laboratory work at the School of Mechanical and Aerospace Engineering and the Singapore Centre for 3D Printing. I also analyzed the data.
- All microscopy analyses, mechanical tests and X-ray diffraction, were conducted by me.
- Dr. Tan Xipeng assisted in the interpretation of the fractured surface of fatigue sample using scanning electron microscopy.
- Dr. Bi Guijun and Dr. Chew You Xiang prepared the samples for tensile, charpy and fatigue tests.

03 Nov 2020

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Abstract

The microstructure of selective laser melting (SLM) processed ASTM A131 EH36 high strength low alloy steel has been studied in-depth [1]. However, there has been no report on the fatigue properties of SLM processed EH36 steel in the literature. The research on direct laser deposited (DLD) EH36 steel has not been reported either. In this research, ASTM A131 EH36 high strength low alloy steel was developed through two additive manufacturing (AM) processes, i.e. selective laser melting (SLM) and direct laser deposition (DLD), using EH36 steel powder. The research included the optimization of 3D printing process and characterization of the microstructures and mechanical properties of the additively manufactured (AMed) EH36 steel samples.

The SLM process parameters were optimized through tuning laser scanning speed from 100 to 400 mm/s, hatch spacing from 0.08 to 0.13 mm, laser power from 145 to 240 W, and preheating temperature from 100 to 200°C with a layer thickness of about 50 μm under a chessboard scanning strategy. It was found that increasing laser power and preheat temperature and lowering scanning speed improved the densification of the built samples to near full density (>99%). With lower laser scanning speeds, the grains were coarser and more like a cellular structure while with higher scanning speeds the grains were finer and more like a cellular-dendritic structure in the samples (around 1 μm). A large plate-like martensitic structure was found in the coarse columnar grains (up to 20 μm). However, different morphologies of the martensite grains were found mainly in cellular, columnar and lath shapes, which probably resulted from different transformation mechanisms and were controlled by cooling rate. Electron back scattered diffraction analysis showed that the prior elongated columnar grains with the martensitic plate-like structure delineated

the columnar grain boundaries. The fractured surfaces of the printed samples, examined with scanning electron microscopy, showed mostly a dimple type of failure. The microsegregation behavior of the samples and the degree of element escaping from the lattices were attributed to this rapid solidification process. The high cycle fatigue behavior, especially fatigue life, of the samples built at lower scanning speeds excelled those built at higher scanning speeds at which porosity induced fatigue failure dominated. S-N curves were built from the experimental data with respect to different scanning speeds and the fatigue failure mechanism was proposed.

The EH36 steel samples were printed using DLD process in four different orientations, namely, horizontal 0° (XY_0°) & 45° (XY_45°) and vertical 45° (XZ_45°) & 90° (XZ_90°). The results showed that all the steel samples printed in the four orientations satisfied the ASTM standards for tensile and charpy impact properties. The microstructure and mechanical properties of the samples could be tuned by controlling the printing process parameters. The different thermal cycles encountered in the different build orientations could also be complemented by adjusting the process parameters. The lack of fusion defects were more prominent in the samples built in the two vertical directions, which was probably due to oxidation that impacted the interlayer bonding. The fatigue life was affected most severely by the lack of fusion defects for the samples printed in the direction XZ_90°. The fatigue fracture mechanism of the DLD printed EH36 steel samples was proposed and the mechanical properties were closely correlated with the microstructure.

In addition, the influence of surface porosity on the fatigue life of AMed EH36 steel samples via selective laser melting (SLM) was studied in-depth by using a numerical method, which the pores were simulative AM 3D pores. The surface pores in both simplified semi-ellipsoid/spheroid and

more realistic triangular shapes were evaluated in the first place. Stress concentration factor K_t analyzed by ABAQUS and fatigue life assessed by FE-SAFE were closely related to the AM 3D pore size, shape, position, orientation as well as their interactions, which confirmed their effects on the crack initiation stage during fatigue testing. The effectiveness of adding a surface finish factor K_t to a smooth surface fatigue model was subjected to internal pore geometry in terms of K_t values ranging from 1 to 5.5. Different algorithms were attempted with the stress-life method gives a higher estimation than a strain-life method while became invalid when the stress concentration situation was rather serious (around $K_t = 2.5$). There was no much difference between the uniaxial and multi-axial fatigue algorithms because the uniaxial tensile cyclic loading was applied to the model. Theoretically calculated total fatigue life (crack propagation) using linear elastic fracture mechanics (LEFM) fell in the same order (within a factor of 2) as the experimental results for all the samples. AMed material parameters were used for fatigue life prediction where the microstructure element was counted. The pores in an AMed material could severely deteriorate its mechanical properties, such as strength, Young's modulus and fatigue lifetime, etc. The statistical analysis showed the scatter band of the SN curves with a factor of 8 with respect to different scanning speeds. All the above results contributed to scientific knowledge and provided a design guideline for possible marine and offshore applications of the AMed grade EH36 steel.

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List of Symbols

σ_u	Ultimate tensile strength
σ_y	Yield strength
E	Young's modulus
ν	Poisson ratio
e	Elongation
ε	Strain
σ_w	Fatigue limit
$\sigma_{n,m}$	Normal mean stress
σ_a	Stress amplitude
ε_a	Strain amplitude
N_f	Number of fatigue life cycles
N_i	Number of fatigue initiation life cycles
σ_f'	Fatigue strength coefficient
b	Fatigue strength exponent
ε_f	Fatigue ductility coefficient
c	Fatigue ductility exponent
$\Delta\sigma$	Stress range
$\Delta\varepsilon$	Total strain range
$\Delta\varepsilon_e$	Elastic strain range
$\Delta\varepsilon_p$	Plastic strain range
$\Delta\varepsilon_n$	Normal strain range revolved to shear stress plane
$\Delta\gamma_{\max}$	Maximum shear strain range

R	Stress ratio
K'	Cyclic strength coefficient
n'	Cyclic strain hardening exponent
K _{IC}	Fracture toughness
K _q	Conditional fracture toughness at a critical point
da/dN	Fatigue crack growth rate
ΔK	Stress intensity factor range
ΔK _{th}	Fatigue crack growth threshold
$\sqrt{area}$	Effective defect size
K _t	Stress concentration factor
K _f	Fatigue notch factor
P	Laser power
v	Laser scanning speed
h	Hatch spacing
R	Solidification rate
G	Temperature gradient
γ	Austenite phase
α'	Martensite phase

List of Abbreviations

CAD	Computer aided design
EBS	Electron backscattered diffraction
EDS	Energy dispersive X-ray spectroscopy
FESEM	Field emission scanning electron microscopy
OM	Optical microscopy
XRD	X-ray diffraction
AM	Additive manufacturing
SLM	Selective laser melting
DLD	Direct laser deposition
EBM	Electron beam melting
LMD	Laser metal deposition
ED	Energy density
HIP	Hot isostatic pressing
BCC	Body centered cubic
CCT	Continuous cooling transformation
TTT	Time temperature transformation
HCF	High cycle fatigue
LCF	Low cycle fatigue
VHCF	Very high cycle fatigue
LEFM	Linear elastic fracture mechanics
H _{IT}	Indentation hardness
H _v	Vickers hardness
HRC	Rockwell hardness measured on the C scale

HB	Brinell hardness
A	Austenite
B	Bainite
C	Cementite
F	Ferrite
M	Martensite
P	Pearlite

Chapter 1 Introduction

1.1 Background

Additive manufacturing (AM) differentiates itself by building materials layer-by-layer from conventional manufacturing that is subtractive. Three typical AM processes are schematically illustrated in Figure 1-1 [2-4]. Among them, selective laser melting (SLM) characterizes itself as a powder bed AM process with priority in making fine features with a typical thin layer of 20 -100 μm as shown in Figure 1-1a. The slow building rate makes SLM lose productivity to powder blown process such as direct laser deposition (DLD) as shown in Figure 1-1b that normally can produce a layer of 200 - 800 μm thick and 600 - 2400 μm wide. DLD technologies are usually adopted in aeronautics industry for repairing high valued components instead of replacing them. Another powder bed process is electron beam melting (EBM) that distinguishes itself with a preheating procedure to a high temperature to avoid the formation of brittle martensitic structure, but its application is restricted by its requirement of building parts in a high vacuum environment.

(a)

(b)

(c)

Figure 1-1 Schematic diagrams of (a) SLM[4], (b) DLD [3], and (c) EBM processes[5]

The adoption of AMed parts has been succeeded in medical sector, e.g., implants and scaffolds as they can satisfy the requirements on surface quality and mechanical properties [6-9]. A great impact has been made to other industries like automotive, aerospace, biomedical, electronics, etc. on design and application with the growth of the AM technologies in creating end-use parts. Various kinds of metals and alloys have been successfully processed by SLM and DLD processes

and even functionally graded parts made by multi-materials are fabricated with their applications in harsh environments [10-12]. However, the adoption of these technologies in Marine and Offshore (M&O) industry is reluctant owing to its harsher service environment and lack of standards and guidance for AM grade of metals.

In Marine and Offshore industry, steels (marine grade) constitute majority of M&O structural components, which are adopted due to their low cost, ease of welding and moderately good mechanical properties. The main stream of manufacturing processes for large scale M&O components is composed of tool making, casting, rolling, machining, welding, etc., where large amounts of effort, space, cost, time and human resources are consumed during these tedious processes. However with AM technologies, the manufacturing processes can be readily simplified in a way of both time and cost effective.

As AM is expanding quickly and the AM market is expected to grow 390% by 2023, the newly added AM technologies shall comprise quality and safety standards [13]. However there are limited guidelines to be used in AM industry. Lloyd's Register's "Guidance Notes for Additive Manufacturing of Metallic Parts" can be adopted to award the certification to those who pass their standards. Meanwhile ISO/TC 261 and ASTM F42 committees are working on developing relevant AM standards which is to qualify AM processes and systems and give to manufacturers and end users confidence for safe, reliable and robust products.

1.2 Problem Statement

In a harsh environment of Marine and Offshore industry, it is important for structural steels to possess both good strength and toughness along with good fatigue performance mainly because brittle fracture and fatigue failure are severe failure modes of ships and offshore structures. However, the current manufacturing methods of complex shaped components in M&O industry, such as star panel, column-to-pontoon nozzle, etc., are time and cost consuming and often involving intensive welding operations that require skilled workers and advanced welding equipment, while the defects of welds often initiate cracks (fatigue, embrittlement, etc.) in ship structures that can lead to a catastrophic failure. As a new emerging technology, AM has attracted industrial partners' interest for its possible transformation of industrialization. The goodness of AM process is well known, such as fabricating complex shape of structure in one step. There are functional parts being manufactured and applied worldwide[14], but less in M&O industry. A study on EH36 grade structural steel for ship construction conducted by Wu et al. [1] provided some preliminary results on tensile, charpy impact properties as well as heat treatment effect on as-built parts, but whether the quality of the AMed metals such as the structural steels for ship construction can be certified by M&O industry needs further validation. In response to the above-mentioned, we have proposed two potentially applicable AM processes, i.e., selective laser melting (SLM) and direct laser deposition (DLD), to develop EH36 steel in this study. Both SLM and DLD are well-known for printing metals, which can provide a one-step manufacturing solution to a complex-shaped component with minimum material waste. This research will focus on optimizing process parameters and evaluating the microstructures and mechanical properties, in particular the fatigue behavior and fracture mechanisms, of the printed EH36 steel samples with reference to ASTM standards. The appropriate processing window and the mechanical properties of the built samples out of this study can be provided to industrial partners as the design guidelines for M&O

applications. The whole investigation process is generic to a family of high strength, low alloy steels including ASTM A131 EH36 and API 5L X65 steels.

Simulation is important to predict the fatigue life of AMed steels. The simulation of fatigue behavior is usually taken by engineers to optimize the design of the component before it is put into production where fatigue testing is not applicable or too time and cost consuming. Meanwhile, simulation work is an alternative when conduction of fatigue test is impossible, e.g. for complex shaped components or under multi-axial loading. Therefore, fatigue simulation through software becomes utmost important[15]. In this research work, an initiative of taking into consideration the microstructure features and the 3D defects/pores is taken to implement the traditional simulation model that never includes microstructural features. The simulated results will be verified with the experimental data.

1.3 Objectives

This Ph.D. research aims to deeply understand the microstructure, toughness, fatigue behavior and fracture mechanism of the additively manufactured high strength, low alloy EH36 steel samples as well as their relationships with the following objectives:

1. To improve the densification of SLM and DLD built EH36 steel samples.
2. To study the microstructures of the SLM and DLD built EH36 steel samples, e.g. density, phases, defects, grains, etc., which serves to understand the relationship between thermal phenomena and microstructures.

3. To evaluate the mechanical properties of the SLM and DLD printed EH36 steel samples with the focus on their fatigue behavior and fracture mechanisms.
4. To correlate the printing process parameters with the microstructures, mechanical properties and performance of the SLM and DLD produced EH36 steel samples.
5. To predict the fatigue life cycle of 3D printed EH36 steel samples by incorporating microstructure features , e.g., defects/pores through numerical study

1.4 Scope

The work scope is as follows:

1. EH36 steel samples will be additively manufactured by both SLM and DLD processes under various process parameters.
2. Optical microscopy (OM), scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDX), electron backscattered diffraction (EBSD), transmission electron microscopy (TEM), and X-ray diffraction (XRD) will be used to investigate the microstructures and fracture mechanisms of the SLM and DLD built EH36 steel samples, e.g. surface morphology, surface roughness, grain structure, porosity, phases, defects, fractured surfaces, etc.
3. Tensile, microindentation and fatigue tests will be used to characterize the mechanical properties of the SLM and DLD built EH36 steel samples, e.g., microhardness, tensile strength, yield strength, and fatigue performance. Statistical models will be established to understand their relationships.
4. The mechanical tests will follow relevant ASTM standards.

5. ABAQUS and FE-SAFE software will be used to do static stress analysis and simulate the fatigue life of SLM built EH36 steel samples. Simulated results will be verified by experimental data.

1.5 Motivations and Challenges

As mentioned above, the conventional manufacturing processes of Marine and Offshore structures have their limitations. This leaves a chance for developing equivalent AM grade of steel components. However, adoption of AMed metals by marine and offshore industry also faces challenges that are rooted from the complex process-microstructure-property relationship and lack of AM standards. Until now, no optimized process parameters have been reported, which can guarantee robust material processing. Process induced porosity in AMed materials is most concerned as it usually does not exist in conventionally manufactured metals. This is due to the fact that even though near full bulk densification is achieved large pores up to a few hundreds of microns are still observed within the built materials [16, 17]. The reason could be due to non-optimized or instable process conditions, which indicates the complexity of process optimization.

Among the mechanical properties, toughness is sensitive to porosity as pores reduce ductility and strength of AMed materials. Pores can act as stress concentrations that can avoid the nucleation stage of cracks and lead to premature failure [18, 19]. But, there is an opinion that a few percent of porosities in AMed materials will not change stress concentration from a large aspect [18]. Meanwhile, the elastic modulus of the materials could be reduced due to the existence of pores. Fatigue is also sensitive to porosity. For high cycle fatigue behavior cracks are mostly initiated

from the surface or sub-surface pores whereby those pre-existing defects can lead to premature failure and decrease the fatigue strength of AMed materials.

The strong anisotropy in the sense of porosity distribution, morphology & orientation of both pores and grains, texture, etc., resulting from the layer-by-layer rapid solidification process causes the properties to vary along the printing orientations. Normally, the vertical build direction is the worst scenario, which is attributed to its higher concentration of detrimental pores aligning along the build.

Knowing the mathematical relationships between properties, microstructure, such as pores and grains, and process parameters makes prediction possible. The data can be fitted according to existing mathematical models and their own correlation parameters can be established [20-23].

The process induced porosity makes traditional models not accurate[24]. For example, models that can bring in microstructure features in fatigue life prediction are needed.

Meanwhile the steels used in Marine and Offshore industry are specially formulated to cater their processes such as rolling, etc. There is a need to explore the suitability of the same compositional steels built through AM. Preliminary work on EH36 grade high strength steel (for ship construction) was conducted through a SLM process [25] with only process parameters, and the tensile and charpy properties being investigated without fatigue data.

Finally, an understanding of the relationship between process-induced microstructure and mechanical properties is necessary to produce quality parts that can enhance industrial partner's confidence in adopting AMed products.

1.6 Novelty and Originality

For the first time, the AM grade EH36 steel samples (both near-net-shape built and cut from built blocks) were manufactured through both SLM and DLD processes and evaluated for their fatigue behavior and fracture mechanisms. Their microstructure forming mechanisms and fracture mechanisms (tensile, charpy impact, fatigue) were detailed by the author in the first place. The comprehensive understanding of the SLM and DLD processes, the microstructure and mechanical properties of the AMed steel samples, and their interrelationships can help both academia and industry for the adoption of additive manufactured EH36 steel, which can be further extended to its entire high strength, low alloy steel family (including API 5L X65 steel). The microstructure forming mechanisms and fracture mechanisms of the AMed samples were explored at nano, micro and macro scales with reference to the theories of solidification and crack initiation/propagation. Meanwhile, for the 1st time, the fatigue behavior of the 3D printed EH36 steel samples was numerically studied with the microstructural features being taken into consideration.

1.7 Organization

The PhD thesis is organized into 7 chapters. Chapter 1 introduces the background, problem statement, objectives, scope, motivations, challenges, novelty and originality of the research work.

Chapter 2 reviews the theories of microstructure forming mechanisms and fracture mechanisms of materials additively manufactured via SLM and DLD processes. The mechanical properties of metals built from the two processes are critically reviewed as well. The theories about fatigue life prediction are also reviewed. Chapter 3 describes the experimental details and methodologies and chapter 4 presents the results from the mechanistic study of microstructure forming for SLM process. Chapter 5 discusses the fatigue performance of the EH36 steel samples built from SLM process and the models predicting fatigue life cycles that are related to 3D microstructural features. Chapter 6 presents the results of the DLD printed EH36 steel samples on microstructure and tensile, Charpy impact and fatigue characteristics. Chapter 7 draws the conclusions and suggests the future work.

Chapter 2 Literature Review

This chapter introduces the materials processed from both conventional manufacturing and AM methods which are commonly used in Marine and Offshore industry as well as their properties. A microstructure incorporated model for prediction of fatigue life is drawn from the literature. A briefing of the state of the art of AM technology is made on SLM, DLD and EBM with their abilities for building marine and offshore structural metals. Then it is streamlined down to one targeted material EH36 steel for an in-depth study through SLM and DLD processes. A comprehensive review is made on the processes, materials, microstructures, properties and intricate property-microstructure-process relationship with a focus on AM built steels. The potential and challenges of SLM and DLD processes are stated.

2.1 Structural Steels Used in Marine and Offshore Industry

Over 90% - 95% of the Marine and Offshore structures are composed of steels[26]. Choosing marine grade of steels is due to their low cost, good weldability and mechanical properties. The steel classification is widely varied across different application areas. Marine and offshore has its separate system that varies between different international codes. In a common ground a prerequisite is sound strength and toughness. They are mostly C-Mn steels with yield strength ranging from 270 MPa to 690 MPa (varying by grades), for example EH36 with carbon content usually $< 0.15\%$ and a yield strength of 355 MPa. Other elements such as Ni, Cr, Mo and Mn are added to control their properties.

Higher strength grade of steels has been motivated by their improved weldability, low temperature toughness and high strength to weight ratio, which is essentially important to build large scale Marine and Offshore construct in a cost effective manner. General practice of strengthening for steels to achieve high strength and toughness is by grain refinement by adding microalloying elements (e.g. small amount of Ti, V and Nb, < 0.1wt%) and through Thermo-Mechanical Controlled Process (TMCP). Those are designed to refine grains by a series of controlled rolling followed by in process cooling. The resultant microstructure typically contains acicular ferrite, bainite and pearlite. In the circumstances even higher strength and toughness are required HTS, HY80 and HY100 grade of steels can be applied. Those are quench hardenable steels through martensitic transformation with additional carbon content. Sometimes bainite can exist at thicker sections due to slower cooling rate.

Stainless steel is also adopted as marine materials owing to their high corrosion resistance that can cope with crucial environment. Examples are the grade of 316L, 310, 17-4PH, 904, 13Cr etc. Nickel based alloy is also used in Marine and Offshore industry due to their high strength, excellent oxidation resistance up to 960°C and excellent weldability. Common grades are 718, 625, 825 and MP-35-N, etc.

2.1.1 C-Fe Binary System

Pure iron is very soft and easily rusted without much application. By adding alloying elements such as C, Mn, Si, etc., the material strength is much improved making steel one of the most popular materials in the modern society. Carbon is the most common alloying element which can

increase strength and hardenability of iron systems. A typical Fe-C phase diagram is shown in Figure 2-1[27], which is well structured for understanding the properties of Fe-C systems as well as being a useful tool to formulate desired steel properties.

Figure 2-1 Fe-C phase diagram [28, 29]

2.1.2 Other Alloying Elements

Other than carbon, there are some other alloying elements added to Fe matrix to form different types of steel including Mn, Si, Cr, Ni, Mo, Cu, Co, W, etc., each contributing to different steel properties and microstructures. With all the elements added to a Fe matrix, they will act together and affect Fe-C phase diagram as well as crystal structure. To have a better control and avoid the harmful effect of the alloying elements, a microscopic-scale understanding of the alloying element state in the Fe matrix is necessary. Table 2-1 listed most of the alloying elements encountered in

iron-carbon alloy systems in steels and the possible compounds they formed with Fe and C. Some elements do not form compounds instead favoring solid solution state by substituting Fe atom in the lattice structure, but some other elements favor forming carbides or nitrides with carbon or nitrogen probably due to their very low compatibility or solubility in the Fe matrix. Carbides or nitrides formed between alloying elements and C or N play a very important role in HSLA steels, which will be discussed in details later. The nonmetal inclusions formed by Al, Si, V, Ti, etc. due to their great affinity to oxygen could be detrimental to the alloy systems

Table 2-1 Alloying elements in steels and their effects[30]

Element in steel	Formation of compounds	Form/state in steel
Ni, Si, Co, Al, Cu and N	Elements do not form carbides in steel	Do not form compounds with iron, only in solid solutions
Cr, Mn, Mo, W, V, Ti, Zr and Nb	Elements form stable carbides in steel	chemical compounds with carbon and iron or present in solid solution
N	>0.015wt% forms nitrides with Fe, V, Al, Ti, and Cr	Nitride
Cu	Dissolves in α -Fe matrix if <1wt% at normal temperature, metal inclusions if >7wt%	N.A.
Al, Si, V, Ti	Deoxidizer and form Al_2O_3 , TiO_2 , V_2O_5 and other oxides	Fine nonmetal inclusions
Most alloying elements except C, N, O, B and metalloids standing far from iron in the periodic table	Co, Ni, Cu, etc standing to the right of iron form solutions in iron only and do not enter iron carbide.	Alloying elements dissolve into ferrite, austenite, cementite of iron-carbon alloys or form special carbides
M_3C , $M_{23}C_6$, M_7C_3 , M_6C	Carbides of group I	Complicated crystal structure, an example is cementite (Fe_3C), or $Cr_{23}C_6$; generally form relatively coarse dispersions

2.1.3 Microstructure of Steels

There are four basic phases in steel: austenite (A), ferrite (F), cementite (C) and martensite (M) each having different properties. The microstructure varies that may contain single phase or mixture of different phases which varies in morphology and size. The being formed microstructure depends on the thermal history and chemical composition, etc. Take hypoeutectic steel for example (with carbon content $< 0.77\%$), under an equilibrium state, the transformed microstructure will be a mixture of ferrite and pearlite phase as shown in Figure 2-2. However the products achieved under non-equilibrium conditions are more complex which can be illustrated by time-temperature-transformation (TTT) diagram and continuous cooling transformation (CCT) diagram shown in Figure 2-3 and Figure 2-4. The TTT diagram, also called isothermal transformation diagram illustrates the microstructure dependence on controlled time and temperature which shows the percentage of phases vs both time and temperature. It can be read that when cooling rate is fast enough martensitic structure can be formed. In another hand the CCT diagram, known as non-isothermal transformation is more helpful in real situation. The end products are mixture of phases depending on the cooling rate and the materials. In a common ground most of the alloying elements can push the TTT or CCT diagrams to the right and downward.

Figure 2-2 Equilibrium transformed microstructure of hypoeutectoid steel with carbon content < 0.77% [29, 31]

Figure 2-3 TTT diagram with martensite transformation [28, 29, 31]

Figure 2-4 CCT diagram of a low carbon steel with 0.2% carbon[32], where B, F, M and P stands for bainite, ferrite, martensite and pearlite, respectively.

2.2 AM Technologies for Metals

The concept of AM started from 1860, but until recently AM started its rapid progressing and commercialization. Over last 30 years, commercialized machines have been developed along with printing materials, printing speed, build size and applications.

2.2.1 AM technologies summary

There are a range of AM technologies so far for processing metals, polymers, ceramic and glass materials. Table 2-2 list some of the typical AM technologies in the market. In this work, the focus is in metal printing technologies which will be further elaborated in the following part.

2.2.2 Laser and Other Heat Source

Laser with an acronym of “light amplification by stimulated emission of radiation” can be used for metal cutting/melting due to its high energy nature. There are many kinds of materials that can be made “lase” including gases, liquid, solid crystalline, glasses and polymers depending on the application. A laser beam is coherent, usually with one wavelength or one color and directional. A typical laser system includes gain medium, laser pumping energy, high reflector and output coupler. From quantum theory when the gain medium is stimulated by pumping energy in the supply of an electric current or light at a different wavelength, the electron is excited from lower energy orbit to higher one. However the electron is not stable in high energy orbit which will decay to the ground level, emitting a photon. Then the light will be further amplified (higher power) by stimulating other atoms through the gain medium when being bounced back and forth by the highly reflective mirror on either end of the gain medium and eventually pass through the output coupler, typically one of the two mirrors which is partially transparent. The laser could be continuous or pulsed which can be further tuned according to the application. There are other types of heat sources such as the electron beam, thermal and piezoelectric, etc. The use of electron beam requires the materials to be conductive so that the materials to be processed by EBM is mostly metals and in vacuum environment. The thermal heat is usually applied for low melting point plastics/polymers which is adopted by FDM, material jetting, etc.

Table 2-2 AM technologies with materials being processed and heat sources[33]

AM Technologies	Processing materials	Heat sources
Selective Laser Melting (SLM)	Metals, Ceramics (powder form)	Laser(Nd:YAG, Fibre)
Direct Energy Deposition (DED)	Metals (powder and wire form), Ceramics	Laser(Fibre,CO ₂),Arc, Electron beam
Electron Beam Melting (EBM)	Metals (powder form)	Electron beam
Selective Laser Sintering (SLS)	Plastics, ceramics, glass and metals (Powder form)	Laser (CO ₂)
Fused Deposition Modelling (FDM)	Plastics (filament form)	Thermal
Stereolithography (SLA)	Photopolymer resin (liquid form)	Laser (Ultraviolet)
Material jetting	Polymers and waxes (Droplet form)	Thermal or piezoelectric method
Binder jetting	Plastics (Powder form)	Adhesive
Sheet lamination	Metals (Sheet, ribbon or paper form)	Ultrasonic or adhesive

2.2.3 Selective Laser Melting

Selective laser melting uses high energy focused laser beam to locally melt metal powder. 3D objects are built layer by layer with the layer thickness of 20 ~ 100 μm . This is to ensure an

effective laser penetration for metallurgical bonding with below solid substrate or built materials. While ideal powder will be spherical shape, medium size with narrow particle distribution (sometime in bimodal mode) for a high initial packing density. The schematic of the SLM process is shown in Figure 1-1(a)[2] where building of parts is taking place within an enclosed environment with size up to a few hundred mm under protection of inert gas Ar, N₂ or operating at vacuum condition. He and H₂ are not used because they can't maintain continuous energy supply[34]. Oxygen level is controlled at 500 ppm which can ensure the processing of reactive metals. The platform can be heated up to 200°C usually, which is taken to prevent stress cracking and delamination during building. In a word SLM allows complex shape of components to be built with good geometrical and property control.

2.2.4 Direct Laser Deposition

Direct laser deposition (DLD) has several synonyms: Laser engineered net shaping (LENS), direct metal deposition (DMD), direct energy deposition (DED), 3D laser cladding (LC) and direct light fabrication (DLF). The typical DLD process is illustrated in Figure 1-1(b)[3]. A nozzle used to deposit melted metal on service plate is mounted on a multi-axis arm that can move in multiple directions. Usually DLD is used to repair military equipment, turbine or other high-end equipment. But it also can be used to build large 3D metal parts e.g. larger than 1 m or 5 m in some case. Material being used can be both powder and wire form. The energy source could be laser, electron beam or plasma arc. Meanwhile the process can be used to deposit polymers, ceramics but mostly metals. Again the DLD process outperforms in building size and rate over SLM.

2.2.5 Electron Beam Melting

Electron beam melting uses a high power electron beam to melt powder layer-by-layer in a controlled vacuum environment. The vacuum level is controlled to 1×10^{-5} mbar throughout the building process. A partial pressure of 1×10^{-3} mbar can be created by introducing He gas to maintain a clean environment during actual printing. A special pre-scanning procedure is used to warm up the entire powder bed to prevent formation of any martensitic structure resulting in stress relieved components. The schematic is shown in Figure 1-1(c).

2.2.6 AM Process Parameters

AM involves an instant melting and rapid solidification process by using a high power heat source. When laser irradiates on powder bed or shining on the substrate, the material covered under the laser spot melts instantaneously and forms a tiny melting pool. In the sense of DLD the melting pool is larger and more close to conventional laser welding process. The width and depth of the melting pool with its behavior is highly related to process parameters and materials thermal physical parameters[35], which will further affect the local thermal cycle and the microstructure formed. Hence it is essential to study the process parameters effects in order to build highly dense and defect free objects.

There are quite some process parameters that can affect quality printing including laser power, scanning speed, hatch spacing, layer thickness, scanning strategy, spot size, preheat temperature, etc. There are also other parameters that could affect AM process stability and printing quality from certain aspect such as the gas flow rate, raw material properties, etc. For DED process, the

laser power, scanning speed, powder delivery rate, etc. will be the controlling factors. Others like gas flow rate and part geometry will be the affecting factors as well. The later factor can influence the process cooling rate from a large aspect due to the relatively large heat accumulation (from large energy input) of DED. All those parameters play together to take effect to the mass and heat transfer within the melting pool and so on to the melting pool behavior.

2.2.7 AM Process Tuning and Optimization

2.2.7.1 Design of Experiment (DOE)

DOE[36], Taguchi method[37, 38], response surface method[39] and Box-Behnken design[40], etc. are widely used methods for optimizing parameters in various fields including AM. The goodness of DOE is that it can evaluate multiple parameters simultaneously (along with interactions) using a lesser amount of runs compared with OFAT (one factor at a time) method[41]. Liao et al.[36] used DOE method to optimize the parameters for the effect of pulse frequency and pulse duration of laser beam, no. of strikes of a pulse and particle size to the amount of porosities formed of the SLS process using Ni powder. However, mostly the process work done using commercial AM printers are mainly concentrating on controlling factors such as laser power, scanning speed, hatch spacing, layer thickness and printing strategies, etc. The rareness on other factors are rather due to the uneasy adjustment or setting. Most of them are fixed according to the guidance of manual in using commercial AM printers. Meanwhile AM is a relatively new process and limited by the few types of commercially available AM printers, the development on controlling factors shows more significance to the new materials to be developed. Based on the

literature review, there are common rules and methodologies for the process tuning and optimization as elaborated in the followings.

2.2.7.2 Experimental Development on Energy Density, Laser Power, Scanning Speed. etc.

A common rule to tune processes for both SLM and DLD process is by a single parameter - energy density (E). E is formulated with process parameters typically including laser power, scanning speed, hatch spacing and layer thickness, powder feed rate, spot size, etc. Here only take laser power, scanning speed and hatch spacing for examples, $P/(s \times h)$ where P is laser power, s is scanning speed and h is hatch spacing. There are some common practices when choosing E value e.g. the threshold value which stands for the minimum energy density for a sound printing for a high densification. The threshold E value is material related.

Process optimization can be made by evaluating a series of E value combined with ranges of varied process parameters [42]. Details will be discussed in the results part. It is known that laser power and scanning speed are two most important parameters to adjust E. Many studies have been conducted regarding energy density to process stability and part forming. Ma et al. have studied the energy input (E in J/mm^2 by varying laser power and scanning speed) effect to metallurgical behavior of 316L in two processes: selective laser melting and laser cladding deposition[20]. The depth-to-width ratio increases with E for SLM process but decreases for LMD process.

Alternatively a simple single track printing could be used to find the correct process parameters as the integrity of AMed parts strongly depends on the single track as long as single layer and the

connection strength between them. Yadroitsev has given a good evaluation of track quality to process parameters through the study of 904L with SLM process[43]. They have demonstrated the changes of track geometry by changing hatch spacing. With too high hatch spacing tracks started to isolate and with too low hatch spacing powder consolidation following denudation could be diminished and lead to an instable melting pool behavior. An illustration is shown in Figure 2-5. Similar rules can be applied to scanning speed. If the scanning speed is too high, as shown in Figure 2-6 [44], wetting is poor due to insufficient heat input that can lead to poor interlayer bonding and low densification. If laser scanning speed is too low, melting pool can become instable as well where spattering happened, which again can reduce printing quality by forming large pores due to irregular shape of melting pool that deteriorated surface roughness. For DED process, the powder feed rate may affect layer thickness and laser power required for proper melting where process tuning involves all parameters. High input energy can also be induced by high laser power as disclosed by Dilip et al. [45] that keyhole-shape melt pool caused vapor being entrapped in the melt pool and form large gas pores. Similarly for laser power as that too high laser power causes larger gas pores due to keyhole-shape melt pool where too high energy causes alloy to melt and vaporize which is entrapped in the melt pool. In a contrast when laser power is not enough, more irregular shape of lack of fusion pore is presented within the 3D matrix.

(a)

(b)

Figure 2-5 Profiles of sequential tracks produced from SS grade 316L powder with (a) 60 μm (50% overlap), and (b) 100 μm hatch distances[43].

Figure 2-6 (a) Cross-section views of laser sintered tracks from SS grade 904L powder on steel substrate and (b) stability map for segmental and free circular cylinders. Φ is the angle defining the contact width, D is the diameter, and L is the wavelength of the molten cylinder[44].

The in-process re-melting is one of the remedies for part quality improvement mainly in the aspect of densification and surface roughness and possible microstructure refining [46-48]. Rashid et al. studied the scanning strategy effect to density and metallurgy behavior of SLM processed 17-4PH and found slight difference in density but large difference in size integrity of printed samples[49]. The percentage and distribution of retained austenite vary by using “O” and “X” scanning patterns, which affect the hardness of the built materials. The cracking behavior and grain structure of Ni based alloy CM247LC processed by SLM was studied by Carter et al. and they related both the bi-modal grain structures with fine elongated grain region and an occurrence of heavy crack region (Ductility-dip cracking) to the overlapping of island scanning[50]. Layer thickness, preheating temperature and environmental effect, etc. have been studied and results are elaborated in another report[34].

To print a new material is rather material's properties related such as viscosity, thermal conductivity, etc. where proper process parameters need to be investigated individually[35]. Mukherjee et al. studied the printability of different alloys based on a simulation model through laser additive manufacturing (LAM) process and concluded that the porosity, lack of fusion, composition change varied with materials as seen in Figure 2-7& Figure 2-8 with alloy AISI 1040 steel behaving moderately among other steels[35]. The chemical compositions of the materials can induce different melting pool behavior, such as thermal gradient and solidification rate which can further affect their printability. The target is to achieve a quality printing for good mechanical properties.

Figure 2-7 Correlation between lack of fusion (LF) and (a) macro-porosity for Ti-6Al-4V, IN 718, SS 316, carbon steel and SS 316 and (b) linear heat input for six different alloys with a constant layer thickness of 0.38 mm[35].

Figure 2-8 Composition change in wt% of the most volatile elements due to vaporization for five alloys [35]

2.2.7.3 Numerical method

Experimental method is the most widely used method for process optimization, however numerical is also an important method for process development and optimization owing to its versatility in evaluating numerous parameters [51]. Tang et al. [52] investigated the physical mechanisms of porosity formation by varying laser power and scanning speed in selective laser melting using DEM (discrete element method) and CFD (computational fluid dynamics). Due to the computational cost of such mesoscopic model using fine mesh grid size to resolve the particle size only single track was studied. Ma et al. designed a 10 factors, 16 runs numerical FEM experiment for a single track fusion in laser powder bed fusion of Inconel 625 metal powder[53]. Krol et al. [54] proposed an efficient procedure to prioritize influential process parameters qualitatively by means of FEM before implementing quantitative study.

2.2.7.4 AI/Machining Learning

As a new data based method, machining learning is gaining its attraction as an easy and systematic method for process development with more focusing on process parameters interactions[38, 55, 56]. The output could be anything such as density, porosity, dimensional accuracy, shrinkage ratio, surface roughness, tensile strength and build time, etc. [56]. The only limitation is that relatively large amount of data is needed. For that FEM-AI combined method can be used[38].

2.3 AM Built Structural Steels for Marine and Offshore Applications

In this section the AMed steels, stainless steels, high strength low alloy steel and EH36 steel will be reviewed. Steels such as maraging steel possess high strength and toughness showing great potential in M&O industry.

