MICROSTRUCTURAL AND MECHANICAL CHARACTERIZATIONS OF METALLIC
PARTS MADE BY POWDER-BED FUSION ADDITIVE MANUFACTURING
TECHNOLOGIES

by

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A DISSERTATION

Submitted in partial fulfillment of the requirements
for the degree of Doctor of Philosophy
in the Department of Mechanical Engineering
in the Graduate School of
The University of Alabama

TUSCALOOSA, ALABAMA

2017
ABSTRACT

Two typical powder-bed additive manufacturing (AM) technologies are selective laser melting (SLM) and electron beam additive manufacturing (EBAM). Due to the complex thermal history and the interactions among the thermal, mechanical, and metallurgical phenomena during the manufacturing process, further study is needed to comprehensively understand the manufacturing process and achieve finishing parts with desired properties for application in the industries. The primary objectives of this research are: (1) investigate the effects of the beam scanning speed and support structure in the EBAM; (2) determine the influences of build height and thermal cycles in the SLM; (3) estimate the distribution of the induced residual stress; and (4) model the microstructural evolution of Inconel 718 in the SLM.

To achieve the research objectives, microstructure characterization technologies including optical microscopy, scanning electron microscopy, Energy-dispersive X-ray spectroscopy, and electron backscattered diffraction have been utilized, and nanoindentation and Vickers indentations were used to evaluate their mechanical properties. In addition, Vickers indentation method was adopted to estimate the residual stress, and the phase field method was developed to model the microstructural evolution.

Based on the results obtained, it is found that (a) a typical columnar and equiaxed microstructure were observed in the X-Plane (side surface) and Z-Plane (Scanning Surface) of the AM parts, respectively. The $\gamma$ phase of Inconel 718 presented a distinct $\{0\ 0\ 1\}$ texture in the $Z$-plane and a strong $\{1\ 0\ 1\}$ texture in the $Y$-plane. The $\alpha$ phase in Ti-6Al-4V had relatively
weak textures of \( \{0 \ 0 \ 0 \ 1 \} \) and \( \{1 \ 1 \ 2 \ 0 \} \) parallel to the z-axis. (2) Fine colonies of cellular
dendrites with a cell spacing of \( 0.511 \sim 0.845 \ \mu m \) were observed in the Inconel 718, which
implied a cooling rate of \( 1.74 \sim 3.88 \times 10^7 \ \text{K} \cdot \text{s}^{-1} \) \( (\circ C \cdot \text{s}^{-1}) \). (3) With increase of the build
height, the width of the columnar structure increased from \( 75 \ \mu m \) till a stable state around \( 150 \ \mu m \), and then slightly decreased to \( 112 \ \mu m \) when closing to the ending process, which results
from the variation of the thermal gradient along the build height. (4) Under continue effects of
thermal cycles, the morphology of Laves phase changed from coarse and interconnected
irregular particles to discrete particles, and the maximum intensity of the texture also increased.
(5) Equiaxed grains were formed at the bottom of the overhang region and then translated into
wider columnar structures. The solid-gas support structure acted as a heat sink to enhance heat
transfer and provided support for the overhang to avoid the occurrence of sink phenomena. (6)
The phase field method is a powerful tool for simulation of microstructure evolution in SLM
process. The manufacturing parameters significantly affected the thermal gradient which plays
an important role in the dendrite growth and a larger temperature gradient resulted in a higher
growth speed. Most of the columnar cellular dendrites have a preferred growth direction of \( 45 \sim 72^\circ \) to the scanning plane. (7) The residual stress is unevenly distributed in the parts with no
notable difference in the X-plane and Y-plane. The beam scanning speed and the build height did
not show significant effects on the residual stress, while the right angle interface of the geometry
induced a stress concentration. (8) The mechanical properties of the parts are comparable with
the count-parts made by traditional methods. (9) The volume fraction of the porosity is below
2\%, and no remarkable effects were found from the thermal cycles and the build height.
<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Full name</th>
</tr>
</thead>
<tbody>
<tr>
<td>AC</td>
<td>Air cooling</td>
</tr>
<tr>
<td>AM</td>
<td>Additive manufacturing</td>
</tr>
<tr>
<td>ASME</td>
<td>The American Society of Mechanical Engineers</td>
</tr>
<tr>
<td>BCC</td>
<td>Body centered cubic</td>
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<tr>
<td>CFD</td>
<td>Computational fluid dynamic</td>
</tr>
<tr>
<td>CNC</td>
<td>Computer numerical control</td>
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<tr>
<td>DDC</td>
<td>Ductility dip cracking</td>
</tr>
<tr>
<td>DLD</td>
<td>Direct laser deposition</td>
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<tr>
<td>DMD</td>
<td>Laser direct metal deposition</td>
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<tr>
<td>EBAM</td>
<td>Electron Beam additive manufacturing</td>
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<tr>
<td>EBSD</td>
<td>Electron backscattered diffraction</td>
</tr>
<tr>
<td>EDS</td>
<td>Energy-dispersive X-ray spectroscopy</td>
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<tr>
<td>FC</td>
<td>Furnace cooling</td>
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<tr>
<td>FCC</td>
<td>Face-centered cubic</td>
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<tr>
<td>FEA</td>
<td>Finite element analysis</td>
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<td>FEM</td>
<td>Finite element method</td>
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<tr>
<td>GA</td>
<td>Gas atomized</td>
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<tr>
<td>HCP</td>
<td>Hexagonal closed packed</td>
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</tbody>
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HIP                Hot isostatic press
IIT                Instrumented indentation technique
LNSM               Laser net shape manufacturing
LRF                Laser rapid forming
MC                 Metal-carbide
OIM                Orientation imaging microscopy
OM                 Optical microscope
PREP               Plasma rotating electrode processed
S1-S8              Sample #1 - #8
SEM                Selective laser melting
SEM                Scanning electron microscope
SF                 Speed function
STA                Solution treatment and aging
UTS                Ultimate tensile strength
WC                 Water cooling
Y-Z plane          Side surface
X-Z plane          Side surface
YS                 Yield strength
Z plane            Scanning surface

<table>
<thead>
<tr>
<th>Symbols</th>
<th>Units</th>
<th>Physical meanings / values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alphabetic</td>
<td></td>
<td></td>
</tr>
<tr>
<td>$a_1$</td>
<td></td>
<td>Constant, 0.8839</td>
</tr>
<tr>
<td>$a_2$</td>
<td></td>
<td>Constant, 0.6267</td>
</tr>
<tr>
<td>$a_{s0}$</td>
<td></td>
<td>Anisotropy strength related parameter</td>
</tr>
</tbody>
</table>
Interpolation function for antitrapping current profile, $\Phi$

Mixture concentration in the dimensionless ratio $c$

Concentrations (in molar fractions) of impurities $B$ at the liquid side of the interface $c_l$

Concentrations (in molar fractions) of impurities $B$ at the solid side of the interface $c_s$

The value of $c$ far from the interface that equals the initial concentration of the alloy $c_\infty$

Equilibrium concentration of the liquid at $T_0$, $c_l^0 = \frac{T_m - T_0}{|m|}$

Equilibrium concentration of the solid at $T_0$ $c_s^0$

Initial solute concentration $c_0$

Loading curvature $C$

Celsius degree $°C$

The specific heat at constant pressure $C_p$

Chemical capillary length, $d_0$

Dimensionless diffusion constant, $D$

Solute diffusivity in the liquid $D_L$

Solute diffusivity in the solid $D_s$

Elastic modulus $E$

Reduced elastic modulus $E_r$

The standard form of a double-well potential, $f(\Phi, T_m) = H \left( -\frac{\Phi^2}{2} + \frac{\Phi^4}{4} \right)$

Phenomenological free-energy function, $F[\Phi, c, T] = \int_{dv} \left[ \frac{\sigma}{2} |\nabla \Phi|^2 + f(\Phi, T_m) + f_{AB}(\Phi, c, T) \right] dp$
\(G\) \(K/m\) Temperature gradient of magnitude

\(h\) Indentation depth in nanoindentation test

\(h_v\) \(W/(m^2K)\) Convection coefficient

\(h_p\) Penetration in thermal analysis

\(h(\emptyset)\) Interpolation source function

\(H\) Barrier height dimensions of energy per unit volume

\(I\) Constant

\(\vec{J}_{at}\) Certain anti-trapping current

\(\vec{J}_c\) The solute current density, \(\vec{J}_c = -M\vec{V}\mu\)

\(\vec{J}_e\) The entropy flux

\(k\) Partition coefficient

\(K\) Interface curvature

\(K_\emptyset(T)\) A kinetic constant that can generally be temperature-dependent

\(l_T\) Thermal diffusion coefficient, \(l_T = \frac{|m|(1-k)c_\emptyset^0}{\mathcal{G}}\)

\(L_f\) \(kJ/kg\) Latent heat of fusion per unit volume

\(m\) Liquidus line of slope

\(m_s\) Solidus line of slope, \(m_s = m/k\)

\(M_S\) Martensite start temperature

\(M(\emptyset, c)\) The mobility of solute atoms or molecules

\(\vec{n}\) Normal vector

\(N_x\) Mesh in \(x\) direction

\(N_y\) Mesh in \(y\) direction

\(q(\phi)\) Interpolation function for diffusivity
\( \tilde{q}(\emptyset) \) A dimensionless function that interpolates between 0 in the solid and 1 in the liquid

\( r \) Mesh Nuclei Radius

\( R \quad \frac{J}{\text{kg} \cdot \text{K}} \) The gas constant

\( R_m \quad \text{m/s} \) Growth rate

\( s_v \) The entropy densities of the solid and the liquid at \( T_m \),

\[ s_v = \frac{\partial f^A}{\partial T} \]

\( S \) Contact stiffness

\( T \quad \text{K} \) Temperature

\( T_0 \quad \text{K} \) Liquid temperature

\( T_p \quad \text{K} \) Equivalent maximum temperature

\( T_L \quad \text{K} \) Liquidus Temperature

\( T_m \quad \text{K} \) Melting temperature

\( T_{\text{max}} \quad \text{K} \) Maximum temperature

\( u \) The chemical potential, \( u \equiv \frac{\delta F}{\delta t} \)

\( U \) Supersaturation, \( U = \frac{c-c_l^0}{(1-k)c_l^0} \)

\( v \) Poisson’s ratio

\( v_0 \) Molar volume of \( A \)

\( V_n \quad \text{m} \cdot \text{s}^{-1} \) Normal velocity of the interface

\( V_p \quad \text{m} \cdot \text{s}^{-1} \) Solidification speed

\( W \) Indentation work

\( W \quad \text{m} \) The width of the diffuse interface, \( W = \sqrt{\frac{\sigma}{H}} \)

\( W_0 \quad \text{m} \) Interface thickness
Solidification direction in directional solidification

Greek

\( \alpha \) \( m^2 s^{-1} \) The thermal diffusion coefficient, \( \alpha \equiv \frac{k}{\rho c_p} \)

\( \alpha \) The hexagonally closed packed phase of titanium and its alloys

\( \alpha' \) Martensitic structure in titanium or titanium alloys

\( \beta \) The body centered cubic beta phase of titanium and its alloys

\( \kappa \) \( W m^{-1} K^{-1} \) Thermal conductivity

\( \rho \) \( Kg \cdot m^{-3} \) Density

\( \tau \) \( s \) Temperature dependent relaxation time, \( \tau = \frac{1}{K_0(T)H} = \tau_0\left[1 - (1 - k)(z - V_p t)/l_\tau\right] \)

\( \tau_0 \) \( s \) Interface kinetics time

\( \Gamma \) \( K \cdot m \) Gibbs-Thomson constant, \( \Gamma = \frac{\gamma T_m}{L} \)

\( \Delta x \) \( m \) Step size

\( \Delta t \) \( s \) Time step

\( \Delta T_0 \) \( K \) Freezing range, \( \Delta T_0 = |m|(1 - k)c_i^0 \)

\( \epsilon' \) A dimensionless parameter that characterizes the anisotropy strength

\( \epsilon_4 \) Anisotropy strength

\( \phi \) Phase field

\( \bar{\lambda} \) The dimensionless ratio of interface thickness times freezing range and the Gibbs-Thomson constant, \( \bar{\lambda} = \frac{RT_m(1-k)c_i^0}{2V_p H} = \frac{l \Delta T_0}{2H T_m} = \frac{1 \Delta T_0^W}{2\Gamma} \)

\( \lambda \) Coupling constant between potentials
\( \gamma \)  
Surface tension, \( \gamma = IWH \)

\( \omega \)  
Grand potential

\( \mu_E \)  
The spatially uniform equilibrium value of the chemical potential

\( \sigma \)  
Energy per unit length

\( \sigma_Y \)  
Yield strength of the material

\( \theta \)  
Dimensionless temperature, \( \theta = \frac{T-T_m-mc_\infty}{L/c_p} \)

\( \Phi \)  
Laser beam diameter in thermal analysis

\( \varphi \)  
The angle between the direction normal to the interface and the \( x \) (horizontal) axis, \( \varphi = \tan^{-1} \frac{\theta_Y}{\theta_X} \)

\( \gamma \)  
Surface tension

\( \mu_k \)  
Linear kinetic coefficient

\( \Delta \)  
Non-dimensional temperature gradient

\( \Omega \)  
Non-dimensional concentration value

\( \varepsilon_{repr} \)  
A representative value of plastic strain

\( \varepsilon_v c \)  
The change of the internal energy density

\( \frac{RT}{v_0} (c \ln c - c) \)  
The dilute form of the mixing entropy
ACKNOWLEDGMENTS

First, I would like to thank my advisor, Dr. Kevin Chou, for his endless encouragement, patience, and support. Dr. Chou’s insights and talented thoughts had a great influence on me of becoming an individual with intellectual curiosity and critical thinking. Without his support and guidance, I would not be where I am today.

Also, I would like to extend my deepest appreciation to my committee members, Dr. Paul G. Allison, Dr. Mark Barkey, Dr. Laurentiu Nastac, and Dr. Alexey N. Volkov, and professors from Department of Metallurgical and Materials Engineering, Dr. Luke N. Brewer, Dr. Lin Li, and Dr. Mark L. Weaver, for their invaluable suggestions on this work and for their support. In addition, I also would like to thank Mr. Johnny Goodwin, Mr. Rob Holler, and Mr. Rich Martens from Central Analytical Facility, and Mr. Robert Fanning from Department of Metallurgical and Materials Engineering for their help and technique support during the experiments.

I also want to give thanks to my friends throughout my study, Dr. Todd M. Butler, Dr. Rogie Rodriguez, Mr. Qianying Guo, Mr. Yudong Li, Ms. Tian Liu, Mr. Tao Liu, Mr. Xuyang Zhou, Mr. Benjamin White, Ms. Rachel White, Mr. Yang Xuan, Ms. Qing Cao, Mr. Neng Wang, Mr. Dustin Avery, Mr. Zhongqi Liu, Mr. Hongda Cao, Mr. Yuan Cao, Mr. Zhong Li, Ms. Wen Chen, Ms. Lian Zhu, Ms. Zhihan Wei, Mr. Seth Kennedy, Dr. Zhigang Dang, Dr. Sicong Qian, Dr. Shaowei Wu, Dr. Juzi Li, Dr. Songqing Yue, Dr. Liming Zhou, Dr. Yi Liu, Dr. Qiang Wu, Dr. Jianwen Hu, Dr. Hui-Chuan “Hannah” Chen, and Dr. Der-San Chen.
Special thanks to the other members of our group, Dr. Ping Lu, Dr. Xibing Gong, Mr. Steven Price, Dr. Bo Cheng, Mr. Subin Shrestha and Ms. Tahmina Keya for their assistance in my research. Special thanks are also extended to UA and the Mechanical Engineering Department for providing me with such a wonderful opportunity to pursue my Ph.D. study in Tuscaloosa.

