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**INVESTIGATION OF GRAIN GROWTH AND
MICROSTRUCTURAL EVOLUTION IN ELECTRON BEAM
MELTING ADDITIVE MANUFACTURING PROCESS**

SHUBHAM CHANDRA

**SCHOOL OF MECHANICAL AND AEROSPACE
ENGINEERING**

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SHUBHAM CHANDRA

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.

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Shubham Chandra

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.

28-Sep-2020

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Date

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Associate Professor Shu Beng Tor

Authorship Attribution Statement

This thesis contains material from one paper published in the following peer-reviewed journals/from two papers accepted at conferences in which I am listed as an author.

Chapter 4 contains material from a conference article published as Chandra, S., Phanikumar, G., Seet, G. L., Tor, S. B., & Chua, C. K. (2016). Multi-scale Modeling Of Additive Manufacturing Process. Proceedings of the 2nd International Conference on Progress in Additive Manufacturing (Pro-AM 2016), 543-550.

The contributions of the author and co-authors are as follows:

- S. Chandra developed the numerical model under the guidance of collaborator A/P G. Phanikumar (IIT Madras).
- S. Chandra analyzed the data and prepared the manuscript.
- A/P S. B. Tor conceived the project and provided the initial project direction.
- All the numerical modelling was carried out by S. Chandra in CAE Lab 1, NTU.
- A/P G. L. Seet and A/P S. B. Tor edited the manuscripts.
- Prof. C. K. Chua provided the funds necessary for the execution of the project.

Chapter 4 also contains material from a journal article published as Tan, X. P., Chandra, S., Kok, Y., Tor, S. B., Seet, G., Loh, N. H. and Liu, E., (2019). Revealing competitive columnar grain growth behavior and periodic microstructural banding in additively manufactured Ti-6Al-4V parts by selective electron beam melting. *Materialia*, 7, p.100365.

The contributions of the author and co-authors are as follows:

- A/P S. B. Tor and Dr X. P. Tan conceived the project and advised the research work.
- S. Chandra developed the numerical modelling framework and performed the experiments on periodic banding formation. S. Chandra and Dr X. P. Tan analysed the results.
- Dr X. P. Tan, S. Chandra and Dr Y. Kok prepared the manuscript. It was revised by A/P S. B. Tor.
- A/P G. Seet, A/P N. H. Loh and A/P E. Liu contributed to the technical discussions.

- S. Chandra performed the microstructural observations in the Materials Lab 1, NTU. The numerical model was developed in the CAE Lab 1, NTU.

Chapter 6 contains material from a conference article published as Chandra, S., Tan, X., Wang, C., Yip, Y. H., Seet, G., & Tor, S. B. (2018). Additive manufacturing of a single crystal nickel-based superalloy using selective electron beam melting. *Proceedings of the 3rd International Conference on Progress in Additive Manufacturing (Pro-AM 2018)*, 427-432.

The contributions of the author and co-authors are as follows:

- A/P S. B. Tor and Dr X. Tan conceived the project and advised the research work.
- S. Chandra performed the experiments, and the results were analysed by S. Chandra and Dr X. Tan.
- S. Chandra prepared the manuscript. It was revised by A/P S. B. Tor and Dr X. Tan.
- Y. H. Yip, C. Wang and A/P G. Seet contributed to the technical discussions
- S. Chandra performed the SEBM fabrication in the ARCAM EBM system located in the SC3DP, NTU. The microstructural observations were performed in Materials Lab 1, NTU.

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The contributions of the author and co-authors are as follows:

- A/P S. B. Tor and Dr X. P. Tan conceived the project and advised the research work.
- S. Chandra performed the experiments, and the results were analysed by S. Chandra and Dr X. P. Tan.
- S. Chandra prepared the manuscript. It was revised by A/P S. B. Tor, Dr X. P. Tan and Dr R. L. Narayan.
- C. Wang and A/P G. Seet contributed to the technical discussions

- S. Chandra performed the SEBM fabrication in the ARCAM EBM system located in the SC3DP, NTU. The microstructural observations were performed in Materials Lab 1, NTU. The numerical model development was carried out in CAE Lab 1, NTU.

25/09/2020

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Date

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Shubham Chandra

Abstract

Selective Electron Beam Melting (SEBM) is a representative powder bed fusion metal Additive Manufacturing (AM) technique that employs a high-energy electron beam for sintering and melting metal powder bed in a layer-wise fashion. High build temperatures (500 – 1100 °C) and vacuum fabrication environment are inherent to SEBM process, leading to residual stress- and contamination-free as-built parts. However, there is a lack of understanding in grain growth behaviour and control during microstructural evolution which eventually governs the mechanical properties of as-built parts. Additionally, the prevailing knowledge on hot cracking during AM of non-weldable Ni-based superalloys lacks a framework to relate the solidification parameters and microstructural features with crack susceptibility.

This study addresses the limitations in state-of-the-art research and utilizes multi-scale experimental and numerical simulation methods to investigate the grain growth behaviour and microstructural evolution with varying build geometries and metal powder sizes for three different engineering alloys, i.e. Ti-6Al-4V, Stainless Steel 316L (SS316L), and Ni-based Single Crystal (SX) superalloy. Furthermore, it also provides an analytical framework to correlate solidification parameters and microstructural features with the contingency of hot cracking in AM of Ni-based superalloys. In general, the key results of this thesis work can be clarified in the following sections:

First, a multi-scale numerical model is created based on SEBM processing of Ti-6Al-4V to investigate the grain growth behaviour and microstructural evolution with varying build geometries, e.g. V-, I-, and A-shaped parts. Model validation was carried out with the aid of a series of quantitative experimental observations on the as-built Ti-6Al-4V microstructure. It is found that the difference in Heat Affected Zone (HAZ) depths caused by varied beam scanning lengths resulted in the periodic microstructural banding in SEBM-built Ti-6Al-4V samples. Moreover, build geometry is determined to impact a dominant effect on solidification parameters, hence on the crystallographic texture of the as-built microstructure. The V-shaped part is identified to favour a strong $\langle 001 \rangle$ crystallographic texture with increasing build height in comparison to the A- and I-shaped counterparts.

Further insight into grain growth control is advanced by SEBM processing of austenitic stainless steel – SS316L. Effects of build geometry and powder size on crystallographic texture in the as-built parts are demonstrated by employing various and multi-scale characterization techniques. Thermal conductivities of the sintered powder bed and as-built samples were

determined using the Laser Flash Apparatus (LFA). Optical Microscopy (OM), Field Emission Scanning Electron Microscopy (FESEM), and Back-Scattered Electron (BSE) imaging were utilized to investigate melt pool topology and single-phase dendritic microstructure. X-ray Diffraction (XRD) and Electron Back-Scattered Diffraction (EBSD) were utilized for bulk phase analysis and crystallographic texture measurements, respectively. Transmission Electron Microscopy (TEM) was employed to determine the deformation mechanisms in the as-built samples subjected to uniaxial tensile testing. It was observed that a fine columnar-dendritic microstructure with primarily $\langle 001 \rangle$ crystallographic texture could be achieved by varying the build geometry. Furthermore, the powder size distribution is found to have an important effect on the as-built crystallographic texture. Changing the powder size from fine to coarse led to a uniform equiaxed-grained microstructure with significant improvement in tensile strength and ductility by ~17% and ~62%, respectively.

Despite its advantages, SEBM processing of Ni-based SX superalloys is still challenging due to their hot cracking susceptibility during rapid solidification. Additionally, elevated build temperature facilitates the precipitation of unfavourable Topologically Close-Packed (TCP) phases. Examination of the as-built microstructure using OM, FESEM, and EBSD revealed severe hot cracking which was confined only to divergent Grain Boundaries (GBs). Investigation of TCP phases using Energy-Dispersive Spectroscopy (EDS) and Atom Probe Tomography (APT) confirmed them as Ta-rich C14 Laves phase. Finally, a generalized hot cracking criterion is presented in this study that can account for the mutual inclination of grains at a GB. This generalized criterion is a major advancement over state-of-the-art and can be used to quantitatively predict cracking susceptibility of a GB under any solidification conditions associated with AM processes universally.

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List of Publications

Throughout his candidature, the author has written and contributed to several publications in international peer-reviewed journals and conferences.

Peer-reviewed Journal articles:

- **Chandra, S.**, Tan, X. P., Narayan, R. L., Wang, C., Tor, S. B., Seet, G. A Generalized Hot Cracking Criterion for Nickel-Based Superalloys Additively Manufactured by Electron Beam Melting. – *Accepted for publication* in Additive Manufacturing.
- Tan, X.P., **Chandra, S.**, Kok, Y., Tor, S.B., Seet, G., Loh, N.H. and Liu, E., 2019. Revealing competitive columnar grain growth behavior and periodic microstructural banding in additively manufactured Ti-6Al-4V parts by selective electron beam melting. *Materialia*, 7, p.100365.
- Xiang, D.D., Wang, P., Tan, X.P., **Chandra, S.**, Wang, C., Nai, M.L.S., Tor, S.B., Liu, W.Q. and Liu, E., 2019. Anisotropic microstructure and mechanical properties of additively manufactured Co–Cr–Mo alloy using selective electron beam melting for orthopedic implants. *Materials Science and Engineering: A*, 765, p.138270.
- Wang, C., Tan, X.P., Du, Z., **Chandra, S.**, Sun, Z., Lim, C.W.J., Tor, S.B., Lim, C.S. and Wong, C.H., 2019. Additive manufacturing of NiTi shape memory alloys using pre-mixed powders. *Journal of Materials Processing Technology*, 271, pp.152-161.

International Conference Publications:

- **Chandra, S.**, Tan, X., Wang, C., Yip, Y. H., Seet, G., & Tor, S. B. (2018). Additive manufacturing of a single crystal nickel-based superalloy using selective electron beam melting. Proceedings of the 3rd International Conference on Progress in Additive Manufacturing (Pro-AM 2018), 427-432.
- **Chandra, S.**, Phanikumar, G., Seet, G. L., Tor, S. B., & Chua, C. K. (2016). Multi-scale Modeling Of Additive Manufacturing Process. Proceedings of the 2nd International Conference on Progress in Additive Manufacturing (Pro-AM 2016), 543-550.

Chapter 1 Introduction

This chapter describes the motivations and objectives of the research. The problem statement is motivated by current state-of-the-art and prospective applications of metal additive manufacturing techniques. The organisation of the thesis is outlined at the end of this chapter.

1.1 Problem statement

Metal Additive Manufacturing (AM) technology has advanced to its current state from Rapid Prototyping (RP) over the last few decades. Though this technology is still nascent, it exhibits immense promise in achieving complete automation, repeatability, and in-process control during manufacturing of complex parts [1–4]. Moreover, high cooling rates and large thermal gradients, achievable easily in metal AM through process parameter alteration, are advantageous in obtaining parts with fine dendritic microstructure and minimum porosity. As a result, additively manufactured metal parts have shown astounding mechanical properties in comparison to their conventionally manufactured counterparts [5–14]. One such metal AM technique termed Selective Electron Beam Melting (SEBM) is a pioneer in achieving single-crystal (SX) microstructure in as-built Ni-based superalloy parts [15–18]. This technique utilizes a high energy electron beam to melt and fuse metal powder bed in a layer-wise fashion [19]. The high-temperature (500 – 1100 °C) fabrication process and a high-vacuum build environment, inherent to the SEBM process, assists in producing residual stress- and contamination-free as-built parts [20]. Profound penetration depths of the electron beam in the powder bed and negligible melt pool convection enables the alignment of dominant heat flow with the build direction. This enforces a nearly unidirectional thermal gradient that favours the formation of highly oriented columnar grains resulting in the evolution of textured or single crystal (SX) microstructure with build height. Though an SX microstructure is desirable for its high-temperature application to gas turbine industries [21,22], it limits the employability of SEBM to a specific set of materials – Ni-based SX superalloys. For conventional industrial applications, the directionally grown grained microstructure is unfavourable as it introduces substantial anisotropy and heterogeneity in mechanical properties of as-built parts [23]. Moreover, prolonged columnar grain boundaries are prone to solidification cracking during AM processing as compared to equiaxed grain ones [24,25].

This aspires a need for robust control in grain growth during microstructural evolution in SEBM-built alloys to achieve an equiaxed grained microstructure. Though it has been realized to an extent in various AM processes through process parameter variations [12,25–30], the introduction of nano-scale inclusions for grain nucleation [24,31], and post-processing [32], the repercussions of these methods can be daunting. For instance, the parametric optimization to initiate columnar-to-equiaxed transition during melt pool solidification is exclusive to the material composition and powder size distribution and thus, varies significantly for different materials. Further, the introduction of nano-scale inclusions changes

the final alloy composition and can lead to the formation of detrimental intermetallic phases. Post-processing method, though maintaining the initial alloy composition, cannot guarantee the uniformity in microstructure, and defies the purpose of micro-scale microstructural control using AM. Hence, it is imperative to conceive alternative grain growth control methods which can be universally applicable to a wide variety of AM techniques and materials while maintaining initial alloy compositions.

1.2 Research Motivation

SEBM has shown significant promise in the fabrication of industrially relevant alloys and superalloys. Its high build temperatures (500 – 1100 °C) and vacuum fabrication environment lead to residual stress- and contamination-free as-built parts. However, there is a need to address the lack of understanding in the grain growth behaviour and microstructural control during melt pool solidification and the phase transformation during subsequent cooling. Moreover, the cyclic reheating of layers during the SEBM process aids the formation of unwanted phases in some complex alloys, which may prove detrimental to the mechanical properties of as-built parts. Hence, there is a need to address the extent of the effect of these phases on the microstructure of as-built parts.

In the absence of grain boundary strengthening elements such as Boron in Ni-based SX superalloys, they have a higher affinity towards solidification cracking during rapid solidification typically associated with metal AM processes. However, the state-of-the-art in the fabrication of Ni-based SX superalloys using SEBM technique involves alleviation of cracking through process parameter optimization. This process is highly dependent on the feedstock material, and the optimum process parameters determined for bulk samples prove unreliable when fabricating industry-relevant parts with complex geometrical features. Thus arises the need for a rigid framework that could relate the solidification parameters as well as the microstructural features to crack susceptibility.

SEBM fabrication and the microstructural evolution in the as-built samples of three alloys – Ti-6Al-4V, Stainless Steel 316L (SS316L), and Ni-based SX superalloy AM1, was investigated in this research due to their industrial significance and severe lack of knowledge in the research till date.

Ti-6Al-4V was chosen due to its significance in the Medical and Aerospace industries. Moreover, there is a lack in consensus about the phase transformation kinetics resulting from the thermal cycling and high-temperature build environment in the SEBM process.

SS316L was chosen, given its indubitable importance in the Marine and Offshore industry. From a metallurgical viewpoint, the single-phase microstructure of SS316L aids in providing a clear picture of the microstructural evolution during solidification and the solidification parameters thereof. Moreover, combining the low-cost benefits of SS316L with an advanced manufacturing process such as SEBM, with enough control of the microstructure, might result in parts with superior performance.

Ni-based SX superalloys have been of prime importance in the Aerospace and Power industry over the past four decades as they form the vital component of high-temperature gas turbines. One such SX superalloy – AM1, was chosen to investigate the detrimental phenomena of solidification-cracking, and the tendency to form deleterious TCP phases in SEBM-built microstructure.

1.3 Research Objectives

The main objective of this work is to obtain an in-depth scientific understanding of grain growth behaviour during the SEBM fabrication process. Paramount focus is given to the effects of build geometry and powder size distribution on the melt pool solidification parameters, and resulting crystallographic texture in the microstructure of SEBM-built Ti-6Al-4V and SS316L alloys. Pertinent to this cause, multi-scale numerical simulations and experimental investigations are utilized extensively. Another predominant focus in this work is dedicated towards grain growth and hot cracking susceptibility in SEBM processing of a Ni-based SX superalloy – AM1.

1.4 Research Scope

The following points lay out the scope of this research in achieving robust control in grain growth by studying the influence of build geometry and powder size distribution on the crystallographic texture of as-built microstructure. A major defect of hot-cracking during SEBM fabrication of a non-weldable Ni-based SX superalloy is investigated to develop a rigid framework for conquering it. The research scope of this study is laid out as below:

1. Development of a multi-scale numerical model of SEBM fabrication of Ti-6Al-4V alloy. This is to understand the effects of build geometry on melt pool solidification parameters which eventually affects the competitive grain growth and crystallographic texture during microstructural evolution.
2. Extension of the study on the effect of build geometry on crystallographic texture in the as-built microstructure to obtain strong textured SS316L alloy.

Moreover, the effect of powder size distribution on the crystallographic texture will be investigated. Utilization of austenitic stainless steel for this task would assist in a direct interpretation of the observed texture with distinct grain orientations. Eventually, the establishment of robust grain growth control by achieving both – a strongly textured microstructure and an entirely equiaxed microstructure, in SEBM-built SS316L alloy without any process parameter variations, material composition variations or post-processing treatment.

3. Study of hot cracking behaviour in SEBM-built Ni-based superalloy – AM1. The crack initiation and propagation in as-built samples will be investigated to correlate their existence and contingency with the microstructural features. The phase analysis of detrimental secondary phase precipitates observed in as-built samples will point out the limitation of SEBM in fabricating industry-oriented SXs. A generalized hot cracking criterion, independent of the AM technique employed, will be proposed that can act as a basis for producing crack-free parts.

1.5 Research Roadmap

Figure 1-1 shows the research road map prepared in line with the objectives and scope of this research. The road map outlines the steps employed to achieve the main objective of this research, that is, investigation of grain growth during microstructural evolution in SEBM as-built parts. First, the literature of the state-of-the-art in grain growth control had to be reviewed. The literature review on the phases and microstructure desired for superior mechanical properties informed the author of the direction in which the grain growth control was to be initiated. Next, the author developed a multi-scale numerical model to predict the variation in solidification parameters with part geometry of SEBM-built Ti-6Al-4V alloy. This process helped the author to understand the effect of part geometry on the resulting crystallographic texture. With that information, part geometry was carefully designed to achieve a very strong textured microstructure in SEBM-built SS316L. This study was further advanced to achieve fine equiaxed microstructure by varying the powder size distribution. This research was concluded by an investigation of hot cracking in SEBM-built AM1 superalloy. The author realized the need to address the hot cracking contingency in metal AM processes before SEBM could advance to a commercial SX manufacturing process. A rigid framework was established that could relate the solidification parameters, microstructural features, and material properties to the contingency of cracking in AM of Ni-based superalloys.

Figure 1-1 Research roadmap

1.6 Organization of thesis

The main content of this dissertation is to establish a framework for achieving application-oriented microstructure in SEBM-built parts – without involving any post-processing techniques. The research work carried out along this direction has been divided into the following chapters:

Chapter 1 provides a brief overview of the SEBM technique and state-of-the-art in achieving a complete grain growth control during manufacturing. This chapter also lays out the motivation, objectives, and scope of this work. It is then concluded with a roadmap followed to achieve the desired objectives.

Chapter 2 provides a review of existing literature on metal AM techniques and their classifications. The grain growth during metal AM of various alloys and the state-of-art are highlighted. Moreover, the current state of multi-scale numerical simulations in predicting the microstructural evolution in AM is presented here. This chapter is concluded with the summary of the literature review and the research gaps in the state-of-art.

Chapter 3 describes the details of the instrument and equipment utilized for sample fabrication, preparation, and characterization. The rationale for using the SEBM system, around which this study is focused, is developed. Fundamental knowledge of numerical simulation methodology employed in the current study, and the principles of using various characterization techniques such as Field Emission Scanning Electron Microscopy (FESEM), Electron Back-Scattered Diffraction (EBSD), X-Ray Diffraction (XRD), Transmission Electron Microscopy (TEM), Micro-Computed Tomography (Mirco-CT), and Atom Probe Tomography (APT) are discussed here as well.

Chapter 4 studies the effect of build geometry on grain growth behaviour during the microstructural evolution of SEBM-built Ti-6Al-4V alloy. This chapter stands as a basis for the development and validation of multi-scale numerical modelling technique utilized in this study.

Chapter 5 lays the essential research framework wherein the microstructural control to obtain both columnar and fully equiaxed grains in SEBM-built SS316L alloy is discussed, and their effects on mechanical properties of as-built samples are presented.

Chapter 6 describes another key achievement of this research wherein the contingency of hot cracking during AM of Ni-based superalloys is related to the solidification parameters, microstructural features, and material properties through a modified criterion for cracking. This

chapter presents a novel and detailed nanometer-scale (nano-scale) characterization of SEBM-built microstructure and highlights the key alloying elements responsible for detrimental TCP phase precipitation during AM.

Chapter 7 summarizes the research work along with the key findings delineated. Prospective future work for the application and advancement of the knowledge is also provided in this chapter.

Chapter 2 Literature Review

This chapter presents a detailed literature review for the relevant topics of this research work. A comparison between different powder bed fusion and powder blown metal AM processes has been carried out to establish the validity of using the SEBM technique in this research. Phases, microstructures, and advantages of the three materials (Ti-6Al-4V, SS316L, and AM1 superalloy) undertaken in this research are also discussed in detail. A review of the state-of-art in metal AM fabrication of Ti-6Al-4V, SS316L, and Ni-based SX superalloys are discussed as well to justify the need for carrying out SEBM fabrication of these alloys. Finally, a research road map is provided to achieve the scope intended in this research.

2.1 Introduction

Achieving mechanical properties that can be benchmarked against industry standards is vital to deem a non-conventional manufacturing technique as a cogent advancement over its conventional counterparts. Metal AM techniques have evolved rapidly over the last three decades due to their ease of access and ever-growing research opportunities. In some specific application areas, these techniques show promise as being more economically and environmentally sustainable, automated, and user-friendly in comparison to the standard subtractive manufacturing techniques. Moreover, the AM process involves material processing at the micron scale. This provides a broad scope for optimizing a part specific to its usage via microstructural engineering and topology optimization. This would result in savings in terms of material consumed and cost of manufacturing. Though state-of-the-art is at a stage when as-built parts show mechanical properties which are comparable to their wrought counterparts [5–11,33–35], there are still a few areas in which the AM processes need significant improvement. For instance, since the metal AM process deals with the manufacturing at a micron scale, the dimensions of the melt pool generated are usually within a few 100 microns in powder bed fusion processes to a few millimetres in the powder blown metal AM processes [36]. As a result, the solidification parameters that control the grain growth are usually in orders of magnitude higher than that obtained in the conventional manufacturing processes. Thus, it is challenging to achieve total control of grain growth in every single melt pool, which results in unpredictable variations within the microstructure. Another issue arises from the epitaxial grain growth that results from remelting of the previously melted layers. For instance, due to a highly unidirectional thermal gradient in SEBM, and Laser Powder-Bed Fusion (L-PBF) for a specific set of process parameters, the grain growth is highly directional which leads to predominantly columnar microstructure resulting in anisotropic mechanical properties [37]. Moreover, the columnar microstructure is prone to hot cracking during solidification [38]. Researchers have countered this limitation by producing equiaxed microstructure through process parameter variation [12,25–30], nano-scale inclusion assisted nucleation [24,31] or by post-processing methods [32]. All these methods have few disadvantages of their own as all of these schemes are material dependent, and need a few expensive trial and errors to get it right.

As the microstructure of a part defines its strengths and suitability in its application, it is vital to understand the fundamentals associated with grain growth and microstructural evolution. Pertinent to this cause, the literature review detailed in this chapter discusses the basis of various metal AM techniques, their applications, advantages, and shortcomings in

manufacturing. Moreover, a review of the phases and microstructures of the three alloys chosen in this research – Ti-6Al-4V, SS316L, and AM1 superalloy, highlight the uniqueness of each of these materials, and the opportunities that they hold for the advancement of the metal AM technology. This review is followed up by the fundamentals of solidification while focusing mainly on the parameters associated with the rapid solidification. Necessary attention is directed towards state-of-the-art numerical modelling of metal AM processes.

2.2 Metal Additive Manufacturing (AM)

Additive Manufacturing (AM) technology traces its roots to Stereolithography (SLA) which was invented back in 1983 by Chuck Hull [39] and patented in 1986 [40]. Initially aimed at producing prototypes with polymers, this technology has since advanced to metals and ceramics and, as a result, found its applications in a myriad of fields such as aerospace [41–43], medical [44–48], electronics [49–52], and many more [1,53–55]. Since the energy utilised is highly focused within micron-scale, the rapid solidification of the melt pool region takes place mostly independent of the rest of the part. This gives tremendous opportunities for tailoring the microstructure to desired specifications [56]. Moreover, AM shows tremendous promise in being more economically viable and sustainable, in certain application areas, in comparison to the conventional subtractive manufacturing techniques [57,58].

2.3 Classification of metal AM processes

Among the vast range of classifications available for the continually advancing AM processes [59], metal AM techniques are categorized predominantly according to the characteristics of the feedstock used – powder-based or wire-based [60,61]. Research on power-velocity process mapping of various powder- and wire-based AM systems has shown that faster beam scanning speeds are achievable in the former [62,63]. Due to this, powder-based metal AM processes form the fundamental components of this work.

Powder-based metal AM techniques are further categorized depending upon whether the high energy beam interacts with a powder bed or whether the powder is fed through a nozzle into the melt pool formed by the high energy beam on a substrate material [33]. Figure 2-1 shows a general classification of these techniques into Powder-Bed Fusion (PBF) and Directed Energy Deposition (DED).

Figure 2-1 Classification of powder-based metal AM techniques

In PBF, the feedstock in powdered form is arranged in a bed, and melting of the powder bed takes place according to the cross-sectional geometry of the part. Once this process is completed, the build table moves downwards by the height of a layer, and a rake system spreads the powder evenly on top of the melted layer. While, in DED, the feedstock (powder) is fed to the highly focused beam by utilizing a carrier gas, argon. The following subsections will discuss each of these processes in detail.

2.3.1 Directed Energy Deposition (DED)

In the DED type AM processes, parts from 3D models are fabricated using a heat source, most commonly a laser, which directs its energy to a focused spot on a substrate to melt and fuse the powder fed [64]. At times referred to as Direct Metal Deposition (DMD), this metal AM process has been adapted and developed to a multitude of variations. Two most researched DED techniques were developed as Laser Engineered Net Shaping (LENSTM)[5,65] at Sandia National Laboratories and Directed Light Fabrication (DLF) [66,67] at Los Alamos National Laboratory.

Figure 2-2 is a 2D schematic of a powder-based DED process [68]. As shown here, this technique utilizes a powder delivery system that consists of coaxial nozzles through which metal powder is fed into the melt pool generated by the high energy beam on the substrate. The entire assembly of the nozzles and the build table is located inside an inert gas chamber, and the inert gas acts as a carrier of the powder as well as shields the melt pool from any oxygen present in the build chamber. Typical melt pool depths observed in DED processes are in the range of 0.1-0.5 mm [64].

Figure 2-2 A schematic of the powder-based DED process [68]

DED systems have shown great promise in AM of biomedical implants, part repair, and fabrication of compositionally graded parts [69][70]. Since the build does not involve a powder-bed, multi-axis build tables form a core of the DED systems to enable non-horizontal printing [71]. Moreover, since the powder is fed through a nozzle system which has a capability of feeding different materials through each nozzle, compositionally, and functionally gradient materials can be printed using this system with varying material compositions in one part [69,72,73].

2.3.2 Powder-Bed Fusion (PBF)

In PBF type AM processes, selective sintering and melting of powder bed are carried out using either a high power laser (L-PBF) or a high energy electron beam (SEBM) [56]. For

dexterity, laser and electron beam-based PBF processes in this dissertation will hereon be referred to as L-PBF and SEBM processes, respectively.

Figure 2-3 shows schematics of these two types of PBF metal AM processes. As seen in Figure 2-3 (a), in an L-PBF system, a laser generation and control system are present which focus, control, and direct a high power laser on to the powder bed surface. The laser energy is absorbed in the powder bed, and effective heat input depends mostly on the absorptivity of the powder bed or the base plate. After the melting of each layer, the build platform moves down by the same amount as the layer height, and the raking mechanism spreads a new layer of powder – to be melted. This complete process takes place inside an enclosure where the argon-rich atmosphere is created to avoid any contamination of the chemically reactive melt pool. The adequate powder size for L-PBF system is 25 – 63 μm .

Figure 2-3 (b) presents a generic schematic of a SEBM system. The electron gun of a SEBM system consists of a tungsten filament, which acts as a cathode, and a copper anode. Under the application of a high voltage of ~ 60 kV, free electrons are generated at the filament. These are accelerated through the anode, and the resulting electron beam is focused and directed on the powder bed by the electromagnetic lens assembly. As the electron beam can interact with the air molecules, the SEBM system requires very high vacuum conditions to operate. Moreover, since the kinetic energy of the electrons is converted to heat energy when they are absorbed in the powder bed, the absorptivity of powder bed does not define the efficiency of the beam energy absorption. Consequently, approximately 90% of electron beam energy is transferred to the powder bed in the form of heat [28,37].

Figure 2-3 Generic schematics of (a) L-PBF and (b) SEBM powder bed fusion techniques.

There are a few critical differences in the L-PBF and SEBM systems which categorize their application-oriented merits and demerits. Since the powder bed in SEBM needs to be densely sintered to avoid powder smoke, the SEBM process is usually carried out at very high build temperatures (500 – 1100 °C). This requirement of the process bodes well in producing residual stress-free as-built parts [74]. Moreover, since the electron beam can only be directed on the powder bed using electromagnetic lenses as opposed to a focusing mirror used in L-PBF processes, it can achieve excellent beam scanning speeds up to ~ 20 m/s resulting in much faster build rates.

Among the powder-based metal AM processes detailed above, PBF process is more advantageous than the DED in many aspects. Due to the presence of a static powder bed in PBF in comparison to a continuously flowing powder in DED, higher predictability in process parameters is possible. Whereas, in DED processes, the powder flow rate and the shielding gas flow rate also play a key role in determining the part quality along with beam scanning speed, hatch spacing, and layer height. Moreover, in DED processes, as the beam is focused on the melt pool which is also the coincident point of the powder flow, the powder particles which interact with the beam before the melt pool can cause shielding effect by scattering the beam to an extent. This results in lower efficiency of the beam in comparison to PBF processes.

Among the PBF processes, L-PBF and SEBM techniques have merits of their own; however, the presence of a vacuum environment and lack of forced convection heat flow makes SEBM an ideal candidate to lead the manufacturing of alloys which require stringent processing conditions such as Ti alloys, Ni-based Single Crystal (SX) Superalloys, etc.

2.4 Engineering alloys and their industrial applications

The three alloys studied in this research – Ti-6Al-4V, SS316L, and AM1 superalloy were chosen upon the significance of the industries they are predominantly applied to. Ti-6Al-4V given its high strength-to-weight ratio that remains undeterred even at high temperatures is most widely used in aerospace industries for manufacturing of airframes and low-temperature gas turbine components [75]. SS316L belongs to a class of austenitic stainless steels which have over hundred years of legacy of application to a wide range of industries some of which are food industries, architecture, manufacturing, marine and offshore, and so on [76]. AM1 superalloy belongs to the class of first-generation Ni-based SX superalloys. The application of these superalloys is limited to gas turbine industries and have evolved over time from Poly Crystal (PX) superalloys only according to their usage in the fabrication of gas turbine blades.

Following subsections will detail the phases and microstructures of these alloys, and the status of metal AM for the fabrication of their parts.

2.5 Ti-6Al-4V

2.5.1 Phases and microstructure

To better understand the temperature-dependent phase transformations and microstructural evolution associated with Ti-6Al-4V alloy, it is imperative to initiate the process from pure titanium, Ti. It is widely known that Ti possesses different crystal structures at different temperatures. At low temperatures, below $\sim 880^\circ\text{C}$, it exists in the form of α phase which has a Hexagonally Close-Packed (HCP) lattice structure. With an increase in temperature, it goes through an allotropic transformation at $\sim 880^\circ\text{C}$ to a Body-Centred Cubic (BCC) lattice structure. This phase is termed as β phase, and the transformation temperature is called β -transus temperature [77,78]. Among the two phases, the BCC β phase titanium has higher plastic deformability compared to the densely packed HCP α phase.

Ti-6Al-4V is an $\alpha+\beta$ dual-phase titanium alloy with a β -transus temperature of 995°C , a highest among all the $\alpha+\beta$ phase titanium alloys. Given its high specific strength (strength-to-weight ratio) and intrinsic research interest, this alloy comprises approximately 50 % of the usage of the commercially available titanium alloys [77]. Moreover, among the α/β phase titanium alloys, Ti-6Al-4V has shown the most vigorous behaviour of superplasticity with elongation $\sim 1000\%$ for specific temperature and strain rate [79]. Among the alloying elements of Ti-6Al-4V, aluminium acts as the α -phase stabilizer while vanadium is the β -phase stabilizer. Figure 2-4(a) shows the phase diagram of Ti-6Al-4V [77]. As seen here, the microstructure is complete β -phase above the β -transus temperature, which has a BCC lattice structure. Between the β -transus (995°C) and martensite transformation (800°C), the microstructure is comprised of the $\alpha+\beta$ phase while below that it is entirely α -phase having an HCP lattice structure. The phase distribution of Ti-6Al-4V microstructure can be classified to fall between two extremities – lamellar and equiaxed. The lamellar microstructure is a result of cooling from above β -transus temperature to the room temperature. Figure 2-4(b) shows the resulting microstructure of Ti-6Al-4V when subjected to slow cooling vs water quenching. As seen here, when slow cooled from a temperature above the β -transus temperature, the β phase transforms completely to lamellar α phase. Under high cooling rates as applied through water quenching, martensitic transformation of the β phase takes place to form a needle-like microstructure [77]. The equiaxed microstructure, on the other hand, forms through

recrystallization when the alloy is subjected to thermomechanical treatments in the $\alpha+\beta$ phase field. Due to the high cooling rates associated with metal AM processes, this research is focused on the as-built microstructure of Ti-6Al-4V with fine lamellar microstructure.

Figure 2-4 (a) Phase diagram of Ti-6Al-4V alloy (b) Effect of cooling rate (Slow (50 °C/h) vs water quenching) on the resultant microstructures [77]

2.5.2 Research status on AM of Ti-6Al-4V alloy

Table 2-1 provides an overview of the directional tensile properties obtained in recent works in as-built and heat-treated conditions of Ti-6Al-4V alloy fabricated from powder-based DED, L-PBF, and SEBM techniques [35,62,80–91]. The direction of loading specified as X and Z implies that the sample was taken from either the beam scanning plane (XY) or from the one aligned to the build direction (XZ or YZ plane) respectively.

Though a majority of these literature report Yield Strength (YS) and Ultimate Tensile Strength (UTS) obtained in as-built conditions much higher than the requirement set by the ASTM standard [92], there are significant improvements required in the ductility of as-built samples. However, as observed from the heat-treated samples in both DED [85] and L-PBF [87,88], there is a significant improvement in ductility of Ti-6Al-4V samples with a compromise in reduced YS and UTS. This is due to the decomposition of the martensitic phase resulting from rapid solidification into the $\alpha+\beta$ phase during heat treatments [85,93]. Here, the advantage of using SEBM for the fabrication of Ti-6Al-4V is evident as in the as-built conditions, the ductility obtained during the tensile testing of samples along X direction far exceeded that of ASTM elongation requirement. As reported by Tan et al. [35], the values of YS, UTS, and elongation decreases with the build direction (z value at X1 < z value at X4). However, they also reported that the α' martensite phase was not observed in SEBM-built samples due to the high-temperature fabrication process. This was also reported by Wysocki et al. [92] by TEM observations, as shown in Figure 2-5, which shows α' martensite observed in L-PBFed Ti-6Al-4V in comparison to SEBM fabricated one.

Another critical difference between the SEBM properties and L-PBF or DED is that the extent of anisotropy in mechanical properties is negligible for SEBM (0.2 – 0.5 % YS) in comparison to that obtained in L-PBFed (~ 3 % YS [89]) or DEDed parts (~ 4 % YS [85]).