Stainless steel and high strength low alloy steel that possess higher strength or corrosion resistance can be used for constructing special components such as gas transport pipeline system. EH36 steel is a common ship structural steel that is widely used to build ship hull and offshore structures. The mechanical properties mainly in strength and toughness of AM built metals through SLM or DLD are reviewed and results are compared with conventional manufactured equivalent or standards. The review eventually demonstrates the suitability of the AM built materials for M&O applications. The relationship between mechanical properties including tensile, toughness, fatigue and microstructure, process parameters will be reviewed and discussed in the later part. Since most structural materials fail by fatigue, a lack of investigation in fatigue and overall performance of SLM/DLD processed EH36 steel has motivated this Ph.D work.

2.3.1 AM Built Steels

The mechanical properties of ultimate tensile strength, yield strength, elongation and charpy impact absorbed energy of AM built vs conventional manufactured steels are listed in Table 2-3. Table 2-4 lists the hardness of some of the AMed steels. From the table tensile strength and hardness of AMed steel materials are usually superior to conventional manufactured equivalent but ductility is lower. Charpy impact absorbed energy of AM built materials are usually lower than conventional manufactured equivalent that could root from the lower ductility. AM built materials usually shows strong anisotropy along building orientations such as higher strength or sometime

in higher ductility in horizontal directions but lower in vertical directions. Reasons could be linked to their microstructure and hence process optimization that will be discussed in the process-microstructure-mechanical properties relationship section. Some results on Ti, Al alloys and Inconel are also listed in the table which are usually adopted by M&O for their special applications. But the focus for this work is on steels.

Table 2-3 Mechanical properties of AM built metals

Material	AM process	UTS (MPa)	σ_y (MPa)	Elongation (%)	Impact energy (J)
316L[16]	SLM	594	487	49	103
316L[57]	SLM	---	---	---	59 (as-built)
316L[16]	HIP at RT/200°C	570/450	220/160	54/42	---
316L [16]	Design criteria	525/415	220/135	45/-	120
316L(parallel)[58]	LMD	639	558	21	---
316L(vertical)[58]	LMD	536	352	46	---
316L[58]	As-annealed rod	586	241	50	---
Maraging steel [59]	SLM				36.3(as-built) 10.1 (after aging)
Maraging steel [59]	Conventional				18(after aging)
Maraging steel_l[60]	SLM	1260±79	768±29	13.9±2	103(K _q ,MPa.m ^{0.5})
Maraging steel_ver[60]	SLM	1324±51	825±96	14±1.5	98.4(K _q ,MPa.m ^{0.5})
Maraging steel[60]	Conventional	1000-1170	760-895	6-15	---

Maraging steel_vertical[60]	SLM+aging	2216±156	1953±87	3.1±0.4	75.6(K _{IC} ,MPa.m ^{0.5})
Maraging steel_parallel[60]	SLM+aging	2088±190	1833±65	3.2±0.6	70.2(K _{IC} ,MPa.m ^{0.5})
Maraging steel[60]	Conventional+aging	1916-2050	1865- 2000	6-8	~70-85
H13 deposited on Cu substrate[61]	DMD	673	---	---	67.12
Inconel 625 [62]	LRM	---	---	---	49
Inconel 718 [63]	SLM	1051	807	21.9	83.8
Ti6Al4V [57]	SLM	---	---	---	11.5
Ti6Al4V [57]	Investment casting	---	---	---	15
AlSi10Mg [64]	SLM	412	242	6.34	2.5
AlSi10Mg [65]	SLM	391	---	5.55	3.94
A360T6 [64]	Conventional	330-365	285-330	3-5	2.5-3.0
AISI 4140(horizontal)[42]	SLM	1535.3	1373.6	13.8	34.7(-18°C)
AISI 4140(vertical)[42]	SLM	1446.5	1289.0	12.3	32.1(-18°C)
AISI 4140[42]	Wrought	1227.9	1141.9	14.6	34.6(-18°C)
AISI 4140[19]	LENS	360±170	235±75	2.3±1.5	---
AISI 4140[19]	Wrought	720	655	4	---
AISI 4340[18]	DMD + stress relief_600°C	1398.65		1.665	
AISI 4340[18]	Annelaed_844°C	745		22	
EH36[66]	Quenched_1000°C	906	795	---	80(Quenched@950°C)

Table 2-4 Hardness and elastic modulus of AMed steels

Material	Process	VHN	E(GPa)
316L[58]	LMD	331-427	
316L[58]	Cold-rolled-stated plate (theoretical)	212	
AISI 4140[67]	LENS	500-930	
SAE 4140[68]	LENS	311-361	
SAE 4140[69]	Quenched martensite	674	
	Q&T_200°C	636	
	Q&T_600°C	386	
	Bainitic structure	330	
	Ferrite-fine pearlite	270	
	Ferrite-coarse pearlite	199	
AISI 4140(horizontal)[42]	SLM	424	
AISI 4140(vertical)[42]	SLM	438	
AISI 4140[42]	Wrought	361	
AISI 1040[69]	Quenched	658	
	Q&T_200°C	575	
	Q&T_600°C	282	
	Bainitic structure	238	
	Ferrite-fine pearlite	208	
	Ferrite-coarse pearlite	178	
AISI 4340[18]	DMD + stress relief_600°C		
	1 st layer	6.391 (H _{IT})	223.887
	2 nd layer	6.929(H _{IT})	206.607

	3 rd layer	7.076(H _{IT})	188.394
AISI 4340[18]	DMD		
	1 st layer	6.845 (H _{IT})	218.292
	2 nd layer	7.666(H _{IT})	210.223
	3 rd layer	8.247(H _{IT})	284.262
AISI 4340[18]	Annealed_844°C	228	190-210
EH36[66]	Quenched_1000°C	313	

2.3.2 AM Built Stainless Steels

Various stainless steels have been investigated through AM processes including 316L, 17-4PH, 15-5PH, 904L, etc. Among them 316L is most studied for its wider applications due to its superior corrosion resistance and mechanical properties. Comparison makes it outstand from other AMed steels in the high strength and relatively good ductility. Charpy and fatigue properties are most close to conventional equivalent among others showing a large potential for direct application. Those are attributed to the solidified microstructure as illustrated by several studies. Zhong et al. has achieved a high densified 316L, >99.8% through SLM process and attributed the high yield strength of SLM processed 316L to the fine intracellular structures reinforced by nano-sized SiO₂ particles without losing its ductility[16]. Even though some un-overlapped region in the size of tens of micron to 150 µm surrounding melting pool region is believed to act as the crack initiation site during tensile loading, the tensile properties seem rather tolerant to the defect and not affected from a large aspect. The chemical composition of the as-built 316L didn't change significantly compared to the powder composition. They suggested a possible remediation for process optimization such as to change the scanning parameters but concluded that it is not possible to get

the porosity to be totally eliminated. They also concluded the possibility of manufacturing 316L with properties suitable for ITER first wall panels.

Zhang et al. also achieved high yield (558 MPa) and UTS strength (639 MPa) of LMD processed 316L in the horizontal direction which is again being attributed to the fine dendrite structure resulting from the rapid cooling[58]. However the inferior strength ($\sigma_y = 352\text{MPa}$) along vertical direction was being attributed to oxygen pick up as well as the easily fractured inter-bonding layer. In a contrast the yet higher elongation achieved in vertical direction (46%) than horizontal direction is claimed due to elongated dendrite along building direction that hold better ductility along its growth direction which is also evidenced by the denser and deeper dimples along that direction from the fractured surface. Through their study they also concluded the more significant effect of scanning speed (from 2 mm/s to 10 mm/s) to microstructure morphology, hardness, yield strength, tensile strength and elongation than laser power does (600 W – 1400 W) in the way that temperature gradient and solidification rate is more sensitive to scanning speed. Meanwhile they also suggested low laser power since high laser power deteriorates material properties resulting in undesired grain coarsening and wasting of energy.

2.3.3 AM Built Low Alloy, High Strength Steels

There is limited research work on low alloy high strength steel until now mostly because of their application and the less profit as compared with other high valued metals such as Ti and the mold making tool steels that can utilize AM's outstanding geometric flexibility in building complex shapes for conformal cooling channels and other circumstances. However there are a few

initiations being taken on AISI 4140, AISI 4340 alloys via SLM and DLD processes. The results are largely scattered and mostly post heat treatment is suggested for its further application.

Kadiri et al. [70] and Amano et al. [68] reported the hardness of LENS processed AISI/SAE 4140 low alloy steel but with no tensile properties being tested. In their studies the hardness varies from HV500 – 900 (varies within 12 mm building height) and HV311 – 361 (varies within 20 mm in building direction) respectively that root from the different cooling rates. Microstructure varies from different process parameters and in-process thermal history along the building. Both of them have reported the hardness variations along the building direction which assures the large influences of in-process thermal history to the material properties that may affect the direct application of the as-build part.

However Wang et al. reported a near full dense fabrication of AISI 4140 low alloy steel through powder bed fusion (PBF-L) process with comparable mechanical properties to wrought grade[42]. And they argued that the excellent mechanical properties (tensile strength and Charpy V-notch impact toughness) was due to more cleanness of AM grade AISI 4140 (with oxide only) than wrought grade in the sense of inclusion (oxide and sulfide) because they believed the inclusion associated fracture was the main failure mode as evidenced from the fractured tensile samples. They also claimed that a much finer martensite lath and bainite mixed microstructure determined the high strength compared with that of wrought 4140 steel. However the ductility is slightly lower than wrought 4140 steel and the author suggested the necessary work on post-weld heat treatment and process optimization. They concluded the achieved excellent mechanical properties of AISI

4140 steel make powder bed fusion process feasible for industrial production showing great potential in the area of aerospace, automobile and machinery industries.

Sun et al. achieved a higher tensile strength in DMD built AISI 4340 low alloy steel (after stress relief) with a much lower elongation compared with annealed grade[18]. The high strength was claimed due to fine microstructure and solid strengthening effect resulting from rapid solidification process and the low ductility was due to high porosity level (3.3%). They claimed that a few percent of porosity would not affect strength, stiffness, large stress concentration or stress redistribution but behaves critical to fatigue (micron sized pore) and ductility that was in agreement with cast steel as the void nucleation stage was bypassed. A heat effect from the subsequent deposition decreased the previous layers' residual stress and nano-hardness. And the rapid solidification process resulted in a decreased ferrite recrystallization temperature and M_s (Martensite start temperature) which was then increased in the stress relieved samples. They suggested changing of scanning strategy to reduce porosity so as to improve properties because the interlayer de-bonding may play detrimental effect to material behavior, and an appropriate heat treatment to tailor the mechanical properties (increased ductility, reduced residual stress, lowered elastic modulus and hardness, etc.). Kim et al. devoted the inferior UTS of LENS processed AISI 4140 low alloy steel (360 ± 170) to the lack of fusion, carbide precipitation and oxide particles inclusions that may act as the crack initiation site for cleavage fracture and premature failure [19].

Based on above study, the results for low alloy high strength steels vary from process to process and mostly the lack of fusion issue needs to be resolved through process optimization to achieve sound properties. A post heat treat could be necessary.

2.3.4 AM Built A131 EH36 steel

EH36 is a type of commonly used ship structural steel that is conventionally manufactured by rolling. The process is to refine grain for targeted mechanical properties. The standard grade of EH36 steel is not for quench and tempering (Q&T) where Q&T grades are categorized by grades EH40, FH32, FH36, and FH40. However they may possess the same chemical composition with the only difference of being processed through different process procedures that is controlled rolling followed by either accelerated cooling or quench & temper. Meanwhile there is reported data for Q&T EH36 steel that can be used for comparison study as both of them possess martensitic structure.

To develop EH36 steel through AM process one of the concerns is the printability – whether or not it can be formed in good integrity. Preliminary results have shown a positive result with the building height up to 10 mm without obvious cracking and delamination[25]. However for larger building height it remains unknown but important when building large scale of components, which would be the 2nd phase task. By using the similar concept from welding owing to two their similarity in processes with only difference in scale, that is, the weldability to assess the materials fusing ability. Steel weldability is assessed by hardenability as they are in inverse proportional relationship. Hardenability is quantified by carbon equivalent (CE) which takes carbon and other alloying element into account as stated in the equation $CE = C + Mn/6 + (Cr + Mo + V)/5 + (Ni + Cu)/15$ [1], where C, Mn, Cr, Mo, V, Ni and Cu all can contribute to the hardenability of steels. The calculated CE value of EH36 steel is about 0.38 which represents very good weldability.

A preliminary work on SLMed EH36 steel has some results on microstructure, tensile and charpy impact properties conducted by Marine and Offshore industry partner who attempted to build node structures using AM technique[25]. The both high strength (over 1000 MPa in UTS) and low toughness (charpy impact energy) being achieved was attributed to obtained martensitic structure from the rapid solidification process (a quenched phase) and ASTM standard was breached for charpy impact energy even though strength is higher than quenched equivalent [66]. The results could be a combined effect of fine lath/block/packet grain size, dislocation, solid solution strengthening, precipitation strengthening and internal pores, etc. A post heat treatment conducted at 650°C recovered certain toughness without losing too much strength. However this post processing stage adds on complexity of downstream processing and is rather expensive that deviates from the purpose of using AM technique. On the other hand the processing chamber of SLM is limited to the size of a few hundred mm, which renders a rethink of an alternative AM process that can build large scale and complex shape of Marine structures. One option is the DLD process which is known for its higher productivity and capability in building large size of parts amongst other AM processes. As a new trial, the properties of DLD built EH36 steel samples will be evaluated against ASTM standards (through rolling) and results will be present in this paper. Meanwhile fatigue performance will be evaluated for both processes. To do so is because SLM gives better geometrical and mechanical property control.

2.3.5 Applications of AMed Steels in M&O Industry

There are some potential applications of AMed components to be adopted by M&O industry benefiting from its unique priorities in producing complex shape of parts in one step. Figure 2-9

shows the column to pontoon structure which is one typical structure that can be potentially produced by AM process. The goodness of using AM for the structure is AM is more cost and time efficient than conventional casting process. As the AM process is not ready for mass production, how to increase the productivity is a key for industrialization.

Figure 2-9 Column to pontoon structure

2.4 Microstructural Evolutions of AM Built Steels

The rapid solidification process with high cooling rate creates unique structures giving unique properties for AMed metals. The mechanism of structure forming is studied in the first position as an understanding of process influence on microstructure formation. Herein the thermal history, theory of rapid solidification, a resultant macro-micro-nano hierarchical structures will be discussed in-depth.

2.4.1 Thermal History of AM Processes

Rapid solidification is defined as a solidification process with cooling rate above 10^4 K/s. It normally accompanies with directional solidification coupled with high temperature gradient (G) and solidification rate (R). This very high temperature gradient limits the distance of solute redistribution resulting in fine cellular/dendritic structures that differs it from conventional manufacturing processes such as laser welding and casting, etc.[2].

However there is much common ground with conventional laser cladding process where the melting pool is created by the high energy laser spot with temperature highest at the center and lowest near the boundaries. This generates a large temperature gradient both near the air-liquid interface and inside molten metal. The resultant Marangoni effect resulted from the surface tension gradient near the air-liquid interface could generate strong convection flow within the melting pool as proved by plenty of research[71]. That can accelerate circulation flow within the melting pool and homogenize the chemical concentration which can further influence the temperature gradient and solidification rate. Meanwhile the materials related properties such as viscosity, etc. will affect the convection flow inside the melting pool.

In AM the thermal history within the melting pool determines the melting pool behavior and final microstructures so as to the mechanical properties. It largely depends on temperature gradient and solidification rate, while temperature gradient can affect heat transfer and fluid flow as mentioned above (Marangoni flow driven by surface tension gradient) within the melting pool so as to the size and shape of the melting pool and the solute concentration as well. Temperature gradient is determined by laser power, scanning speed, spot size, laser energy distribution, etc. Solidification rate can be affected by alloy composition, concentration and materials thermal physical properties.

2.4.2 Macro-scale Features

Melting pool, pores and inclusions characterizes macro-scale structure.

Melting Pool and Boundary

As a basic constitutional unit, melting pool plays significant role in bulk material properties. A Gaussian distribution type of laser energy created a half circle shape of melting pool where the size varies from around 100 μm for SLM to a few mm for DLD process. The depth and width characterize the melting pool morphology which is closely related to process parameters. Ma et al. has studied the energy input (E in J/mm^2 by varying laser power and scanning speed) effect to the metallurgical behavior of 316L in two processes: selective laser melting and laser cladding deposition[20]. The depth-to-width ratio n increases with E for SLM process but decreases for LMD process. The relativity of E to microstructure and properties will be discussed in the following part.

There are many descriptions on melting pool that are formed under different conditions. Thijs et al. defined three different zones that distinguished by coarse MP, fine MP and HAZ in SLM built AlSi10Mg and claimed it was due to different cooling rate across the melting pool [72]. Tan et al. found equiaxed grains at centre of the melting pool due to low G/R ratio in SLM built maraging steel which may not happen homogenously [73]. Wang et al. disclosed the stray grains formation within dendrite grains within the melting pool both experimentally and numerically through laser surface-melted single crystal superalloy [74]. Such kinds of melting pool behavior are often seen

in traditional laser welding, e.g. Amrei et al. defined similar zones like columnar, tempered and fine martensite across the melting pool for multipass welded 13Cr4Ni and attributed it to different heat cycles[75]. It is deduced that the AM process is somewhat very similar to traditional laser welding process but only differed in scale and regarded as very locally melting.

The phenomena of melting pool boundary detachment are often observed in AMed metals. In most cases they are believed to be the crack initiation point that is summarized based on the observation of fractured surface using SEM [16, 76-78]. Often MPB is coexisting with defects likely lack of fusion, oxides and precipitates and it is deduced that MPB is the favorable place for defects to form based on the analysis in the reports[18]. Care must be taken when dealing with printing and not to deteriorate melting pool boundaries. Again melting pool characteristic is a result of complex thermal history within it and highly related to process parameters.

2.4.3 Micro-scale Features

2.4.3.1 Grain Morphology and Size

During SLM process a cooling rate of $10^3 \sim 10^6$ K/s can be achieved [79, 80] and DED of $10 \sim 10^5$ K/s [20, 58, 68, 81]. The instantaneous melting and solidification process is illustrated in Figure 2-10 where temperature can shoot to nearly 2000°C and drop back to 200 -300°C within a period of less than 1 s for the 1st layer and stabilize around 500 - 600°C for the subsequent several building layers (7 – 8 layers)[67]. From the Figure 2-10 it can be seen that the heat effect of the following building layers to the below 3 - 4 layers is prominent which may generate a heat affected zone with temperature that can reaches over 1000°C. With high temperature gradient, up to 10^6 K/m,

new metastable phases and changes in microstructure feature (mostly in scale) could be expected or even amorphous phase during this non-equilibrium solidification process[2].

Figure 2-10 (a) The thermal history of the single-walled deposit calculated at the mid-point of each deposited layer by finite element simulations using a three dimensional model that was developed as a subroutine in the SYSWELD software tool[13], and (b) the minimum temperature of C_t (the minimum temperature of the cycle) cycle immediately following the last melting cycle for each layer in comparison with the martensitic starting temperature (350°C)[70].

The higher cooling rate favors finer grain structures and enhances the limit of solid solute being trapped in the lattice. The distance of solute redistribution or called micro-segregation is shortened and mainly accumulated at sub-grain boundaries. Ma et al. studied the metallurgical behavior of both SLM and LMD processed 316L and results showed that cooling rate slowed down when E became higher resulting in coarser grains as well as larger grain aspect ratio (grain length L /grain width λ) for both processes[20]. The typical cellular or dendritic grains from AM process are around $1 \pm 0.5 \mu\text{m}$ as reported resulted from the rapid cooling rate of $10^3 \sim 10^6 \text{ K/s}$. With substrate acting as a heat sink, grains grow from bottom of the melting pool to the centre of it following the heat dissipation direction. It could be an epitaxial growing as found in many different kinds of alloy and steels[72]. Overall grains can vary from planar, to cellular, cellular/dendritic, columnar dendritic or equiaxed as reported[73]. This is elucidated by most authors from the theory of solidification that constitutional supercooling determines the morphology of liquid/solid front[82]. As stated by Yan et al. constitutional supercooling accompanied solute redistribution that caused the temperature at solidification front cooler than liquidus temperature. The degree of constitutional supercooling is determined by the ratio of G/R [82]. With a schematic relationship between constitutional undercooling and grain morphology shown in Figure 2-11 [83], the grain evolution can be stated in the followings. Near the melting pool boundary to a distance of a few microns temperature gradient is the highest and solidification rate is almost zero where planar crystals dominate. Along the way to MPB centre temperature gradient decreases gradually with solidification rate increases to the maximum (equals scanning speed). Grains may evolve into cellular or dendritic structure. Even equiaxed grains could be found near the surface of the melting pool provided they are still remained and not being remelted. And the fineness of grains is determined by cooling rate which is taken as $G \times R$. Higher the cooling rate, finer the grains.

Figure 2-11 Schematic relationship between constitutional supercooling and grain morphology [83]

Above is based on the solute redistribution theory, however there was a recent report in a SLM built 316L showing no micro or even nano-segregation across the cellular grain boundaries [84]. This was supported by the results from examination of chemical composition using EDS under TEM. It was elucidated by a very high cooling rate of 1.7×10^6 °C/s achieved by using a pulsed laser with exposure time of 80 μ s and a spot size of 75 μ m at a very fast scanning speed of 5000 mm/s. The author attributed this solidification behavior to this very rapid cooling rate together with formation of Mn, Si oxide that avoids their segregation to the cellular grain boundaries. It seems that the constitutional supercooling theory can't be used here to explain the non-segregation solidification behavior. A new theory is needed to explain the non-equilibrium phenomenon.

The strong anisotropy characterized by elongated cellular/dendritic grains is dictated due to the rapid solidification process together with a range of variety of morphologies and sizes of resulted

grains which illustrate the complexity of structure formation during the rapid solidification process. Coarse, equiaxed and columnar grains were observed at different locations of melting pool and inter melting track areas as disclosed in SLM built maraging steel where the complex local heat cycle played an important role to the grain formation[73]. Occasionally coarsened grain zone could be observed within one melting pool which was elucidated due to slower local cooling rate that was affected by heat accumulation from adjacent layers [73]. Meanwhile heat affected zone coupled with grains evolution undergoing recrystallization, grain growth or precipitation, etc. is expected. The metastable nature and high concentration of dislocation of the grains may favor or accelerate these changes[72]. Complexity in phase composition could be expected also in regard with the complex local heat flow. Kadiri et al. has managed to mark the possible phases within laser deposited AISI 4140 steel[70]. To a large extent the phases identified covered from martensite, bainite, ferrite, carbide to some metastable new phases which are characterized with OM, SEM and TEM and all these were not observable from conventional processed materials. All these anisotropy and local inhomogeneity can cause directional mechanical properties. Detailed discussion will be followed in the next section.

2.4.3.2 Pores

Voids in AMed metals consist mainly of lack of fusion and small spherical pores. Lack of fusion usually locates at inter-building layers formed due to insufficient energy penetration or sometime to oxide. They are usually coexisting with unmelted particles. Small spherical pores are often dispersed within the matrix that is usually gas trap which come from raw powder, or process induced gas evaporation that can't escape from the molten metal. Among them lack of fusion is most concerned due to their strong stress concentration effect that can initiate crack and pre-mature

failure. Sun et al. evaluated the lack of fusion zone in laser-deposited AISI 4140 steel on their synergistic oxidation effect for subsequent crack failure under 3 point bending and it could significantly degrade fatigue properties associated with increasingly intergranular fracture features and secondary grain boundary cracking [18]. Mostly lack of fusion is process related and can be minimized through process optimization but completely avoiding is difficult[85]. In a pre-laid powder bed laser cladding processed H13 tool steel, a minimum 2.3% of porosity was achieved and the reason was claimed mainly due to spattering and balling[86]. Beal et al. gave such conclusion by evaluating 4 different types of scanning strategies using high power (550W) Nd:YAG laser (1.06 μm). They also suggested possible solutions: using bimodal distribution powder, preheating powder bed or use pressurized shield gas such as N_2 or He to reduce plasma and spatter. However based on quite some researcher's work, the density of selective laser melting and laser metal deposition processed metals can reach near full density that is above 99% or 99.5% presenting closed and more regular shape of pores [42, 60, 87].

2.4.3.3 Inclusions

The inclusions found in AM built steels are usually oxides. Thijs et al. attributed the inclusion (10~100 μm) found in SLM processed maraging steel to be oxide formed by surface oxidation of molten melt that left in between building layers with mostly irregular and elongated shapes, which may also be trapped inside melting pool. They also concluded that the oxide (Al_2O_3 and Ti_3O_5) intends to form at periphery of melting pool due to the relatively low temperature at MPB[88]. Stanford et al. also found plates oxide inclusion (some >100 μm in length) with composition of $\text{TiO}_2\text{:Al}_2\text{O}_3$ deposited at interfacial building layer of a DMLS processed maraging steel[89]. However Wang et al. claimed a cleaner build of SLM processed 4140 low alloy steel than wrought

grade in the sense of lesser oxide and no sulfide inclusions which was believed as the reason for the higher tensile and CVN impact properties[42].

2.4.4 Nano-scale Features

Micro/nano-segregation and precipitation will be discussed in this section. As mentioned above: from conventional theory of alloy solidification segregation accompanies solidification, however microsegregation is not identified within some materials which put forward a task that new theory needs to be developed to explain the rapid solidification behavior that is not coupled with micro-/nano-segregation. This puts forward a need to study the segregation behavior and its forming mechanism which may help in understanding the rapid solidification process.

Segregation is found and reported in various SLM or LMD processed metal as it has been said that even for the rapid solidification process with as high as 10^6 K/s cooling rate, it is difficult to trap all solute in the lattice[81]. And in these circumstances microsegregation is normally found at interdendritic or intercellular layers for SLM and DLD processed metals where enriched solute from microsegregation renders precipitation energetically favorable[81]. Saidei et al. verified the Mo segregation to the cellular subgrain boundaries with a diameter of 0.5 μm using STEM and EDS in laser melted 316L[90].

A mechanism study on microsegregation and precipitation behaviour was conducted by Jäggle et al. who defined the precipitation behavior into two types: precipitation upon deposition (intermetallics or precipitates within SLM processed Inconel 738LC and Scalmaooy), precipitation through intrinsic heat treatment (IHT) (Ti clusters in maraging steel processed by LMD induced

by heat effect of subsequent deposition)[81]. But they didn't conduct the precipitate examination in SLM processed maraging steel since they rather expected no precipitation happened during this very rapid cooling process but suggested that it could be analyzed using statistical of APT datasets. However massive nanoprecipitates with the size of 5 -20 nm were indeed identified within the columnar martensite matrix (200 nm) of as-fabricated maraging steel by Tan et al. by using TEM examination. They were regarded as precipitation during intrinsic heat treatment of SLM process[73]. The micro-/nano-segregation behavior is closely related to the rapid solidification process. Microsegregation induced precipitates could be undesired compared with the desired (within grain precipitates using normal aging process). Microsegregation may affect mechanical properties from aspects.

2.5 Process-Microstructure-Property Relationship

As known properties are closely related to microstructures and hence process parameters and their relationship has been correlated over most of the studies[16]. Sometimes due to limited resources specimen built at optimized process parameters are evaluated for their specific properties. Among them tensile and hardness have been studied extensively but limited on charpy and fatigue performance. There are experimental, numerical as well as newly emerging AI method for the process-microstructure-property analysis, each has its pros and cons. A brief review on the methods and methodologies are discussed below. More detailed review will be focusing on experimental studies on tensile, hardness, toughness and fatigue with process related microstructure and densification correlated.

2.5.1 Methods and Methodologies of Process-Microstructure-Property Study

The microstructure determines macroscopic properties such as mechanical properties, while microstructure can be tuned through process parameter adjustment. To understand their inter-relationship becomes super important to understand the mechanistic behind as well as to achieve good properties. The process-microstructure-property relationship has been studied both experimentally[91-93] and numerically[93-97], while mostly by experimental method due to its robustness. Another important branch is numerical methods. The complex and effort-intensive relationship of process-microstructure-properties can be easily conducted using numerical method. Machine learning/AI prompted data-driven material design[97, 98].

2.5.2 Densification

As properties like ductility, toughness and fatigue are sensitive to porosity, it is crucial to build an AM part with high enough density. As known near full density can be achieved by AM process but complete prevention of porosities is difficult. Casalino et al. studied the dependence of mechanical properties on density for maraging steel using SLM[99]. The research indicated the correlation among UTS, hardness, elongation and density were positive. A proportional relationship also existed between density and energy input (J/mm^2). Based on their statistical analysis there was a significant interaction among process related scan speed, laser power and density, hardness and UTS as shown in the contour plot in Figure 2-12.

Figure 2-12 Contour plots for relative density (%) with respect to the process parameters[99]

2.5.3 Tensile Properties

Both yield strength and ultimate tensile strength of AMed metals are excellent that can compare or in most cases higher than that of conventional manufactured[100]. This is mainly attributed to its finer grain or sub-grain structures resulted from rapid cooling rate[16]. Ductility is low especially for Ti and Al alloys[100]. This is explained by the deterioration effect of defects such as lack of fusion voids and other defects within AMed parts acting as stress concentration and promoted pre-mature failure[78], however 316L is the only material that can achieve both strength and ductility. Zhong et al. attributed the superior yield strength to the intracellular segregation network with randomly distributed nano-oxide inclusions acting as dispersive strengthening that increased the defect tolerance and yield strength of SLM processed 316L without sacrificing ductility[16]. They also mentioned strengthening by pinning effect of dislocation introduced by Mo segregation to the intercellular boundaries. Qiu et al. discovered that nano-needle/cellular

structures coupled with dislocation within and at the boundary of nano-needle/cellular grains was crucial to the superior tensile properties for SLM processed 316L[84].

Mechanical properties of AMed metals behave anisotropic with some properties could fall below standards[101]. The reasons could be linked to their microstructure features such as lack of fusion (sometime due to oxide formed at interlayers) and the strong anisotropy, etc. In most cases the strength and ductility in vertical direction is lower than that in other directions. The large and elongated lack of fusion void (mostly extended along horizontal direction) deteriorates both strength and ductility of vertical direction more than that in other directions. Casati et al. claimed that the defects such as pores and unmelted powders with surfaces orthogonal to loading direction is the reason for lower mechanical response of vertical direction[78]. He also claimed that partially melted powder are believed to be the reason for the scattered ductility in the vertical direction and this could also be responsible for the lower vertical strength due to reduced effective cross-section, however there is research demonstrating much higher ductility in vertical direction instead.

Smith et al. achieved a higher ductility in vertical direction (50%) with Inconel 718 using SLM (after heat treat with a porosity level of 0.15%)[102]. It was observed the mean free path of dislocations moving along the loading direction was the longest (less work hardening accumulated in the material) in the vertical sample grain structure characterized with weak $\langle 001 \rangle$ texture fiber and elongated grain structure. This was rationalized to be the reason for low yield stress in that direction as well but it needed to be confirmed by further research. Similar higher ductility in vertical directions were achieved by other researchers as well such as SLM processed 316L reported by Song et al. that a 50% elongation was achieved that can exceed wrought standard, 40%

in American Society for Metals (ASM) handbook[100, 103]. Zhang et al. attributed the higher elongation along vertical direction to the elongated dendrite that held better ductility along the growth orientation[58].

As stated above the higher ductility in vertical direction is probably a consequence of elongated grain that allows more dislocation movement, however it could be balanced out by the defects which may substantially lower the ductility. So the high ductility scenario may happen only when there are no detrimental defects influencing its properties in that direction that is with a higher density and this is especially true for those defect sensitive materials. Moreover the texture in the building direction could be an additional effect for this[104]. Again both of them may be responsible to the yield stress drop in vertical direction [105].

A worse ductility scenario in horizontal direction is reported by Carroll et al.[101]. They achieved an elongation of 14% along vertical direction in DED processed Ti6Al4V which is higher than 11% in horizontal direction. The lowered elongation was attributed to the prior β grain morphology and the presence of α grain boundary that serves as a Mode I opening failure.

Despite of the superior achievable strength of AM built parts there are studies showing that a further homogenization could bring AMed parts to another level. Tan et al. compared the tensile strength and ductility of SLM processed maraging steel and concluded that an after process full solution treatment followed by age hardening can render the printed sample reaching standard wrought level superior than the direct age-hardened sample[73].

Even though the relationship between tensile properties and microstructure has been studied extensively, there are still unknown whereas they may affect the strength as well. Some of those are intermetallics and excess solid solution being trapped in lattice. The micro-segregation or intrinsic heat treatment induced intermetallic and precipitates located at inter-cellular/dendritic layer has been reported. Their effect to bulk material strength is not well understood[2]. Still the effect of over trapped solute element within lattice structure due to rapid cooling is not well elucidated yet.

Again the tensile properties are closely related to process. Casalino et al. proved through experimental study that the UTS, hardness and elongation of SLM processed maraging steel obeyed a strict proportional relationship with density which was nothing but process related[99]. Ma et al. disclosed the metallurgical behavior of both SLM and LMD processed 316L and showed that for both of the processes microhardness, UTS and yield strength showed a general proportional or reciprocally proportional trend with ED, but not that straightforward for ductility where ductility in SLM process increased with ED but seemed insensitive to the changes of energy in LCD process[20].

2.5.4 Hardness

The hardness of the AM built parts is normally higher than conventional manufactured equivalent such as cast and rolling. This is due to the finer grain or sub-grain structures that resulted from the rapid cooling rate and possible higher dislocation density and solid solution strengthening effect, etc. And there is scenario that the nano-oxide found dispersed throughout the matrix can act as precipitation strengthening effect that can affect hardness as well[16].

But the variations of hardness along build direction are observed or in some extremes even within one melting pool which in most cases is highly related to microstructure inhomogeneity. Zhong et al. observed the hardness fluctuation along building direction of SLM processed 316L and attributed it to more nano-oxide inclusions at bottom of the parts due to higher oxygen level at the beginning of the printing job[16]. Kadiri et al. measured the microhardness of LENS processed AISI 4140 steel and cyclic variations of hardness values were observed within the building layers (minimum value at fusion line) and a variation along build direction[67]. It was deduced from tempering effect from the heat of repeated scanning and the heat accumulated on the substrate/processed materials which may reduce the cooling rate of upper part of the materials and resulted in coarsened grains with lower hardness value. This is especially important for building a large scale of component where material properties may vary in a wide span from the bottom of the top.

Heat treatment trials have been implemented to the AM built parts and the microstructure inhomogeneity can be easily removed after proper treatment. But mechanical properties deterioration needs to be taken into consideration as most heat treat will coarsen the microstructure in due.

2.5.5 Toughness

Toughness is the scale of measurement to material embrittlement. In material science and metallurgy, toughness is the ability of a material to absorb energy and plastically deform without fracturing. It requires a balance of strength and ductility and usually measured in a way of charpy

impact using a small size of specimen. It can also be measured in the way of fracture toughness K_{IC} (as stress intensity factor in linear elastic fracture toughness) and J_{IC} (elastic plastic fracture toughness). Steel specifically ferritic steels (BCC, body centered cubic structure) may undergo brittle fracture at low temperature. That is a transition from ductile to brittle failure so it is good for steels for ship construction to operate above this temperature namely ductile to brittle transition temperature T_0 (DBTT) so that catastrophic failure won't happen.

Table 2-3 lists the charpy impact energy (and tensile properties) of AM metals against that from conventional manufactured. The toughness of AM built metals is normally lower than conventional manufactured metals such as forging and rolling. As grain refinement can do good to both strength and toughness, the lowered toughness of AMed metal is said due to the defects like porosity, oxygen pick up or meta-stable phases, etc. The reasons are toughness is sensitive to ductility and ductility is sensitive to porosity, etc.[18].

2.5.6 Fatigue Properties

The fatigue property of AMed parts is less investigated compared with tensile and AM process parameters. The limited work mainly focuses on high cycle fatigue behavior in the way of S-N curve, that is the stress-strain curve or crack propagation behavior (da/dN vs ΔK). Basically fatigue properties behave sensitive to intrinsic AM pores even in the size of micron level which doesn't show much inferior effect to monotonic tensile strength[18]. As known, fatigue performance is affected by various factors including surface roughness, defects, microstructure, residual stress, sample shape & size, environment and loading, etc. Some of the studies related to AMed parts were summarized in this section.

2.5.6.1 Effect of Surface Roughness, Pores, Residual Stress, Heat Treatment and Building Orientations

Riemer et al. studied the surface finishing, residual stress and internal porosity effect to the fatigue life and fatigue crack growth behavior on SLM processed 316L [106]. It was found the fatigue limit was doubled for turned surface compared with as-built condition. And surface turned specimen after HIPping gained 18% increase in fatigue limit. However residual stress didn't affect the fatigue limit as much as the other does and only a 10% difference was achieved after heat treatment at 650°C. The threshold value ΔK_{th} of HIPped sample is only slightly increased compared with as-built sample and both behaved similar crack propagation rate. However the threshold value of sample in vertical building direction increased by 40% than that of horizontal sample which was attributed to crack path deflection effect as illustrated in Figure 2-13 that larger energy encountered to more deflected crack route. The orientation related fatigue behavior is also dictated in Figure 2-14 [100, 107] for SLM processed Ti6Al4V. The X-direction performs better than Y-direction followed by Z-direction. The fatigue properties are significantly lower (77%) than wrought material. Again this proved that porosity is the key contributor to the reduction in fatigue life. Meanwhile residual stress and surface conditions behaves important to for a better fatigue performance. As seen from the Figure 2-14 [106] for both Ti6Al4V and 316L it seems fatigue life is largely lowered for the as-built samples.