The research is supported by NASA under award No. NNX11AM11A and the additional support from National Science Foundation (CMMI 1335481), which are also acknowledged. Also, I acknowledge the financial support from AL EPSCoR GRSP.

Last but not least, I would like to thank my beloved grandparents, parents, sisters, and brothers and my extended families, for their love, understanding, patience, and encouragement when it was needed.
CONTENTS

ABSTRACT ....................................................................................................................................... ii

LIST OF ABBREVIATION AND SYMBOLS .............................................................................. iv

ACKNOWLEDGMENTS ............................................................................................................. xi

LIST OF TABLES ..................................................................................................................... xviii

LIST OF FIGURES ...................................................................................................................... xx

CHAPTER 1 INTRODUCTION .................................................................................................... 1

1.1 Background and Motivation ................................................................................................. 1

1.2 Research Objectives and Scope .......................................................................................... 4

1.3 Outline of Dissertation ...................................................................................................... 5

References ....................................................................................................................................... 7

CHAPTER 2 LITERATURE REVIEW ....................................................................................... 10

2.1 Introduction .......................................................................................................................... 10

2.2 Powder-bed Additive Manufacturing Processes ................................................................. 11

2.2.1 EBAM Process .................................................................................................................. 11

2.2.2 SLM Process .................................................................................................................... 13

2.3 Microstructure ....................................................................................................................... 14

2.3.1 Ti-6Al-4V ........................................................................................................................ 14

2.3.2 Inconel 718 ...................................................................................................................... 16

2.3.3 Effects of manufacturing conditions ................................................................................. 19
<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.3.3 Defects</td>
<td>25</td>
</tr>
<tr>
<td>2.4 Mechanical Properties</td>
<td>28</td>
</tr>
<tr>
<td>2.4.1 Tensile Testing</td>
<td>28</td>
</tr>
<tr>
<td>2.4.2 Hardness Testing</td>
<td>33</td>
</tr>
<tr>
<td>2.4.3 Other Testing</td>
<td>34</td>
</tr>
<tr>
<td>2.5 Conclusions</td>
<td>35</td>
</tr>
<tr>
<td>References</td>
<td>37</td>
</tr>
<tr>
<td>CHAPTER 3 PROCESSES PARAMETRIC EFFECT ON THE FINAL PARTS: BEAM SCANNING SPEED</td>
<td>46</td>
</tr>
<tr>
<td>3.1 Introduction</td>
<td>46</td>
</tr>
<tr>
<td>3.2 Experimental Procedure</td>
<td>48</td>
</tr>
<tr>
<td>3.3 Results and Discussions</td>
<td>50</td>
</tr>
<tr>
<td>3.3.1 Microstructures</td>
<td>50</td>
</tr>
<tr>
<td>3.3.2 Texture analysis</td>
<td>52</td>
</tr>
<tr>
<td>3.3.3 Mechanical Properties</td>
<td>59</td>
</tr>
<tr>
<td>3.4 Conclusions</td>
<td>63</td>
</tr>
<tr>
<td>References</td>
<td>66</td>
</tr>
<tr>
<td>CHAPTER 4 EFFECTS OF THERMAL CYCLES</td>
<td>70</td>
</tr>
<tr>
<td>4.1 Introduction</td>
<td>70</td>
</tr>
<tr>
<td>4.2 Experimental procedures and methods</td>
<td>72</td>
</tr>
<tr>
<td>4.3 Results and discussion</td>
<td>75</td>
</tr>
<tr>
<td>4.3.1 Microstructure analysis</td>
<td>75</td>
</tr>
<tr>
<td>4.3.2 Effects of the thermal cycles</td>
<td>79</td>
</tr>
<tr>
<td>4.3.3 Texture analysis</td>
<td>85</td>
</tr>
</tbody>
</table>
6.4.3 Build height effects ................................................................. 138
6.4.4 Beam scanning speed effects .......................................................... 140
6.4.5 Comparison of the Residual Stresses in EBAM and SLM ......................... 141
6.5 Conclusions.................................................................................................. 142
References............................................................................................................. 144

CHAPTER 7 EFFECT OF SUPPORT STRUCTURE ON THE FINAL PARTS ................. 148
7.1 Introduction................................................................................................. 148
7.2 Experimental Procedure .............................................................................. 150
7.3 Results and Discussions ............................................................................ 153
  7.3.1 Microstructure characteristics .............................................................. 153
  7.3.2 Advanced analysis of microstructure ..................................................... 161
  7.3.3 Texture analysis .................................................................................... 168
  7.3.4 Microhardness ...................................................................................... 170
  7.3.5 Residual stress ...................................................................................... 172
7.4 Conclusions.................................................................................................. 175
References............................................................................................................. 177

CHAPTER 8 MICROSTRUCTURE SIMULATION OF INCONEL 718 ................. 182
8.1 Introduction................................................................................................. 182
8.2 Mathematical formulation ........................................................................... 184
  8.2.1 Thermal analysis model ........................................................................ 184
  8.2.2 Phase Field Model ................................................................................ 187
8. 3 Results and discussion ............................................................................. 203
  8.3.1 Temperature gradient and cooling rate .................................................. 203

xvi
LIST OF TABLES

Table 2.1 Detail of the heat treatment methods used in the studies .............................................. 22
Table 2.2 Hardness of Inconel 718 parts built with laser-based technologies ......................... 34
Table 4.1 Chemical compositions of IN718 powder .................................................................... 74
Table 4.2 Manufacturing parameters used in this study ............................................................... 74
Table 4.3. Predicted cooling rate based on the statistic results from experiments ................. 79
Table 5.1 Manufacturing parameters used in this study ............................................................. 107
Table 5.2 Measured characteristic sizes SLM samples with build height ......................... 112
Table 7.1 Compositions of Ti-6Al-4V powder [35] ................................................................. 151
Table 8.1 Parameters used in thermal analysis of SLM Inconel 718 alloy ......................... 186
Table 8.2 Parameters used in the phase field modeling on Inconel 718 alloy [32,33] ......... 202
Table 8.3 Temperature gradient, growth rate and cooling rate at the fusion boundary of the melt pool during SLM process of Inconel 718 at the first pass, layer 44 ............ 209
Table 8.4 Changing trend of melt pool during SLM process of Inconel 718 ......................... 215
Table 8.5 Temperature gradient, growth rate and cooling rate at the fusion boundary of the melt pool during SLM process of Inconel 718 at beam scanning speed of 200 \( mm/s \) ........................................................................................................................ 217
Table 8.6 Temperature gradient, growth rate and cooling rate at the fusion boundary of the melt pool during SLM process of Inconel 718 at beam scanning speed of 600 \( mm/s \) ........................................................................................................................ 218
Table 8.7 Temperature gradient, growth rate and cooling rate at the fusion boundary of the melt pool during SLM process of Inconel 718 at beam scanning speed of 1000 \( mm/s \) ........................................................................................................................ 218
Table 8.8 Temperature gradient, growth rate and cooling rate at the fusion boundary of the melt pool during SLM process of Inconel 718 at beam scanning speed of 1500 mm/s ........................................................................................................................ 219

Table 8.9 Temperature gradient, growth rate and cooling rate at the fusion boundary of the melt pool during SLM process of Inconel 718 at different beam scanning speed.... 219

Table 8.10 Changing trend of melt pool during SLM process of Inconel 718 ...................... 224

Table 8.11 Temperature gradient, growth rate and cooling rate at the fusion boundary of the melt pool during SLM process of Inconel 718 at the first pass, layer 2 ................. 225

Table 8.12 Temperature gradient, growth rate and cooling rate at the fusion boundary of the melt pool during SLM process of Inconel 718 at the first pass, layer 79 ............... 226

Table 8.13 Temperature gradient, growth rate and cooling rate at the fusion boundary of the melt pool during first pass SLM process of Inconel 718 at different heights ......... 227

Table A.1 Input in the calculations ............................................................................................. 246

Table A.2 Physical properties of Inconel 718 ............................................................................. 247
LIST OF FIGURES

Figure 1.1 (a) The Arcam A2X system [9], and (b) Concept Laser M2 cusing system [10] .......... 2

Figure 2.1 Schematic drawing of an Arcam EBAM machine [8] .................................................. 12

Figure 2.2 Schematic of the SLM machine layout [17] .................................................................. 14

Figure 2.3 Microstructures of Ti-6Al-4V specimens made by (a) EBAM vs. (b) Wrought [32]. 16

Figure 2.4 Optical micrographs of Inconel 718 specimens: (a) SLM, cross section [6], (b-c) vertical section [51,52] and (d) Casting [53] .............................................................. 17

Figure 2.5 SEM micrographs of As-deposited SLM IN718 samples: (a) cross section and (b) vertical section [6] ....................................................................................................... 18

Figure 2.6 SEM micrographs showing the intermetallic micro-segregation in (a) Inside γ-phase grains [51], (b) lower inset shows the δ needles at stacking faults and carbides at higher magnification [38], (c-d) Micrograph and the respective chemical compositions of the various precipitates [38] ........................................................................ 21

Figure 2.7 Micrographs in vertical section of SLM-processed IN718 samples with heat treatment: (a) Solutioned (980 °C/1 h)+ Aged [6], (b) Aged, (c) Solutioned (1040 °C/1 h) and (d) Solutioned (1100 °C/1 h) and aged [51] .................................................. 24

Figure 2.8 Micrograph of EBAM produced Ti-6Al-4V alloy, (a) [34] and (b) [76] ..................... 26

Figure 2.9 Porosity defects observed in the SLM-processed of Inconel 718 at various laser densities: (a) 180 J/m, (b) 275 J/m, (c) 330 J/m [7] ............................................................. 26

Figure 2.10 Comparison of the mechanical properties between EBAM vs. wrought Ti-6Al-4V specimens. (A1 [80], A2 [81], A3 [79], A4 [82] , Wrought (ASME) ) ................ 29

Figure 2.11 Mechanical properties of Inconel 718 ....................................................................... 30

Figure 2.12 Strengths of Inconel 718 after heat treatment ............................................................ 32

Figure 2.13 Elongation compared with after heat treatment .......................................................... 32
Figure 2.14 Tensile strength of four series Inconel 718 specimens built with various orientation ................................................................. 33

Figure 3.1 Ti-6Al-4V alloy: (a) As-fabricated EBAM block built with SF50, and (b) XRD patterns obtained for the raw powder ................................................................. 48

Figure 3.2 Triboindenter (a) appearance and (b) nanoindentation setup, and (c) applied load function ........................................................................................................ 50

Figure 3.3 Typical EBAM Ti-6Al-4V microstructure under optical microscope (a) Y-plane and (b) Z-plane ........................................................................................................ 51

Figure 3.4 Microstructure on Z-plane of Ti-6Al-4V under scanning electron microscope ...... 52

Figure 3.5 As-fabricated EBAM Ti-6Al-4V samples and typical EBSD data ......................... 53

Figure 3.6 EBSD data from Y plane of EBAM Ti-6Al-4V parts ........................................ 55

Figure 3.7 EBSD data from Z plane of EBAM Ti-6Al-4V parts ........................................ 57

Figure 3.8 Average diameter of grains of Ti-6Al-4V alloy .............................................. 58

Figure 3.9 EBAM Ti-6Al-4V alloy: (a-b) typical misorientation angle maps, and (c-f) distribution of misorientation angle ................................................................. 59

Figure 3.10 Typical load vs. displacement curves ............................................................ 60

Figure 3.11 Comparison of (a) elastic modulus and (b) hardness of EBAM Ti-6Al-4V alloy (SF 36) between test values and literature values (Note: Literature hardness results are microhardness) ................................................................. 61

Figure 3.12 Hardness of Ti-6Al-4V specimens built with different SFs: (a) Z-plane, (b) Y-plane .................................................................................................................. 63

Figure 3.13 Elastic modulus of Ti-6Al-4V specimens built with different SFs: (a) Z-plane, (b) Y-plane .................................................................................................................. 63

Figure 4.1 The pre-alloyed raw IN718 powder, (a) SEM morphology and (b) histogram of IN718 particle-size distribution; (c) as-fabricated staircase block, and (d) schematic drawing showing the location of the specimens ........................................... 74

Figure 4.2 Optical microscopy images showing the microstructures of the SLM IN718 samples (a-c) Z-plane and (d) Y-plane ........................................................................... 76
Figure 4.3. SEM morphologies of $\gamma$ matrix and Laves phase in the microstructure of SLM IN718: (a-b) BSE micrographs of sample 1.0Y1, and (c-d) SEM micrographs of specimen 3.1Y1

Figure 4.4. SEM images showing the microstructures of Y-plane at different build heights of SLM IN718 (a-f), and statistic results of (g) the fraction of Laves phase and (i) the width of the cellular dendrites

Figure 4.5. SEM microscopy images showing microstructure of inside Y-plane at bottom of the SLM IN718 part (a-f), and statistic results of (g) Laves phase fraction, and (i) width of the cellular dendrites

Figure 4.6. SEM micrographs showing microstructures in Z-plane with and without thermal cycles in SLM 718 parts (a-f), and statistic results of (g) fraction of Laves phase, and (i) width of the cellular dendrites

Figure 4.7. EBSD analysis of SLM IN718 sample (1.0Z1), a larger scanning area: (a) inverse pole figure (IPF) colored map, (b) grain map, (c) pole figure (PF) maps, (d) grain size plot and (e) misorientation angle plot; and two enlarged areas: (f-h) IPF colored map, (g) grain boundary map corresponding to (f), and (g) grain map corresponding to (h)

Figure 4.8. EBSD analysis of SLM IN718 sample (2.1Y2), a larger scanning area: (a) IPF colored map, (b) grain map, (c) PF maps, and (d) grain size plot; and an enlarged area: (e) IPF colored map, (f) grain boundary map, and (g) misorientation angle plot corresponding to (f)

Figure 4.9. EBSD results of samples from various build height without further thermal cycles: (a) 3.1Y1, (b) 2.1Y1, (c) 1.0Y3, (d) 3.1Z1, and (e) 1.0Y3

Figure 4.10. EBSD results of samples from various build height with further thermal cycles: (a) 3.1Y1, (b) 3.1Y3, (c) 1.2Y1, (d) 2.1Y1, and (e) 2.1Y2

Figure 4.11. EBSD results of samples from different build height with/without further thermal cycles: (a) 1.0Y3, (b) 1.1Y1, and (c) 1.2Y1

Figure 4.12. IN718 samples, 1.1Y1: (a) pores under optical microscope at the location close to the boundary of the part, A1-small spherical pores, and A2-big pores and irregular pores, (b-d) locations of the samples and the statistic values of the porosity fraction