Given the high-vacuum fabrication environment of SEBM, evaporation of light alloying elements, such as Al in Ti-Al alloys, takes place. Juechter et al. [94] determined that the variation in Al concentration was observed in a layered fashion along the build direction which was directly related to the line energy (beam power/scanning speed). Maximum deviations in the Al concentration from original were observed either for increasing line energies at constant scanning speed or increasing scanning speeds for constant line energy. Coincidentally, the line energy-scanning speed combinations, which resulted in the least deviation in Al concentration were the same as that required for producing fully dense parts. A

maximum loss of 30 % in Al was observed for the extreme conditions. Zhou et al. [95] reported that the Al evaporation during the SEBM fabrication of Ti47Al2Cr2Nb alloy increased drastically with decreasing pressure in the build environment (from 1 Pa to 4E-03 Pa). Recently, they reported the successful fabrication of a Functionally Graded Material (FGM) by employing techniques to increase the evaporation of Al from the melt pool [96]. By increasing the evaporation of Al, an α -phase stabilizer, from the melt pool, an $\alpha+\beta$ phase microstructure was obtained, which resulted in better mechanical properties.

Table 2-1 Tensile properties obtained in powder-based metal AMed Ti-6Al-4V

AM technique	Direction of loading	YS (MPa)	UTS (MPa)	Elongation ϵ (%)	Young's Modulus E (GPa)
DED – As-built [84] (Water-cooled base plate)	Z1	945 $\pm$ 13	1041 $\pm$ 12	18.7 $\pm$ 1.7	-
	Z2 < Z1	970 $\pm$ 17	1087 $\pm$ 8	17.6 $\pm$ 0.7	-
	X	960 $\pm$ 26	1063 $\pm$ 20	13.3 $\pm$ 1.8	-
DED – As-built [85]	X	1080 $\pm$ 4	1139 $\pm$ 12	13.1 $\pm$ 0.7	118 $\pm$ 2
	Z	1038 $\pm$ 28	1080 $\pm$ 13	8.7 $\pm$ 1.8	109 $\pm$ 5
DED – As-built [86]	X	922.6	992.8	8	-
	Z	868	956.8	13.8	-
DED – As-built [62]	X	960 $\pm$ 26	1063 $\pm$ 20	10.9 $\pm$ 1.4	-
DED – As-built [80]	X	1105 $\pm$ 19	1163 $\pm$ 22	4 $\pm$ 1	-
DED – Heat Treated [85]	X	762 $\pm$ 20	813 $\pm$ 16	13.8 $\pm$ 2.3	100 $\pm$ 4
	Z	808 $\pm$ 38	858 $\pm$ 27	14.3 $\pm$ 3.5	108 $\pm$ 10
L-PBF – As-built [87]	X	1075 $\pm$ 25	1199 $\pm$ 49	7.6 $\pm$ 0.5	113 $\pm$ 5
	Z	967 $\pm$ 10	1117 $\pm$ 3	8.9 $\pm$ 0.4	119 $\pm$ 7
L-PBF – Stress Relieved [87]	X	974 $\pm$ 7	1065 $\pm$ 21	7.0 $\pm$ 0.5	112 $\pm$ 6
	Z	937 $\pm$ 9	1052 $\pm$ 11	9.6 $\pm$ 0.9	117 $\pm$ 6
L-PBF – As-built [88]	Z	1065	1241	6	120.1

HT800 [88]		996	1223	7	117.5
HT900 [88]		869	945	18	116.2
HT920 [88]		850	933	15	115.8
HT950 [88]		837	919	12	116.7
HT1080 [88]		840	979	4	117.1
HIP (900 °C, 120 MPa) [88]		839	941	19	115.3
<hr/>					
	X	857.60	935.57	14.1	111.55
L-PBF – As-built [89]	45 ° from X	882.76	963.50	17.5	113.26
	Z	888.42	952.97	16.8	115.87
<hr/>					
L-PBF – As-built [92]	X	1273 ± 53	1421 ± 120	3.2 ± 0.5	-
	Z	1150 ± 67	1246 ± 134	1.4 ± 0.5	-
<hr/>					
L-PBF – As-built [81]	X	1195 ± 19	1269 ± 9	5 ± 0.5	-
	Z	1143 ± 30	1219 ± 20	4.89 ± 0.6	-
<hr/>					
L-PBF – Annealed [82]	X	929.5	1032	15.6	-
	Z	953	1035	17.7	-
<hr/>					
SEBM – As-built [35]	X1	851.8 ± 5.8	964.5 ± 0.3	16.3 ± 0.8	-
	X2	836.6 ± 8.7	953.7 ± 4.3	15.2 ± 1.2	-
	X3	827.9 ± 0.3	944.5 ± 5.8	14.0 ± 0.5	-
	X4	823.4 ± 0.1	940.5 ± 6.5	13.2 ± 0.7	-
<hr/>					
SEBM – As-built [83]	X	783 ± 15	833 ± 22	2.7 ± 0.4	-
	Z	812 ± 12	851 ± 19	3.6 ± 0.9	-
<hr/>					
SEBM – As-built [92]	X	846 ± 7	976 ± 11	15.0 ± 2.0	-
	Z	845 ± 9	972 ± 14	14.2 ± 1.5	-
<hr/>					
SEBM – As-built [81]	X	899 ± 4.7	978 ± 3.2	9.5 ± 1.2	-

	Z	869 ± 7.2	928 ± 9.8	9.9 ± 1.7	-
SEBM – As-built [90]	X	1006	1066	15	-
	Z	1001	1073	11	-
SEBM – As-built [91]	X	982.9 ± 5.7	1029.7 ± 7	12.2 ± 0.8	-
	Z	984.1 ± 8.5	1032.9 ± 12.9	9.0 ± 2.9	-
ASTM F136 [92]	-	> 795	> 860	> 10	-

Figure 2-5 Comparison of TEM images of Ti-6Al-4V in as-built condition obtained from (a) SEBM and (b) L-PBF [92]

Apart from the heat treatment deemed fit to reduce anisotropy in mechanical properties of Ti-6Al-4V, quite recently researchers have started to utilize the addition of trace elements to the Ti-6Al-4V feedstock to obtain superior properties. For instance, Xue et al. [97] reported that during Ti-6Al-4V fabrication by DED, the addition of amorphous Boron powder with the Ti-6Al-4V feedstock using ball milling results in an overall decrease in the anisotropy in mechanical properties. While the anisotropy in YS and UTS showed a general trend of reduction with increasing Boron wt.% from 0 – 0.25, the anisotropy in elongation % showed an increasing trend but reduced to its lowest value at 0.25 wt.% B. Though the anisotropy in properties is minimum at 0.25 wt.%, the elongation % is recorded at the lowest value of 7.5 %. This is due to the formation of brittle phase TiB intermetallics which were shown to increase the overall strength of the Ti-6Al-4V-xB alloy but compromise the ductility of the as-built alloy.

Similar findings were reported during L-PBF of Ti-6Al-4V feedstock mixed with nano yttria-stabilized zirconia (nYSZ) [98] where an addition of 2.5 wt.% nYSZ lead to significantly higher compressive strengths but with compromised ductility.

Addition of these trace elements modifies the resultant microstructure of as-built alloy resulting in the formation of brittle load-bearing intermetallics as well as refining the grain sizes of the microstructure by acting as nucleation sites [99]. However, the refinement of the grains can also be achieved by varying the solidification parameters. As shown in [100], competitive grain growth is dominant in controlling the microstructural evolution in as-built Ti-6Al-4V samples, and that the resulting crystallographic texture is dependent on part geometry. Hence, this research will focus on utilizing numerical simulations to control the as-built texture and provide non-intrusive and inexpensive ways of achieving desired microstructure.

2.6 SS316L

2.6.1 Phases and microstructure

Stainless steel type 316L is a low carbon chromium-nickel-molybdenum austenitic steel [101]. These alloys were first discovered first by Léon Guillet in 1906, and this discovery was complemented with that of Philip Monnartz in 1908 [76]. Monnartz determined the necessary requirement of at least 12 % Cr for the remarkable corrosion-resistant properties of austenitic stainless steels. Since the discovery of these alloys, a lot of modifications in the elemental compositions have been carried out depending on specific needs and applications. Over the years of development and compositional modifications, stainless steels – especially austenitic ones have been applied to an endless number of fields such as food, architecture, aircraft, and automotive industries [76].

Among the alloying elements of SS316L, high content of Cr (16 – 18 wt. %) is responsible for providing corrosion resistance through the formation of protective oxide layers when exposed to corrosive environments while the high content of Ni (10 – 14 wt. %) increases the stability of the Face-Centred Cubic (FCC) austenite phase [102]. Figure 2-6 shows a part of the iron-carbon binary alloy phase diagram that shows different Fe-C phases which exist at various temperatures for 0 – 6.70 wt.% C [103]. This region is essential as the carbon content of stainless steels lies well below 6.70 wt.%. As seen here, pure Fe exists in the form of BCC α -ferrite at room temperature until 912 °C. At this temperature, it undergoes allotropic transformation to an FCC γ -austenite phase which remains stable until 1394 °C. Beyond 1400

°C, Fe exists as a BCC δ -ferrite which melts at a temperature of 1538 °C. With the addition of C to Fe, a compound in the form of orthorhombic Fe_3C cementite phase coexists either with α -ferrite until 727 °C at a solubility of min. 0.022 wt. % C or with γ -austenite from there on until 1147 °C at a solubility of min. 2.14 wt. % C. This phase improves the mechanical strength of stainless steels but results in lower ductility due to its inherent brittle nature [103,104].

Figure 2-6 Iron-Carbon binary alloy phase diagram [103]

SS316L has a carbon composition of ~ 0.03 wt. % [101]. Thus, without the addition of other alloying elements, it is impossible for this alloy to exist in a pure austenite phase. Pertinent to this cause, Ni is added to the alloy as it is a γ -austenite phase stabilizer. To determine the effect of constituting elements on the stable phases of stainless steels, one must refer to the Schaeffler diagram [105]. Figure 2-7 shows a Schaeffler-Delong constitution diagram constructed for stainless steels [104]. This diagram is an important tool for predicting the phase constitution of stainless steel for different element additions. The additional elements are divided into two categories: ferrite forming (Cr, Mo, Si, Nb, W) and austenite forming (Ni, C, Cu, Mn, N). Each of these categories has been deemed chromium-equivalent (Cr_{eq}) and nickel-equivalent (Ni_{eq}), respectively.

Figure 2-7 Schaeffler-Delong constitution diagram for stainless steels [104]

Presence of Cr, Mo, and Mn as integral constituents of SS316L exposes it to a risk of formation of intermetallic phases when exposed to high temperatures. These precipitates can exist in the form of Topologically Close-Packed (TCP) sigma (σ), chi (χ) or Laves (η) phases [102]. σ phase precipitates mainly at the grain boundaries between a temperature range of 550 – 900 °C in SS316L with a composition of 55% Fe-29% Cr-11% Mo-5% N [106]. χ phase in SS316L has been identified similar in composition with that of σ phases with a higher solubility of carbon. Molybdenum contents between 2 – 3 wt.% in SS316L favours the formation of FeMn₂ type laves phases. The driving factor for the formation of these precipitates is the exposure of the alloy to long annealing times. Due to the complex thermal histories during metal AM fabrication processes, it poses a strong challenge to fabricate SS316L parts without the formation of these detrimental phases.

2.6.2 Research status on AM of SS316L alloy

Table 2-2 summarizes the tensile properties obtained in the recent research on the as-built SS316L parts obtained from powder-based metal AM processes. As evident here, the majority of focus on metal AM of SS316L has been directed toward the L-PBF process in comparison to DED or SEBM. However, the anisotropy in mechanical properties has been touched rarely. Moreover, the study of anisotropy in properties is found contradictory among different powder-based processes. For instance, the mechanical properties obtained in DED fabricated SS316L parts show higher ductility when tested along the build direction [107,108], while the L-PBF-built samples show a lower ductility along the same direction [109]. Similarly, for SEBM-built parts, better mechanical properties in terms of YS, UTS, and elongation was observed when tested along the build direction [13]. Interestingly enough, only SEBM-built parts show lower anisotropy in terms of YS (~ 15 %), but the ductility along the x-direction was compromised due to the presence of brittle σ -phase precipitates at the columnar grain boundaries which also explains a lower UTS value. Effect of process parameters on as-built microstructure and mechanical properties was demonstrated by Sun et al. [12] where higher laser powers applied during L-PBF of SS316L lead to a primarily $\langle 110 \rangle$ crystallographic texture in as-built samples resulting in impressive mechanical properties in comparison to low power samples with primarily $\langle 001 \rangle$ crystallographic texture. Ziętała et al. [107] reported the presence of δ -ferrite at the sub-grain boundaries surrounding the austenite matrix, which would explain the improved mechanical properties in comparison to the literature [108,110].

It is clear that the thermal conditions to which the melt pool was subjected to determine the as-built microstructure and the resulting mechanical properties of metal AMed SS316L. However, as seen in the state-of-art, there is insufficient work done in SEBM fabrication of SS316L, which has shown promise in the fabrication of residual stress-free as-built parts with inherent annealing effect. Hence, alternative methods should be sought to achieve a more uniform microstructure with reduced directional anisotropy in as-built condition from SEBM processes.

Table 2-2 Tensile properties obtained in powder-based metal AMed SS316L

AM technique	Direction of loading	YS (MPa)	UTS (MPa)	Elongation ϵ (%)
DED – As-built [107]	X	576	776	33
	Z	479	703	46
DED – As-built [108]	X	558	639	21
	Z	352	536	46
DED – As-built [110]	Z	261	530	43.4
L-PBF – As-built [109]	X	554.0 $\pm$ 4.6	684.7 $\pm$ 4.7	36.3 $\pm$ 2.1
	Z	-	580.7 $\pm$ 14.5	25.7 $\pm$ 12.2
L-PBF – As-built [111]	X	-	~ 590	~ 21
L-PBF – As-built (22 °C) [112]	Z	487 $\pm$ 3	594 $\pm$ 4	49 $\pm$ 4
L-PBF – As-built (250 °C) [112]	Z	376 $\pm$ 3	461 $\pm$ 3	31 $\pm$ 4
L-PBF – As-built [113]	Z	343.2 $\pm$ 11	428.8 $\pm$ 11.5	58.4 $\pm$ 3.9
L-PBF – As-built [12]	-	~ 550	~ 700	~ 55
SEBM – As-built (22 °C) [114]	Z	253 $\pm$ 3	509 $\pm$ 5	59 $\pm$ 3
SEBM – As-built (250 °C) [114]	Z	152 $\pm$ 3	386 $\pm$ 3	46 $\pm$ 3
	X	342.9 $\pm$ 22.8	436.5 $\pm$ 23.2	9.6 $\pm$ 2.3

SEBM – As-built [13]	Z	395.8±9.0	651.7±8.5	30.6±3.0
Wrought [13]	-	170	480	40
ASTM A240/A240M-16a [115]	-	≥ 170	≥ 485	≥ 40

2.7 Ni-based SX superalloys

2.7.1 Phases and microstructure

Ni-based SX superalloys were evolved from their polycrystalline ancestors in the early 1980s by suppression of grain boundary strengthening elements like carbon, boron, zirconium, and hafnium. In addition to this, large amounts of refractory elements like tungsten, tantalum, and molybdenum became an essential part of the superalloys; improving the high-temperature creep properties, and making them vital to the aerospace and power industry as high-pressure gas turbine components [21]. In their entirety, Ni-based SX superalloys have been classified into six generations: first three of which are classified depending upon rhenium weight percentages of 0, 3, and 6 % present in the first, second, and third generations respectively [116], and, later three on ruthenium weight percentage additions [117,118].

Ni-based SX superalloys are some of the most complex structural engineering materials used in high-temperature gas turbine applications. The alloying elements of a Ni-based SX superalloy include aluminium, chromium, cobalt, molybdenum, rhenium, ruthenium, tantalum, titanium, and tungsten [22]. The two phases (γ and γ') that constitute the microstructure of a typical heat-treated Ni-based SX superalloy are shown in Figure 2-8 [119].

Figure 2-8 Microstructure of fully heat-treated Ni-based SX superalloy - D99 [119]

2.7.1.1 γ phase

γ phase is the matrix in which all the other phases reside. It is primarily of FCC lattice structure, and is composed of alloying elements with atomic radii comparable to Ni. Among the alloying elements: Cr, Mo, Re, Ru, and W are γ phase strengtheners and partition to the austenitic γ matrix.

2.7.1.2 γ' phase

γ' phase, which is based on the Ni_3Al compound, is a precipitate phase which is uniformly distributed within the γ matrix and imparts strength to the superalloy. The crystal structure of this phase is orderly cubic, L_{12} , with Ni atoms situated at the face centres and Al atoms, at the corners of the lattice cube.

2.7.1.3 Topologically Close-Packed (TCP) Phase

Ni-based superalloys containing a high amount of refractory elements such as tungsten, tantalum, and molybdenum exhibit a strong tendency to form Topologically Close-Packed (TCP) phases, such as μ , σ , δ , P, R, and Laves phases when exposed to elevated temperatures [120–125]. These phases are detrimental to the high-temperature strength of SX superalloys as during their formation, and they drain the surrounding region from solid solution strengtheners, thus weakening the γ/γ' dual-phase microstructure. In particular, the Laves based TCP phase deplete the matrix from the γ' phase forming elements such as Ti and Ta. This results in a

decrease in overall creep resistance of the microstructure. Moreover, the TCP phases are highly brittle in nature, and hence have a higher affinity towards crack nucleation.

The development of Ni-based SX superalloys over the course of history implies that the later generations of superalloys, though promise quite significant increase in creep strength over first-generation superalloys, require extreme care in terms of balancing the heavy elements in the γ phase to avoid the formation of defects like Topologically Closed Packed (TCP) phases [119,126] and Secondary Reaction Zones (SRZs) [127] which inadvertently results in inferior creep strength. Moreover, higher composition percentage of heavier metals in second and later generations of SX superalloys results in a substantial increase in alloy density dictating the cost of manufacturing turbine components and fuel consumption by the turbine during operation. First-generation SX superalloys, on the other hand, have an advantage in terms of better microstructural stability due to absence of Re. Latest research has shown a trend in developing lighter, Re-free SX superalloys which show creep strength comparable to second, and later generations of heavier and relatively expensive SX superalloys [128,129]. Along with these advancements, significant improvements are due in the conventional manufacturing process of Ni-based SX superalloys [130].

2.7.2 Conventional processing of SX superalloys: directional solidification

At present, directional solidification technique is the most successful and widely accepted for manufacturing of SX superalloy components, especially for high-pressure turbine blades [22,131,132] [5][12]. Requirements in terms of cost reduction and fuel efficiency have driven the gas turbine industry towards utilization of hollow turbine blades with internal channels to allow passage of cold air thus restricting highest temperatures within the blades hence increasing the fuel efficiency by an overall weight reduction and increase in operating temperatures of the gas turbine [21]. Directional solidification technique also helps in eliminating the columnar grain boundaries by crystal selection, thus easing the manufacture of an SX part. Directional solidification process is carried out by establishing a nearly uniform positive thermal gradient and constant solidification rate. For this, a ceramic mould filled with molten metal is moved away from a heating zone to a cooled area at a constant speed. The following section provides a brief description of the methods developed to carry out the production of directionally-solidified SX cast components for aircraft engine and gas turbines:

2.7.2.1 Bridgman method

Bridgman method, at present, is the industrial method employed for the production of castings for SX turbine blades for high-pressure gas turbine applications. This method consists of directional solidification caused by radiation heating and cooling of the ceramic mould and casting [132]. The modern adaption of this method involves uniform heating of a ceramic mould (resting on a copper chill plate) inside the heating furnace, subsequent pouring of induction melted nickel superalloy into the mould and withdrawal of the mould containing solidifying metal at a constant rate from the heating chamber. For directional solidification of the SX superalloy castings, the nominal withdrawal speed is in the range of 2-4 mm/min [133]. Figure 2-9(a) shows a schematic of the Bridgman method.

Figure 2-9 Schematic of (a) Bridgman and (b) LMC method used for directional solidification of SX superalloy castings [132]

2.7.2.2 Liquid metal cooling method

In the Liquid Metal Cooling (LMC) method, liquid metal is employed as a coolant for the hot ceramic mould and casting during withdrawal from the heating chamber. Usually,

aluminium or tin in molten form is used as a coolant in this method. Figure 2-9(b) shows a generic schematic of the LMC method, which is quite like the Bridgman method in practice.

2.7.2.3 Fabrication of SX castings - grain selection and seeded growth

Two techniques - grain selection and seeded growth, are employed during the industrial manufacturing processes for the production of SX superalloy castings [22,132]. These assist in the selection and growth of grains of desired crystallographic orientations resulting in final SX microstructure.

A grain selector is, most commonly, a spiral structure (Figure 2-10(a), (b), and (c)) which is designed in the ceramic mould prepared for the SX casting process. It assists the growth of a single grain, which is randomly selected, during the solidification of the liquid metal in the selector. The grain selector follows the starter block of the casting and is utilized for elimination of highly misoriented grains before the solidification interface reaches the selector. A starter block utilized in an industrial manufacturing process is of height 20 - 30 mm, and constricts the grain deviation angles to a maximum of 20° [132]. The geometrical parameters, utilized in designing a spiral grain selector, affect the final height at which only one grain is selected for further propagation. A smaller value of take-off angle - θ , shown in Figure 2-10(a) increases the efficiency of the selector; while, the wire diameter - d_s and spiral rotation diameter - d_r have little to no effect on the grain selection process.

In order to obtain an SX casting of a particular crystallographic orientation, an SX seed of the desired orientation is used in the ceramic mould placed directly in contact of the copper chill plate [22,132] (schematically shown in Figure 2-10(d)). When molten metal is poured in the mould, grain growth from the top surface of the crystal seed takes place during melt solidification, and the crystallographic orientation of the seed dominates during the directional solidification of the casting.

Figure 2-10 (a) Geometrical features of a grain selector. Schematic of industrial application of a grain selector - (b), (c), and of a crystal seed - (d) for directional solidification of SX superalloy castings [132]

2.7.2.4 Limitations of the conventional manufacturing processes

Though Ni-based SX superalloys have witnessed nearly four decades of advancements in material composition, spread across their six generations, negligible improvements have been imposed on the manufacturing processes - which yield a component ever so vital to the gas turbines used in aerospace and power industries. The entire process of directional solidification with crystal seeding or grain selection is relatively simple in theory, though its application to obtain an SX part, suitable for industrial application is another story. Defects arising in directional solidification such as stray grain formation, grain misorientation, and high-angle grain boundaries (formed due to recrystallization during heat treatment [119,134]), along with the tight tolerances in terms of weight, dimensions, and application, results in low yield rate and adds on to the overall cost of manufacturing a gas turbine. Statistical analysis of the conventional manufacturing process shows that the percentage yield obtained, due to various defects associated with the traditional process of fabrication, remains below 70 % even for industry significant third-generation superalloys [22]. Hence, there is a need to modernize the current manufacturing technique for Ni-based SX superalloy parts due to the vast array of defects. The answer to this can be sought out in metal AM technology.

2.7.3 Hot cracking

Cracking during the manufacturing of Ni-based superalloys is a major defect resulting from the residual stresses which accumulate during solidification. During solidification, severe intergranular cracks are formed during manufacturing [38]. Since cracks form during the building process, the process of cracking is referred to as ‘hot cracking’, which is also sometimes referred to as solidification cracking or hot tearing in literature [135–138].

Broadly speaking, hot cracking [139] can be classified into three types according to the temperature of operation, i.e. solidification cracking, liquation cracking, and ductility dip cracking or strain age cracking. Solidification cracking operates in the solidification interval when the liquid to solid transformation takes place in the melt pool through the dendritic mode of solidification. This type of cracking is accompanied by a dendritic fracture surface with nearly no trace of melting [140]. Liquation cracking occurs in the Heat-Affected Zone (HAZ) [139–141] that affects the deposited layers exposed to reheating cycles during metal AM processes [142,143]. For additively manufactured Ni-based superalloy IN718, it was reported that the liquation cracks were accompanied with the precipitation of Laves phases which distinguishes them from precipitate-free solidification cracks [142]. Cracking that occurs at temperatures lower than liquation cracking is termed as ductility dip cracking or strain age cracking [139]. The primary cause of this cracking is the reheating of material to temperatures that cause precipitation of fine particles at the GBs [144], which in turn reduces the ductility. The fracture morphology accompanying ductility dip cracking exhibits a ductile intergranular fracture surface [139]. To investigate the cracking during metal AM processes, studies have referred to criteria developed by Kou, which has initially been applied to understand intergranular cracking during welding processes [145]. This criterion suggests that cracking occurs due to the combined action of thermal strains and solidification shrinkage during rapid solidification. Alternately, a higher growth rate of neighbouring grains towards each other and flow of liquid metal to the mushy zone combine to resist crack initiation and growth. A significant limitation of the prevailing criterion for intergranular cracking during solidification [145] is that it only takes the unidirectional growth condition of columnar grains into consideration but overlooks the process of competitive grain growth. Since competitive grain growth governs microstructure development in metal AM processes [100], hot cracking cannot be modelled by this criterion, in its present form.

2.7.4 Research status on AM of Ni-based SX superalloy

Application of metal AM techniques for fabrication of Ni-based superalloy SXs began with Scanning Laser Epitaxy (SLE) [146] – a technique derived from L-PBF wherein a high power laser was used to melt and deposit a single layer of superalloy powder (~ 2 mm high) on top of an SX substrate of the same composition. Mainly directed towards repair of turbine components, this technique was utilized over the years for investigation the microstructural evolution for Ni-based superalloys such as CMSX-4 [146–148], IN100 [149], Rene N5 [150], and MAR-M247 [151]. Figure 2-11 shows the as-built microstructure of CMSX-4 SX superalloy obtained from SLE [148]. As seen in Figure 2-11(c), the top surface of the deposited layer shows a strong presence of stray grains and Columnar-to-Equiaxed Transition (CET).

Figure 2-11 As-built microstructure of CMSX-4 obtained from SLE. (a) Long-section and (b) transverse section micrographs showing the transition of microstructure from SX base plate (coarse microstructure) to deposited layer (fine microstructure). (c) EBSD orientation map of the long-section showing strong $\langle 001 \rangle$ crystallographic texture until ~ 1.5 mm of the build (Adapted from [148])

It was observed that the SX base material acts as a crystal seed, and governs the crystallographic texture of the microstructure at the start of solidification. This crystallographic texture was not sustainable for the entire deposited layer as nearly 1/4th of the top region suffered defects such as CET and misoriented grains formation. Though promising at first, this technique is mainly limited by its efficient use for repair, and not for the fabrication of bulk SX parts.

A significant breakthrough in the use of metal AM for the fabrication of SX superalloys can be credited to the achievement of SX crystallographic texture in SEBM-built CMSX-4

[15]. Here the presence of highly unidirectional thermal gradient played a vital role in achieving $\langle 001 \rangle$ crystallographic texture from SEBM technique without the use of an SX base plate. Figure 2-12 shows the microstructure observed in the as-built part [15]. Figure 2-12(a) is the etched micrograph of the as-built sample with build height ~ 55 mm. As seen here, the bottom region of ~ 15 mm contains contrasting grains which indicate their mutual misorientation. This region acts as a grain selection region, wherein the misoriented grains are eliminated by competition with well-oriented ones leading to a strongly textured microstructure in the rest of the part. Severe intergranular cracking was observed in the polycrystalline (PX) regions of the sample, implying that the hot cracking associated with the SEBM-built parts is directly related to the mutual misorientation of the grains. Figure 2-12(b) shows orientation maps of the top regions obtained from EBSD along the $\langle 001 \rangle$ and $\langle 110 \rangle$ directions. A strong crystallographic texture is observed in the bulk regions of the sample, while the contour regions remained PX throughout the part. Being directed towards the achievement of an SX microstructure, this study did not undertake the investigation of hot cracks observed in the PX regions of the microstructure. Following this work, similar studies were carried out for the successful achievement of SX microstructure by SEBM fabrication of a non-weldable Ni-based superalloy by process parameter variation to achieve crack-free parts [17].

Figure 2-12 SX microstructure in SEBM-built CMSX-4 superalloy [15]

Hot cracking during SEBM fabrication of Ni-based superalloys was investigated in a study by Chauvet et al. [152]. They concluded that the hot cracking at a Grain Boundary (GB) was related to the angular misorientations between two grains forming the GB, and categorized two types of GBs observed in SEBM-built samples as High-Angle Grain Boundaries (HAGBs) and Low-Angle Grain Boundaries (LAGBs). According to them, the cracking was limited only to HAGBs. This study, however, did not consider the mutual growth of the grains towards each other at a GB, which is a factor deemed vital by a widely accepted criterion for cracking during solidification [145].

A review of the state-of-the-art highlights the potential of SEBM technique in fabricating Ni-based superalloy SXs. However, there is a vast gap in the knowledge that needs to be addressed before establishing SEBM as a suitable advancement over conventional manufacturing techniques employed for SX turbine blade fabrication. For instance, these studies do not investigate the contingency of cracking with solidification parameters, microstructural features or materials properties. Moreover, rarely focus has been given in investigation of secondary-phase (or TCP phase) precipitation likely to happen in Ni-based superalloys due to the cyclic reheating phenomena intrinsic to the metal AM processes as observed in DEDed Inconel 718 [142], L-PBFed Inconel 718 [153], and SEBMed Inconel 718 [154]. These limitations in the state-of-the-art are addressed in this research.

2.8 Solidification and grain growth

The grain growth accompanying the solidification of a melt pool can take place in the following modes: planar, cellular, columnar dendritic, and equiaxed dendritic, as shown in Figure 2-13. These modes correspond to the morphology of the solid/liquid interface during solidification.

Figure 2-13 (a) Planar, (b) Cellular, (c) Columnar dendritic, and (d) Equiaxed dendritic solidification modes [155]

For a constant growth rate, with decreasing thermal gradient, the final microstructure varies from planar to cellular to columnar dendritic, and finally to equiaxed dendritic, as shown in Figure 2-14 [68]. This variation is similar for constant thermal gradient and increasing growth rate. The product of the thermal gradient and solidification rate, also known as Cooling Rate (CR), governs the Primary Dendrite Arm Spacing (PDAS) for the dendrite mode of solidification.

Figure 2-14 Effect of the thermal gradient, growth rate, and cooling rate on grain growth during solidification [68]

Large values of CR assist in the formation of a fine microstructure, whereas lower values generate coarse grains. The solidification mode in a pure material is essentially planar, and for alloys, however, the solidification mode also depends upon the cooling rate and the thermal gradient to growth rate ratio [155]. In the case of alloys and impure metals, constitutional supercooling might exist [156], which occurs due to the temperature of the liquid being less than the liquidus temperature predicted by the equilibrium diagram.

2.9 Numerical modelling of metal AM processes

Most advanced fields of science and technology are not new to the idea of numerical modelling and simulation in assisting the scholars pursuing those fields to understand the underlying physics of the complex phenomenon, and for predicting effects of various process parameters on the same. Be it the kinematic modelling of the Mars rover [157] or most accurate visualization of a black hole [158] which wouldn't be possible to do physically with the state-of-the-art. This section discusses the most vital numerical simulation techniques employed for simulating macro- and/or micro-scale physics for various metal AM techniques.

2.9.1 Macro-scale modelling

This scale corresponds to the millimetre scale or system scale. The most general process of numerical modelling of metal AM at this scale involves solving the energy conservation and Navier Stoke's equation either using Finite Element Method (FEM) [159–168][169][170][171][172], Finite Volume Method (FVM) or Finite Difference Method (FDM) [28,173–176]. Recently, Lattice-Boltzmann method has been utilized to consider the powder bed as discrete particles instead of the usual continuum assumption [177–180][181]. This technique is more accurate than FEM, and FVM for metal AM processes when visualizing the effect of relative powder density, balling, capillary, and wetting phenomena [182] but is computationally intensive when compared to the two when simulating large domains especially concerning the determination of melt pool solidification parameters such as thermal gradient and cooling rate.

Apart from the numerical modelling of the system scale of metal AM process, analytical models derived from Rosenthal's equation [183], for a moving heat source, have been utilized for assisting with the understanding of the resultant microstructure in manufacturing processes that involve a moving heat source [184–187]. Many of the literature utilized thermal gradient and solidification rate thus obtained to develop processing microstructure selection maps [184,185] by using a modified form of Hunt's equation [188] which predicts the CET ahead of an advancing solid/liquid interface with respect to the thermal gradient in the melt pool and solidification rate of the interface. Raghavan et al. [28] utilized the modified form of Hunt's equation applicable to Ni-based superalloys [184] for the numerical model development and validation for SEBM of Inconel-718. Moreover, they employed the Analysis of Variance (ANOVA) method to determine the significance of process parameters, such as electron beam diameter, beam current, beam on time, and preheat temperature, on the volume fraction of equiaxed grains in the simulated melt pool. It was concluded through their study that, among these parameters, preheat temperature was the most significant while the electron beam diameter was least significant. Another work [189] on the numerical modelling of SEBM of Ti-6Al-4V alloy investigated the effect of beam scanning speed, beam power, and beam diameter on the geometry of a simulated melt pool. It was determined through ANOVA method that all the three parameters had a significant effect on the cross-sectional area of the melt pool while the melt pool length-to-depth ratio was primarily affected by beam diameter and beam scanning speed. Hence, the application of statistical methods such as ANOVA to the numerical

modelling of metal AM processes has proven fruitful in gaining new insights into the effect of process parameters on the solidification parameters or microstructural features.

Numerical modelling of metal AM process, be it of DED or PBF processes, assists in understanding the underlying complex physics associated with a multi-layer and multi-scan transient process which is usually not accounted for in an analytical formulation. On a micro-scale and meso-scale, numerical modelling of the grain growth during solidification supersedes analytical modelling by accounting for solute diffusion, visualization of the dendritic sub-grain structures and CET visualization. The next section covers in detail, various numerical techniques widely utilized for modelling and visualization of microstructural evolution during metal AM.

2.9.2 Micro-scale modelling

To date, microstructural evolution during various manufacturing processes, especially casting, has been modelled by utilization of various techniques such as vertex models, phase-field modelling, Monte Carlo pots, and cellular automaton model [190]. Among these, Phase Field (PF) modelling [191–195] and Cellular Automata (CA) [196,197] have been utilized extensively for simulation of microstructure formation during melt solidification of the binary and multi-component alloy system.

A PF model is based on a set of thermodynamically-based partial differential equations. In this modelling technique, a diffuse solid/liquid interface is considered, which is defined by continuously varying order parameter ϕ . The advantage of a PF model is in realistic microstructure simulation during solidification of ternary and multi-component alloy systems [198–201]; while being computational intensive even for small scale simulations.

A CA-based model is advantageous in terms of being computationally efficient with the capability of modelling both meso-scale grain-growth and sub-grain dendrite morphology while being limited to application in binary alloys. The dendrite tip growth velocity determination can be carried out using Kurz-Giovanola-Trivedi (KGT) model [202] – as a function of thermal undercooling at the tip, or; by solving the transport equations for both solid and liquid phases with solute conservation boundary condition at the solid/liquid interface. There have been a few efforts towards the development of an approach for the successful coupling of CA and PF techniques [203] in order to exploit their respective advantages at meso- and micro-scale, respectively. Raghavan et al. [204] devised a method for constructing pseudo-binary phase diagrams for multi-component Ni-based superalloys according to the partitioning

behaviours of its constituent elements to the γ and γ' phases respectively. They went on to utilize CA-based model to simulate the segregation of each of the constituent elements during solidification distinctly with reasonable accuracy.