There are few investigations on very high cycle fatigue (VHCF) [108] and low cycle fatigue (LCF) behaviors [109]. The crack initiation location intends to shift towards interior region for a laser additive manufactured Ti6Al4V steel and results show no fatigue limit up to 10^9 cycles [108]. As

for LCF, steady state crack propagation dominates the whole fatigue life and the defects dominated mechanism is not as much as HCF and a different mechanism shall be interpreted[109].

Figure 2-13 Schematic illustrations depicting (preferential) grain orientation and morphology dependent on building direction and heat treatment. Elongated grains in building direction are present in the as-built condition and after heat treatment at 650°C. Closely spaced grain boundaries act as barriers and cause the crack arrest at relatively higher values for DK in (a) due to a more tortuous crack path in comparison to crack growth parallel to the elongated grains illustrated in (b). (c) Following HIP a bimodal microstructure evolved leading to intensified crack–microstructure interactions.[106]

Figure 2-14 Fatigue results of SLM processed (a) Ti6Al4V ($R = -0.2$, $K_t = 1.0$)[110] and (b) 316L stainless steel [100, 107]

2.5.6.2 Fatigue Models

The effect of defects to fatigue life and strength has been studied using conventional El-Haddad model and the results are very similar to conventional manufactured equivalent like casting and forging or sometimes even better. As shown in Figure 2-15 the kitagawa diagram provides a very good picture of trend of fatigue properties to the defect size for AM built Ti6Al4V and is able to explain the large scattering for the different fatigue datasets[23]. However the models described is based on the experimental evidence of without damaging cracks or defects that can decrease fatigue limit (compared to that of unnotched specimen) and on ΔK_{th} dependency of crack size as $\sqrt{area}$ [22]. There are other fatigue related models that give either good or not results which will be discussed in the following sections in “mathematical model” [22, 111] and “numerical studies on fatigue for AMed metals & alloys”. However, these models didn’t consider microstructural effect to the fatigue crack behavior. Instead a multi-stage fatigue model as introduced by McDowell et al. can be a useful microstructural sensitive fatigue model that is proven to bring

microstructure details, such as grain size, porosity, void size and spacing to link them to fatigue behavior[105]. Xue et al. has practiced this model to LENS-processed 316L steel and found the incubation life at un-melted powder was about one-third that of a LENS built sample with pores of similar size[112]. The mechanistic study involving microstructural effect can be easily conducted through FEM by incorporating micro-crack initiation model such as the Tanaka-Mura model[113]. For total fatigue life calculation, multi-scale algorithm by adding in the LEFM Paris law model for crack propagation stage and the stress intensity factor range and angle of crack propagation can be both calculated with ABAQU[114]. More review on numerical fatigue studies were discussed in the following section “numerical studies on fatigue for AMed metals & alloys”.

Figure 2-15 Fatigue limit $\Delta\sigma_w$ of Ti6Al4V as a function of crack $\sqrt{\text{area}}$: (a) $R = 0.1$ and (b) $R = -1$ (gray regions corresponding to the data for conventional processes) [23].

2.5.6.3 Fatigue Failure Mechanism of AMed Metals and Alloys

The fatigue properties of AM built metals is largely dominated by defects including pores, unmelted particles and inclusions, etc. [18, 109]. The anisotropy of fatigue properties are elucidated by defects between building layers and the microstructure related to tortuosity of crack path [60, 115, 116]. Depending on the size and location of the defects fatigue strength and lifetime can be greatly scattered. There is a debate that the effective stress calculated based on effective cross section area could be the reason to influence crack initiation sites[105]. Ductility of materials leads them to less defect sensitive[117, 118]. To achieve comparable fatigue properties with wrought materials post processing including machining, HIPping and heat treatment is necessary and Hipping is the most effective way as it can close internal pores[105, 109].

Leuders et al. studied the fatigue behavior of Ti6Al4V manufactured by SLM and concluded that the pores within the samples had drastic effect to SLM-processed Ti6Al4V in the high cycle regime (HCF)[119]. This was supported by the large difference in fatigue life between as-built, heat treated and HIPped sample failed at 2.7×10^4 , 2.9×10^5 and 2×10^6 respectively and it was concluded that the extension of crack initiation phase could only be achieved by reducing porosity. This led to an improved fatigue limit of 620 MPa tested by staircase method which was comparable to conventionally manufactured Ti6Al4V. The stress relief was said affecting the subsequent crack growth phase only. At higher crack growth rate all curves coincided with the reference material. Similar studies on 17-4PH[120, 121], maraging steel[60], etc. have been conducted in the literature[122]. Large difference between as-built and post processed samples can be found for Ti6Al4V and 17-4PH, etc. metals compared with 316L and maraging steel [60, 106]. It is reasonably deduced that the ductility plays an effect to the fatigue behavior that ductile material shows less crack sensitivity[123].

For metals and alloys the fatigue crack usually initiate from surface or near surface defects such as pores, inclusions, grain boundaries or other material inhomogenities. Without defects, fatigue fails mostly by shear bands generated through intrusion and extrusion from cyclic loading. It is said also that the crack initiation is strongly influenced by their shape and fatigue limit is strongly influenced by the defect size. Once the crack has initiated from the inhomogeneity, inclusions or defects, the size of the crack determines the stress intensification at the crack tip and accordingly the threshold condition[22]. Based on above literature review the pore initiated fatigue failure mode is believed highly related to the sharp corners of the pores/lack of fusions resided inside AMed parts.

Many fatigue failure mechanisms have long been well recognized, such as 2-stage[124], 4-stage[125] and 3-stage multistage fatigue model (MSF) [126] based on the micromechanisms at different stages. McDowell described crack incubation (INC), microstructurally small crack (MSC) and long crack (LC) in the later evolved 3-stage MSF. During the incubation stage, a crack-like damage is nucleated at micronotch. The crack length of MSC covers a few grains mostly 1 to 3, which may extend to 1 mm varied to microstructural inclusion morphology and texture in the matrix[112]. The length for LC is in the range of 10 - 20 microstructural (MS) length scale depending on the amplitude of fatigue loading and the corresponding extent of microplasticity ahead of the crack tip[112]. The MS has been defined by different researchers that may vary with the manufacturing process and formed microstructure[127]. The crack initiation stage including crack incubation and MSC is known as microstructurally sensitive, while LC is microstructurally insensitive. The above mentioned can be further dictated by intrinsic pores entrapped in AMed metallic materials. Whether the conventional crack initiation/propagation mechanism is fulfilled or deviated for the AMed EH36 steel was investigated in this study. The results provided a guideline for the adoption of AMed EH36 steel by marine and offshore industry.

2.5.6.4 Comparison with Conventional Manufactured Counterpart

From the literature review fatigue properties of AM built metals behaves in a similar way to conventional manufactured materials. That is crack is always initiated from surface or subsurface defects like voids or inclusions, etc.[128]. Fatigue limit and crack initiation threshold is comparable with wrought parts given they are machined (remove surface roughness of as-built

parts) and stress relieved (relieve residual stress) or HIPped in some case (to close internal pores especially for defect sensitive material such as Ti6Al4V, etc.)[91]. The classical models such as El-Haddad model, etc. can be used to correlate defects, inclusions and inhomogeneity with fatigue limit[22, 111].

2.5.7 Fractography Analysis

The fractography analysis is conducted to look for the cause of failure. Fractured surface is usually examined to identify origin of crack and the crack propagation pattern that can be used to determine failure mode. For metal materials it is common for crack to initiate from defects like voids, crack, inclusions, and large secondary phases, etc. [19].

Casati et al. examined the fractured surface of the SLM processed maraging steel tensile samples and found ductile failure mode and the relatively large crack originated from adjacent layer tracks[129]. Kim et al. examined the fractured surface of LENS produced AISI 4140 steel tensile samples using SEM and EDS and found three types of defects from fractured surface: large carbide particles, lack of fusion and oxide particle inclusions. All those may act together to lead to cleavage fracture resulting in scattered tensile results [19]. Sun et al. evaluated the defect density using SEM on fractured surface of laser deposited AISI 4340 steel tensile samples and explained the oxidation behavior related to the large lack of fusion region assisted by EDS[18]. Those again were the sites for nucleation and propagation of high temperature oxidation cracks. Zhang et al. examined the fractured surface of fatigue samples from SLM processed 316L using SEM and found the origin of crack always started from surface or near-surface defects and in mixed mode of ductile and brittle failure[21]. Obviously the fractography analysis is crucial to help understand the failure

mechanism of AMed metals which in turn help to improve AM process to achieve better material properties.

2.6 Mathematical Models, Correlation and Validation

2.6.1 Mathematical Models and Correlations

Building mathematical models from experimental data is helpful to investigate the relationship between variables. Typical mathematical equations obtained through curve fitting from raw data is regression model that can predict the values of the variables based on the other. Meanwhile a correlation analysis gives the strength of the linear relationship between two variables.

The effect of process parameters, materials parameters, defects, etc. to the properties and other responses we concerned is an important research topic. As a new manufacturing process, the relationship of different variables for AMed parts are worth study. Works on mathematical models investigated is summarized here and in the next section. Meanwhile, from the literature review the properties of AM built parts are rather unknown and occasionally away from known practice. This is believed strongly associated with its intrinsic unique microstructure, which may lead the results away from conventional counterparts.

Some of the mathematical models established from conventional manufacturing method was examined to AM as proved from some pioneer studies[130, 131]. For example the high yield strength of SLM processed 316L steel agrees well with Hall-Petch relation (shown in equation 2-

1) if the fine subgrain size is used instead of grain size [16, 132, 133]. This attributes the high yield strength of AM 316L to the fine cellular structure resulting from high cooling rate. Meanwhile the mechanical properties of AMed metals scatter. This is

$$\sigma = \sigma_0 + kd^{-1/2} \quad 2-1$$

known to be microstructure related also and can be traced back to grains, solid solution, precipitate, lattice strain, dislocations and other defects. Tan et al. practiced the Orowan relationship in SLM processed maraging steel martensitic structure and calculated results conformed well to experimental one [73]. Ma et al. practiced the Hall-Petch relationship for both SLM and LMD processed 316L and correlated UTS, yield strength, hardness to the grain width[20]. The UTS, yield strength and elongation were fitted valid well against Hall-Petch relationship for both fine grain in SLM and coarse grain in LCD. And 316L produced from SLM is much stronger than LCD but with lower elongation.

A few of fatigue models have also been examined for the AMed metals and alloys, such as the Murakami equation in the form of

$$\sigma_{FL} = 1.43 \frac{HV+120}{(\sqrt{area})^{1/6}} ((1-R)/2)^\alpha (\alpha = 0.226 + Hv \times 10^{-4}) \text{ (for surface crack)} [22, 111] \quad 2-2$$

where σ_{FL} is fatigue limit, Hv is microhardness, $\sqrt{area}$ is the defect size projected to the plane perpendicular to the loading direction, R is stress ratio. The Murakami equation is known giving good prediction of fatigue life by correlating with initiated defect size for the conventional

manufactured parts. However the transferability to AM process is not so successful as stated by Leuders et al. for the SLMed TiAl6V4[134], however rather good applicable to the SLMed EH36 steel from our previous work[135]. Those models help to predict the fatigue limit or critical defect size within the built parts which is important for ease of engineering adoption. Further studies on other correlating models such as the correlation between fatigue limit and UTS, Hv as shown in equation 2-3 can help understand the AM built metal fatigue behaviors[21].

$$\sigma_{FL} = 0.5 \text{ UTS and } \sigma_{FL} = 1.6 \text{ Hv [136]} \quad 2-3$$

There are dozens of mathematical models which can be related [20]and examined for the AMed parts. More of them will be discussed in the following section specifically for fatigue life prediction.

2.6.2 Model Validation

Model validation is to use experimental measurement to compare with the predictions from a mathematical model[137]. If discrepancies are exceeding criteria, model tuning is needed to meet the criteria[138]. A simple statistical method checking such as goodness of fit may not be adequate, and a hypothesis test is considered as a minimum for model validation[139]. Very often a simple statistical method for strength of correlation is conducted, that is calculating the coefficient of correlation (r) [140], which lies in the range of -1 to +1, such as the Karl Pearson Coefficient. The strength of association will depend on the r values, that is if r is larger than 0.7, there is strong linear correlation between the variables. If the r is between 0.5 and 0.7, it is considered moderate, and between 0.3 – 0.5 is weak and less than 0.3 is treated as none or very weak.

2.7 Numerical Methods on Fatigue Life Prediction

2.7.1 Background

Over 80 - 90% of engineering failure comes from fatigue failure, especially high- cycle fatigue (HCF). For HCF, the fatigue initiation stage occupies a large portion of the total fatigue life. When a crack grows to an engineering detectable size, the crack growth rate is very high leading to a fatigue failure in a catastrophic manner. So, it is necessary to study the crack initiation stage and its influencing factors. Conducting fatigue test is usually time and cost consuming. Therefore, fatigue modelling and simulation become utmost important[15] in order to optimize the design of component before it is put into production. It can be a good approach to study the effects of pores with various combinations of shape, position, size and orientation on the fatigue initiation life cycles through simulation. Even though a simulation through software may not accurately reflect the actual situation, it can help better understand their complex interactions.

2.7.2 Numerical Studies on Fatigue for Conventionally Manufactured Metals & Alloys

Pores entrapped in additively manufactured metals are one of the critical factors affecting the fatigue behavior of the metals because the pores especially in large size and complex shape can elevate local stress concentration and induce complex stress field that favors fatigue crack formation. Sigl et al. [141] evaluated the influence of internal shrinkages on strength, elongation and young's modulus of cast steel 8630 (quenched and tempered, HRC = 34) with two types micro-

and macro-sized as well as their fatigue lives both experimentally and numerically. They found that the method of local strain-life was valid for assessing the fatigue life of materials containing only micro-sized shrinkages while the method of linear elastic fracture mechanics (LEFM) was valid for neither. They claimed that the non-conservative prediction of LEFM method to the macro-sized shrinkages was attributed to the complex shape and large size of the shrinkages that induced uncertainty of responsible actual sizes for failure, and lack of good crack front propagation models catered for material surrounding the more tortuous macroscopic pores. The unrealistic calculations of LEFM with micropores were dictated as inappropriate usage of extrapolating the Paris equation into the small crack growth regime and lack of fatigue crack growth properties for microporosity-contained specimens also. Fan et al. [142] studied the effect of 2D pores/inclusions on local cyclic plasticity at the micronotch root of a cast Al-Si alloy with varied size, spacing, aspect ratio and clustering for both realistic and idealized geometries using finite element method (FEM). Based on a simple local form of Coffin-Manson law and shear-based criterion study, the fatigue incubation life (N_{inc}) was highly related to the maximum range of cyclic plastic shear strain ($\Delta\gamma_{max}$), which acted as the driving force for fatigue crack formation. Zhao et al.[143] experimentally and numerically studied the super-long life fatigue behavior of a welded EH36 steel and found that a more severe stress concentration was coupled with larger size as well as near surface pores and inclusions. They also observed that the fatigue cracks intended to initiate from subsurface impurities, which was attributed to a higher stress concentration induced by inclusions rather than pores with same sizes. High strength steels usually behave more defect sensitive than their low strength counterparts. Nicoletto et al.[144] reported the realistic 3D pore morphology inside a cast Al-alloy using X-ray computed tomography and calculated the stress concentration factors using ABAQUS and found that the stress concentration factor of 3D pores ($K_t = 3.5$) was

only half of that of 2D pores ($K_t = 7.186$). They indicated that the stress concentration effect induced by the complex shape of 3D internal pores was dependent on not only notch root radius but also loading direction. Lu et al. [145] studied two types of 2D pores with simplified circular shape and awkward star shape using FEM and found that the stress distribution varied with the pore geometry but the stress concentration factors were within a 10% variation.

2.7.3 Numerical Studies on Fatigue for AMed Metals & Alloys

Numbers of experimental reporting on fatigue behavior of AMed materials have been made, but fewer on simulation [24, 146, 147]. Biswal et al. [148] assessed the local cyclic plastic behavior near the isolated gas pores and their fatigue performance of AMed titanium alloy through conducting an elasto-plastic analysis in ABAQUS and found that the simulated stress concentration factor varied with pore shape and location where higher K_t (up to 4.36 at a distance of $0.02d$ from the surface, where d is the major axis diameter of a pore) incurred with subsurface pores and lower K_t (2.08 – 2.5) with internal/surface pores. The local stress-strain, stress ratio and plastic deformation at root area of the pores were also evaluated where higher K_t coupled with higher cyclic plastic strain and hence a lower fatigue life was determined. They also claimed the highest strain occurred at the mouth of the surface hemispherical pore but the highest stress occurred at the root of the pore.

Other models such as Murakami method [149, 150], Murakami-Takahashi diagram [151], El HADDAD and TOPPER approach [151], Danninger-Weiss models [149] use crack length to predict fatigue strength which count for the crack location effect as well. Those models show good

prediction capability to small sized cracks up to a few hundred microns, but not larger sized cracks. The discrepancy between experimental results may come from other factors except size and location, such as crack shape, etc. Instead of single pore evaluation, Siddique et al. [152] evaluated the overall pore influence on stress concentration and fatigue life inside SLMed Ti6Al4V, where the overall pores are captured by CT scanning and analyzed in FEM software. There was also simulation work done by Wycisk et al. [151] using LEFM-software AFGROW to predict fatigue strength of SLMed Ti-6Al-4V. The simulation gives good consistency at long crack range, but overestimated the fatigue strength for smaller crack size, which is presumed due to its linear elastic nature that didn't take into consideration of non-linear effect and micro-crack propagation below the stress intensity threshold ΔK_{th} . Statistical analysis was also adopted by Günther et al.[150] for predicting failure probability of various defects including pores, lack of fusion and α -phase initiated fatigue crack types.

The unique AM process may render the fatigue behavior of AMed samples away from traditionally manufactured grades, including distinction in strength, ductility, Young's modulus, pores and microstructure, etc. The simulation work done by Siddique et al.[152] adopts radiographs to reconstruct the intrinsic pores existed inside the sample to obtain the pore distribution. However, the resolution of X-CT is constrained to tens of microns, so the simulated results might not be accurate.

2.7.4 Fatigue Life Prediction Methods and Baseline Theories

2.7.4.1 Theories on Fatigue Failure Mechanism

The fatigue life prediction methods[153] and the widely spreaded baseline fatigue theoretical models[154] are briefly introduced in this section. Chowdhury et al.[154] divided the fatigue failure theories into two major groups, that is fracture mechanics (FM) and dislocation-based theories. Wang et al. [153] generally divided the abundant fatigue life prediction models into another two categories, that is fracture mechanics and cumulative damage theories. In view of the other work published in the field of fatigue, the cumulative damage theory can be added as the third theory above Chowdhury et al. work. FM deals with both macro- and meso-scale fatigue studies depending on which theories are involved from LEFM, geometrical or plastic accumulation based on the summary of Chowdhury et al.[154]. Among FM, LEFM is most popular theory that deals with more on macro-scale fatigue behavior with pre-existing small cracks. Dislocation theory touch on mesoscale plastic deformation mechanism on the level of dislocation behavior. Cumulative damage theory covers both macro- and meso- scopes depending on the scales that is involved. There are latest atomistic scale studies, which uses Molecular dynamics (MD) replying on numerical methodology.

2.7.4.2 Fatigue Life Prediction Methods

Regarding fatigue life prediction, traditional three methods[155] stress-life, strain-life and Paris law equation are widely used today. The stress-life method uses nominal stress which didn't consider the local stress and strain. The underlying assumption is for same stress concentration factor and loading, the fatigue life cycle is the same[156]. The local stress and strain can be

calculated using methods such as the critical distance, highly stressed volume and gradient based methods[157], which can be imported into S-N curve to calculate the fatigue life. Strain-life method is a local method which count the local stress and strain (with local cyclic plastic deformation) at stress concentrators, which can be used for both high cycle and low cycle fatigue life analysis. The underlying assumption of local strain-life method is that the life for crack nucleation and small crack propagation at notched component is similar to the laboratory specimen under the same cyclic distortion.

Other existing analytical models which are essentially mutations of the above three methods and derived from the original models contributing individually to certain aspect[146]. The above three methods are considered phenomenological models that describes fatigue behavior from macroscale which didn't tell anything regarding the underlying mechanism. So development on mesoscale attributed to recognize the reasons of fatigue failure from the mechanistic aspect. One of the goodness down to mechanistic level is that it can study the microstructural effect by incorporating a statistical method to produce varied grain size and crystallographic orientation into FEM for fatigue crack initiation and propagation[113, 114]. The micro-level corresponds to the atomistic scale which studies the fatigue behavior from lattice resistance. Many of them utilize numerical methodologies (such as fitting a regression curve through experimental data sets) that are regarded as numerical methods. Qiu et al.[158] classified numerical methods for structural fatigue problem as stress-life, strain-life, Paris law and FEM methods as listed in the top rows of Table 2-5, However there are still other methods that can be classified as numerical methods as added to the lower part of the table along with the mathematical equations and their predicting scope for fatigue crack initiation (N_i), propagation (N_p) or both of them. Some of the commercially

available software for fatigue life prediction is summarized in Table 2-6, each with their pros and cons. Most of the software can only predict the fatigue initiation life using S-N and ϵ -N methods using FEM based input of stress and strain. Only a few of the software can simulate the crack propagation with both crack path and the remaining life cycles, while by incorporating fatigue initiation algorithms, the software can also predict the fatigue crack initiation life cycles. Few of the software use equations instead of FEM input such as NASGRO. The Fe-Safe software is a well-recognized and world leading package for durability analysis based on FEA with its capabilities being developed to meet the most demanding industry applications[159]. It is also the first commercial multiaxial strain-based fatigue analysis software. It is chosen for the analysis of the fatigue life cycles in this work.

On the other hand, finite element method (FEM) is a modern powerful numerical method for studying the fatigue crack propagation[160]. Microstructural effect can be incorporated for evaluation from the grain-scale level. Even though numerical methods are time and cost saving compared to experimental fatigue study. However, the experimental method is still an important and most reliable method for fatigue life measurement. Meanwhile due to the uncertainty (scattering in nature) of fatigue life owing to microstructure variations, etc. probabilistic methodology is often used as a useful skill for data analysis of fatigue life cycles. Pure analytical models can be used but the accuracy of the predicted results are highly relying on the material parameters adopted and microstructure homogeneities of the materials being studied. The composite methods combining FEM and analytical models can study the fatigue behavior from multi-scales.

The stress-life method is usually adopted for HCF [161], where materials is assumed to fail by a maximum stress. The strain-life method is usually taken for LCF with the damage parameter being a normal strain. Nowadays, the strain-life method becomes more popular as it considers localized plastic deformation[117, 162]. The two methods are based on uniaxial fatigue algorithm. However, in reality, a uniaxial stress or strain state is very difficult to happen, so multi-axial fatigue theories are recommended for practical 2D or 3D structures. Brown-miller (BM) algorithm is a commonly used multi-axial fatigue criterion (strain-life method) that is using a critical plane analysis with the damage parameter of shear ($\Delta\gamma_{\max}$, maximum shear strain) and normal strains ($\Delta\epsilon_n$, a normal strain range) [163, 164]. This algorithm is recommended for most of the metals in fatigue analyzing software FE_SAFE, mostly for ductile metals. For brittle metals a maximum principal strain algorithm is recommended as there is not much plastic deformation at crack tip. All above three methods are used for the uniaxial tensile HCF analysis in this work with their differences to be discussed. A mean stress correction can be applied for the tests conducted at stress ratios other than $R = -1$. There are two well-known mean stress corrections that are applied to the strain-life method, i.e. Morrow [165]and SWT[166]. The Paris law equation deals with crack propagation based on LEFM theory that is evaluated for the pre-existing cracks as pores, inclusions, etc. All the three methods will be evaluated in this work for their applicability for SLMed EH36 steel samples.

Table 2-5 Numerical methods for fatigue life prediction[154, 158]

Numerical method	Mathematical equations	Fatigue life prediction	Scale	Fatigue Theory
Stress-life (S-N curve)	$\frac{\Delta\sigma}{2} = \sigma'_f(2N)^b$	N_i	Macro	---
Strain-life (ϵ -N curve)	$\frac{\Delta\epsilon}{2} = \frac{\Delta\epsilon_e}{2} + \frac{\Delta\epsilon_p}{2} =$ $\frac{\sigma_f}{E}(2N_f)^b + \epsilon'_f(2N_f)^c$	N_i	Macro	---
Paris law	$\frac{da}{dN} = A (\Delta K)^n$	N_p	Macro	LEFM
Finite element method (FEM)	---	N_p	Macro/Meso	---
Extended Finite Element Method (XFEM)	---	N_p	Macro/Meso	
Finite difference method (FDM)	---	N_p	Macro/Meso	---
Boundary element method (BEM)	---	N_p	Macro/Meso	---
Mesh-less method	---	N_p	Macro/Meso	---
Miner's rule	$D = \sum_{i=1}^n \frac{n_i}{N_i}$	N_i	Macro	Cumulative damage

Molecular dynamics (MD)	---	N_i & N_p	Meso/Atomistic	---
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Table 2-6 The commercial available software used for fatigue life prediction

Software	Method	Fatigue life prediction
ABAQUS	FEM	N_p
ANSYS	FEM	N_p
COMSOL	FEM	N_p
Nastran	FEM	N_i
MSC Fatigue	FEM	N_i & N_p
NASGRO	---	N_p
AFGROW	FEM	N_i & N_p
nCode	FEM	N_i & N_p
Altair OptiStruct	---	N_i
LS-Dyna	FEM	N_i & N_p
FRANC	FEM	N_p
FatiguePlus	FEM	N_i
SFAT	FEM	N_i

LIMIT	FEM	N_i
Sesam Wind Manager	FEM	N_i
I-DEAS	FEM	N_i
Fe-Safe	FEM	N_i

2.7.5 Notch Effect

The crack growth behavior at the root of a hole, notch or other stress concentrators are different from macroscopic behavior described by Paris law[167]. That is the small crack growth is governed by local plasticity and crack closure effects, etc. coming from the large scale local plastic deformation induced at the notch root from severe stress concentration or applied loading. Even though the small crack behavior is not studied specifically in this work, the local stress and strain field at the surroundings of the notches were studied through FEM method. The stress and strain field calculated from FEM will be used for the fatigue life cycle studies in the following steps. Instead of using the EPFM model, the LEFM based model was investigated on the SLMed EH36 steel samples with assumption of pre-existing small cracks (the pores).

2.8 Summary

In summary, metals fabricated from AM are stronger and harder compared to those fabricated with conventional manufacturing methods mainly due to high cooling rate, solid solution strengthening, etc., incurred during AM processing. However, the ductility, toughness and fatigue strength of

AMed metals are lower, which are mostly attributed to the internal porosities. To achieve good quality AM products for real applications the density of AMed materials has to be high enough to ensure sound mechanical properties of the AMed products, which is determined by process parameters and material properties.

The strong anisotropic microstructure coupled with AM process again determines the anisotropic mechanical properties that vary with orientations. In some cases, post-process homogenization (compositional, structural, etc.) could be applied to meet the standards for wrought materials. The inhomogeneous meta-stable microstructure (fine cellular structure) and phases (due to element segregation, etc.) formed from rapid cooling process may undergo evolution upon heat or mechanical treatment due to recrystallization from dislocations[168]. As those studies are obviously not enough, more studies are needed for possible solutions to improve the material integrity and guarantee consistent quality printing[169]. The process needs to be further optimized for the ultimate target of fabricating pore free solid part. The process-microstructure-property relationship needs to be further studied for materials adopted. The applicability of traditional mathematical models and new mathematical models suitable for AMed steels are necessary to be evaluated and set up for their benefits. Meanwhile, the fatigue failure mechanism of AMed metals need to be studied through both experimental and simulation work, especially for the intrinsic pore effect.

The availability of AMed materials is limited especially the high strength steels for ship construction and further development is needed to provide more choices for industrial applications. This review provides a critical assessment on the mechanical properties, microstructures and

processes of AMed materials with reference to conventional manufactured materials for understanding of their suitability for Marine and Offshore applications. The promising properties achieved from AM built steel materials make development of ship structural steels, e.g. EH36 grade, worth of further development. Filling of a gap in fatigue performance investigation is needed for a comprehensive understanding of the material mechanical properties. In this work the feasibility study is on the EH36 steel to be built with both SLM and DLD processes while both have their pros and cons. The results obtained would be used as a guideline for certification of AM built EH36 steels in assisting industrial partner's adoption.

Chapter 3 Experimental Details and Simulation Methodology

3.1 EH36 Steel Samples Built with SLM Process

3.1.1 EH36 Steel Powder

An ASTM A131 powder (EH36 grade, gas atomized) with particle sizes in the range of 20 - 53 μm was provided by Sandvik Osprey with the chemical composition listed in Table 3-1 and the morphology shown in Figure 3-1.

Table 3-1 Chemical composition of EH36 steel powder (wt%)

C	Si	Mn	P	S	Cr	Mo	Ni	V	Cu	Nb	Ti	Al	Ca	Fe
0.14	0.39	1.07	0.01	0.006	0.5	0.03	0.2	0.11	0.01	0.04	<0.01	0.01	<0.002	Balance

Figure 3-1 SEM micrograph of EH36 steel powder used for printing samples via SLM and DLD processes

3.1.2 Design of Coupons and Printing Parameters for SLM Process

Rectangular EH36 steel blocks were printed using an SLM process with a size of 95 mm x 20 mm x 50 mm with their schematic diagram shown in Figure 3-2. The blocks were cut into tensile and fatigue test coupons. Miniature tensile coupons were used for tensile property evaluation with the dimensions shown in Figure 3-3. The drawing of the fatigue coupons is shown in Figure 3-4. The process parameters evaluated include laser power, scanning speed, hatch spacing and preheat temperature with the details shown in Table 3-2, 3-3 & 3-4. The dimensions of the start plate used were 100 x 100 x 25 mm. The design of experiment was based on a previous work in which scanning speed showed a more significant effect on microstructure and properties based on the study of LMD (laser metal deposition) processed 316L while the solidification rate and temperature gradient were more sensitive to scanning speed than laser power[58].

Figure 3-2 Schematic diagram of printed rectangular blocks to be used to cut into tensile and fatigue coupons

Figure 3-3 Drawings for tensile test coupons with all dimensions in mm

Figure 3-4 Drawings for fatigue test coupons with all dimensions in mm

Table 3-2 SLM process parameters with varying scanning speed and hatch spacing

Sample No.	Laser power (W)	Scanning speed (mm/s)	Hatch spacing (mm)	Layer thickness (mm)
1	175	100	0.08	0.05
2			0.09	
3			0.1	
4			0.11	
5			0.12	
6			0.13	
7		200	0.08	
8			0.09	
9			0.1	
10			0.11	
11			0.12	
12			0.13	
13		300	0.08	
14			0.09	
15			0.1	
16			0.11	
17			0.12	
18			0.13	
19		250	0.12	

Table 3-3 SLM process parameters with varying laser power (hatch spacing = 0.11 mm, layer thickness = 50 μ m)

Laser power (W)	145	155	165	175	185	195	210	225	240
Scanning speed (mm/s)	100								
	200								
	250								
	300								
	400								

Table 3-4 SLM process parameters with varying preheat temperature (laser power = 175 W, hatch spacing = 0.11 mm, layer thickness = 50 μ m)

Scanning speed (mm/s)	Preheating temperature ($^{\circ}$ C)	
100	100	200
200		
250		
300		
400		

3.2 EH36 Steel Samples Built with DLD Process

3.2.1 Design of Coupons and Printing Plan

The EH36 powder used here was the same as that used in SLM printing. Samples were built with a laser assisted additive manufacturing (LAAM) system which is equivalent to direct laser deposition (DLD) process developed by Singapore Institute of Manufacturing Technology, with a 6 kW laser integrated to an industrial robot. The system was also equipped with a coaxial powder feeding nozzle and an Argon gas protection system. Near-net shaped coupons in 4 different orientations, noted as XY_0 $^{\circ}$ (9pcs), XY_45 $^{\circ}$ (15pcs), XZ_90 $^{\circ}$ (15pcs) and XZ_45 $^{\circ}$ (15pcs), were built under optimized process conditions using 1 kW laser power, 1200 mm/min scanning speed, 1 mm overlapping and 8 g/min powder feed rate. The beam size is 2 mm with beam focus size of 0.6 mm, however defocusing of the beam is adopted for printing at a beam size of 2 mm. The resultant layer thickness is about 0.5 mm. A continuous printing strategy was applied to the two horizontal directions, while an intermittent printing strategy was adopted for the two vertical

directions after the process optimization. The printing strategies adopted with different orientations were illustrated in Figure 3-6. The layout of the near-net-shaped samples is shown in Figure 3-5 with all the samples. The start plates were not preheated. Tensile, charpy and fatigue test coupons were machined from the near-net-shaped coupons with the dimensions (in mm) shown in Figure 3-7.

Figure 3-5 Photographs showing EH36 steel samples built in (a) two horizontal directions XY_0° and XY_45°, (b) vertical direction XZ_90°, and (c) vertical direction XZ_45° via DLD process

Figure 3-6 Schematic illustration of scanning strategies in the four directions of: XY_0° (b) XY_45° (c) XZ_90°, and (d) XZ_45°

Figure 3-7 Drawings of testing coupons for (a) Charpy impact, (b) tensile and (c) fatigue tests with all dimensions in mm

3.3 Sample Preparation and Characterization

Metallographic preparation of the samples was done under standard procedures. The mainly vertical cross-section examination along the build direction was done by cutting through the middle of the samples using electrical discharge machining (EDM) followed by grinding and polishing until 1 μm diamond paste. The freshly prepared surfaces were then etched by a saturated picric acid or 2% nital for a few to tens of seconds using an immersion method. The etched samples were immediately washed thoroughly with purified water and then ethanol.

TEM samples were prepared with the following procedure: a slice was sectioned off from each block sample and thinned down to a thickness of about 100 μm by sand papers. A disk of 3 mm in diameter was formed by blanking and mounted for further dimple grinding (Gatan Model 656) to a thickness of about 10 μm using 1 μm diamond paste. Finally, ion milling (Gatan Model 691) was used to make the disk even thinner until perforated. The ion milling operations were run at a large angle of 8° for 30 min followed by a small angle milling at 4° and 2° respectively until the sample was perforated. The sample was taken out and checked under optical microscope every 15 minutes to prevent over milling and sample damage.

The microstructure and surface morphology of the samples were characterized using optical microscopy (Zeiss and Olympus), scanning electron microscopy (JEOL JSM-5600LV) and transmission electron microscopy (JEM 2010), FE-SEM (JEOL JSM-7600F). X-ray Diffraction (Philips) was used for phase determination (with Cu anode).. Elemental analysis was analyzed by using energy dispersive X-ray spectroscopy (FE-SEM, JEOL JSM-7600F). The hardness of the samples was measured by Vickers micro indenter (FM-300e) under a fixed load of 50 g or 300 g for 10 s – 15 s. Tensile testing was conducted with a universal testing machine under a maximum loading of 50 kN (Instron 5569). The error of the force sensor of the tensile testing machine is 0.1% and the extensometer is 0.01%. Fatigue testing was conducted on a MTS machine with a dynamic hydro servo system (MTS 810) using a sinusoidal loading under a load ratio of $R = 0.1$ and a frequency of 5 Hz. The surface roughness of the machined tensile, charpy and fatigue coupons are measured by Surftest SJ301 surface profilometer (Mitutoyo). The testing coupons were machined to achieve a surface roughness of $R_a < 0.2$ for all test coupons.

The density of the SLM built samples was measured by Archimedes method at room temperature. At least 3 repeated measurements (wet weight in ethanol) were taken to get an average density value. The 2D analysis of the porosities was conducted by using ImageJ software. The 3D analysis of the porosities was conducted by using a custom-made computerized tomography (CT) system with an XWT-225KV-XC microfocus transmission tube and a PerkinElmer a-Si Digital X-Ray Detector. The scanning speed effect on densification, microstructure and mechanical properties were studied and compared between samples: 100 mm/s (denoted as S100, with hatch spacing at 0.08 mm), 200 mm/s (denoted as S200, with hatch spacing at 0.1 mm), 250 mm/s (denoted as S250, with hatch spacing at 0.12 mm) and 300 mm/s (denoted as S300, with hatch spacing at 0.12 mm). The samples used for fatigue test were from different printing lots with slightly different laser powers around 150W, which were denoted with a prefix of SS, that is SS100, SS200, SS250, SS300 and SS400 representing the scanning speeds of 100, 200, 250, 300 and 400 mm/s, respectively. At least 3 pcs of tensile coupons were tested to get an average value. The S-N curves were built using 5 stress levels with 1 or 2 pcs of fatigue samples being tested at each stress level. For the DLD printed samples in each orientation, 3-5 pieces of tensile coupons, 3-5 pieces of charpy impact coupons and 1-5 pieces of fatigue coupons (for each stress level) were tested. The fatigue samples exceeding 10^6 cycles were considered run-out. The chemical elements of the samples were analyzed by Carbon Sulphur Analyzer for C and S, Oxygen Analyzer for O, and ICP-OES for P, Mn, Si, Cr, Ni, Mo, Cu, Nb, V, Ti, Al and Fe. Eventually for the both processes the relationships between mechanical properties, microstructures and process parameters were established.