Figure 5.1 (a) Laser Concept M2 Cusing System, (b) island scanning pattern and (c) as-deposited part with stress relieved

Figure 5.4 Optical micrograph showing etched microstructure in the Y-plane (a) S1, (b) S2, (c) S4 and Z-plane (d) S8
Figure 5.5 Statistic results of porosity in the Inconel 718 samples: (a) X-plane and (b) Y-plane .......................................................................................................................... 112

Figure 5.6 (a-d) are orientation maps from Y-plane of the samples #1, #2, #3 and #4, respectively, and (e-h) are pole figures corresponding to (a-d), sequentially........... 114

Figure 5.7 (a-b) are orientation maps from Z-plane of the samples #5 and #8, respectively, and (c-d) are pole figures corresponding to (a-b) ................................................................. 115

Figure 5.8 (a, b) are the typical misorientation angle map from Y-plane (#2) and Z-plane (#8), and (c, d) are the statistics results corresponding to (a, b) ............................... 116

Figure 5.9 Load vs. displacement curve from the nanoindentation test ........................................ 119

Figure 5.10 Young’s Modulus for the SLM-processed Inconel 718 alloy ................................. 119

Figure 5.11 Nano-hardness of SLM-processed Inconel 718 alloy ............................................. 119

Figure 6.1. Schematic of the geometry of the Vickers indentation tests [18], and (b) Schematic of the nominal projected contact area Annom ....................................................... 127

Figure 6.2. Fabricated metal parts: SLM Inconel 718 (a) as-fabricated with stress relieved block, (b) as-fabricated staircase part, and EBAM Ti-6Al-4V (c) speed function part ............................................................................................................................ 132

Figure 6.3. (a) Vickers indentation test stage, and (b-d) typical residual profiles of Vickers indentation test on the surface of as-fabricated Inconel 718 samples.............................. 133

Figure 6.4 Comparison of Vickers indentation test results of X-plane and Y-plane Inconel 718 samples, (a, c) Vickers hardness, and (b, d) Residual stresses ...................... 135

Figure 6.7 Y-plane of stress relieved SLM Inconel 718 alloy: (a) Residual stress, (b) Vickers hardness, and SEM images of (c) S1 and (d) S4....................................................... 139

Figure 6.8 Z-plane of stress relieved SLM Inconel 718 alloy: (a) Residual stress, (b) Vickers hardness..................................................................................................................... 140

Figure 6.9. Hardness of Ti-6Al-4V specimens built with different SFs....................................... 141

Figure 6.10. Residual stresses in the Ti-6Al-4V samples.............................................................. 141

Figure 7.1. Schematic of Ti-6Al-4V overhang parts (a) tooth contact (TC), (b) solid-gap (SG), and (c) locations of the samples (S1-4) cut from the part .............................. 151

Figure 7.2. OM images of the microstructures of Ti-6Al-4V samples from TC part: (a, b) S1, and (c, d) S2 ............................................................................................................. 154
Figure 8.4 Schematics showing the edge and tail monitoring locations selected to analyze the solidification conditions: (a) 3D view, and (2) 2D cross-section view [35]............. 205

Figure 8.5 Schematic of the melt pool in selective laser melting process................................. 207

Figure 8.6 Inconel 718: (a) Longitudinal section, and (b) Thermal gradient at the fusion boundary, of the melt pool at the first pass layer 44 in SLM process.............................. 209

Figure 8.7 Example of measuring the angle between the dendrites growing direction and the beam scanning direction ......................................................................................... 210

Figure 8.9 Phase field profile of Inconel 718 at the average dimensionless thermal gradient of 0.498 (Temperature gradient $\Delta T = 1.04 \times 10^6 \degree C/m$ ).............................................. 212

Figure 8.10 Concentration profile of Inconel 718 at the average dimensionless thermal gradient of 0.498 (Temperature gradient $G = 1.04 \times 10^6 \degree C/m$).............................. 213

Figure 8.11 Example of measuring second dendrites arm spacing (a) experimental test, (b) microstructure simulation ................................................................................................. 214

Figure 8.12 Longitudinal section of melt pool during SLM Inconel 718 at different beam scanning speed: (a) 200 mm/s, (b) 600 mm/s, (c) 1000 mm/s, and (d) 1500 mm/s ................................................................................. 215

Figure 8.13 Thermal gradient at the fusion boundary of the melt pool during SLM process of Inconel 718 at the beam scanning speed of 200 mm/s................................................. 217

Figure 8.14 Thermal gradient at the fusion boundary of the melt pool during SLM process of Inconel 718 at the beam scanning speed of 600 mm/s................................................. 217

Figure 8.15 SLM process of Inconel 718 at beam scanning speed of 1000 mm/s ................. 218

Figure 8.16 Temperature gradient at the fusion boundary of the melt pool during SLM process of Inconel 718 at beam scanning speed of 1500 mm/s................................. 219

Figure 8.17 Microstructure evolution of SLM Inconel 718 at $t = 1 ms$ with the laser beam scanning speed of 200 mm/s (Dimensionless thermal gradient $U = 1.027$)....... 221

Figure 8.18 Microstructure evolution of SLM Inconel 718 at $t = 1 ms$ with the laser beam scanning speed of 600 mm/s (Dimensionless thermal gradient $U = 1.187$)....... 221

Figure 8.19 Microstructure evolution of SLM Inconel 718 at $t = 1 ms$ with the laser beam scanning speed of 1000 mm/s (Dimensionless thermal gradient $U = 1.442$)....... 222
Figure 8.20 Microstructure evolution of SLM Inconel 718 at $t = 1$ ms with the laser beam scanning speed of 1500 $mm/s$ (Dimensionless thermal gradient $U = 1.068$) ...... 222

Figure 8.21 Characteristics of the simulated microstructure at different beam scanning speeds in SLM process: (a) Secondary dendritic arm spacing, and (b) Growth rate, of the dendrites ................................................................................................................................. 223

Figure 8.22 Longitudinal section of melt pool during SLM Inconel 718 at different heights: (a) layer 2, (b) Layer 44, and (c) Layer 79 ........................................................................................................................................... 224

Figure 8.23 Inconel 718: (a) Longitudinal section, and (b) Thermal gradient at the fusion boundary, of the melt pool at the first pass layer 2 in SLM process .............................. 225

Figure 8.24 Inconel 718: (a) Longitudinal section, and (b) Thermal gradient at the fusion boundary, of the melt pool at the first pass layer 79 in SLM process .............................. 226

Figure 8.25 Microstructure evolution of SLM Inconel 718 for layer 2 at $t = 1$ ms and dimensionless thermal gradient $U = 0.907$ .................................................................................................................................. 228

Figure 8.26 Microstructure evolution of SLM Inconel 718 for layer 79 at $t = 1$ ms and dimensionless thermal gradient $U = 0.529$ .................................................................................................................................. 228

Figure 8.27 Characteristics of the simulated microstructure at first pass with different layers in SLM process .................................................................................................................................. 229

Figure A.1 Schematic of the element $(i, j)$ and its adjacent elements $\Delta x = \Delta y$ ) .............. 242

Figure A.2 Flow chart for the FORTRAN code in microstructural simulation ...................... 248
1.1 Background and Motivation

Additive manufacturing (AM) has grown up organically from the early days of rapid prototyping [1]. According to the standard, ISO/ASTM52900-15 [2], AM is “a process of joining materials to make parts from 3D model data, usually layer upon layer, as opposed to subtractive manufacturing and formative manufacturing methodologies.” Metallic part fabrication has been of great interest in industries due to the possibility of fabricating of net (near-net) shape components directly from 3D-models using raw metal powder [3,4]. During additive manufacturing, the near net shape part is made by melting only the required amount of materials in a controlled fashion, which is quite different with the conventional subtractive manufacturing processes like machining [5], in which the desired shape and size of the part is obtained by removing the excess material from a large casting or forging. Compared to conventional manufacturing methods, AM processes have the following perceived advantages [6]:

(1) Material efficiency. AM uses raw materials efficiently by building parts layer by layer, and leftover materials can often be reused with minimum processing, while a lot of materials need to be removed in conventional subtractive manufacturing [7].

(2) Resource efficiency. AM does not require auxiliary resources, such as jigs, fixtures, cutting tools, and coolants in addition to the main machine tool that required in conventional manufacturing processes. This presents an opportunity for improved supply chain dynamics.
(3) Part flexibility. AM is possible to build a single part with varying mechanical properties (flexible in one part and stiffer in another part) because there are no tooling constraints in AM and no need to sacrifice part functionality for the ease of manufacture. This opens up opportunities for design innovation [8].

(4) Production flexibility. In AM, the quality of the parts depends on the process rather than operator skills. Therefore, the production can be easily synchronized with customer demand. In addition, the problems of line balancing and production bottlenecks are virtually eliminated because complex parts are produced in single pieces.

According to the type of raw material input and the energy source used to consolidate or form the part, the majority of metal AM systems can be divided into two main categories, powder bed fusion and directed energy deposition [1]. The starting material for powder bed fusion systems is metal powder while for directed energy technologies is either powder or wire [5]. In powder bed AM systems, the build envelope is an enclosed chamber that can be operated in vacuum or filled with an inert gas to prevent oxidation of reactive metal powders, such as titanium and aluminum. The chamber is preheated to a pre-determined temperature depending on the process, around 100°C for laser based and 700°C for the electron beam. In the center of the chamber, a reservoir of metal powder is smoothed using a leveling system with a layer thickness of 20 to 200 µm. The laser or electron beam is then selectively scanned over the surface of the metal powder particles to melt and fuse them to the underlying layers following the predefined pattern of the part. The build reservoir is then lowered a single layer thickness, a leveling system provides fresh powder on top of the part, and the process is repeated until the final build is finished. This study will focus on two typical powder-bed AM systems, Selective Laser Melting (SLM) and Electron Beam Melting (EBM), as the final parts produced by them usually have a
higher strength than other metal AM technologies [4]. The typical systems for SLM and EBSD are shown in Figure 1.1.

![Figure 1.1 (a) The Arcam A2X system [9], and (b) Concept Laser M2 cusing system [10]](image)

SLM completely melts the metal powder with a high-power laser beam to form a metallic part that is almost completely dense and does not require post-processing [4]. This results in mechanical properties equal to or even better than those of rolled metal sheets. The manufacturers of commercial SLM equipment include the MCP Realizer, EOS, and SLM Solutions. Currently available alloys used in this process include Stainless steel, Inconel alloy, and Titanium alloy. One of the key advantages of this process is that the powder bed serves as an in-process support structure for overhangs and undercuts, and, therefore, complex shapes with a high geometrical accuracy of +/- 0.05 mm can be manufactured with this type of process which separates this AM technology from the others [11].
Electron beam based processes have significantly higher power density [12] than laser-based processes, and also, the electron beam based Arcam® process has comparatively faster melting rate than laser-based processes because of its higher energy density and higher scanning speed. Parts fabricated using the electron beam process also tend to have significantly lower residual stresses [13] compared to parts manufactured using laser-based fabrication process. It has the capability of creating fully dense parts in a variety of metal alloy applications. These characterizations have made it a promising process to produce Ti-6Al-4V functional parts for aerospace application, and medical industry for creating implants [14–16].

As a dynamic field of study, AM has acquired a great deal of significant progress in the practical application. For example, additive manufacturing has revolutionized the production of turbines and turbos which are regarded as complex to produce as they have to be as light as possible and ultra-safe to meet the needs of the aerospace industry. Another significant implication is in the medical industry, such as dentistry and orthodontics industry, which is full of one-off, expensive designs. In addition, there is a fast growth in tool repairing as the fine layers of metal powder can be melted to repair the area of the tool affected that used has to be replaced the whole tool. Moreover, in Jewelry industry, Additive manufacturing allows for a variety of metals and gemstones to be used in a number of ways not previously possible. Furthermore, it also has been adopted to produce models and prototypes during a product’s development phase and produce parts of high geometrical complexity, which can not be produced by means of conventional manufacturing. However, they are still some drawbacks in the product fabricated by additive manufacturing processes, which are not fully understood and still need further study.
(1) Porosity (void and pores) has been a historical issue with the AM process even though there have been some improvements towards fully dense parts [17,18].

(2) The deposition rate is very low for all the powder bed processed, and the fabricated component size is limited to the bed size, which restricts its application to relatively small components.

(3) The process is difficult to control due to the large energy input to melt metal particles. The layer-by-layer fusion step in powder-bed fusion systems leads to a complicated thermal history which has a significant influence on the microstructural evolution and mechanical properties of the final part [19].

(4) The part’s surface finish is not as good as the counterpart fabricated by traditional methods. The complex of the process causes problems such as residual stress development and part deformation [20]. The observed errors of the AM parts are significantly larger than those of typically machined parts, and even delamination for some layers [21].

These issues still limit its growth in production applications, even though there has been particular interest in aerospace [22–24] and biomedical industries [25,26] owing to the possibility of making high performance parts with reduced overall cost for manufacturing, such as Ti-6Al-4V and Inconel 718 which have been widely used in many industries due to their desirable and versatile combination of good mechanical and chemical properties.

1.2 Research Objectives and Scope

The primary objectives of this study are to evaluate the microstructure and mechanical properties of EBAM-produced Ti-6Al-4V and SLM-processed Inconel 718 alloys. The objectives of this research include the following:
(1) Investigate the effects of manufacturing parameters on the microstructure and mechanical properties of the EBAM Ti-6Al-4V and SLM Inconel 718 parts, and study the difference of morphology and microstructure among different manufacturing parameters.

(2) Evaluate the residual stresses in the final EBAM Ti-6Al-4V and SLM Inconel 718 samples and compare the two processes.

(3) Apply the phase field method [27–29] to simulate the microstructure evolution during the solidification process in selective laser melting. The growth of microstructure will be studied, and the morphology and concentration field will be investigated. In addition, the influence of the undercooling and anisotropy on the microstructure will be studied.

This work would contribute to the understanding of microstructure evolution and resulting mechanical properties of EBAM and SLM produced parts.

1.3 Outline of Dissertation

The layout of this proposal is as follows.

Chapter 2 presents a thorough literature review of the powder-based technologies by focusing on EBAM process for Ti-6Al-4V and SLM process for Inconel 718. The chapter introduces the general aspects of EBAM and SLM processes, including their characteristics, advantages, and challenges. Moreover, it includes extensive discussions of microstructures, mechanical properties, and defects, which affect the application ranges of Ti-6AL-4V and Inconel 718 alloys.

In Chapter 3, the influence of the electron beam scanning speed on the microstructure, texture and mechanical properties of the EBAM-processed Ti-6Al-4V parts is investigated. The
relationship of the scanning speed and its resulted microstructure, texture and mechanical properties is studied.

In Chapter 4, the effects of the repeated heating thermal cycles from the layer-by-layer fabricating process are investigated through characterization of an SLM staircase part. Its influence on the microstructure evolution, texture, and mechanical properties are found.

In Chapter 5, the influence of the build height on the microstructure and the mechanical properties of the SLM-processed Inconel 718 part is found. The relationship between the build height and resulted microstructure, and its resulted microstructure, texture and mechanical properties is obtained.