In metal AM, intense research has been carried out towards coupling of macro-scale FE model with CA to simulate grain growth and accurately predict resultant crystallographic texture on a large scale in SEBM of CMSX-4 superalloy [205,206]. Recently, Shi et al. [207] presented a multi-scale numerical modelling framework for PBF of Ti-6Al-4V alloy. This framework coupled macro-scale numerical model developed using hybrid FEM and FVM method for the transient heat transfer with the CA-based micro-scale model for simulating the grain growth and crystallographic texture, and with the PF based microscale model for simulating the nucleation of α -phase from the β -grains during subsequent cooling. Their framework is a good example of the implementation of various numerical techniques towards the improvement of the knowledge of microstructural evolution during metal AM. Another interesting work by Zinovieva et al. [208] presented a novel approach towards simulating the crystallographic texture evolution of L-PBFed Ti-6Al-4V. Instead of employing different techniques for modelling the physics at different scales, their CAFD based numerical model simulates the solidification phenomena at a meso-scale. As a result, their numerical model can simulate the melting/solidification phenomena associated with a moving melt pool on a solid layer to the crystallographic texture evolution in the melt pool. Though their numerical modelling had some limitations such as lack of grain nucleation and solute diffusion, it shows promise of CA-based models in metal AM processes at different scales.

Though these works utilized KGT model for the determination of dendrite tip velocity – hence lacking in the dendrite morphology estimations, they show great promise in the usage of coupled modelling approach in metal AM especially in predicting the microstructural evolution for a given set of process parameters. Hence, CA seems to be the most applicable technique for the simulation of large-scale microstructures generated in metal AM processes.

This research utilized a Cellular Automata Finite Difference (CAFD) open-source code developed at the Imperial College London and validated extensively for simulation of grain growth, porosity growth for various casting processes [209–217]. This CAFD technique has been coupled with macro-scale numerical models for Direct Chill (DC) casting of aluminium-magnesium alloys [209], for an industrial casting of aluminum-silica-copper alloy [212], for investment casting of dental prostheses [216], and for vacuum arc remelting process for manufacturing Inconel 718 superalloy discs [215] with exquisite validation of microstructural

evolution with experimental results. An application of this CAFD technique for modelling of stray grain formation during directional solidification of superalloy CMSX-4 was carried out using a binary alloy approximation with Ni-Ta [214], and the work reported in good agreement with the experimental results.

2.10 Research Gaps

The detailed literature review on metal AM techniques and their state-of-the-art shows that the AM technology has come a long way since its advent three decades ago. In its present state, this technology has shown abundant promise in the fabrication of functional parts which would require minimum post-processing treatment. L-PBF and SEBM are two main techniques which have benefitted most from increasing research interest in metal AM. Among these two, SEBM shows promise in advancing to a mainstream manufacturing technique in sectors such as aerospace, marine and offshore, and medical manufacturing due to inherently residual stress-free parts with superior as-built mechanical properties. However, there is still a lack of research establishing robust control in grain growth during microstructural evolution in SEBM-built parts. Through the extensive literature survey presented in this chapter, the following research gaps in the state-of-the-art have been identified:

1. The majority of literature in metal AM processes to date has focussed on process parameter variation or the introduction of trace elements to the melt pool to achieve control in the grain growth during microstructural evolution, which is always limited by the material under investigation or the technique itself. Hence, there is an opportunity to explore inexpensive and non-intrusive methods of grain growth control by influencing the solidification parameters through build geometry variations.
2. The multi-scale numerical modelling adopted in metal AM research to date lacks the consideration of solute diffusion in the melt pool, which may lead to micro-scale predictions which are far from reality. It is widely known that solute diffusion plays an integral role in inducing CET during the solidification process. Hence, accurate modelling of solute diffusion during solidification at micro-scales could lead to an improved understanding of the solidification parameters which would affect the grain growth control during microstructural evolution.
3. The research in PBF metal AM techniques lacks a conclusive argument regarding the effect of powder size distribution on the thermophysical properties of the

powder bed. Moreover, there is a significant research gap in terms of the properties of powder bed at high temperatures, ideal to SEBM process.

4. The research status on the fabrication of Ni-based superalloys has several shortcomings which need to be addressed before metal AM techniques become a front runner in the fabrication of industry-relevant parts such as SX gas turbine blades. First and foremost, hot cracking is quite prominent in metal AMed Ni-based superalloys due to inherent rapid solidification phenomena. Though a workaround was figured by some researchers through process parameter optimization, there is still a need of a rigid analytical framework to correlate cracking susceptibility to solidification parameters, microstructural features and material properties. This framework would act as a tool which will enable one to work in the advantages of metal AM techniques – such as fine dendritic microstructure into the material properties of an SX superalloy. Given, its high-temperature and high-vacuum build environment, SEBM has proven to be the most suited for fabrication of Ni-based SX superalloys. However, the research lacks a dedicated investigation of TCP phases otherwise observed in nearly all of the metal AM techniques such as SEBM, L-PBF, and DED. A detailed investigation of these phases can prove beneficial in designing a new generation of Ni-based SX superalloys suitable only for metal AM techniques.

Chapter 3 Materials and Methods

This chapter provides a description of the Electron Beam Melting (EBM) system employed for sample fabrication in this research. Powder size distribution and the elemental constitutions of Ti-6Al-4V, SS316L, and AM1 superalloy powders, and their respective sample preparation techniques are detailed here as well. The numerical modelling techniques utilized for simulating grain growth during melt pool solidification are discussed here. These samples were analyzed using a variety of characterization tools detailed in this chapter such as Optical Microscope (OM), Field Emission Scanning Electron Microscope (FESEM), Energy Dispersive Spectroscopy (EDS), Electron Backscattered Diffraction (EBSD), X-Ray Diffraction (XRD), Transmission Electron Microscope (TEM), Micro-Computed Tomography (Micro-CT), and Atom Probe Tomography (APT). Uniaxial tensile testing was carried out for SS316L samples, and micro-hardness testing was performed on AM1 superalloy samples at room temperature. The thermophysical properties of sintered powder bed specimens and as-built SS316L samples were measured using a Laser Flash Apparatus (LFA) and a Differential Scanning Calorimeter (DSC).

3.1 Electron Beam Melting (EBM) system

Figure 3-1(a) shows the ARCAM A2XX EBM system employed in this research for sample fabrication. This machine offers a build envelop that is $\Phi 420$ mm in diameter and 380 mm high. The generation of electron beam takes place in an electron gun unit where the cathode-anode assembly is present. The electron beam column which houses this assembly is accessible via the column hatch located at the top of the electron gun unit. The vacuum chamber houses the hoppers, build platform, and rake assembly. Here the selective sintering and melting of the powder bed take place. The control cabinet houses the high voltage (HV) unit and the control computer. The printing process can start only when the column pressure, chamber pressure, and the door backing pressure reaches $\sim 10^{-6}$ mbar, $\sim 10^{-4}$ mbar, and $\sim 10^{-1}$ mbar respectively.

The SEBM process begins with a 3D model which is oriented in the ARCAM A2XX build environment to maximise build efficiency by using the Materialise Magics software. The necessary tolerances with regards to thermal contraction are provided, and necessary support structures are generated for overhanging structures in this stage. The oriented model and the support structures are saved as .stl files which are then loaded into the ARCAM's build assembler software. This software is used to assign the different sets of parameter definitions to desired parts and their support structures. The build file thus generated is loaded in the EBM Control 3.2 software present in the EBM control computer.

After the completion of each build, the densely-sintered powder bed is put through the Powder Recovery System (PRS) shown in Figure 3-1(b). In the PRS, a small amount of powder feedstock is mixed and cycled through – using compressed air (pressure of ~ 6 bars). This mixture of powder and compressed air is used to break the sintered powder bed and recycle it for consecutive builds. The PRS also performs an essential function of separating very small-sized particles, unfit for SEBM, from the reusable feedstock. These particles can cause smoke under the influence of negative charge transferred from the electron beam, which might result in the build failure.

Figure 3-1 (a) ARCAM A2XX EBM system employed for sample fabrication in this research. (b) Powder Recovery System (PRS) for recycling of powder

Figure 3-2(a) shows a schematic of the vacuum chamber and the electron gun unit during the SEBM process. Figure 3-2(b) and (c) show the tungsten filament (F125) and the anode compatible with the ARCAM A2XX EBM system. When a high voltage (60 kV) is applied to the tungsten filament, a large number of electrons are generated at the filament at a temperature of $\sim 2000^{\circ}\text{C}$. When a substantial electric potential is applied between the filament, acting as a cathode, and the anode, the electrons accelerate through the generated electric field resulting in a highly defocused beam. This beam is focused and directed to the powder bed with the help of the electromagnetic lens assembly, as shown in Figure 3-2(a).

Figure 3-2 (a) Schematic of the vacuum chamber and electron beam unit during SEBM process. The F125 tungsten filament (b) and the anode (c) used in the ARCAM A2XX EBM system

Stepwise description of various stages during the printing of each layer in the ARCAM A2XX EBM system is as follows:

Once the EBM system is entirely ready for use, and desired pressures are achieved in the column and build chamber respectively, preheating of the base plate is initiated, with a highly defocused beam, to a temperature of 730 – 1000 °C depending upon the feedstock. The build temperature information is gathered from a K-type thermocouple embedded in the powder bed, placed in contact with the base plate.

Once the desired baseplate temperature is achieved, the SEBM starts the actual printing process, which, is carried out in three steps for each layer. First, the powder bed is sintered, which pre-conditions it for efficient melting during the in-fill hatching step. It also ensures that the powder bed is strong enough to avoid smoking of powder particles due to the repulsion effect under the influence of a negative charge. Next, contouring precedes in-fill hatching which improves the surface finish of the as-built part. Finally, in-fill hatching melts the powder

within the contours according to the shape information provided to the control software, and the process parameters specified. Once the desired height of the part is achieved, the machine pumps down, and slow cooling of the build platform takes place. Once the temperature reaches a safe level, the part can be taken out with the sintered powder bed and is transferred to the PRS for powder recycling, after which the base plate is extracted with the parts in it.

3.2 Materials

This section details the three different engineering alloys utilized for this research – Ti-6Al-4V, SS316L, and AM1 superalloy.

3.2.1 Ti-6Al-4V powder

Pre-alloyed Ti-6Al-4V ELI (Grade 23) powder was utilized in this study for sample fabrication mainly for the multi-scale numerical model development and validation. The powder, with a size distribution of 45 – 106 μm , was supplied by ARCAM AB in 2014, as shown in Figure 3-3(a). The nominal elemental compositions of the pre-alloyed powder were Ti-6Al-4V-0.03C-0.1Fe-0.15O-0.01N-0.003H(wt.%) [100]. As shown in the micrograph, the powder morphology is ideally spherical with negligible satellites. It is known that spherical powder particles result in a higher packing density of the powder bed [218]. The multiscale numerical model was first developed around the fabrication of rectangular samples while for investigating the effect of build geometry on the resultant crystallographic texture, A-, I-, and V-shaped samples were printed in 100 mm heights [100]. Figure 3-3(b) shows an illustration of the as-built parts with their dimensions. The base of the A-shaped samples and the top surface of V-shaped samples were 20 mm broad and 10 mm thick, which was also same as the cross-sectional dimensions of the I-shaped part. All of these parts were printed without any supports where the vertex angles of both A- and I-shaped parts were $\sim 11^\circ$.

Figure 3-3 (a) SEM micrograph of pre-alloyed Ti-6Al-4V ELI powder utilized in this study. (b) Illustration of the A-, I-, and V-shaped SEBM-built parts (all dimensions are in mm).

3.2.2 SS316L powder

SEBM fabrication of SS316L parts forms an essential component of this research. As mentioned in Chapters 1 and 2, investigating the effects of build geometry and powder size distribution on the crystallographic texture in as-built microstructure constitutes a vital content of this research. To this cause, two different batches of pre-alloyed SS316L powders, provided by TLS Technik GmbH in 2016, were processed using the SEBM technique. The nominal elemental compositions of pre-alloyed powder were Fe-11.8Ni-17.1Cr-2.3Mo-0.4Mn-0.02C-0.8Si (wt.%) [13]. These powder batches exhibit different powder size distributions as well as morphologies. These two batches of powder were classified as fine and coarse powders depending upon their particulate size arrange. Figure 3-4 shows the particle size distributions of the fine and coarse powders. Figure 3-4(a) and (b) show the SEM micrographs of fine and coarse powders, respectively. As seen here, the morphological quality of the fine powder is far better than that of the coarse powder. Majority of the particles are nearly spherical with a few faceted surfaces while the coarse powder shows the presence of a large number of satellite particles fused to the surfaces of sizeable particles. Since powder size distribution is crucial to the SS316L fabrication using SEBM in this research, a laser particle size analyser was utilized to measure the same. Figure 3-4(c) shows the Malvern Panalytical's Mastersizer 3000 [219], utilized in this research, that uses laser diffraction to measure particle sizes ranging from 10 nm to all the way up to 3.5 mm. For this purpose, the powder sample was first loaded into the Aero S adapter's hopper. The powder then vibrated slowly down through the channel feeder to the laser particle-size analyzer. The feed rate for the sample feeder was set to 10%, and the air pressure used for the analysis was set to 4 bar. A laser beam was passed through the dispersed powder sample, and, as a result, the incident light was scattered at small angles by large-sized powder particles and high angles by small-sized ones as shown by the schematic in Figure 3-4(d). The angular variation in the intensity of light was analyzed to calculate the size of the particles according to the Mie scattering [220,221] and reported as the diameter of a volume equivalent sphere.

Figure 3-4 Particle size distribution analysis of (a) fine and (b) coarse SS316L powders. (c) Malvern Panalytical Mastersizer 3000 with Aero S adapter. (d) Powder-size distribution obtained from the Mastersizer

Figure 3-4(e) shows the powder-size distribution obtained from the Mastersizer. As seen here, the fine powder particle-size varies from 5 μm to 140 μm while the coarse powder particle-size distribution is in a range of 35 μm to 175 μm . The D50 diameter values were measured as $\sim 50 \mu\text{m}$ and $\sim 80 \mu\text{m}$ for the fine and coarse powder batches, respectively.

The effect of build geometry on the crystallographic texture observed in the as-built microstructure is studied using the fine powder as feedstock. These samples were fabricated after the determination of the optimum set of process parameters to achieve parts with minimum porosity and distortion [13]. For investigating the effect of a varying cross-section on the formation of crystallographic texture, X-shaped rectangular and circular samples were fabricated with vertex angles of 60°, 90°, and 120°, as shown in Figure 3-5. These samples were 10 mm high, with the width of the prismatic samples was 15 mm. The maximum width of the samples was determined by an effort to keep the necking region, which was $\sim 500 \mu\text{m}$ wide, at the midway of the sample height. These samples were classified with the nomenclature of A-B- α , where A is the shape along the build direction in the longitudinal-section, B is representative of the cross-sectional shape of the sample in each layer, and an α value of 1, 2 or 3 correspond to the vertex angles of 60°, 90°, and 120° respectively. For instance, X-R-1 implies the part has an X shape in the longitudinal section, rectangular shape in the cross-section and a vertex angle of 60°. Though the illustration does not show cuboidal and cylindrical specimens, they were fabricated, as shown in Figure 3-6, and were utilized for comparative crystallographic texture investigations.

Figure 3-5 A schematic of the SS316L fine powder samples fabricated using SEBM. All dimensions are in mm

A necking region of $500\ \mu\text{m}$ width and cylindrical supports of diameter $400\ \mu\text{m}$ were modelled to assist in the grain selection phenomena, and transitioning of $\langle 001 \rangle$ texture from weak to strong in the upper section of the fabricated parts. Four batches of these parts were fabricated onto a 150 mm base plate, as shown in Figure 3-6.

Figure 3-6 As-built fine powder parts fabricated using SEBM

Samples in sections 1 and 2 were fabricated with the Focus Offset (FO) and Speed Function (SF) values of 10 mA and 170, respectively. Parts in sections 3 and 4 were fabricated with FO of 12 mA and SF 150, respectively. Moreover, sections 2 and 4 were fabricated without contours to avoid the microstructural variations that occur in the contour region with respect to the bulk. Four outer cylinders were fabricated to increase the preheating area onto the base plate, and for carrying out density measurements. Apart from these samples, one cylindrical and one rectangular sample were also fabricated for every section for carrying out comparative investigations.

Another set of X-shaped circular samples were fabricated using SEBM technique, to demonstrate the effect of build geometry on the crystallographic texture obtained in as-built samples. Figure 3-7 is a 3D schematic of the as-built samples with their orientation on the base plate. The bottom half of the X-shape for all three shapes was identical – a right cone with 10 mm base diameter and 4.5 mm height. The samples had the same cone angle of 90° while the diameter varied from 10 – 30 mm between the three samples. All of these samples were printed

successfully without any supports. However, the narrow neck region and heavy top halves of the samples resulted in a break-off from the neck immediately after the removal of surrounding sintered powder bed.

Figure 3-7 Schematics of X-shaped samples with varying top section diameters. All dimensions are in mm

3.2.3 AM1 superalloy powder

Pre-alloyed AM1 superalloy powder was utilized for investigating the grain growth and its effect on the contingency of hot cracking during SEBM fabrication process. AM1 is a Ni-based SX superalloy with first-generation Re-free compositions of Ni–5.5Al–7.4Ta–4.4W–7.1Co–7.5Cr–1.2Ti–2.0Mo (wt.%) [222,223]. This alloy is non-weldable because it contains an aggregate Al and Ti contents of 6.7 wt.% [224], and enriched in refractory elements such as Mo, Ta, and W (13.7 wt.%), which makes it highly susceptible to strain age cracking. Figure 3-8 shows the powder size distribution of the pre-alloyed AM1 superalloy powder used in this research. As seen from the SEM micrograph of the virgin powder in Figure 3-8(a), the particles are mostly spherical with some satellites and irregularly shaped particles in the mix. The powder size distribution, as shown in Figure 3-8(b), was obtained from image analysis using the ImageJ software. A D50 diameter of 35 μm was obtained from these measurements.

Short samples with build heights of $\sim 3\text{mm}$ were fabricated using SEBM process. As detailed in Chapter 6, this height proved efficient in investigating the equiaxed-to-columnar transition region in the as-built microstructure, which was also recognized as the crack initiation region.

Figure 3-8 (a) SEM micrograph of pre-alloyed AM1 superalloy powder. (b) Powder size distribution obtained from image analysis

3.3 Electric Discharge Machining (EDM)

For extracting the SEBM-built samples from the base plate, and cutting specimens for microstructural characterization and grain growth investigations, TROOP's TP-50 Electric Discharge Machining (EDM), a.k.a. Wire-cut was used shown in Figure 3-9.

Figure 3-9 TROOP's TP-50 wire-cut system

This technique carries out the cutting via concentrated melting of the wire-metal contact surface. The diameter of the electrode wire in an EDM wire-cut system is $\sim 200\ \mu\text{m}$ resulting in a highly concentrated region for melting and cutting. As-built samples were sectioned along the build direction (Z-direction) and the beam scanning direction (X-Y plane) for further characterizations.

3.4 Thermophysical property measurements

For investigating the effect of powder size distribution on melt pool solidification parameters and resultant crystallographic texture, thermophysical property measurements are crucial. Consequently, temperature-dependent thermal conductivity measurements were carried out for SS316L sintered powder bed and SEBM-built samples using a Laser Flash Apparatus (LFA) while heat capacity measurements of the sintered powder bed were carried out using Differential Scanning Calorimetry (DSC). Figure 3-10(a) shows the NETZSCH's

LFA 457 Microflash® utilized for temperature-dependent thermal conductivity measurements carried out in this research. Figure 3-10(b) show the specimens prepared from the sintered-powder bed for LFA measurements. In comparison, Figure 3-10(c) shows specimens prepared from the SEBM-built samples. These specimens were made to adhere to the sample holder dimensions of 6 mm × 6 mm. The width of these samples remained between 1 – 1.5 mm. The sintered-powder bed samples were extracted from the well-sintered powder located below the start plate. A clean, sharp blade was used to shape the powder bed to the required dimensions. The specimens from as-built samples were first sectioned to cuboids of 8 mm × 8 mm × 3 mm dimensions. These were then manually ground to 6 mm × 6 mm × 1.5 mm to get rid of the wire-cut surface. All of these specimens were coated with graphite spray to increase their light absorptivity.

Figure 3-10 Thermophysical property measurements of SS316L sintered powder bed and SEBM-built samples. (a) LFA utilized for measurement of temperature-dependent thermal conductivity. (b) Sintered powder bed specimens from fine and coarse powders. (c) As-built ground samples

3.5 Numerical modelling techniques

3.5.1 Macro-scale modelling of the melt pool formation and solidification

Multi-scale numerical modelling of the SEBM fabrication of the three alloys utilized in this study was carried out. The macro-scale numerical simulations were carried out using the COMSOL Multiphysics software. It is a commercial Finite Element Analysis (FEA) package and is quite popular in the modelling research of AM processes [225–229]. It should be noted that due to the absence of forced convection and keyhole formation in the SEBM process, only heat transfer equations considering solid/liquid phase change were solved during macro-scale

simulations. These simulations were carried out for Ti-6Al-4V, SS316L, and AM1 superalloy. The material properties and the SEBM parameters utilized for macro-scale simulations of Ti-6Al-4V powder are shown in Table 3-1. The parameters of heat source utilized for meso-scale modelling are enlisted in

Table 3-2. It should be noted that temperature-independent material properties were utilized for multi-scale modelling of Ti-6Al-4V. This step was necessary to reduce the computational time required for the simulations. To ensure that this assumption was justified, a comparative study was carried out in 2D where temperature-dependent values of thermal conductivity and specific heat capacities were utilized for Ti-6Al-4V from [53]. The comparative analysis is shown in Figure 3-11. As seen here, the error in the temperature measurements at a point in the melt pool during solidification is 0.15 % and 0.65 % at liquidus and solidus temperatures respectively. In comparison, the same for thermal gradient measurements is 18 % and 13 % respectively.

Table 3-1 Ti-6Al-4V material properties utilized for macro-scale numerical simulations

Material Property	Value
Ambient temperature (T_o)	298.15 K
Solidus temperature (T_{sol})	1888 K [168]
Liquidus temperature (T_{liq})	1947 K [168]
Thermal conductivity of solid phase (k_{sol})	28.3 W/m.K [230]
Thermal conductivity of liquid phase (k_{liq})	37 W/m.K [230]
Thermal conductivity of sintered powder bed (k_{sint})	2.4 W/m.K [37]
Specific heat capacity of solid phase ($C_{p_{sol}}$)	848 J/(kg·K) [231]
Specific heat capacity of liquid phase ($C_{p_{liq}}$)	921 J/(kg·K) [231]
Specific heat capacity of sintered powder bed ($C_{p_{sint}}$)	848 J/(kg·K)
Density of solid phase (ρ_{sol})	4200 kg/m ³ [37]
Density of liquid phase (ρ_{liq})	4100 kg/m ³ [37]
Density of sintered powder bed (ρ_{sint})	2500 kg/m ³ (50% porosity) [37]
Absorptivity (α)	0.9 [28,37]

Table 3-2 SEBM process parameters utilized for macro-scale numerical simulations

Parameters	Value
Power (P)	900 W
Current (I)	15 mA
Beam scanning speed (Vs)	3220 mm/s
Spot radius (r_o)	254.8 μm
Hatch spacing (d_s)	100 μm
Layer height (h_l)	50 μm

Figure 3-11 Effect of temperature-dependent thermal conductivity and specific heat capacity on macro-scale numerical simulation results. (a) Temperature variation at a fixed point in the melt pool. (b) The thermal gradient variation at a fixed point in the melt pool

Table 3-3 provides the material properties utilized for multi-scale numerical simulations carried out in this research for the SEBM fabrication of SS316L. The thermophysical properties for modelling were chosen for SS316 due to their availability in literature. For modelling the moving heat source, only the scanning speed, V_s was changed to 1063 mm/s and 3035 mm/s for coarse and fine powder, respectively, while keeping the rest of the parameters the same as in

Table 3-2. Temperature-dependent thermal conductivity and specific heat capacity measurements for fine and coarse sintered powder bed were carried out using LFA and DSC. A comparative study on these properties, their usage in numerical simulations, and effect on resultant crystallographic texture shall be discussed in detail in Chapter 5.

Table 3-3 Material properties of SS316L utilized for macro-scale numerical simulations

Material Property	Value
Ambient temperature (T_o)	298.15 K
Solidus temperature (T_{sol})	1658 K [232]
Liquidus temperature (T_{liq})	1723 K [232]
Thermal conductivity (k)	$7.31+(27.2e-03) \times T - (7e-06) \times T^2$ for $T < 1658$ K [232] $355.93 - (196.8e-3) \times T$ for $1658 \text{ K} < T < 1723$ K [232] $443 + 0.2 \times T - (8e-07) \times T^2$ for $1658 \text{ K} < T < 1723$ K [232]
Specific heat capacity (C_p)	770 for $1658 \text{ K} < T < 1723$ K [232]
Density of solid phase (ρ_{sol})	7995 kg/m ³ at T_o [233]
Density of liquid phase (ρ_{liq})	6966 kg/m ³ at T_{liq} [233]
Density of sintered powder bed (ρ_{sint})	4000 kg/m ³ (50% porosity)
Absorptivity (α)	0.9 [28,37]

Table 3-4 shows the material properties utilized for macro-scale numerical simulations of SEBM fabrication of Ni-based superalloy – AM1. Given the scarcity of thermophysical property measurements carried out for this alloy, the numerical models were developed on the

basis of the data obtained from another Ni-based SX superalloy – CMSX4. This alloy is specifically chosen as it is compositionally similar to the alloy used in the study (its Al + Ti wt.% is ~6.6 wt.%), and its properties are available in the literature [234].

Table 3-4 Material properties of CMSX-4 superalloy utilized for macro-scale numerical simulations

Material Property	Value
Ambient temperature (T_o)	298.15 K
Solidus temperature (T_{sol})	1593 K [234]
Liquidus temperature (T_{liq})	1653 K [234]
Thermal conductivity of sintered powder bed (k_{sint})	2.4 W/m.K
Specific heat capacity of sintered powder bed ($C_{p_{sint}}$)	690 J/(kg·K)
Density of sintered powder bed (ρ_{sint})	4141.5 kg/m ³ (50% porosity)
Absorptivity (α)	0.9 [28,37]

Temperature-dependent thermal conductivity, specific heat capacity and density values were taken from [234]. The data was provided in a tabulated form, as shown in Table 3-5. Linear interpolation was used to get the property values at the missing data points.

Table 3-5 Temperature-dependent data for thermophysical properties of CMSX-4 [234]

Temperature (K)	k (W/m.K)	Cp (J/Kg.K)	ρ (kg/m ³)
298	-	397	8700
400	9	419	8652
600	12	448	8559
800	15.4	471	8466
1000	19.9	540	8374
1200	21.9	650	8283
1400	24.4	925	8193
1593	-	-	8107

1653	-	630	7754
1700	25	630	7710

For the SEBM-parameters, a power rating of 660W, and beam scanning speed of 1976 mm/s were provided to the macro-scale model while the rest of the parameters were the same as provided in

Table 3-2. Further details of material-specific simulations will be discussed in chapters 4, 5, and 6, respectively.

3.5.2 Meso-scale modelling of grain growth during solidification

In order to visualize the competitive grain growth phenomena occurring during solidification process inside the moving melt pool, a Cellular Automate Finite Difference (CAFD) model [211,235] was developed for two-dimensional (2D) rectangular domains using an open-source code μ MatIC. Fixed nucleation grains at the bottom of the melt pool were carried out to simulate the epitaxial grain growth inherent to SEBM-built microstructures. The thermal gradient and cooling rate data obtained from the macro-scale FE model were coupled with this CAFD model to visualize the microstructure development during melt-pool solidification.

A significant modification was carried out in the open-source code's library files to incorporate a time-dependent variation of the thermal gradient to the third degree. Details of this coupling will be provided in the model development section of Chapter 4.

3.6 Specimen preparation

The specimens obtained from the wire-cut were manually ground and subsequently polished to a mirror finish using the Struer's LaboForce-50 shown in Figure 3-12. The grinding process was carried out using SiC papers of grit sizes 200, 600, 800, 1000, and 2000. Polishing of the specimen surfaces to a mirror finish was carried out using DiaPro Dac (3 μ m suspension) and MD-Nap (1 μ m diamond suspension) provided by Struers.

Figure 3-12 Struers LaboForce-50 employed in this research for sample grinding and polishing

For the macro-scale microstructural evolution investigations, etched specimens were observed under Optical Microscope (OM) and Scanning Electron Microscope (SEM). The etching was carried out on Ti-6Al-4V and SS316L specimens using Kroll's reagent (1 – 3% HF, 2 – 6% HNO₃, and 91 – 97% H₂O). The AM1 specimens were etched with the Kalling's No. 2 reagent (5 gm CuCl₂, 100 ml HCl, 100 mm CH₃CH₂OH) for 10 – 15 s for observations under the OM and FESEM for revealing the grain morphologies and γ/γ' distribution. An extra step of polishing with OPS-suspension on MD-Chem was carried out for specimens destined for EBSD analyses. For the observations of crack surfaces of SEBM-built AM1 superalloy, specimens from the as-built samples were sectioned off to a width < 1 mm, and broken from the crack to keep the crack surface contamination-free.

3.7 Characterization techniques

This section details the characterization techniques utilized in this research for investigating the grain growth and microstructural evolutions.

3.7.1 Optical Microscopy (ZEISS Axioskop 2MAT)

OM was used to study the microstructure of as-built samples. A magnification range of 50x to 500x was utilized to investigate the grain growth in SEBM fabricated samples. For Ti-6Al-4V specimens, the columnar microstructure and heat-affected zone were identified, whereas, for SS316L specimens, the effect of melt pool topology on grain growth was investigated. For specimens from as-built AM1 superalloy samples, crack distribution in the microstructure was investigated.

3.7.2 Scanning Electron Microscopy (SEM)

In this research, two different SEM setups were utilized in order to benefit from their respective advantages. JEOL JSM-6700F, fitted with an Oxford Instruments' Energy Dispersive Spectroscopy (EDS) system, shown in Figure 3-13(a) is utilized exclusively for the characterization of powder feedstock used in this research. A significant working distance achievable for a clear image quality makes this technique ideal for capturing the powder-size distribution at 100x – 200x magnification. While the sharp images, obtained even at 1000x – 2000x magnification, as shown in Figure 3-13(b) and (c), enables a user in carrying out accurate compositional analysis of distinct powder particles. Figure 3-13(d) shows the JEOL's JSM 7600F FESEM utilized in this research for the precise investigation of grain growth and microstructural evolution in as-built samples. Due to FESEM's capability to distinguish nano-scale features, this technique has proven advantageous in understanding the effect of build geometry and powder size distribution on as-built crystallographic texture. Moreover, this technique has assisted in determining the dependence of hot cracking on microstructural features in non-weldable Ni-based superalloys. Crisp, clear images of AM1 microstructure capturing the bright secondary-phase precipitates (Figure 3-13(e)) and primary-phase precipitation (Figure 3-13(f)) were obtained at magnifications of 5000x and 15000x, respectively. The FESEM system is equipped with Oxford Instruments's EDS system, which was utilized extensively for the compositional analyses of the secondary-phase precipitates observed in SEBM-built AM1 microstructure. Electron backscatter diffraction (EBSD) analysis of the OPS-polished specimens of as-built SS316L and AM1 superalloy samples was carried out using the JEOL JMS-7600F FESEM. The orientation mapping was carried out at

an accelerating voltage of 20 kV and a probe current setting of 18. The map settings were provided to the AZTEC software associated with the FESEM. The EBSD maps were utilized for crystallographic texture investigations and grain size analysis using the Oxford Instruments' Tango software.

Figure 3-13 (a) JEOL JMS-6700F Scanning Electron Microscope (SEM) utilized in this work for imaging the powder particles at 1000x magnification (b) and (c). (d) JEOL JMS-7600F Field Emission SEM (FESEM) employed to investigate as-built microstructure. (e) FESEM micrograph of as-built microstructure photographed at 5000x magnification showing clear evidence of secondary-phase precipitation. (f) FESEM micrograph of primary-phase precipitates photographed at 15000x magnification

3.7.2.1 Crystallographic texture investigations

For the investigation of the crystallographic texture obtained in SEBM-built SS316L and AM1 superalloy samples, EBSD mapping of OPS-polished specimens was carried out at various magnifications and areas. For the determination of texture dependence on build geometry, large-scale mapping was carried out on specimens prepared from SS316L fine powder X-shaped samples. For understanding the effect of powder size distribution on grain size and crystallographic texture, comparative EBSD analyses of specimens from fine and coarse powder samples was carried out. The EBSD mapping of fractured fine and coarse powder samples was also performed to understand their respective deformation mechanisms. A region 2 mm away from the fracture surface was chosen for both the samples. The crystallographic texture maps were generated, and the grain size measurements were carried out using Oxford Instruments' Tango software. Grain size measurements for both fine and coarse powder specimens were carried out with six grain-misorientation thresholds of 0.5 °, 1 °, 2 °, 5 °, 10 °, and 15 ° to avoid any errors arising due to the presence of a large number of well-aligned grains.

For microstructure of the SEBM-built AM1 superalloy, large-scale EBSD mapping of longitudinally sectioned samples was carried out. 56 fields of 119 μm $\times$ 90 μm at a magnification factor of 1000x and step size of 1 μm were mapped out in a sequential manner, and later montaged to a macro-scale orientation map – 1.08 mm high and 0.42 mm wide using the AZTEC software. The grain size measurements were performed using the Oxford Instruments' Tango software using a misorientation threshold of 5 °.

3.7.2.2 Primary Dendrite Arm Spacing (PDAS) measurements

PDAS obtained in an as-built microstructure provides a direct indicator of the cooling rate during solidification of the melt pool. Hence, PDAS measurements are crucial for understanding the solidification parameters affecting a melt pool. Hence, PDAS measurements

of the as-built microstructure for SS316L and AM1 superalloy were carried out using the ImageJ software from the SEM micrographs.

3.7.3 Transmission Electron Microscopy (TEM)

TEM is beneficial in distinguishing the nano-scale features and deformation modes of fractured samples. Thus, TEM studies were carried out for SS316L and AM1 superalloy samples. For SS316L, specimens were prepared from fractured samples (subjected to uniaxial tensile testing) for a detailed comparative investigation of the deformation modes in fine and coarse powder samples. For AM1 superalloy samples, specimens were prepared to investigate the nano-scale microstructural features such as primary- and secondary-phase precipitations. Moreover, TEM investigations assist in the determination of lattice parameters by the Selected Area Electron Diffraction (SAED) technique. Figure 3-14(a) and (b) shows the setup used for TEM specimen preparation. Samples were sectioned and manually ground to ~50 μm in thickness. $\Phi 3$ mm disks were punched out from these specimens which were subsequently ground on the Gatan's 656-model dimple grinder (Figure 3-14(a)), and successively milled in the Gatan's 691-model precision ion polishing system (Figure 3-14(b)) at 3.5 kV and a milling angle range of 4 - 8 $^\circ$. These were analysed using the JEOL JEM-2010 TEM (Figure 3-14(c)), with a beam accelerating voltage of 200 kV. Micro-scale compositional analyses of the same samples were carried out using the Oxford Instruments' EDS. The SAED pattern was first calibrated using known lattice parameters of pure polycrystalline Al thin-film specimen and the calibrated column length of 1361.5 mm was utilized for SAED pattern indexing using the CrysTBox software [236].