3.4 Simulation of Fatigue Life

3.4.1 Static Strength and Young's Modulus

The ultimate tensile strength (UTS) varied from 971 to 855 MPa from the highly densified samples to the more porous samples obtained from previous test results[135] and re-listed in Table 3-5. The effect of scanning speed to densification has been thoroughly discussed in previous results and here only the values of their UTS were used for simulation. The microstructural effect was incorporated into UTS without too much detailed study on its effect to fatigue life. Young's modulus of the AMed steel samples could be a bit away from conventionally manufactured same grade of steel due to process induced defects such as pores (lack of fusion and gas pore) and other factors. The internal pores may reduce the effective loading area as well as Young's modulus based on previous research on materials manufactured by conventional processes[170, 171]. Fatigue properties were proved to be seriously deteriorated by pores for conventional manufactured steels/irons [170-172] as well as AMed grade [150, 152]. However, there was no experimental data available for it and further investigation would be needed to achieve more accurate results. Here the porosity effect to Young's modulus was counted by a simple linear relationship and would be discussed in the following fatigue parameters section.

Table 3-5 Tensile properties of the samples[135]

Sample	UTS (MPa)	$\sigma_{0.2}$ (MPa)	Elongation (%)
S100	971 $\pm$ 43	874 $\pm$ 111	8.7% $\pm$ 1.8%
S200	921 $\pm$ 28	867 $\pm$ 8	6.3% $\pm$ 1.4%
S250	891 $\pm$ 38	845 $\pm$ 24	5.0% $\pm$ 0.9%
S300	855 $\pm$ 16	809 $\pm$ 15	4.3% $\pm$ 0.9%
S400	808 $\pm$ 50	751 $\pm$ 49	3.6% $\pm$ 0.4%

3.4.2 Pore Characterization

Figure 3-8 showed the computed tomography (CT) image giving the 3D morphology, size and distribution of pores inside the sample SS300. Most of the pores were in complex shapes in a scale of tens of microns to a few millimeters. Some pores were much extended in one direction and highly branched (protruding outward) along the way. The narrow part was estimated to be as penny as tens of microns in the transverse directions of the pores. The irregular contour may elevate stress at locations such as corners with small radius. The estimated volume of the pores was around 3 vol%, which is slightly lower than 2D ImageJ measured density (6%) but comparable to Archimedes method (3%)[173]. From the CT image the pores were vastly allocated through the whole volume and small pore-pore distance was observed forming pore clustering. However those pores were mainly located between layers which belong to lack of fusion due to insufficient laser penetration during the printing process. The lack of fusion porosity had been thoroughly evaluated at previous work[135]. One of the OM images was illustrated here in Figure 3-9 showed the positions of the lack of fusions for sample SS300. The densities of the printed samples are directly related to the amount of lack of fusions, which have been measured as 99%, 97%, 94% and 94% for the sample of SS100, SS200, SS250 and SS300 using Archimedes method from previous work[135].

Figure 3-8 CT scan image for sample SS300 showing the 3D view of the pores in the sample[135]

Figure 3-9 OM image for sample SS300

3.5 Simulation Background and Methodology

3.5.1 Modelling Pores on Surface

As stated above, inner pores were believed to directly cause crack propagation and contribute to less bearing area and reduced Young's modulus, strength, etc. Therefore, surface types of pores were proposed in a way to mimic the actual AMed pore sizes and shapes for their effect on fatigue behavior. However, complex tri-axial stress fields could be induced for real pores with above

shapes found in CT scan. To be simplified, triangular (tri) shaped pores (a pentahedron in 3D morphology) were proposed with various aspect ratios to be studied for their effect on stress concentration/distribution and fatigue crack initiation. Further simplified semi-ellipsoidal (elli)/semi-spheroidal (sphe) shaped pores were also proposed to be studied as a comparison to more realistic complex shape of pores. The triangular pores were designed with sharp corners and studied in both isolated (with single triangular segment) and clustered forms (dual triangular pores, pores with three triangular segments and pores with crossover segments). The clustered triangular pores were to present the effect of pore-pore interactions on the induced stress fields. Among them dual triangular pores separated by a small distance of 10 μm and triangular pores with multiple triangular-segments, and triangular pores with crossover segments were studied as well. The configurations of such pores would be discussed in the following parts.

The 2D sketch of semi-spherical or semi-elliptical (in varied aspect ratios) pores were illustrated in Figure 3-10a and their 3D geometries were obtained by revolving along either x, y or z axis. Here, x-y-z was denoted as the radii of 3D semi-spheroidal/ellipsoidal pores along three axes x, y & z. Those semi-spheroidal pores were studied at seven radii in 0.01, 0.025, 0.05, 0.075, 0.1, 0.15 and 0.2 mm, respectively. The semi-ellipsoidal pores were studied at 0.01, 0.025, 0.05, 0.075, 0.1, and 0.15 mm at two axes and at 0.25, 0.5, 0.75, 1.0 and 1.5 mm at the rest axis. The loading direction was along the y axis. The triangular pores were denoted in the form of x-y-z as well, but the x, y and z doesn't follow the axis of the above semi-ellipsoidal pores, while it will depend on the side (either short side or long side) and orientations (perpendicular or parallel) being resided to the model as illustrated in Figure 3-10b. But all triangular pores are denoted literally as x fixed at 0.2 mm and y fixed at 0.1 mm (with 10 μm root radius for the top triangular corner) and varying

length scale z from 0.05 mm to 2.9 mm (through pore along short side) or 5.8 (through pore along long side). The 3D morphologies of semi-ellipsoidal/spheroidal and triangular pores were shown in Figure 3-11.

The effect of the aspect ratios to stress concentration were studied from 1 – 50 for semi-ellipsoid/spheroid pores, which was calculated by major axis over minor axis. For the ratio of z/x or z/y , z is fixed at 500 μm and x & y were varied from 10 - 500 μm . For x/y or x/z , x was fixed at 500 μm and y & z were varied from 10 – 50 μm . The aspect ratios of the triangular pores were ranged from 0.25 – 14.5, which were calculated by length z divided by x or y , where z was varied from 0.05 mm – 2.9 mm and x & y was fixed at 200 μm . There is an option in FE-SAFE software for a value of surface finish factor K_t to be assigned to the analyzed model which can count for potential stress concentration effect for the analyzed model. To evaluate the effectiveness of adding this equivalent K_t to count for pore induced stress concentration. A series of K_t was assigned to a smooth surface model with the same design (smooth model without pore incorporated). The K_t values ranged from 1.0 – 5.5 and results of predicted fatigue life will be compared with pore-contained model.

Figure 3-12 showed the studied positions and orientations of the various semi-ellipsoidal/spheroidal & triangular pores. All the semi-ellipsoidal pores were allocated to the short side (ShS) surface (xy plane as shown in Figure 3-10b) of the fatigue model. The perpendicularly oriented triangular pores were allocated to both the short side (ShS) and long side (LoS) surface (yz plane as shown in Figure 3-10b) and the parallel oriented triangular pores were allocated to both ShS and LoS surface. Three positions were studied, side centre (SC), side edge (SE) and

middle edge (ME). Side centre refers to the middle position of the surface and side edge refers to the corner position near to the edge of the model. Middle edge means the side edge further moved down inside the mid of the body. Meanwhile to compare the shape and size effect with triangular pores, a simple cylindrical pore with the similar size (diameter of 0.2 mm and varied in length scale being allocated to side centre of short side) as shown in Figure 3-12 will be studied for their stress concentration effect as well as a singular triangular pores which is formed by revolving (being assigned to side centre of short side). The effects of proposed pore shape, size, position and orientation on K_t and fatigue crack initiation life were studied. The dual pores, crossover pores and pores with multiple triangular segments were studied to compare their stress amplification/reduction effect to single isolated triangular pores. Different algorithms in FE-SAFE were used and compared. In the end, a theoretical crack propagation lifetime was calculated using LEFM model. By using the LEFM model, the crack propagation life was regarded as total fatigue life assuming the whole life cycle was dominated by crack propagation stage[141]. Then results will be compared with crack initiation life predicted by FE-SAFE software. In FE-SAFE the crack initiation life was treated to be the cycles that caused a 1 ~ 2 mm crack length with an assumption that crack initiation dominated the whole fatigue life. The accuracy of the FE-SAFE software and LEFM model will be validated with experimental tested fatigue life for all the samples. Possible reasons will be discussed for the inaccuracies caused.

Figure 3-10 Schematic illustrations of the (a) 2D surface semi-elliptical/spherical pores and revolving the directions to 3D surface semi-spheroidal/ellipsoidal pores, and (b) the axis allocation of the model

Figure 3-11 Schematic diagrams showing the shapes and dimensions of 3D ellipsoidal/triangular pores (a) $x = y$ and z varies with different length scales (0.1 – 1.5 mm), (b) $y = z$ and x varies with different length scales (0.1 – 1.5 mm), (c) $x = z$ and y varies with different length scales (0.1 – 1.5 mm), and (d) triangular pore with two dimensions fixed at 0.2 (x in this case) and 0.1 mm (y in this case), respectively, and another dimension varies with different length scales (0.15 – 2.9 mm), where the x or y axis may vary depending on which plane and orientation the triangular pore is resided.

Figure 3-12 2D layouts of triangular (tri) and elliptical (elli) pores with positions at the side edge (SE) or side center (SC) of the sample surface located at gauge area. The 3-segment triangular pore represents more complex shape of triangular pores. The cylindrical pore is illustrated and will be compared with triangular pores. The singular triangular pore which is revolved will be compared as well. Note: If the pore is allocated to short side, a prefix of “ShS” will be added to the denotation, same as long side as “LoS”. This is appeared in the following part. ME means middle edge with the pores moving from the side edge to the middle/centre of the body (to the z direction as shown in Figure 3-10b). All the images illustrated are viewed from the short side of the model. The 3D morphology of crossover pore is also inserted which doesn’t show the cross section view but located at LoS_ME_Perp.

3.5.2 Determination of Fatigue Property Parameters

The material parameters adopted for the both stress-life and strain-life methods are listed in Table 3-6. The experimental Young's modulus (E) was not available for the SLMed EH36 steel which would be varied due to geometrical effect and manufacturing process[174]. A value of E = 200 GPa and the poison ratio of $\nu = 0.3$ for standard steel materials were used for most of the simulation for the SLMed EH36 steel. Due to the potential significant influence of E to the fatigue properties, a virtual Young's modulus (E') was studied for its effect to fatigue lifetimes. The virtual Young's modulus was calculated using a simple linear relationship with porosity (vol%) of the individual sample, that is $E' = E \times (1 - \text{vol}\%)$, where here E is defined as 210 GPa instead of 200 GPa. As the E value of AMed steels could vary from conventional manufactured equivalent, here 210 GPa was only a general estimation which couldn't represent the real value. Fatigue strength coefficient σ_f' and fatigue strength exponent b were obtained from the fitted SN curves. The fatigue ductility coefficient ϵ_f' was estimated using the model $\epsilon_f' = \frac{0.32(\text{HB})^2 - 487(\text{HB}) + 191000}{E}$ proposed by Roessle et.al.[161], where HB is the Brinell hardness and E is Young's modulus. The microhardness data was taken from previous work for SLMed EH36 steel[135]. A value of -0.56 was also suggested for the fatigue ductility exponent c. The strain controlled fatigue test was not available for the AMed EH36 steel sample, so cyclic strength coefficient K' and cyclic strain hardening exponent n' were estimated with compatibility method[175] in the form of $n' = \frac{b}{c}$ and $K' = \frac{\sigma_f'}{\epsilon_f'^{1/c}}$. Noted that the ϵ_f' and c was obtained from literature review which was correspondent to zero mean stress (R = -1) condition, while the experiment in current work was conducted at R = 0.1. The mean stress effect was ignored at this moment.

Table 3-6 Material parameters used for simulations with respective symbols of notations

Parameter	Symbol	SS100	SS200	SS250	SS300
		values			
Porosity	Vol%	1%	2%	6%	6%
UTS	UTS	971	921	891	855
Microhardness, Hv_0.05[135]	Hv	288	334	345	345
Brinell hardness	HB	288	330	342	342
Young's modulus (GPa)	E	200	200	200	200
Virtual Young's modulus (GPa)	E'	210	205.8	197.4	197.4
Poisson ratio	ν	0.3	0.3	0.3	0.3
Fatigue ductility coefficient	ϵ_f'	0.386	0.325	0.309	0.309
Fatigue ductility exponent	c	-0.56	-0.56	-0.56	-0.56
Fatigue strength coefficient (MPa)	σ_f'	2,483	1870	3073	3425
Fatigue strength exponent	b	-0.183	-0.174	-0.222	-0.241
Cyclic strength coefficient (MPa)	K'	3399.4	2649.5	4915.6	5675.2
Cyclic strain hardening exponent	n'	0.33	0.31	0.40	0.43
SN curve method (Max. stress at 10^7 and 10^4 cycles, R=0.1)	-	901/255	742/223	758/163	700/132

3.5.3 Simulation Methodology

The simulation steps were illustrated in Figure 3-13. The static stress and strain was analyzed by FEM method with ABAQUS at a stress level of 400 MPa (nominal stress in the gauge area). The stress concentration factor (K_t) was calculated using maximum elastic stress (equivalent Von Mises) divided by the nominal stress (400 MPa). The stress data obtained from ABAQUS was then put into endurance analyzing software FE-SAFE for fatigue life analysis of the proposed models for the models incorporated with various surface pores. To calculate fatigue life at other stress levels, e.g. 500, 600 and 700 MPa, scale factors were used in FE-SAFE to be multiplied by 400 MPa. All studied stress levels were below the yield stress of the AMed EH36 steel samples. Only was elastic stress analysis conducted with ABAQUS, while the elastic-plastic behavior was not. For multi-axial plasticity correction, Neuber's rule in FE-SAFE could be turned on to convert stress into an elastic-plastic stress/strain to count plasticity effect.

Figure 3-13 Flow chart of static stress/strain analysis and fatigue life prediction

A few types of algorithms were evaluated for predicting the fatigue life [161]. In stress life method (S-N), also known as Basquin equation is shown as:

$$\frac{\Delta\sigma}{2} = \sigma_f' (2N)^b \quad 3-1$$

Where $\Delta\sigma/2$ is stress amplitude and $2N_f$ is reversals of fatigue life cycles. σ_f' is fatigue strength coefficient, b is fatigue strength exponent. The other method strain-life method (ϵ -N) known as Coffin-Manson relationship is shown as:

$$\frac{\Delta\epsilon}{2} = \frac{\Delta\epsilon_e}{2} + \frac{\Delta\epsilon_p}{2} = \frac{\sigma_f'}{E} (2N_f)^b + \epsilon_f' (2N_f)^c \quad 3-2$$

Where $\Delta\epsilon/2$, $\Delta\epsilon_e$ and $\Delta\epsilon_p$ are strain in total, elastic and plastic respectively. ϵ_f' is fatigue ductility coefficient and c is fatigue ductility exponent. Nowadays the strain-life method becomes more popular as it considers localized plastic deformation [117, 167]. The above two equations are considered uniaxial fatigue algorithm. However uniaxial stress or strain state is very difficult to happen, so multi-axial fatigue theories are recommended for practical 2D or 3D structures. Brown-

millers algorithm was one of the most commonly used multi-axial fatigue criterion as the strain-life method that were using critical plane analysis with the formula shown in equation (3) without mean stress correction [163, 164].

$$\frac{\Delta\gamma_{\max}}{2} + \frac{\Delta\epsilon_n}{2} = 1.65 \frac{(\sigma'_f)^2}{E} (2N_f)^b + 1.75 \epsilon'_f (2N_f)^c \quad 3-3$$

The life cycles of $2N_f$ could be solved using above equation at the node, where $\Delta\gamma_{\max}$ was the maximum shear strain, $\Delta\epsilon_n$ was the normal strain range revolved to shear stress plane. The critical plane methods resolve the strain onto a number of planes, and calculate the damage on each plane[176]. This form of analysis must be applied for criteria such as principal stress/strain, maximum shear stress/strain, and the Brown-Miller criterion for complex strain signals with varying phase relationships. It can show the orientation of the most damaged plane. In the case of combination with Brown-Miller method, the critical plane was the plane with maximized value of the sum of the two terms on left of the above equation revolved into the three shear planes. For each of three planes, critical plane is searched from the 18 subsidiary planes spaced at 10° increments from 0° to 180° using the three principle strains which were converted from in-plane principle stresses calculated from the 6 components of stress tensor coming from the FEA analysis. The damage parameters was then cyclically counted and summed for total fatigue damage. The plane with the shortest life was defined as the plane of crack initiation. This algorithm was recommended for most of the metals in FE_SAFE, mostly for ductile metals. For brittle metals maximum principal strain algorithm is recommended. All parameters are needed for simulation to be conducted. And FE-SAFE was using tensorial stress.

3.5.4 Settings in ABAQUS

The boundary conditions were set such that one end of the fatigue model was restricted at all degrees of freedom ($U1 = U2 = U3 = UR1 = UR2 = UR3 = 0$) and a uniform tensile loading of 400 MPa, was set at the other end as shown in Figure 3-14a. The meshed model was shown in Figure 3-14b which is the assigned mesh by ABAQUS with auto-refined mesh near surface pore region. Tetrahedron elements were used to mesh the fatigue test sample with quadratic shape functions. The meshed model was also shown in Figure 3-14b. The current FEM analysis is focusing on trend analysis, further refining of the mesh may increase the accuracy of the results.

Figure 3-14 (a) Boundary conditions and loading for a fatigue model analyzed in ABAQUS for static stress/strain, and (b) the meshed model using tetrahedron element. The mesh is finer near surface incorporated pore.

Chapter 4 Microstructure and Mechanical Properties of SLM Printed EH36 Steel Samples

4.1 Phase Determination

A basic BCC structure is confirmed by the XRD patterns shown in Figure 4-1 where all the three samples S300, S250 and S200 demonstrate similar diffraction patterns with five characteristic peaks indexed as (from left to right) $(110)_{\alpha'}$, $(200)_{\alpha'}$, $(211)_{\alpha'}$, $(220)_{\alpha'}$ and $(310)_{\alpha'}$, which are attributed to a martensitic structure[177]. A minor peak is also detected in the XRD patterns, which belongs to retained austenite indexed as $(111)_{\gamma}$ as indicated. There could be iron carbide phases as well but they are not detectable due to their low quantities.

The mixed phases can be related to the EH36 steel composition and the SLM process parameters. Firstly, high martensite start (M_s) and finish (M_f) temperatures make a near complete martensitic transformation possible. The M_s is estimated to be over 400 °C from the equation: $M_s (K) = 731 - 227(C+N) - 17.6Ni - 22.5Mn - 17.3Cr - 16.2Mo$ [177]. For this low carbon low alloy steel, M_f shall fall at around 300 °C. With the preheating temperature lowered to 100 °C, a temperature difference of about 200 °C makes a possible full martensitic transformation. Secondly, the SLM process can induce a high cooling rate ($10^3 - 10^6$ K/s) that is sufficient for the martensitic transformation to happen. From the CCT diagrams of steels a high cooling rate is usually achieved by a quenching process. Here, the SLM process can achieve a very high cooling rate up to 10^6 K/s, which is realized through directed heat dissipation where the build platform acts as a heat sink[80]. These

two are believed to be the main factors that lead to a mainly martensitic structure in the SLM printed EH36 steel samples.

Figure 4-1 XRD patterns of selected EH36 samples identified as martensite, retained austenite and iron carbide

The excess retained austenite in the samples could result from the high cooling rate as well. It was reported that a higher cooling rate renders higher undercooling[178] and reduced M_s (due to finer grain structure, etc.)[179] compared to conventional water/oil quenching process, where the martensite transformation is suppressed. A higher retained austenite content is evidenced by the small FCC peak detected in the sample S300, which incurred with the highest cooling rate among the three samples with the highest scanning speed. A similar report claimed a higher amount of

retained austenite in AM processed steels, which was attributed to two factors that can 1) suppress $\gamma \rightarrow \alpha'$ transformation due to strains at high-angle grain boundaries, γ stabilizing elements and environment of powder & part manufacturing and 2) reduced M_s temperature due to finer grain structure and higher dislocation density [180].

Higher retained austenite in SLM processed 17-4PH stainless steel has been reported compared to wrought one of the same steel [180], which was attributed to a core-shell dendritic structure with α' phase in the core and γ phase in the shell (based on a Scheil-Gulliver non-equilibrium solidification simulation) with following possible reasons: nitrogen absorption that stabilized austenite phase, lowered M_s due to fine dendrite of submicrons and microsegregation caused austenite stabilizers that suppressed the martensitic transformation. Meanwhile, the influence of residual stress was concluded based on as-built 4340 steel (DMD process) and stress relieved equivalent [18]. Results implied the existence of residual stress decreased M_s (measured through CCT experiment).

A small amount of iron carbide is believed to be formed either upon solidification or through tempering by the applied heat source. The element segregation is believed to make precipitation thermal dynamically favorable. The heat comes from three main sources: laser scanning, subsequent laser scanning and preheating, which may affect the microstructure in terms of martensite self-tempering, tempering and aging, etc.

Moreover, due to very low local cooling rate and high drop back temperature (above M_s), etc., some bainite or ferrite phases may also form. However, it is difficult to differentiate them from the martensitic BCT (body centered tetragonal) crystal structure because of the low tetragonality of

this low carbon steel. The martensite, retained austenite and possible iron carbide precipitates are further characterized using TEM and EBSD in the following part.

4.2 Macro-scale Features

Melting pool, pores and inclusions, etc. characterize the macro-scale features of the SLM printed EH36 steel samples. Figure 4-2 displays the optical images of the vertical cross sections for the etched samples S300 and S200, which are featured by melting pool and lack of fusion. The image of the sample S200 is darker and shows more significant lack of fusion that can range up to hundreds of microns compared to the sample S300. The average melting pool depths for the samples S300 and S200 are about 70 - 90 μm and 100 μm , respectively. A re-melted layer of 20 - 40 μm in depth is estimated from the average melting pool depth compared with the top-most layer of the sample S300. This ensures an enough penetration depth for a thorough melting of a 50 μm powder layer and metallurgical bonding with the previous solidified layers. Process parameters can influence melting pool behavior that may result in different grain structures formed subsequently as well as different features like lack of fusion, inclusions, etc. Here the two samples built with different scanning speeds demonstrate different macro-features that can in turn affect their mechanical behavior.

The average melting pool depth of the sample S200 is larger than that of the sample S300, which is due to a higher energy input due to the lower scanning speed. This is consistent with the findings on SLM processed SS 316L steel that the higher, the deeper the melting pool obtained is [20]. Meanwhile, the maximum void size suggests lack of fusion in the sample S200 is larger than that

in the sample S300. This is most probably due to more irregular melting pool morphology that deteriorates the surface finish and hence the interlayer bonding. The melting pool irregularity is most probably generated from a higher energy input as a result of a lower scanning speed[181]. The small round pores shown in Figure 4-2 are mainly due to trapped gas that cannot escape from the molten metal. More small porosities in the sample S200 are probably induced from more vigorous evaporation at higher energy input.

Figure 4-2 Optical images of vertical cross sections for etched samples: (a) S300 and (b) S200

Lack of fusion zone is usually coexisting with unmelted powder particles and inclusions. Those unmelted particles are usually present in the lack-of-fusion regions where energy is not enough to penetrate through and melt the layer underneath. A certain degree of melt pool surface oxidation results in formation of small metal oxides that are left in the inter-bonding layer. The kinetics of oxide formation in an SLM processed maraging steel is explained by Thijs et al.[88], while voids or cracks could be formed along with it [19]. In most AM printed metals the lack of fusion and unmelted powders are regarded as unpreventable [43]. Pores together with unmelted powders could possibly initiate cracks as often observed in the fractured surfaces of AM built metals [18,

19], which could also deteriorate the ductility, elastic modulus, toughness and fatigue properties of the materials due to their stress concentration effect[18]. Sometimes, lack of fusion can promote synergistic oxidation that may deteriorate the mechanical properties of the AMed steels from a large aspect[18]. Those kinds of unmelted or partially melted powder are not obvious in the sample S200 due to the higher energy input in the printing process from the lower scanning speed.

4.3 Micro-scale Features

Micro-scale features cover cellular-dendritic grains with their forming and evolution together with possible microsegregation-precipitation accompanied the rapid solidification process. Aside the cellular-dendritic grains the rapid solidification process induces various shapes of grains including acicular and coarsened columnar grains that will be discussed together with the possible martensitic transformation mechanism within the columnar grains.

As seen from Figure 4-3(a) the optical image of the etched vertical cross section of the sample S300 shows some cellular-dendritic grains within the melting pool and some equiaxed grains ($5 - 6 \mu\text{m}$) near the centre of it. The average size of the cellular-dendritic grains is around $1 \pm 0.5 \mu\text{m}$. The acicular shape of grains with the size of $1-2 \mu\text{m}$ characterized by smaller aspect ratios is observed within the same melting pool. The elongated cellular-dendritic grains with large aspect ratios grow from the bottom of the crescent-like melting pool to its center along the heat dissipation direction. However, the optical image of the etched vertical cross section of the sample S200 in Figure 4-3(b) shows indistinct cellular grains with darker intercellular regions that are dominated by a more cellular-like shape and obviously coarsened. The size of the cellular grains can reach

about 2 μm . In some regions short and wide acicular shaped grains with the size of 2 -3 μm are observed. For the both samples a near fusion white band distinguishes the melting pool boundaries.

In the sample S300, a grain evolution from planar (a region enclosed with a dotted line) to cellular and cellular-dendritic morphology (with dendrite arm) is observed in the SEM image in Figure 4-3(c). The cross section of the etched sample shows that the sample S300 has a mixture of cellular and dendritic grains with dendritic grains dominating, which in many cases are defined as cellular-dendritic grains[46, 182]. However, the cross section of the etched sample S200 shows indistinct cellular grains. No obvious evolution phenomena are observed and the cellular boundaries are rather unclear. There are some acicular shaped grains with the boundaries decorated by tiny precipitates as shown in Figure 4-3(d). The tiny precipitates are analyzed by EDX for their chemical composition later.

Figure 4-3 (a & b) Optical images of etched vertical cross sections showing: (a) S300 with cellular-dendritic grains within melting pool and some equiaxed grains near the centre of it, and (b) S200 with indistinct cellular grains within melting pool and darker intercellular regions, and (c & d) SEM images of etched vertical cross sections showing: (c) S300 with cellular grains with a planar zone (enclosed region with dotted line) and some polygonal grains below it, and (d) S200 with indistinct cellular grains and some acicular shapes with sharp grain boundaries decorated by tiny precipitates

The metastable grains and their evolutions are related to G/R ratio, where G is the temperature gradient and R is the solidification rate. At the melting pool boundary, a high G/R ratio (high G and low R) favors the formation of planar grains. Moving towards the melting pool centre, with gradually decreased G and increased R , the grains may evolve into cellular, cellular-dendritic, dendritic or even equiaxed grains. The equiaxed grains are mostly found in the center near the surface of the melting pool probably due to the isotropic heat dissipation [183, 184]. Similar mechanism was also reported in an SLM processed 300 maraging steel[73]. Because R equals scanning speed at the center of the melting pool, a higher G/R ratio in the slower scanning speed case favors the formation of cellular grains [46, 182] which phenomena is observed in the sample S200 with more cellular like grains. A lower G/R ratio favors the cellular-dendritic grains as observed in the sample S300.

The formation of microstructure can usually be explained by the theory of solidification [82, 185], according to which a rapid solidification is caused by constitutional undercooling. Constitutional undercooling is coupled with solute redistribution that helps achieve necessary undercooling in the solidification front (below liquidus temperature), where constitutional supercooling is determined by the G/R ratio. The morphology of liquid/solid interface is determined by the constitutional undercooling. However, this constitutional supercooling theory based on solute redistribution is challenged by a recent study[84], in which no micro-/nano-segregation can be found at the intercellular grain boundaries of an SLM processed SS 316L. Instead the study claimed that the phenomenon was probably due to largely suppressed element movement caused by a high cooling rate of about 1.7×10^6 K/s that was achieved by a pulsed laser at a high scanning speed of 5000 mm/s. This has put forward a need for a new theory to explain the rapid solidification behavior.

On the other hand, the grains formed during an SLM process are again related to the thermo-physical properties of the materials and the process parameters that are related to $G \times R$ value. Based on the solidification theory cooling rate is governed by $G \times R$ value. With increased $G \times R$ value, cooling rate increases. This explains the microstructural characteristics of the two samples printed at different scanning speeds. With a higher scanning speed, the cooling rate could be higher thus resulting in finer grains. With a lower cooling rate the grains formed are coarser as observed from the sample S200. Similar findings were reported by other researchers, including fine cellular or cellular-dendritic subgrains with sizes of $1 \pm 0.5 \mu\text{m}$ in many alloys [20, 84], e.g. SLM processed 316L[183] and maraging steel[99] that resulted from a rapid solidification process[79, 186]. The effect of slower scanning speed has been reported to lead to a lower cooling rate due to its higher energy input[20] that caused microstructure coarsening of the printed material. Meanwhile, a

higher energy input could influence the microstructure in the way of phase transformation, precipitation and release of residual stress through larger heat input from subsequent laser scanning resulting in more vague intercellular grains as reported [73, 81, 187]. Here, in the SLM processed EH36 steel the darker color in the sample S200 is probably due to a large amount of tiny precipitates, while the enlarged dark holes are due to over-etching. The microsegregation/precipitation behavior is discussed as follows.

As seen from Figure 4-3 the optical images of the etched vertical cross sections show that the sample S200 has darker intercellular boundaries compared to the relatively white intercellular/dendritic interface of S300. The SEM image in Figure 4-4(a) shows the sample S200 with possible tiny precipitates distributed along its indistinct cellular or acicular grain boundaries. The EDX spectrum of the tiny precipitates in Figure 4-4(b) illustrates mainly Fe and C and a small amount of Si, where the percentages may not be stoichiometric owing to the small size of the precipitates.

Figure 4-4 (a) SEM image showing indistinct cellular grains with precipitates distributed along intercellular region in the sample S200, and (b) EDX spectrum of the tiny precipitates in (a)

The observation and EDX analysis confirms there is precipitation behavior at 200 mm/s. The mechanism of precipitation is believed to be related to microsegregation that accompanies the rapid solidification process that makes precipitation energetically favorable. For the sample S200 longer retaining time at high temperature prompts a higher degree of element migration to the intercellular regions during this lower cooling rate scenario. The relatively white intercellular/dendritic interface of the sample S300 seems indicating the absence of microsegregation or there could be in such a level that is a much lesser amount. A greater degree of suppression of element redistribution is believed to be due to a higher cooling rate that incurs in the sample S300.

It has also been reported that a high cooling rate in an AM process can enhance the limit of trapped solutes in the crystal lattice, while a complete avoidance of microsegregation is not possible even up to 10^4 - 10^6 K/s[81]. The microsegregation phenomena are found in various AMed metals such as Mo segregation into intercellular grain boundaries in SLM processed SS 316L [16], Ti segregation into intercellular grain boundaries in LMD processed maraging steel [81], precipitates found in intercellular and interdendritic regions in SLM processed Inconel 738LC [81], etc.

Sparsely found polygonal grains ($\sim 2 \mu\text{m}$) located right below the white band region are believed to be within a heat affected zone where the grains may have undergone recrystallization upon the laser scanning of next layer. Chemical segregation or carbide precipitation may occur within this regime. The heat effect of the laser source is not only constrained to the heat affected zone. A higher amount of heat accumulation can be another consequence.

4.3.1 Coarsened Columnar Grains

As shown in Figure 4-5 for the both samples S300 and S200, the melting pools consisting of the coarse columnar grains are observed, where the columnar grain size can go up to 20 μm . Within the coarsened columnar grains are the differently oriented plate-like structures that delineate the columnar grain boundaries (as indicated by red arrows). Though the exact forming mechanism of those coarsened columnar grains is not clear yet, it is believed to be attributed to a lower cooling rate. The lower cooling rate is probably caused by heat accumulation from adjacent track or layer as reported[73]. The plate-like structures are formed with the martensite grains, where the coarsened columnar grains are due to some prior phase (e.g. γ austenite) before the martensitic transformation takes place within it. The array pattern of differently oriented martensite grains constrained inside the bigger columnar grains seems to demonstrate a $\gamma \rightarrow \alpha'$ transformation and details will be discussed in the following phase transformation section.

In some scenarios metastable submicron-sized cellular grains can grow into bigger cellular grains upon excess heat influence that comes from adjacent or next layer of laser scanning, which is observed by other researchers whereas timing and temperature are crucial for this merging to happen[90, 168]. The optical micrograph in Figure 4-5(a) shows one of the coarsened columnar grains in the topmost layer which assures its formation upon solidification.

Figure 4-5 (a & d) Optical micrographs showing coarse columnar grains with big plate-like martensite structure at different orientations inside (a) S300 and (d) S200 (both etched with 2% nital) , and (b, c, e & f) SEM images showing coarse columnar grains and some finer regions with acicular shape of grains in etched samples of (b) S300 and (e) S200 with respective enlarged views in (c & f) showing plate-like martensite structure at different orientations inside the coarse columnar grains from area A in (b) and area B in (e), respectively

Occasionally, some stray grains are found in between the arrayed cellular/dendrite grains. This most probably results from a varied local heat flow as reported in literature[74]. Microstructural variation within a melting pool is observed in the SLM processed EH36 steel implying the complexity of its thermal cycles, which is usually a consequence of rapid solidification. Meanwhile, novel meta-stable structures and phases may form along with the non-equilibrium solidification process as reported [70]. All those phenomena can be linked to process settings that result in tailored microstructures [50, 75], which can be realized by process adjustment through scanning speed, laser power, etc. In the SLM process this results in different heat input that can lead to

different thermal cycles thus tailoring for desired microstructures so as to desired mechanical properties of the printed samples.

4.4 Nano-scale Features

Martensite grains are observed using TEM with multiple types of grain with different morphologies. The TEM images (a-d) of the sample 300 in Figure 4-6 show the bright field and dark field microstructures disclosing lath, cellular and columnar shapes of grains and (e & f) of the sample S250 show the cellular and columnar shapes of grains.

A SAED (selected area electron diffraction) pattern inserted shows that each lath is a single crystal in a body centered cubic (BCC) phase as shown in Figure 4-6(a). It confirms that the martensite laths with lath width of about 100 to 200 nm are parallel-oriented in a band and the retained austenite (the dark field image with SAED inserted showing an FCC phase in Figure 4-6(d)) lies in between. The sizes of cellular grains and columnar grains are 500 - 1000 nm and up to 300 nm, respectively, which are located at different sites of the sample. The cellular grains seem slightly smaller than the cellular-dendritic grains that are observed using optical microscope and grow from the bottom of the melting pool to the center of it. The sizes of cellular grains and columnar grains in the sample S250 are up to 1 μm and 500 nm, respectively which are slightly coarser than those in the sample S300. The SEAD pattern inserted confirms a single BCC martensite crystal of each columnar shaped grain. There are no lath martensite and retained austenite found in the sample S250, which could be due to a higher amount of retained austenite in the sample S300 resulting

from the higher cooling rate. The dislocation density in the cellular shaped martensite grains seems to be higher than that in the columnar and lath shaped grains for both samples S300 and S250.

As shown in Figure 4-6(b & e) with the TEM images, the cellular boundaries of S250 are obviously vague that is in contrast to the clear cellular boundaries of S300. The inserted SEAD pattern (around cellular grain boundaries) confirms the BCC martensite matrix with carbide precipitates. The EDX measurement of the sample S250 shows a higher concentration of Cr element around the intercellular region and slightly higher concentrations of Ni, C and Si as well compared to the inner grain region. The vague cellular grain boundaries are probably due to an increment of dislocation density resulted from microsegregation or nanoprecipitates to the boundaries.

From the above observation, the columnar shaped martensite grains of a few hundreds of nm are transformed from some prior grains as shown in Figure 4-6(c). The TEM observation is consistent with the above optical graphs (coarsened columnar grains partitioned by plate-like martensite structures). For the type of cellular shaped martensite grains the transformation mechanism is not so clear. It could be dictated that the cellular shaped martensite grains have partitioned the prior austenite cellular grains (which is larger in size) upon solidification while both are in similar cellular shapes but differing in scale. It may follow a conventional hierarchical transformation scheme as dictated in a DMD built AISI 4340 low alloy steel that was reported similar to a conventional heat treat process[18]. They also disclosed for the first time in the literature a Kurdjumov-Sachs relationship between martensite and austenite in the DMD processed 4340 steel and other similar findings were reported earlier in a DMD processed tool alloy steel [188]. However, the cooling rate of SLM process is higher than that of DMD process.