In Chapter 6, the distribution of the residual stress in the EBAM-produced Ti-6Al-4V parts and SLM-processed Inconel 718 is studied and compared.

In Chapter 7, the effects of the supporting structures during EBAM process are studied by analyzing the evolution of the microstructure and mechanical properties.

In Chapter 8, the microstructure evolution of Inconel 718 in SLM process is simulated using a phase field model. In addition, the beam scanning speed and the build height effects on the microstructure evolution are also predicted using the phase field method.

The last chapter proposes the future work of this project.
References


CHAPTER 2
LITERATURE REVIEW

2.1 Introduction

Powder-bed additive manufacturing is an innovative technique, in which parts are built through melting the metal powder layer by layer. As a developing manufacturing technique, achievement of excellent mechanical properties in the final part is of paramount importance for the mainstream adoption of this technique in industrial manufacturing lines [1,2]. Electron beam additive manufacturing (EBAM) utilizes a high-energy electron beam as a moving heat source to produce parts in a vacuum chamber [3,4]. Therefore, it can be used to process high melting point and reactive metallic materials, such as Ti-6Al-4V alloy for the aerospace and biomedical applications. Selective laser melting (SLM) can produce geometrically complex components with high dimensional precision and good surface integrity using a computer-controlled high energy laser beam as the energy source [5]. As the super alloy parts with complex structures, high dimension precision, and further elevated mechanical properties are on higher demand, the application of the novel non-traditional processing technology is necessary to the net shape production of parts with complex configurations and high performance [6].

However, the desired microstructures of the final AM-produced parts are inevitably affected by complicated physical and chemical behaviors within the molten pool, as a result of the non-equilibrium processing technique of the beam [7]. For example, the rapid thermal cycles induced by the layer-by-layer fusion step in powder-bed fusion systems. Moreover, the previous
layers experience a thermal history during printing and this further introduces a different thermal history for each subsequent layer. These characterizations of the process will lead to the presence of defects, such as porosity, incomplete melting and residual stress in the parts, which contributes to the ultimate mechanical properties of the final part. Therefore, understanding how the AM process influences the part microstructure and mechanical properties are of paramount importance.

In order to ensure the final printed parts for structural applications have outstanding mechanical properties, significant research efforts are still required to focus on microstructures evolution during the manufacturing process and mechanical properties of the fabricated parts under various processing conditions. In this regard, the objective of this work will focus on electron beam additive manufacturing (EBAM) and selective laser melting (SLM) processed, which will be used for fabricating Ti6Al4V and Inconel 718, respectively. The effects of the building condition on the microstructures and associated mechanical properties of the final parts will be investigated. In addition, to achieve the control of microstructure by tailoring the AM machine parameters, the microstructure evolution during the manufacturing processes is modeled and discussed.

2.2 Powder-bed Additive Manufacturing Processes

2.2.1 EBAM Process

A conceptual schematic of an EBAM machine is shown in Figure 2.1 [8]. A heated tungsten filament, in the upper column, emits electrons which are collimated and accelerated to a kinetic energy of about 60 keV. The electron beam is controlled by two magnetic coils, which are housed in the lower column. The first one is a magnetic lens, which focuses the beam to the
desired diameter, and the second one deflects the focused beam to the desired point on a build platform. The electron-beam gun itself is fixed, no moving mechanical parts involved in beam deflections. The beam current is controlled in the range 1 to 50 mA and the beam diameter can be focused down to about 0.1 mm.

![Schematic drawing of an Arcam EBAM machine](image)

Figure 2.1 Schematic drawing of an Arcam EBAM machine [8]

The raw materials used in EBAM are metallic powder and the characteristics and quality of powder strongly affect the process performance. In general, fine powder, with a diameter between 45 and 100 µm, are used. Figure 2.2 shows scanning electron microscopic (SEM) images of Ti-6Al-4V powder: (a) low magnification showing a population of particle sizes and (b) high magnification of an individual powder. In the chamber of the lower part of the machine, the metal powder layers are formed by a raking mechanism. The typical layer thickness is in the range 0.07 to 0.15 mm. The computer-controlled electron beam scans over the powder layer in a predefined pattern and consolidates the desired areas into solid and dense metals. The beam has to first scan at a high speed (order of 2 m/s) in multiple passes to preheat powder to a sintered
state; while a beam scan approximately ~0.5 m/s is used during the melting cycle. A new powder layer is laid on top and the scanning process is repeated until all layers are completed.

The entire process takes place under a high vacuum. The typical pressure of residual gasses in an EBAM machine is 10-1 Pa in the vacuum chamber and 10-3 Pa in the electron gun [8,9]. During the melting process, a low pressure of inert helium gas (10-1 Pa) is added to the vacuum chamber to avoid build-up of electrical charges in powder. When all layers have been completed, the built part is allowed to cool inside the process chamber, which is then filled up with helium as to assist cooling. Because of radiation from electrons, the process observation is not as accessible as other AM technologies, only through a leaded-glass viewport.

2.2.2 SLM Process

For the powder-bed laser AM methods, the precursor powder is melted by a high-energy laser beam in a layer-by-layer manner. Selective laser melting (SLM), embodying the most typical features of the powder-bed laser technologies, has been recognized as a promising AM technology due to its flexibility in feedstock and shapes [10–13]. In SLM, a computer controlled scanning laser beam is applied as the energy source to selectively melt the pre-spread powder particles layer by layer [14,15]. Furthermore, geometrically complex components with high dimensional precision and good surface integrity can be obtained precisely by the SLM process without subsequent process requirements, with which the conventional methods cannot easily keep pace with [5,16]. The SLM process utilizes an inert argon or nitrogen gas as the atmospheric environment. The schematic view of an SLM system is shown in Figure 2.2.
2.3 Microstructure

2.3.1 Ti-6Al-4V

Titanium and titanium alloys have been utilized in numerous applications due to their low density, high strength, and excellent corrosion resistance [18,19]. With the highest strength to density ratio and a high melting temperature (1670 °C), titanium alloys are always selected over other competing metallic materials, such as high-strength aluminum alloys and magnesium alloys [20–22], for many high-temperature aerospace applications. Ti-6Al-4V is by far the most commonly used and extensively studied titanium alloys [23–25], which was developed in 1950 at Illinois Institute of Technology USA. Its microstructures and mechanical properties depend upon the processing history and heat treatment [26]. The microstructures of Ti-6Al-4V mainly consist of hexagonal closed packed (HCP) α phase and some retained body centered cubic (BCC) β phase [27]. Upon the cooling rate and thermal processing history, the final equilibrium state microstructures can be categorized into different groups, grain boundary allotriomorph α, globular or primary α, Widmanstätten α platelets, transformed β phase, and possibly martensitic phase. The solid phase transformation occurs at a temperature of 882 ± 2 °C, which is known as β transus temperature. The β phase transforms into globular type of α at slow cooling rates from
above β transus temperature at a low cooling rate, and into α platelets growing from grain boundary of prior β grain to the inside of grain at moderate to high cooling rates with their length and thickness determined by the cooling rate [25,28]. In addition, at a rapid cooling (quenching) process with a cooling rate above 410 °C/s [29] will generate the martensitic phase, which is enriched in β stabilizing element and either α′ phase or orthorhombic α” phase depending upon the composition of the β phase before quenching. The ageing in the range between the β transus and martensitic start temperature results in decomposition of the α′ martensitic phase into equilibrium α and β phases. Ageing at about 700 °C for about thirty minutes will fully decompose the phase into the equilibrium α and β phases [30,31].

Figure 2.3 displays the typically ordered lamellar microstructure in EBAM-processed Ti-6Al-4V samples. The extremely fine primarily acicular α-plates and Widmanstätten-like (α + β) microstructure were observed under the optical metallography. While the wrought sample shows slightly elongated α grains and intergranular β grains with the size of them are much coarse than that of the EBAM specimens [32]. In addition, some studies also found α′-martensitic platelets or mixtures of α and α′ platelets in EBAM samples, which accounts for the increased hardness and strengths [33]. This can be expected by the thermal characteristics of the EBAM process, such as small melt pool and rapid cooling. There are also studies reporting different microstructures (grain size and morphology) obtained from the EBAM, which may be attributed to noticeably different cooling rates [34,35] and re-melting [36] results from different manufactured conditions.
2.3.2 Inconel 718

The base phase in Inconel 718 alloy is γ phase, also called γ matrix and the major precipitates are disk-shaped γ” phase and spheroidal γ’ phase. There are also some needle/plate-like δ phase, discrete metal carbide (MC) particles and round, island-like Laves phase [37]. γ” phase, having the composition Ni3Nb and a body-centered tetragonal (bct) crystal structure, and γ’ phase, having the composition Ni3(Al, Ti, Nb) and a cubic (ordered face-centered) crystal structure, are the major strengthening phases which are coherent with the γ matrix. An orthorhombic δ phase, having the composition Ni3Nb, always precipitates at grain boundaries in a needle-like form. Laves phases are irregularly shaped phases which form due to Nb segregation with the other alloying elements with a typical composition of (Ni, Fe, Cr)₂(Mo, Nb, Ti), instead of γ” (Ni₃Nb). This phase is detrimental to mechanical properties but it can be dissolved in the matrix by proper heat treatments [38,39].

Within traditional manufacturing methods, such as cast and wrought, the components present coarse grain size and heavy dendritic segregation [40,41], which is caused by the low cooling rate during solidification [42,43], as shown in Figure 2.4d. Solidification defects, such as shrinkage cavities and porosity, also form in the castings at the same time [44–46]. In wrought
products, the formation of macro-segregation, freckles, Laves phase and white spots results in a large scatter in the mechanical properties [47–49]. Thus, homogenization treatment and hot isostatic pressing (HIPing) are required to homogenize the microstructure to close the internal pores and improve the casting quality [50]. However, with the new AM technologies, such as SLM, the specimen shows regular fine microstructure due to the rapid cooling rate induced by high laser energy density, as shown in Figure 2.4(a-c).

Figure 2.4 Optical micrographs of Inconel 718 specimens: (a) SLM, cross section [6], (b-c) vertical section [51,52] and (d) Casting [53]

Under the optical microscope with lower magnifications, the SLM formation features are shown clearly, such as the regular laser melted tracks on the cross-section corresponding to laser
scanning strategy and laminar material structure and columnar architecture throughout the vertical section with geometry determined by the specimen building strategy, such as the scanning pattern, hatch spacing and thickness of layers. The cut ends of melted tracks in the form of a series of arcs on the vertical section induced by the Gauss energy distribution of laser [6]. At higher magnification, it can be seen that regular columnar microstructures composing of parallel dendritic cells grew along the building direction with an averaged dendrite arm spacing around 2 μm on the vertical section of the samples [6], as shown in Figure 2.5(b). Gong et al. [54] also found that strong orientation of columnar structure grew along the build direction and perpendicular to the melted layers determined by the vertical heat flux related to heat transfer into the substrate.

![Figure 2.5 SEM micrographs of As-deposited SLM IN718 samples: (a) cross section and (b) vertical section [6]](image)

However, the cross section of the samples exhibits a typical equiaxed grain microstructures, as shown in Figure 2.5(a), which is caused by horizontal heat flux related to the movement of the heat source. Therefore, similar to the directionally solidified microstructure,
such type of microstructure is a result of epitaxial, dendritic grain growth in the direction determined by heat flux direction and crystallographically favored orientation [55]. If viewed in a three-dimensional section, the grains would be in a rod-shape, which has also been mentioned by Amato et al. [52] and Gong et al. [27].

2.3.3 Effects of manufacturing conditions

The manufacturing conditions, such as the parts’ configuration and the operating parameters adopted during the manufacturing process [34,35], have significant impact on the thermal stories of the final parts and further influence part characteristics [56–59], quality consistency and process performance.

Based on the study the effects of beam power, diameter, and speed, as well as the preheat temperature in EBAM processing of Ti-6Al-4V from Murr et al. [34], the variations in melt scan, beam current, and scan speed affect the defects in the final parts, such as porosity, and may cause significant property-performance variations. Gaytan et al. [60] found that the differences in melt-scan parameters result in variations of microstructure characterizations, such as grain sizes (dimensions), the composition ratio of phases and dislocation density variations [60]. That is because variations in the beam power and speed can alter both solidification cooling rates and thermal gradients by several orders of magnitude, which have a significant effect on resulting microstructures, as reported by Bontha et al. [35]. Rapid self-cooling, which results in the EBAM layer building/bonding mechanism, has been noted to be different upon comparing different melt-scan parameters. Results specifically for Ti-6Al-4V suggest that process size-scale can have a significant effect on microstructures, including a transition from columnar to equiaxed microstructure at higher powers.
For the manufacturing process of Inconel 718 using SLM, Jia et al. [7] revealed that with an increase of laser energy density, the microstructures of Inconel 718 will change successively in the order of coarsened columnar dendrites, clustered dendrites, to slender and uniformly distributed columnar dendrites. Generally, at high cooling rates, macrosegregation would be completely prevented and microsegregation of the chemical composition in Inconel 718 could occur during the rapid dendritic/cellular growth of columnar grains resulting from the primary dendrite shape altered to a more cell-like form at a high cooling rate [58]. As shown in Figure 2.6, Laves and MC-type carbides are observed between aligned dendritic cells and in the region of an overlapping interface between two adjacent laser scanning tracks or layers. By EDS, Parimi et al. [38] also observed white irregularly shaped Laves phases with size of ~1–2 μm in the interdendritic regions, as shown in Figure 2.6(c-d), and Want et al., [6] found δ phase and Laves phase at grain boundaries, as shown in Figure 2.5(b).

In general, the δ phase cannot be precipitated during the process of SLM due to the high cooling rate in SLM and the small content of Nb caused by the occurrence of abundant Laves phase. But, the precipitation of δ phase occurs following aging for less than 100 h at a temperature range of 750 °C to 1000 °C with maximum precipitation at 900 °C at the grain boundaries [61]. Although δ phase is generally detrimental to the mechanical properties, the proper morphology of these precipitates at grain boundaries could improve the creep property of these materials [62].
The desirable microstructure is a stable $\gamma$ phase strengthened with coherent and dispersive precipitates of the phases $\gamma'$ and $\gamma''$ as IN718 parts are supposed to work in a strongly corrosive environment at low and high temperatures. The non-equilibrium structure was obtained and macrosegregation of alloy elements was sufficiently inhibited due to the inherent rapid solidification rate associated with laser deposition. To enable the precipitation of strengthening $\gamma'$ and $\gamma''$ phases and relieve the residual stresses [63,64] post heat treatment is necessary. After solution and double aging heat treatment, Wang et al. [6] found that there are numerous niobium-rich precipitates ($\delta$ and Laves phases) along the developed curved grain boundaries on both
sections with the regular dendritic crystals demolished due to reheating, diffusion and recrystallization, as shown in Figure 2.7(a). Generally, the δ phase precipitated at grain boundaries with the dissolving of Laves, γ′ and γ” phases dissolving in the matrix in the solution treatment at 980 °C, and then γ′ and γ” phases dispersed and precipitated further in the matrix to strengthen the alloy in the process of double aging treatment. Similar results have obtained in annealed samples by Amato et al. [52] recrystallized grain boundaries developed with precipitating of δ phase, γ” phase precipitates parallel to the laser beam or build direction after hot isostatic pressed (HIP) treatment and γ′ precipitates distributed in a dense field of fine γ” precipitates in the ~50% recrystallized regions after annealed (1160°C for 4 h) treatment.