Figure 3-14 Transmission Electron Microscopy (TEM) of SEBM-built samples. (a) Gatan's 656-model dimple grinder. (b) Gatan's 691-model precision ion polishing system. (c) JEOL JEM-2010 TEM showing the CRT monitor (inset in yellow) and the viewing chamber (inset in blue)

3.7.4 X-ray Diffraction (XRD)

XRD is beneficial for bulk phase analysis and qualitative texture analysis of AM as-built samples. For this research, this technique was advantageous to gain an understanding of the prevailing crystallographic texture in the microstructure qualitatively. Thus, XRD analyses of the as-built samples were performed using the PANalytical Empyrean system with an applied voltage of 40 kV and a current of 40 mA. XRD analyses of SS316L samples were carried out at the cross-sectional surface near the top while for AM1 superalloy samples, specimens were chosen at two cross-sections: one close to the top surface and other 1.5 mm below the top surface. XRD peaks were collected over a 2θ range of $30 - 100^\circ$ with a step size of 0.02° . Removal of background noise, smoothing, and peak search was performed on the raw data using the HighScore software.

3.7.5 Micro-Computed Tomography (Micro-CT)

Given its non-destructive nature and a scanning resolution well below the minimum particle size of fine and coarse SS316L powders utilized in this research, micro-CT can prove beneficial for providing a quantitative estimation of the density of the sintered powder bed. Hence, Bruker's Skyscan 1173 was employed to analyse the sintered powder beds of fine and coarse stainless steel powders. Samples measuring 5 mm × 5 mm × 1 mm for both of the powder beds were extracted from the region located beneath the base plate. The 3D micro-CT analyses were performed using a 130 kV X-ray source and a spot size of 5 μm. The raw data generated from the analyses were processed through the 3D.SUITE software provided with the Bruker system.

3.7.6 Atom Probe Tomography (APT)

To get the exact elemental constitution information of the primary- and secondary-phase precipitates in the microstructure of SEBM-built AM1 superalloy, the powerful and unique APT technique for quantitative chemical mapping in 3D, and at atomic scale was employed. Specimens were prepared by using the focused ion beam (FIB) technique on an FEI Helios dual-beam via the site-specific lift-out method. APT specimens were examined by the local-electrode atom probe (LEAP) 3000X HR at 70 K and a gauge pressure $<2.0\text{e}^{-11}$ Torr under voltage mode. Data analysis was performed using IVAS 3.8.2 software, and compositional analysis was carried out by using both the proximity histogram (proxigram) and one-dimensional (1D) concentration profile methods. As a bonus, APT analysis of SS316L coarse powder specimens was carried out to investigate the solute distribution.

3.7.7 Uniaxial tensile testing

Uniaxial tensile testing of SEBM-built SS316L samples was carried out under a constant loading speed of 0.2 mm/min using Shimadzu AGX 10 kN equipped with a non-contact digital video extensometer. Tensile specimens were prepared from SEBM-built blocks of dimensions 30 mm × 30 mm × 25 mm, as shown in Figure 3-15, and were tested at room temperature. The gauge length of samples was 10 mm for horizontal direction and 5 mm for the vertical direction. The initial cross-sectional area for both types of samples was 3 mm². The strain rate was 3.3E-04 s⁻¹ for the horizontal specimens and 6.7E-04 s⁻¹ for vertical specimens.

Figure 3-15 (a) Schematic of tensile specimens extracted from the block samples, (b) Sample along the vertical direction, (c) Sample along the horizontal direction (All dimensions are in mm)

3.7.8 Micro-hardness measurements

Vickers micro-hardness (HV) tests were performed in the XY cross-sections of the as-built samples of SEBM-built AM1 superalloy using the Future-Tech's FM-300e with a load of 300 g. These measurements were conducted in crack-free regions at room temperature. A mean microhardness value was determined from 9 indentations taken in a 3×3 matrix, where each indentation was 0.5 mm apart.

Chapter 4 SEBM Fabrication of Ti-6Al-4V – Microstructural Characterization, Multi-scale Numerical Model Development and Validation

This chapter provides a detailed overview of the multi-scale numerical modelling of SEBM process implemented in this research. At the macro-scale, Finite Element Analysis (FEA) was used to solve heat transfer equations associated with a moving heat source in a domain which consists of specific regions representative of Ti-6Al-4V sintered powder bed, as-built Ti-6Al-4V, and stainless steel base plate. The phenomenon of melt pool solidification was modelled by the inclusion of solid/liquid phase transformation with temperature. The meso-scale modelling of the melt pool solidification was accomplished by the Cellular Automata Finite Difference (CAFD) method. Microstructural characterization of SEBM-built Ti-6Al-4V samples was performed by OM and FESEM. It was determined using macro-scale simulations that the banded microstructure observed in the longitudinal micrographs of as-built Ti-6Al-4V, identified by different contrasts under OM, is due to the Heat Affected Zones (HAZs) and their varying depths via different beam scanning lengths leading to the formation of periodic banding pattern. The periodic variation in the band thickness is geometry dependent, and constant thickness of bright and dark bands is observed for square geometries. The part geometry was determined to affect the solidification parameters of the melt pool in different regions of the part. Amongst the A-, I-, and V-shaped geometries investigated in this research, the V-shaped geometries are seen to be most supportive of strong <001> crystallographic texture.

4.1 Introduction

SEBM-built Ti-6Al-4V parts have shown promise in producing parts with negligible anisotropy in mechanical properties [90,91]. Moreover, the mechanical properties obtained surpassed the ASTM standard requirements [92]. However, SEBM-built Ti-6Al-4V parts have also shown a variation in these properties with degradation in YS, UTS, and elongation % along the build height [35]. This variation has been attributed to the equiaxed-to-columnar transition of the microstructure observed in as-built samples, and a gradual coarsening of the prior- β grain and the α/β dual phase microstructure with build height – for 100 mm high samples. However, Prior- β grain refinement strengthening will be more effective in improving the mechanical properties as these grain boundaries have a higher capacity to absorb dislocations in comparison to the α/β interfaces [35]. Hence, with its ever-growing applications in the fields of aerospace and biomedical, it is necessary to investigate the columnar grain growth in SEBM-built Ti-6Al-4V parts. Achieving a robust control of the solidification parameters which govern the grain growth can lead to a significant advancement in the metal AM technology.

Grain growth control in metal AM processes has hitherto been limited to either process parameter variation or post-processing methods. These methods involve a lot of trial and errors since the microstructural evolution during metal AM of alloys takes place through rapid solidification, and the formation of various phases during the process is unpredictable due to segregation of alloying elements at the grain boundaries (GBs). Hence, there is a need for achieving grain growth control during microstructural evolution by techniques that would eradicate the extensive trial-and-error methods.

Metal AM processes involve a complex mixture of a variety of heat transfer physics associated with them including melting and solidification of the powder bed, remelting and solidification of deposited material, fluid flow within the melt pool and at the surface, and many more. These processes together govern the microstructural evolution in the as-built parts. An accurate visualization of these processes is nearly impossible by using state-of-the-art imaging techniques. Even the microstructural observations of the parts using a variety of characterization techniques can only provide a snapshot of information of the end product but not about the complex heat transfer physics associated with every layer that led to the final microstructure. This is where numerical simulations can lead the way in providing one with the ability to predict/estimate the solidification parameters and resulting microstructure, specific to a metal AM technique independent of the material utilized. Multi-scale numerical

simulation techniques can even assist in visualizing elemental segregation, and thus provide valuable insights in tailoring the solidification parameters to achieve desired phases in the as-built microstructure.

This chapter discusses the development and validation of a multi-scale numerical model associated with the SEBM fabrication of Ti-6Al-4V alloy. It provides a novel insight into the effect of build geometry on the solidification parameters and resultant crystallographic texture in the as-built AM microstructure.

4.2 SEBM fabrication process

For the SEBM fabrication of Ti-6Al-4V parts investigated in this research, build themes provided by ARCAM AB for Ti-6Al-4V were utilized, namely, Ti-6Al-4V-preheat-50 μ m and Ti-6Al-4V-melt-50 μ m. The process parameters settings provided in these themes have been identified by ARCAM AB for optimum part density. The thermocouple temperature specified for a 150 mm $\times$ 150 mm base plate, at which the building process can start, for Ti-6Al-4V was 730 °C. Once the desired build temperature was achieved, the building proceeded in the order of preheating, melting, and then post-heating for each layer. For melting, a beam current of 15 mA and beam scanning speed of 3220 mm/s were employed. Each layer height was kept as 50 μ m and a hatch spacing 100 μ m.

4.3 Numerical modelling of the SEBM process

Multi-scale numerical modelling, representative of melting/solidification phenomena associated with SEBM process, is carried out in this research and is detailed in this chapter. The macro-scale model is developed in an FEA software, COMSOL, while the meso-scale modelling of the grain-growth phenomena during melt pool solidification is carried out using a CAFD based open-source code - μ MatIC [212].

4.3.1 Macro-scale modelling of melting/solidification

The macro-scale numerical modelling of the melting/solidification phenomena accompanying the SEBM process was carried out in a sequential manner as shown by the flow diagram in Figure 4-1. To begin with, a 3D macro-scale numerical model was generated for determining the heat flow along the x, y, and z directions, and for investigating the HAZ of Ti-6Al-4V. For the sake of clarity, an AM standard was followed during modelling where the z-direction was aligned with the build direction and the x-direction with the beam scanning direction. Following this sequence ensured efficient utilization of the computational resources

while providing maximum insight to the complex heat transfer physics that accompanies melting/solidification in a SEBM process.

Figure 4-1 Flow diagram for multi-scale numerical simulations

4.3.1.1 3D model

Figure 4-2 shows a schematic of the 3D model constructed for transient heat transfer simulations and HAZ investigations. As seen here, the model consists of three distinct domains – deposited material, sintered powder bed, and base plate. A radiative heat transfer boundary condition (BC) is applied to the top surface of the domain to account for the loss of heat through radiation. Since in the actual SEBM process, the melted region is surrounded by highly insulating powder bed, insulated BC is applied to the sides and at the base of the domain. An initial condition (IC) of a uniform temperature of 630 °C was applied to the entire model. This

temperature value correlates directly with the thermocouple measurements recorded during the SEBM fabrication Ti-6Al-4V samples.

Figure 4-2 3D numerical model showing the melt pool schematic (All dimensions in mm)

For the deposited material and the sintered powder bed, the material properties were specified as detailed in Table 3-1. The base plate was modelled with the material properties of stainless steel available in the COMSOL material database. The dimensions of the modelled domain are summarized in Table 4-1. It also provides the values of the diffusion lengths calculated for each of these domains. Diffusion length is vital in determining whether the dimensions of a domain modelled for heat transfer simulations is large enough to be representative of the process independent of the dimensions. It is given by:

$$L_D = 2\sqrt{Dt} \quad (4-1)$$

Where D is the thermal diffusivity of the material in the domain and t is the time. For each of these domains, the diffusion length was calculated for with their respective material properties and the time it takes for an electron beam to scan a 10 mm long powder bed three times, i.e. 0.009 seconds. Here the diffusion length values of the base plate and the sintered powder bed is most vital since, for the deposited material, it is necessary for the powder bed to affect the thermal history of the melt pool to relate more closely to the actual process where the deposited material is always surrounded by the powder bed.

Table 4-1 Dimensions of the simulated domains

Domain	Dimension (mm)			Diffusion length (mm)
	x	y	z	$L_D = 2\sqrt{Dt}$
Stainless steel base plate (Heat transfer in solid)	7	3	5	0.4
Sintered powder bed (Heat transfer in solid)	7	3	2	0.2
Deposited Material (Heat transfer with solid/liquid phase change)	5	1	2	0.5

Transient heat transfer equations associated with solid/liquid phase change were solved for the deposited material domain with the incorporation of a moving heat source with a gaussian distribution of $\sigma = 127.4 \mu\text{m}$. This 3D model also incorporated the multi-pass beam scanning of 3 passes. This 3D model was also utilized for simulating the difference in HAZ between short and long pass beam scans. The length of a single scan for a short pass was 2.5 mm – half of the long pass.

4.3.1.2 2D model

The macro-scale modelling was extended further in 2D in a yz-plane associated with $x = 3.5 \text{ mm}$ of the deposited material domain shown in the 3D model with the heat source moving along x-direction and perpendicular to the yz-plane as shown in Figure 4-3. The material properties and the process parameters for the 2D simulations were specified same as that for the 3D simulations, with the exception of the presence of adaptive meshing in 2D, to improve the accuracy of the simulations.

Figure 4-3 2D adaptation of the 3D numerical model (All dimensions in mm)

This 2D approximation of the SEBM process relied on the observations from 3D simulations that the thermal gradient within the melt-pool along the beam scanning direction (x-direction) was $< 17\%$ of the gradients in y- and z-directions during solidification. The resultant thermal gradient which governed the grain growth in the melt pool was inclined to the z-direction at an angle less than 4 degrees towards x-direction; the solidification of the melt pool, hence, was mainly in the yz-plane.

This simplification proved beneficial in terms of lower computational resources required to carry out the simulations. The time taken for 2D simulations in man-hours was 90 times less than that for 3D simulations. Due to this reason, this study in two dimensions was extended to incorporate the effect of build geometry on the resulting thermal history in the melt-pool with build height. The 2D heat transfer simulations were extended for SEBM simulation of A-, I-, and V-shapes for large domain sizes comparable to that of the experiments with the build height variation of 10 - 100 mm as shown in Figure 4-4.

Figure 4-4 Schematic of 2D macro-scale simulations. S0-S5 indicate various heights for simulations
(All dimensions in mm)

4.3.1.3 Governing equations

In the case of 3D modelling, heat transfer physics related to phase change during melting/solidification was considered, hence, only the energy equation was solved for the 3D domain. Since the SEBM process takes place in a vacuum chamber, heat transfer due to forced convection is absent. The BCs for the model only consisted of radiative boundary condition on the top surface and insulation on every other boundary. The governing equation for energy conservation solved using COMSOL Multiphysics software is given as [238]:

$$\frac{\partial}{\partial t}(\rho H) + \nabla \cdot (\rho V H) = \nabla \cdot (k \nabla T) + S_h \quad (4-2)$$

Where H is the enthalpy of the material expressed as:

$$H = h + \Delta H \quad (4-3)$$

Here, ΔH is latent heat, and h is sensible heat given by,

$$h = h_o + \int_{T_o}^T C_p dT \quad (4-4)$$

With, h_o and T_o being reference enthalpy and temperature, respectively. The latent heat ΔH is obtained from:

$$\Delta H = (1 - s)L \quad (4-5)$$

Where, the solid fraction s is defined as,

$$\begin{aligned} s &= 1 & \text{for } T \leq T_s \\ s &= \frac{T_l - T}{T_l - T_s} & \text{for } T_s < T \leq T_l \\ s &= 0 & \text{for } T > T_l \end{aligned} \quad (4-6)$$

The source term in Eq. (4-2) can be written as [238]:

$$S_h = \frac{\partial(\rho\Delta H)}{\partial t} + \nabla \cdot (\rho V \Delta H) \quad (4-7)$$

4.3.1.4 Design of Experiments (DOE)

Since the numerical model being developed and detailed in this chapter was intended to transcend across different materials, a Design of Experiments (DOE) approach was employed to estimate the influence of the individual process parameters. The process parameters in question were chosen to be the beam power, beam scanning speed, and the preheat temperature – as these are the ones which are varied primarily during SEBM fabrication of different materials. Beam power is varied through the beam current, and the beam scanning speed through the Speed Function (SF). A full factorial ANOVA was performed on a model developed with these three parameters, each having three levels ($3^3 = 27$ runs) in the statistical software - Minitab. 2D macro-scale numerical simulations were performed where the responses for the DOE were taken as solidification parameters – Thermal Gradient (G), Growth Rate (R), and the ratio of G and R (G/R). The values of G and R were recorded at a point in the middle of the melt pool – 50 μm deep from the melt pool surface when the temperature at these points reached $T_{\text{liq}} = 1947 \text{ K}$. G/R was treated as a possible indication of the CET, where a lower value improved the tendency of CET as shown in Figure 2-14 of Chapter 2. The levels of beam power were taken as 800 W, 900 W, and 1000 W. Same for the preheat temperature were taken as 750 °C, 850 °C, and 950 °C. The beam scanning speed was varied as 2000 mm/s, 3000 mm/s, and 4000 mm/s. A null hypothesis was chosen that neither these parameters nor their interactions have any significant effect on G, R, and G/R, and a significance level of 0.05.

4.3.2 Meso-scale modelling of grain growth during melt pool solidification

This section details the 2D numerical simulations carried out at meso-scale of melt pool solidification. These simulations showcased the competitive grain growth phenomena taking

place during melt pool solidification in SEBM processing. A varying thermal gradient value determined from the macro-scale 2D simulations was provided as an input to the meso-scale model. A major modification to the CAFD source code was carried to incorporate a varying thermal gradient value to a third-degree polynomial in time. Figure 4-5 shows the thermal gradient data obtained at 70 μm depth in the centre of the melt-pool and the parameters of polynomial fit from the start of solidification at that point.

Figure 4-5 Time-dependent variation of the thermal gradient along build direction at a point in the centre of the melt pool. The polynomial fitting is carried out at the advance of solidification

This thermal gradient fitted curve was translated along the time axis since the melt pool solidification in macro-scale numerical simulation started at $t = 0.0007$ seconds when the temperature reached 1947 K during cooling. Hence the input of time-dependent thermal gradient to the 2D meso-scale model was provided as per the equation given by:

$$\frac{\partial T}{\partial z} = 2.46e07 - 5.06e10 \times (t - 0.0007) + 4.07e13 \times (t - 0.0007)^2 - 1.1e16 \times (t - 0.0007)^3 \quad (4-8)$$

A 2D domain was modelled simulating the melt pool dimensions. The width and height of the domain were taken as 180 μm and 100 μm , respectively. Fixed nucleation of 15 grains with random orientations ranging from -15° to $+15^\circ$ deviations from the growth direction was carried out at the bottom of the domain. The material properties specified for the meso-scale simulations were the same as that for the macro-scale modelling. A binary phase alloy approximation is assumed with a 10 wt.% solute concentration.

4.4 Results

4.4.1 Macro-scale modelling

4.4.1.1 Grid convergence study

The 3D numerical model was developed to realize the significant direction of heat transfer during solidification of moving melt-pool. During the development process, a grid convergence study carried out for four mesh sizes ranging from around 26000 elements to 400,000 elements, revealed a substantial increase in computational round-off error during thermal history calculations at a point in the symmetry plane just below the maximum melt pool depth - with the increasing number of mesh elements. The mesh was generated using the physics dependent meshing provided by default in COMSOL. Figure 4-6 showcases the superimposed thermal history curves - with same peak temperatures and cooling rates for 57,000 elements and above but with a drastic increase in the computational round-off error.

Figure 4-6 Grid convergence study of the macro-scale 3D model carried out for 4 mesh sizes

Deriving from the grid convergence analysis, 57000 mesh elements were chosen for the study. For improving the mesh accuracy in regions of very high thermal gradients and cooling rates, user-defined meshing was carried out with specifications of boundary layers in regions with expected high thermal gradients in y and z directions respectively. The smallest element size close to the free boundary was chosen to be $4\text{ }\mu\text{m}$. The resultant thermal history recorded in the melt pool for 57000 mesh elements, shown in Figure 4-7 indicates the absence of round-off error which was prominent in the default mesh.

Figure 4-7 A thermal profile recorded in the melt-pool for 57000 elements with boundary layer meshing

4.4.1.2 Validation of the macro-scale 3D numerical model

Since the melt pool boundaries were not visible in the etched micrographs of SEBM-built Ti-6Al-4V, depth of the HAZ for the last SEBMed layer was utilized for the validation of the 3D model. Pertinent to this cause, comparison of HAZ depths obtained from the 3D numerical simulations with that observed from the optical micrographs of the longitudinal sections of SEBM-built Ti-6Al-4V samples was carried out. Since the HAZ is a long-term effect, it is highly affected by the beam scanning speeds and the number of beam passes.

Figure 4-8 shows this comparison between the HAZ obtained from the 3D simulations of short pass beam scan and the observed HAZ in the micrograph. As seen in the optical micrograph of the longitudinal section shown in Figure 4-8(a), the top bright region shows a HAZ, a region where $1220\text{ K} < T < 1888\text{ K}$, depth of $\sim 330\text{ }\mu\text{m}$. Figure 4-8(b) shows the temperature contours obtained from a moving melt pool indicating a HAZ depth of $345\text{ }\mu\text{m}$ and width of $1010\text{ }\mu\text{m}$ obtained after three passes.

Figure 4-8 Validation of macro-scale numerical simulations. (a) Optical micrograph of SEBM-built Ti-6Al-4V (b) Contour plot showing temperature distribution below the melt pool obtained from the 3D numerical model

4.4.1.3 Effective thermal gradient during solidification of Ti-6Al-4V melt pool

Figure 4-9 shows the contour plots of the thermal gradient along the x, y, and z directions (Figure 4-9(a), (b), and (c) respectively), and the resultant thermal gradient

magnitude (Figure 4-9(d)) represented by T_x , T_y , T_z , and T_R respectively, obtained during the long pass beam scanning. The plots were obtained in a cut plane at $x = 3.5$ mm towards the end of the last beam scan. These plots are superimposed with the melt pool obtained from the simulations as well, the boundaries of which are indicated with the red lines on the plots. The lower melt pool boundary corresponds to the solidus temperature, $T_{sol} = 1888$ K while the upper boundary corresponds to the liquidus temperature, $T_{liq} = 1947$ K. The region enclosed between the two boundaries is the mushy region where both solid and liquid phases of the alloy exist. The solid phase is in the form of dendrites while the liquid phase is present in the form of film at the GBs and sub-GBs. At the advent of solidification, this mushy region width is negligible and grows continuously as the solidification progresses. A melt pool depth of ~ 100 μm and width of ~ 500 μm was obtained from these simulations after three beam passes.

The colour legends provided above each of the contour plots correspond to their respective colour mapping in the contour plots. As seen here, the contour plot of T_z is almost identical to that of T_R . An average difference between their respective values being less than 0.5 %. The thermal gradient along the y-direction was maximum near the top surface, and the maximum thermal gradient along the x-direction was $< 17\%$ than that along the z-direction. However, near the melt pool boundary, T_z dominated the grain growth near the bottom of the melt pool while both T_y and T_z governed the grain growth in the top regions of the melt pool. This distribution trend of thermal gradient would support the growth of prior β grains whose $\langle 0002 \rangle$ crystallographic orientations are well aligned with the build direction as the effective thermal gradient which causes solidification would be directed along the build direction.

Figure 4-9 (a) Thermal gradient along z-direction, b) y-direction, and c) z-direction. (d) The resultant thermal gradient

It is evident from the contour plots that the thermal gradients along the y- and z-directions governed the overall grain growth during solidification. Hence, it can be concluded

that the heat transfer in the x-direction, in this case, will not contribute towards solidification; making the development of 2D models valid in both macro- and meso-scales.

4.4.1.4 Microstructural banding formation in SEBM-built Ti-6Al-4V

The appearance of the banded microstructure in AM of titanium alloys has been reported [73,239–242]. The common features between the reported banded microstructure are that the repeating pattern has approximately the same thickness as each layer thickness and that the top-most region often consists of no bandings. The microstructure bands have been reported to be formed through the eutectoid reaction when the β phase transforms into the $\alpha+\beta$ phases during cooling. A study based on the direct laser deposited Ti-6Al-4V alloy reported that the microstructural banding gradually disappeared at the top of the build sample [241].

From Figure 4-8, the thickness of the banded microstructure formed in the SEBM-built sample was not equivalent to the build layer height of 50 μm . It was found out that this was due to differences in the depth of HAZs obtained for different beam scan lengths for the rectangular geometry with a cross-sectional dimension of $10 \times 20 \text{ mm}^2$. A recent study also reported that there were differences in the microstructure for varied scan lengths in SEBM-built Ti-6Al-4V for different beam current values [243].

3D numerical simulations of multi-pass scans were performed to investigate the effect of build cross-sectional geometry on the HAZ. In the SEBM process, the snake-type scanning pattern was rotated by 90° in every consecutive layer. Therefore, for a build part with either one of x or y dimensions larger than the other, the beam scan lengths are different for alternating layers, such as the case in this research. This implies that within the same time span, the shorter beam scan length should witness a higher number of passes, and hence affecting the melting and HAZ in that layer due to heat accumulation. Figure 4-10 shows that for the long beam scan length, which in this case is twice than that of the short scan length, the HAZ resulted from the moving heat source – a region of $1220 \text{ K} < T < 1888 \text{ K}$, had a width of $\sim 806 \mu\text{m}$ and a depth of $\sim 275 \mu\text{m}$, while the HAZ with a width of $\sim 1010 \mu\text{m}$ and a depth of $\sim 345 \mu\text{m}$ was formed for the short beam scan length.

Figure 4-10 Computational simulations of the melt pool. (a) shows the HAZ formed by the longitudinal scan of 10 mm with a melt pool width of 690 μm and a depth of 270 μm while (b) shows the HAZ by the transverse scan of 5 mm with a melt pool width of 845 μm and a depth of 300 μm

The difference in the HAZ depths for the two consecutive layers during melting results in the non-uniform microstructural banding, schematically illustrated in Figure 4-11. The simulated HAZ depth is shown to be in good agreement with the OM observation in Figure 4-11(a). Figure 4-11(b) clearly demonstrates the formation process for the periodical microstructural banding of alternate $\sim 20 \mu\text{m}$ and $\sim 100 \mu\text{m}$ in thickness. In order to verify the formation mechanism of the microstructural banding in SEBM-built Ti-6Al-4V, a square block sample of 15 mm $\times$ 15 mm was also examined. It was found that a uniform microstructural banding consisting of $\sim 50 \mu\text{m}$ bright and $\sim 50 \mu\text{m}$ dark bands were formed. All these further helps to prove that the microstructural banding was formed due to the overlapped HAZs from consecutive layerwise melting and solidification.

Figure 4-11 (a) OM image showing the experimental observation of the microstructural banding with HAZ at the top-most region. (b) Schematic of the formation mechanism of the non-uniform microstructural banding based on computational simulation results. (c) Resolved views of (b) in YZ and XZ planes showing the formation of the banding in 4 consecutive layers

4.4.1.5 Validation of the macro-scale 2D numerical model

The 2D macro-scale model was validated with the experimental observations from SEBM fabricated thin-walled Ti6Al-4V samples. The melt pool width obtained, of 520 μm, from 2D simulations was validated with the width of thin-walled Ti-6Al-4V specimens which are basically single-scan multi-layer depositions; the OM image and width observations of which are shown in Figure 4-12.

Figure 4-12 OM image of thin-walled Ti-6Al-4V specimen (Top) and corresponding width measurements using ImageJ (Bottom). The scale on the micrograph is 200 μm

The average value of the width of the thin-wall for ten observations along the build height is 470 μm . The variation in this value for different observations can be attributed to the thermal contraction and adherence of semi-melted powder particles on the melt pool wall.

4.4.1.6 Design and Analysis of Experiments (DOX)

Table 4-2 presents the full factorial combinations of process parameters employed for 2D numerical simulations, and the values of G , R , and G/R measured at a point in the melt pool at the advent of solidification.

Table 4-2 3³ combinations for numerical simulations, and the corresponding G, R, and G/R values

Number	Power (W)	Temperature (° C)	Scanning speed (mm/s)	Thermal Gradient, G (K/m)	Growth Rate, R (m/s)	G/R (K-s/m ²)
1	800	750	2000	8.73E+05	3.98E-02	2.19E+07
2	800	750	3000	2.42E+06	5.30E-02	4.57E+07
3	800	750	4000	4.18E+06	9.05E-02	4.61E+07
4	800	850	2000	5.04E+05	1.47E-01	3.42E+06
5	800	850	3000	1.66E+06	6.37E-02	2.60E+07
6	800	850	4000	3.11E+06	6.44E-02	4.82E+07
7	800	950	2000	3.40E+05	2.10E-01	1.62E+06
8	800	950	3000	1.09E+06	3.75E-02	2.90E+07
9	800	950	4000	2.29E+06	6.82E-02	3.35E+07
10	900	750	2000	1.21E+06	2.85E-02	4.26E+07
11	900	750	3000	2.00E+06	5.17E-02	3.87E+07
12	900	750	4000	3.23E+06	5.51E-02	5.86E+07
13	900	850	2000	3.77E+05	1.66E-01	2.27E+06
14	900	850	3000	1.24E+06	3.60E-02	3.44E+07
15	900	850	4000	2.43E+06	5.10E-02	4.78E+07
16	900	950	2000	1.77E+05	3.60E-01	4.91E+05
17	900	950	3000	7.98E+05	8.81E-02	9.05E+06
18	900	950	4000	1.69E+06	5.08E-02	3.32E+07
19	1000	750	2000	4.31E+05	1.58E-01	2.72E+06
20	1000	750	3000	1.46E+06	6.05E-02	2.40E+07
21	1000	750	4000	2.65E+06	4.44E-02	5.98E+07
22	1000	850	2000	2.40E+05	1.79E-01	1.34E+06
23	1000	850	3000	1.07E+06	5.99E-02	1.79E+07
24	1000	850	4000	1.93E+06	5.46E-02	3.54E+07
25	1000	950	2000	9.97E+04	4.35E-01	2.29E+05

26	1000	950	3000	6.02E+05	9.17E-02	6.56E+06
27	1000	950	4000	1.22E+06	6.30E-02	1.94E+07

The statistical significance of process parameters on the solidification parameters (G, R) and the tendency for CET (G/R) was investigated by the ANOVA method. R. sq values of 99.58 % and 96.90 % were obtained for the models with G and R as response variables, respectively, and the same of 92.1 % was obtained for the model with G/R as the response variable. Table 4-3, Table 4-4, and Table 4-5 display the results from the ANOVA of the design with G, R, and G/R as response variables, respectively. A P-Value > 0.05 would imply that the null hypothesis is valid. Moreover, factors and their mutual interactions for which the F-value is less than 4.46 are deemed insignificant in affecting the response variable.

As seen in Table 4-3, the interaction between power and temperature is insignificant towards the variation in the thermal gradient in the melt pool as $F < 4.46$ and $P > 0.05$. Scanning speed, with an F-value of 617.08, is the most influential towards determining the thermal gradient.

As seen in Table 4-4, the beam scanning speed is also the most influential to the variation in growth rate during solidification. Along with this, the preheat temperature and its interaction with the scanning speed is another influential factor towards the growth rate. In terms of the G/R ratio, only scanning speed and preheat temperature are statistically significant towards the predicted CET in the melt pool.

Table 4-3 ANOVA of the design with G as the response variable

Source	Adjusted Sum of Squares	Degrees of Freedom	Adjusted Mean of Square	F-Value	P-Value
Power (W)	2.52E+12	2	1.26E+12	81.51	0.000
Temperature (° C)	5.78E+12	2	2.89E+12	186.96	0.000
Scan Speed (mm/s)	1.91E+13	2	9.53E+12	617.08	0.000
Power*Temperature	1.79E+11	4	4.47E+10	2.90	0.094
Power*Scan	7.57E+11	4	1.89E+11	12.25	0.002
Temperature*Scan	7.41E+11	4	1.85E+11	11.99	0.002
Error	1.24E+11	8	1.54E+10		
Total	2.92E+13	26			

Table 4-4 ANOVA of the design with R as the response variable

Source	Adjusted Sum of Squares	Degrees of Freedom	Adjusted Mean of Square	F-Value	P-Value
Power (W)	8.09E-03	2	4.05E-03	4.19	0.057
Temperature (° C)	3.97E-02	2	1.99E-02	20.55	0.001
Scan Speed (mm/s)	1.03E-01	2	5.17E-02	53.56	0.000
Power*Temperature	7.93E-03	4	1.98E-03	2.05	0.179
Power*Scan	1.71E-02	4	4.26E-03	4.41	0.035
Temperature*Scan	6.53E-02	4	1.63E-02	16.9	0.001
Error	7.73E-03	8	9.66E-04		
Total	2.49E-01	26			

Table 4-5 ANOVA of the design with G/R as the response variable

Source	Adjusted Sum of Squares	Degrees of Freedom	Adjusted Mean of Square	F-Value	P-Value
Power (W)	6.60E+14	2	3.30E+14	3.47	0.082
Temperature (° C)	2.41E+15	2	1.21E+15	12.69	0.003
Scan Speed (mm/s)	5.18E+15	2	2.59E+15	27.27	0.000
Power*Temperature	2.17E+14	4	5.42E+13	0.57	0.692
Power*Scan	1.92E+14	4	4.80E+13	0.5	0.734
Temperature*Scan	1.73E+14	4	4.32E+13	0.45	0.767
Error	7.60E+14	8	9.51E+13		
Total	9.60E+15	26			

4.4.1.7 Effect of build geometry on melt pool solidification conditions

2D numerical simulations were utilized to investigate the influence of build geometry on the solidification parameters that, eventually, dictates the grain growth during melt pool solidification. Thermal gradients and cooling rates at varied build heights along the build and transverse directions at a fixed point inside the melt pool - a depth of 70 μm from the surface, were simulated for A-, I-, and V-shaped samples respectively.

Figure 4-13(a) shows that the thermal gradient along build direction (T_z) during solidification decreases with build height for A-shaped sample, remains constant regardless of build height for I-shaped sample, and increases with build height for V-shaped sample. Moreover, the variation of the thermal gradient along the transverse direction (T_y), shown in Figure 4-13(b), to be like T_z . However, the influence of T_y on grain growth could be negligible since it is one order of magnitude lower than T_z .

Figure 4-13 Thermal gradient plots inside the melt pool at different heights for A-, I-, and V-shapes (left to right)

Figure 4-14 Thermal history variation for A-, I-, and V-shapes at 10, 20, 40, 60, 80, and 100 mm locations with regard to the base plate

Figure 4-14 shows the thermal history variation among A-, I-, and V-shaped samples at various build heights. The slope of the temperature curve during solidification yields the cooling rate (CR). As shown here, the CR does not vary significantly with build height for all the samples. However, it is noteworthy to point out that the thermal heat variation is relatively larger with the increase in build height in the A- and V-shaped samples, and it remains constant throughout the I-shaped build. Therefore, build geometry, particularly build cross-section, plays an important role in determining the thermal distribution and the resultant microstructure in SEBM builds.

Though the thermal history plots shown above provide valuable insights regarding the thermal history variation with build geometry at a fixed point in the melt pool, it is imperative to observe the thermal gradient variations with build height and build geometry in the entire melt pool. Hence, contour plots of thermal gradient magnitude in the melt pool were compared with increasing build heights for A-, I-, and V-shapes, as shown in Figure 4-15.

Figure 4-15 Contour plots of thermal gradient magnitude for A-, I-, and V-shapes - left to right, and for 10 - 80 mm heights on samples 100 mm high - bottom to top

Like Figure 4-9, the melt pool boundaries are indicated by black curves with the lower curve corresponding to the solidus temperature of 1888 K and upper curve to the liquidus temperature of 1947 K, respectively. The contour plots for all the sections were generated at the same simulation times. The maximum melt pool depth recorded from the 2D simulations was $\sim 90\text{ }\mu\text{m}$ that is quite similar to the 3D simulations. In the contour plots shown in Figure 4-15, the depth of the melt pool is $80\text{ }\mu\text{m}$ indicative of the initiation of solidification at the simulation time for which the plots were generated. The direction of the resultant thermal gradient magnitude can be devised from the solid/liquid interface during solidification. It is well known that the resultant thermal gradient at a point at the interface will always be normal to the interface at that point.

4.4.2 Meso-scale modelling

Figure 4-16 shows the simulated grain growth during directional solidification in a layer at four consecutive time steps. The orientation angle deviating from $\langle 001 \rangle$ build direction for each specific grain is labelled and provided in Table 4-6. The simulation results show that the columnar prior β grain growth did not always obey the classical competition model. For instance, well-orientated grains (e.g. grain 13) was overgrown by less-orientated grains (e.g. grains 12 and 14). Hence, there is a possibility of the competition of the grains to not follow the classical model.