Figure 4-6 TEM images of (a-d) sample S300 showing (a) bright field microstructure, (b) cellular grain structure, (c) columnar grain structure, and (d) dark field microstructure, and (e & f) sample S250 showing (e) cellular grain structure and (f) columnar grain structure

A direct precipitation of martensite grains with no prior austenite phase formation cannot be ruled out. Such early precipitation of posterior phase was reported by Kadiri et al. on a LENS built AISI 4140 low alloy steel. A direct nucleation of primary lower bainite from the prior δ ferrite grain boundaries that cross a few γ prisms is found while it is not following the normal transformation mechanism of δ ferrite $\rightarrow$ γ austenite $\rightarrow$ bainite [70]. It could be also the same scenario here for the SLM processed EH36 steel, where a very high cooling rate renders that a high temperature phase has no time to form and the molten metal is directly solidified into the martensite crystals.

This is based on the assumption that the very high cooling rate promotes the formation of novel and metastable structures and phases as well as a new transformation mechanism in due.

There is another assumption that the martensite structures within the SLM processed steel samples could be away from the conventional packet/block/lath hierarchical structures with support by several findings, e.g. it has been reported that lath martensite cannot be formed due to this high cooling rate in an LMD processed maraging steel [189]. Jäggle et al. reported martensite blocks (boundaries with misorientation above 50°) confined to a single dendrite that belongs to the same prior austenite grains with retained austenite between dendrites in the LMD built maraging steel but saying that it was not following the conventional hierarchical structure [189]. They also claimed the stabilized austenite in the interdendritic regions due to solute segregation of Ti, Mo and Ni.

However, the lath structure and smaller size of columnar martensite grains are indeed identified in another researcher's work in spite of their micro cellular-dendritic morphologies[73]. The martensite transformation mechanism in AM built metals remains largely unknown and the perspective is rather uncertain. However, the variety in martensite grains and shapes is believed to be related to their individual thermal cycles, which evidences the complexity of melting pool behavior. As a usual practice there are two types of martensite in steels: lath martensite and needle-shaped martensite. Low carbon steel usually takes the shape of lath martensite that is less brittle. The high strength and fair toughness of lath martensite, that have long been believed characterized by a high density of dislocations, make direct applications possible. However, a high density of dislocations is only found in very low carbon steels [177]. A general rule may apply that the higher

the cooling rate, the finer the structures obtained hence for a better strength and ductility [190, 191].

The retained austenite (RA) is found in the sample S300 as shown in the TEM image in Figure 4-6(d). The retained austenite grains lie in between the martensite laths. These RA grains have no much difference from a conventionally quenched RA that usually coexists with the lath martensite grains and lies in between. However, it has been reported that the position of RA in a laser metal deposited maraging steel mainly exists at the intercellular grain boundaries [189]. Here, EBSD analysis is used to further investigate the nano-scale features of the samples from a larger point of view including the various martensite grains, retained austenite and possible carbide precipitates within the matrix.

An EBSD image with an inverse pole figure (IPF) shown in Figure 4-7 again clearly depicts a region with the big plate-like martensite structures distributed within some band substructures as shown in the lower part of the image with the band size of about 7 - 20 μm . The boundaries of those bands are delineated by various colored plate-like or block martensite crystals that are consistent with optical microscopic observation. The patches in different colors demonstrate different crystallographic orientations, which clearly show that the plate-like or block shaped arrays are in certain angles with the band boundaries and there are a few different colors within one band. This could be a sign of some prior phase of grains formed first during solidification before martensitic transformation taking place within them. The predominant phase is a BCC structure and the grain sizes measured mostly fall around 1 μm . Due to large step size (0.2 μm) used or a similar crystallographic orientation of martensite crystals the measured grain size could

be reduced if a smaller step size is used. In the upper part of the image, some acicular shapes dominate. From the image, there is no obvious preferred orientation of crystal growth observed.

Figure 4-7 EBSD image of sample S300 with IPF

A small amount of FCC phase is detected, which should belong to the retained austenite with the grain size ranging from about 0.2 to 0.4 μm. The retained austenite grains are spread at different locations within or along grain boundaries, which again may be reduced further by using smaller step size such as 0.1 μm. A trace amount of Fe₃C phases is also detected by EBSD scanning where it scatters mainly along the grain boundaries. The microstructure evaluation through EBSD depicts the elongated columnar shaped grains with the overall length up to a few tens of microns in accordance to the melt pool size measured from optical microscopy.

This is the first time that the detailed features of the SLM produced EH36 steel samples are reported including the martensite grain morphologies and orientations, retained austenite, and possible precipitates using the method of TEM, EBSD, OM, SEM and XRD.

4.5 Mechanical Properties

As described above tuning of the process for a higher densification of the built samples can be done through adjusting energy density (ED), where some of the key process parameters are correlated with this single factor, $ED = P / (S \times H)$, where P is the laser power, S is the scan speed and H is the hatch spacing. With different energy density or heat input, the amount of heat that enters the scanning regime or laser spot region is different, which affects the melting pool behavior and the cooling rate. As a result the densification and final microstructure could be tuned.

In this work the laser power is initially fixed at 175W and the layer thickness is fixed at 50 μ m for all the cubes built by a 3 x 6 matrix where varying scanning speed and hatch spacing are conducted to find the optimized parameters. The results of densification versus energy density are shown in Figure 4-8, which shows that the material density increases with the energy density until a certain level of about 8 J/mm², beyond which the densification appears to have reached a maximum (>99%, indicated by the grey arrow).

The high densification values fall within a wide range of ED from about 8 J/mm² to 22 J/mm² (as indicted by the green arrows) in Figure 4-8. This could be linked to the downside effect of smaller hatch spacing and lower scanning speed as explained in the literature[43, 44], which indicates that

an excess energy input can turn the melting pool into an unstable state thus deteriorating the densification.

Figure 4-8 Densification versus energy density for EH36 samples

Figure 4-9 illustrates the effect of scanning speed on densification. At hatch spacing of 0.08 mm, 0.09 mm and 0.13 mm, the slower the scanning speeds, the higher the density is. The maximum densification happens at the scanning speed of 100 mm/s. However, at hatch spacing of 0.10 mm, 0.11 mm and 0.12 mm, the highest densification of up to about 99.5% is achieved at the scanning speed of 200 mm/s instead. The contour plots and 3D response surfaces for the effect of scanning speed and laser, scanning speed and hatch spacing to densification are shown in Figure 4-10a&b and Figure 4-11a&b, respectively.

Figure 4-9 Density of EH36 samples with respect to scanning speed

The trend of densification versus hatch spacing is not obvious and there seems to be an optimal hatch spacing at each scanning speed. For example, an optimum hatch spacing is about 0.12 mm with the scanning speed of 300 mm/s, which is shifted to a smaller hatch spacing of about 0.11 mm at the lower scanning speed of 200 mm/s. The graph illustrates that the process for the EH36 steel can be optimized by using scanning speeds lower than 300 mm/s. The results from varying scanning speed and hatch spacing can be used as a guideline for processing the EH36 steel.

Then the effect of laser power was studied with P varied from 145 – 240 W and scanning speed from 100 – 400 mm/s and layer thickness fixed at 50 μm and hatch spacing fixed around 0.11 mm. The densification of the samples vs laser power and scanning speed is listed in Table 4-1. Obviously density increases with laser power and decreases with scanning speed. This is again demonstrating a proportional correlation between densification and energy density. The effect of

preheat temperature on densification at different scanning speeds is shown in Table 4-2. Obviously, increasing preheat temperature to 200°C effectively improves densification. All the samples up to a scanning speed of 300 mm/s achieve a very high density (> 99.4%).

Table 4-1 Effect of laser power on densification

Densification (%) Scanning speed (mm/s)	Laser Power (W)	145	155	165	175	185	195	210	225	240
100		98.0	99.1	99.5	99.3	99.5	99.6	99.7	99.6	99.7
200		99.1	99.4	99.2	99.4	99.4	99.5	99.6	99.6	99.7
250		98.9	98.8	99.2	98.8	98.7	99.1	99.2	99.5	99.6
300		98.1	98.7	97.9	98.2	97.7	98.9	98.6	99.1	99.4
400		98.6	98.4	97.2	96.7	95.6	97.1	97.6	97.8	97.0

Table 4-2 Effect of preheat temperature on densification

Scanning speed (mm/s)	Preheat temperature (°C)	Densification (%)
100	200	99.5
200	200	99.9
250	200	99.7
300	200	99.4

Figure 4-10 a) the contour plot, and b) the response surface showing the effect of laser power ($p = 0.849$) and scanning speed ($p = 0.000$) to densification

Figure 4-11a) the contour plot, and b) the response surface showing the effect of scanning speed ($p = 0.000$) and hatch spacing ($p = 0.995$) to densification. Note: The p value is calculated by one-way ANOVA using Minitab software and $\alpha = 0.05$. Scanning speed has significant effect while

Microhardness is measured on the polished vertical cross sections of the samples with a measuring interval of 0.05 mm. Figure 4-12 shows the microhardness readings of the samples S300 and S200, which are recorded starting from near the top surface of the samples downward along the vertical build direction with the average values of $HV_{35}1_{0.05}$ and $HV_{35}4_{0.05}$ for the samples S300 and S200,

respectively. The sample S300 shows a larger degree of fluctuation. The coefficient of variation (standard deviation over average) is 0.06 and 0.08 for sample SS200 and SS300, respectively. The upmost layers of the both samples hold high hardness values of around $HV_{0.05} 400$, which represents the freshly solidified layer hardness. Below the top layers, both tempering and aging factors result in the variations of the hardness values along the build direction. The hardness of the sample S200 decreases obviously, which is due to tempering caused by the laser heat source through subsequent scanning. The overall hardness for the sample S200 fluctuates less, which demonstrates a more homogeneous microstructure of the sample. This homogenization could be linked to the indistinct cellular structures resulted from the higher heat input. Other phases like carbide and ferrite may be formed as well during the printing process due to very local thermal cycles.

Figure 4-12 Micro-hardness profiles of samples S200 and S300 measured along vertical cross sections

The theoretical hardness of a full martensitic structure containing 0.18% of carbon is around 450 Hv [192], which is higher than the results measured from the EH36 steel in this study possibly due to a certain percentage of ferrite or retained austenite formed in the samples. However, the hardness values of the AMed samples are higher than that of water quenched EH36 steel (311 HV) with above 90% martensitic transformation [66] by more than 10%. The grains in the printed samples examined under TEM are even larger than the conventional martensite lath/packet sizes. Meanwhile, the low angle cellular grain boundaries may not inhibit dislocation movement as good as the high angle martensite blocks or packets. The relatively higher hardness of the AM samples is possibly due to a combined strengthening effect from solid solution, dislocation and highly dispersed precipitate.

The ranges of tensile strength (UTS), yield strength (σ_y) and elongation of the samples S250 and S300 are shown in Table 4-3 against a quenched EH36 steel equivalent. The average UTSs of the samples S250 and S300 are 1040 MPa and 1028 MPa, respectively. The yield strengths of S250 and S300 are taken from a 0.2% offset and averaged at 943 MPa and 864 MPa, respectively, with their respective average elongations of about 5% and 6%. The sample S300 shows higher ranges (maximum – minimum) than the sample S200 for the UTS (R = 114 MPa vs 5 MPa), σ_y (R = 174 MPa vs 9 MPa) and the ranges are used to illustrate the degree of fluctuation. The UTS results shown here are measured using sub-sized specimens, which are comparable with the reported results (1100 MPa) measured using standard tensile coupons under similar conditions[25]. The slight lower UTS values of the samples from this study could be due to the size effect. The SLM built EH36 steel samples with a near full martensitic structure possess slightly higher UTS and yield strength than the water quenched EH36 steel equivalent by about 13 - 15% and 15 - 19%, respectively.

As discussed previously the high tensile and yield strengths of the AM built metals benefit from the finer subgrain structures resulted from high cooling rate along with the rapid solidification process [16]. For this high strength EH36 steel built through the SLM process the high strength comes from the martensitic structure combined with possible solid solution, dislocation and precipitation strengthening [190]. The martensite grains of either cellular, columnar or lath shape found from the AMed samples used are not smaller than the conventional martensite lath structure (100 – 200 nm). However, it is believed that it is the martensite packet size that determines the strength of the steel instead of martensite lath size. The elongated cellular martensite grains are believed to be a downside effect on the strength. As explained above, the size of a cellular is around 1 μm with its length in tens of microns, which is larger than the conventional martensite lath or block/packet. And those low angle cellular grains are believed to be weaker in strength compared to the conventional martensite packet, block and lath hierarchical structures. The low level of retained austenite may contribute to the certain aspects including ductility. Even though the elongations of the AMed samples are relatively low, a 5% elongation for martensitic steels is believed to be decent [190].

The yield strength for the sample S250 is higher than that for the sample S300, by about 9%. However, the tensile strength of the sample S250 is only slightly higher than the sample S300, by about 1%. The smaller difference in the tensile strength could possibly be due to the higher yield strength of the sample S250 so that the plastic deformation or work hardening stage is shortened especially for the smaller tensile samples [76, 122, 193, 194]. Meanwhile, the smaller grains could possibly give a higher work hardening exponent as demonstrated by the sample S300 with the highest tensile strength of about 1101 MPa but a smaller elongation of about 6%.

Table 4-3 Tensile properties of samples S300 and S250

Sample	UTS (MPa)	$\sigma_{y,0.2}$ (MPa)		Elongation (%)
		measured	calculated	
S300	987-1101	763-937	930	3.5-6
S250	1037-1042	938-947	865	4.5-7
Quenched EH36@1000°C	906	795		

Moreover, the measured yield strengths are away from the theoretical values converted from the measured microhardness using the empirical equation: $\sigma_s = 3.28 \times HV - 221$ [130, 131], which are 930 MPa ($HV_{0.05} = 351$) and 865 MPa ($HV_{0.05} = 331$) for the samples S300 and S250, respectively. For the sample S250, the measured value is about 7% higher than the calculated, while for the sample S300, the measured value is about 8% lower than the calculated, whose differences could be related to the local defects such as pores, microstructure variation and anisotropy (with high aspect ratios for cellular grains) that are resulted from the rapid solidification process.

Again the detrimental effect of microstructural inhomogeneity is reflected from the more varied data for the sample S300. The pores acted as a stress concentration can lead to premature failure. More consistent results obtained from the sample S250 benefit from a higher densification containing a lesser amount of detrimental pores. This could be verified by the fractured surfaces for both the samples. A higher density is helpful to rule out the porosity deterioration effect, thus improving the mechanical properties, but it could be a trade-off from grain coarsening resulted from a lower cooling rate accompanied with a slower scanning speed. Since grain refinement is an important strengthening mechanism for steels it is always good to have a comprehensive

understanding from process, densification to microstructures and their correlated mechanical properties for a real “optimal” process.

The fractured surface morphologies are examined using SEM for both the samples. A large number of shallow dimples and micro-voids are found on the fractured surfaces of both the samples as shown in Figure 4-13(a-f) and Figure 4-14(a-f). For the sample S300, porosities, lack of fusion containing un-melted or partially melted powder particles (as indicated in yellow arrows) are all observed. A large flat surface is shown in Figure 4-13(b) aside a big void of lack of fusion that is spread with the shallow dimples on the fractured surfaces. The dimple size is around 1 μm , which is comparable with the cellular grain size. The large lack of fusion void with the size more than 100 μm possibly acts as a stress concentration for a crack to initiate from. There is also de-bonding of melting pool boundaries (MPB) identified in the sample as shown in Figure 4-13(e) demonstrating brittle fracture features. Moreover, some equiaxed grains attached to the surface of the partially melted powders as shown in Figure 4-13(d) can be due to the growth promoted by the unmelted powder in the melting pool, which act as nucleation sites during solidification[187].

Figure 4-13 SEM images of fractured surface for sample S300: (a) overview of fractured surface with lack of fusion and unmelted powder as indicated with yellow arrows, (b) enlarged view of area A in (a) where a big flat surface is covered by shallow dimples spread on it, (c) detailed view of the shallow dimples shown in (b), (d) partially melted powder particles with equiaxed grains, (e) melting pool boundary de-bonded during tensile loading, and (f) detailed view of dimples shown in (c)

Compared to the sample S300, the fractured surface of the sample S250 shows a lesser amount of defects but in similar types, that are mainly porosities and lack of fusions, while smaller amounts of unmelted powder and inclusions are observed in the lack of fusion regions with some de-bonding of the melting pool boundaries as shown in Figure 4-14(a, d & e) (as indicated by the yellow arrows). Meanwhile, intercellular debonding is observed in the sample S250 as shown in Figure 4-14(f) (as indicated by the yellow arrows) with the shallow dimples (around 1 μm) spreading over their surfaces and the dimples are possibly initiated from the intercellular precipitates. Figure 4-15(a) shows that some small particles with the size of a few hundred nanometers are dispersed along the de-bonded melting pool boundaries. The elemental analysis by

EDX as shown in Figure 4-15(b & c), confirms the main elements of Mn, O, Fe, C, Si and Cr for the larger particles and C and Fe for the smaller particles.

It is said that totally avoiding lack of fusion for the SLM process is difficult. All those observations have proved that the laser scanning at the speed of 250 mm/s is able to melt the powder more efficiently than the laser scanning at 300 mm/s and thus resulting in a better densification.

Figure 4-14 SEM images of fractured surface for sample S250: (a) overview of fractured surface where lack of fusion and unmelted powder particles are indicated with yellow arrows, (b) enlarged view of area A in (a), which shows a number of dimples, (c) detailed view of dimples shown in (b), (d) inclusions, (e) melting pool boundary de-bonded during tensile loading, and (f) intercellular debonding with shallow dimples on the surface

The small particles are believed to form during the powder melting/solidification or tempering by subsequent laser scanning, which weaken the intercellular or melting pool boundaries. A trace

amount of oxygen inside the building chamber may provide an oxygen source to form metal oxides. The phenomenon was explained in literature that the metal oxide droplets later form spherical particles that were not compatible with the metal melt and dispersed inside the matrix during cooling[21]. It was reported that oxides and impurities are intended to stay at melting pool boundaries, which may affect interlayer bonding and form cracks[88] leading to premature failure. The carbide may precipitate upon solidification or subjected to heat source from adjacent or next layer laser scanning. The areas near the melting pool boundary or fusion line experienced the highest temperature become a heat affected zone (HAZ) where the precipitates could form. The precipitates intend to accumulate at the intercellular grain boundaries and melting pool boundaries.

Figure 4-15 (a) SEM image showing melting pool boundary detached from fractured surface of sample S250, (b) EDX spectrum of particles measured at spot 1 in (a), which indicates a high level of oxygen and other elements, and (c) EDX spectrum measured at spot 2 in (a), which depicts high concentrations of carbon and iron

The fracture mechanism and modes for the AM built materials have been elaborated from different aspects including intergranular fracture, transgranular fracture (ductile or brittle), melting pool boundary fracture, etc. [16, 76]. The large defects such as lack of fusion, holes or voids ranging up to a few hundred of microns can be the critical points for crack initiation. Interestingly, mainly ductile dimples are observed with a small fraction of brittle fracture and melting pool debonding that is weakened by small particles or second phase precipitates. The fracture is believed to be mainly in a transgranular mode in which a large amount of small particles located at the bottom of the dimples could be the sites for ductile failure to occur. The sizes of the dimples are at submicron to micron level, which are comparable to the cellular/dendrite sizes. In general, the defects induced from the SLM process do not deteriorate the tensile properties of the built samples with high densification in a large scale.

4.6 Summary

The microstructure, densification and mechanical properties of EH36 steel samples additively manufactured through an SLM process were systematically studied by varying laser scanning speed and hatch spacing using a checker board scanning strategy. The effects of laser power and preheat temperature on densification were also presented.

A high density of above 99.4% in the printed samples was achieved at a lower scan speed of 200 mm/s and a smaller hatch spacing of 0.11 mm. The densification showed a nearly linear relationship with the scanning speed, i.e., the lower the scanning speed, the higher the densification

was. Increased laser power also benefited the densification, but increased preheat temperature was more efficient in improving the densification with all the samples having achieved very high densification ($> 99.4\%$) up to the scanning speed 300 mm/s. An average melt pool size of 70 - 90 μm was observed from the sample S300 that had mainly cellular-dendritic grains. With a higher heat input corresponding to a lower scanning speed, a deeper and wider melt pool was formed in the sample S200 but the cellular structure became indistinct possibly due to phase transformation, precipitation or stress relief. Due to a large heat input there was a larger chance of chemical segregation or precipitation to happen at the intercellular grain boundaries. A lower scanning speed also led to coarser grains and lower microhardness in the samples. A very high cooling rate suppressed the martensitic transformation leading to a high level of retained austenite in the sample S300 that experienced the highest cooling rate during printing.

Single crystal martensite grains in cellular, columnar and lath shapes were observed using TEM, which contained retained austenite grains and possible carbide precipitates as revealed through EBSD. The martensitic transformation mechanism possibly followed a conventional packet/block/lath hierarchical pattern, though it remained unclear. The variety of martensite grains dominated by the elongated cellular or acicular features together with the columnar and lath shapes demonstrated a complex local heat flow during a rapid cooling process.

The tensile properties of the samples were correlated to their microstructures such as grain size, precipitation, lack of fusion, void, etc. and hence they were printing process related. A high yield strength was believed to be the reason for the smaller difference to UTS of the sample S250 at a lower scanning speed with reduced plastic deformation or work hardening compared with the larger difference of yield and UTS for sample S300. The instable tensile properties of the sample

S300 were due to the intrinsic defects like pores. The calculated yield strengths of the printed samples based on their microhardness values were within a 10% variation from the measured values, which indicated that the specimens were quite tolerant to the detrimental effect of defects.

Chapter 5 Fatigue Behavior of SLM Built EH36 Steel Samples via Experimental and Numerical Studies

5.1 Experimental Results and Discussion

5.1.1 Microstructure

Figure 5-1a&b shows the microstructure of the etched sample printed at scanning speed of 300 mm/s. The melting pool is clearly seen with fine cellular-dendritic grains grown inside from the bottom to the centre of it. The size of the cellular-dendritic grains is $1 \pm 0.5 \mu\text{m}$ and the depth of the melting pool is 70 -100 μm . Lath martensite or other shapes may align in different directions inside the cellular-dendritic grains, which has been confirmed from our previous work[173].

Figure 5-1c&d shows the etched microstructure of the sample SS400 consisting of mainly martensite lath structures. However, when the scanning speed is lowered to 100 mm/s, substantial changes of microstructure are observed. As seen from Figure 5-1e, the overall microstructure is dominated by fine ferrite grains with a size of 2 - 3 μm (finer grain bands). Within the brighter bands, the ferrite grains can be further coarsened to about 5 – 7 μm . Martensite lath patches are found sparsely dispersed in the ferrite matrix with a quantity of less than 30%. Intact coarse martensite lath shape is only observed in the top 3 layers as shown in Figure 5-1f where their transformation within large prior columnar grains is clearly seen (the large pores are due to over etching and the medium sized holes are indented holes from the micrhardness test). Without further heat influence coming from repeated laser scanning, the contrast reflects microstructure

evolution happened to the below deposited materials. The undergone evolution is caused by the heat coming from laser scanning of next layer or adjacent tracks. Obviously, the heat induced temperature is high enough to cause a large extent of phase transformation and recrystallization. Growth of ferrite grains along the prior austenite grain boundaries is triggered, which delineate the columnar shape of the prior grains with the size of 10 to 20 μm as shown in Figure 5-1g. The martensite laths are either transformed into ferrite phase or tempered.

In some areas when the temperature is above austenization temperature, austenization can happen and γ phase may transform back into martensite again during cooling depending on the local cooling rate and cooling temperature. This is proved by the observed martensite lath structure within 2nd and 3rd top layers where the large heat effect from 1st top layer is believed to be able to extend far into the below layers and raise the temperature of the below 2 layers to above austenization temperature where austenite to martensite transformation happened two or three times. The heat effect further into deeper materials is believed to only make recrystallization and tempering possible. The resultant graded structure in terms of the exhibited fine and coarse ferrite bands (shown in Figure 5-1(e)), remanent martensite lath lands, demonstrates varied local thermal cycles. The higher heat input also creates a wider and deeper melting pool. A large amount of tiny precipitates in nano to submicron sizes is formed as shown in Figure 5-1g in black dots (indicated using yellow arrows) located along martensite lath/ferrite grain boundaries or within them.

The large heat effect makes precipitation energetically favourable and more discussion can be found from Chapter 4. Overall, the higher heat effect at slower scanning speeds induces larger heat affected zone (HAZ), higher degree of tempering effect to previous deposited materials,

coarser and more homogeneous microstructure or even phase transformation & recrystallization. More detailed discussions regarding the microstructure forming mechanism and the characterization of other samples printed at different scanning speeds can be found in our previous work[173]. The in-process thermal cycle results in a mixed structure with tempered martensite and fine ferrite at a random distribution, which may render macroscopically favoured mechanical properties coming from both of them and it is proven by the following obtained tensile and fatigue mechanical properties.

Figure 5-1 Optical micrographs showing the microstructures of vertical cross sections of (a&b) sample SS300: (a) overview and (b) enlarged view, (c&d) sample SS400: (c) overview and (d) enlarged view, and (e-g) sample SS100: (e) overview, (f) top 3 layers and (g) enlarged view of interface between top 3 layers and below layer

Defects, mainly pores, with their shapes and sizes are characterized for all the samples and shown in Figure 5-2a-e with the polished samples at their vertical cross sections for the samples SS100, SS200, SS250, SS300 and SS400. It is clearly seen that the sample SS400 has the highest porosity level with rather irregular pores and the sample SS100 has the lowest porosity level. The

densification is 99%, 98%, 94%, 94%, and 91% for the samples SS100, SS200, SS250, SS300 and SS400, respectively, analyzed by Image-J software and the density gradually decreases with the increase of scanning speed.

The pores are mostly lack of fusion in narrow elongated shapes (indicated in (a)) or in triangular-like shapes (indicated in (b) with or without elongated narrow portions or tails). Enlarged and more irregular shape of pores and mostly with multiple sharp corners and uneven contour profile are found in samples SS250, SS300 and SS400 (indicated in (e)) which is mainly due to non-enough laser penetration at higher scanning speed. The overall length can range up to 500 - 1000 μm . Unmelted particles are observed increasingly in the samples SS250 followed by SS300 and SS400, as indicated in Figure 5-2b-e with white arrows. The unmelted particles increase roughness/unevenness of the pore surface. It may enhance stress concentration on top of others when connected together. Inclusions in the size of tens of microns are revealed in the fractured surface under SEM in the later part. All those defects can lead to complex stress state singly or interacted and lead to fatigue crack. The higher energy input resulted from lower scanning speeds ensures a deeper laser penetration that improves the densification and contributes to improved mechanical properties of the as-built samples as proved in the following part of tensile behavior and fatigue behavior. However above characterization of pores is based on 2D metallography. The actual morphology of the pores in 3D may not be accurately reflected. The CT scan image in Figure 3-8 shows the 3D morphology of the pores formed in the sample SS300, which overviews the pore size and distribution. Most of the pores are in complex shapes in a scale of tens of microns to a few millimeters. Some pores or portion of the pores are in elongated shapes, which are estimated to be as narrow as tens of microns in the transverse directions of the pores. The porosity level

measured by the CT scan is around 3 vol%, which is slightly lower than that measured by means of the ImageJ software probably due to the resolution limitation of the CT system used in this study.

Figure 5-2 Optical micrographs of polished samples at vertical cross sections showing (a) SS100 with lowest porosity level, and (b-e) increased porosity levels in samples (b) SS200, (c) SS250, (d) SS300, and (e) SS400

5.1.2 Tensile Behavior

The effect of scanning speed on the tensile properties and microhardness of the printed samples is shown in Figure 5-3. As seen with decreasing of scanning speed, the tensile strength, yield strength and elongation of the samples increase. However, microhardness reduces with decreased scanning speed. From the discussion of Chapter 4 this is due to the thermal effect which has a higher tempering effect from higher heat input to the martensite microstructure induced by slower

scanning speed [173]. The tempered martensite structures may vary in lattice strain, solid solution, dislocation density, etc. from less tempered martensite. Formation of fine ferrite grains as observed from above OM images contributes to the lowered hardness to sample SS100. In other samples small amount of ferrite grains may form due to varied local thermal cycles, such as the polygonal grains found within the HAZ in sample S300 known from Chapter 4. The slightly lowered Hv of the sample SS400 due to softer phases possibly retained austenite and ferrite, etc. was also discussed together with some others' similar findings [18, 178-180]. From literature the higher amount of retained austenite in AM processed steels are mainly due to factors that can 1) suppress $\gamma \rightarrow \alpha'$ transformation (strain at high-angle grain boundaries, γ stabilizing elements, environment of powder & part manufacturing) 2) reduce M_s temperature (finer grain structure, higher dislocation density) [180]. The martensitic transformation was proved in the SLM processed EH36 steel by XRD patterns, TEM and morphology observation using OM and SEM. The higher hardness value at the upmost layer (above HV400_{0.05}) was coming from brittle martensite structure followed by a drop to around HV350_{0.05} below the top layer as disclosed in our previous discussion in Chapter 4.

Microstructure governs material's macroscopical behavior, such as chemical compositions, phases, grain size & its orientation, defects (solid solution, elemental segregation, dislocation, grain/sub-grain boundaries and others), crystallographic orientation, etc. It was disclosed from our previous discussion in Chapter 4[173] that the high strength and ductility of the low carbon steel (grade EH36) printed at scanning speed of 200 – 300 mm/s is mainly due to the tempered martensitic structure. The tempering is happened through auto-tempering during its transformation or by the heat coming from later laser scanning. The tempered martensite structure could be further

strengthened by precipitation, solid solution and dislocation coupled with this rapid solidification process. Tempered martensite is more applicable than fresh martensite (without tempering) due to its good strength and ductility. Here when the scanning speed is lowered to 100 mm/s, dual phase structure including fine ferrite and highly tempered martensite is obtained. This dual phase structure gives a high strength of about 1000 MPa and a fair elongation of up to 10.5% and Hv280. Other microphases like retained austenite and bainite, etc. could further enhance the resultant mechanical properties. Retained austenite (confirmed by XRD and TEM from our previous work) and other softer phases within SLMed EH36 possess better plasticity that may contribute to tensile and fatigue behavior in different aspects.

For the sample SS100 the measured yield strength is lower than the theoretically predicted value by about 5% using an empirical relation $\sigma_y = 3.28 \times \text{Hv}^{0.22}$ [130, 131], but 30% lower for the sample SS400, where σ_y is yield strength and Hv is microhardness. Possessing finer grains and strong martensitic structure, sample SS400 from higher cooling rate didn't achieve higher strength, however the highest strength was achieved by sample SS100 which contains coarser and softer ferrite grains. All these demonstrate the detrimental effect of pores to the mechanical properties. Tensile behavior shows to be pore-sensitive, especially for the porous samples like SS400 that contains 9% of porosity. Others like microstructure variation and anisotropy (with high aspect ratio for cellular grain and texture, etc.) that are resulted from the rapid solidification process may render bulk data away from the calculated [100].

Figure 5-3 Effect of laser scanning speed on UTS, yield strength, elongation and microhardness of the printed EH36 steel samples

5.1.3 Fatigue Behavior

5.1.3.1 S-N curve

The S-N curves built under different stresses (in max. stress) for the samples SS100, SS200, SS250 and SS300 are shown in Figure 5-4. The S-N curves are fitted using a power law Basquin relation in the form of $\sigma_a = \sigma'_f (2N)^b$ [147, 195, 196], where σ'_f is the fatigue strength coefficient that is approximated to be the true tensile strength and b is the fatigue strength exponent that is the slope of the logarithmic S-N curve. Due to a high porosity level of sample SS400, its fatigue behavior is

not studied in this paper. The overall trend of the S-N curves demonstrates a porosity dominated fatigue behavior. As seen from the figure the higher the porosity level, the lower the fatigue life cycles achieved by the samples are. The data show more scattering in the higher cycle regime compared to that at lower cycle that is due to the more serious influence of pores on higher cycle fatigue behavior.

A finer microstructure is believed to be able to resist fatigue initiation better than coarser grains due to its higher strength[197], larger amount of microstructural obstacles from grain boundaries and possible higher degree of crack tortuosity[106] within early stage of crack propagation. Known crack initiation stage dominates total fatigue life of HCF. Here finer grain structure didn't give longer fatigue life and it seems the above mentioned merit of finer microstructure is overwhelmed by other adverse effect. Those are mostly defects like internal pores, inclusions and others as these are strong stress concentrations that can usually cause severe stress concentration and lead to crack formation easily. Larger the quantity, size and complexity more complex and severe stress state could be so as to the faster crack growth rate. Another adverse effect of pores is reducing the loading areas resulting in elevated nominal stress/strain being applied to the samples during the actual test. All those are the commonly observed phenomena for AMed metal materials [21, 147]. Further examination of the fractured surfaces of the EH36 steel samples discloses the crack initiation at pores, unmelted particles and other defects with their detrimental effects confirmed to fatigue. Possessing less porosity, high strength and fair ductility (equivalent to higher toughness) sample SS100 gives potentially good fatigue properties. The higher strength allows lower elastic strain than lower strength upon same fatigue loading. With better plasticity the stress concentrated ahead of crack tip can be effectively relocated and crack growth can be delayed. The detrimental

effect of pores can be diminished with fair plasticity as well. All these prolong the sample's total fatigue life. Therefore, the fine structure together with less porosities guarantees good fatigue performance. For the AMed metals, the large size & amount, ubiquitous distribution and complex shape of pores become a crucial factor to their fatigue behavior, which may lead to different failure modes. Moreover, the microstructure variation and anisotropy within the AMed metals may render fatigue behavior further away from those deductions.

Figure 5-4 S-N curves of samples SS100, SS200, SS250 and SS300

5.1.3.2 Prediction of Fatigue Limit

Basquin Relation

The fatigue limits corresponded to a life of 10^6 cycles (in stress amplitude) are calculated from the fitted curves using the Basquin relation shown above and tabulated in Table 5-1. The sample SS100 gives a highest fatigue limit of 175 MPa followed by SS200, SS250 and SS300. No absolute positive proportional relationship is observed between fatigue strength and precipitation hardening owing to the complex microstructure-precipitate behavior as reported in [198, 199]. The σ'_f and b values obtained for the SLMed EH36 steel samples are away from a conventionally manufactured equivalent [161] and their fatigue behavior may challenge the usual practice, which has been raised for various AMed steels and alloys by many reports [136, 147]. Hence, new mathematical models and correlations need to be established.

Empirical Mathematical Models

Empirical correlations regarding fatigue limit (σ_{FL}) for traditional steels and alloys are used for AMed counterparts, e.g. $\sigma_{FL} = 1.6 Hv \pm 0.1Hv$ (mean stress = 0, $R = -1$), where σ_{FL} is the fatigue limit and Hv is the Vickers microhardness [22]. This empirical equation is valid for $Hv \leq 400$. For hard steels with $Hv \geq 400$, the fatigue limit is not following this linear relationship but much lower than it. However the fatigue limit calculated using this formula is much away from experimental results. The ratios of σ_{FL}/Hv for the SLMed EH36 steel samples are far below 1.6 as seen in Table 5-1. They fall into the range of 0.4 – 0.8. With microhardness measured away from pores, it seems there are other factors highly influencing the usual linear proportional relationship governed for traditional steels, most probably the intrinsic pores and other defects from AM process with possibly the grain orientation effect as discussed in the following part.

Table 5-1 Predicted fatigue limits and σ_{FL}/H_v & σ_{FL}/UTS ratios for samples SS100, SS200, SS250 and SS300

Sample	Effective crack size (μm)	UTS (MPa)	$H_{V0.05}$	σ'_{FL} by Murakami, R = -1(MPa)	σ_{FL} by curve fitting, R = 0.1(MPa)	σ_{FL}/H_v (R = -1)	σ_{FL}/UTS (R = -1)
SS100	168	971	288	248	175	0.8	0.2
SS200	313	921	334	249	150	0.6	0.2
SS250	406	891	345	244	123	0.4	0.2
SS300	627	855	345	227	104	0.4	0.1

Another famous correlation $\sigma_{FL} = 0.5UTS$ for traditional steels[22] is used to examine the fatigue limit of AMed EH36 steel. However obtained fatigue limits of the SLMed EH36 steel samples are not reaching 50% of their UTS. The resultant coefficients of σ_{FL}/UTS for all the samples are tabulated in Table 5-1. All the ratios falling between 0.1 – 0.2 are much away from engineering practice. Similar reasons can be deduced as above to be pores/defects and grain orientation, etc. A new mathematical equation with a constant term added discloses a linear positive correlation between σ_{FL} and UTS as displayed in Figure 5-5. Similarly a linear positive relationship between σ_{FL} and ε is correlated as well and shown in Figure 5-5. The above obtained mathematical models in respect with strength and elongation demonstrate proportional linear relationships with fatigue limit. It is clearly seen that sample SS100 shows highest endurance limit and survives for longest life cycles. The high strength with fair ductility resultant from the obtained microstructure of sample SS100 printed at lower scanning speed may benefit both low and high cycle fatigue performance. Those samples show higher tolerance to larger pores. The improved density prevents possible increment of effective nominal stress/strain applied to the sample during fatigue test. All above contribute to a better fatigue performance.