Table 2.1 Detail of the heat treatment methods used in the studies

<table>
<thead>
<tr>
<th>Types</th>
<th>Heating Methods</th>
<th>Heat Treatments</th>
<th>References</th>
</tr>
</thead>
<tbody>
<tr>
<td>SLM</td>
<td>Aging</td>
<td>720 °C/8 h (FC/100°C/h) + 620 °C/10h (AC)</td>
<td>Chlebus et al. (2015)</td>
</tr>
<tr>
<td></td>
<td>Solution + Aging</td>
<td>980/1040/1100 °C/1h (WC) + 720 °C/8 h (FC/100°C/h) + 620 °C/10h (AC)</td>
<td></td>
</tr>
<tr>
<td>SLM</td>
<td>Solution + Aging (double)</td>
<td>980 °C/1h (AC) + 720 °C/8 h (FC) + 620 °C/8h (AC)</td>
<td>Wang et al. (2012)</td>
</tr>
<tr>
<td>SLM</td>
<td>HIP</td>
<td>1163 °C(0.1GPa)/4h</td>
<td>Amato et al. (2012)</td>
</tr>
<tr>
<td></td>
<td>Annealing</td>
<td>1160 °C/4h</td>
<td></td>
</tr>
<tr>
<td>LRF</td>
<td>Homogenization +</td>
<td>1080 °C/1.5 h (AC) + 980 °C/1h (AC) + 720 °C/8 h (FC) + 620 °C/8 h (AC)</td>
<td>Zhao et al. (2008)</td>
</tr>
<tr>
<td></td>
<td>Solution + Aging</td>
<td></td>
<td></td>
</tr>
<tr>
<td>DLD</td>
<td>Solution + Aging</td>
<td>965 °C/1h (WC) + 718 °C/8 h (FC, 56°C/h) + 620 °C/8h (AC)</td>
<td>Zhang et al. (2011)</td>
</tr>
<tr>
<td>DLD</td>
<td>Solution + Aging</td>
<td>980 °C/1h (AC) + 720 °C/8 h (FC) + 620 °C/8h (AC)</td>
<td>Blackwell et al. (2005)</td>
</tr>
<tr>
<td>LNSM</td>
<td>Homogenization +</td>
<td>1093 °C/1.5 h (AC) + 982 °C/1h (AC) + 718 °C/8 h (FC) + 621 °C/10 h (AC)</td>
<td>Qi et al. (2009)</td>
</tr>
<tr>
<td></td>
<td>Solution + Aging</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Chlebus et al. [51] also compared the effect of different heat treatments and the conclusion is subsequent heat treatment is necessary for the SLM-processed IN718 alloy with parameters used in solution and double aging heat treatment determined by the as-deposited microstructure. The microstructure maintained its columnar nature and traces of layered build of the material after direct aging, as shown in Figure 2.7(b). From the Figure 2.7(c), it can be seen that residual particles of the Laves phase and δ phase occur on grain or subgrain boundaries and along the layer interfaces, which states that the solution annealing parameters used appears insufficient for complete homogenization of the γ phase. Complete dissolution of residual Laves particles requires the proper selection of temperature and time of the annealing.

As can be seen in Figure 2.7(d), homogenization of γ solid solution occurred (1100 °C/1 h + aged) obtained with boundaries decorated with MC carbide. This heat treatment variant was found optimal from the viewpoint of satisfying the degree of microstructure homogenization and aging effects. Thus, the recommend heat treatment is using higher temperature (i.e. 1100 °C) than that normally used to homogenize the alloy and slow furnace heating to avoid local subsolidus liquation of the material and possible propagation of metastable liquid along grain boundaries. This also has been mentioned by Qi et al. [65], who also studied the microstructure of Inconel 718 under different heat treatment conditions of as-deposited, direct aged, solution treatment and aging (STA), and full homogenization followed by STA. The homogenized STA heat treatment can completely dissolve the Laves phase but enable substantial grain growth with isotropic appearance.
Chlebus et al. [51] deduced that the driving force is not likely the uneven distribution of residual thermal stresses [66] but more probably the higher grain boundary migration, which is much higher than that of the traditionally manufactured alloy. Because the microstructure in the as-deposited parts is unstable due to high energy accumulated in the form of grain boundaries and dislocations. The detail of the heat treatment methods used in the studies is listed in Table 2.1.

Figure 2.7 Micrographs in vertical section of SLM-processed IN718 samples with heat treatment: (a) Solutioned (980 °C/1 h)+ Aged [6], (b) Aged, (c) Solutioned (1040 °C/1 h) and (d) Solutioned (1100 °C/1 h) and aged [51]

To avoid these internal stresses, one can reduce the temperature gradients by heating the substrate plate or increasing the temperature inside the processing chamber [67,68]. Using a
double-scanning strategy will also minimize the level of residual stresses in the parts [1].
Moreover, the post-processing heat treatment [69,70] could be adopted to relieve the residual
stress in the final components.

2.3.3 Defects

Even though the powder-bed AM technologies have a lot of advantages compared to
traditional machining methods, it also has disadvantages, such as pores, unmelted powder and
bonding defects. The pores in the volume of laser-based AM parts, which affect the density and
porosity of the samples, stem from process-induced defects originating from initial powder
contaminations, evaporation, or local voids after powder-layer deposition.

Figure 2.8 presents the typical pores in the EBAM-processed Ti-6Al-4V parts. The small
spherical gas voids or porosities usually have a small void size (order of 10 µm), and they may
not impact the mechanical properties of EBAM-built parts [71,72]. However, the spherical or
irregular pores are supposed to be detrimental to fatigue properties [73,74]. Some gas bubbles
actually came from the producing of the powder particles or the recycled powder, which also has
been reported in SLM processed parts. Wang et al. have observed some micro-pores on both
cross-section and vertical section, as shown in Figure 2.5(b). Zhang et al. [70] found pores with
a spherical shape present in the deposits. The spherical-shape pores are most likely entrapped gas
bubbles in the molten metal bead that could not rise and escape to the top surface before
solidification. The porosity is probably entrapped from the GA process during powder
manufacturing [75]. Most of the powder particles have a spherical shape with internal porosity
infrequently observed in them, and very fine satellite particles attached to them [38]. Another
important factor causing the porosity is the laser’s high scan speed, as reported by Jia et al. [7].
During the process of manufacturing, the influence of high viscosity at a high scan speed (600 mm/s) impeded the Inconel 718 liquid from spreading out smoothly, which in turn caused the formation of open pores, as seen in Figure 2.9(a).

![Micrograph of EBAM produced Ti-6Al-4V alloy](image1)

Figure 2.8 Micrograph of EBAM produced Ti-6Al-4V alloy, (a) [34] and (b) [76]

![Porosity defects observed](image2)

Figure 2.9 Porosity defects observed in the SLM-processed of Inconel 718 at various laser densities: (a) 180 J/m, (b) 275 J/m, (c) 330 J/m [7]

Besides, the linear laser energy densities (η) also plays an important part in the surface finishes as it has a critical effect on the dynamic viscosity and balling phenomenon. By comparing Figure 2.9(a) to (c), it can be seen that the smooth sound surface morphology without
any pores or balls was obtained with the $\eta$ increasing properly to 330 J/m, due to the weakened dynamic viscosity and balling phenomenon. Niu and Chang [77] has studied the instability the scan tracks and mentioned that the balling phenomenon will occur in high energy density processes. Due to the presence of the steep thermal gradient between the center and edge of the melt pool at the surface, a variation of the surface tension, which is a function of temperature, will occur between them and further induce a Marangoni flow from a region of low surface tension to a region of high surface tension. This fluid flow will produce an extra force exerted on the molten track of the samples, which results in breaking up of the fluid to smaller volumes, such as a row of spheres, to minimize surface free energy. Also, the protective gas, argon, may be encapsulated inside the pores, with which the chamber was filled in order to avoid contamination of the processed metal with oxygen and nitrogen during AM processing.

Eventually, these pores will act as strong stress raisers and finally lead to failure, especially under fatigue loading. Right now, these pore-like defects cannot be totally avoided. However, the porosity can be greatly reduced by using PREP powder [65,75] or fine size GA powder combined with a high $\eta$ (laser power/scanning speed) [65]. The balling phenomenon can be inhibited by higher $\eta$ when the laser wavelength $\lambda < 2\pi R$, where $R$ is the initial radius of an unperturbed liquid cylinder. At a constant laser wavelength $\lambda$, lower scan speed accompanied with higher energy input could lead to a rapid increase of radius $R$ of the cylinder, which will weaken the instability of the liquid cylinder. Therefore, at a high $\eta$, there is no need for the liquid to alter its shape to reduce the surface energy and discontinuous scanning tracks will disappear. Another method is HIPing of the final solid components, which is used to achieve reduction of pore size or even the closure of these features in traditional powder metallurgy. However, the complete closure of these pores will be hard to achieve with these established techniques.
The second defect is unmelted powder particles. Some particles (similar in size to the powder particles applied) may appear on the top surface or at the boundary of the deposited layers. Up to the melting conditions, these particles may have good metallurgical bonding or lack of bonding with the deposited metal, which might result from spattering.

2.4 Mechanical Properties

Mechanical properties of AM parts have been frequently investigated. The studies indicate that mechanical properties of AM parts are comparable with cast or wrought counterparts. It is also indicated that processing parameters, such as power and speed, may cause the remarkable change in the mechanical properties [38,74,75,78]. In addition, the properties will have a significant change after the post-heat treatment, such as aging, solution, and HIPing.

2.4.1 Tensile Testing

Tensile testing has been widely used to characterize the mechanical properties of EBAM Ti-6Al-4V parts. Facchini et al. [79] found that the ultimate tensile strength (UTS) of EBAM built specimens is higher than the wrought or annealed ones, with a lower ductility. However, Koike et al. [80] reported that both the UTS and ductility of the cast and wrought Ti-6Al-4V specimens are higher than those of EBAM counterparts which have an average yield strength (YS) of 735 MPa, UTS of 775 MPa and a ductility of 2.3% elongation. This could result from the variation in the operating parameters, which leads to different structures, such as composition, structures, pore size, and porosity distribution. The comparison of the mechanical properties of EBAM, cast and wrought Ti-6Al-4V parts are shown in Figure 2. 10.

28
For the Inconel 718 alloy, some studies reported that the tensile properties of SLM specimens are comparable with the wrought Inconel 718 alloy [6,52] while others presented that the tensile strength and yield strength of the as-deposited samples are inferior to the typical wrought, while the elongations are relatively high [51,65,75]. The detailed comparison of the mechanical properties of laser-based AM technologies versus wrought Inconel 718 is shown in Figure 2.11. It can be seen that the tensile strength of the as-deposited parts is comparable or even superior to the casting samples but inferior to wrought ones. Same is the case with the elastic modulus. It is quite comparable to that of the wrought counterparts. As SLM is a totally/fully powder melting manufacturing process, it can be seen that the yield strength and ultimate tensile strength of the Inconel 718 samples built with SLM [6] are superior to that from the other three laser-based manufacturing technologies, LNSM, LRF and DLD. In addition, the LNSM built parts [66] has a slightly lower yield strength, which is caused by the dynamic heat transfer of the moving heat source and the layered material formation mechanism. During the building process, a thin nucleation zone at the layer interface is formed as the molten pool solidifies, which will grow into fine grains at the boundaries. As these layer interface regions are
usually associated with sharp changes in grain size and degree of micro-segregations, they can be the sites of weakened tensile stress.

Figure 2.11 Mechanical properties of Inconel 718

The heat treatment has a significant effect on the tensile strength of the AM-built Inconel 718 parts. Based on the studies of Chlebus et al. [51], the heat treatment will cause the yield
strength, tensile strength, and the hardness to increase by 84% (72 ~ 95%), 38% (30 ~ 46%), and 48%, respectively. According to Zhao et al. [75], the tensile strength of the heat treated components increased approximately 1.5 times that of the as-deposited samples and is comparable with that of wrought Inconel 718, which has also been reported by Qi et al. [65]. The detailed tensile properties of Inconel after post-heat treatment are shown in Figure 2.12. However, the ductility of the samples was reduced significantly due to fine Laves particles that remained at the interdendritic regions [75], as shown in Figure 2.13. After homogenization treatment, the alloy presents slightly lower tensile strength of 1194 MPa, but even better elongation of 19.9% [65]. Through the tensile test, Chlebus et al. [51] also studied the effect of orientation on tensile strength, and he found that there is no significant difference for the B, C, and D series because of the large fraction of grains with small shape aspect ratio in the microstructure. The series A has the lowest tensile strength, which is caused by the perpendicular orientation of overlapping interfaces between layers to the loading direction and the weaker strengthening of the grain boundaries.
Figure 2.12 Strengths of Inconel 718 after heat treatment

Figure 2.13 Elongation compared with after heat treatment
2.4.2 Hardness Testing

Some hardness tests, such as Vickers test and Rockwell test, are also applied to evaluate the hardness of laser-based AM components. The study from Chlebus et al. [51] showed the hardness increased 48% after heat treatment. Amato et al. [52] and Wang et al. [6] have the same findings. According to Amato et al. [52], the microindentation (Vickers) hardness was 3.9 GPa for the as-fabricated materials, 5.7 GPa for the HIP material, and 4.6 GPa for the annealed material. This also has been reported by Zhao et al. [75], The average hardness for heat-treated samples is 3.9 GPa (HRC 41), which has increased remarkably from 2.3 GPa (HRC17) for as-deposited samples. Wang et al. [6] have also found that the SLM-processed parts showed direction independence without large fluctuations in various directions. Zhang et al. [83] reported that the hardness of the layer produced by laser direct metal deposition (DMD) did not show obvious differences in the height of the layer, either for the as-deposited layer or for the heat-treated layer. The detail of the hardness test results is listed in Table 2.2. Generally, it can be seen that the hardness of the as-deposited parts processed with SLM has higher hardness values than the parts built with other laser technologies, LRF and DMD. However, there is no big difference among them after post-heat treatments.
Table 2.2 Hardness of Inconel 718 parts built with laser-based technologies

<table>
<thead>
<tr>
<th>Ref.</th>
<th>Methods</th>
<th>Test</th>
<th>Values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Amato et al. 2012</td>
<td>SLM</td>
<td>Vickers</td>
<td>3.9 GPa</td>
</tr>
<tr>
<td></td>
<td>SLM + HIPing</td>
<td>Vickers</td>
<td>5.7 GPa</td>
</tr>
<tr>
<td></td>
<td>SLM + Annealed</td>
<td>Vickers</td>
<td>4.6 GPa</td>
</tr>
<tr>
<td>Wang et al. 2012</td>
<td>SLM</td>
<td>Vickers</td>
<td>3.58 GPa (Hv 365)</td>
</tr>
<tr>
<td></td>
<td>SLM+HTed</td>
<td>Vickers</td>
<td>4.61 GPa (Hv 470)</td>
</tr>
<tr>
<td>Chlebus et al. 2015</td>
<td>SLM</td>
<td>Vickers</td>
<td>3.07 GPa (Hv1 313)</td>
</tr>
<tr>
<td></td>
<td>SLM+HTed</td>
<td>Vickers</td>
<td>4.54 GPa (Hv1 463)</td>
</tr>
<tr>
<td>Jia et al. 2014</td>
<td>SLM</td>
<td>Vickers</td>
<td>3.88 GPa (Hv0.2 395.8)</td>
</tr>
<tr>
<td>Murr et al. 2012</td>
<td>SLM</td>
<td>Vickers</td>
<td>5.6 GPa</td>
</tr>
<tr>
<td>Zhao et al. 2008</td>
<td>LRF</td>
<td>Rockwell</td>
<td>2.3 GPa (HRC 17)</td>
</tr>
<tr>
<td></td>
<td>LRF + Heated</td>
<td>Rockwell</td>
<td>3.9 GPa (HRC 41)</td>
</tr>
<tr>
<td>Zhang et al. 2011</td>
<td>DMD</td>
<td>Vickers</td>
<td>2.94 GPa (Hv 300)</td>
</tr>
<tr>
<td></td>
<td>DMD + Heated</td>
<td>Vickers</td>
<td>4.90 GPa (Hv 500)</td>
</tr>
</tbody>
</table>

2.4.3 Other Testing

Some other studies have been conducted to investigate the properties of Inconel 718. Jia et al. [84] have investigated the oxidation behavior by an isothermal oxidation test. The result shows that the oxidation kinetics of SLM-processed Inconel 718 parts follows the parabolic law and shows a good high-temperature oxidation performance when the applied volumetric energy density increases from 70 to 130 J/mm³. This is contributed to the refined and uniformly distributed microstructures and the improved relative density of the parts. Besides, Jia et al. [84] also conducted the wear tests with the conclusion that the Inconel 718 parts have a considerably
low friction coefficient of 0.36 without any apparent fluctuation and a decreased wear rate of 4.64×10×4mm3/N m. The improvement of the wear performance was attributed to the influence of elevated microhardness in combination with the formation of adherent tribo layers.