Figure 4-16 Columnar grain growth during directional layerwise solidification process as obtained from the meso-scale CAFD model: (a) Grain orientation mapping of totally 15 columnar grains with the respective misorientation angles indicated and (b) solute concentration maps showing the competitive growth of columnar grains (initial value - 10 wt.%)

Table 4-6 Orientation of grains w.r.t the $\langle 001 \rangle$ growth direction

Grain	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Orientation (deg.)	-3.2	-10.3	-7.5	8.0	13.7	-3.1	1.4	11.4	0.16	-4.7	18.7	-8.3	4.6	8.7	-14

Competitive grain-growth was observed for fixed nucleation of 15 grains with random orientations and positions at the bottom of the melt-pool. Specifically, grains 4, 5, 6, 7, 11, and 13 are superseded by their neighbouring grains. The elimination of grains 4 and 5 by grain 3 and grain 11 by grains 10 and 12 support the classical competitive model - the grain well

oriented to the thermal gradient is favoured for growth; the elimination of grain 6 by 3, 7 by 6 and 8, and 13 by 12 and 14 clearly violates the classical model.

4.4.3 Effect of build geometry on crystallographic texture evolution

Figure 4-17 shows the XRD analysis of the SEBM-built Ti-6Al-4V samples for investigation of the phase constitution and qualitative crystallographic texture. Three main peaks designating prismatic (1010), basal (0002), and pyramidal (1011) crystal planes of the α phase were determined by the peak search, and are selectively highlighted. Due to its limited volume fraction of $\sim 3\%$ in the as-built microstructure [35], residual β phase is rarely to be detected by conventional XRD technique in SEBM-built Ti-6Al-4V. It is noteworthy that the relative intensity of the $(0002)_\alpha$ peak varies for different geometries and, within them, along the build heights. In the A-shaped sample, the relative intensity of $(0002)_\alpha$ peak is seen to decrease with build height. A contrasting behaviour of this peak is seen in the V-shaped sample while in the I-shaped sample, it decreased until halfway of the build height. It is then seen to increase with build height. The relative intensity of $(0002)_\alpha$ peak could be an important indicator of the crystallographic texture of prior β grains formed during solidification in different Ti-6Al-4V build geometries.

Figure 4-17 Bulk phase and qualitative crystallographic texture analysis of A-, I-, and V-shaped samples at different build heights

4.5 Discussion

Melt pool solidification parameters such as thermal gradient and cooling rate govern the grain growth during microstructural evolution in any solidification-assisted manufacturing process. With the advent of metal AM techniques and their prospective applications towards end-product manufacturing raises a need to control the grain growth during rapid solidification at the micron scale.

The multi-scale numerical modelling of the SEBM fabrication of Ti-6Al-4V alloys provides valuable insight into the effect of build geometry on the resultant crystallographic texture. It is well known that CR and thermal gradient (G) are two solidification parameters that govern the grain growth during solidification. This subsection will discuss the effect of build geometry on these two parameters.

The effect of build geometry on the CR is evident from Figure 4-14. The slope of the temperature variation with during solidification determines the CR. In the thermal history plots shown in Figure 4-14, linear fitting was carried out for the range of solidification temperature. It is evident that the CR does not vary with the height of the beam scan for A and I shapes respectively. However, there is a substantial increase in the CR with increasing build height for V-shape. The effect of this variation in CR and along with thermal gradient can be implemented on stray grain formation during solidification, which in turn affects texture strength in a SEBM fabricated part.

Figure 4-15 highlights the effect of build geometry on the thermal gradient. The thermal gradient distribution in the I-shaped sample remains uniform throughout the build height while the same changes dramatically for A- and V-shaped geometries. Similarly, for an A-shaped sample, the thermal gradient distribution remains constant until the build height of ~ 40 mm, and after that, it changes to resemble the thermal gradient distribution obtained in V-shaped samples in reverse order. The contour plot for A-shape at 60 mm height is similar to that obtained at 40 mm in the V-shape. Same goes for A-shape at 80 mm and V-shape at 20 mm. The dimensions of the melt pool curve in all the contour plots, except V-shape 10 mm and 20 mm, is similar. This implies that the cooling rates were the same in these regions as well. A closer look at the contour plot obtained for V-shaped sample at 10 mm reveals that the melt pool is much deeper and wider in comparison to the melt pools observed at different geometries at the same simulation time. This implies that the V-shaped part cooled at a much slower rate at the height of 10 mm. For the same magnitude of resultant thermal gradient and a lower CR

value would reduce the tendency of columnar-to-equiaxed transition [155], thus assisting in continuous improvement in crystallographic texture with build height. This conclusion is supported by the qualitative crystallographic texture observations from the XRD data shown in Figure 4-17.

With regards to SEBM-built Ti-6Al-4V, it was reported that there exists no variant selection operated during the SEBM process due to the weak α phase texture in the as-built material [187,244]. Moreover, the reconstructed β phase was often presented to have a strong $[001]_\beta$ texture due to the $\langle 001 \rangle$ crystal orientation being the preferred direction of grain growth in cubic metals [187,244,245]. It is noteworthy pointing out that the $(0002)_\alpha$ peak does not represent the crystal orientation of the prior β grains due to the unique phase decomposition process of $\beta \rightarrow \alpha' \rightarrow \beta + \alpha$ that occurs in SEBM-built Ti-6Al-4V. A recent study revealed that under a $\beta \rightarrow \alpha$ variant selection mechanism during cooling, the α variant is more incline to orientate close to the initial grain growth direction due to a texture memory effect [246]. This is supported by prior studies, where one such study reported that at elevated temperatures the rolled commercially pure (CP) titanium possessed a $(001)_\beta$ texture though when cooled a dominant $(0002)_\alpha$ texture was obtained instead [247]. A more recent study also observed that after Hot Isostatic Pressing (HIP) process the $[0002]_\alpha$ texture close to the build direction was preferentially selected, and variants that were perpendicular to the build direction disappeared [248]. Thus, it was hypothesized that $(0002)_\alpha$ peak can correspond to the (001) peak of the prior β grains, i.e. the strong $\langle 0002 \rangle_\alpha$ texture indicates the prevailing $\langle 001 \rangle_\beta$ texture.

The height of every layer specified in the ARCAM Ti-6Al-4V process theme is 50 μm . However, due to $\sim 58\%$ porosity of the powder bed, the very first melted layer has a height of $\sim 20\ \mu\text{m}$. However, since the build table moves by 50 μm for every layer, the height of each powder bed layer ready for sintering and melting is $\sim 80\ \mu\text{m}$. However, the macro-scale numerical simulations provide a maximum melt pool depth of $\sim 90\ \mu\text{m}$ from single-scan 2D simulations or at best $\sim 100\ \mu\text{m}$ from multi-scan 3D simulations. This implies that for every layer, only 20 μm of the previous layer gets re-melted. This implies that the grain growth which occurs during solidification to a height of 30 μm contributes to the epitaxial grain growth for the next layer, and to the final microstructure in the deposited part.

When focusing on the contour plots shown in Figure 4-15, to a height of around one-third of the melt pool depth, it becomes apparent that the resultant thermal gradient magnitude is predominant in z-direction in both higher build heights of A-shape and lower build heights of V-shape which assists in grain growth in $\langle 001 \rangle$ direction.

Earlier research [188] quantified a condition for equiaxed grain growth during solidification according to the theory of columnar-to-equiaxed transition (CET), which can be written as:

$$G < 0.617N_o^{\frac{1}{3}} \left\{ 1 - \frac{\Delta T_N^3}{\Delta T_c^3} \right\} \Delta T_c \quad (4-9)$$

Where G is the imposed thermal gradient, N_o is the total number of substrate particles initially available for nucleation, ΔT_N is the undercooling at the heterogeneous nucleation temperature, and ΔT_c is the undercooling of the columnar growth front.

This was further modified for rapid solidification [249][250][184], which is given by,

$$\frac{G^n}{V} < a \left\{ \sqrt[3]{-\frac{4\pi N_o}{3 \ln[1 - \phi]} \frac{1}{n + 1}} \right\}^n \quad (4-10)$$

Writing solidification rate V , in terms of CR and G , this can be rewritten as:

$$\frac{G^{n-1}}{CR} < a \left\{ \sqrt[3]{-\frac{4\pi N_o}{3 \ln[1 - \phi]} \frac{1}{n + 1}} \right\}^n \quad (4-11)$$

The value of parameter ϕ varies from 0 - indicating no stray grains, to 1 - indicating nucleation of equiaxed grains, thus dominating over columnar grains and interrupting epitaxial grain growth during microstructural evolution.

For the same material and constant cooling rate, the tendency of equiaxed grain formation increases with decreasing thermal gradient. Hence, for A-shape, stray grain formation is predominant at shorter build heights which results in an overall weak <0002> texture in A-shape. The increase in thermal gradient and cooling rate with build height for V-shape results in an effective decrease in growth rate and parameter ϕ . This, in turn, assists in epitaxial grain growth hence resulting in a stronger <0002> texture.

4.6 Summary

The multi-scale numerical modelling carried out in this chapter has proven vital in highlighting the effect of build geometry on the crystallographic texture. The novelty of the numerical modelling detailed in this chapter lies in the fact that the CA-based meso-scale numerical model takes into account the solute diffusion while accounting for the time-

dependent variation of the thermal gradient in the melt pool – both of which are a significant advancement over the state-of-the-art in numerical modelling of metal AM processes. The meso-scale CAFD model has shown that deviations to the classical competitive grain growth model do occur during melt pool solidification, wherein grains with their $\langle 001 \rangle$ growth direction oriented along the effective thermal gradient are superseded by the misoriented ones. Moreover, the macro-scale 3D numerical simulations revealed the phenomena behind microstructural banding observed universally in additively manufactured Ti-6Al-4V that was previously inscrutable. The HAZ caused by the moving electron beam has different depths depending upon the length of the beam scan. This leads to an extra step of heat treatment of the as-built microstructure trapped in the region of overlapping HAZs leading to a eutectoid phase transformation.

The macro-scale 3D model led to an understanding of the effective thermal gradient which governs the solidification process. It was determined that for SEBM fabrication of Ti-6Al-4V using the standard ARCAM build themes, the thermal gradient along the beam scanning direction had the least effect on the grain growth. It was also determined that primarily, the SEBM process favours epitaxial grain growth resulting in elongated columnar grains in as-built microstructure since the thermal gradient along the build direction is the most significant during solidification. The thermal gradient along the direction transverse to that of the beam scan direction influenced the grain growth only at the top regions of the melt pool.

With the help of macro-scale 2D models and XRD analyses, the effect of build geometry on the resultant crystallographic texture was investigated. The information gathered helped in determining that among A-, I-, and V-shaped geometries, V-shaped part will inherently support strong $\langle 0002 \rangle$ crystallographic texture in SEBM-built Ti-6Al-4V parts.

Chapter 5 SEBM Fabrication of SS316L – Investigation of Grain Growth and Microstructural Characterization

This chapter provides a detailed overview of the investigation of grain growth during the microstructural evolution of SEBM-fabricated SS316L alloy. Two different methods have been devised based on the literature review, and numerical modelling to obtain both a strong $\langle 001 \rangle$ crystallographic textured and a fully equiaxed microstructure in the as-built condition. Coincidentally, the effects of build geometry and powder size distribution on the as-built microstructure of SS316L have been investigated independently. For the build geometry studies, fine pre-alloyed SS316L powder was utilized, while coarse powder was utilized for investigation of the impact of powder size distribution on as-built microstructure. Microstructural characterization of SEBM-built samples was performed by OM and FESEM. Electron Backscatter Diffraction (EBSD) was employed for the crystallographic texture analysis. X-ray Diffraction (XRD) was utilized for the bulk phase analysis and determination of the qualitative crystallographic texture. Uniaxial tensile testing was performed on the as-built samples. The investigations of the deformation mechanism of as-built samples were performed on a Transmission Electron Microscope (TEM). For the fine powder samples, strong $\langle 001 \rangle$ crystallographic texture was observed in X-shaped samples in comparison to I-shaped ones. Moreover, with increasing prism angles of the X-shape, the strongest texture was obtained for an angle of 90° . With this prism angle, samples were printed, which showed a technical SX texture (misorientation $< 15^\circ$) in bulk. Among the fine- and coarse-powder samples, the latter showed a fully equiaxed microstructural evolution during SEBM. Also, nearly isotropic mechanical properties were obtained in the coarse-powder samples under uniaxial tension. The investigation of the deformation mechanism

by EBSD and TEM analyses of deformed specimens revealed the presence of a large number of deformation twins in the coarse powder samples.

5.1 Introduction

Exhaustive control of the grain growth during microstructural evolution in as-built metal AM parts has been challenging ever since the advent of Rapid Prototyping (RP) into massive AM production. The ability to tailor the microstructure of as-built parts can lead to a significant advancement of the metal AM techniques by improving their capabilities towards the fabrication of functionally graded parts, topologically optimized parts or parts with predictable mechanical properties.

Metal AM, where the core process involves remelting of deposited layers to produce the next layer of a part, suffers from an inherent tendency to assist epitaxial grain growth. When a deposited layer is remelted, the grains from its layer, which lie at the advent of the melt pool boundary, act as heterogeneous nucleation sites, and the newly generated grains tend to follow the same crystallographic orientation until they are impeded by another grain. Moreover, large thermal gradients present in a melt pool of a typical metal AM process support growth of columnar grains over equiaxed grains. This results in the formation of elongated columnar grains, aligned with the build direction, and lasting for a few layers of the as-built parts [100]. Large aspect ratios of these columnar grains result in anisotropic mechanical properties, where the tensile properties along the build direction deteriorate due to lack of grain boundaries to resist dislocation movements. A significant alternative to columnar grain growth is the equiaxed one which results in a homogeneous microstructure and isotropic mechanical properties. Moreover, since the equiaxed grain growth is a random process with a large number of nuclei forming and growing at the same time, ultra-fine grain sizes can be achieved that can improve the overall mechanical strength of the part [12] or even achieve superplasticity [79,251–253]. Hence, the ability to achieve an equiaxed microstructure in the as-built condition is both desirable and necessary for metal AM research.

Over the years, researchers have achieved equiaxed grain microstructure either by process parameter variations [12,25,26], nano-scale inclusions [24,32] or post-processing treatments [32]. However, these processes have a few limitations of their own. Process parameter variations require intense trial and errors, and similar variation in these parameters may not result in similar microstructural evolution for different materials. For instance, Sun et al. [12] showed that increasing laser power for L-PBF fabrication of SS316L alloy resulted in an increase in the formation of equiaxed grains, which led to excellent mechanical properties. On the other hand, Montero-Sistiaga et al. [26] showed that for L-PBF-fabricated Hastelloy X, high power of laser resulted in a primarily columnar microstructure with stronger

crystallographic texture. Nano-scale inclusions are the most effective in equiaxed grain nucleation and growth, but a careful evaluation of their weight fraction during addition to the feedstock is required since a large amount of them can affect the elemental compositions of the as-built part or form detrimental compounds or intermetallics. Post-processing treatments, such as Hot Isostatic Pressing (HIP), add to the cost of the fabrication process, and may not be as effective for a part with graded microstructure. Hence, alternative methodologies of varying the solidification parameters through variations of build geometry and powder size distribution are sought and addressed for SEBM-built SS316L alloy in this chapter.

Figure 5-1 is a flowchart depicting the process sequence employed for this study. As seen here, two independent methods of achieving control during grain growth in the microstructural evolution of SEBM-built SS316L were utilised – variation in build geometry and powder size distribution. The effect of build geometry was investigated on fine powder samples where the effect of powder size distribution was observed amongst fine and coarse powder samples. The flowchart also depicts the determining factor on which more focus is given (red dashed box). The sections detailed herewith will follow the flow chart.

Figure 5-1 A flow diagram depicting the sequence of various studies carried out in this chapter

5.2 SEBM fabrication process

The SEBM fabrication of SS316L parts was carried out on the basis of the optimized set of process parameters [13]. Fabrication of fine powder samples with varying geometries was carried out for an SF value of 150 and FO of 12 mA. The thermocouple temperature specified for a 150 mm × 150 mm base plate, at which the building process could begin, for these samples was 820 °C. Once the desired build temperature was achieved, the building proceeded in the order of preheating, melting, and then post-heating for each layer. For melting, a beam current of 12 mA and beam scanning speed of 2500 mm/s were employed. Each layer height was kept as 50 µm and the hatch spacing as 100 µm. For the fabrication of both fine and coarse powder samples for the investigation of powder size distribution effect, all the process parameters were kept the same to have an unbiased comparison. To obtain fully dense parts for both of these powders, a build start temperature of 850 °C was specified, and the part fabrication was carried out with an SF value of 170 and FO of 10 mA.

5.3 Results

5.3.1 SEBM as-built SS316L

For validation of the optimised set of process parameters employed for SS316L part fabrication in this research, density measurements were performed on as-built samples using a densitometer according to the Archimedes principle. The as-built samples were wire-cut from the start plate and were ground to remove the contaminated wire-cut surface of ~ 300 µm. These samples were then weighed first in an ethanol bath and then in the air. Then the density of each sample was determined using the formula:

$$\rho_s = \rho_l \left(\frac{w_a}{w_a - w_l} \right) \quad (5-1)$$

Where, ρ_s is the density of the sample, ρ_l is the density of ethanol, w_a is the measured weight of the sample in air, and w_l is the measured weight of the sample in ethanol.

The densities of 4 cylindrical samples were measured in this way, which were printed alongside the parts investigated in this study, and the results of their relative density measurements are shown in Figure 5-2.

Figure 5-2 Relative density results of four SEBM-built SS316L samples

As seen here, the density variations in the as-built parts are $< 1\%$ between the samples with the lowest measured density being 98.7% , and the highest being 99.5% . These measurements were felicitous to confirm that the process parameters utilised for the SEBM fabrication of SS316L alloy were adequate for producing fully dense parts.

Next, the bulk phase analysis of the fine powder samples was carried out using non-destructive XRD method. The top surfaces of the samples were analysed to obtain information about their phase compositions. Figure 5-3 shows the XRD plot of fine powder sample compared with that of the feedstock powder utilised for this study. The dashed drop lines are indicative of the phases (BCC α -ferrite and FCC γ -austenite), and their respective crystal planes present in a duplex steel microstructure.

Figure 5-3 Bulk phase analysis of the powder feedstock utilised and SEBM-built SS316L sample using XRD

As seen here, the XRD peaks of the powder feedstock correspond to 2theta values of 43.54 °, 44.5 °, 63.8 °, 74.56 °, 81.44 °, and 90.52 ° in the specified range of 40 – 100 °. The values of d-spacing for the crystal planes at these values can be calculated using Bragg's law [254]:

$$n\lambda = 2d\sin\theta \quad (5-2)$$

Where n is a positive integer, λ is the wavelength of the incident wave, d is the interplanar spacing, and θ is the angle of incidence. Since the X-rays were generated using Cu, the wavelength λ was taken as 0.154 nm.

The d-spacings corresponding to the 2theta values obtained above are 0.2076 nm, 0.2033 nm, 0.1457 nm, 0.1271 nm, 0.1180 nm, 0.1084 nm. These d-spacings conform with d-(111)γ, d-(110)α, d-(200)γ, d-(200)α, d-(220)γ, d-(211)α, and d-(311)γ, respectively. As seen

in Figure 5-3, the feedstock utilised for SEBM part fabrication is in the form of dual-phase steel while the as-built samples were comprised only of the γ -austenite phase. Hence, it is worth noting that the build conditions in SEBM inherently support the formation of pure austenite phase steel.

Next, the microstructure of the SEBM-built fine powder samples is characterized. Figure 5-4 is the optical micrograph of the longitudinal section of a fine powder block sample. As seen here, the melt pool boundaries are visible due to severe elemental segregation. Six grains numbered 1 – 6 are illustrated with distinct colours. Epitaxial grain growth is prominent in as-built samples, and as a result – prolonged grains $\sim 100\ \mu\text{m}$ wide, are observed which extend to a few scan layers. Moreover, grain coarsening is observed in a few grains which happen at the expense of neighbouring grains. The alignment of the subgrains with growth direction is indicative of their crystallographic texture. Hence, it is seen that competitive grain growth following the classical model occurred. For instance, grain 2, whose subgrains are well aligned with the build direction, widens from $\sim 120\ \mu\text{m}$ width to $\sim 150\ \mu\text{m}$ as it grows along the build direction. Contrary to this, grain 3, visibly misaligned with the build direction, was overgrown by grains 2 and 4. This implies that competitive grain growth is a significant factor in shaping the final microstructure. It is also observed, that the columnar subgrains do not align with what would be the direction of the resultant thermal gradient at the melt pool boundary (normal to the boundary), which is due to good epitaxial grain growth.

Figure 5-4 Optical micrograph of the longitudinal section of SEBM-built fine powder specimen.

Varied colours were super-imposed to differentiate different columnar grains

5.3.2 Effect of build geometry on as-built microstructure

The X-shaped samples fabricated with the fine SS316L powder were characterized using OM, FESEM, EBSD, and XRD. The initial observations were performed to determine that grain growth in the necking region of X-shaped samples. This region is vital because it acts as a zone of crystal selection which would eventually determine the crystallographic texture of the upper half of the as-built part. Figure 5-5 presents the analysis of the observed microstructure in X-R-2 sample. As seen in Figure 5-5(a), the sample ~ 2 mm wide on either side of the necking region, the X-shape does not have any visible deformities. The thin columnar struts at the ends of the X-shape are columnar with delamination at the ends. Delamination of layers is observed at the ends of the X-shape to about ~ 100 μm . Figure 5-5(b) is a magnified view of the necking region with melt pools of ~ 500 μm wide and a height of ~ 26 μm between consecutive layers. The microstructure shows elongated grains with the subgrains aligned well with the build direction. Figure 5-5(c) shows the EBSD mapping of the necking region shown in Figure 5-5(a). The orientation map shows a strong $\langle 001 \rangle$ crystallographic texture in this region with the grains being ~ 2 mm long. These results confirm that the necking region, in fact, acts as a crystal selection region wherein the strong $\langle 001 \rangle$ crystallographic texture prevails.

Figure 5-5 (a) Micrograph of the transverse section of X-R-2. (b) An etched micrograph showing the melt pool geometry in the necking region. (c) EBSD mapping of the necking region

The purpose of using this geometry modification over the conventionally built I-shaped parts was to achieve a stronger $\langle 001 \rangle$ crystallographic texture in the as-built microstructure. Thus, it was only viable for a comparison study between the X-shaped and I-shaped samples. To this cause, comparative XRD was carried out between varying prism angles of X-shaped

parts and the I-shaped parts printed in the same build. Figure 5-6 shows the bulk phase analysis of the two shapes performed at the top surfaces of the as-built samples.

Figure 5-6 Comparison of bulk phase compositions of I- and X-shaped as-built samples

As seen from the XRD plots in Figure 5-6, both the I- and X-shaped samples are comprised of entirely austenite phase with an FCC crystal structure. However, the stronger peak is observed for the (111) plane which is indicative of a substantial $\langle 111 \rangle$ crystallographic texture in the as-built I-shaped samples as well. Qualitative texture analysis of various X-shaped and I-shaped samples was carried out using XRD as well. For this purpose, X-shaped - X-R-1, -2, and -3, and I-shaped samples were utilised, and the pole figures obtained from the XRD texture analysis of the top surfaces of these samples are shown in Figure 5-7.

Figure 5-7 Pole figures comparing (200) texture for different X-shaped samples. (a) X-R-1. (b) X-R-2. (c) X-R-3. (d) I-shaped sample

The pole figures, as shown above, were taken in the cross-sectional plane of the samples. They present a fascinating trend regarding the crystallographic texture variation with build geometry. It is noteworthy that a very strong $\langle 200 \rangle$ (or $\langle 100 \rangle$) crystallographic texture

free from substantial noise is obtained in SEBM-built SS316L samples that had not been reported in the literature so far. Further, sample X-R-2 having a prism angle of 90° showed the best quality among all these samples. The worsening of the texture for X-R-3 implies that there is a transition point among the prism angles in terms of crystallographic texture which lies at 90° . However, it is worth noting from the pole figures shown in Figure 5-7 that X-R-1 and I-shaped samples have a close resemblance in the crystallographic textures, while X-R-3 shows the second strongest crystallographic texture among the four samples investigated.

For understanding the reason behind this phenomena, macro-scale numerical modelling of the SEBM of SS316L alloy was carried out for V-shaped domains with varying vertex angles from 60° to 120° . Figure 5-8(a), (b), and (c) show the contour plots of the thermal gradient magnitude obtained during solidification for build geometries X-R-1, X-R-2, and X-R-3, respectively. A magnified view of each of the melt pools is also shown in the inset next to the plots. The arrows are vector representations of the resultant thermal gradient with their sizes being proportional to its magnitude. The colouring of the arrows also corresponds to the magnitude of the thermal gradient provided in the legend associated with the contour plot.

Figure 5-8 Numerical simulations of melt pool solidification for varying part geometry. (a) X-R-1, (b) X-R-2, and (c) X-R-2 with magnified views of their respective melt pools

As seen here, there is a continuous increase in the maximum thermal gradient magnitude from 5.26×10^6 to 5.55×10^6 K/m from X-R-1 to X-R-3. However, it is noteworthy

that for X-R-1 (Figure 5-8(a)) relatively strong thermal gradient magnitude exists slightly away from the datum point of the melt pool – indicated by red arrows with a value of $\sim 4.55 \times 10^6$ K/m. Given that this thermal gradient is sufficiently inclined from the build direction, it would promote grain misorientation by affecting the $\langle 001 \rangle$ grown grains. Similar thermal gradients exist in the melt pools of X-R-2 and X-R-3 however, their magnitude values are relatively lesser than the maximum thermal gradient along the build direction. As one moves along the melt pool boundary towards the surface of the melt pool, the thermal gradient during solidification tends to align more with the horizontal direction. The higher the magnitude of this gradient, the stronger it will negatively affect the $\langle 001 \rangle$ crystallographic texture. Among the three build geometries, this gradient value is highest for X-R-3 at $\sim 2.18 \times 10^6$ K/m. Hence, this is the reason for a weaker crystallographic texture obtained in X-R-3.

Further investigations on the effect of build geometry on as-built crystallographic texture were carried out for a prism angle of 90° , and by increasing the cross-sectional diameter of the parts. Three SS316L parts 25 mm high and varying diameters from 10 – 30 mm were fabricated, as shown in Figure 3-7 in Chapter 3. Figure 5-9 shows large-scale EBSD orientation mapping of these samples along the build direction in the longitudinal/transverse plane. Figure 5-9(a), (b), and (c) correspond to diameters 10, 20, and 30 mm respectively. These maps were taken for half of the area of the longitudinal/transverse plane but are assumed to represent the other half. The geometry below the necking region is identical for all the samples with 10 mm diameter. The base plate region lies below the dashed yellow horizontal line. Pole figures along with the three primary directions - $\langle 100 \rangle$, $\langle 110 \rangle$, and $\langle 111 \rangle$ for each of the top and bottom regions are shown above and below the regions, respectively.

Figure 5-9 EBSD orientation maps for X-shaped samples of (a) 10 mm diameter, (b) 20 mm diameter, and (c) 30 mm diameter

The EBSD maps and their adjoining pole figures, as shown in Figure 5-9, show that there is a significant improvement in the $\langle 001 \rangle$ crystallographic texture from the bottom through the necking region and to the top of the samples. Among the three samples, the strongest and best quality of the crystallographic texture is observed for 30 mm wide sample which was quite similar to the 20 mm wide sample (Figure 5-9(a)). Moreover, the 10 mm wide sample has regions with strong misorientation among grains (encircled in black). This presence

of these regions is likely due to lack-of-fusion porosity due to which epitaxial grain growth gets interrupted, and the grains grow from the powder bed present below the melt pool.

The information gathered from the XRD texture analysis of the samples, shown in Figure 5-7, when coupled with the knowledge gained from numerical simulations of the melt pool observed in the three geometries, shown in Figure 5-8, proves that there is indeed an effect of build geometry on the $\langle 001 \rangle$ crystallographic texture in the SEBM-built SS316L alloy parts. The X-shaped geometry assists the competitively grown $\langle 001 \rangle$ grains in comparison to the I-shaped geometry. However, there exists a sweet spot at the prism angle of 90° for which strongest $\langle 001 \rangle$ crystallographic texture – an almost Single Crystal (SX) microstructure can be obtained as shown in Figure 5-7 and Figure 5-9. This information can prove beneficial in SEBM fabrication of SX parts which do not confine to a rectangular geometry. For instance, an X-shaped geometry can be provided as a support structure for overhangs to avoid the growth of misoriented grains in the region.

5.3.3 Effect of powder size distribution on as-built microstructure

For powder bed metal AM processes, the thermophysical properties of powder bed play a vital role in governing the melt pool topology and its solidification parameters. Since the powder bed particles are loosely in contact with each other, the thermophysical properties are a few orders of magnitude smaller than that of the bulk material. SEBM has an advantage in this regard over L-PBF processes as the preheating and sintering steps before the actual melting process result in improved contact between the particles by the generation of necking between neighbouring particles due to localised melting. A higher value of thermophysical properties such as thermal diffusivity and thermal conductivity could lead to a lower thermal gradient in the melt pool. An earlier study [255] has shown that the thermal diffusivity of the Ti-6Al-4V sintered powder bed obtained from SEBM can be doubled by increasing the beam passes. It also suggested that the thermal conductivity of the powder bed is more dependent on the powder bed density than on the diffusivity. Another study [256] has determined that the contact thermal conductivity of a powder bed is directly proportional to the linear dimension of contact between the powder particles. It was also determined that the increasing particle diameter of the powder feedstock along with increasing powder bed density resulted in an increased thermal conductivity [257]. However, the presence of smaller particles in the powder bed has a lesser effect on the resultant thermal conductivity than larger ones.

Thus, the next part of this research on the fabrication of SS316L part investigates the effect of powder size distribution on the thermophysical properties of the powder bed. Since, these properties determine the melt pool topology and the solidification parameters, a substantial change in the resultant microstructure should be eminent.

Two batches of powder feedstock – fine and coarse powder were utilised for fabrication of block parts from SEBM (details provided in Chapter 3). Figure 5-10 shows the temperature measurements obtained by the thermocouple below the start plate for the two powder batches (referred hereafter as fine and coarse powder). During SEBM fabrication process, these measurements are vital for successful build since the temperature recorded here is used as an input by the EBM system for determination of various points in the process such as the starting temperature for the actual printing process. However, these measurements can prove beneficial when attempting to understand the real-time heat transfer trends occurring during the build.

Figure 5-10 Thermal profiles obtained from the thermocouple located below the base plate

There are a few key points to be focussed on in the plots shown above. Point 1 is when the heating of the base plate is initiated, and a fast, defocussed beam scans the base plate in a

predefined fashion. Once the specified start plate temperature is reached at point 2, 850 °C or 1123.15 K in this case, the build table moves down by the height of one layer, 50 μm , and rake system spreads a layer of powder to be sintered and melted. This is where the temperature plot gets interesting. Ideally, once the new layer of powder is spread, the temperature of the thermocouple decreases for a few layers since the thermal conductivity of the sintered powder bed is lower than that of the bulk base plate due to thermal contact resistance. This fall of temperature goes on until the powder bed gets sintered enough or the melting of the parts is long enough for the majority of the heat conduction to happen along the direction of the thermocouple. This is when the temperature begins to rise with each layer until a steady-state is achieved. As seen in Figure 5-10, this behaviour is observed in the thermal history plot for the fine powder. Whereas for coarse powder, the temperature rose above the melting initiation temperature and kept on increasing for a long time before a steady-state temperature could be achieved. This is an indication that the thermal conductivity of the powder bed was, in fact, sufficient for a large amount of heat transfer during the printing process. To confirm this speculation, the thermal conductivities of fine and coarse sintered powder bed were measured using LFA.

Figure 5-11 showcases the size distribution in the sintered fine and coarse powder bed samples which were utilized for LFA measurements. The SEM micrographs of fine (Figure 5-11(a)) and coarse (Figure 5-11(b)) powders show the visual dissimilarities between the two. As seen here, the fine powder particles mostly merged as a lower amount of energy to heat or even melt them. However, there is a lack of interconnectivity between the particles, as seen here. The coarse powders, as seen in Figure 5-11(b), are still quite large in comparison to the fine powders with a large number of small satellites all around their periphery. This should improve the connectivity between the particles resulting in a better apparent density and, consequently, higher thermal conductivity. Figure 5-11(c) and (d) are the powder size distribution curves obtained for the two batches of powder before and after sintering, respectively. As seen here, before sintering, the minimum powder size of the fine powder was $\sim 5 \mu\text{m}$, and the maximum powder size was $\sim 140 \mu\text{m}$. However, after the sintering process, a significant reduction is observed in the maximum measured size of fine powder particles to $\sim 70 \mu\text{m}$. Whereas, there is negligible change in the particle size of the coarse powder due to sintering. As observed from Figure 5-11(c) and (d), the coarse powder batch retained its maximum powder size as $\sim 175 \mu\text{m}$. This should imply that the coarse powder should possess better thermophysical properties, as proved in earlier research [257].

Figure 5-11 Size distribution in the sintered powder bed. (a) and (b) are the SEM micrographs of fine and coarse powders, respectively. (c) The size distribution of the loose powder obtained from Mastersizer 3000. (d) The size distribution of the sintered powder bed obtained using image analysis tool ImageJ

Thermal diffusivity measurements of both the sintered powder bed and SEBM-built SS316L samples from the two powder batches were carried out using LFA. These measurements are shown in Figure 5-12. As seen here, there is no observable difference between the thermal diffusivities of the fine and coarse as-built samples. Both of these samples show a trend of increasing thermal diffusivity with temperature.

On the other hand, significant differences are observed between the fine and coarse sintered powder bed samples. The room temperature thermal diffusivity of coarse powder bed is nearly twice than that of the fine powder bed. The difference between the two values increases gradually with temperature until ~ 975 K. After this temperature, a sudden dip and a sudden rise are observed in the diffusivity values of coarse and fine sintered powder bed respectively. Ideally, the differences between the thermophysical properties of the two powder beds should diminish gradually with rising temperature and should disappear at the solidus temperature ~ 1658 K. It is noteworthy that the diffusivity values are nearly 10 % of the as-

built samples, implying that the heat flow will be more prominent in as-built regions during printing.

Figure 5-12 Thermal diffusivity measurements of (a) SEBM-built SS316L and (b) sintered powder bed prepared from fine and coarse powder batches

It is well known that the thermal diffusivity in m^2/s is given by the formula:

$$\alpha = \frac{k}{\rho C_p} \quad (5-3)$$

Where k is the thermal conductivity in $\text{W}/\text{m}\cdot\text{K}$, ρ is the density in kg/m^3 and C_p is the specific heat capacity in $\text{J}/\text{kg}\cdot\text{K}$. Thus, in order to determine the thermal conductivity of the powder bed, specific capacity and density measurements are required as well.

To this cause, the specific heat capacities of both the fine and coarse sintered powder bed were measured using Differential Scanning Calorimetry (DSC). Due to the limitations of the testing system, measurements were only possible at temperatures up to 450°C . Thus, specific heat capacities of the two samples were measured at room temperature (avg. value of 5°C to 35°C) and high temperature (avg. value of 430°C to 460°C) and plotted as shown in Figure 5-13. The function to determine C_p value for bulk SS316L was taken from the literature [232] and plotted against temperature, named wrought, as shown in Figure 5-13. This variation is given as:

$$C_p = 412 + 0.2T - 8 \times 10^{-5}T^2 \quad (5-4)$$

Figure 5-13 C_p for coarse and fine powder bed (wrought information from [232])

In order to plot the C_p variation with temperature for fine and coarse powder beds, an assumption was made that these values approach that of the bulk SS316L at the solidus temperature. This assumption assisted in a parabolic fitting of the data to result in the plots shown in Figure 5-13. The fitting function for the two powder beds can be written as:

For coarse powder bed:

$$C_{p/c} = 441.11 + 0.22T - 1.04 \times 10^{-4}T^2 \quad (5-5)$$

For fine powder:

$$C_{p/f} = 413.15 + 0.3T - 1.4 \times 10^{-4}T^2 \quad (5-6)$$

Determination of the density of a powder bed can be challenging due to its porous structure. Hence, Micro-Computed Tomography (micro-CT) was employed to compute the densities of the sintered powder beds. Figure 5-14(a) and (b) are the volumetric renderings of fine and coarse sintered powder beds, respectively, reconstructed from the micro-CT scans. As

seen in the magnified views associated with each of the 3D renderings, substantial necking between the powder particles had occurred due to the high sintering temperature ($\sim 850^{\circ}\text{C}$) utilized during the SEBM process.