Figure 5-5 Correlations between fatigue limit (σ_{FL} with $R = -1$ being converted using a Goodman mean stress correction) and ultimate tensile strength (UTS) and elongation (ϵ) for samples SS100, SS200, SS250 and SS300

On the other hand Murakami method is famous in estimating fatigue limit by using actual defect size. The usual formula of Murakami method is $\sigma_{FL} = 1.43 \frac{Hv+120}{(\sqrt{area})^{1/6}}$ (for surface crack)[22], where $\sqrt{area}$ is the effective crack size (μm) and Hv is microhardness (kgf/mm^2). The crack is regarded as the crack origin, while its size can be measured directly from fractured surface. Therefore the effective size of the crack can be calculated by Murakami $\sqrt{area}$ method[22, 200, 201], which are 168, 313, 406 and 627 μm , respectively for samples SS100, SS200, SS250 and SS300. Crack of single origin is used to calculate the effective crack size. For multiple crack origins, equation needs to be modified. Here in this work all defects are regarded as surface crack (sub-surface crack uses another formula). Since this method is applicable to reverse loading (stress

ratio $R = -1$) scenario while the current fatigue test is conducted at $R = 0.1$, a correction factor needs to be applied in the way of $((1-R)/2)^\alpha$ ($\alpha = 0.226 + H_v \times 10^{-4}$)[201]. Nevertheless the experimental data is not well conformed to the usual Murakami linear relationship, but to $H_v/\sqrt{area}$ as displayed in Figure 5-6. Instead of using usual form of Murakami equation this new mathematical model (shown on the Figure 5-6) demonstrates higher accuracy to the experimental data.

Figure 5-6 Correlation between fatigue limit (σ_{FL} (with $R = -1$) being corrected using a Goodman mean stress relationship) and $H_v/\sqrt{area}$ with the data from samples SS100, SS200, SS250 and SS300

Lower results away from 1.6 Hv and 0.5 UTS relationships were also reported on SLM processed stainless steel SS630 steel and SS316L [120, 136, 147]. More accurate predictions by using Murakami equation for fatigue limit were reported in high strength steels in very high cycle fatigue (VHCF) regime[200]. The violation of traditionally well established relationships between fatigue limit and Hv and UTS reflects the unique characteristics of the AM processed metal materials.

Compared with thermo-mechanically controlled processed (TMCPed) and welded EH36 steels which fall at 299 MPa and 212 MPa, respectively (for fully reversed condition ($R = -1$))[143, 202, 203], the fatigue limit of 224 MPa of SLMed EH36 steel sample SS100 (after conversion using Goodman relationship to count for the mean stress effect) is lower than TMCPed grade but slightly higher than welded grade. In view of the microstructure, heat affected zone (HAZ) of welded grade of EH36 steel often becomes the crack initiation place with predominant phases of white ferrite, granular bainite and gray lamellar pearlite and a hardness of around Hv220, where crack can propagate along the fusion line. The microstructure of TMCPed EH36 steel plate is usually consisting of ferrite and pearlite with acicular ferrite, Widmastatten ferrite, lower bainite, upper bainite and some microphases giving a microhardness of around Hv185[203]. While the microstructure of SLMed EH36 steel sample mainly consists of fine ferrite, tempered martensite and other microphases with the hardness of around Hv288 for sample SS100. The lowered fatigue strength of SLM printed EH36 steel is deduced mainly due to the porosity/inclusions and other factors, such as increased hardness, grain orientation and retained austenite located at lath interface, etc. Firstly, large and complex shape of pores induces severe stress concentration around them and fatigue crack can form easily. The ubiquitous distribution of the pores increases the chance of multiple crack origins as disclosed later from the fractured surface of the fatigue samples, which may multiply the crack grow rate. Meanwhile unmelted particle or inclusions can induce strong stress/strain concentration in the way of either increasing surface roughness or amplifying the stress/strain concentration state around them[112]. Secondly, high strength steel is more sensitive to defects while low strength steel can easily absorb microdefects. The hardened steel subjected to surface defects with a hardness of Hv288 ~ 392 makes crack initiation and propagation easier[143]. Thirdly, for horizontally built samples, elongated columnar grains are oriented along the vertical

built direction that is perpendicular to the loading direction. For the crack initiated from grain boundaries or pores it can easily propagate along the longitudinal grain boundaries or within them with less blockage/resistance from microstructural level resulting in a less tortuous way. The less deflected crack propagation consumes lower energy where fatigue life could be shortened. The early crack propagation along longitudinal lath/block is observed from the fractured path of the fatigue sample SS200 tested at 400 MPa as shown in Figure 5-13. Fourthly, the effect of retained austenite on crack initiation/propagation is more argued, which may contribute to fatigue behavior in certain aspects[204]. A lowered fatigue strength of welded EH36 steel than TMCPed one is probably due to the welding defect, increased brittleness and hydrogen embrittlement where hydrogen is inevitably introduced into welding process.

5.1.4 Fracture Behavior

Fracture behavior is studied to serve as a better understanding to the fracture mechanism of the SLM built EH36 steel, mainly the focus on pores with their sizes & shapes to the crack initiation. The effects of stress levels, the density and microstructure on fatigue crack initiation and propagation are discussed. The ultimate target is to design a strong & tough AMed EH36 steel suitable for industrial application with possible adoption by M&O industry.

5.1.4.1 Multiple Crack Origins

A phenomenon about multiple crack origins is observed, which is common for many samples that are subjected to various loadings, from low to high. Examples are shown in Figure 5-7 for the SEM

micrographs with fractured surfaces for the sample SS100 tested at 500 MPa (O1, O2 & O3), in Figure 5-8 for the sample SS200 tested at both 400 (O1, O2 & O3) and 700 MPa (O1, O2 & O3), and in Figure 5-9 for the less dense sample SS300 tested at 400 (O1, O2 & O3), 600 (O1) and 700 MPa (O1 & O2). The reasons are believed inevitably due to the pores' ubiquitous distribution in the 3D matrix. Exposure to surface engenders the pores equal chance to initiate crack. Origins are even located at different planes or heights as observed in Figure 5-8a&b. Within one crack origin, multiplicative crack nucleation is formed as shown in Figure 5-7b indicated by yellow arrows. They may develop within the MSC (microstructurally small crack) regime and reduce fatigue life with a small portion of effect.

From the SEM observation, the pores initiated cracks are mostly in narrow elongated shapes without or with one or multiple triangular-like segments, which are similar to those shown in the previous OM images. The origin O1 in Figure 5-7a shows a narrow elongated shape with a total length of about 500 μm and a height of 10 μm (indicated with a white double arrow). O2 shows a triangular-like shape with a side length of about 125 μm (indicated by a white double arrow). O3 has multiple triangular-like segments connected by narrow slit like portions and narrow elongated tails with a total length of about 150 μm and heights of about 25, 25, 15 and 7 μm for the four respective triangular-like portions and about 5 μm for the narrow slit like segment and tail. The four triangular-like pore segments are about 15 - 50 μm wide and separated by a distance of about 25 - 50 μm . In view of their 3D morphology shown in the CT image (Figure 5), they may belong to one single pore. The narrow sections could be the extended elongated portion of the 3D pores and the triangular-like sections could correspond to the sharp corners of the 3D pores. Similar lack of fusion pore is observed in sample SS200 shown in Figure 5-8 but in a larger scale (with marked

up characteristic length scale in double headed white arrows). The pore has multiple triangular-like shape sharp corners. The above pores are described in 2D dimension and their 3D morphology could be simplified into sharp roof top configure with all sides in curved profile that coupled with multiple sharp corners and small radius side corners. Depending on their orientations and loading direction different sharp corners may act as stress concentration points that can lead crack initiation. The forming mechanism of such pore configure can be the lack of fusion between adjacent layers and tracks rooted from improper process parameters. The lack of fusion may locate at the inter-track where energy penetration is not enough. The size of the lack of fusion geometry is up to 500 - 1000 μm . More complex shapes could be formed at boundaries of chess boards in a crossover manner. The length scale of those pores is submit to chess board settings, such as size of the chess board, rotation angle of vector and other factors. Smaller chess board and random vector scanning may reduce the length scale of the pore. Therefore, pores cannot be eliminated without proper process parameter setting and enough laser penetration through increasing heat input. The other reasons for such complex shaped pores are also possibly caused by the surface roughness of the printed layer where gaps are left between the tracks or layers.

Internal pores along the crack propagation path deep into the sample show crack nucleation traces in ridge features highlighted with a yellow dotted circle in Figure 5-7a where the nucleation from the internal pores could be started concurrently with surface cracking or through interactions with a main crack when passing by. Similar phenomena are found in other samples, such as SS300 in Figure 5-9a&d, where defects like inclusions and delamination cracks change the initial crack path but follow the same direction macroscopically. The large pores inside the AMed parts are believed to be difficult to prevent even for the highly dense samples. The distinction between MSC and LC

is not very clear from the SEM observation. Stable crack propagation (SCP) including MSC and LC stages is characterized hereafter. Tearing topology surface (TTS) is observed near crack origin and before fast fracture regime as shown in Figure 5-7c&d. Dimples are found spreading over fast fracture regimes as shown in Figure 5-7f and Figure 5-8d. Fan feature is observed in sample SS100 being tested at 600 MPa as shown in Figure 5-7g. The fans are usually observed at final fracture region showing brittle features.

Figure 5-7 SEM micrographs of the fractured surfaces of sample SS100 tested at the stress level of 500 MPa (max. stress) showing (a) overview of crack origins, (b) enlarged view of the white dashed box A in (a), (c) enlarged view of the white dashed box C in (b) showing the tearing topology surface (TTS) near crack initiation area, (d) enlarged view of the white dashed box B in (a) showing the tearing topology surface (TTS) before fast fracture regime, (e) overview of fast fracture regime, and (f) enlarged view of the white dashed box D in (e), and (g) the fan features in sample SS100 tested at 600 MPa.

Figure 5-8 SEM micrographs of fractured surfaces of (a-c) sample SS200 tested at 400 MPa (max. stress) showing (a) one crack origin (O1), (b) two other crack origins (O2 and O3), and (c) enlarged view of the white dashed box in (b), and (d-e) sample SS200 tested at 700 MPa (max. stress) showing (d) three crack

origins (O1, O2 & O3), SCP and fast fracture regime, and (e) dimples at fast fracture regime. In (a) & (b) origin 1 is located at different planes from origins 2 & 3. The white double arrows in (c) indicate the length scales of the lack of fusion pores with different segments (triangular-like sharp corners and connected areas).

Figure 5-9 SEM micrographs showing the fractured surfaces of sample SS300 (a) tested at 400 MPa for the overview of crack initiation (O1 & O2), SCP and fast fracture regimes, (b) the overview of fast fracture regime, (c) tested at 600 MPa for the overview of crack origin, SCP and fast fracture regimes, and (d) tested at 700 MPa for the overview of crack origins (O1 & O2), SCP and fast fracture regimes

As reported, under low stress levels (e.g., 400 MPa) sample normally fails by one main crack, while at high stress levels (e.g., 700 MPa) multiple crack origins may propagate simultaneously with all of them contribute to the total fatigue life[146]. Here for SLMed EH36 steel multiple crack

origins are commonly seen in samples subjected to both low and high stresses. This unusual consequence incurred on AMed metals shows detrimental effects of pores on its fatigue behavior. The ubiquitously distributed pores highly exposed to sample surface make crack initiation favorable at pores due to their strong stress/strain concentration ability coming from their large sizes and complexity of shape. Those are demonstrated in Figure 5-8e where sharp corners and uneven bulging along the strait sides of pores show multiple crack nucleation and propagation emanating from those points. The other detrimental effects of pores include elevated effective nominal stress/strain, cyclic softening and pore-pore interactions that can accelerate crack propagation during the cyclic fatigue process.

The reason of crack nucleation favoring sharp corners and surface uneven points is understandable because the sharp tips of the defects can raise local stresses as strong stress concentrations. This is consistent with Murakami's finding that the crack initiation is strongly influenced by its shape[22]. In some cases as shown in Figure 5-7 a subsurface triangular-like shape of pore (O2) is found near the surface crack origin O3 where they may interact to affect local plastic strain and lead crack propagation as well[112, 117, 205, 206]. They may originate from one single pore which is unveiled from 2D image. In view of this, a model describes fatigue initiation mechanism for SLMed EH36 steel samples is proposed for typical lack of fusion pores and illustrated in Figure 5-10. The 2D geometry of the pore shows that sharp triangular-like tip can be highly stress concentrated subject to loading direction. Due to the multiple sharp corners along the contour of the 3D pores orientated in different directions, the points as strong stress concentrations are in a larger quantity. Its 2D illustration (may represent one of the scenarios) is described in Figure 5-10b. Similarly the surface unevenness can be highly stress concentrated in the form of unmelted

particles, inclusions or micronotch. The round dots represent unmelted particle/inclusions. All these stress concentration points may lead cracks in the very first place followed by other sites with lessened concentration effect subjected to loading direction. Similar fatigue initiation mechanism was proposed by Liu et al. for a 6005A CMT welded joint[207]. Actual pores in more complex 3D shape with multiple sharp corners and small radius edges can cause an even more complex stress state. Overall these can act together to affect the plastic strain of the surroundings and lead to fatigue crack propagation.

Figure 5-10 Schematic diagrams showing crack initiation mechanisms from triangular-like lack of fusion pores with narrow elongated tails (a) a simple triangular-like form of pore, and (b) a multiple triangular-like form of pore with the loading directions indicated. The unmelted particles or inclusions are indicated with red dashed arrows.

Mostly unmelted particles or round particles are found within crack origins that are due to non-enough energy penetration to melt all powders and possible laser sputtering caused surface roughness. If the particles are detached from matrix, it is treated as pores. If the particles are connected to the matrix the mechanism could be deduced as that the incubation life at inclusions is much less [143] or quantified by one third of pores with equivalent size being reported in a multistage fatigue model by Xue et al.[112]. As known HCF is dominated by crack initiation stage[208]. For porous AMed steel samples, the crack initiation stage mainly MSC could be largely reduced or even by-passed while total fatigue life can be largely reduced in due. Despite the unmelted particles and other defects that may amplify the complex stress distribution around them, HCF is highly influenced by pores including their quantity, position, ubiquitous distribution, size and shape. Large complex shape of pores together with the possible increased effective nominal stress/strain can largely increase crack growth rate. The microstructure around the vicinity of the pores may contribute to the crack initiation from certain aspect, such as grain orientation and size, crystallographic orientation and defects, etc. The fracture mechanism is then highly related to its unique microstructure formed during the SLM process.

5.1.4.2 Fracture Behavior at Low Stress Level

The overall crack path delivers brittle like features. Under a low stress level a crack starts from interacting with microstructural features (e.g. martensite laths/blocks) and then propagates into a fully developed region (showing more surface unevenness) until fast fracture. As seen from Figure 5-7a a crack is well developed before entering the fast fracture region. The fractured surface shows a long stable crack propagation zone (stage SCP) up to a few millimeters. The enlarged views of

the fractured surface in Figure 5-7c&d show mainly a tearing topology surface (TTS) throughout the whole crack propagation stage. The TTS appearance is a sign of ductile or microplastic tearing on a very fine scale[209]. Similar fracture pattern is found in martensitic AerMet100[209] and HY130 high strength steel[210]. Dimples are observed spreading over the fast fracture regime as shown in Figure 5-7f.

Striations are sparsely found along the stable crack propagation zone, which are mixed with TTS feature but not shown here. The reason for the occurrence of striations is elucidated related to the material ductility. Usually, striations happen in metals which are not too brittle or too soft. The crack tips inside the sample lead to a high local stress above yield stress that can generate a local plastic deformation where the opening and closure of cracks give the striation features. Striations under uniaxial tensile fatigue loading are observed in other metallic materials [211], where the re-sharpening and blunting of the crack propagation caused by the change of the fracture velocity resulting in formation of the fatigue striation (Mode I) was reported. The TTS takes the form of coarser features or ductile micro-tearing topology at lower hardness[212], while when the hardness increases the TTS features are finer or even goes featureless. Retained austenite has been reported in our previous discussion in Chapter 4 in a higher amount in sample SS300 printed at a higher scanning speed coupled with higher cooling rate which is confirmed by XRD and TEM. Those soft phases could induce striations as well. In Figure 5-11 TEM bright field images of sample S300 show (a) cellular-dendritic grains with retained austenite located at intra and inter cellular-dendritic grains (dark in color, indicated by yellow arrow), and (b) growing cellular-dendritic grains in different orientations (indicated in white arrows) with retained austenite grains (indicated by yellow arrows) located at boundaries and inside the grains as well. The detailed forming

mechanism of martensite grains and retained austenite were discussed in our previous work[173] together with lath shape of martensite shown in that work.

Figure 5-11 TEM bright field images of sample S300 showing (a) cellular-dendritic grains with retained austenite located in intra and inter cellular-dendritic grains (dark in color, indicated with yellow arrow), and (b) growing cellular-dendritic grains in different orientations with retained austenite grains located at the boundaries and inside the grains as well

5.1.4.3 Fracture Behavior at High Stress Level

Under a high stress level, a crack enters a fast fracture region right after initiating from the lack of fusion pores and other defects. The crack propagation stage, mainly the stable crack propagation (indicated as stage SCP) is largely reduced as shown in Figure 5-8b for the sample SS200 tested at 700 MPa. The SCP distance is less than 500 μm for each of the 3 crack origins that propagate concurrently leading to reduced fatigue life of this sample. Similarly, a SCP distance of $< 1\text{ mm}$ is measured from the sample SS300 in Figure 5-9a,c&d tested at 400, 600 and 700 MPa, respectively.

The enlarged views of the fast fracture areas in the samples SS200 and SS300 show dimple features as seen in Figure 5-8d demonstrating large plastic deformations and void coalescence failure mechanism in this stage. The reduction of stable crack propagation is often observed in sample SS200 and SS300 that have higher level of porosity regardless of the applied stress level, which discloses the possible increased effective nominal stress/strain being applied to the sample during the fatigue test. The fast fracture zone shown in Figure 5-9b of sample SS300 tested at 400 MPa is obviously enlarged. As seen crack initiates from large surface pores, and after a short propagation of about 1 ~ 1.5 mm (stable crack propagation) crack enters into its fast fracture regime in Figure 5-9b. While for healthy stable crack propagation the propagation distance can be up to a few mms. Hence for porous sample being tested at low stress level the whole fractured surface is still dominated by fast fracture zone. There are other factors that may contribute to the fatigue life from certain aspects, such as the strong stress concentration effect of pores, multiple origins, incremental of nominal stress/strain, interactions between large internal pores, which may change the crack growth rate. Change of crack path is observed in porous samples. As seen from Figure 5-9d along the crack path initiations around inclusions (indicated in red thin arrow), pores and inter-layer delamination-like long crack (indicated in thick red arrows) are observed that may resume propagation along previous directions (indicated in red dotted line). Similar crack jumping can be seen from Figure 5-9a at internal defects indicated in red arrows. The overall fractured surface shows ductile and brittle features that may appear in an alternative way along the path demonstrating the complexity of fatigue failure mode.

5.1.4.4 Effect of Density on Fracture Behavior

The characteristics of crack initiation and propagation of the porous sample are mentioned above and the samples with low density show mainly a porosity dominated failure mode. However, for the highly dense sample, marks of slipping (in dotted loop) are observed as shown in Figure 5-12 near the crack initiation pore in the sample SS100 being tested at low stress level of 400 MPa. The morphology of the slip marking may take the form of extrusion as indicated in Figure 5-12d and it may happen within coarsened ferrite grains or martensite lath. The mechanism of slip generation within coarsened ferrite grains has no much debate. More elaboration is needed for harder martensitic structure as it was reported slippery bands were not possible for brittle martensitic steel[209]. However this type of slip feature was found in 12%Cr martensitic steel along the vicinity of lath boundaries which has been explained by Seidameova et al. in a few mechanisms like vacancy diffusion induced redistribution of matter, grain boundary sliding, plane slipping and soft phase of retained austenite located at lath boundaries[204]. Similar slip markings were observed in SLMed SS630[120]. While in this SLMed printed EH36 steel sample SS100, coarsened lath and ferrite grains could be an important factor for the occurrence of slip markings. It was observed that within finer grain matrix, e.g., a few microns, pore initiation is preferred, while within coarser grain matrix, e.g., tens of microns, intra-grain slippery bands generated within coarser grains type of initiation preferred[197]. As discussed above a low scanning speed produces coarse grains upon deposition due to the low cooling rate and transformed ferrite with heat from repeated laser scanning[173]. The coarser lath is considered as “soft spots” characterized by larger size, less defect and higher auto-tempering effect[213]. In contrast finer lath are highly dislocated and less auto-tempered. Therefore the crystallographic slip can be activated within soft coarse grains. For martensite lath induced slipping it may be further enhanced by intra-lath-plane slipping subject to the preferable crystallographic and grain orientations[213]. The slip mechanism induced

by coarse grains may extend the coarser microstructure's fatigue life by an extra stage of generating slip markings, while in the finer microstructure matrix pore failure mechanism may shorten its fatigue life by starting crack propagation straight away especially when the stress concentration effect is severe. Based on these the merit of fine grains is deemed depending on their individual system with pore as a crucial factor.

Figure 5-12 SEM micrographs of fractured surface of sample S100 tested at 400 MPa showing (a) overview of crack initiation, (b) enlarged view of the white dashed box A in (a), (c) the enlarged view of the white dashed box B in (b), and (d) the enlarged view of the white dashed box C in (c) with a small area featured by slip markings

Another possible mechanism for the slip is grain boundary gliding which is an important mechanism for the plastic deformation for lath martensite that has been reported mainly along the flat block or packet boundaries where the orientation of the boundary plane with respect to the loading direction is a crucial factor[213]. Morsdorf et al. found out that high shear stresses along 45° orientation (approximately) enabled most pronounced interface plasticity[213]. Moreover unmelted particle induced higher plasticity could possibly enhance the slip generation as reported by Xue et al.[112]. Since the occurrence of slip marking is located at the triple point enclosed by unmelted particle indicated in Figure 5-12c which is broken during crack propagation, the stress state at surroundings is believed to be very complex as illustrated in Figure 5-10. However the effect of soft retained austenite film located at martensite lath interfaces on the local plastic deformation was argued that may contribute to the plastic deformation in certain aspect[213]. Crack continues to propagate beyond the slip marking as shown in Figure 5-12c. Another nucleation site indicated in Figure 5-12b shows a different microstructural feature of crack propagation mainly in tearing ridges radiating from the corner of the narrow tail to the center of the sample. Mode of competing fatigue failure with pores and slipping markings is observed for sample SS100. Such pores and slippery bands competing fatigue failure mode was reported elsewhere for Ti-6Al-4V fabricated from powder metallurgy [197].

The crack initiation stage of AMed metal materials are deduced highly dependent on pores, inclusions and other defects. If the pore and other defects induced stress concentration is too high, crack initiation stage, mainly MSC could be largely reduced or even bypassed in some extreme cases. The soft microstructure of SLMed EH36 steel sample printed with higher heat input at slower scanning speed with a hardness of Hv288 shows certain tolerance to pores with a size of

hundred over microns. However for hard steel samples with higher hardness, e.g. $\geq 400\text{Hv}$, to have good fatigue strength, reducing the size of pores could be crucially important beyond microstructure adjustment such as chemical compositions and heat treatment[22].

5.1.5 Fatigue Failure Mechanism

The fractured path of the sample SS200 tested at 400 MPa is shown in Figure 5-13. As seen from Figure 5-13b, a crack initiates from a surface pore. In the early stage, the crack propagates along martensite lath/block boundaries (indicated with black arrows) for about 20 – 30 μm in a straight path with 2 or 3 turning points. Or, to be more accurate, the crack is propagating at the vicinities of the martensite lath/block boundaries, which were also reported in the literature[204, 214]. Then crack transits into transgranular mode (indicated with yellow arrows in Figure 5-13b) in a more zig-zag pattern. The micromechanisms of crack propagation at different stages have been reported [112, 120, 154, 204, 208, 215].

The early stage along longitudinal lath/block boundaries that are oriented in certain angle to loading direction could be controlled by shearing mode. The straight path with a range up to 10 – 20 μm without much tortuosity is regarded as easy propagation with low energy consumption. The possible mechanisms could be plane slipping, grain boundary gliding and retained austenite, etc. as discussed above in slip marking section. From the observation, the conventionally defined crack incubation stage mainly the INC stage driven by maximum shear stress seems not bypassed even in the presence of pores [112]. However further study is needed. Here the point is to address the grain orientation effect to the early crack propagation. To bear in mind that the fine elongated

cellular-dendritic grains are oriented along the building direction grown from bottom and perpendicular to the melting pool boundary to the centre of it. Even though martensitic transformation within the prior grains increases the randomness of grain orientation, the orientation along the building direction is still dominating. Presume the crack initiates from intergranular boundary or lath/block boundaries with the longitudinal boundary plane of the lath or cellular-dendritic grains in preferable orientation, crack can propagate easily in a long path with less microstructural obstacles, mainly grain boundaries. Fatigue life could be reduced. Similar for the intra lath/block propagation with preferable crystallographic orientation in the longitudinal direction[213]. In addition the microstructure in the vicinity of the pores may affect crack propagation and failure mechanism. The intergranular defects such as element segregation, nano-precipitates and residual stress, etc. may enhance the intergranular crack propagation as well.

Figure 5-13 SEM micrographs showing sample SS200 tested at 400 MPa with (a) the overview of fracture path, (b) the enlarged view of dashed box A in (a) near a crack origin, and (c) the enlarged view of the dashed box B in (a) within a fast fracture region

All those influence the fatigue behavior and may lead to a shortened fatigue life. In contrast the crack is more frequently deflected after its transit into a transgranular mode, thus resulting in a more tortuous path. This is controlled by shearing or in a mixed mode. Therefore, both grain size and morphology and its orientation influence the crack propagation of the SLMed EH36 steel samples. Near the fast fracture region, large deformations of grains are observed as shown in Figure 5-13c and voids are formed both within the laths/grains and at their boundaries demonstrating a void coalescence failure mode. No much deformation of grains is observed from the locations far away of the fractured surface.

5.2 Simulation Results and Discussion

Porosity has a crucial effect on the samples' fatigue performance, which needs to be studied in depth using numerical methods. In this work pores with accurate sizes and new shapes were proposed at the first position with their effects on stress concentration and fatigue life being studied and compared with experimental data. The scope covered pore shape (both more realistic triangular and simplified), position, orientation, size and pore-pore interaction. The proposed pore morphology and size were rationalized from CT scan results and experimental observations from the fractured surface of AMed EH36 steel samples. The elastic stress concentration factors were studied. Then several stress-life and strain-life algorithms set in Fe-Safe software were adopted for fatigue life evaluation. The predicted fatigue life was validated with experimental results as well as the calculated lifetimes using LEFM model. The main contributions of this paper are:

- Disclosed fracture behavior of additively manufactured EH36 steel, fatigue crack initiating types, morphologies and proposed a new concept of fatigue life prediction method which could be extended to other additively manufactured metals and alloys
- The additively manufactured steel materials parameters were used for the prediction including ultimate tensile strength, stress-strain curve (SN curve) and related fatigue parameters
- Studied the influence of different types of pores on stress concentration factor K_t and fatigue lifetimes including simplified semi-spheroid, semi-ellipsoid and more realistic triangular pores
- Evaluated pore size, shape, location as well as pore-pore interactions effect on stress concentration and fatigue lifetimes

- Evaluated conventional stress-life and strain-life methods and linear elastic fracture mechanics model (LEFM) on additively manufactured EH36 steel for predicting fatigue life cycles using a surface-pore incorporated model

5.2.1 Short Review from Experimental Work

Overall, the printed EH36 steel samples demonstrate a porosity dominated fatigue behavior. The samples printed at higher scanning speed show larger sized and amount of lack of fusion (LOF) resulted from deficit laser penetration, which has been reported elsewhere for the SLMed EH36 steel[173]. As discussed in the previous work, finer microstructure is resulting from the higher cooling rate with higher scanning speed, which shall benefit their strength and fatigue performance. But meanwhile larger amount of LOF is resulted from higher scanning speed which gives the density in a decreasing trend as 99%, 97%, 94% and 94% for sample SS100, SS200, SS250 and SS300, respectively. LOF is the main failure mode which is more detrimental to fatigue performance of AMed parts than smaller and more regular shape of gas pores. In view of this, the fatigue performance is mainly defect dominated (lack of fusion, gas pore, unmelted powder and inclusions, etc.) and LOF is the reason here for the SLMed EH36 steel. The role of unmelted powder is difficult to evaluate as it always incurred inside LOF which is regarded as LOF only. The deterioration of LOF to the fatigue life is demonstrated in Figure 5-14 with the SN curve for the sample SS300 fallen below those of the samples SS250, SS200 and SS100 gradually with fitted equations shown in the figure. This shows that the larger the LOF, the lower the fatigue life for the samples printed at higher scan speeds. The data falls within the scatter band of a factor 8 with respect to the different scanning speeds. More details can be found in the previous work for the

fractured mechanism[135]. Meanwhile, when the samples' porosity is high, e.g., SS250 and SS300, at higher stress levels, e.g., 700, 600 and 500 MPa, their lifetimes are similar. This means that their lower life performance (higher applied stress) is less affected by pores. When the stress level is low (e.g. 400 MPa), the lifetime difference is significant. This can be explained from the failure mechanism of LCF and HCF. For HCF, crack initiation stage mainly incubation plus short crack propagation dominates total fatigue life. The crack initiation phase could be largely shortened when large pore initiate crack. The varied pore size, distribution and shape, etc. lead to largely scattered fatigue life for the HCF. For LCF, the crack propagation phase dominates and the initiation phase is a relatively small portion and this is why the big gap diminishes. The fatigue limits corresponding to 10^6 cycles are calculated according to the fitted SN curves for all the samples. The obtained fatigue limits have not been corrected for mean stress effect using Goodman relationship[147] to a stress ratio of $R = -1$ due to its unforeseen inaccuracy of the conversion.

Figure 5-14 Fatigue SN curves for samples SS100, SS200, SS250 and SS300 built at stress levels of 700, 600, 500 and 400 MPa (max. stresses) with scatter band of a factor of 8.

The fractured surface shown in Figure 5-15 reveals the pores that initiate fatigue failure. Basically, all the samples fail by LOF. For samples with higher densification such as SS100 at low stress level of 400, there is only one crack origin. For other less densified samples and at higher stress levels, multiple crack origins are observed as shown in the yellow dashed boxes in Figure 5-15a,b,c&d. The shapes of the 2D LOF are either narrowly elongated with wider triangular segments or largely irregular with multiple sharp corners (with inclusions or unmelted powder particles inside). Their sizes can range up to a few hundreds of microns. Even though inclusions were reported to be able to induce a higher stress concentration factor than pores[143], the inclusions/unmelted particles inside the LOF regions are mostly isolated, which makes their effects difficult to be differentiated from the pores. So inclusions are considered to only increase surface roughness while all the fatigue crack origins are due to large pores of LOF in SLM manufactured EH36 steel samples. As disclosed from CT scan, the 3D LOF pore is elucidated to be a simple sharp roof top shape, defined as triangular pore. The length scale can vary with process parameters used and the settings of the chess board strategy, which can range from 100 - 200 μm to 1000 - 1500 μm . The triangular pores inside the samples printed at lower scanning speed (e.g. 100 mm/s) with higher heat input are narrower and shorter, while the wider, longer and more irregular pores inside the samples printed at higher scanning speed are due to limited laser penetration. Those contour sharp corners are likely to concentrate stress and initiate fatigue failure upon far end loading direction. In view of this, a few factors that may affect K_t and fatigue crack initiation life are studied using numerical methods for the isolated triangular pores and pore-pore interactions. The width and height of the triangular pores are fixed at 200 μm and 100 μm respectively, while the length scale and locations are varied. To be more generalized, the pores are further simplified

into semi-spheroidal or semi-ellipsoidal surface pores for their effects to be compared with the above more realistic pores.

Figure 5-15 SEM micrographs showing the fractured surfaces of sample SS100_400MPa for the (a) overview and (e) enlarged view of the yellow dashed box in (a), and sample SS200_400MPa for the (b) overview and (f) enlarged view of the dashed box in (b), and sample SS250_600MPa for the (c) overview, and sample SS250_700MPa for the (d) overview and (g) enlarged view of the yellow dashed box in (d) of partial of the crack initiation point.

5.2.2 Static Stress Analysis of Fatigue Models using ABAQUS

5.2.2.1 Examination of Stress Concentration Point

As fatigue failure often initiates from pores, inclusions or other defects induced stress concentration, the locations where largest stresses can be induced are studied first. The static stress analysis results with respect to various shapes of pores proposed are shown in Figure 5-16 and Figure 5-17. The stress clouds (shown in equivalent Von Mises stress) in Figure 5-16 describe the peak stress points, which are found located at the minimum radii near subsurface of mouth location or nearly the mid position between mouth and root (the variation may come from the meshing) for the perpendicularly oriented triangular surface pores, similarly for the dual triangular pores (in Figure 5-16d), triangular pores with triple triangular segments (Figure 5-16e) and crossover triangular pores (in Figure 5-16f) which show the peak stress location could be at crossover joint point at minimum root radii. For the triangular pore with through thickness the peak stress occurs at the minimum radius position along the thickness as shown in Figure 5-16b. However, when the triangular pores are parallel oriented to the loading direction the locations of maximum stresses shift to the sharp corners of the 90° side walls as shown in Figure 5-16g&h. For perpendicular oriented triangular pores, the K_t of pores located at side centre (SC in Figure 5-16c) is comparable with those located at side edge (SE in Figure 5-16a) positions, while slightly higher than those at

middle edge (ME). The cylinder pore in Figure 5-16i with the similar size of triangular pores show mostly lower K_t than triangular pores that can avoid the higher concentration induced by the sharper radius and possibly the complex shapes coupled with the triangular pores. In a contrast, the maximum stresses for most semi-spheroidal/ellipsoidal pores are mostly located near the subsurfaces of the open surface at the smallest radii along the planes normal to the loading directions as shown in Figure 5-17a-d. The maximum stress points of y-series of ellipsoidal pores shift to the middle root positions. The stress concentration factor K_t for dual triangular pores is not higher than single triangular pore, but K_t of tri-segment pore is very high ($K_t = 13$) making the pore clustering (with small radius) super dangerous to the fatigue properties. The singular revolved triangular pore in Figure 5-16j shows higher K_t ($K_t = 3.0$) than spheroidal pore (mostly around $K_t = 2.0$ for all the surface spheroidal pores, as shown in Figure 5-17a), which is comparable with ellipsoidal pore of $\text{elli_}0.5\text{-}0.1\text{-}0.1$ ($K_t = 2.8$ as shown in Figure 5-17c with refined mesh) but lower than other triangular pores demonstrating that the radius and the shape are key factors that can influence the stress concentration. Based on the above study, a rule of stress concentration locations is generalized in Figure 5-18. The near mouth locations and the sharp corners/radii of the open pores residing along the plane normal to the loading direction favors fatigue crack. For triangular pores the points are mostly located along the root of the pore near subsurface or in middle positions. For the semi ellipsoidal and semi-spheroidal series, the stress concentration points are also near to the sub-surface areas as illustrated in Figure 5-18.

(a) tri_ShS_SE_Perp_0.2-0.1-0.5 ($K_t = 5.51$)

(b) tri_ShS_SE_Perp_0.2-0.1-2.9_through ($K_t = 5.75$)

(c) tri_ShS_SC_Perp_0.2-0.1-0.1 ($K_t = 4.77$)

(d) tri_Dual triangular pore_ShS_SE_Perp_0.2-0.1-0.5 ($K_t = 4.83$)

(e) Triangular pore with 3 triangular segments_ShS_SE_Perp ($K_t = 13.28$)

(f) Crossover triangular pore_LoS_ME_Perp_joined with triangular pore 0.2-0.1-2.9 and half triangular pore 0.1-0.1-0.5 ($K_t = 1.9$)

(g)tri_LoS_SC_Para_0.2-0.1-0.5($K_t = 2.32$)

(h)tri_LoS_SE_Para_0.2-0.1-0.5($K_t = 2.26$)

(i)cylinder_ShS_SC_Perp_0.1-0.1-0.1($K_t = 2.13$)

(j)Tri_ShS_SC_Perp_0.2-0.1_revolved($K_t = 3.0$)

Figure 5-16 Stress clouds of triangular pores of (a)ShS_SE_Perp_0.2-0.1-0.5, (b)ShS_SE_Perp_0.2-0.1-through, (c)tri_ShS_SC_Perp_0.2-0.1-0.1 (with refined mesh), (d)Dual triangular pore_SS_SE_Perp_0.2-0.1-0.5, (e)triangular pore with 3 triangular segments_ShS_SE_Perp, (f)crossover triangular pore_ShS_SC_Perp_joined with triangular pore 0.2-0.1-0.5 and another half triangular pore 0.1-0.1-0.5, (g)tri_LoS_SC_Para_0.2-0.1-0.5, and (h)tri_LoS_SE_Para_0.2-0.1-0.5, (i)cylinder_ShS_SC_Perp_0.1-0.1-0.1 (with refined mesh), and (j)Tri_ShS_SC_Perp_0.2-0.1_revolved (with refined mesh). Note: Dual triangular pores separated by a distance of 10 μm .