2.5 Conclusions

Powder-bed additive manufacturing technologies, such as electron beam additive manufacturing (EBAM) and selective laser melting (SLM), can efficiently assist in producing complex, difficult-to-fabricate components. However, the EBAM and SLM process are complex, in which the process parameters have critical effects on the characteristics of the final parts’, and still not well understood. There have been various studies of EBAM build Ti-6Al-4V and SLM-processed Inconel 718. This study intends to further understanding the EBAM and SLM technologies by review the operating principles, microstructures and mechanical properties of the final parts. The collected and analyzed information from this review is summarized as below.

(1) It is recommended to use PREP (plasma rotating electrode processed) powder or fine GA powder (Gas-atomized) in order to achieve acceptable mechanical properties.

(2) In general, EBAM-processed Ti-6Al-4V parts have fine microstructures with some porosity, which can be mostly eliminated by HIP. The as-deposited Inconel 718 parts fabricated with selective laser manufacturing (SLM) present a fine columnar dendrites structure with internal micro-segregation in inter-dendritic regions.

(3) The EBAM Ti-6Al-4V parts typically have comparable (to wrought counterparts) or superior mechanical properties, high YS and UTS and higher hardness, but a lower ductility. The tensile strength of the as-fabricated Inconel 718 parts is comparable or even superior to the casting samples but inferior to wrought ones.
(4) The heat treatment has a critical effect on the microstructure and mechanical. The desirable microstructure can be obtained by proper post-heat treatment. After post-heat treatment, the yield strength, tensile strength, and the hardness of the parts would have a remarkable increase about 84%, 38%, and 48%, respectively, which are comparable (or superior) to the wrought ones, but the ductility of the samples would have a large drop (-21.5%).
References


[71] Gaytan, S. M., Murr, L. E., Martinez, E., Martinez, J. L., Machado, B. I., Ramirez, D. A.,


CHAPTER 3

PROCESS PARAMETRIC EFFECT ON THE FINAL PARTS: BEAM SCANNING SPEED

3.1 Introduction

EBAM has several unique advantages, such as high energy efficiency and rapid scan speed, which is most appropriate for small production runs with expensive materials and is consequently largely of interest to the aerospace and biomedical industries [1]. Even though EBAM shows various advantages over traditional manufacturing technologies, it still exhibits several process challenges, such as part accuracy and property scattering [2].

Microstructures of EBAM Ti-6Al-4V samples contain a mixture of phases such as α (HCP), β (BCC) and α’ martensite (HCP). Wang et al. [3] noted columnar prior β grains in the longitudinal section of the samples due to the high thermal gradient along the Z axis. The columnar prior β grains contains grain boundary α and transformed α + β structure which includes lamellar colony and basket weave (also called Widmanstätten pattern) morphology. This also has been reported by Sirdharan et al. [4] In addition, it is also reported that only α phase and a relatively small fraction of β phase exist in the present plate [5,6]. Compared to cast samples which are characterized by a coarse acicular (α+β) and thick prior β grain boundaries, the microstructure of the EBAM specimen consists of fine acicular (α+β) and thin prior β grain boundaries [7]. The thickness of the α-lath is around 1.4-2.1 µm for different EBAM samples [8]. In addition, α’-martensitic platelets exist in EBAM parts [9], which may contribute to the increased strength and hardness but lower ductility [10]. In summary, the EBAM-processed
Ti-6Al-4V samples display a fine Widmanstätten (α+β) microstructure and α’ phase due to the thermal characteristics of the EBAM process, such as a small melt pool and a rapid cooling process. However, the AM machine parameters during manufacturing process have critical effects on the thermal cycling duration, which affects the microstructure in the final parts [11].

Several studies have investigated the effects of the process parameters. Based on the experimental results of Murr et al. [8], small deviations from optimum build parameters, such as beam current and scan rate, will lead to the built defects, including unmelted or unsintered powder regions which maybe account for microstructure-property variations in the final product. Jamshidinia et al. [12] developed a coupled Computational Fluid Dynamic (CFD) – Finite Element Method (FEM) model to study the heat and thermal stress distribution in EBM. He found that heat input and cooling rate are the dominant factors influencing the level of thermal stresses developed during melting and cooling steps, respectively. In our group, Gong et al. [13] also studied the effect of the beam scanning speed on the microstructure evolution. Despite research on modeling and experiments of EBAM, there are few systematic studies have been carried out on the relationship between the scanning speed and the mechanical properties of the EBAM parts.

The objective of this experimental study is to better understand the effect of the beam scanning speed on the texture of the microstructure and the mechanical properties in EBAM built Ti-6Al-4V parts. A commercial EBAM system was used to fabricate specimens with different speed functions, which are related to beam speed during the same build cycles.
3.2 Experimental Procedure

To study the beam speed effects on the final build parts, 4 rectangular blocks were fabricated at four different speed functions (SFx) (SF20, SF36, SF50 and SF65) by an Arcam S12 EBAM machine located at NASA’s Marshall Space Flight Center (Huntsville, AL) using fine gas atomized (GA) Ti-6Al-4V powder with a diameter between 45 and 100 µm. To identify the phases in the raw powder, X-ray diffraction (XRD) test was carried out with a XPERT Pro-type diffractometer and Cu as the anode material. All the blocks have identical dimensions of 60 mm length, 5.5 mm width, and 25 mm height, and they were built layer by layer with a thickness of 70 µm. The detail of the manufacturing process can be seen in our former studies [13]. The final finishing part built with SF50 is shown in Figure 3.1a, in which both the side surface (Y plane) and the scanning surface (Z plane) have been studied to examine the anisotropic conditions in the microstructure, as indicated by specimen 1 and specimen 2 in the Figure 3.1a, respectively.

![Figure 3.1 Ti-6Al-4V alloy: (a) As-fabricated EBAM block built with SF50, and (b) XRD patterns obtained for the raw powder](image)
The cut samples were prepared using standard metallographic procedures, which includes sectioning, mounting, grinding and polishing. To reveal the microstructures, the final polished specimens were etched with a hydrofluoric acid-based solution, which contains 1 mL hydrofluoric acid (50 wt. %) and 3 mL (68 wt. %) nitric acid in 20 mL distilled water. The etched metallographic samples were examined using a Keyence VHX-5000 digital microscope, and a JEOL 7000 FE scanning electron microscope (SEM). To characterize of the texture of the samples, Electron beam backscatter diffraction (EBSD) measurements were performed using a JEOL 7000 FE-SEM equipped with a detector for EBSD. EBSD samples were final polished by a vibratory polisher using colloidal silica (0.05 μm). The AZTEC data acquisition software from Oxford Instruments was used in the test. The texture analysis was conducted at a beam voltage of 20 kV with a scanning area of 500 μm by 500 μm in 0.75 μm steps. Post processing of the data was done using orientation imaging microscopy (OIM) software from EDAX.

The nanoindentation tests were performed using an in-situ nanomechanical testing system Hysitron TriboindenterTM equipped with a Berkovich tip with a radius of 100 nm and an included angle of 142.3°, as shown in Figure 3.2. The maximum load is 10.0 mN with a resolution of 1 nN, and the maximum depth is 20 μm with displacement resolution of 0.04 nm. An open-loop trapezoidal shape load function with maximum load of 5000 μN was applied to the specimens during nanoindentation test, as illustrated in Figure 3.2c. In trapezoidal shape method, the loading force on the indenter increases at a constant load rate until reaching maximum value with a dwell time of 10 seconds, then followed by a constant unload rate until reaching zero. To obtain the precise results, several nanoindentation tests were conducted on different areas of the specimen. Each run of the tests consisted of a series of 25 indents arranged in a 5 × 5 square pattern with 5 μm spacing between indent locations. The spacing ensured that the stress-strain
fields of previous indents did not affect the behavior of any subsequent indent in the series. The sample and tip were allowed to come to thermal equilibrium over 30 min before each test.

![Triboindenter](image)

Figure 3.2 Triboindenter (a) appearance and (b) nanoindentation setup, and (c) applied load function

3.3 Results and Discussions

3.3.1 Microstructures

The typical optical morphology and microstructure of EBAM Ti-6Al-4V under a microscope are shown in Figure 3.3. The Y-plane presents columnar solidification structures along the Z-axis and their length might go through multiple layers in the sample. When the temperature was above the β-transus temperature (about 980 °C) at initial rapid solidification, the nucleation and growth of columnar grains of prior β took place, which typically proceeds in a very stable manner due to the narrow solidification range. They nucleate in the fusion zone towards the former solidified layer, build upon those crystals with the same orientation and growth direction, and grow along the steepest temperature gradients depending on the rapid cooling from the melt. This process combines epitaxial growth mechanism and layered melting deposition techniques [14]. This results in a columnar shaped morphology with their length go
through multiple layers as crystallographic orientation is inherited across layers [15]. However, the Z-plane is full of thoroughly equiaxed grains, as shown in Figure 3.3b.

Figure 3.3 Typical EBAM Ti-6Al-4V microstructure under optical microscope (a) Y-plane and (b) Z-plane

The microstructures of the Z-plane under the SEM with a higher magnification are shown in Figure 3.4, in which the β, α and α′ phases can be identified. In solid phase transformation, the prior β columnar grains are transformed into fine α laths or α′ solid phases depending on the cooling rate. The (α+β) structure is formed by a diffusion controlled solid phase transformation, in which V diffuses into β while Al diffuses into α [16]. Due to the preferential growth of the phase parallel to the (110) family of crystallographic planes of the β phase, a fine Widmanstätten (α+β) structure is formed inside of large, equiaxed, previous β grains following the Burgers relation, indicating a rapid cooling rate in EBAM. Further, the classical α-lath structure is the primary phases in Ti-6Al-4V, and only a very small amount of β phase was revealed in the α boundaries. This also can be seen from the XRD pattern for the raw powder shown in Figure 3.1b, in which an asterisk represents α-Ti. There are also some plates-like martensitic phase, α′, which was transformed from the β phase due to a very high cooling rate [17]. The α′ phase is
commonly observed in Ti-6Al-4V alloy subject to rapid solidifications such as selective laser melting [18] and electron beam welding [19,20].

![Figure 3.4 Microstructure on Z-plane of Ti-6Al-4V under scanning electron microscope](image)

3.3.2 Texture analysis

Figure 3.5 includes the typical EBSD results. The image quality (IQ) maps have a good overall quality, as can be from Figure 3.5a,d. The SF20 Y plane (Figure 3.5b) presents the basket weave microstructure showing in the inverse pole figure (IPF) with an averaged grain size of 5.65 µm. Because the α-laths in the colonies share the same orientation leading to low-angle boundaries between the laths and their neighboring laths and thus makes up one grain [21], as shown in the grain boundary map in Figure 3.5c. The blue lines stand for a misorientation angle over 15°, named high-angle grain boundaries, which could strongly affect the residual stress [22–24] and distribution of the cracks [25,26] in the as-fabricated material. This could be improved by performing heat treatment.

The IPF from SF36 Y plane (Figure 3.5e) shows columnar microstructure, which is commonly observed. The secondary dendrites can be seen clearly in Figure 3.5e, which resulted from the high cooling rate due to the fast-cooling solidification process in EBAM process. The
analysis of the microstructure above has revealed that the $\alpha$-lath and acicular $\alpha'$-lath structure precipitated within elongated columnar grains of the parent $\beta$ phase formed along the build direction. The boundary of the columnar $\beta$ structure can be seen clearly in Figure 3.5a,d. Based on the size of the dendrites, the conclusion can be made that one grain is filled with many dendrites, as the dendrites forming low angle grain boundaries with neighboring dendrites during columnar solidification. Two distinct morphologies, equiaxed and lamellar, of the $\alpha$ phase are clearly shown at these regions, which is the standard bimodal microstructure of Ti-6Al-4V alloy.

Figure 3.5 As-fabricated EBAM Ti-6Al-4V samples and typical EBSD data
Figure 3.5h shows the $\alpha$ laths growing from the equiaxed $\beta'$ grain boundaries toward its interior [27]. Figure 3.5i is the phase map corresponding to Figure 3.5h. The statistic results show that beta phase takes about 1.4 percent fraction of the total scanned area. The $\beta$ phase presents in a discontinuous form because the width of $\beta$ laths is about 60 nm (Figure 3.4b) and only larger $\beta$ phase areas at triple points can be detected due to the limitation of the step size in SEM.

Figure 3.6 includes the EBSD data from the Y plane of the samples. A morphology of colony or basket weave microstructure presents within the $\beta'$ columnar structure (Figure 3.6a-d). Most of $\alpha$ grains in the Y plane of SF20 shows a preferred orientation of $<0001>$ direction (Figure 3.6g) with a strength of texture 13.5 times of random, which might be caused by the lack of variant selection during the phase transformation from the high-temperature $\beta$ to the room-temperature $\alpha$ and $\beta$ phases [4]. The elongated pre-solidified $\beta'$ grains have preferential growth directions leading to the presence of a fiber-like texture. Then, the texture of the $\beta'$ phase is transmitted to a texture of $\alpha$ phase in diffusion-controlled solid phase transformation from $\beta'$ phase to $\alpha$ and $\beta$ phases.