Figure 5-14 Representative 3D rendering of (a) Fine and (b) Coarse sintered powder beds analysed using micro-CT scanning

Packing density values of 76.9 % and 80.7 % were obtained for the fine and coarse powder beds, respectively, from the micro-CT measurements. Hence, the apparent density of the powder beds at 298.15 K can be estimated as 6148.2 kg/m^3 and 6452 kg/m^3 for fine and coarse powders, respectively (Density of bulk SS316L at 298.15 K taken as 7995 kg/m^3 [233]). Among the two, coarse powder bed possess a higher packing density which can be attributed to a broader particle size distribution, wherein, smaller particles fill the interparticle voids created by large-sized particles resulting in a higher packing of the powder bed. It is noteworthy that the packing density measured for both fine and coarse sintered powder beds is much higher than that reported in the literature, for non-sintered powder beds [258–261].

The differences in the powder bed density, with particle size distribution and the sintering process, presents an opportunity to devise a framework for varying powder size distribution and extent of sintering. The first step in this direction was taken in this research, that involved simulation of the particulate microstructure by utilizing the powder size distribution obtained using ImageJ for the sintered powder bed shown in Figure 5-11(d). To this cause, an opensource toolbox named Mote3D [262] was utilized for the generation of particulate microstructure. Within this code, the particle overlap factor was varied to effectively simulate a non-sintered powder bed as well as a heavily sintered one. Figure 5-15 shows the simulated fine and coarse powder beds for zero overlaps between neighbouring powder particles. The domain sizes for fine and coarse powder beds were chosen as $100 \text{ }\mu\text{m}$ and $150 \text{ }\mu\text{m}$ respectively. These particulate microstructures were loaded into the FEA software – ABAQUS. Mass of each of these models was calculated by assigning SS316L density to the powder particles and vacuum as the rest of the space. It was then divided by the volume of each of the cubical domains to result in the density of the powder bed.

Figure 5-15 Particulate microstructures obtained for (a) fine powder bed and (b) coarse powder bed using Mote3D [262]

A density value of 2200 kg/m^3 was obtained for fine powder bed and 3200 kg/m^3 obtained for coarse powder. This simulated powder bed density matches well with the literature [257] regarding particle size-density relation. To correlate the simulated powder beds with that obtained from the SEBM process, the overlap factor was increased until the packing densities of the simulate powder beds were similar to that obtained from the micro-CT measurements. Packing densities of 72 % and 77.5 % were obtained for fine and coarse powders, respectively, for an overlap of 38 % between neighbouring particles. This trend is similar to the values obtained from micro-CT measurements with an approximate error of 5 %. Hence, with this technique, it is indeed possible to simulate powder beds subjected to different sintering conditions, for varying powder size distributions.

From the values of α , ρ , and C_p determined by the methods mentioned above, the thermal conductivity of the coarse and fine sintered powder bed was calculated using Eq. (5-3), and was plotted against temperature, as shown in Figure 5-16. A similar assumption, as for the C_p , was made here that the thermal conductivity of the sintered powder bed approaches that of the bulk SS316L at solidus temperature.

Figure 5-16 Thermal conductivity variation of the coarse and fine sintered powder beds

As shown in Figure 5-16, the coarse powder bed possesses a higher value of thermal conductivity in comparison to the fine powder bed in the sintered form. At room temperature, these values were determined as 1.02 W/m·K and 0.43 W/m·K for coarse and fine powder, respectively. At ~ 1100 K, these values changed to 1.65 W/m·K and 1.01 W/m·K, respectively, as shown in the inset in Figure 5-16. This temperature-dependent thermal conductivity data was utilised in the macro-scale numerical simulations.

The thermophysical properties obtained for both coarse and fine powder beds were utilised for the macro-scale numerical modelling of their SEBM fabrication process. It should be noted that, for the automated mode of printing, ARCAM's EBM system employs the calculations of 1D heat transfer, and sets the beam speed and current according to the temperature specified for the build. It was found out, from the build log files, that for the coarse and fine powder, and the same initial process parameters and the desired build temperature of 850 °C, the ARCAM's A2XX software utilised a current value of 12 mA for both coarse and fine powders respectively. However, the EBM control software may have determined that the heat transfer in the coarse powder bed was higher than that in the fine powder bed which was

also evident from their respective thermocouple measurements as shown in Figure 5-10. Hence, towards a steady fabrication process, the final scanning speed set by the EBM control software for the coarse and fine powder batches came out to be 1063 mm/s and 3035 mm/s, respectively. These values were employed for the numerical modelling of the melt pool solidification during SEBM of the respective powder beds.

Figure 5-17 shows the plots of the solidification parameters obtained from the numerical simulation of melting/solidification of the coarse and fine SS316L powder beds. The values plotted were recorded in the melt pool domain at a depth of 70 μm , from the top surface for a single-layer-single-pass melting/solidification. Figure 5-17(a) shows the temperature variation in the coarse and fine powder samples. As shown, during the heating process, the temperature for both the powders goes well above the liquidus temperature of SS316L (1723 K – dashed horizontal line), hence indicative of melting phenomena. Moreover, a higher temperature is achieved in the melt pool of the coarse powder bed due to a slower beam scanning speed. The curve beyond the maximum temperature indicates cooling, which causes the solidification of the melt pool region. The time values when the solidification begins for both the coarse and fine powders were identified for the determination of their respective cooling rate (CR) associated with the solidification process. The CR values were determined by taking the 1st differential of the curve with time when $T = 1723\text{ K}$, which came out as $7.7 \times 10^4\text{ K/s}$ and $1.8 \times 10^5\text{ K/s}$ for coarse and fine powders, respectively. The thermal gradient magnitude curves are shown in Figure 5-17(b) show their variation with time for coarse and fine powders. Like the CR values, the thermal gradient magnitude values were determined as $8.5 \times 10^5\text{ K/m}$ and $4.7 \times 10^6\text{ K/m}$ for coarse and fine powders, respectively, at the advent of solidification.

Figure 5-17 Solidification parameters, at a fixed point in the melt pool, obtained from macro-scale numerical simulations. (a) Temperature variation, and (b) thermal gradient magnitude variation

Since, the point at which the G and CR values were obtained lies at the mid of the melt pool, the velocity of the solidification front, or growth rate, at this point is directed towards the build direction. Its value can be determined by the relation:

$$V = \frac{CR}{G} \quad (5-7)$$

Thus, for coarse and fine powders, the growth rates came out as 0.09 m/s and 0.04 m/s respectively. From these values, the $\frac{G^n}{V}$ parameter was determined for the coarse and fine powder, for $n = 3.4$, as 1.6×10^{21} and 1.3×10^{24} , respectively. A lower value of this parameter for coarse powder samples indicates a stronger tendency of columnar-to-equiaxed transition (CET) in comparison to fine powder samples. Figure 5-18 shows the CET curve for SS316L adapted from [263]. As seen here, the point conforming to the solidification parameters obtained for the coarse powder lies in the equiaxed region while the same in the columnar region for the fine powder. Hence, under the current SEBM processing conditions, it is highly likely that equiaxed microstructure will be observed in the coarse powder samples.

Figure 5-18 CET curve (adapted from [263]) showing the tendency of CET in coarse and fine powder samples with the solidification parameters

The as-built microstructures for both fine and coarse powder bed were analysed using OM imaging. Figure 5-19 presents the optical micrographs, of transverse/longitudinal sections of SEBM-built specimens to provide a visual comparison between the two as-built microstructures. As seen in Figure 5-19(a), the microstructure obtained from the SEBM fabrication of fine powder is primarily columnar while the same for the coarse powder is equiaxed as seen in Figure 5-19(b). Moreover, there is a significant observable difference between the melt pool topologies of the two microstructures as originally predicted from the macro-scale numerical simulations. The melt pool obtained in the fine powder samples appears to be shallower and wider than that of the coarse powder.

Figure 5-19 SEBM-built microstructure using (a) fine powder, and (b) coarse powder.

To confirm the equiaxed grain growth in the coarse powder samples, EBSD mapping of the transverse/longitudinal sections of the specimens from SEBM-built samples was carried out. Figure 5-20 shows the crystallographic orientation maps produced from EBSD scans of fine and coarse powder samples, with their corresponding pole figures. These maps were superimposed with the FESEM micrographs where the dark lines indicate the melt pool

boundaries observed at the regions to put the melt pool size in perspective w.r.t. the grain sizes obtained in the as-built samples.

Figure 5-20 Crystallographic orientation maps obtained for the transverse/longitudinal sections of specimens from (a) fine powder, and (b) coarse powder by utilising the EBSD method

As seen here, there is a legitimate difference between the melt pool topologies observed in the SEBM-built fine and coarse powder samples. The melt pool in the fine powder samples, for instance, appears wider and shallower than that observed in the coarse powder samples. The measurements of the melt pool dimension from the micrographs reveal that average depth of $\sim 110\ \mu\text{m}$ and width of $\sim 410\ \mu\text{m}$ is observed in the fine powder samples. At the same time, an average depth of $\sim 140\ \mu\text{m}$ and width of $\sim 300\ \mu\text{m}$ is observed in the coarse powder samples. As seen in Figure 5-20(a), columnar grain growth resulting in a strong $\langle 001 \rangle$ crystallographic texture prevails in the fine powder specimens. Whereas, Figure 5-20(b) shows that a primarily equiaxed microstructure is observed in the coarse powder samples.

The variation in grain size with Misorientation Threshold (MT) for the two samples, determined from EBSD data is shown in Figure 5-21. It is observed that, for the fine powder measurements, the average grain size depends heavily on the specified MT – ranging from $52\ \mu\text{m}$ for an MT of 0.5° and $85.5\ \mu\text{m}$ for an MT of 15° . The same for the coarse powder measurements, however, varied little from $10.3\ \mu\text{m}$ for an MT of 0.5° and $10.5\ \mu\text{m}$ for an MT of 15° . Grain size values were chosen at the lowest MT value of 0.5° as representatives of the fine and coarse powders. A smaller variation in the grain size with MT for coarse powder samples implies that the resultant microstructure is predominantly composed of High Angle Grain Boundaries (HAGBs).

A smaller average grain size causes grain boundary strengthening by reducing the dislocation pile-ups at the grain boundary (GB), and as a result, should result in better yield strength.

Figure 5-21 Grain width measurements using EBSD analysis for (a) fine powder sample, and (b) coarse powder sample and their variation with grain MT

The aspect ratio of a grain is the ratio of its length and the width. Complimentary to smaller grain size, an aspect ratio close to one is indicative of the homogeneity in microstructure, and implies the extent of isotropy in the mechanical properties. For the fine powder samples, at an MT of 0.5 °, the average aspect ratio was measured as ~ 8 with a maximum value of ~ 21. For the coarse powder samples, the average and maximum values of the aspect ratio was measured as ~ 2 and ~ 8, respectively. Hence, it can be established that the SEBM-built microstructure obtained in coarse powder samples was finer and homogeneous in comparison to that obtained in the fine powder samples. As a result, better isotropic mechanical properties can be expected from the coarse powder samples.

Uniaxial tensile testing was performed on the tensile coupons sectioned from the as-built samples. Figure 5-22 and Figure 5-23 summarise these results with plots showing the engineering stress-strain, true stress-strain and work hardening rate curves. The coupons from each of the fine and coarse powder samples were tested along the horizontal and vertical directions to establish the extent of anisotropy in mechanical properties. As shown here, plots names C and F designate the coarse and fine powder samples, respectively, and H and V designate the horizontal and vertical direction of testing. The green transparent box in Figure 5-22 envelopes the ASTM A240 [115] requirements for UTS and elongation percentage.

Figure 5-22 Engineering stress vs engineering strain curves obtained from the uniaxial tensile testing of coarse and fine powder samples

Figure 5-23 True stress vs true strain and work hardening rate curves obtained from the uniaxial tensile testing of coarse and fine powder samples

As seen in Figure 5-22, the YS for the coarse powder is 310 MPa when tested along the horizontal and vertical directions (CH and CV) showing good isotropy. This value varies from 210 to 316 MPa when the testing direction is changed from horizontal to vertical. This value is well above the YS requirement of the ASTM standard (170 MPa) [115]. Much higher values of UTS and elongation were obtained in the coarse powder samples in comparison to the fine powder samples, which were well above the ASTM standard requirements. Similar observations were made for fracture strains as well. The fracture strain for CH, CV, FV, and FH were measured to be 70 %, 58 %, 36 %, and 33 %, respectively. Overall, ~ 17 % and ~ 62 % improvements are observed in both UTS and elongation % in the coarse powder samples over fine powder ones. The work hardening rate curves presented in Figure 5-23 show that the

work hardening starts at the lower true strain value of ~ 0.017 for coarse powder coupons, and ~ 0.025 and ~ 0.043 for fine powder coupons tested along the vertical and horizontal directions respectively. The reduction in area for fine powder samples was obtained as $\sim 57\%$ while the same for coarse powder samples was obtained as $\sim 70\%$.

The deformation modes during the tensile testing of the samples prepared from the two sets of powder batches were further investigated by observing the fractured samples under FESEM and TEM. The comparative studies were carried out between the coarse and fine powder samples tested along the horizontal direction.

Figure 5-24 summarises the FESEM investigations of the fracture surfaces. Figure 5-24(a) and (b) are the micrographs obtained for the coarse powder samples, and Figure 5-24(c) and (d) were obtained for the fine powder samples. As seen here, the fracture surfaces of the samples are covered in microscopic dimples indicative of a ductile fracture. However, the fracture surface of the coarse powder sample is macroscopically flat (Figure 5-24(a)) and a large amount of dimples is seen under high magnification (Figure 5-24(b)) indicating higher plastic deformability of the samples under tension. Conversely, an uneven fracture surface is observed in the fine powder sample associated with intergranular fracture, which is most likely caused by the sigma-phase precipitates as was reported in an earlier study [13]. The differences in the mechanical properties shown in Figure 5-22 and Figure 5-23, and fracture surfaces observed under FESEM are indicative of different deformation mechanisms predominate in the coarse and fine powder bed samples when subjected to uniaxial tensile testing. Further investigations on the distinct deformation behaviours of these samples were carried out using EBSD and TEM.

Figure 5-24 Fractography of the SEBM-built SS316L samples. FESEM micrographs of the fracture surface obtained from coarse powder, (a) and (b), and fine powder (c) and (d) samples

The EBSD analyses of the deformed region in the fractured samples were carried out to investigate the deformation mechanisms. Figure 5-25 shows the comparative EBSD maps of coarse and fine powder samples. The regions of interest (ROIs) were chosen 3 mm from the fracture surface. The tensile testing direction is shown with the outwards pointing arrows in the plane of the maps. Figure 5-25(a) and (b) are the crystallographic orientation maps obtained in the coarse and fine powder samples, respectively. The colours associated with each grain corresponds to the IPF scale attached to the figures. The $\langle 001 \rangle$ directions of most of the grains in fine powder samples are oriented along the build direction. While, the crystallographic orientations of the grains in the coarse powder samples are random – like the as-built microstructure. A large number of deformation twins (white boundaries showed with blue arrows) are observed in the coarse powder samples, while, within the ROI, twin boundary is rarely seen in the fine powder sample.

Figure 5-25 EBSD analysis of the deformed regions in the fractured (a) coarse powder, and (b) fine powder samples. Black arrows indicate the tensile testing direction

Further, TEM investigations were performed on fractured coarse and fine powder samples to affirm the EBSD observations of deformation modes associated with the two samples. Figure 5-26 shows the Bright Field (BF) TEM images obtained for both of the deformed coarse and fine powder samples with their respective Selective Area Electron Diffraction (SAED) patterns shown as insets. The imaging was carried out along the $\langle 011 \rangle$

zone axis for both the cases. A large number of nano-scale twins (nano twins) marked by red arrows are observed in the BF image of the deformed coarse powder sample, as shown in Figure 5-26(a). The particular SAED pattern shows the numerous streak lines. Moreover, the SAED pattern can be resolved into two distinct patterns mirrored along the $\langle 111 \rangle$ in-plane direction, confirming the presence of deformation twins. The BF image of the deformed fine powder sample, shown in Figure 5-26(b), also contains a few planar defects (red arrows). However, the presence of a large number of dislocations in the region, and lack of twin pattern in the associated SAED pattern confirms these to be Stacking Faults (SF). Hence, it can be confirmed that the deformation in the coarse powder samples under uniaxial tension occurred through twinning. Whereas, the same occurred through dislocation slip in the fine powder samples.

Figure 5-26 (a) A Bright-field TEM image of the deformed coarse powder sample. (b) A bright-field TEM image of the deformed fine powder sample. Both images and their respective SAED patterns were photographed along $\langle 011 \rangle$ zone axis

5.4 Discussion

Obtaining a complete equiaxed microstructure in additively manufactured parts is highly desirable due to its effect on the mechanical properties. Equiaxed grain growth results in a homogenous microstructure and isotropic mechanical properties. However, fabricating sizeable parts with equiaxed microstructure has been a challenging ordeal so far. Earlier works attempted by careful variation of process parameters, the addition of nano-inclusions to initiate homogenous nucleation and post-processing. These processes require a lot of trial and error experiments and might result in an undesired alloy composition.

The study on SEBM fabrication of SS316L alloy carried out in this research has utilised novel solutions such as build geometry modification and powder size variation to achieve a certain level of grain growth control which was hitherto impossible.

A unique X-shaped geometry was improvised to achieve a nearly SX crystallographic texture in as-built SS316L parts, which was confirmed by scans at two different sections, that have not been reported in the literature so far. In the process, this research has pointed out a few novel key points which determine the solidification parameters associated with the melt pool solidification in SEBM-built parts. For instance, Figure 5-7 presents the dependence of the crystallographic texture with the prism angles of X-shaped samples. The presence of a distinct prism angle of 90° for the strongest crystallographic texture implies that the geometry of a part has a significant effect on the solidification parameters associated with a melt pool. Moreover, the most suitable parameters, which would result in a strong crystallographic texture, cannot be obtained by merely choosing the extreme build geometries. To eradicate the need for tedious trial and errors, a suitable alternative of numerical modelling is presented, which can predict the optimal build geometry for achieving the strongest crystallographic texture. As evidenced in Figure 5-8, the thermal gradient magnitude that favours the growth of $\langle 001 \rangle$ oriented grains in the melt pool is highest for a prism angle of 90° . With increasing prism angle, a larger thermal gradient exists at the melt pool boundary near the top surface, which would favour the growth of non- $\langle 001 \rangle$ oriented grains. This study was further progressed to achieve a technical SX microstructure, where the grain misorientation $< 15^\circ$, presented in Figure 5-9.

Another remarkable achievement during the investigation of grain growth behaviour of SEBM-built SS316L, considered in this research, is the tailoring of thermophysical properties of powder bed by varying the powder size distribution. As shown in Figure 5-11, a variation

of powder size distribution from fine to coarse results in a powder bed which possesses a large range of powder particle sizes in the sintered powder bed. Moreover, the particles in the coarse powder bed were enriched with smaller satellite particles which would improve the contact thermal conductivity [257]. Consequently, a higher value of thermal diffusivity and resultant thermal conductivity was measured for coarse powder bed samples which are clearly demonstrated in Figure 5-12 and Figure 5-16. This increased thermal conductivity for coarse powder bed resulted in large heat dissipation to the thermocouple of the ARCAM EBM-system, and it might have forced the control software to utilize a beam scanning speed much lower than that for the fine powder feedstock even for the same initial process parameters. As a result, a fine equiaxed microstructure was obtained in the coarse powder samples as shown in Figure 5-19 and Figure 4-13, that too without any tedious trial and errors or manual process parameter variations. Sizeable parts ranging up to 30 mm cross-sectional dimensions were produced with the same set of process parameters, and no human intervention to prove the repeatability of the technique employed.

Macro-scale numerical simulations, results of which are shown in Figure 5-17, provide a vital inference that, in comparison to the SEBM-fabrication of fine powder samples, smaller thermal gradient and lower cooling rate govern the solidification process in the coarse powder samples. As described in detail in chapter 4, the modified equation to predict the CET in rapid solidification processes [249][250][184] is given by,

$$\frac{G^n}{V} < a \left\{ \sqrt[3]{-\frac{4\pi N_o}{3 \ln[1 - \phi]} \frac{1}{n + 1}} \right\}^n \quad (5-8)$$

The value of parameter ϕ varies from 0 - indicating no stray grains, to 1 - indicating nucleation of equiaxed grains, thus dominating over columnar grains and interrupting epitaxial grain growth during microstructural evolution.

The values of G and V obtained from the macro-scale numerical simulations were plotted in a CET curve for SS316L adapted from literature [263] and depicted in Figure 5-18. The data point confining with the G and V values of coarse powder lies within the equiaxed region, implying that the CET will take place during solidification. Moreover, since the values of the solidification parameters were calculated at a fixed point near the bottom and the centre of the width of the melt pool, a high tendency of CET at this point would imply that the rest of melt pool would undergo CET as well since it has been shown from macro-scale numerical

simulations that the tendency of epitaxial grain growth over CET is the strongest at the bottom of a melt pool.

Due to this CET, the fine equiaxed microstructure was observed in the as-built coarse powder samples with a weak crystallographic texture. The resultant microstructure observed in the EBSD scan shown in Figure 5-20, shows grains with their crystallographic orientations aligned with $\langle 001 \rangle$, $\langle 110 \rangle$, and $\langle 111 \rangle$ directions. The grain growth during microstructural evolution in coarse powder samples is mostly independent of the shape of the melt pool interface. However, some $\langle 110 \rangle$ oriented grains were seen to grow from the melt pool interface confirming that this equiaxed microstructural evolution did not take place from dynamic recrystallisation process, and a classical CET took place during solidification where the nucleating equiaxed grains impede the growth of columnar grains [188].

The effect of the as-built equiaxed microstructure on the mechanical properties is depicted in Figure 5-22 and Figure 5-23. A significant improvement over the mechanical properties obtained in the fine powder samples was the isotropic yield strength value of 310 MPa. Moreover, the plastic deformation curve obtained for the testing along the horizontal and vertical directions of the coarse powder samples remained identical until 45 % elongation at which the vertically tested sample achieved a UTS value of 621 MPa which is 0.6 % lower than that of the horizontal sample. On the one hand, the equiaxed grains in coarse powder samples improved the isotropy in the mechanical properties, on the other, presence of a large number of $\langle 011 \rangle$ oriented grains caused twinning during deformation thus improving the ductility. In fact, a maximum elongation to fracture, of ~ 65 %, and an ultimate elongation of ~ 55 % obtained in coarse powder sample tested along the horizontal direction is far higher than any reported works on the fabrication of SS316L parts using metal AM processes [107–112].

The abundance of deformation twins in the coarse powder samples, even for a finer microstructure, in comparison to the fine powder samples can be attributed to the dominant crystallographic texture of the as-built microstructure. Gutierrez-Urrutia et al. [264] reported, for an Mn-rich Twinning-Induced Plasticity (TWIP) steel, that the refinement of grain from 50 μm to 3 μm did not suppress deformation twinning. They also observed that, at small strain, deformation twinning mainly occurred in grains oriented close to $\langle 111 \rangle$ while the ones oriented along $\langle 001 \rangle$ remained essentially free from deformation twins. This was due to the fact that the maximum resolved shear stress for twinning, τ_{tw} , was larger than that for slip, τ_{s} for $\langle 111 \rangle$ oriented grains. Rüsing et al. [265] made a similar conclusion by investigating the

effect of local texture in a TWIP steel. Shterner et al. [266] reported that among the grain orientations of $\langle 111 \rangle$, $\langle 110 \rangle$, and $\langle 001 \rangle$, grains with their crystallographic orientation of $\langle 111 \rangle$ aligned well with the tensile testing direction depict the maximum tendency for deformation twinning. This effect is least for $\langle 001 \rangle$ oriented grains while $\langle 110 \rangle$ grains show an intermediate effect. These observations are quite similar and relatable to the results obtained in this study. As shown in Figure 5-20, the $\langle 001 \rangle$ is the prevailing crystallographic texture in SEBM-built fine powder samples (Figure 5-20(a)), while the as-built microstructure of the coarse powder samples is diffuse with grains oriented along the three directions of $\langle 111 \rangle$, $\langle 110 \rangle$, and $\langle 001 \rangle$. Due to this, twinning was dominant over slip during the plastic deformation of coarse powder samples resulting in a larger elongation to fracture under tensile loading.

5.5 Summary

The research carried out in this chapter on SEBM fabrication of SS316L alloy has provided an advancement to the state-of-the-art grain growth control during microstructural evolution. The effects of build geometry on the solidification parameters and resultant crystallographic texture were validated by experimental and numerical simulation investigations. The macro-scale 3D model led to an understanding of the effect of part geometry on the melt pool topology and on the solidification parameters – G and CR governing the melt pool solidification process. At the same time, the experimental investigations assisted in the validation of this idea. Consequently, a strong $\langle 001 \rangle$ crystallographic texture was obtained in the SEBM-built SS316L parts which have not been reported in the literature so far.

A significant contribution of this research is the effect of the powder size distribution on the thermophysical properties of the powder bed. This understanding can prove fruitful in the advancement of the powder bed fusion process, and its utilisation can result in the development of new materials – especially ones which possess low thermophysical properties deemed inadequate for metal AM. An equiaxed microstructure was successfully obtained in the SEBM-built part. A significant improvement in the mechanical properties is reported in this chapter for coarse powder samples over fine powder ones. Strong isotropy and improved ductility in the as-built samples form a core result of this chapter and constitute a significant advancement over the state-of-the-art. This technique can prove beneficial in fabricating parts with superior and predictable mechanical properties.

Chapter 6 EBM Fabrication of AM1 Superalloy – On the Nanometer-scale Microstructural Evolution and a Generalized Hot Cracking Criterion

In this research, ARCAM's EBM system was employed for manufacturing a Ni-based single crystal superalloy – AM1. The as-built samples were ground, polished to a mirror finish, and analyzed using optical and electron microscopy. Compositional analysis of the mirror finish specimens was carried out using EDS equipped with the FESEM. The crystallographic texture of as-built specimens was characterized using EBSD analysis. Bulk-phase analysis of the samples was carried out using XRD. TEM and APT were employed for distinguishing nanometer-scale (nano-scale) features of the microstructure. Micro-hardness measurement of the as-built samples was carried out using Vicker's testing method. It was found that fine as-built microstructure with ubiquitous nano-scale γ' precipitation via the spinodal decomposition mechanism is formed. Therefore, an exceptional microhardness of 480.0 ± 6.7 HV at room temperature was obtained for the SEBM-built samples. However, severe intergranular hot cracking and topologically close-packed (TCP) precipitation occurred at the grain boundaries (GBs). Hence, the tendency of cracking and TCP phase precipitation at three different types of GBs - divergent, convergent, and sub-GBs, through advanced characterization techniques and numerical simulations, were systematically examined. A criterion for cracking was proposed to determine the likelihood of a GB to crack for given solidification conditions. Also, the TCP precipitates of the C14 Laves phase in the form of NiTa_2 were unexpectedly observed at the GBs where no cracking occurred. Finally, a range of material properties, solidification parameters, and microstructural features was identified to obtain crack-free samples.

6.1 Introduction

Single crystal (SX) Ni-based superalloys have excellent creep and fracture resistance which makes them ideal for making gas turbine blades and vanes that operate in high-temperature conditions [22]. The exceptional high-temperature mechanical properties of these alloys can be attributed to the precipitation of the L12 ordered γ' phase – a geometrically close-packed (GCP) phase that forms coherently or semi-coherently with the parent face-centred cubic (fcc) γ phase, and the absence of grain boundaries, which if present, would have weakened at higher temperatures [22,125]. To obtain a microstructure free from grain boundaries, directional solidification is employed during casting. It facilitates competitive grain growth, wherein grains that are aligned with the effective thermal gradients – ones having the $\langle 001 \rangle$ crystallographic growth direction, eliminate the misoriented ones. In conventional manufacturing, directional solidification is achieved either by grain selection or SX seeding in a continuous, directional casting process. However, since these processes involve high manufacturing costs, low yield rates, and complex technical control parameters, alternate manufacturing methods were sought. The most popular alternative in this context is additive manufacturing (AM) or three-dimensional (3D) printing technique. Apart from providing a high yield, employing these techniques also eliminates the need to perform post-manufacturing processing as the produced parts can be used directly in the as-built condition.

Amongst the commercial 3D printing techniques, selective electron beam melting (SEBM) has shown significant promise in the fabrication of bulk Ni-based superalloy single crystals as it inherently introduces ultrahigh unidirectional thermal gradients ($\sim 10^6$ K/m) in a high-temperature build environment [35,267,268]. Moreover, employing this technique to make components reduces homogenization and ageing heat treatments due to the as-built fine dendritic microstructure, which is typically associated with the rapid solidification process [16]. Despite these advantages, SEBM-built Ni-based superalloys, which have a high content of Al + Ti (>6.0 wt. %) undergo intergranular cracking along the build direction. Ramsperger and Körner [15], Chauvet et al. [17,152], Barr et al. [144], and Xu et al. [269] tried to alleviate cracking by either increasing the energy density or by increasing the substrate preheat temperatures. Both these strategies lower the cooling rate, which in turn increases the probability of columnar-to-equiaxed transition (CET) [68]. However, the occurrence of CET at the expense of competitive grain growth of columnar grains, which is a necessary condition for eliminating GBs and forming an SX microstructure, is undesirable. Moreover, lower cooling rates promote coarsening of the primary dendrites and compromise the advantages

associated with fine microstructure in the as-built part. Therefore, mitigating cracking, while producing an as-built fine microstructure, using the SEBM technique remains a significant challenge.

Keeping these aspects in context, the motivation of this work is to thoroughly investigate the hot cracking process and microstructural features of a Ni-based superalloy additively manufactured by the SEBM process via experiments and numerical simulations. In particular, hot cracking was analysed from a quantitative and qualitative point of view, and a generalized criterion for cracking was proposed. Factors influencing the cracking process were critically evaluated against this criterion, and the validity of our theoretical modelling was evaluated. Furthermore, the impact of precipitation of TCP phases, which was rarely observed in SEBM-built superalloys, on the mechanical properties and cracking behaviour was also discussed in detail. Based on the results of this study, it is suggested that the mutual misorientation of neighbouring grains during solidification would eventually determine GB's resistance to hot cracking during SEBM process. Finally, measures to inhibit hot cracking in SEBM-built Ni-based superalloy SXs were proposed.

6.2 SEBM fabrication process

The powder bed is first sintered and subsequently melted according to the cross-section of the build file, and then the build platform moves down by a layer height of 50 μm . The sintering parameters were established to maintain a surface temperature of $\sim 950\text{ }^{\circ}\text{C}$. For the sintering (preheating) step of SEBM fabrication of the Ni-based superalloy, a beam speed of 16000 mm/s and a focus offset (FO) of 90 mA were utilized with a high average current of 31 mA to ensure a uniform build temperature of $\sim 950\text{ }^{\circ}\text{C}$. Optimum process parameters for the melting step were determined by the Design of Experiments (DoE) analysis for fabricating square-shaped samples with side 30 mm and height 3 mm each. This sample height was adequate for the feasibility study and for observing the columnar grain growth trend in the superalloy samples while proving economically viable. Figure 6-1 summarizes the DOE study with respect to the two vital and independent process parameters – speed function (SF) and FO, where the former was varied from 50 to 130, and the latter – from 5 to 15 mA. Figure 6-1(a) shows the variation in surface topography of as-built samples with SF and FO. It is shown here that for smaller SF and FO values, severe swelling occurred on the top surface, whereas for large SF and FO values, critical porosity was observed. Figure 6-1(b) tabulates the process parameter combinations for which swelling or porosity was observed – indicated by red and blue colours, respectively. The parameter combinations which have green colours

showed a flat surface – free from swelling or porosity. Among the two parameters investigated, SF was found to be more vital in defining the morphological characteristics of as-built samples. For the SF value of 90, completely dense samples were obtained without any visible morphological deformities, regardless of the FO used. Eventually, SF 90 and FO 11 mA were chosen for the fabrication of Ni-based superalloy samples to be utilized for experimental and numerical simulation investigations.

Figure 6-1. Process parameter optimization for SEBM of Ni-based SX superalloy

6.3 Results

In this section, the examination of the as-built microstructure is presented, which was carried out using various multi-scale characterization techniques. Moreover, a generalized

analytical criterion for cracking has been laid out systematically to account for the solidification parameters, material properties, and microstructural features.

6.3.1 Meso-scale microstructure and cracking in as-built samples

Figure 6-2(a) shows a pseudo-3D representation of the microstructure in the as-built superalloy samples. In these images, three long cracks, two along the build direction and one perpendicular to it are visible. Magnified images of the regions containing cracks in the longitudinal section and cross-section are displayed in Figure 6-2(b) and (c), respectively. From these images, it was determined that all cracks in the sample were intergranular in nature and that they extended from the bottom of the build to the top of it.

Figure 6-2(a) Pseudo-3D optical micrograph of etched as-built superalloy samples showing severe cracking and grain orientation indicated by the yellow arrows. FESEM micrographs taken at the specific sections shown in (a): (b) cross-section and (c) longitudinal section

As seen here, the microstructure surrounding the cracks consists of fine and columnar dendrites, which indicates that the cooling rate was high, and the thermal gradient along the build direction was unidirectional. In general, the GBs of Ni-based superalloys broadly fall into the following three categories – convergent, divergent, and sub (a.k.a. dendritic). This categorization stems from previous studies that examined the effect of GB type, i.e., convergent or divergent, on competitive grain growth during directional solidification processes [270,271].

In a purely definitive sense, a divergent GB is one where the dendrites constituting the grains grow outward from it, whereas in a convergent GB, the inverse is observed, i.e. the dendrites grow inward. This definition can be misleading as two neighbouring grains in 3D can potentially exhibit either converging or diverging grain growth behaviour depending on the chosen 2D plane of reference. Figure 6-3(a) is a 3D illustration, which was simulated using the meso-scale CAFD model, of two neighbouring grains (G1 and G2) that are inclined at 10° to the growth direction (Z).

Figure 6-3. Illustration of two grains inclined at 10° from growth direction. (a) 3D representation of grains during solidification. (b) 2D representation in YZ plane and (c) XZ plane

Figure 6-3(b) and (c) show the sectional view of the GB between G1 and G2 as observed in YZ and XZ planes, respectively as solidification proceeds. In the YZ plane, the dendrites within the grains are seen growing towards each other. Thus, the boundary shared by the two grains is designated as a convergent GB. In contrast, the growth directions of dendrites within the grains, in the XZ plane, are nearly parallel to each other, which implies that they share a neutral GB – as it is neither convergent nor divergent. Hereinafter, these terms will be used to describe the nature of grain growth across GBs broadly. Although, strictly speaking, grain growth is a 3D process, for the sake of simplicity and clarity, multi-scale numerical simulations and experiments in this work were conducted and analysed in a 2D frame of reference. However, any reference to the results of these 2D simulations applied for

understanding the nature of grain growth in three dimensions must specify the 2D plane of reference. Based on this terminology, in the present study, a closer look at Figure 6-2(a) and (c) reveals that while a crack is present along a divergent GB, the neutral and convergent GBs appear to be crack free.