(a) elli_ShS_SC_0.1-0.1-0.1 ($K_t = 1.92$)

(b) elli_ShS_SC_0.1-0.5-0.1 ($K_t = 1.36$)

(c) elli_ShS_SC_0.5-0.1-0.1 ($K_t = 2.8$ with refined mesh)

(d) elli_ShS_SC_0.1-0.1-0.5 ($K_t = 2.6$)

Figure 5-17 Stress clouds of semi-spheroidal and semi-ellipsoidal surface pores of (a)elli_SC_0.1-0.1-0.1 indicated with mouth and root position,(b)elli_SC_0.1-0.5-0.1,(c)elli_SC_0.5-0.1-0.1(with refined mesh),and (d)elli_SC_0.1-0.1-0.5

Figure 5-18 Schematic illustration of stress concentration points for different shapes of pores from left to right: triangular, spherical/elliptical_z, elliptical_y and elliptical_x.

5.2.2.2 Stress Concentration Factor

Effects of proposed pore size, position, shape and orientation on stress concentration factor K_t is evaluated. Some of the results are shown in Table 5-2 for triangular surface types of pores. Those data was plotted in Figure 5-19a for the correlation of K_t to the length of pores. For different pores, K_t essentially shows a wavy pattern along with the length of a pore. Overall, the triangular pores show higher K_t values than the semi-spheroidal/ellipsoidal pores for most of the configurations, which are mainly due to the sharp radius of the triangular pores set as $10\text{ }\mu\text{m}$, while the radius of most of the semi-ellipsoidal pores is much larger than $10\text{ }\mu\text{m}$. When the crack length increases, K_t increases first and then decreases or bounces back a few times. Those may come from errors of coarse mesh size or the stress constraint state that may change due to the ratio changes of pore length to model size [148, 152]. Meanwhile, K_t for the perpendicularly orientated triangular pores are higher than those of parallel oriented, which is also dictated mainly by the sharp stress concentrated radius and the elongated pore length along the plane to the loading direction (equivalent to longer crack length). As discussed above for all the perpendicular oriented triangular pores, the K_t of side edge (SE) location is similar to that of side centre (SC) but slightly higher than that of middle edge (ME). It seems mean that increasing the pore depth into the body making their stress concentration effect less effective. Other than the side centre and side edge distinction, the short side (ShS) and long side (LoS) location also affect K_t as shown in Figure 5-19a that pores allocated to the ShS gives higher K_t than that allocated to LoS. This is somehow consistent with experimental observations that more than 50% of the crack origins are found along the ShS. The rest is found along the LoS, while few is found initiating from corners. And 3D semi-ellipsoidal pores show lower K_t (< 3.0) than the 2D elliptical pores, which has been reported by Nicoletto et al. [144] and Yang et al.[216]based on their simulation works. As shown in Figure 5-16 the

triangular pores with three segments show much higher K_t than the simple triangular pores. The double triangular pore also gives higher K_t than single triangular pore with the same size. However the crossover triangular pore gives lower K_t than each of the single pores. Those showing the complex pore shapes and pore-pore interactions may induce varied stress amplification or reduction to the surroundings. Overall the stress concentration factor can be affected by a series of factors including pore size, shape, position and orientation, as well as pore-pore interaction so as to affect the crack initiation points/locations. The effect of aspect ratio is further studied and results are shown in Figure 5-19b. From the figure it seems K_t saturates at certain point and drops back to stabilize. For elli-SC-Perp series of pores, K_t maximizes to 2.8 at the aspect ratio of around 5.0. It appears that when the surface pores turn to be more prolate, a further increase of aspect ratio (corresponded to reduced pore radius) does not elevate stress concentration. For the triangular pores K_t shows a similar, but more fluctuated pattern.

Table 5-2 The stress concentration factors K_t for different types of triangular surface pores

Pore type	K_t						
	ShS_SE_Perp	ShS_ME_Perp	ShS_SC_Para	LoS_SE_Perp	LoS_ME_Perp	LoS_SC_Para	LoS_SE_Para
tri_0.2-0.1-0.25	5.37	4.69	2.25	5.28	4.68	2.33	2.30
tri_0.2-0.1-0.5	5.51	4.79	2.40	5.46	4.50	2.32	2.26
tri_0.2-0.1-0.75	5.68	5.20	2.24	5.98	4.67	2.13	2.12
tri_0.2-0.1-1.0	5.82	4.86	2.25	5.81	4.93	2.12	2.07
tri_0.2-0.1-1.5	5.80	4.86	2.84	5.52	4.68	2.61	2.55
tri_0.2-0.1-2.0	5.80			5.63			
tri_0.2-0.1-2.9_through	5.68	4.83					
tri_0.2-0.1-5.8_through				5.95	6.18		
tri-segments	13.28						
double tri_0.2-0.1-0.5	4.83						
tri_crossover_ 0.2-0.1-2.9 + 0.1-0.1-0.5					1.96

Figure 5-19 (a) Stress concentration factors of triangular and ellipsoidal pores in different orientations and length scales, and (b) Effect of aspect ratio on stress concentration factor with data from elliptical and triangular pores

5.2.3 Fatigue Life Simulation with FE-SAFE

5.2.3.1 Effect of UTS, E and Fatigue Parameters

To evaluate the influence of the materials parameters on prediction of fatigue life, different UTS, E and fatigue parameters are studied which is assigned into the categories of SS100, SS200, SS250 and SS300 according to actual testing results where the suffix of E and E' stands for the ground modulus and virtual one, that is 200 GPa and corrected values according to pore volume within the samples. The values used are from Table 3-6. The predicted fatigue life results are shown in Table 5-3 for typical surface pore shape of elli_ShS_SC_0.1-0.1-0.1, elli-ShS_SC_0.1-0.1-0.25 and tri_ShS_SE_Perp_0.2-0.1-0.25 with experimental results inserted. Clearly seen, sample SS100 printed at slower scanning speed outperforms the rest. This is consistent with experimental study due to its higher strength, Young's modulus, etc. with less detrimental pores effect. The predicted

fatigue lives of the rest three samples show similar trend as experimental results, that is higher the pores, lower the fatigue lifetimes. Higher Young's modulus also benefits fatigue lifetimes as illustrated in Figure 5-20 of strain-life method results. The results using stress-life method is not affected by E because stress-life method uses S-N curve method without E values (the E effect is incorporated into S-N curve already). As seen the elli_0.1-0.1-0.1 pore is more close to actual tested values of experimental data. Meanwhile for ellipsoidal pores the strain-life method (ϵ -N) gives more conservative estimation to fatigue life than stress-life method, while for triangular pores, stress-life method gives more conservative estimation. This is probably due to the linear elastic nature of the stress-life method which invalidates it when stress-concentration is too high. Further studies will be shown in the following part.

Table 5-3 The predicted fatigue life cycles of surface pore elli_0.1-0.1-0.1, elli_0.1-0.1-0.25 and tri_0.2-0.1-0.25 with experimental results

	Fatigue life cycles, x10 ⁵								
	Exp	ε-N	S-N	ε-N	S-N	ε-N	S-N	ε-N	S-N
		elli_ 0.1-0.1- 0.1	elli_ 0.1-0.1- 0.1	elli_ 0.1-0.1- 0.1	elli_ 0.1-0.1- 0.1	elli_ 0.1-0.1- 0.25	elli_ 0.1-0.1- 0.25	tri_ 0.2-0.1- 0.25	tri_ 0.2-0.1- 0.25
	K _t	2.1				2.5			
Max. Stress (MPa)	SS100	SS100_E'	SS100_E'	SS100_E	SS100_E	SS100_E	SS100_E	SS100_E	SS100_E
400	9.43	0.67	15.26	0.65	7.22	0.14	2.49	0.0069	0.0209
500	1.55	0.27	4.50	0.26	2.19	0.06	0.74	0.0034	0.0062
600	1.05	0.13	1.66	0.13	0.83	0.03	0.27	0.0019	0.0023
700	0.45	0.07	0.71	0.07	0.36	0.02	0.12	0.0012	0.0010
	SS200	SS200_E'	SS200_E'	SS200_E	SS200_E	SS200_E	SS200_E	SS200_E	SS200_E
400	4.10	0.32	6.43	0.32	6.29	0.07	0.96	0.0037	0.0063
500	0.64	0.13	1.78	0.13	1.79	0.03	0.27	0.0018	0.0018
600	0.38	0.07	0.63	0.06	0.64	0.02	0.09	0.0011	0.0006
700	0.17	0.04	0.26	0.04	0.27	0.01	0.04	0.0007	0.0003
	SS250	SS250_E'	SS250_E'	SS250_E	SS250_E	SS250_E	SS250_E	SS250_E	SS250_E
400	1.77	0.26	2.86	0.27	4.23	0.07	0.65	0.0617	0.4007
500	0.47	0.12	1.05	0.12	1.27	0.04	0.24	0.0308	0.1591
600	0.41	0.07	0.46	0.07	0.48	0.02	0.10	0.0177	0.0748
700	0.14	0.04	0.23	0.04	0.21	0.01	0.05	0.0112	0.0395
	SS300	SS300_E'	SS300_E'	SS300_E	SS300_E	SS300_E	SS300_E	SS300_E	SS300_E
400	0.81	0.21	1.58	0.21	2.39	0.06	0.40	0.0047	0.0107
500	0.52	0.10	0.63	0.10	0.79	0.03	0.16	0.0025	0.0043
600	0.12	0.06	0.29	0.06	0.32	0.02	0.07	0.0015	0.0020
700	0.16	0.03	0.16	0.03	0.15	0.01	0.04	0.0010	0.0011

Figure 5-20 Predicted fatigue life for different scanning speeds with different material parameters like UTS and E. The data is taken from model elli-0.1-0.1-0.1.

5.2.3.2 Comparison of Algorithms used for Assessing Fatigue Life

Different algorithms are compared as well with the results shown in Figure 5-21a-h. The data used for plotting is extracted from the series of triangular pores ShS_SE_Perp_0.2-0.1-1.0 and ShS_SE_Perp_0.2-0.1-0.25, and semi-ellipsoidal pores of 0.1-0.1-0.5, 0.1-0.1-0.25, and semi-spheroidal pores 0.2-0.2-0.2, 0.15-0.15-0.15 and 0.1-0.1-0.1, respectively. Experimental results are also put in the figures. As can be seen, the uniaxial stress-life method mostly predicts the largest fatigue lifetime while strain-life while biaxial Brown-Miller (strain-life, CP) methods are similar. This is the usual case for other shapes of pores. When the stress concentration factor is too high, strain-life method predicted lifetimes may exceed that of stress-life method as seen in

Figure 5-21a&b. Among the different types of pores, elli-0.2-0.2-0.2 pore lifetimes using stress-life method can match the experimental results of sample SS100 which may act as an equivalent crack initiation size for the sample SS100. For the other two samples SS200 and SS300, there are several types of pores that can match the experimental results using different algorithms. The

simulated fatigue lives using different algorithms fall within a factor of 100 which seems still within actual HCF life probabilistic data (2 - 100)[217] due to the large scattering nature of HCF behavior coming from varied pore shape and distribution. The SN curve is obtained from experimental results making the stress-life method suitable for the HCF estimation. Whether the strain-life method can be suitable will also depend on the materials parameters adopted for the simulation as strain-controlled fatigue experiment is not conducted on the AMed EH36 steel. The effective crack sizes converted from a series of semi-spheroidal surface pores are correlated to their fatigue lives as shown in Figure 5-21h with the three methods in uniaxial stress-life, strain-life and biaxial Brown-Miller (strain-life, CP) method, where the effective crack sizes are converted using Murakami's effective crack size method[22]. The curves show wavy features along with increased semi-spheroidal radius and the stress-life method predicts a higher number of life cycles than the strain-life method (without mean stress correction) and Brown-Miller method. Overall whether the stress-life method will produce accurate results depends on the severity of the stress concentration effect induced by the surface pores or the complexity of the 3D stress field.

(a)

(b)

(c)

(d)

(e)

(f)

Figure 5-21 Predicted fatigue life for surface triangular pore (a) 0.2-0.1-1.0($K_t = 5.82$), (b) 0.2-0.1-0.25($K_t = 5.37$), and semi-ellipsoidal pores (c) 0.1-0.1-0.5($K_t = 2.69$), (d) 0.1-0.1-0.25($K_t = 2.50$), and semi-spheroidal pores (e) 0.2-0.2-0.2, (f) 0.15-0.15-0.15, (g) 0.1-0.1-0.1, using different algorithms and (h) Influence of effective crack size on calculated fatigue life (effective crack size converted from semi-spheroidal pore series 0.2-0.2-0.2($K_t = 2.01$), 0.15-0.15-0.15($K_t = 2.15$), 0.1-0.1-0.1($K_t = 1.92$), 0.075-0.075-0.075($K_t = 1.85$), 0.05-0.05-0.05($K_t = 1.65$), 0.025-0.025-0.025($K_t = 2.0$), and 0.01-0.01-0.01($K_t = 1.61$)) (data from strain-life method without mean stress correction)

5.2.3.3 Effects of Size, Shape and Orientation of Pores on Crack Initiation Life

The effects of different pores to the fatigue crack initiation life are discussed in this section. Some of the results of predicted fatigue crack initiation lifetimes (N_i , assessed by FE-SAFE software) are listed in Table 5-4 for the triangular surface type of pores. Again those data are plotted in Figure 5-22a showing the predicted fatigue life and K_t (assessed by ABAQUS) of the semi-ellipsoidal pores versus major elliptical radius. Overall, the predicted fatigue life goes in an opposite way to K_t , that is, the higher the K_t , the lower the N_i obtained. For the z-series semi-ellipsoidal pores that are aligned perpendicular to the loading direction (with the pore extended deep into the body), when K_t goes higher, the predicted fatigue life first decreases and then falls

to a low value with no much fluctuation after the 1st point drop-off. Similar pattern is found with x series of ellipsoidal pores. For the y series of semi-ellipsoidal pores varying in the y axes, their K_t is lower than that of the z- & x-series of pores, while the predicted life demonstrates a wavy pattern, but basically following the opposite trend to K_t . Obviously, increases of x & z dimensions worsen the fatigue resistance of the steel because it makes the major axis lying along the plane normal to the loading direction. However, extending crack length along the y dimension has no detrimental effect. A contour plot in Figure 5-22b further illustrates the adverse effect of increasing x and z dimensions on the fatigue crack initiation life ($y = 0.1$ mm). The K_t & N_i versus length for triangular pore is shown in Figure 5-22b with more fluctuation compared with ellipsoidal pores. The lifetimes are shorter than that of ellipsoidal pores for both perpendicular oriented triangular pores and the parallel oriented triangular pores.

Table 5-4 Predicted fatigue crack initiation life cycles for different types of triangular surface pores

Pore type	Predicted life cycles (N_i , x 10 ³) (uniaxial strain-life method, without mean stress correction)						
	ShS_SE_Perp	ShS_ME_Perp	ShS_SC_Para	LoS_SE_Perp	LoS_ME_Perp	LoS_SC_Para	LoS_SE_Para
tri_0.2-0.1-0.25	2.1	2.7	58.8	1.8	2.3	16.8	28.0
tri_0.2-0.1-0.5	1.0	1.1	4.7	1.8	2.7	17.1	8.2
tri_0.2-0.1-0.75	0.6	1.5	30.5	0.6	2.5	48.6	42.3
tri_0.2-0.1-1.0	0.9	1.3	57.8	0.5	1.6	60.6	42.7
tri_0.2-0.1-1.5	0.4	0.7	11.0	1.1	2.5	17.2	20.6
tri_0.2-0.1-2.0	0.4	0.0		0.4			
tri_0.2-0.1-2.9_through	0.4				0.6		
tri_0.2-0.1-5.8_through				0.2			
tri-segments	0.03						
double tri_0.2-0.1-0.5	1.0						
tri_crossover_ 0.2-0.1-2.9 + 0.1-0.1-0.5					20.2

Figure 5-22 (a)&(c) Predicted life cycles (N_i assessed by FE-SAFE) and stress concentration factors (K_t) of (a)semi-ellipsoidal surface pores and (c) triangular surface pores (The data comes from the algorithm of strain-life method without mean stress correction.), and (b) Contours of fatigue crack initiation life cycles (using strain-life method) vs diameters of x & z (only elliptical 0.1 series with constant y = 0.1 mm).

The fatigue life contour plots of the semi-spheroidal 0.1-0.1-0.1 surface pores are shown in Figure 5-23(a-c). The lowest fatigue life point predicted by stress-life method (indicated in the Figure 5-23a) doesn't coincide with max. stress point from the above static analysis, but both of them

reside along the plane normal to the loading direction and near the sub-surface location. The min. fatigue life points for other two methods of strain-life and Brown-Miller (strain-life method, CP) methods are also different as shown in Figure 5-23b&c. Same thing happens for triangular pore tri_0.2-0.1-0.5 as shown in Figure 5-23(d-f). This maybe caused by the complex stress/strain field around the 3D pores.

Figure 5-23 Fatigue life contour plots showing the shortest fatigue life along the plane normal to the loading direction for elli_ShS_SC_0.1-0.1-0.1 pore with (a) stress-life method, (b) strain-life method, and (c) Brown-Miller (strain-life, CP) method, and for tri_ShS_SE_Perp_0.2-0.1-0.5 pore with (c) stress-life method, (d) strain-life method, and (e) Brown-Miller (strain-life, CP) methods.

5.2.3.4 Effectiveness Examination of Assigned Surface Finish Factor K_t to Smooth Surface Fatigue Model

The relationship between surface finish factor K_t and N_i is studied in this section. Firstly, the results of assigned surface finish factor K_t to smooth surface are presented. Specifically the K_t in FE-SAFE is defined as surface finish factor the same as fatigue strength factor which is the ratio of fatigue strength in smooth (polished) condition to that of surface finishing condition[218]. The

stress amplitude should be multiplied by K_t in fatigue evaluation. The calculated SN curves for K_t from 1 – 5.5 are shown in Figure 5-24. When K_t increases the S-N curves move to the left hand side, that is, to the lower fatigue life region. With reference to the experimental results, the SN curves of sample SS100 lies between $K_t = 1.0$ and 1.2, sample SS200 lies between $K_t = 1.0$ and 1.4 and sample SS300 lies between $K_t = 1.4$ and 2.0.

Figure 5-24 Effect of assigned K_t value on fatigue crack initiation life with experimental data points (data from strain-life method without mean stress correction). Note: The max. stress is nominal stress.

The N_i of the semi-ellipsoidal pores is correlated to K_t with the results shown in Figure 5-25a. The predicted number of cycles N_i for the smooth model (assigned with surface finish factor K_t) overwhelms that of the pore-incorporated model with the same K_t for x&z series all the way. The difference is more significant before about $K_t = 2.5 - 3.0$. While for y series, at $K_t = 1.3 - 1.4$ there are pores with N_i higher than that of assigned K_t scenarios. This may underline the severeness of the x&z series of pores that create stronger stress concentration effect to the surroundings. Same thing is observed to triangular pores as shown Figure 5-25b, the perpendicular oriented pores

associated N_i is similar to that of assigned K_t scenarios that could be due to the large K_t ranges (above 4) they both fall into. While for the less harmful parallel oriented triangular pores with smaller K_t (from 2 - 3), their predicted N_i is less than that of assigned K_t scenarios, which could be largely affected by the complex stress/strain field around the pores which severely deteriorate their fatigue performance. Similar phenomena are observed for the stress-life method that provides a more conservative prediction to fatigue life under the condition of larger stress concentration or more complex stress field, while providing fair results when stress concentration is rather small or simple. This is because the stress-life method is strictly used for HCF but not LCF, while the strain-life method can be applied for both HCF and LCF. For similar K_t values, ellipsoidal pores associated N_i are closer to that of assigned K_t series than triangular pores do. This illustrates inappropriateness to assign an equivalent surface finish factor K_t value to a smooth model or others without knowing the actual pore geometry.

Figure 5-25 (a) Effect of K_t on N_i for semi-ellipsoidal surface pores (data from strain-life method (without mean stress correction) at max. stress of 400 MPa for the series of 0.1 mm ellipsoidal pores with varying

length in x, y or z ranging from 0.1 – 2.0 mm), and (b) Effect of K_t on N_i for triangular pores (data from strain-life method at max. stress of 400 MPa)

5.2.4 LEFM Model for Crack Propagation of SLMed EH36 Steel Samples

Sigl[141] has used LEFM model to calculate total fatigue life of cast 8630 steel who treated shrinkages as pre-existing cracks within the component with the assumption of crack propagation consuming the majority of the fatigue life. Here the LEFM model is adopted to calculate fatigue crack propagation life of SLMed EH36 steel. The total fatigue life will be equated to the crack propagation life (N_p) based on above assumption. Then the predicted N_i using FE-SAFE and calculated N_p using LEFM will be compared with the experimental data in the end. The LEFM model is expressed as $\frac{da}{dN} = A (\Delta K)^n$ with $\Delta K = \Delta \sigma F \sqrt{\pi a}$, where A and n are the material constants, ΔK is the stress intensity factor range, $\Delta \sigma$ is the stress range, a is crack depth and F is a shape factor that relates crack front geometry to crack depth. Through integration from the initial crack size a_i to the final crack size a_f , the total number of fatigue crack propagation cycles (N_p) is expressed as

$$N_p = \int_0^{N_f} dN = \int_{a_i}^{a_f} \frac{da}{A(\Delta \sigma \sqrt{\pi a} F)^n} \quad 5-1$$

Forman[219] has proposed a model to estimate the shape factor $F(a/D_{sp})$ (D_{sp} is the diameter of a round specimen) for the semi-circular cracks grown in round bars

$$F\left(\frac{a}{D_{sp}}\right) = g\left(\frac{a}{D_{sp}}\right) \left(0.725 + 2.02 \frac{a}{D_{sp}} + 0.37 \left(1 - \sin\left(\frac{\pi}{2} \frac{a}{D_{sp}}\right)\right)^3\right) \quad 5-2$$

$$g\left(\frac{a}{D_{sp}}\right) = 0.92 \left(\frac{2}{\pi}\right) \left(\tan\left(\frac{\pi}{2} \frac{a}{D_{sp}}\right) / \left(\frac{\pi}{2} \frac{a}{D_{sp}}\right)\right)^{1/2} / \cos\left(\frac{\pi}{2} \frac{a}{D_{sp}}\right) \quad 5-3$$

However, in this work, the proposed crack is simulated from a rectangular fatigue specimen. For simplicity, the shape factor of $F = 0.65$ for surface cracks and 0.5 for subsurface cracks are

suggested by the Murakami equation [22] and all the pores are treated as surface cracks, so $F = 0.65$ is applied here.

To estimate the initial crack size a_i there are a few methods. One method is to estimate a_i directly from the fractured surfaces of tested fatigue samples. Due to the complex shape of a pore that initiates a crack, a converted size in the form of $\sqrt{area}$ is used, where $\sqrt{area}$ is the square root of the projected defect area. The length and width of the complex shape of pore was firstly measured from the fractured surface under SEM which will then be used to convert to $\sqrt{area}$ using online software (Altair) that treated the pore as microcrack using small defect analysis[200]. The underlying basis of the online Altair software is to simplify the pore into a microcrack with the input of length and width, from which the $\sqrt{area}$ can be calculated and is equated to a_i . The estimated initial crack sizes using this method for the samples SS100, SS200, SS250 and SS300 are 168 μm , 313 μm , 406 μm and 627 μm , respectively.

Another commonly used method to estimate a_i is to calculate it using fatigue limit from the Murakami equation of $a_i = \sqrt{area} = (1.43 \frac{Hv+120}{\sigma_{FL}})^6$ [22], where Hv is the microhardness, σ_{FL} is the fatigue limit and $\sqrt{area}$ is the square root of defect projection area. Alves et al.[220] and others [221, 222] also proposed a similar method in estimating a_i using the Kitagawa-Takahashi diagram in the form of $a_i = \frac{1}{\pi} (\frac{\Delta K_{th}}{Y \Delta \sigma_f})^2$, where ΔK_{th} is the stress intensity threshold, $\Delta \sigma_f$ is the fatigue limit and Y is the geometric function with the stress intensity factor $K = \sigma Y \sqrt{\pi a}$. The fatigue limits can be calculated using the fitted SN curves from the experimental data that have been shown above for the sample SS100, SS200, SS250 and SS300, which are 224, 187, 147 and 122 MPa, respectively.

Those will be used to estimate the sizes of the cracks that initiate the fatigue failure. Thus, a_i can be estimated as 315, 1754, 8405 and 26385 μm , respectively for the samples SS100, SS200, SS250 and SS300. Obviously the estimated crack size is far larger than the actual crack size that initiated the fatigue failure. This is mostly due to the large amount of pores existed inside those porous samples, which elevates the actual local stress during the fatigue test to a much higher level. Thus, the a_i values are taken directly from the experimental measurements. Note that the crack propagation stage was possibly extrapolated to a small crack propagation regime with the LEFM method (depending on the critical distance of the AMed EH36 steel for long crack propagation[223]). The final crack length corresponds to the point of yielding, or where the critical stress intensity factor K_c is reached. For simplicity, the final crack length, a_f , is defined as half the thickness of the rectangular samples used in this study, i.e., 1.45 mm. For more accurate calculation, actual final crack length shall be used where a_f is longer for more densified sample (longer period of stable crack propagation) such as sample SS100 and about this value for more porous samples (shorter period of stable crack propagation) such as samples SS250 and SS300. The final crack length is also dependent on the stress amplitude applied. This will not affect the calculated SN curve from a large amount for densified samples (within 3% for sample SS100 and 8% for sample SS200 compared with $a_f = 2.45$ mm).

In the calculation of total fatigue life, A is taken as 1×10^{-13} , n is taken as 4.5 for the EH36 steel from literature[224], and $\Delta\sigma = \sigma_{\text{max}} - \sigma_{\text{min}}$. The calculated SN curves (in dotted lines) for the samples SS100, SS200, SS250 and SS300 are shown in Figure 5-26 with the experimentally obtained SN curves (in solid lines) included. The LEFM model shows comparable SN curves to those experimentally determined for all the samples. As LEFM assumes crack propagation stage

dominates total fatigue life, it is demonstrated that majority of the fatigue life of the samples could be spent at the crack propagation stage. This is also based on theoretical understanding that the critical distance for long crack propagation (applicable for LEFM mode) can be in the size of tens of microns[223]. It is known that the calculated SN curves for all the samples are based on the actual crack sizes as indicated above. The lowered fatigue strength of the more porous samples, mainly SS200, SS250 and SS300, is mainly due to the much higher porosity levels found in these samples, which affects both crack propagation and effective nominal and local stress applied during the fatigue tests. However as the unique microstructure of AMed steels may defer their fatigue behavior away from conventionally manufactured grade, experimental studies will be need to prove this[151].

For the sample SS100 and SS200 with certain plasticity (lower hardness), in the longer fatigue life regime (lower stress amplitude), the predicted total number of life cycles are lower than the experimental values, which could be due to the microstructure interaction in the earlier stage of small crack regime where the fine AMed grains may block the crack propagation effectively[151]. This essentially invalidates the LEFM model while crack initiation approach may suit better by considering local plasticity near crack tip in a shear dominated failure mode. For the samples SS250 and SS300 with less plasticity but higher brittleness (both higher hardness and higher porosity level), the plasticity may not be critical to them. And the internal porosities may have deteriorated their fatigue properties. A study of using a simplified shape of crack to represent an actual complex shape of crack is practically meaningful as it is not always possible to get the accurate morphology of the actual pores/cracks/defects inside the samples due to the limitations of actual measurements.

The calculated results may vary depending on the material parameters used. A sensitivity study was conducted for a series of materials parameters n , A , F and a_f for sample SS100 and the results are illustrated in Figure 5-27a-d. As seen, the factors of n and A have significant effect to the fatigue life cycle predicted. The effect of F and a_f is not that significant.

Figure 5-26 LEFM calculated fatigue life (N_p) with experimental results for samples SS100, SS200, SS250 and SS300.

(a)

(b)

(c)

(d)

Figure 5-27 Material parameter sensitivity study on a) n , b) A , c) F and d) a_f together with experimental results (fitted with solid line)

5.3 Summary

5.3.1 Experimental

- A131 EH36 steel samples with high strength and fair ductility were additively manufactured via selective laser melting by using relatively low laser scanning speeds (e.g., 100 mm/s). A higher heat input improved the densification of the samples and triggered effective in-process tempering and extensive phase transformation and recrystallization as well, which contributed to the better tensile and fatigue properties of the built samples.
- S-N curves were studied and a porosity dominated fatigue failure mode was observed from all the fatigue tested samples. A crack mostly originated from a large lack of fusion void (about hundreds of microns in size) with a complex shape and sharp corners, which was embedded in the highly densified samples (>99% densification). The effective nominal stress/strain applied to the samples during the fatigue tests could be increased due to the ubiquitously distributed intrinsic pores inside the SLMed EH36 steel samples. Unmelted particles and inclusions could contribute to HCF in a large aspect as well.
- It was found that the lower the stress level, the longer the stable crack propagation distance was and vice versa. The whole fractured surface was dominated by a fast fracture zone for the highly porous sample SS300. For all the other samples, tearing topology surface failure mode was observed together with weak striations along the stable crack propagation stage while a void coalescence failure mode was also observed in the form of dimples along with the fast fracture stage. Many small cracks were found within MSC.
- Experimentally determined fatigue limits were far below predicted values based on an empirical relationship with Hv and UTS for traditional steels, but relatively comparable with predicted values based on Murakami equations with the effective crack sizes estimated from the fractured surfaces. New models correlating σ_{FL} , Hv, ϵ , and UTS were proposed.

- Grain orientations for the horizontally built samples might favor early crack propagation and lowered fatigue life. Some soft phases such as retained austenite located at martensite lath boundaries and prior columnar grain boundaries or within them were believed to contribute to the fatigue behavior of the samples as well.
- The high strength coupled with fair ductility of the 3D printed EH36 steel samples resulted from the AM process parameter tuning made in-process material design possible.

5.3.2 Simulation

- Fatigue crack initiation life was affected by the newly proposed triangular & semi-ellipsoidal/spheroidal pores in terms of their shape, position, size, orientation and pore-pore interaction. The more realistic surface triangular pores showed a higher stress concentration effect than the semi-ellipsoidal pores due to the sharper corners/radii of the triangular pores that could shorten the fatigue crack initiation stage subjected to the loading direction. The surface pores located near the edge of the surfaces induced a higher K_t than those located near the centre of the surfaces.
- K_t increased with the aspect ratios of the pores till a peak value and then reduced to a stabilized value.
- Assuming an equivalent surface finish factor (same as fatigue strength factor) K_t value for a smooth surface model or other models might not be accurate, which was dependent on the actual geometry and size of the pores.
- The size and shape of the pores along with induced complex stress state worked in a complex manner to affect the fatigue behavior of the models. Basically, increasing the

dimension in the y axis that was the loading direction did not show a detrimental effect on the model's fatigue crack initiation life, but increasing the dimensions in the x & z axes worsened the fatigue life. When the K_t values or the dimensions of the pores in the x and z axes were large enough, the crack initiation stage could be largely shortened.

- The K_t is mainly determined by the radius of the sharp corner, however pore-pore interactions induced a different stress concentration behavior from the single pores. Different stress concentration behaviors were also expected for the pores with more complex shapes, which may lead to the fatigue behavior away from the single pores in both crack initiation and propagation stages. The 3D pores gave lower stress concentrations than the 2D pores.
- The stress-life method could predict a larger fatigue life than the strain-life method and Brown-Miller biaxial methods. When the stress concentration induced was relatively high, it invalidated the stress-life method due to its high cycle nature. The uniaxial fatigue algorithm gave similar results as the multi-axial fatigue algorithm (Bi-axial Brown-Miller) due to the applied uniaxial tensile cyclic loading. With Neuber's plasticity correction, the fatigue life cycles did not change demonstrating the brittle nature of the SLM processed EH36 steel.
- The AMed materials parameters were applied for fatigue life prediction. Higher UTS and E gave longer fatigue lifetimes.
- The LEFM method could be effective in predicting the total fatigue life for the AMed EH36 steel samples that would depend on the initial crack size and whether the small crack propagation could be neglected.

Chapter 6 Microstructure and Mechanical Properties of DLD Printed EH36 Steel Samples

The results of the DLD processed EH36 steel samples will be presented in this chapter. With the evaluations of the chemical compositions, microstructures, and tensile, charpy impact and fatigue properties along the 4 built orientations, the suitability of DLD process as a potential advanced manufacturing technique will be evaluated.

6.1 Chemical Composition Analysis

The results of chemical element analysis for DLD processed EH36 steel sample are listed in Table 6-1. Most elements are within ASTM standard demonstrating the conformity of DLD as an alternative process to conventional manufacturing method in producing equivalent grade of steel. The slightly lowered Al value in the printed steel sample is due to its low concentration in the raw powder as shown in the table.

Table 6-1 Chemical element analysis of DLD processed EH36 steel sample

Elements	C	Si	Mn	P	S	Cr	Mo	Ni	V	Cu
Printed sample	0.078	0.336	1.227	< 0.001	0.007	0.143	0.024	0.058	0.124	0.013
ASTM Standard	0.18	0.1-0.5	0.9-1.6	0.035	0.035	0.2	0.08	0.4	0.05-0.1	0.35
Elements	Nb	Ti	Al	O	Fe					
Printed sample	0.06	< 0.001	< 0.001	0.094	98.444					
ASTM Standard	0.02-0.05	0.02	Min 0.015	---	Balance					

6.2 Phase Analysis

In Figure 6-1 the XRD patterns of printed EH36 steel samples XY_0°, XY_45°, XZ_90° and XZ_45° show mainly a BCC crystal structure that belongs to ferrite or martensite. The five characteristic peaks are indexed as (110), (200), (220), (211) and (310). Other phases such as austenite or carbide are not detected. The later found small amount of carbide using EBSD is in a tiny amount which can not give clear XRD peaks. Consistent phase formation can be observed in the printed samples in all directions.

Figure 6-1 XRD patterns of EH36 steel samples printed in XY_0°, XY_45°, XZ_90° and XZ_45°

6.3 Microstructure

The optical micrographs of vertical cross sections of samples built in XY_0°, XY_45°, XZ_90°, XZ_45° are shown in Figure 6-2a-d, respectively. The density being analyzed using image J software is above 99.5% for samples XY_0° and XY_45°. Small pores are mostly in a spherical shape and in the size of a few microns, which are uniformly dispersed within the matrix (indicated in the optical micrograph). These could be the trapped gas not being able to escape from the molten metal during printing. Large amount of Ar gas used tends to increase the chance of gas porosity formation. There are large pores being found in sample XZ_90° from the OM as well as the later SEM micrographs on fractured surface of fatigue sample. The pores are elongated which seems lined up ranging up to a few mms with inclusions/un-melted powder inside. They are believed to be lack of fusions due to un-optimized process parameters or complex melting pool behavior. Those big pores are sometimes caused by too much energy input induced unstable melting pool behavior. Oxidation can occur under such conditions with too much heat and high temperature retention. The lack of fusions and big pores can affect mechanical properties in various aspects to tensile, charpy and fatigue. It will be discussed in the mechanical properties section.

All the microstructure shows periodically appeared layered feature with melting pool depth ranging about 1 mm as shown in Figure 6-3a. Within the melting pool, large columnar prior austenite phase grains are still visible grown from the bottom of the melting pool to the centre of it. Their boundaries are delineated by either short elongated ferrite grains (indicated in Figure 6-2a), martensitic structure (indicated in Figure 6-2c) or mixture of bainitic & martensitic structures (indicated in Figure 6-2d) which have transformed within the columnar mother grains. The short elongated ferrite grains are probably transformed phases from prior austenite grains. Sometimes phase changes or grain growth could also occur upon reheating from the heat of repeated laser

scanning. They are preferentially growing along the boundaries so that the prior grain morphology is delineated. The complex or graded thermal cycles of DED process along the built height can be deduced from the findings of the top layer microstructure as shown in Figure 6-3b which consist mainly martensite and widmanstatten like ferrite. Without any further heat cycles above the top layer it shows that the heat has been taking effect on the previous built materials along the entire built direction and microstructure evolution are induced.

Overall samples in horizontal directions printed by continuous strategy possess mainly ferrite grains in short elongated shape. For the two vertical directions, due to very high laser energy input smaller contact areas encounters low heat dissipation rate that is suspected to excess heat. The influence of excess heat includes excess heat accumulation, high cooling back temperature and possibly low cooling rate. The effect will be either over soften the microstructure within the built samples or promoting pore formation so as to the inferior mechanical properties, so printing strategy is changed from continuous to intermittent to avoid the detrimental effect of excess heat. Compared with continuous printing strategy being applied to the two horizontal directions where each time one sample was built until finished, the intermittent printing is to finish one layer for all the 6 pieces of samples simultaneously before another layer is deposited. Consequently intermittent printing strategy allows time intervals for the samples to cool down to a relatively lower cooling back temperature whereas cooling rate is obviously accelerated as well. In a result the microstructure of samples XZ_{45°} and XZ_{90°} is rather finer than those printed using continuous strategy in horizontal directions. The main phase of sample XZ_{45°} contains tempered martensite, bainite, acicular ferrite and finer ferrite. Sample XZ_{90°} contains mainly martensite as shown in Figure 6-2d&c. The excess heat accumulation is avoided for the vertical samples and

structure differs resulting in lesser degree of phase transformation and smaller heat affected zone, etc. Microstructure inhomogeneity along the building direction especially for large height or size of the component needs to be examined to ensure sound mechanical properties.