With the increase of the beam speed, the texture of $\alpha$ phase becomes weak and more random. This results from the increased fraction of $\alpha'$ phase [13], which has more random crystallographic orientations due to the relatively high number of $\alpha'$ variants within each $\beta'$ grain [28]. The appearing of $\alpha'$ leads to a lack of partitioning of alloying elements and this results in a non-noticeable contrast of the SF50 IQ image (Figure 3.6e) compared to SF20 (Figure 3.5d) and SF36 (Figure 3.5g). In addition, the dark IQ map indicates a poor relative image quality, which attributes to the higher internal strains associated with the $\alpha'$ formation that would degrade the
quality of the EBSD image obtained at these analysis points [29]. With the increase of the speed function, the texture will decrease and disperse randomly at several points (Figure 3.6g-j).

Figure 3.6 EBSD data from Y plane of EBAM Ti-6Al-4V parts
Figure 3.7 presents the EBSD data from the Z plane of the as-deposit Ti-6Al-4V samples. The surfaces are characterized by equiaxed β' grains with a basket weave and colony microstructure within them (Figure 3.7a-d). Generally, most α grains in the Z plane present several preferred orientation directions and show a restively stronger orientation of $<10\bar{1}0>$ (Figure 3.7g-j). The intensity of the texture would decrease first with the increase of the SF and then increases, which will enhance the mechanical properties of Ti-6Al-4V alloy in varied directions. The SF20 specimen shows a strongest preferred orientation of $<0001>$ and SF50 has the weakest texture in the 4 samples. The Same changing trend for the mechanical properties and the average grain size (Figure 3.8). The average grain size is 3.06 μm to 5.65 μm for the X-plane, and 2.97 to 4.79 μm for the Z plane, which falls in line with former findings [30]. So the finest grain sizes and the weakest texture intensity in SF50 more likely contribute to its highest hardness and elastic modulus.
Figure 3.7 EBSD data from Z plane of EBAM Ti-6Al-4V parts
Figure 3.8 Average diameter of grains of Ti-6Al-4V alloy

The typical grain boundary maps and the statistic results obtained from the EBSD tests are illustrated in Figure 3.9. In Figure 3.9a-b, different colors represent different boundary rotation angles. It can be seen that ninety percent of the boundary angles are above 15°, which is also called high-angle grain boundaries. It is known that a high-level grain misorientation angle at the grain interfaces will lead to the instability of the microstructure due to the high energy accumulated in the form of grain boundaries or dislocations. It may act as the driving force of recrystallization during the post heat treatment process, which is a necessary process to obtain the needed homogenized microstructure. The migration of the higher grain boundary was induced by the reduction of the grain boundary area itself, discontinuous dissolution of columnar arrayed phases and solute diffusion along the boundaries [31]. In addition, the presence of high-angle grain boundaries could strongly affect the density [32], residual stress [33] and distribution of the cracks [25] in the as-fabricated material. Thus, the as-fabricated builds have to be improved by performing heat treatment as they generally have these defects.
3.3.3. Mechanical Properties

An example of the plots of load vs. depth from nanoindentation test on a small area of the Z-plane SF 36 is shown in Figure 3.10. The average maximum indentation depth was about 205 nm, and Young’s modulus of the sample could be calculated by:
\[
\frac{1}{E_r} = \left(\frac{1 - v^2}{E}\right)_{\text{specimen}} + \left(\frac{1 - v^2}{E}\right)_{\text{indenter}} \quad (3.1)
\]

where \(E\) and \(v\) are the elastic modulus and Poisson’s ratio of the specimen and the indenter, respectively. For Berkovich tip, \(E_i=1140\) GPa and \(v_i=0.07\). The hardness was obtained as the ratio of the maximum indentation force to the resultant projected contact area evaluated from the shape function of the indenter and the maximum indent displacement.

Figure 3.10 shows an example of nanoindentation properties from the Y-plane of an EBAM sample, SF 36. The elastic modulus on the Z-plane and Y-plane are 116 GPa and 112 GPa, respectively, while the literature values are ranged from 108 GPa to 128 GPa with an average value of 117.5 GPa. The test results of elastic modulus are comparable with literature values, as shown in Figure 3.11a.

Figure 3.10 Typical load vs. displacement curves
The average hardness on the Z-plane and Y-plane of Ti-6Al-4V parts built with SF 36 are shown in Figure 3.11b. Murr et al. [34] measured the microindentation hardness of the specimen and found that the hardness is between 3.6 GPa and 4.3 GPa, which is very close to the hardness of wrought Ti-6Al-4V alloy, around 4.0 GPa [35]. The value from Baufeld and Biest [36] is only 3.1 GPa, which is much smaller. The hardness obtained from the current study is about 6 GPa for both Z-plane and Y-plane, which is about 30% higher than the literature values. This could result from the difference of the testing method as the literature values are obtained from micro-indentation tests. According to Qian et al. [37], nanoindentation hardness is 10-30% higher than the micro-indentation hardness for the same sample. Considering the method difference, the values here may just slightly higher than the literature results. The elastic modulus for the
wrought or cast Ti-6Al-4V is around 120-125 GPa with the microhardness around 4-4.2 GPa [38]. It demonstrates that the mechanical properties of EBAM Ti-6Al-4V alloy are comparable to those from wrought or cast components. The excellent mechanical properties of EBAM parts could be attributed to the strengthening phase and fine microstructure [10]. Compared to the wrought or cast specimens, the EBAM parts present much finer microstructure owing to the larger cooling rate during solidification [39]. Moreover, the presenting of martensites could work as the strengthening phase in Ti-6Al-4V alloy due to the very rapid cooling in EBAM. The martensites might contribute to the improvement of the hardness [13].

The elastic modulus and hardness of the specimens built with different SFs are shown in Figure 3.12 and Figure 3.13. Generally, the mechanical properties, including the elastic modulus and the hardness, will increase with the increase of the beam scanning speed to SF50 and decrease slightly with speed further increase to 65. The Y-plane have a higher hardness than the Z-plane, which mainly results from the larger fraction of β phase. It is known that α-Ti exhibits weaker strength than β-Ti within two-phase microstructures, and the hardness of α-Ti is less than one-third of β-Ti [40]. Therefore, the loading has a greater opportunity to apply on β in the Y-plane. In addition, Z-plane has smaller standard deviation values in both elastic modulus and hardness. This attributes to the more homogeneous microstructure and the better bounding force in the Z-plane. The bonding force on the Y-plane is weaker, which led to building defects on the Y-plane. Moreover, these defects will become serious with the increase of the beam scanning speed. The poor mechanical properties for the SF 65 sample could be attributed to the defects in the SF 65, including insufficient melt powder and pores [13], that affected by the small melting pool and melting temperature. Furthermore, the maximum difference in elastic modulus between
the X plane and Z plane appears at SF50 as 5.04%, which indicates that there are no significant anisotropic mechanical properties in the parts.

Figure 3.12 Hardness of Ti-6Al-4V specimens built with different SFs: (a) Z-plane, (b) Y-plane

Figure 3.13 Elastic modulus of Ti-6Al-4V specimens built with different SFs: (a) Z-plane, (b) Y-plane

3.4 Conclusions

To advance the rapid manufacturing of metallic parts by a deep understanding of the fundamental process of EBAM, four Ti-6Al-4V parts were built with 4 beam scanning speeds by EBAM to investigate the effects of the beam scanning speed on the final parts by combining microstructure analysis and mechanical properties. The major findings are summarized as follows.
(1) The Y-plane (side surface) of the specimens presents columnar prior β grains with martensitic structures growing along the build direction across multiple layers, and the Z-plane shows equiaxed grains. Additionally, the majority of the grain size is below 10 nm and the primary dendrite arm spacing is approximately 2 nm due to the rapid cooling rate during the manufacturing process.

(2) Most α grains have some preferred growing orientation with relatively stronger orientations of $<0001>$ in X plane and $<10\bar{1}0>$ in Z plane. The maximum intensity of the texture decreases and disperses randomly at several points with the increase of the speed function.

(3) The finest average grain size might contribute to the highest mechanical properties in the part fabricated with speed function 50. There are no significant anisotropic mechanical properties in the parts.

(4) Overall, the texture distribution disperses randomly at several points and the parts did not show significant anisotropic characteristics in mechanical properties. What is more, the majority of the misorientation angles are at a high level which results in the instability of the microstructures. This could be removed by post-heat treatments to satisfy the requirement of the designed working condition.

(5) The elastic modulus of the EBAM Ti-6Al-4V alloy is around 111.7~119.0 GPa, and the averaged hardness is about 5.24~6.52 GPa. The mechanical properties are superior or at least comparable to the wrought Ti-6Al-4V alloy. In addition, the Z-plane has a higher strength (elastic modulus and hardness) than the Y-plane.
(6) From this study, the optimized speed function is between SF 36 and SF 50, which may result in fine microstructures without severe surface pores in the EBAM Ti-6Al-4V parts and higher mechanical properties.
References


4.1 Introduction

Additive manufacturing (AM), which originated from the early days of rapid prototyping [1], is a process of joining materials to fabricate parts layer by layer directly from 3D CAD model [2–4]. Metallic part fabrication has been of great interest in industries due to the potential to fabricate near-net shape components using raw metal powder as feedstock materials [5,6]. One of the typical metal powder AM systems is selective laser melting (SLM), which completely melts the raw powder with one or multiple high-energy laser beams to build parts that could have comparable or even better mechanical properties than those of rolled metal sheets [7–9]. Considering various advantages that are unobtainable with traditional subtractive manufacturing, such as a high geometrical complexity of fabricated components, fabrication of metamaterial [10], and a reduced manufacturing time and cost [11,12], SLM has aroused interest from oil and gas, biomedical, and aerospace industries. It is suitable for producing Ni-based high-temperature components, which are difficult to be manufactured by traditional methods at room temperature due to excessive tool wear and low material removal rates caused by the high hardness of Ni-based super alloys [13–15].

As a nickel based alloy, Inconel 718 (IN718) has been widely applied in aviation and aerospace industries as it can retain its excellent mechanical properties at the elevated temperatures than other alloys [16–18]. Various works were performed to study the
characteristics of IN718 fabricated by SLM [19–24]. Amato et al. [17] reported that fine columnar microstructure with a width of 0.5 ~ 1 μm was observed in the plane parallel to the build direction of the cylindrical components. According to the study of Jia et al. [21], laser energy density has remarkable effects on the surface and it changed from a relatively smooth with few scattered metallic globules to a sound surface with a near-full density of 98.4% (at the recommended laser-beam energy density of 330J/m). Chlebus et al. [20] observed dendritic-cellular grains with Laves phase in inter-dendritic regions in the as-built samples, which also has been revealed by Zhang et al. [25]. In addition, he also mentioned that subsequent solution and double aging heat treatment were necessary for SLM-processed IN718 alloy. The mechanical properties of the SLM IN718 were also studied, and it was found that the specimens with heat treatment had superior mechanical properties but with a lower ductility in comparison with the wrought material [25,26]. Wang et al. [27] reported that there were no significant differences in mechanical properties along the build height of the parts. Moreover, according to former studies, there were some defects in the finishing parts, such as porosity and incomplete fusion powder [28], in addition, residual stress [29–33]. These defects might have strong negative effects on the performance of the finishing parts, as the fatigue life of components can be significantly decreased by the pores trapped in the melt pool during solidification [34].

Even though extensive studies have been conducted on SLM IN718, very few studies have focused on the effects of the thermal cycles on the microstructure evolution in the part. In the SLM processes, the complicated physical and chemical processes within the melt pool would unavoidably affect the microstructures in the fabricated components [21,35]. In addition, solidified microstructures in the previous layers are repeatedly subjected to the thermal cycles from the deposition of subsequent layers during the printing process, which leads to the variation
of the thermal histories for different layers. To further explore potential industrial applications, a staircase block IN718 part was built to study the effects of the thermal cycles on the microstructure evolution in the final part. The effects of the thermal cycles on porosity in the part also were also evaluated and the relationship between SLM build height and the microstructure was established.

4.2 Experimental procedures and methods

As indicated in the scanning electron microscope (SEM) image, Figure 4.1a, the majority of the raw IN718 powder are in a smooth sphere shape. A small fraction of them has some small satellite balls attached to them. The distribution of the spherical powder diameters was measured from the SEM images using the ImageJ software. The statistic results were plotted in a histogram, as displayed in Figure 4.1b. The average diameter of the particles is 29.1 um with a range from 11 um to 65 um. The details of chemical compositions of the powder measured from XRD are shown in Table 4.1. Figure 4.1c is the as-fabricated IN718 staircase part, and Figure 4.1d is the schematic drawing of the part to illustrate the location of the specimens. The staircase block part has initial dimensions of 20 mm in width and 90 mm in length, which was divided into three steps with each of them having a length of 30 mm. The first step only have a build height of 3 mm, the second and the third steps have a height of 60 mm and 90 mm, respectively. This design could achieve the research goals with the exclusion of other potential factors as it can provide several samples from different locations that subjected to various periods of thermal cycles.

A commercial SLM equipment, Concept Laser M2 Laser Cusing System, was used to fabricate the solid staircase block part. It utilizes a computer-controlled continuous wave fiber
laser, which can be manipulated by scanner system to form multiple laser beams with scanning velocity up to 7 m/s and to give a small spot size of 70 ~ 200 μm. In this study, ‘island’ scan strategy/pattern, also called ‘chessboard’, was adopted in raster-scans. As represented by Figure 4.1c, the area inside of the contour was divided into $5 \times 5$ mm squares named as islands. These islands were selectively melted in a random order with vectors perpendicular to each other in the neighboring islands and adjacent layers. To reduce the possible interference between straight boundaries of individual islands from same layer or neighbor layers, the whole pattern is rotated 45 degrees relating to the substrate plate [36], and the island pattern is shifted by 1 mm in both the X and Y directions for each subsequent layer, as illustrated in Figure 4.2a. For each island, a standard alternating x/y raster strategy, also called ‘antiparallel’, was adopted. The beam scanning speed is 600 mm/s and the beam scan spacing is 105 μm, and the detail of the parameters are given in Table 4.2.