In order to further understand the initiation and propagation of cracks, large-scale EBSD scans were conducted on the longitudinal sections of the SEBM-built superalloy samples. Figure 6-4(a) is one such EBSD orientation map of the entire build. From the lower edge of the map, which is close to the base plate to about a height of 200 μm , the microstructure consists of nearly-equiaxed grains. As the build progresses, the grain morphology does not remain equiaxed and transitions to an entirely columnar microstructure. A fine crack initiates at the bottom of the build extends vertically (along the build direction) and passes through the equiaxed region. This crack terminates at the boundary of a sizeable columnar grain with a strong $\langle 001 \rangle$ crystallographic orientation. Near the terminal point of this crack, another crack forms from an adjacent grain boundary and extends to the top of the build. It is worth noting that the grains on either side of these cracks have a substantial difference in their crystallographic orientations, which further confirms that all the cracks in this sample were intergranular. The crack that originates from the bottom of the build terminates at the equiaxed-to-columnar grain transition region (as encircled in black in Figure 6-4(a)). Furthermore, the two correlative SEM images of the cracked regions shown in Figure 6-4(b) and (c), reaffirm that a divergent GB is likely to undergo cracking in the SEBM-built superalloy samples. In addition, a PDAS of $4.0 \pm 0.3 \mu\text{m}$ was obtained with fine precipitates in the interdendritic region and at the GBs (Figure 6-4(b) and (c)). Given that all observed cracks are intergranular in nature, it can be asserted that this alloy does not undergo transgranular fracture during solidification.

Figure 6-4 (a) EBSD map of the transition region of equiaxed to columnar grains. Encircled in black is a region where crack termination occurs due to the presence of a well $\langle 001 \rangle$ oriented grain. (b) and (c) FESEM images showing dendritic microstructure near crack propagation and initiation regions of the crack respectively indicating a presence of divergent GB at the crack

Figure 6-5 is another large-scale EBSD orientation map of a sufficiently dense longitudinal section, $\sim 800 \mu\text{m}$ high and $\sim 1000 \mu\text{m}$ wide, and free from severe cracking (black arrows). As seen here, a predominantly $\langle 001 \rangle$ crystallographic texture prevails in the crack-free regions of the as-built microstructure. It is also apparent that the SEBM-built Ni-based superalloy microstructure is free from any sizable porosity defects. Hence, a small-scale mechanical testing process like micro-hardness measurement should be able to ascertain the mechanical strength of the crack-free microstructure with primarily $\langle 001 \rangle$ crystallographic

texture obtained from the SEBM of AM1 superalloy. The average and maximum grain widths were determined from this map to be $29.1 \pm 3.0 \mu\text{m}$ and $\sim 130.7 \mu\text{m}$, respectively.

Figure 6-5 A large scale EBSD orientation map showing a primarily crack-free region with a predominant $\langle 001 \rangle$ crystallographic texture

6.3.2 Nano-scale microstructure and TCP phase precipitation

The specimen cross-sections were characterized using BSE and XRD. BSE images of the columnar region (upper section of the build) and equiaxed-to-columnar transition region (lower section of the build) in the sample are shown in Figure 6-6(a) and (b), respectively. These two regions have been labelled as Region 1 (R1) and Region 2 (R2), respectively. As can be seen from these images, a large number of precipitates, which have a bright contrast, appear at the equiaxed GBs in R2. Alternately, a significantly lesser number of particles appear at both sub-GBs and columnar GBs in R1. In fact, images obtained from a more comprehensive SEM investigation of the entire build reveals that these precipitates are even fewer at the top of the build. Overall, this gradient in the distribution of the precipitates renders a hierarchical appearance to the overall microstructure.

Figure 6-6 (a) and (b) BSE images of unetched specimens showing abundant bright particles near the bottom layers of as-built samples (Region 2). (c) XRD profiles of the pre-alloyed powder and regions 1 and 2

The XRD scans of these R1 and R2, along with that of the pre-alloyed superalloy powder are displayed in Figure 6-6(c). Since the XRD scan of R1 indicates a single peak, it can be asserted that the majority of the grains in R1 have a strong $\langle 001 \rangle$ crystallographic texture. In contrast, multiple non-indexed peaks (indicated by black arrows in Figure 6-6(c)) are observed in the XRD scan of R2 that are not present in the scan obtained from R1. It is also observed that some of these peaks are not present in the XRD scan of the powder material, which suggests that they correspond to the precipitate phases observed at GBs in R2.

Figure 6-7 shows the hierarchical microstructure observed in the as-built samples. Figure 6-7(a) is a stitched BSE micrograph of a longitudinal section, $\sim 300 \mu\text{m}$ high. As seen here, the fusion region between the stainless steel base plate and deposited Ni-based superalloy is $\sim 50 \mu\text{m}$ high, and thus conforms to the very first melted layer of the SEBM process. This region is likely rich in the constituent elements of both stainless steel and Ni-based superalloy, and hence, acts as a transition between the two. Three regions of interest (ROIs) were chosen in this longitudinal section, and high magnification BSE images were taken to capture the smallest feature distinguishable within the microscope's limit. ROI-1 comprised the transition between the base plate and fusion region, ROI-2 – between the fusion region and deposited Ni-based superalloy, and ROI-3 resides in the deposited Ni-based superalloy region. Figure 6-7(b)

is a high-magnification micrograph of ROI-1, while (c) – (e) of ROI-2, and (f) – (h) of ROI-3, respectively.

Figure 6-7 Hierarchical microstructure observed in as-built Ni-based superalloy fabricated via SEBM.

(a) FESEM-BSE micrograph of longitudinal section $\sim 300\ \mu\text{m}$ high showing three distinct microstructures – stainless steel base plate, fusion layer, and as-built Ni-based superalloy along the build direction. (b) High-magnification micrograph of ROI-1. (c) – (e) High magnification micrographs of ROI-2. (f) – (h) High-magnification micrographs of ROI-3

At first glance, Figure 6-7(a) depicts a large number of bright precipitates in the fusion region in contrast with the rest of the build. A high-magnification micrograph of ROI-1, Figure 6-7(b), reveals an abundant number of these precipitates, which are irregular in shape and distribution, and are $\sim 500\ \text{nm}$ in size. Also, the population density of these precipitates is seen to increase towards the fusion region, implying primarily that the constituent elements of the Ni-based superalloy contribute towards their formation. Moreover, the area surrounding the precipitates is devoid of the primary phase precipitates, which form in Ni-based superalloys subjected to high-temperatures. This indicates that the primary-phase precipitate strengtheners – Al, Ti, and Ta, are the fundamental constituents of the precipitates observed in this region.

Figure 6-7(c) – (e) show the transition from the fusion layer to the deposited Ni-based superalloy region. Figure 6-7(d) shows an area below the boundary in the fusion layer that is free from any secondary phase precipitates. Moreover, the region above the boundary is seen enriched in the bright primary-phase precipitates indicating clearly that the thermal conditions in the region support the precipitate strengthening. A few bright primary phase precipitate were also observed in the as-built superalloy region near the boundary as seen more clearly Figure 6-7(d).

Figure 6-7(f) – (h) are the BSE micrographs of the equiaxed-to-columnar transition region observed universally in the SEBM-built alloys [35]. As seen in Figure 6-7(g) and (h), the entire area is rich in primary phase precipitation which is confined entirely to the grain boundaries. Moreover, their population density of reduces along the build direction as evident from Figure 6-7(f), which can be correlated to the more substantial ageing of the bottom layers due to cyclic reheating. Thus, it is evident that within the as-built superalloy, the hierarchical microstructure is predictable from the thermal history. However, within the fusion layer, the presence of alloying elements from both the base plate and Ni-based superalloy changes the precipitation behaviour and demands further analysis.

Figure 6-8 shows the concentration area maps of key constituting elements of AM1 superalloy obtained in the crack-free regions of unetched specimens using FESEM-EDS. Figure 6-8(a) is the cross-section examination of as-built microstructure near the top surface while Figure 6-8(b) and (c) are the maps which were retrieved at the longitudinal sections at different heights of the same specimen. As evident from the elongated GBs shown in the FESEM micrograph of Figure 6-8(b), the EDS mapping results are from the columnar region of the as-built microstructure. At the same time, Figure 6-8(c) is the result of chemical analysis in the base plate-superalloy fusion layer. In a macro-scale reference, Figure 6-8(b) corresponds to the chemical analysis of ROI-3 in Figure 6-7 while Figure 6-8(c) corresponds to the region between ROI-1 and 2, as indicated in Figure 6-7.

Figure 6-8 Chemical analysis of unetched specimens using FESEM-EDS. (a) Chemical analysis of the cross-section located near the top surface. Chemical analysis of a longitudinal section located in the (b) columnar microstructure region of the specimen and (c) fusion layer of the specimen

As seen in Figure 6-8(a), there is a clear segregation of Ta, W, Mo, and Ti. Since there are no visible precipitates observed in the attached micrograph, the segregation areas represent the GBs. This implication was further verified by the segregation behaviours observed in Figure 6-8(b). However, Cr and Co also show slight segregation quite visibly at the GBs. The EDS mapping in Figure 6-8(c) corroborate that the bright precipitates observed in abundance in the fusion layer are exclusively rich in Ta and W while Mo and Ti are still present, they lack in intensity. Thus it can be reasonably assumed that Ta and W rich precipitates are observed in the fusion region.

Figure 6-9 displays the chemical analysis performed on the longitudinal sections in crack-free regions for an etched specimen. The EDS maps linked to the SEM micrograph show

a large number of primary-phase precipitates (indicated by black arrows in Figure 6-9). As can be seen from the image, precipitation is limited to the GBs and interdendritic regions. Moreover, EDS mapping indicates that the precipitates are rich in Ta, W, and Mo.

Figure 6-9 FESEM micrograph of as-built superalloy sample together with its compositional analyses clearly showing a divergent GB with secondary precipitates observed as bright particles at both divergent and sub-GBs (black arrows). The corresponding elemental maps show obvious segregation of Ta, W, and Mo in the precipitates

To characterize the individual phases in the alloy, TEM investigations were conducted on thin-film specimens extracted from different portions of the build. Figure 6-10 is a bright-field TEM image of the portion extracted from the upper build cross-section of the SEBM as-built sample. In this image, γ' precipitates were identified and labelled within the γ matrix. The corresponding SAED pattern of this image along the $[111]_{\gamma/\gamma'}$ zone axis is shown in the inset of Figure 6-10. Presence of satellite reflections in the SAED pattern (green arrows) indicates precipitation of ordered γ' precipitates from supersaturated γ solid solution [222] via the spinodal decomposition mechanism [272–274]. These γ' precipitates are nearly spherical in shape and have an average size of ~100 nm.

Figure 6-10 Bright-field TEM image showing γ' precipitation through the spinodal decomposition as observed along $[111]_{\gamma/\gamma'}$ zone axis. The corresponding SAED pattern shows satellite reflections associated with the spinodal decomposition (green arrows)

Table 6-1 compiles the compositions of the γ/γ' dual-phase region from Figure 6-10, which was measured using the EDS attached with the TEM. It should be noted that the measured composition of γ and γ' matches closely with the data obtained from previous APT studies on a similar alloy, which was subjected to homogenization, followed by rapid cooling (~ 1273 K/s) [222]. Minor differences in the TEM data recorded in this study with the reference APT data are the result of limited resolution capabilities of TEM. Nevertheless, it is evident that Al, Ti, and Ta are present in the γ' precipitates while rest of the constituent elements are present in the γ matrix.

Table 6-1 Chemical compositions (at. %) of the pre-alloyed superalloy powder and TEM-EDS data recorded at γ matrix, γ' phase, and TCP phase precipitates

	Ni	Cr	Co	Mo	W	Ti	Al	Ta
Powder	65.63	8.55	7.15	1.22	1.41	1.48	12.15	2.41
γ	63.34 $\pm$ 2.68	14.36 $\pm$ 3.54	9.42 $\pm$ 1.14	1.66 $\pm$ 0.71	1.54 $\pm$ 0.21	1.37 $\pm$ 0.37	5.86 $\pm$ 1.56	2.46 $\pm$ 0.69
γ'	66.41 $\pm$ 2.20	6.98 $\pm$ 2.75	6.42 $\pm$ 1.23	0.89 $\pm$ 0.34	1.28 $\pm$ 0.24	2.11 $\pm$ 0.60	12.07 $\pm$ 1.31	3.84 $\pm$ 0.34
Precipitate	11.43 $\pm$ 0.43	3.39 $\pm$ 0.16	1.46 $\pm$ 0.05	3.95 $\pm$ 0.34	3.40 $\pm$ 0.25	25.55 $\pm$ 0.75	1.26 $\pm$ 0.46	49.57 $\pm$ 1.20

TEM was further utilized to analyse the primary phase precipitates in the SEBM-built superalloy samples. Figure 6-11 shows the bright-field TEM images of the GB precipitates and their corresponding SAED patterns from the columnar region of as-built samples. Majority of these precipitates have irregular shapes and are ~200 – 400 nm in size. The SAED patterns of the precipitates are a combination of at least two indexable patterns. Figure 6-11(a) shows that smaller precipitates labelled as 1, 2, and 3 clusters together. The corresponding SAED pattern was indexed along the $[111]\gamma // [1\bar{1}01]C14$ zone axis. Figure 6-11(b) is a magnified image of the precipitate 1 labelled in Figure 6-11(a) with its corresponding SAED pattern indexed along the $[211]\gamma // [11\bar{2}3]C14$ zone axis. Similarly, Figure 6-11(c) is a TEM bright-field image of another 400 nm-sized cuboidal precipitate with its corresponding SAED pattern along the $[\bar{5}504]C14$ zone axis.

Figure 6-11 Bright-field TEM images showing TCP phase precipitation in as-built samples with their characteristic SAED patterns. (a) Agglomeration of 3 irregular C14 Laves TCP phase precipitates observed along $[111]_{\gamma}$ zone axis. (b) A magnified view of precipitate 1 from (a) as seen along $[211]_{\gamma}$ zone axis. (c) A cuboidal shaped C14 Laves TCP phase precipitate as seen along $[\bar{5}504]_{C_{14}}$ zone axis

Based on these reflections, it can be inferred that the secondary precipitates present at GBs are C14 Laves phase that has an HCP structure. Several previous studies on Ni-based superalloys have also reported the formation of these phases, and have referred to them as TCP precipitates [120,121,123,124,275–278]. These precipitates are generally irregular in shape and form when the alloy is aged at high temperatures for extended periods [278]. Note that during the SEBM manufacturing process, each layer of deposited material is subjected to a reheating cycle when another layer is built over it. Hence the material close to the base plate (or substrate plate) is exposed to higher temperatures for more extended periods of time than that of the layers deposited over them. This is why a higher volume fraction of TCP precipitates are present at the bottom of the build (R2 in Figure 6-6(b)). The process of reheating is also responsible for the formation of symmetric and regular shaped precipitates (see Figure 6-11(c)) as it promotes solid-solid phase transformation mechanisms. It is however interesting to note that the top part of the build, which does not experience as much prolonged heating, also contains some of these TCP precipitates. Therefore, it is plausible that TCP precipitation also occurred during the terminal stages of solidification.

From the elemental compositions of the precipitates listed in Table 6-1, it is evident that they are rich in Ta (~ 50 at. %) and Ti (~25 at. %). Since the former has a higher concentration than the latter, it is more likely to combine with Ni and form the MgZn₂ prototype of C14 Laves phase. Therefore, the stoichiometric formula of the TCP phase should be NiTa₂. The composition and precipitation mechanism of these TCP phases is summarized as follows. During dendritic solidification of Ni-based superalloys, the process of solute enrichment leads to variation in partition coefficients of refractory elements. When these solute rich regions are exposed to higher temperatures for longer durations, which happens during ageing or annealing or--as observed in this study--reheating during SEBM process, TCP phases are formed [120,121,123,124,275–278]. It is believed that the presence of Laves based TCP phases in as-built specimens is the outcome of the inherent non-equilibrium nature of solidification in AM processes. Non-equilibrium solidification facilitates the micro-segregation of heavy elements such as Ta and W, which in turn, favours the formation of Laves based TCP phases in the Ni-based SX superalloy considered in the present study [279].

For further quantitative analysis of the composition of the nano-scale γ/γ' dual-phase microstructure and the secondary-phase precipitates, APT was utilized. Figure 6-12(a) shows a FESEM micrograph with two ROIs – labelled 1 and 2, respectively. ROI-1 is located at the interface between the stainless steel base plate and the deposited Ni-based superalloy, and

represents the 1st printed layer of the build. A higher magnification image of this region in the BSE mode of the FESEM reveals the presence of several bright TCP phases at the GBs and within the grains. ROI-2 is located a few layers above ROI-1 in the equiaxed-to-columnar transition region. In the γ/γ' dual-phase microstructure, Al and Cr undergo reverse partitioning, and the former preferentially partitions to γ' precipitates, while the latter is more abundant in the γ matrix, respectively. Hence, these two elements were taken as a reference to indicate the presence of γ/γ' phases in the APT reconstructed volume (RVs). Figure 6-12(b) and (c) show the APT images of two RVs that are 5 μm apart within ROI-1. These are labelled as RV-1.1 and RV-1.2. The RV obtained from APT of ROI-2 is referred to as RV-2 and is shown in Figure 6-12(d). Concentration values of 10 at. % and 15 at. % Cr were chosen to determine the isoconcentration surfaces (isosurfaces) for RV-1.1 and RV-2 respectively as they were the approximate averages of Cr concentration in γ matrix and γ' precipitate phase obtained from the proxigrams and 1D concentration profiles. While, several 30 nm-sized γ' precipitates were found embedded within the γ matrix in RV-1.1, RV-1.2 appears to be homogeneous, and Cr is uniformly distributed in it. RV-2 contains a 100 nm-sized γ' precipitate, which is surrounded by the γ matrix. Two curved γ/γ' interfaces are observed on the top and bottom edge of RV-2, which indicates that the γ' precipitate has a near-spherical morphology. This observation is in good agreement with the TEM image shown in Figure 6-10. Tan et al. [222] determined that employing a high cooling rate (~ 1273 K/s) during solution heat-treatment of Ni-based AM1 SX superalloy results in precipitation of 30-50 nm-sized, spherical γ' precipitates with diffused γ/γ' interfaces. On the contrary, solutionized alloy subjected to a lower cooling rate (~ 274.25 K/s) contained 70-100 nm-sized γ' precipitates with well-defined γ/γ' interfaces. Hence, the differences in precipitation behaviours observed in RV-1.1 and RV-2 infer that the thermal history of deposited layers in AM closely resembles that of a solution heat-treated superalloy. Since the γ' precipitates in RV-1.1 are much smaller than that in RV-2, it can be asserted that ROI-1 was subjected to a higher cooling rate than ROI-2 during AM processes.

Figure 6-12 APT of SEBM-built Ni-based SX superalloy. (a) Micrograph indicating the two ROIs chosen for APT analyses. (b) APT reconstructed volume showing the γ' precipitation in the fusion layer between stainless steel base plate and SEBM-built superalloy. (c) The APT reconstructed volume of the bright intermetallic phase observed in abundance at the GBs in the fusion layer. (d) APT reconstructed volume showing the γ' precipitation in the equiaxed-to-columnar transition region in pure SEBM-built superalloy

Figure 6-13 presents a quantitative compositional analysis of RV-1.1 with 10 at. % Cr isosurfaces. The γ' precipitates, which are reconstructed as pink coloured spheres, are seen being rich in Ni, Al, Ti, and Ta whereas Cr, Mo, and Co partition to the γ matrix. Alternately, W is distributed more uniformly across the γ/γ' dual-phase region. It should also be noted that some constituent elements from the stainless steel base plate, such as Fe, Si, P, and Mn, partitioned only to the γ matrix, and thus did not contribute to the primary phase precipitation.

Figure 6-13 (a) RV-1.1 showing spherical γ' precipitates ~20 nm in size. (b) Proximity histograms (proxigrams) showing the concentration profiles of the alloying elements across the γ/γ' interface

The quantitative compositional analysis of RV-1.2 is presented in Table 6-2. Since it was evident from Figure 6-12(c) that unlike RV-1.1, there are no γ' precipitates in RV-1.2. Tentatively, this suggests that this RV may exclusively contain the γ matrix. However, its compositional analysis, shown in Table 6-2, reveals that it is rich in Ta (13.99 at. %), Mo (9.73 at. %), and W (6.28 at. %).

Table 6-2 Chemical compositions (at. %) of the γ/γ' dual-phase microstructure and TCP phase precipitates measured by APT in the ROI-1 (RV-1.1 and RV-1.2) and ROI-2 (RV-2) along with their comparison with the chemical composition of the γ/γ' dual-phase microstructure observed in solutionized AM1 superalloy subjected to high cooling (HC) and low cooling (LC) [222]

	Ni	Cr	Co	Mo	W	Ti	Al	Ta
RV-1.1								
γ (APT)	32.98 $\pm$ 0.18	18.81 $\pm$ 0.15	4.35 $\pm$ 0.08	1.25 $\pm$ 0.043	0.90 $\pm$ 0.04	0.45 $\pm$ 0.03	3.92 $\pm$ 0.08	0.16 $\pm$ 0.02
γ' (APT)	67.35 $\pm$ 0.44	0.99 $\pm$ 0.09	1.20 $\pm$ 0.10	0.77 $\pm$ 0.08	0.83 $\pm$ 0.08	3.63 $\pm$ 0.17	17.44 $\pm$ 0.35	3.50 $\pm$ 0.17
HC								
γ (APT) [222]	59.13 $\pm$ 0.10	19.03 $\pm$ 0.08	10.54 $\pm$ 0.06	2.41 $\pm$ 0.03	1.85 $\pm$ 0.03	0.38 $\pm$ 0.01	5.57 $\pm$ 0.05	1.09 $\pm$ 0.02
γ' (APT) [222]	69.34 $\pm$ 0.24	2.92 $\pm$ 0.14	4.78 $\pm$ 0.07	0.62 $\pm$ 0.04	0.86 $\pm$ 0.05	1.95 $\pm$ 0.03	15.38 $\pm$ 0.12	4.14 $\pm$ 0.06
RV-1.2								
Precipitate	15.52	14.17	4.58	9.73	6.28	1.81	0.29	13.99
RV-2								
γ (APT)	51.25 $\pm$ 0.26	26.54 $\pm$ 0.23	16.52 $\pm$ 0.19	2.69 $\pm$ 0.06	1.38 $\pm$ 0.07	0.14 $\pm$ 0.03	1.4 $\pm$ 0.08	0.06 $\pm$ 0.03
γ' (APT)	69.82 $\pm$ 0.35	2.64 $\pm$ 0.10	4.14 $\pm$ 0.18	0.87 $\pm$ 0.05	1.73 $\pm$ 0.10	2.25 $\pm$ 0.10	15.06 $\pm$ 0.28	3.28 $\pm$ 0.13
LC								
γ (APT) [222]	50.86 $\pm$ 0.55	26.84 $\pm$ 0.69	14.91 $\pm$ 0.20	2.94 $\pm$ 0.19	1.69 $\pm$ 0.11	0.07 $\pm$ 0.02	2.23 $\pm$ 0.02	0.46 $\pm$ 0.01
γ' (APT) [222]	70.37 $\pm$ 0.10	1.89 $\pm$ 0.07	3.97 $\pm$ 0.07	0.74 $\pm$ 0.01	1.04 $\pm$ 0.05	1.89 $\pm$ 0.04	16.18 $\pm$ 0.07	3.92 $\pm$ 0.07

Since the corresponding concentrations of Ta, Mo, and W in the γ matrix of RV-1.1 is much lower, this region does not correspond to γ matrix. Moreover, the possibility that these elements originated from the base plate can be ruled out as they were not present in the stainless steel plate used in our study. Note that the elemental composition of RV-1.2 matches well with that of the TCP phase, which was measured using TEM-EDS (Table 6-1). The partitioning behaviour of Cr, Co, Mo, W, and Ta in RV-1.2 is also similar to that observed in the C14 Laves based TCP phase. Based on these observations, it is confirmed that RV-1.2 contains one of many TCP precipitates observed in the FESEM images of ROI-1 (See Figure 6-12(a)).

Figure 6-14(a) displays the APT image of RV-2 with 15 at. % Cr isosurfaces. Figure 6-14(b) shows the ROI cylinder for which 1D concentration profiles were plotted and displayed in Figure 6-14(c). The concentration profiles, shown in Figure 6-14(c), indicate that while the γ' precipitate is rich in Ni, Al, Ti, and Ta, the γ matrix is nearly devoid of Ta and Ti.

Figure 6-14 (a) RV-2 showing a γ' precipitate ~ 100 nm in size with distinct nearly parallel γ/γ' interfaces. (b) ROI cylinder for which the 1D concentration profiles are generated. (c) 1D concentration profiles of the alloying elements across the ROI cylinder

Much like what was seen earlier in RV-1.1, W partitions uniformly across the γ and γ' phases. Overall, it was determined that the microstructure is composed of nano-scale γ' embedded in the γ matrix. Micro-hardness measurements in the XY-plane of as-built samples in these regions yield a hardness value of 480.0 ± 6.7 HV or 4.7 ± 0.1 GPa. This implies that the nano-scale γ' precipitates impart superior mechanical properties to the SEBM built SX Ni-based superalloy, and are hence highly desirable.

6.3.3 A generalized criterion for cracking

The term hot cracking broadly refers to cracking of materials during manufacturing processes such as casting, welding, forging, as well as AM [135–137,144,280]. For a detailed investigation of cracking observed in as-built samples, the inner surfaces of the cracks were observed under FESEM. Figure 6-15(a) and (b) display images of two adjacent crack faces observed in the AM sample. Both faces contain smooth, clear dendritic arms inherent to the sub-grains or columnar dendrites. In Figure 6-15(b), a region on the crack face, which does not possess the distinct dendritic pattern as observed elsewhere on the micrograph, is highlighted by enclosing it within a white dotted rectangle. It is proposed, based on prior studies, that this region contains rapidly solidified liquid film that filled in the gaps between grains during the terminal stage of solidification [281]. The presence of such features implies that cracking occurred at the terminal stage of solidification, and not during the subsequent cooling stage, thus confirming this as a case of solidification cracking [138,140,152,282]. Figure 6-15(c) contains the FESEM-EDS maps of Figure 6-15(b). With the exception of Al and Mo, the elemental distribution was not resolved accurately, due to the unevenness of the crack surfaces.

Figure 6-15 (a) and (b) Crack surfaces showing smooth dendritic microstructure. (c) SEM-EDS mapping of the suspected liquid film region in (b)

Kou [145] proposed a criterion for cracking during solidification. The mathematical formulation of this criterion is,

$$\left\{ \frac{d\varepsilon_{local}}{dT} > \sqrt{1-\beta} \cdot \frac{d\sqrt{f_s}}{dT} + \frac{1}{(dT/dt)} \cdot \frac{d}{dz} [(1 - \sqrt{1-\beta} \cdot \sqrt{f_s}) \cdot V_z] \right\}_{\sqrt{f_s} \rightarrow 1} \quad (6-1)$$

Where, $d\varepsilon_{local}/dT$ in the left-hand side (L.H.S) is the local separation term that represents cracking due to solidification shrinkage and thermal contraction. T is temperature and ε_{local} is the local strain. Since fracture of ductile solids is governed by a strain-based fracture criterion, ε_{local} is an appropriate parameter for describing the onset of cracking [283]. The first term in the right-hand side (R.H.S) is the growth term, which accounts for the growth of neighbouring grains toward each other. f_s is the fraction of solid in the semisolid zone, and β is the solidification shrinkage. The second term in the R.H.S is referred to as the *feeding* term, which accounts for the molten liquid being fed to the mushy zone between dendrites. A higher feeding rate of the liquid typically enhances the resistance to cracking. Here, t is time and V_z is the velocity of liquid being fed to the mushy zone along the growth direction, z . The local separation term in Eq. (5-1) consists of the local strain, $\varepsilon_{local} = D_{local}/\varphi$, where D_{local} is the local deformation and φ is the grain width. When the local separation term (L.H.S) exceeds the sum of the growth term and the feeding term (R.H.S), at $(f_s)^{1/2} = 1$, cracking is expected to occur. It is to be noted that when the value of $|dT/d\sqrt{f_s}|$ is high for a particular GB, the growth term becomes lower in magnitude. This implies that a GB is more susceptible to cracking when $d\varepsilon_{local}/dT$ at which cracking initiates also becomes lower. Since $|dT/d\sqrt{f_s}|$ is a quantitative indicator of the susceptibility to cracking, Kou defined it as the cracking index [145]. This index correlates the resistance to cracking of a GB with the dendritic coalescence between two neighbouring grains at the GB and the length of the liquid channel for feeding. A high value of $|dT/d\sqrt{f_s}|$ at $(f_s)^{1/2} = 1$ physically signifies that grains will have a lower tendency to grow towards each other, and that a longer liquid channel needs to be fed with liquid is required to prevent cracking.

Although this criterion provides valuable quantitative insight on cracking behaviour during solidification, which had hitherto been an intractable problem in the field of solidification processing, it has a glaring limitation. The formulation of this criterion implicitly assumes that the columnar GB is parallel to the grains associated with the GB. This means that it can only be applied to manufacturing processes such as continuous casting, where the as-processed microstructure satisfies this condition. In contrast, the evolution of SEBM-built

microstructures depends mainly on the competition of grain growth between adjacent misoriented grains. As shown earlier in Figure 6-4(b) and (c), the mutual inclination of two grains neighbouring the crack, within the plane, was observed to be as much as 12° and 16° respectively (black arrows indicate the grain growth direction). Hence, to generalize the application of Kou's original criterion to AM processes, it was necessary to incorporate the mutual angle of inclination between two grains as an additional parameter.

Figure 6-16 is a schematic of the growth of two misaligned grains during solidification. Figure 6-16(a) shows an illustration of two neighbouring grains with grain 2 inclined at an angle of θ to grain 1 that is oriented along the growth direction. Figure 6-16(b) is the cross-section of the two grains at a height z . It is assumed that, in fully solidified states, grains 1 and 2 have an equal grain width, φ . Furthermore, it is also assumed that the morphology of grain 1 transitions from circular to hexagonal during solidification [145]. To introduce an angular component, grain 2 is assumed to undergo a morphological transition from that of an ellipse to a deformed hexagon whose two sides are longer, as shown in Figure 6-16(c). In this process, the half-size of grain increases from $l' = l/\cos\theta$ to $L' = \varphi/2\cos\theta + z\tan\theta$, where l is the initial characteristic radius of grain 1. Figure 6-16(d) is schematic of a liquid channel region through which feeding occurs at a height z . $\emptyset$ is the width of the region at height z .

Figure 6-16 Schematic of grain growth during solidification (a) in a 2D plane along the build direction, (b) cross-section in XY-plane at a particular height z . (c) Grain cross-section at the terminal stage of solidification and (d) Liquid channel at the grain- boundary during solidification

The systematic derivation of the modified cracking criterion, which considers the inclination between grains, θ (where, $-90^\circ < \theta < 90^\circ$), is as follows:

The areas of circular and elliptical grains in their initial states are given as:

Cross-sectional area of a circle,

$$a_c = \pi l^2 \quad (6-2)$$

Cross-sectional area of an ellipse,

$$a_e = \frac{\pi l^2}{\cos \theta} \quad (6-3)$$

The effective volume fraction of solid is taken as-is from the literature [145] as:

$$f_{se} = \alpha + (1 - \alpha)f_s \quad (6-4)$$

Where, f_{se} is the effective volume fraction of solid at z and α is the ratio of volume liquid fraction within the grain with the total volume liquid fraction.

At the terminal stage of solidification,

$$[f_{se}]_{f_s \rightarrow 1} \cong [f_s]_{f_s \rightarrow 1} \quad (6-5)$$

The effective volume fraction for each grain at a given z can also be given as the ratio of the initial circular cross-sectional area of the grain, a , and the final hexagonal cross-sectional area, A .

For grain 1:

$$a_1 = \pi l^2 \quad (6-6)$$

$$A_1 = \frac{\sqrt{3}\phi^2}{2} \quad (6-7)$$

Hence,

$$f_{se_1} = \frac{\pi l^2}{\frac{\sqrt{3}\phi^2}{2}} \quad (6-8)$$

For grain 2:

$$a_2 = \frac{\pi l^2}{\cos \theta} \quad (6-9)$$

$$A_2 = \frac{\sqrt{3}\phi^2}{2\cos \theta} + \frac{\phi}{\sqrt{3}} \times z \tan \theta \quad (6-10)$$

Hence,

$$f_{se_2} = \frac{\sqrt{3}\pi l^2}{\frac{3\phi^2}{2} + \phi \times z \sin \theta} \quad (6-11)$$

Figure 6-16(c) shows the final geometry of the grains during the terminal stage of solidification. A schematic of the region of the liquid channel through which feeding occurs is shown in Figure 6-16(d), the width of which at height z is given as:

$$\phi = \left(\frac{\varphi}{2} - l'\right) + \left(\frac{\varphi}{2\cos\theta} - \frac{l'}{\cos\theta}\right); \quad (6-12)$$

$$\phi = \left(1 + \frac{1}{\cos\theta}\right) \cdot \left(\frac{\varphi}{2} - l'\right); \quad (6-13)$$

Now, the modified criterion can be written as:

$$\begin{aligned} w \cdot [V_{local} \cdot \Delta z] - w \cdot \Delta z \cdot \left[\frac{dl'}{dt} + \frac{1}{\cos\theta} \cdot \frac{dl'}{dt} + \tan\theta \cdot \frac{dz}{dt} \right] \\ > \\ w \cdot \left[\left(\frac{\varphi}{2} - l'\right) \cdot \left(1 + \frac{1}{\cos\theta}\right) \cdot V_z \right]_{z+\Delta z} - w \cdot \left[\left(\frac{\varphi}{2} - l'\right) \cdot \left(1 + \frac{1}{\cos\theta}\right) \cdot V_z \right]_z; \end{aligned} \quad (6-14)$$

$$\begin{aligned} V_{local} - \left[\frac{dl'}{dt} \cdot \left(1 + \frac{1}{\cos\theta}\right) + \tan\theta \cdot \frac{dz}{dt} \right] \\ > \\ \frac{1}{\Delta z} \cdot \left[\left(\frac{\varphi}{2} - l'\right) \cdot \left(1 + \frac{1}{\cos\theta}\right) \cdot V_z \right]_{z+\Delta z} - \frac{1}{\Delta z} \cdot \left[\left(\frac{\varphi}{2} - l'\right) \cdot \left(1 + \frac{1}{\cos\theta}\right) \cdot V_z \right]_z; \end{aligned} \quad (6-15)$$

$$V_{local} - \left[\frac{dl'}{dt} \cdot \left(1 + \frac{1}{\cos\theta}\right) + \tan\theta \cdot \frac{dz}{dt} \right] > \frac{d}{dz} \left[\left(\frac{\varphi}{2} - l'\right) \cdot \left(1 + \frac{1}{\cos\theta}\right) \cdot V_z \right]_z; \quad (6-16)$$

$$V_{local} > \left[\frac{dl'}{dt} \cdot \left(1 + \frac{1}{\cos\theta}\right) + \tan\theta \cdot \frac{dz}{dt} \right] + \frac{d}{dz} \left[\left(\frac{\varphi}{2} - l'\right) \cdot \left(1 + \frac{1}{\cos\theta}\right) \cdot V_z \right]_z \quad (6-17)$$

From Kou's criterion [145]:

$$l' = \frac{\varphi}{2} \sqrt{1 - \beta} \cdot \sqrt{f_{se}}; \quad (6-18)$$

$$\frac{dl'}{dt} = \frac{\varphi}{2} \sqrt{1 - \beta} \cdot \frac{d\sqrt{f_{se}}}{dT} \cdot \frac{dT}{dt} \quad (6-19)$$

By substituting Eq. (6-19) into Eq. (6-17), it can be rewritten as:

$$V_{local} > \left[\frac{\varphi}{2} \sqrt{1-\beta} \cdot \frac{d\sqrt{f_{se}}}{dT} \cdot \frac{dT}{dt} \cdot \left(1 + \frac{1}{\cos\theta}\right) + \tan\theta \cdot \frac{dz}{dt} \right] + \frac{d}{dz} \left[\frac{\varphi}{2} \cdot (1 - \sqrt{1-\beta} \cdot \sqrt{f_{se}}) \cdot \left(1 + \frac{1}{\cos\theta}\right) \cdot V_z \right]_z ; \quad (6-20)$$

Since, $\frac{d\sqrt{f_{se}}}{dT}$ and $\frac{dT}{dt}$ are inherently negative for solidification, they can be rewritten as $-\left|\frac{d\sqrt{f_{se}}}{dT}\right|$ and $-\left|\frac{dT}{dt}\right|$ respectively. Hence, Eq. becomes:

$$V_{local} > \left[\frac{\varphi}{2} \sqrt{1-\beta} \cdot \left|\frac{d\sqrt{f_{se}}}{dT}\right| \cdot \left(1 + \frac{1}{\cos\theta}\right) \cdot \left|\frac{dT}{dt}\right| + \tan\theta \cdot R \right] + \frac{d}{dz} \left[\frac{\varphi}{2} (1 - \sqrt{1-\beta} \cdot \sqrt{f_{se}}) \cdot \left(1 + \frac{1}{\cos\theta}\right) \cdot V_z \right]_z ; \quad (6-21)$$

The final form of the new criterion when $f_s \rightarrow 1$ is,

$$\frac{d\varepsilon_{local}}{dt} > \left[\frac{\sqrt{1-\beta}}{2} \cdot \left|\frac{d\sqrt{f_s}}{dT}\right| \cdot \left(1 + \frac{1}{\cos\theta}\right) \cdot |CR| - \frac{\tan\theta}{\varphi} \cdot \frac{|CR|}{G} \right] + \frac{d}{dz} \left[\frac{1}{2} (1 - \sqrt{1-\beta} \cdot \sqrt{f_s}) \cdot \left(1 + \frac{1}{\cos\theta}\right) \cdot V_z \right]_z \quad (6-22)$$

In this derivation, besides θ , several additional parametric terms have been added to the growth and feeding terms of the criterion with the intention of fine-tuning its ability to predict hot cracking. Note that the growth term (first term in R.H.S) contains an additional contribution from the cooling rate (CR), the thermal gradient (G), and grain width (φ). While CR was already present in Kou's penultimate equation [145] in the form of dT/dt , G and φ are new parameters. In essence, if φ is ignored and $\theta \sim 0^\circ$, Eq. (6-22) can be transformed to Eq. (5-1) after dividing it by CR. The values of CR and G can be determined through numerical simulations and φ can be directly measured from optical micrographs. Alternately, the CR can also be estimated from the PDAS [142,284]. Much like discussed earlier in connection with Eq. (5-1), when the L.H.S exceeds R.H.S in Eq. (6-22), hot cracking is expected to occur. Given that CR and $d\sqrt{f_s}/dT$ are inherently negative for solidification, and G is always positive in AM, the first part of the *growth* term in Eq. (6-22) will always be positive. The second part of the growth term, $(-\tan\theta/\varphi \times |CR|/G)$, can assume negative or positive values depending on whether the GB is divergent ($\theta > 0^\circ$) or convergent ($\theta < 0^\circ$), respectively. In the former condition, the magnitude of the overall *growth* term will be relatively smaller than that in the latter.