As mentioned above the obtained microstructure shows highly dependency to the thermal cycles within the melting pool. According to theory of solidification, the grain morphology and size is mainly depending on the temperature gradient (G) and solidification rate (R)[82, 185], etc. Those factors are affected by process parameters (such as laser power, scanning speed, powder feed rate, etc.) and material physical properties, etc. Specifically the G/R ratio determines grain morphology and the $G \times R$ value determines the cooling rate which will affect the fineness of the grains accordingly. The complexity of AMed microstructure can be further influenced by the cooling back temperature, which will determine the composition of the resultant phases for this high strength low alloy steel. When the cooling back temperature is above martensite start temperature (M_s), there will be no martensite phase formation. If the cooling back temperature is below martensite finish temperature (M_f) and meanwhile the cooling rate is fast enough to avoid the nose in the continuous cooling transformation (CCT) diagram, full martensitic transformation can be expected. In other situations the final microstructure will be mixtures of different phases or structures that may contain one or more of them such as martensite, ferrite and bainite, retained austenite, carbide, pearlite or other metastable new structures. Compared with the smaller size of dendrite grains resultant from selective laser melting process the prior austenite dendrite grains from DED process are larger (a few microns to tens or even hundreds of microns)[225], where phase transformation takes place within those large dendrite grains.

It is clearly seen from the horizontal samples, zones with refined grains (being circled out in Figure 6-2a&b), in smaller aspect ratio, are regarded as heat affected zones (HAZ). The austenization or recrystallization may take place within HAZ as it is close to the fusion line of upper layer melting pool, which may experience high temperature effect. The HAZ separates the layered structures filled with elongated columnar grains along the directions indicated by the yellow double headed arrows. An illustration diagram in Figure 6-4 describes the printing layers with the 1st layer being printed in 0° (parallel to the paper plane) and the 2nd layer being printed in 90° (a rotation of 90° to perpendicular to paper plane) in stripe scanning strategy, which illustrates an alternating of 90° being applied between two printing layers. The double headed arrows indicate the melting pool depth in a size of a few hundred microns (500 μm). The dotted lines indicate the heat affected zones located just below the melting pool boundaries of the upper layer in a range of a few hundreds of microns depending on the extending of heat effect. The green double arrow indicates the re-melting layer. For direct laser deposition process, the HAZ is relatively large compared with SLM process due to its higher energy input and larger spot size, with a relatively lower cooling rate. This lower cooling rate together with much slower scanning speed incurred in powder blow system of DED lead to a longer retaining time at high temperature. The long time retaining at high temperature makes the deposited material have a larger chance to be heat affected and forming a larger HAZ, where re-austenization or phase transformation may take place. In a comparison, the heat effect in SLM process may only make tempering possible[173].

Figure 6-2 Optical micrographs of etched EH36 steel samples built in (a) XY_0°, (b) XY_45°, (c) XZ_90°, and (d) XZ_45°, with the heat affected zones separated by dash lines in (a) & (b)

Figure 6-3 Optical micrographs of etched EH36 steel samples built in (a) XZ_90 showing the melting pools periodically appeared along the build orientation, and (b) XY_0° showing the top layer microstructure of the samples

Figure 6-4 Schematic illustration of printed layers with melt pool structures formed during LAAM process in a stripe scanning strategy by alternating of 90° between each two consecutive layers

Similar to optical micrographs, the EBSD IPF maps (with inverse pole figures inserted) and phase maps shown in Figure 6-5, display the characteristics of grains for the samples XY_0°, XY_45°, XZ_90° and XZ_45°. Mainly two phases Iron BCC (body centered cubic) and Fe₃C are recognized. As a low carbon steel (0.2% C), the tetragonality of the martensite structure of this steel is low which makes it difficult to differentiate BCT (body centered tetragonal) from BCC. Therefore the iron BCC could be ferrite or martensite. There could be some upper bainitic and pearlite phases due to varied local thermal cycles but cannot be distinguished[226]. The estimated bainite starting

temperature is 628°C from the empirical equation for low carbon low alloy steel (continuous cooling): $B_s = 810 - 270C\% - 90Mn\% - 37Ni\% - 70Cr\% - 83Mo\%$ and the B_s and B_f range = 195°C[227]. For sample XY_0° and XY_45° the Iron BCC phase is ferrite with clear crystallographic recognition and grain features as being recognized by the software. The IPF mappings show part of the melting pools due to small areas scanned. The grains are rather similar to that observed by above microscope. For sample XZ_90°, the Iron BCC phase is martensite or tempered martensite where the EBSD IPF image gives very low hit rate for the sample XZ_90° due to its highly strained structure. Too much lattice distortion makes matching of crystallographic parameters difficult. For sample XZ_45°, a mixed structure consisting of martensite, ferrite and minor bainite can be determined based on the observation of grain morphology. The dark region is again due to low hit rate from highly strained structures such as martensite. As EBSD can only match the database with known lattice constants, the unmatched structure (with low hit rate) within the EH36 steel samples are regarded as martensitic structure in this work.

(c)

(d)

(e)

(f)

(g)

(h)

Figure 6-5 EBSD IPF maps (a,c,e,g) with inverse pole figures inserted and phase maps (b,d,f,h) of EH36 steel samples printed in XY_0°, XY_45°, XZ_90°, and XZ_45°, respectively

6.4 Microhardness and Integrity of Deposited Material

The microhardness of the deposited EH36 steel is shown in this section together with the integrity measurement of the deposited materials.

6.4.1 Integrity of the Deposited Material

Figure 6-6 shows the cross-section views of the deposited EH36 sample (3 layers). It can be observed that crack-free deposition can be achieved. Density is about 99.6%. The pore size is smaller than 50 microns.

Figure 6-6 Cross-section view of the deposited EH36

6.4.2 Microhardness

Figure 6-7 shows the hardness profile measured in the cross-section, as shown in Fig. 5. It can be observed that microhardness in the deposited material is very homogeneous (average 230 HV). It is also equivalent to the specified hardness of cast EH36.

Figure 6-7 Micro-hardness of deposited EH36 from the top surface to the base material

6.5 Mechanical Properties

6.5.1 Tensile Properties

The tensile properties of all samples are summarized in Table 6-2. After process optimization, tensile properties of the printed EH36 material in all the 4 orientations satisfy ASTM standards. The yield strength of vertical sample XZ_45° is even better than that of the samples printed in horizontal direction. As discussed earlier this is due to the stronger phase of tempered martensite or bainitic structures formed in the vertical samples XZ_45°, which is possibly strengthened further by dislocation, solid solution, etc. A typical stress-strain curve is shown in Figure 6-8 with sample XZ_45°.

However, the slightly lowered strength for vertical sample XZ_90° is attributed to the process induced porosity. It is confirmed by the SEM micrographs of the fractured surface of tensile sample XZ_90° as shown in Figure 6-9a. A few pores in the size of up to 100 μm (indicated by yellow

arrow) are revealed. The enlarged view of dimples for all samples ranging up to a few microns is shown in Figure 6-9b. The phase, grain morphology/size, solid solution, dislocation density, precipitation and intrinsic defects can act together and influence the strength and ductility of the samples built in different orientations from different thermal cycles. In general, DLDed EH36 steel shows better tensile properties than conventional grade which is about 500 MPa and 600 MPa for yield and UTS, respectively as shown in Table 6-2[228]. This roots from variations in being formed microstructure. The chemical compositions of the EH36 raw powder and AMed EH36 steel samples differ from that of traditionally used EH36 raw materials. Additional alloy elements can increase the hardness and strength of the material, and harder phases like martensite, bainite and pearlite possess higher strength than softer phase such as ferrite and austenite. Conventional EH36 steel mainly contains ferrite and small amount of pearlite[228], where harder phase may also form depending on the control of process thermal cycle. AMed grade of EH36 steel contains finer ferrite grains and quite some amount of harder bainite, martensite and microphases obtained from its rapid cooling rate, which is more similar to welded EH36 microstructure. However welded grade has large & hardened heat affected zone and welding cracks, etc., which may deteriorate materials' mechanical properties which has been proved by plenty of research work [143, 202, 203, 228]. In comparison, AMed EH36 has smaller heat affected zone in micron scale that may not affect the mechanical properties significantly.

Table 6-2 The tensile properties of the samples printed along the 4 orientations

Orientation	XY_0°	XY_45°	XZ_90°	XZ_45°	Conventional	ASTM standard
UTS (MPa)	708±10.4	695±2.1	644±8.6	690±15.2	572[228]	490-620
σ_y (MPa)	612±21	600±5.5	578±12.1	624±24	500[228]	355
ϵ (%)	28±0.9	33.3±0.3	26±2.5	24±0.7	22[228]	22

Figure 6-8 Stress strain curve of sample XZ_45°

Figure 6-9 SEM micrographs showing the fractured surfaces of tested tensile sample in XZ_90° (a) overview and (b) enlarged view of the dimples spread over the entire fractured surface

The UTS, yield strength (σ_y) and microhardness (Hv) of samples printed along horizontal directions under continuous printing strategy, consisted with mainly ferrite grains are correlated to grain size (d) using Hall-petch relation in the form of $\sigma = \sigma_0 + kd^{-1/2}$ [20, 229, 230], where σ_0 and k are interpreted as resistance for dislocation movement from lattice and grain boundary,

respectively. The data are fitted to linear relationship with $d^{-1/2}$ and shown in Figure 6-10 with fitted equation inserted, however those fitted mathematical models differs from conventionally manufactured grade[231, 232], e.g., Hall-petch parameter k with the fitted value of $981.8 \text{ MPa } \mu\text{m}^{1/2}$ for σ_y is larger than the reported values of conventional manufactured ferritic steels, $600 \text{ MPa } \mu\text{m}^{1/2}$. As the data are not well fitted in linear relationship, the difference could not interpret a sole dependency of yield strength to grain size [232-234], where other defects may also influence the strength and microhardness, such as dislocation, solid solution, grain orientation, etc., which is unique for the rapid solidification process of DED.

Figure 6-10 Correlation between UTS, σ_y , Hv and grain size ($d^{-1/2}$) measured from the samples printed using continuous printing strategy. The short diameter of the elongated grain is taken as the grain size d .

6.5.2 Charpy Impact Properties

The charpy impact properties of all samples printed along the 4 directions are listed in Table 6-3. All the samples satisfy ASTM standard of 35 J (at -40°C). Figure 6-11 shows the SEM micrographs

of fractured surface of tested charpy impact samples XY_0° (a-c), XZ_90° (d-f), and XY_45° (g-i) for crack initiation region, brittle to ductile transition region and dimples in ductile region. Crack origins locate at a few hundred microns below the fracture initiation region (surrounded dotted line), as illustrated in the samples in Figure 6-11 and indicated by yellow arrow, with the radiation lines spreading from the crack origins. The dimple sizes for all samples are ranging from 1-2 to 5-6 μm which is equivalent to grain size. There are no obvious defects being found from the fractured surfaces of most samples including the two vertically printed samples. The lowest absorbed energy incurred with samples XZ_90° are due to their hard martensitic structures as being discussed above. Compared with conventional manufactured EH36 steel, 429 J (TMCP) and 110 J (welded) listed in Table 6-3[228], the toughness of AMed EH36 steel is much lower. This can be chased back to microstructure difference. Among them, the TMCP EH36 steel has most toughest microstructure consisted with mainly soft ferrite (with minor pearlite) at a microhardness of HV185. With a slightly higher a hardness of HV210[203] welded EH36 steel consists fine ferrite, bainite and some interlath microphases, where fine ferrite is believed good for crack resistant as it enhances both strength and toughness simultaneously [235]. The lower toughness of AMed EH36 steel than TMCPed EH36 grade is attributed to both higher hardness and inherent pores.

Table 6-3 The charpy impact properties of samples printed along the 4 orientations

Orientation	XY_0°	XY_45°	XZ_90°	XZ_45°	Conventional	ASTM standard
Hv _{0.3}	220±11.9	220±7.6	281.5±18.3	279.4±15.2	185[228]	
Charpy impact energy (J, tested at @ -40°C)	48.2	83.9	39	63.3	429[228] 110(welded) [228]	35

(a)

(b)

(c)

(d)

(e)

(f)

(g)

(h)

(i)

Figure 6-11 SEM micrographs of fractured surfaces of tested charpy impact samples printed in XY_0° (a-c), XZ_90° (d-f), and XZ_45° (g-i), showing crack initiation points, brittle to ductile transitions, and enlarged views of dimples in final fracture regions, respectively. The dotted line in (a) illustrates the crack initiation region in sample XY_0°.

6.5.3 Fatigue Behavior

6.5.3.1 Correlation Between Fatigue Life and UTS, Yield and elongation

The fatigue life of printed samples tested at 400 MPa are shown in Table 6-4. Results at other stress levels are not shown here. 400 MPa is below the yield strength for all the samples along 4 orientations. The data used for illustration in Figure 6-12 are all tested at 400 MPa. As seen from Table 6-4 samples XY_{45°} & XZ_{45°} run out at this stress level. Sample XY_{0°} outperforms sample XZ_{90°}. The obvious difference between the samples implies defect dominant fatigue failure mode as confirmed from the later fractographic study. Similarly for sample XZ_{90°} large lined-up defect makes its fatigue life inferior to other samples. Meanwhile the brittleness of martensitic structure of sample XZ_{90°} may further reduce its fatigue resistance as high strength steel is more sensitive to defects, which may accelerate crack initiation and propagation.

As displayed in Figure 6-12 strong positive proportional trend can be correlated between fatigue life and ultimate tensile strength, yield strength with the Karl Pearson coefficient of $r = 0.71$, while a moderate correlation between fatigue life and elongation with the Karl Pearson coefficient of $r = 0.65$. Even though there are unavoidable internal voids existed inside the AMed samples, the strong correlation with UTS and σ_y still holds. The slightly less correlation with elongation could be due to the more sensitive effect of pores to elongations[209]. All the above interpret that the defects like pores, cracks, inclusions and anisotropic grain affect the high cycle fatigue behavior at a large aspect including increased local stress concentration, decreased young's modulus & ductility, varied crack propagation path, etc. Therefore the samples' strength/ductility, defect as

well as the grain orientation may balance each other and deem the high cycle fatigue behavior of the AMed EH36 high strength steel.

Table 6-4 The fatigue life cycles for the samples printed along 4 orientations (tested at 400 MPa of Max. stress)

Orientation	XY_0°	XY_45°	XZ_90°	XZ_45°
Fatigue life cycle (x10 ⁵ , tested at $\sigma_{\max} = 400$ MPa)	4.2	Run-out	1.5	Run-out

Figure 6-12 Relationships between fatigue life cycles, ultimate tensile strength, yield strength and elongation of EH36 steel samples printed in the four orientations

6.5.3.2 S-N curve and Fatigue Limit

The SN curves are built for samples XZ_90°, XZ_45° against Basquin relation in the form of $\sigma_a = \sigma'_f (2N)^b$ ($R = 0.1$) [147, 195, 196], where σ_a is stress amplitude, σ'_f is material's true tensile

strength and b is the slope of logarithmic SN curve as shown in Figure 6-13. The fitted σ'_f and b values are listed in Table 6-5. Fatigue limit predicted are 146 MPa and 170 MPa, respectively for sample XZ_90° and XZ_45° using the above fitted curve corresponding to a cycle of 10^6 . The higher fatigue limit of the sample XZ_45° is believed mainly attributed to its less amount, which will be enclosed from fractured surface.

Figure 6-13 S-N curves for EH36 steel samples printed in XZ_90° tested under 550, 500 and 400 MPa (max. stress), and in XZ_45° tested under 600, 550, 500 and 450 MPa (max. stress).

Table 6-5 Predicted fatigue limits and correlated constants

Orientation	UTS (MPa)	HV _{0.3}	σ'_f (MPa)	b	Fatigue limit ($\times 10^6$) (MPa)		
					Curve fitting	σ_{FL}/UTS	$\sigma_{FL}/HV_{0.3}$
XY_0°	708	220	---	---	---	---	---
XY_45°	695	220	---	---	---	---	---
XZ_90°	644	281.5	1546.0	-0.171	146	0.2	0.5
XZ_45°	690	279.4	3761.0	-0.224	170	0.2	0.6

6.5.3.3 Mathematical/Empirical Models

The traditional relationship of $\sigma_{FL} = 0.5 \text{ UTS}$ and $\sigma_{FL} = 1.6 \text{ Hv}$ is also examined for the two samples. The ratios of σ_{FL} to UTS are 0.2 for both samples, while the ratios of σ_{FL} to Hv are 0.5 for sample XZ_90° and 0.6 for sample XZ_45°. They are below the traditional relationships. The lowered ratio is again mainly attributed to the intrinsic pores of AMed metals that deteriorate its fatigue performance [136] as discussed in previous report[135]. Murakami equation is believed to give more accurate estimation to fatigue limit by using actual crack size, in the form of $\sigma_{FL} = 1.43 \frac{HV+120}{(\sqrt{area})^{1/6}} ((1-R)/2)^\alpha$ ($\alpha = 0.226 + Hv \times 10^{-4}$) (for surface crack)[22, 111]. Here crack size is the converted value from measured dimensions of initiating defect using online software Altair[214], which are 560 μm and 300 μm , respectively for samples XZ_90° and XZ_45°. The calculated fatigue limits using converted crack size is 200 MPa and 221 MPa for sample XZ_90° and XZ_45°, respectively, which differed by a 23 - 27% from that of experimental curve fitting. The difference could be related to the complex shape of 3D crack or intrinsic porosity induced higher effective nominal stress being applied to the test samples during fatigue test.

6.5.3.4 Comparison with Conventionally manufactured EH36 Steel

Compared with conventional manufactured EH36 steel, the fatigue strength of TMCPed steel plate and welded steel is 299 MPa and 212 MPa, respectively (fully reversed condition ($R = -1$))[143, 202, 203]. The fatigue limit of sample XZ_45° is 230 MPa (by using Goodman relationship to count mean stress effect) which compares or slightly better than that of welded EH36 grade, but

lower than TMCPed steel plate. In view of the microstructure, the heat affected zone of welded grade of EH36 is consisted of white ferrite, granular bainite and gray lamellar pearlite with a hardness of around HV220, where crack propagates along the fusion line. The microstructure of XZ_45° sample consists of bainite, martensite, ferrite and some microphases with similar hardness of around HV220. The similarity in microstructure and microhardness essentially equate fatigue strength of DLD printed EH36 steel with welded grade of EH36 steel. The slightly lowered fatigue strength of welded grade could be due to the hydrogen embrittlement where hydrogen is inevitably introduced into welding process. The lowered fatigue strength of DLD printed EH36 steel than TMCPed is mainly due to the porosity defect.

6.5.3.5 Fracture Behavior

The fractured surfaces of tested fatigue samples are viewed by SEM with the micrographs in Figure 6-14 show the crack origins in (a,c,e,g) and the dimples in the fast fracture regions in (b,d,f,h) for XY_0°, XY_45°, XZ_90° and XZ_45°. All the cracks originate from surface voids with inclusions inside demonstrating a defect dominated fatigue failure mode. The inclusions are about a few hundreds of microns, which are mainly unmelted particles or oxides that are formed during the printing process. The crack initiating pore size of sample XZ_45° is smallest which explains the best fatigue life cycle for this sample followed by XY_0°. Among the samples, sample XZ_90° shows the largest defects (indicated by yellow arrows). Those lined-up voids are lack of fusions due to improper process parameters. The periodic appeared lined-up defects along the same plane perpendicular to the loading direction reduces effective nominal stress and makes multiple crack initiations, where the fatigue life is severely reduced. Meanwhile the closely interacted voids may change the surrounding stress field by amplifying the stress concentration factors that can worsen

its fatigue performance. The dimple size is again a few microns, which is consistent with the grain size. Striations are found in samples and shown in Figure 6-15 with (a) XY_0° (b) XY_45° and (c) XZ_45°. The distances of striation are 138, 304, 289 nm, respectively. The striations are signs of plasticity that are caused by local plastic deformation[211] and formed by crack open and close upon tension cyclic loading[211]. Striation is usually appeared in stable crack propagation stage. For those samples with larger striation distance they are found in the relatively longer crack regime.

(a)

(b)

(c)

(d)

(e)

(f)

(g)

(h)

Figure 6-14 SEM micrographs of fractured surfaces of tested EH36 fatigue samples built in XY₀°, XY₄₅°, XZ₉₀° and XZ₄₅° tested under a maximum stress of 400 MPa (max. stress) showing (a,c,e,g) crack origins and (b,d,f,h) dimples in the fast fracture region (Note: The sample printed in XZ₄₅° was tested under 450 MPa.)

(a)

(b)

(c)

Figure 6-15 SEM micrographs showing striations in EH36 steel samples built in (a) XY_0°, (b) XY_45° and (c) XZ_45° using the continuous scanning strategy

6.5.3.6 Fatigue Failure Mechanism

The fractured path is examined for a purpose of mechanism study using sample XY_45° tested at 400 MPa (max. stress), which is shown in Figure 6-16. As seen, near crack initiation region within a very short range of ~ 100 μm indicated as segment (1) (the size may vary as the cutting may not across the centre of the main crack path), the crack path is more straight with one or two reflections along certain angle to load direction. Beyond that the crack goes in medium sized straight lines for another few deflections within the 100 μm regime. And followed by a more zig-zag patterns in a minor size indicated by (2) for another about 300 μm . Along its way of further propagation the crack goes more flat. The crack is originating from surface pore which seems starting along the maximum shear plane of the adjacent grain so that the crack line aligns near 45° with external loading direction. The being observed change of orientation maybe incurred when passing through the boundary of the grain initiated crack before entering another differently orientated grain. From the theory of fatigue crack formation[236, 237], the crack is deemed formed after passing by one or two grains and then it may propagate intergranularly in a cleavage manner into small crack regime. Following is the zig-zag pattern where the crack is believed experiencing some resistance from microstructural level which could be the micro-scale features such as grain boundaries, inclusions, etc. [207]. From the SEM image in Figure 6-16, the length of the small zig-zag straight line is about 10 to 20 μm that is comparable with the scale of grain size. It propagates mostly in shear mode which could be along preferred crystallographic plane. When the line goes randomly or in curvature, it is said could be frequently reflected by refined grains or non-preferential crystallographic orientation according to Liu et al.[207] and He et al.[208]. The reflection either

by grain boundaries or non-preferential crystallographic plane for crack propagation results in a more tortuous way that can increase the crack propagation energy which is beneficial to fatigue performance. After crack initiation stage, the crack is less affected by the microstructures showing a macroscopically straight path as indicated by (3). Similar crack propagation pattern was reported [208], where most of the fatigue life was consumed for initiation of crack. It is suspected that the complex shape of pores inside 3D printed samples may reduce the crack initiation stage (or avoided in some extreme cases) due to the sharp corners along the irregular contours[135]. But it seems the initiation stage (stage I) is still there as can be seen from Figure 6-16 corresponding to segments (1) plus (2), while (3) is regarded as stage II of the long crack regime.

A schematic illustration for the crack initiation and propagation mechanism to the DLDed EH36 steel is shown in Figure 6-17, where crack may initiate from stress concentrated areas, mostly the surface porosity/inclusions/unmelted powder of 3D printed samples based on this study. The crack may initiate in shearing mode (the nucleation/incubation stage) if the stress concentration factor is not too high[154], through a small distance across few grains, around tens of microns ($\sim 30 \mu\text{m}$ here indicated by the yellow dashed circle in Figure 6-16). Then, the crack may propagate in intergranular mechanism, most probably due to restricted slip (another tens of microns distance) in the small plastic zone at low values of stress intensity factor K_{max} [215]. After that crack goes in a zig-zag way, together with cleavage stage cracks enters into small crack region, about $400 \mu\text{m}$ observed here. Beyond that is long crack region crack propagates macroscopically along the plane perpendicular to the loading direction. In this case the fatigue initiation life is not affected too much by the surface crack induced stress concentration. However, if the stress concentration is high, like the case of highly porous AMed samples the initiation stage could be much shortened

and it may go directly along the plane perpendicular to the loading direction after a short distance of small crack propagation. The entire crack initiation stage could be bypassed in some extreme cases with very strong stress concentration.

Figure 6-16 SEM micrograph of EH36 steel sample printed in XY_{45°} tested under a stress of 400 MPa (max. stress) showing the fractured path. The yellow arrow indicates loading direction.

Figure 6-17 Schematic illustration of crack initiation and propagation from the stress concentration area on sample surface through the grains

The enlarged view of crack initiation and final fracture regime is shown in Figure 6-18 (a) with the SEM micrographs for sample XY_{45°} tested at 400 MPa (max. stress). It is seen that the grains

near the fractured surface at final fractured region is stretched along the loading direction. The grains are obviously deformed showing large degree of plastic deformation happened at the fast fracture area. And voids are found within this area indicating a large loading applied leading to a different failure mechanism from crack initiation stage. The void is formed both within the ferrite grains and from the grain boundaries. They may coalesce and lead to crack propagation. Large amount of ductile dimples are disclosed on the fractured surface of the fast fractured area. Instead less grain deformation is expected at crack initiation and propagation regimes as illustrated in Figure 6-18(b) where the crack may go in transgranular way most of the time as illustrated in Figure 6-17 in a more brittle manner[209].

It shows that the microstructural features such as grain size, grain morphology, grain orientation or phases, etc. can interact with crack propagation. In the initiation stage where crack size is small (the crack driving force, i.e. the range of stress intensity factor ΔK is small) and propagation is slow, finer grains with higher density of grain boundary are able to prevent crack propagation better than coarser grains from microstructural interactions in a way of increasing the tortuosity of crack path. However when crack propagates into long regime the effect of grains, either size or others in inhibiting crack propagation is diminishing because the crack goes more rugged driven by a large ΔK value. Only plasticity will be an effective factor during this stage which is also explained by the low cycle fatigue behavior. Fine grains which are the normal practice for AMed metals increase hardness of the samples and the high cycle fatigue performance especially in crack initiation stage. However the benefit of the finer grains is overwhelmed by pores, brittleness and others[197]. It is an option to the end users in pore-containing 3D printed parts.

Figure 6-18 SEM micrographs of EH36 steel sample built in XY_45° tested under a max. stress of 400 MPa showing the grains and cracking path near (a) final fracture region and (b) crack initiation region. The yellow arrows indicate loading direction.

6.6 Summary

The microstructure and mechanical properties including tensile, charpy impact and fatigue properties of ASTM A131 EH36 steel samples printed by DLD in four different orientations were studied in detail. From the results clear relationships of process-microstructure-properties were identified.

The mechanical properties of the samples printed in all investigated deposition directions can meet ASTM standards after process optimization. The slightly lowered tensile properties and fatigue performance for the vertically built samples in the orientation of XZ_90 ° were mainly attributed to process-induced lack of fusions appeared in a periodic manner where increased nominal stress deteriorated the material properties of the high strength low alloy EH36 steel from large aspect.

Higher fatigue strengths of the samples printed in 45° along the horizontal planes were attributed to their high densities (containing lesser defects).

Austenization, phase change, grain growth, secondary phase transformation and relatively large heat affected zones were all attributed to the large heat input of the DLD process and non-effective heat dissipation due to a small contact area with the support. By using a continuous printing strategy a higher amount of carbide precipitates phase change and grain growth were revealed from the samples printed in the two vertical directions due to a longer retaining time at high temperatures rooted from less effective heat dissipation compared to the horizontal layout. An intermittent scanning strategy, however, effectively improved thermal cycles as well as the microstructure and mechanical properties of the samples by reducing heat accumulation through increased cooling intervals. The fatigue crack initiation and propagation mechanisms for the DLD processed EH36 steel samples built in the four orientations were elucidated.

Chapter 7 Conclusions and Future Work

7.1 Conclusions

7.1.1 Development of EH36 Steel through Selective Laser Melting

- The SLM process parameters were optimized through the study of laser power, scanning speed, hatch spacing and preheat temperature to target a high enough densification of the AMed EH36 steel samples (e.g. > 99%). The higher the laser power & preheat temperature and the lower the scanning speed, the higher the densification obtained was.
- Rectangular blocks of EH36 steel were printed through SLM to prepare the tensile and fatigue testing coupons, where light cracks were found near the edges of the blocks due to residual stresses accumulated during the printing process. Slower cooling rate could be one of the measures to reduce the residual stress in the printed EH36 steel samples.
- The resultant martensitic structure in the printed samples was due to the very high cooling rate of SLM process ($10^4 - 10^6$ K/s), which induced the high strength and hardness of the printed EH36 steel samples. The hierarchical macro, micro, nano-scale structures were explained based on the thermal history incurred during the rapid solidification process and the theory of solidification.
- Different shapes of martensite grains found from the TEM analysis rendered a need for deeper understanding of the phase transformation mechanism during the rapid solidification process.

- In-depth study has linked the tensile properties of the AMed EH36 steel to its microstructure and hence the printing processing parameters. The superior strength of the AMed EH36 samples was mainly due to the fine microstructure and martensitic phases (combined with possible strengthening of solid solution, dislocation, precipitation, etc.) resulted from high cooling rate incurred in the rapid solidification process.
- The process induced defects such as porosity could be the reason for the lowered yield strengths measured from the samples than the calculated values from the model for the martensitic structure.
- The high ductility coupled with the high strength of resultant tempered martensite and ferrite dual phase structure was obtained through in-process tuning of parameters mainly by reducing laser scanning speed. High enough energy input achieved by using slow laser scanning can induce efficient tempering to achieve the desired mechanical properties that are normally favored for a structural steel.
- Through the study of various scanning speeds, the high cycle fatigue behavior showed a porosity dominated failure mode for the samples with a low density and a failure mode of mixed porosity and slippery bands for the samples of a high density. Increasing density and reducing defects effectively improved the high cycle fatigue life of the SLM printed EH36 steel samples.
- New mathematical models correlating stress-life cycles, fatigue limit/life cycles, UTS, Hv, σ_y , ϵ , and effective defect size were established for the SLM processed EH36 steel samples. The fatigue behavior of the SLM printed samples was away from that of traditionally manufactured equivalents due to the intrinsic porosities in the printed samples.

7.1.2 Simulation of High Cycle Fatigue Life of SLM Processed EH36 Steel Samples with Surface Porosity

- The effect of surface porosity on the fatigue initiation life of the SLM processed EH36 steel samples were evaluated through numerical methods. The predicted initiation life and stress concentration factors were closely related to the size, shape, position, orientation and interactions of the pores in the samples.
- The sharper the crack tip radius and the deeper the crack propagated into the sample along the plane perpendicular to the loading direction, the higher the stress concentration factor K_t is thus leading to a lower fatigue life of the sample.
- The irregular shapes of the pores formed in the AMed steel samples could induce complex stress concentrations around their vicinities making the simulated pore profiles away from the simplified ellipsoidal/spheroidal pores, similarly for the pore-pore interactions.
- AMed material parameters/properties were used for the prediction.
- The effectiveness of adding a stress concentration factor to a smooth surface was subject to internal pore geometry based on the studies on a series of K_t values from 1 to 5.5.
- Both stress-life and strain-life methods could be used to effectively simulate the HCF behavior of the SLMed high strength low alloy steel samples.
- The predicted fatigue initiation life using FE-SAFE endurance software was validated by the experimental results.
- The effectiveness of LEFM model for calculating the fatigue total life cycle would depend on the initial defect size and whether the small crack propagation could be neglected.

7.1.3 Development of EH36 Steel through Direct Laser Deposition

- Small scale near-net-shape testing coupons were built using the DLD process. The process parameters in all the 4 printing orientations (XY_0°, XY_45°, XZ_45°, and XZ_90°) were optimized with high densifications of > 99% being achieved for the DLD processed EH36 steel samples.
- The samples printed in the two vertical orientations showed different microstructures due to different thermal cycles caused by the different heat dissipation areas and scanning strategy. Ferrite phases dominated the samples printed along the two horizontal directions with lower cooling rate, while martensite and bainite phases dominated the samples printing along the two vertical directions due to higher cooling rate. The microstructure could be controlled through tuning the printing process parameters and strategy, which can affect thermal cycles during the printing process.
- All the samples printed in the four orientations passed the ASTM standards for the tensile and charpy impact properties of EH36 steel. The fatigue performance of the XY_90° samples was slightly lower than that of the samples printed in the other three directions which were due to process induced pores.
- The positive results obtained from this work make direct application of additively manufactured EH36 steel in marine and offshore industry possible.

7.2 Future Work

There are some future works that are worthy to be explored. For the SLM process, the martensitic transformation mechanism of the SLM processed EH36 steel is not clear yet due to the high cooling rate of the process. New and meta-stable phases and structures were found by Kadiri et al. [70] for the LENSEd AISI 4140 low alloy steel. The martensitic transformation mechanism worthy for further study along with the forming mechanisms for the cellular-dendritic, lath and columnar structures, etc. for the EH36 steel through the SLM process due to its even higher rapid cooling rate. Those will help for the understanding of the physical phenomena of the SLM process benefiting for both academia and industry. Meanwhile the directional mechanical properties of the SLM printed EH36 steel samples need to be evaluated for potential application of AMed EH36 steel in marine and offshore industry. The mechanical properties are mainly tensile, charpy impact and fatigue properties. As the as-built SLMed EH36 steel samples are brittle in nature due to the martensitic structure from rapid solidification which limits their direct applications. Further investigation of after heat treatment is needed such as the stress relief, etc.

As a new process, the mechanical properties of the SLMed EH36 steel is different from conventional manufactured equivalent. The fatigue crack propagation behavior and the influencing material parameters of SLMed EH36 steel needs to be studied both experimentally and numerically. The experimental data can be used to validate the numerical models. Meanwhile the elastic stress strain near surface porosities of SLMed EH36 steel has been evaluated in this work through ABAQUS. Local plastic deformation is not considered yet, which could play significant role to the fatigue behavior of the SLMed EH36 steel. Those will help to set up the database for AMed EH36 steel and provide guideline to real applications.

Due to the largeness in size of the marine and offshore components, the development of large sized parts through DLD process shall be further conducted as DLD has priority in building large and complex shape of components. The potential problem of building large sized component would need to be addressed such as the structural integrity, the residual stress, microstructural and properties inhomogeneity, etc. for its applicability in real situation. Until now only one type of high strength low alloy steel, that is EH36 steel is studied for its entire microstructure, tensile, charpy and fatigue properties. It shed a light to the whole high strength low alloy steel family. Whether the AMed steel parts of the family can be adopted by industrial partner will depend on the quality and robustness of the fabricated parts from capable process or in another word the defect or pore-free parts, which is the ultimate goal of AM processing. Meanwhile the AM process is now still in single part one run and not ready for mass production yet. How to improve the productivity is another future target.

List of Publications

Journals

1. **Wang JJ**, Wu WJ, Jing W, Tan XP, Bi GJ, Tor SB, Leong KF, Chua CK, Liu EJ*, Improvement of densification and microstructure of ASTM A131 EH36 steel samples additively manufactured via selective laser melting with varying laser scanning speed and hatch spacing, *Materials Science & Engineering A* 746 (2019) 300-313.
2. **Wang JJ**, Zhang M, Tan XP, Bi GJ, Li H, Tor SB, Liu EJ*, Fatigue behavior of ASTM A131 EH36 steel samples additively manufactured with selective laser melting, *Materials Science & Engineering A*, 777, 10 March 2020, 139049.
3. **Wang JJ**, Zhang M, Wang B, Tan XP, Wu WJ, Liu Y, Bi GJ, Li H, Tor SB, Liu EJ*, Influence of surface porosity on fatigue life of additively manufactured ASTM A131 EH36 steel samples, *International Journal of Fatigue*, 142, January 2021, 105894
4. **Wang JJ**, Chew YX, Wu WJ, Jing W, Tan XP, Weng F, Liu Y, Sharine Tan YJ, Lee YH, Aziz Merchant, Tor SB, Chua CK, Bi GJ*, Liu EJ*, Robust ASTM A131 EH36 steel samples printed in different orientations via laser aided additive manufacturing, *Materials Characterization*, (submitted)
5. **Wang JJ**, et al., Effect of process parameters on porosity, microstructure of ASTM A131 EH36 steel via selective laser melting, (under preparation)
6. **Wang JJ**, et al., Evaluation of porosity effect on fatigue life of selected laser meted ASTM A131 EH36 steel using cyclic plasticity model, (under preparation)

Conferences

1. **Wang JJ**, Tor SB, Liu EJ, Impact and Fatigue Characteristics of Additively Manufactured Steel Materials: A Review, 3rd International Conference on Progress in Additive Manufacturing (Pro-AM 2018), Singapore, 14 – 17 May 2018.
2. Jing W, **Wang JJ**, Tan XP, Tor SB, Liu EJ, Mechanical Properties and Fracture Analysis of Additively Manufactured EH36 Steel Parts by Laser Engineered Net Shaping Process, 3rd International Conference on Progress in Additive Manufacturing (Pro-AM 2018), Singapore, 14 – 17 May 2018.
3. **Wang JJ**, Liu EJ, Tor SB, Chua CK, Metal 3D Printing Via Selective Laser Melting and Direct Metal Deposition: Materials, Properties and Applications, 2nd International Conference on Progress in Additive Manufacturing (Pro-AM 2016), Singapore, 16 – 19 May 2016, 270-275.

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