6.4 Discussion

The TCP phase precipitation in SEBM-built Ni-based superalloys is discussed here in detail. Multi-scale numerical simulations were utilized to explain the solute segregation tendencies at the sub-, divergent and convergent GBs. Moreover, the effect of solidification parameters, microstructural feature and material properties on the contingency of hot cracking has been laid out systematically.

6.4.1 Elemental segregation and TCP phase precipitation

TCP precipitation in Ni-based superalloys depends on the segregation tendencies of microconstituents and the service temperature [120,121,123,124,275–277]. It is important to examine the elemental segregation at the micro-scale to better understand the unexpected TCP precipitation in the SEBM-built superalloy. Chemical analysis of the γ/γ' microstructure, observed in Figure 6-10, by TEM-EDS, confirms that while Ni, Co, Cr, W, and Mo are present in both γ matrix and γ' precipitates, Al, Ti, and Ta are present only in γ' precipitates. Table 6-2 summarizes the composition profile of nano-scale γ/γ' dual-phase regions obtained from ROI-1 and ROI-2 and the TCP phases observed in ROI-1 in Figure 6-12. It is noteworthy that the partitioning behaviour of the constituent elements in SEBM-built Ni-base superalloy is quite similar to that observed in a conventionally heat-treated AM1 superalloy [222]. At high and low post-homogenization cooling rates, this alloy exhibits microstructural features similar to that present in RV-1.1 and RV-2, respectively. The γ' precipitate phases are observed to be notably rich in Al, Ti, and Ta. Moreover, even though ROI-1 lies in the fusion zone of the stainless steel base plate and SEBM-built SX superalloy, the constituent concentrations in the γ' precipitates do not deviate much from that of the conventional AM1 superalloy. Alternately, the difference in the constituent concentration of the γ matrix in RV-1.1 and that in solutionized AM1 superalloy subjected to high cooling (HC – 1273 K/s) obtained in a prior study ([222]) is due to the partitioning of constituents, such as Fe, Si, P, and Mn, from the stainless steel base plate to the γ matrix.

Based on this information and the analysis of an earlier work [204], the multi-component superalloy was simplified into a pseudo-binary alloy of A-62.1 wt.%B for meso-scale numerical simulations. Table 6-3 compiles the initial elemental compositions of the pseudo-binary alloy in wt.% for the individual constituent elements in both A and B.

Table 6-3 Chemical compositions (in wt.%) of the initial pre-alloyed superalloy powder, and weight fractions of each constituent element in the pseudo-binary alloy – A-B and the segregation ratios (SRs) for alloying elements calculated using multi-scale numerical simulations

	Ni	Cr	Co	Mo	W	Ti	Al	Ta
Initial (wt.%)	64.97	7.50	7.11	1.97	4.37	1.19	5.53	7.36
A	0.59	0.16	0.14	0.03	0.08	-	-	-
B	0.69	0.02	0.03	0.01	0.02	0.02	0.09	0.12
SR	1.11	0.09	0.23	0.37	0.27	2.94	2.93	2.93

Figure 6-17 shows the 2D meso-scale simulation results for the solidification of a melt pool, i.e. 100 μm in-depth, along the build direction – solidified up to 30 μm . The contour plots in Figure 6-17(a) display solute (B) concentration variation in the dendritic and interdendritic regions. It should be noted that the PDAS in the meso-scale simulations is smaller than that observed in micrographs. This is due to a slightly higher cooling rate in the single-track macro-scale simulations when compared to a multi-pass multi-layer melt pool solidification associated with the actual SEBM process. However, it is believed that this particular aspect does not affect the inferences made from the multi-scale simulations and that they are useful in predicting the rapid solidification conditions and segregation behaviour of constituent elements in a SEBM-built multi-component Ni-based superalloy.

Figure 6-17(b) shows the SEM images of crack-free GBs, where the precipitation of TCP phases is marked with red arrows, and the grain growth direction is marked by yellow arrows. The 1D solute concentration profiles measured along the sub-, divergent, and convergent GBs obtained through meso-scale simulations is shown in Figure 6-17(c). It is worth noting that amongst the three types of GBs, the solute concentration is as high as ~98 wt.% at divergent GB. As can be seen in Figure 6-17(b), dendritic coalescence occurs at convergent GBs, which creates intermittent regions of solute enrichment. In contrast, the sub-GB has a solute concentration profile similar to that of the divergent GB but has a lower extent of solute segregation. Moreover, as solidification progresses, secondary dendrite arms also disappear. The steep trenches observed in the 1D concentration profiles are due to the presence of secondary dendrite arms, where the solute concentration at their axes is the lowest. Figure 6-17(d) schematically illustrates dendritic growth under an ideal rapid solidification process

with the absence of secondary dendrite arms. Figure 6-17(e) represents a general trend for solute concentration variation at the three different types of GBs, as shown in Figure 6-17(d). Much like what was seen in experiments, the solute concentration at divergent and sub (neutral)-GBs show a similar trend. However, due to the excess segregation of solutes at divergent GBs, the dendrite arms surrounding it will be devoid of solute. The absence of solute results in a weakened γ/γ' microstructure during ageing. Furthermore, a higher solute concentration at a GB decreases the permeability of the surrounding mushy zone, which retards liquid feeding. This is particularly detrimental for the already crack prone divergent GBs as liquid feeding alleviates strains associated with solidification shrinkage [145,281]. It is, therefore, evident that microsegregation of solutes to divergent GBs promotes cracking. Alternately, the solute concentration profile at a convergent GB has a sinusoidal appearance with the troughs corresponding to points where dendrites coalesced.

Figure 6-17 Solute segregation during dendritic solidification of a pseudo-binary A – 62.1 wt.% B alloy. (a) Contour plot showing segregation of solute (B) during solidification obtained via 2D numerical simulations. (b) Variation of B concentration along the build direction at the sub-,

divergent, and convergent GBs. (c) A micrograph of as-built Ni-based superalloy sample showing TCP phase precipitation at divergent GB (Red arrows) and sub-GBs (bright particles). (d) A schematic of rapid solidification devoid of secondary dendrite arms. (e) A trend curve of solute concentration profiles for the sub-, divergent, and convergent GBs along the build direction

Figure 6-18 displays colour contrast plots of the elemental distribution obtained from the meso-scale numerical simulations. These plots are obtained by deconvoluting the solute segregation behaviour of B in terms of the chosen pseudo-binary system. As shown in Figure 6-18(b), (c), and (d), the γ' -forming elements -- Al, Ta, and Ti -- tend to segregate to all types of GBs. While their segregation contributes to cracking at the divergent GB, at the convergent and sub-GBs they facilitate the precipitation of C14 Laves phases. Table 6-1 and Table 6-2 list the chemical compositions of the Laves phase precipitates observed at the GBs. Clearly, these precipitates are enriched in Ta and Ti. Figure 6-18(e) – (h) show that the γ -forming elements mainly segregate to the dendrite arms. For the further analysis of the nature of segregation, a Segregation Ratio (SR) is defined as the ratio of solute concentration in the interdendritic region to its concentration at the dendrite core. SR will be used as a quantitative index for microsegregation. For instance, for any constituent element, the closer the value of SR is to 1, the lower is its tendency to segregate [285].

Figure 6-18 Contour plots from the meso-scale simulations indicating the solute segregation of each constituent element. (a) Concentration variation plot of Ni. (b), (c), and (d) are the concentration variation plots of γ -forming elements – Al, Ti, and Ta respectively indicating their segregation behaviour mainly to the sub-GBs and GBs. (e)-(h) are the concentration variation plots of γ -forming elements – Cr, Co, Mo, and W respectively. These elements possess a tendency to segregate more to the dendrite cores

Table 6-3 lists the average *SR* of each element, obtained from the simulated composition maps displayed in Figure 6-18. It is evident that Ni, Al, Ti, and Ta segregate to interdendritic regions whereas Cr, Co, Mo, and W segregate to dendrite cores. Such segregation behaviour is typical in superalloys and was reported earlier in a CMSX-4 superalloy [286]. These results are compatible with the observed compositional profiles obtained from TEM and APT analysis of the Laves phase precipitates and can explain the occurrence of severe TCP phase precipitation only at the GBs and not at the dendrite cores. Owing to severe microsegregation of Ti and Ta TCP phase precipitation at GBs becomes highly favourable during the SEBM of Ni-based SX superalloys. It should be noted that the individual segregation behaviour of constituents simulated in the meso-scale model does not consider the influence of other constituent elements [287]. Therefore, the results obtained from mesoscale simulations are at best, a qualitative estimate of the elemental segregation behaviour.

In all, meso-scale simulation results confirm that solutes such as Al, Ti, and Ta segregate at all GBs, irrespective of its nature. However, divergent GBs, which have weaker dendritic arms, provide a preferential site for severe solute segregation. In contrast, convergent GBs are both crack resistant and exhibit intermediate segregation. This behaviour can be attributed to the tendency of dendrites to coalesce at convergent GBs [288].

Solute segregation at the advancing solid interface during solidification leads to a transition from planar to dendritic solidification. To ensure uniform distribution of constituents, diffusion of elements with lower solid/liquid diffusion coefficients should be facilitated by employing slower cooling rates. Otherwise, these elements can segregate to interdendritic and intergranular regions [289]. In this study, it was shown that due to the fast cooling rates inherent to the process of additive manufacturing, severe micro-segregation followed by TCP phase precipitation occurs in SEBM built Ni-based superalloys. Moreover, high cooling rates lead to a high solid fraction rate. As this limits liquid feeding and dendritic coalescence, ROS increases significantly and promotes hot cracking. It was also determined that hot cracking and TCP phase precipitation occur at the terminal stage of solidification, although they are independent processes. However, the phenomenon of hot cracking is considered to be more deleterious than TCP phase precipitation as a high hardness value is obtained in fully dense regions regardless of the presence of the latter.

This study clearly underlines the ideal conditions required for manufacturing Ni-based SX superalloy samples using the SEBM technique. Apart from using high beam power, it is

necessary to eradicate divergent GBs via competitive grain growth or SX seeding. Employing such strategies should favour a crack-free microstructure.

6.4.2 Contingency of hot cracking

The local separation term, $d\varepsilon_{local}/dt$, in Eq. (6-22) consists of two additional contributions, the rate of solidification shrinkage (ROS) and the rate of deformation (ROD). A detailed explanation of these terms is provided in [281]. The ROS is defined as

$$ROS = \frac{\partial V}{V} \cdot \frac{1}{\partial t} = -\frac{1}{\rho} \cdot \frac{\partial \rho}{\partial t} = \frac{\beta}{\beta \cdot f_s + 1} \cdot \frac{df_s}{dt} \quad (6-23)$$

with the shrinkage factor β , given as:

$$\beta = \frac{\rho_y - \rho_l}{\rho_l} \quad (6-24)$$

where, ρ_y and ρ_l are the densities of solid and liquid phases, respectively. The shrinkage factor calculated using the thermophysical properties of CMSX-4 superalloy [234] is 4.6×10^{-2} and df_s/dt , determined from the meso-scale simulations at the terminal stage of solidification, at $f_s = 0.99$, gave a value of 1555.51 s^{-1} . The resultant ROS is calculated to be 68.44 s^{-1} .

Now,

$$ROD = -\alpha \cdot \frac{dT}{dt} \quad (6-25)$$

where, α is the coefficient for thermal expansion, the value of which was taken at $\sim 1600 \text{ K}$ as $1.9 \times 10^{-5} \text{ K}^{-1}$ for CMSX-4 superalloy [234]. The value of cooling rate, dT/dt , is determined from the macro-scale simulations as $4.0 \times 10^4 \text{ K/s}$ near the terminal stage of solidification. Therefore, the ROD value is 0.76 s^{-1} which is at least two orders of magnitude smaller than the ROS. Hence, for the current study of superalloy manufacturing using SEBM technique, solidification shrinkage is the dominant driving force for crack propagation, and the local separation term is 68.44 s^{-1} .

For the present case, the maximum value of $|d\sqrt{f_s}/dT|$, determined using meso-scale simulations, is $3.15 \times 10^{-3} \text{ K}^{-1}$. The average grain size obtained from the EBSD grain orientation map was $29.1 \text{ }\mu\text{m}$, while the CR and G determined from macro-scale simulations are $4.0 \times 10^4 \text{ K/s}$ and $2.2 \times 10^6 \text{ K/m}$, respectively. The first and second parts of the growth term in Eq. (6-22) become $61.53 \times (1 + 1/\cos\theta)$ and $626.96 \times \tan\theta$, respectively. The liquid feeding velocity

in the feeding term is derived for an average PDAS value of 4.0 μm and a liquid channel length of 42 μm obtained from meso-scale simulations to be in the order of 10^{-3} m/s [281] as shown here:

As shown in [281], the permeability of the mushy zone is given by,

$$K = \frac{C_2 \lambda_1^2 f_l^3}{(1 - f_l)} \quad (6-26)$$

With,

$$C_2 = 4.53 \times 10^{-4} + 4.02 \times 10^{-6} (f_l + 0.1)^{-5} \quad (6-27)$$

Where, λ_1 is the PDAS and f_l is liquid fraction.

Using the above equations, the value of K can be determined for an average grain width of 4 μm and a f_l of 0.01 as 5×10^{-18} m². Using this value, the liquid feeding velocity can be determined from the following equation [281]:

$$v_l f_l = -\frac{K}{\mu} \cdot \frac{\Delta P}{L} \quad (6-28)$$

Where ΔP is the pressure difference, L is the length of the liquid channel, and μ is the dynamic viscosity. ΔP is experimentally determined in [281] to be in the order of 10^4 Pa hence is followed here as well, and μ is taken as 4.9 mPa s from [281]. L is calculated from mesoscale simulations as 42 μm .

v_l comes out to be $\sim 10^{-3}$ m/s. Given that the *feeding* term in Eq. (6-28) is orders of magnitude lower than the *growth* term, its contribution to the RHS can be ignored. Therefore, in the case of SEBM-manufactured Ni-based superalloy, cracking will occur if the local separation term exceeds the growth term.

Using the obtained values of these parameters, the susceptibility for cracking is examined for three values of θ . The growth terms for a convergent GB ($\theta = -10^\circ$), for a neutral GB ($\theta = 0^\circ$), and a divergent GB ($\theta = 10^\circ$) is 234.56 s^{-1} , 123.06 s^{-1} , and 13.46 s^{-1} , respectively. Hence, according to the above-stated criterion, it is obvious that the growth term is lesser than the local separation term only for the case of the divergent GB. Therefore, the criterion predicts that divergent GBs are susceptible to hot cracking, which is consistent with our experimental observations. Although it appears that parameter optimization to limit the formation of

divergent GBs may offer a simpler solution for hot cracking, it is not a necessary and sufficient condition for cracking.

Note that the criterion in the form of Eq. (6-22) correlates the cracking susceptibility to solidification parameters (df_s/dt , CR, and G), microstructural parameters (θ and φ), and material properties (β). Their explicit contribution to cracking and qualitative estimation of how they can be optimized to achieve a crack-free microstructure is as follows:

- i. Rate of solidified fraction, df_s/dt and shrinkage factor, β : Since it was determined that ROS contributes overwhelmingly to the separation term in Eq. (6-22), either or both parameters associated with shrinkage, df_s/dt and β , can be reduced to prevent cracking. df_s/dt can be further deconvoluted as a product of df_s/dT and CR. An earlier work on solidification of an aluminium alloy B206 has shown that for the high magnitude of CR (>13 K/min), the evolution of f_s with T is roughly proportional to the variations in CR [290]. Hence, it is safe to assume that the reduction in the magnitude of CR will lead to a proportional reduction in df_s/dt . β is a material property, and its magnitude scales directly with the density difference between the solid and liquid. For the purpose of illustrating this, the crack susceptibility is compared with that of another alloy, CM186 [234], whose $\beta \sim 0.0065$ is significantly smaller than that of the alloy used in our study. For the same value of df_s/dt (1555.51 s^{-1} at $f_s = 0.99$), the ROS of this alloy calculated using Eq. (6-23) is only 10.05 s^{-1} . Hence it is expected that CM186 alloy, owing to a lower value of β , has a superior resistance to hot cracking under the processing conditions employed in this study.
- ii. Cooling rate, CR and thermal gradient, G: The growth term in Eq. (6-22) consists of two parts. While CR exclusively contributes to the first part, both CR and G contributes to the second part. In addition, the second term also contains θ , which becomes positive in the presence of divergent GBs. As mentioned earlier, a divergent microstructure may not necessarily imply that hot cracking will inevitably occur. There are two methods to reduce the possibility of cracking in a microstructure that has divergent GBs. Both of these methods involve increasing the growth term. First, if the magnitude of CR is lowered, the second part of the growth term reduces more in magnitude compared to the first part, which in turn increases the overall magnitude of the growth term. However, at lower CR, the as-built microstructure coarsens significantly, and the advantages associated with fine microstructures are compromised [15,17,18]. Moreover, since CR is associated with both terms, there is a critical lower limit of CR

beyond which the overall magnitude of the growth term cannot be increased by reducing CR. The second way of reducing the growth term is to increase G , which exclusively reduces the magnitude of the second part of the growth term. This is a more favourable method to prevent cracking as it produces alloys with fine microstructures. G can be increased by increasing the input power and beam scanning speed [68].

- iii. Angle of inclination, θ and grain width, φ : θ plays an important role in determining the resistance of a GB against hot cracking. As shown in Eq. (6-22), both terms of the R.H.S contain θ as a parameter. As mentioned earlier, for divergent GBs, the overall magnitude of the growth term is lesser than that of the local separation term as the second part of the growth term is negative. However, the second part of the growth term will be less negative if $\theta \rightarrow 0$ in a divergent GB. This will increase the overall magnitude of the growth term, and improve the GB's resistance to hot cracking. Therefore, if the extent of divergence of grains can be limited, cracking can be prevented even at divergent GBs. This condition of lower divergence can be facilitated by employing strict grain growth control measures. Nevertheless, one fairly conclusive argument can be made w.r.t θ . The susceptibility to hot cracking at GBs is lowest for convergent GBs, intermediate for neutral GBs and highest for divergent GB. It is also worth noting that for a divergent GB, a higher φ will limit the magnitude of the term containing $\tan\theta$ (where $\theta > 0$). This implies that the magnitude of the growth term will increase as the second part of the growth term is negative. Hence, the growth term for a large angle divergent GB can still be significantly large compared to the local separation term, provided the grain width is significantly high. This should imply that the cracking tendency of wider grains must be lower. However, one report on SEBM-built Ni-based superalloy [152], revealed that although the columnar grain width increased substantially along the build direction, its resistance to hot cracking did not increase. Therefore, it appears that manipulating φ may not significantly alter hot cracking behaviour SEBM-built Ni-based superalloy parts.

An earlier work [17] has reported that crack-free Ni-based SX superalloy samples were successfully fabricated by reducing the CR to ~ 30 °C/s. However, their microstructure, with an average PDAS of ~ 30 μm , was coarser than that obtained in our study. Another work [16] featuring SEBM-built CMSX4 superalloy has reported a low density of cracks with high power or high scanning speed, through the design of experiments which is similar to the method proposed in point ii above. Hence, it is evident that this criterion can predict the conditions to

obtain crack-free microstructure in the SEBM process for different superalloys with a reasonable degree of accuracy. Moreover, the modified cracking criterion is expected to be universally applicable to any metal AM process, provided that the microstructural evolution is broadly dominated by columnar grain growth.

It is, however, necessary to mention that the influence of different parameters on cracking behaviour is not uniform (see point iii). Some parameters exert a greater influence on the cracking, and in certain conditions, the mutual dependence of certain parameters makes it impossible to ensure process control to achieve crack-free structures (see point (ii)). In light of these considerations, it is always prudent to treat the solidification of each alloy on a case-by-case basis while treating the above-mentioned parameters as general thumb-rules.

6.5 Summary

SEBM processing of a Ni-based single crystal superalloy was carried out, and its microstructure was examined in this study. Detailed microstructural characterization and multi-scale numerical simulations were conducted so as to achieve an in-depth understanding of the two prevailing microstructural features in the SEBM-built superalloys, i.e. hot cracking and TCP phase precipitation. The following conclusions, thus can be drawn.

1. Nano-scale γ/γ' microstructure with a PDAS of $4.0 \pm 0.3 \mu\text{m}$ was obtained in the SEBM-built Ni-based single crystal superalloy, which exhibited an extraordinary microhardness of $480.0 \pm 6.7 \text{ HV}$. However, severe intergranular cracking and TCP phase precipitation were observed to take place at specific GBs in the SEBM-built samples.
2. Preserved dendritic microstructures were observed on crack surfaces. TCP phase precipitation was not observed on the crack surface and at the GBs where cracking occurred. It was then deduced that hot cracking occurs during the terminal stages of solidification.
3. The experimental results demonstrate that the solidification cracking was governed by the type of GB, and to quantify this, a modified criterion for cracking was proposed that can correlate the solidification parameters to the tendency of cracking at the divergent, convergent or neutral GB. It was found that a divergent GB is more prone to suffer from solidification cracking. A convergent GB, on the other hand, is found completely resistant to solidification cracking under the SEBM parameters utilized in this study. Unlike a convergent GB, the divergent GB also lacked the tendency to promote dendritic coalescence that can potentially mitigate crack initiation and propagation.

4. TEM and APT analyses reveal that the nano-scale γ' precipitation occurred in the as-built samples via the spinodal decomposition due to the rapid solidification rate involved in the SEBM process. Along the build direction, the size of γ' precipitates increased gradually as different layers were subjected to post-reheating at different cooling rates. A higher cooling rate led to the nano-scale spherical γ' precipitates (~ 20 nm) with diffuse γ/γ' interfaces whereas, a lower cooling rate results in near-spherical γ' precipitates of ~ 100 nm in size with well-defined γ/γ' interfaces. The elemental composition of γ/γ' dual-phase region obtained via SEBM is similar to that achieved in a conventional heat-treated counterpart. Hence, the rapid solidification associated with the SEBM processes did not alter the elemental partitioning behaviour.
5. Multi-scale numerical simulations of the melt pool solidification revealed micro-segregation of Al, Ti, and Ta – the γ' forming elements, to the GBs which leads to TCP precipitation of the C14 Laves phase due to the high-temperature build environment inherent to SEBM technique. The TEM and TEM-EDX analyses of the TCP phases observed in the columnar region reveal them to be type C14 laves phase rich in Ta and Ti in the form of NiTa_2 . Additionally, the APT analysis of the TCP phase observed in the fusion zone of stainless steel substrate and deposited SX superalloy identifies them as Ta-rich Laves phase of type NiTa_2 as well. TCP phase precipitation in Ni-based superalloys may prove detrimental to their creep strength. Hence, this is a major roadblock in the application of SEBM, or any other metal AM techniques for manufacturing of Ni-based SX superalloys.

Chapter 7 Conclusions and Future Work

This chapter details the major conclusions of this research and presents the scope for future work. A major emphasis of the research work in this dissertation has been on the investigation of grain growth in the AMed parts using SEBM technique. The investigation of the effect of build geometry on the as-built microstructure of Ti-6Al-4V using experimental observations and numerical simulations highlights that a V-shaped geometry possesses an inherent tendency to strengthen the $\langle 001 \rangle$ crystallographic texture with build height. An application of this understanding to SEBM-built SS316L parts led to a technical SX microstructural evolution in X-shaped parts with a prism angle of 90°. Moreover, a fully equiaxed microstructure was obtained in as-built condition for coarse powder samples that resulted in improvement in the mechanical properties in terms of isotropy, strength, and elongation under tensile loading conditions. Another vital inference is from the SEBM fabrication of AM1 superalloy. The investigation on the secondary phase precipitates observed in the as-built samples confirms the occurrence of Ta-rich C14 Laves TCP phase precipitates due to the strong annealing effect inherent to the SEBM process. Moreover, solidification cracks observed in the as-built samples are found to be dependent on the behaviour of grains at the GB that underwent cracking. Through the microstructural investigations and the development of a modified criterion of cracking under rapid solidification and competitive grain growth, it can be concluded that a divergent GB has the highest susceptibility to crack when grown rapidly. Further, the modified criterion presents a direct interpretation of the effect of solidification parameters, material properties, and grain growth behaviour on the contingency of cracking in AMed alloys.

7.1 Main Findings

The comprehensive research carried out on the fabrication of Ti-6Al-4V, SS316L, and AM1 superalloy using SEBM technique has provided an in-depth understanding of the role of solidification parameters, thermophysical properties, and processing parameters on the microstructural evolution in as-built parts. The specific novel findings of this research can be outlined as below.

- The numerical simulation and experimental investigations of the SEBM-fabricated Ti-6Al-4V provide valuable insight into the effect of build geometry on the melt pool solidification parameters. The 3D numerical simulations highlight that, for a rectangular cross-section, the scan length of the beam determines the depth of the HAZ. For a multi-scan melting process, a deeper HAZ is obtained for a shorter scan length. This results in the periodic microstructural banding observed in the SEBM-built Ti-6Al-4V samples.
- Extensive macro-scale modelling of the melt pool geometry at different build heights for A-, I-, and V-shaped samples, with a prism angle of $\sim 10^\circ$, reveals that the solidification parameters at the bottom layers of A-shaped samples favour a tendency of CET in these regions. In contrast, the solidification parameters obtained in the top regions are similar to that of the lower sections of the V-shaped samples which favour the evolution of a strong $\langle 001 \rangle$ crystallographic texture. Hence, there is a possibility of combining these two shapes to obtain a strong $\langle 001 \rangle$ crystallographic texture in as-built samples.
- The meso-scale simulations of the microstructural evolution in the SEBM-built Ti-6Al-4V samples show a strong competition among grains. However, the competitive grain growth did not always follow the classical model as a few well-oriented grains were overshadowed by the misoriented ones. This highlights the need for a grain selector for assisting the growth of well-oriented grains in obtaining a strong $\langle 001 \rangle$ crystallographic texture.
- The SEBM fabrication of SS316L X-shaped parts, with varying prism angles, was carried out using the fine powder as the feedstock. The texture analysis of the as-built samples using XRD indicates strong $\langle 001 \rangle$ crystallographic texture in specimens with 90° prism angle. A weakening in this texture is found with the increasing prism angle beyond 90° . The macro-scale numerical simulations support this novel finding,

and it is observed that the melt pool solidification parameters for the X-shaped sample with 90 ° prism angle support the grains whose <001> growth directions are well aligned with the build direction. Consequently, a technical SX microstructure is obtained for these samples measuring 30 mm in diameter. These findings can prove beneficial, especially when designing supports for overhang structures for SX gas turbine blades.

- Another vital result presented in this dissertation is on the fabrication of SS316L parts with fully equiaxed microstructure. By utilization of coarse powder feedstock, having a size distribution between 10 – 170 µm, the sintered powder bed with higher thermal conductivity and density is achieved. This resulted in the employment of much lower beam scanning speed for the fabrication of fully dense parts. A lower thermal gradient value is obtained in the melt pool for a high cooling hence increasing the tendency of CET. The as-built parts, with an avg. grain size of ~ 10 µm, show better homogeneity in microstructure with a random crystallographic texture.
- Isotropic mechanical properties are obtained in the SS316L coarse powder as-built samples under uniaxial tension. YS value of 310 MPa for both horizontal and vertical samples, maximum UTS of 625 MPa and maximum elongation of 55 % for horizontal samples are much higher than their fine powder counterparts and surpass the ASTM requirements. A major difference in deformation modes is observed between the coarse and fine powder samples. While the deformation twinning dominates the coarse powder fracture mechanism, fracture in fine powder samples takes place through dislocation slip. These findings show a promise in SEBM fabrication of high strength parts with homogeneous and isotropic microstructure for engineering alloys.
- The SEBM fabrication of Ni-based AM1 superalloy highlights the detrimental effect of high-temperature build environment on the microstructural evolution in the as-built parts. Large amounts of TCP phase precipitates were observed at the GBs. The TEM and APT investigations of these precipitates reveal them as Ta-rich C14 Laves precipitates. Multi-scale numerical simulations of the grain growth during microstructural evolution reveal severe segregation of Ta at the GBs. These results show the prospect of multi-scale numerical simulations in predicting the segregation of elements during metal AM fabrication of multi-component alloys and superalloys.

- Apart from the TCP phase precipitation, severe cracking at the GBs was observed in the SEBM fabricated AM1 superalloy samples. A comprehensive investigation of the optical and FESEM micrographs reveal the cracking to be limited to the divergent GBs. A fractography analysis shows fully preserved dendritic microstructure in the fractured surfaces indicative of solidification cracking in the as-built samples. In order to account for the effect of the type of GB on the cracking contingency, a modification is performed in the prevailing criterion for cracking. The modified criterion thus considers the converging or diverging behaviour of grains at the GB. Moreover, the effects of cooling rate, thermal gradient and grain width are involved in the modified criterion to provide a better interpretation of the effect of the process, microstructural and solidification parameters, and material properties on the susceptibility of an AMed alloy to crack under rapid solidification. It is determined that for a competitively grown PX, divergent GBs are most susceptible to cracking in comparison to convergent and neutral GBs. This information can prove beneficial in fabricating crack-free microstructure in non-weldable Ni-based superalloys.

7.2 Limitations and Future Work

The experimental work carried out in this research on the SEBM fabrication of SS316L alloy can be extended to other engineering alloys. For instance, the X-shaped geometry can be applied to induce SX crystallographic texture evolution in overhang sections of AMed high-temperature gas turbine blades. Moreover, modified support structures in accordance with the X shape can prove beneficial in tailoring microstructure in Topologically Optimized (TO) parts.

The predominant inference from this research is the successful fabrication of completely homogenized equiaxed microstructure in SEBM fabricated SS316L parts. The understanding gained in this regard can be applied to alloys such as Ti-6Al-4V to fabricate parts with ultra-fine equiaxed microstructure ($\sim 10\ \mu\text{m}$ grain size) and explore the possibility of superplasticity in AMed parts.

While the numerical modelling framework detailed in this research shows the promise of utilizing numerical techniques to the advantage of understanding grain growth in SEBM process, the research can be continued to include fluid flow physics at both macro- and meso-scales. The macro-scale modelling employed in this research was limited to the melt pool solidification behaviour in one solidified layer at a time due to economic and computational

constraints. A more accurate prediction of the solidification parameters can be from a multi-pass and multi-layer process which can be applied to future studies. Moreover, the investigation of microstructural evolution during SEBM-fabrication of fine and coarse SS316L powders performed during this study identifies a substantial effect of powder size distribution on the thermophysical properties of the sintered powder-bed. A fruitful extension along this direction would be the implementation of a Lattice-Boltzmann based numerical model for consideration of the powder bed instead of continuum assumptions [177–181]. Another continuation can be in terms of the CA-based meso-scale modelling of multi-component alloys [212]. This would assist in the determination of solute segregation more accurately for individual alloying elements, and provide one with the predictive capabilities in terms of secondary phase formation in SEBM-built components, and reduce the need for destructive analysis of the as-built parts. The multi-scale numerical modelling presented in this thesis is also limited to the solidification process only. However, a possible future extension of this work can be in the simulation of phase transformation during subsequent cooling [208].

In the present state, a one-way coupling between the models at different length scales is employed, where the thermal history data obtained at a point in the melt pool from the macro-scale model is used as an input to the meso-scale model, whereas, no input is provided vice-versa. Hence, a multi-scale model with a two-way coupling such that the solute segregation near a dendrite tip or at the GB region can govern the fluid flow in the melt pool. This would assist in modelling the fluid flow in the melt pool accurately, and visualize the liquid feeding phenomena.

Since the numerical modelling presented in this thesis is confined to the effect of process parameters on the solidification parameters and solute segregation, it would be interesting to develop a framework for predicting the mechanical properties from the as-built microstructure. One such framework could be developed by coupling an architecture for computational microstructure such as Dream3D with a crystal plasticity simulation package such as DAMASK [291].

For the determination of powder bed density for SS316L fine and coarse powders, particulate microstructure modelling technique was employed. This technique shows promise in modelling the powder bed effectively and can be used for determination of the thermophysical properties, which in this research were determined using LFA and DSC.

From the application perspective, the knowledge gained on the grain growth during SEBM-fabrication of SS316L parts, and the modified analytical criterion developed for cracking during solidification can be applied to the fabrication of SX Ni-based superalloy parts or better yet an entire gas turbine blade with the complex shapes as that would be a crucial step in establishing SEBM or any metal AM technique for that matter, as the primary manufacturing technique for the aerospace and power industries.

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