The finishing part was cut into small samples at the locations shown in Figure 4.1d. The well-prepared samples were etched to show the microstructure. The etched metallographic samples were observed using a digital microscope and a scanning electron microscope. EBSD was also conducted using a scanning electron microscope equipped with an EBSD camera to analyze the texture in the specimens.
Figure 4.1 The pre-alloyed raw IN718 powder, (a) SEM morphology and (b) histogram of IN718 particle-size distribution; (c) as-fabricated staircase block, and (d) schematic drawing showing the location of the specimens

Table 4.1 Chemical compositions of IN718 powder

<table>
<thead>
<tr>
<th>Element</th>
<th>Cr</th>
<th>Mo</th>
<th>Al</th>
<th>Ti</th>
<th>Fe</th>
<th>Nb</th>
<th>C</th>
<th>Ni</th>
</tr>
</thead>
<tbody>
<tr>
<td>wt%</td>
<td>18.4</td>
<td>4.2</td>
<td>0.3</td>
<td>0.9</td>
<td>17.7</td>
<td>5.1</td>
<td>0.08</td>
<td>Balance</td>
</tr>
</tbody>
</table>

Table 4.2 Manufacturing parameters used in this study

<table>
<thead>
<tr>
<th>System</th>
<th>Laser Type</th>
<th>Spot size, μm</th>
<th>Power, W</th>
<th>Layer thickness, μm</th>
<th>Hatch spacing, μm</th>
<th>Scanning speed, mm/s</th>
<th>Scanning Pattern</th>
</tr>
</thead>
<tbody>
<tr>
<td>Concept Laser M2</td>
<td>CW Fiber</td>
<td>54</td>
<td>180</td>
<td>30</td>
<td>105</td>
<td>600</td>
<td>Island, 5mm</td>
</tr>
</tbody>
</table>
The distribution of the porosity in the part was investigated by optical microscope which has a measuring accuracy as high as that of the X-ray computed tomography (XCT) [34]. All the samples were final polished and photographed by a microscope at a magnification of 100. Then, each of them was processed using the ImageJ software by reducing it to 8-bit gray image and adjusting the function threshold level. The pore data and area fraction of porosity were obtained using the particle analyzing function in the ImageJ software [37,38]. For each of the samples, twenty-two measurements were carried out on different images with a total area of 22.7 $mm^2$. The obtained area fractions of porosity in different measurements were averaged as the porosity of the whole surface observed.

4.3 Results and discussion
4.3.1 Microstructure analysis

Figure 4.2 is the optical metallographic images, which show the morphologies of the IN718 samples. The island scanning pattern with the laser beam scanning tracks is seen clearly in the Z-plane (Figure 4.2a). In addition, the 1 mm shifting of the ‘island’ between two adjacent layers can be easily told by the difference of the color results from the binding between the boundaries of the islands, as indicated by the arrows in Figure 4.2a. The morphology of the solidified macrostructure in Figure 4.2b shows clearly that the beam antiparallel scanning paths were used as the hatch strategy within each ‘island’. The width of each track was about 100 $\mu m$ (Figure 4.2c), and equiaxial microstructure was revealed on the Z-plane.

While on the Y-plane, a series of wave-like arcs presented in the cross-section of the layers, which were induced by the Gauss energy distribution of laser beam in the melt pool [26], as displayed in Figure 4.2d. The microstructure on the Y-plane features a directional fine
columnar architecture, which is determined by the specimen building strategy. There are a lot of elongated dendrites growing through several layers with an orientation nearly parallel to the Z axis, as also can be seen from the BSE images in Figure 4.3a-b. This was attributed to the vertical heat flux corresponding to the heat transfer into the stainless steel sub-plate, which was affected by the re-melting process due to the overlap of the scanning tracks.

Figure 4.2 Optical microscopy images showing the microstructures of the SLM IN718 samples (a-c) Z-plane and (d) Y-plane

In addition, two quite different melt pools were observed on the Y-plane, shallow ones and deep ones, as shown in Figure 4.2d and Figure 4.3c. The deep melt pools were revealed to be the residual profile of the laser hatching scan tracks at turning points, which can be determined
by the pattern appeared on the Y-plane in Figure 4.2c-d. Moreover, the cellular dendrites within
the deep melt pools have random growth directions, and the majority of them are vertical to the
boundary of the melt pool with some degree of vertical to the Y-plane, as revealed in Figure
4.2c. This is determined by the determinate cooling rate during solidification.

The dark regions under SEM are the basic $\gamma$ phase [39]. There are also some bright
features formed in interdendritic regions with a long-chained interconnected morphology, which
was revealed as precipitated Laves eutectic, as labeled in Figure 4.3d. Their formation results
from the segregation of Nb and other elements, which could weaken the precipitation of $\gamma''$ and $\gamma'$
phases as the formation of Laves phase would consume a mass of Mo, Ti, and Ni. In addition,
Laves phase is brittle and deleterious to the mechanical properties of IN718 [39,40]. The back-
scattered electron (BSE) micrograph reveals that the elongated columnar microstructure in the Y-
plane has a morphology of cellular, as shown in Figure 4.3a-b. The transversely cut dendritic
cells were also presented in some melt pools, which result from the laser beam scanning paths
that are nearly vertical to the Y-plane (Figure 4.3d).
Based on former studies, the cooling rate can be calculated according to the empirical equations shown below.

\[
\lambda = 104.47 \times \dot{T}^{-0.31} \quad (1)
\]

\[
\lambda = 156.52 \times \dot{T}^{-0.349} \quad (2)
\]

\[
\lambda = 319.4 \times \dot{T}^{-0.378} \quad (3)
\]

where \(\lambda\) is the width of the cell spacing and \(\dot{T}\) is the thermal cooling rate. They were developed by Wang et al. [41], Bouse and Mihalisin, [42], and Won et al. [43], respectively. Based on the statistical results from the experiments, the characteristic size of the cell spacing falls in the
range of 0.511 ± 0.047 μm to 0.845 ± 0.195 μm. This implies that the SLM process is a fast solidification process with a very large temperature gradient. Therefore, the estimated cooling rate values for this case are in the range of $1.74 \times 10^6$ K · s$^{-1}$ (°C · s$^{-1}$) to $3.88 \times 10^7$ K · s$^{-1}$ (°C · s$^{-1}$), which are in accordance with former studies [44,45]. The detailed results from the empirical equations are listed in Table 4.3.

Table 4.3. Predicted cooling rate based on the statistic results from experiments

<table>
<thead>
<tr>
<th>*Eq.</th>
<th>$\lambda_{\text{Min}}, \mu m$</th>
<th>$\lambda_{\text{Max}}, \mu m$</th>
<th>Constant</th>
<th>Constant</th>
<th>$\dot{T}_{\text{Max}}, ^\circ C/s$</th>
<th>$\dot{T}_{\text{Min}}, ^\circ C/s$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.464</td>
<td>1.04</td>
<td>104.47</td>
<td>-0.31</td>
<td>$3.88 \times 10^7$</td>
<td>$2.87 \times 10^6$</td>
</tr>
<tr>
<td>2</td>
<td>0.464</td>
<td>1.04</td>
<td>156.52</td>
<td>-0.349</td>
<td>$1.75 \times 10^7$</td>
<td>$1.74 \times 10^6$</td>
</tr>
<tr>
<td>3</td>
<td>0.464</td>
<td>1.04</td>
<td>319.4</td>
<td>-0.378</td>
<td>$3.22 \times 10^7$</td>
<td>$3.80 \times 10^6$</td>
</tr>
</tbody>
</table>

Note: Eq.1.-Wang et al. [41], Eq. 2.-Bouse and Mihalisin, [42], Eq. 3.-Won et al. [43]

4.3.2 Effects of the thermal cycles

(a) Samples close to top surface of the staircase block part

The microstructures in the Y-plane from different heights are shown in Figure 4.4. In general, the Y-plane presents a fine columnar cellular structure. At the initial state, the cellular dendrites have multiple growth directions and they were easily interrupted by melt pools from subsequent layers (Figure 4.4a). With the increase of the build height, the cellular structures are more likely to grow upwards as the growth directions of the cellular dendrites along the build direction becoming narrow, as can be seen from Figure 4.4a-c. They might go through multiple layers without being interrupted by the re-melting process in SLM. This was attributed to the combined effects of the thermal gradient, nucleation, and growth of the dendrites [46].
At the initial building stage, the heterogeneous nucleation of γ phases would occur and dendrites grow subsequently along the thermal gradient. For the first melted tens of layers, the main the heat loss is from heat sink effect of the stainless steel substrate plate. However, there is minor heat loss diffusing peripherally from the melt pool in the plane, which may take a relatively larger fraction of the total heat loss with a thin build layer. Therefore, the grains at the bottom part have a titled growth direction with the angle depends on the fraction of the heat loss through various paths.

With the build-up of the part layer by layer, the epitaxial nucleation would be taken over the nucleation and the growth of the dendrites. Because the latter beam scanning re-melt the previously solidified layers, and the former layer grains at the fusion boundaries of the melt pool would play the role of the substrate for nucleation of the liquid metal in the melt pool [46]. Then, epitaxial nucleation occurs and grows from the former layer to the next layer. In addition, the heat loss in the plane decreased gradually with the growth of the fabricated component. In this way, the highest thermal gradient would be from the melt pool to the sub-plate, which leads to a nearly vertical growth direction of the dendrites, as shown in Figure 4.4a-c.
In addition, the width of the cellular structures increased with the build-up of the part (Figure 4.4i), which is attributed to the variation of the cooling rate. At the initial stage of manufacturing process, the cooling rate was very high due to the higher heat conductivity between the melt pool and the subplate (300 °C). Therefore, more nuclei were generated, resulting in the formation of smaller columnar grains [1] (Figure 4.4a). Then, the cooling rate decreased with the increase of the build height [47,48], which leads to fewer nuclei and a slower growth rate for the dendrites. This results in wider dendrites (Figure 4.4c).
Moreover, the Laves phase morphologies and the Nb segregation patterns are clearly shown in Figure 4.4d-f, which are mostly located at the intercellular regions and triple points with a white color under SEM. The volume fractions of the Laves phase in the specimens were measured by estimating the fraction of the white area using ImageJ, as demonstrated in Figure 4.4d. Figure 4.4g presents the statistic results of the fraction of Laves phase particles in the three specimens. Generally, it increases first and then goes down with the build-up of the part. The slight increase from sample 3.1Y1 to 2.1Y1 was owing to the decreased cooling rate in the fabrication process of the component. At a higher cooling rate in rapid solidification, the dendrite growth rate and the solute trapping can be improved as there is no sufficient time available for Nb to diffuse from the solid phase to the liquid phase. Nb elements are more likely to be trapped in the solid phase and this would limit the formation of Laves phase due to the lack of Nb elements [49]. Another reason is the strong eutectic reaction of $L \rightarrow \gamma + \text{Laves}$ that occurred at the juncture corner of the part, due to the heavy segregation of Nb, as shown in Figure 4.4e. Therefore, sample 2.1Y1 would have a weak mechanical performance due to the brittle nature of Laves phase [50]. However, it goes down in the sample 1.0Y2, which was caused by the smooth growth of the dendrites. As shown in Figure 4.4c, the cellular dendrites are more likely to go through multiple layers without distinct change of growth direction, while in Figure 4.4a, a lot of Laves phase was observed at the interlayers with the change of the growth direction of the dendrites.

(b) Samples from the bottom region of staircase block part

SEM micrographs of the samples with different durations of reheating thermal cycles are shown in Figure 4.5. With the extension of the reheating thermal process, the cellular dendrites
are more likely to align themselves with the sharpest thermal gradient, which can be seen by comparing Figure 4.5a-f. In sample 3.1Y1, the variation of the growing direction for the dendrites between neighboring layers is extremely obvious, while it gets less distinct with the increase of the thermal cycles.

Figure 4.5. SEM microscopy images showing microstructure of inside Y-plane at bottom of the SLM IN718 part (a-f), and statistic results of (g) Laves phase fraction, and (i) width of the cellular dendrites.

Moreover, the morphology of the Laves phase also has changed under the continuing effects of the thermal cycles. Coarse and interconnected Laves phase were revealed in sample 3.1Y1 while discrete Laves phase particles were revealed in sample 1.2Y1. As shown in Figure
4.5g, there is a big drop in the estimated fraction values of Laves-phase, which is attributed to the reheating process as Laves phase can be dissolved at proper heat treatments [51]. This continuing thermal cycle process also has effects on the size of the solidified microstructure in the part. As shown in Figure 4.5i, the longer the thermal cycle period is, the coarser the grains are.

3.3 Microstructures with and without further thermal cycles

Figure 4.6 displays SEM micrographs of the samples at the same location with different heights. In general, the width of the cellular dendrites increases with the build-up of the height, as plotted in Figure 4.6i, which is attributed to the decreased cooling rate at a higher build height in addition to the effects of the thermal cycles. Comparing to the statistic results listed in Figure 4.4i, it was also found that the width of the cellular dendrites increased under the effects of thermal cycles, which is accordance with the conclusion in section 3.2. The same trend also was revealed in Laves phase. As displayed in Figure 4.6g, the fraction of the Laves phase showed a slight increase with the increase of the build height, and its morphology changed from coarse and interconnected to discrete particles. By comparing the results from sample 2.1Y1 (Figure 4.4g) and 1.1Y1 (Figure 4.6g), the fraction of the Laves-phase dropped significantly under the continuing effects of the thermal cycles, which agrees well with the findings above in section 3.2.
4.3.3 Texture analysis

(a) Typical features in EBSD results

EBSD tests were performed to characterize the basic $\gamma$ phase crystallographic texture of the as-fabricated IN718 samples with the typical results from Z-plane shown in Figure 4.7, in which the larger scanning area is 2.1 mm by 1.3 mm. From the inverse pole figure (IPF) colored map (Figure 4.7a), the Z-plane is characterized by equiaxial grains with morphology leftover by the part build strategy (Figure 4.7b), such as beam power, scanning pattern, scanning speed, and hatching space.
Figure 4.7. EBSD analysis of SLM IN718 sample (1.0Z1), a larger scanning area: (a) inverse pole figure (IPF) colored map, (b) grain map, (c) pole figure (PF) maps, (d) grain size plot and (e) misorientation angle plot; and two enlarged areas: (f-h) IPF colored map, (g) grain boundary map corresponding to (f), and (g) grain map corresponding to (h)

In general, the Z-plane shows a strong texture of \{0 0 1\} as most grains exhibit preferential solidification in the \(< 0 0 1>\) direction with respect to the building direction.

However, some grains showing different orientations were observed in the binding regions among neighbor islands from different layers, as indicated by the spacing of them, about 1 mm. Under an enlarged magnification, the inter-islands zone shows fine equiaxial grains with chaotic orientations (Figure 4.7f-g), while the region within one island presents a relatively larger grain size with a very strong orientation of \(< 0 0 1>\) (Figure 4.7h-i). In addition, multiple sub-grains
were observed in each grain, which indicates that many low angle grain boundaries exist in this area (Figure 4.7h-i).

Figure 4.8 presents the typical EBSD analysis results of the Y-plane. In general, the Y-plane is characterized by columnar grains with the majority of them having a preferred orientation of <1 0 1> with respect to the Y direction. As shown in the inverse pole figure (IPF) colored map Figure 4.8a, the texture analysis results show that there is a strong texture of \{1 0 1\}. However, irregular shape grains with various orientations were observed in a narrower zone, which was found to be the inter-region of the islands. They were caused by the scanning strategy, in which the spacing between two neighbor zones is about 1 mm.

In addition, the long axes of the columnar grains in the Y-plane approached millimeters in length, which means the grains have grown through tens of layers if considering the layer thickness is only 30 um. This grain structure was a result of the combined impact of the re-melting process, epitaxy and melt pool solidification kinetics [40]. Moreover, the growth of the grains also depends on the local heat flow directions which are significantly affected by the scanning pattern adopted in SLM [40]. The maximum heat flow approach is from the melt pool of the present build layer to the substrate layers via conduction, which is in a zigzag way through the build direction. This results from the behavior of the highly elongated melt pool and the infill “hatching” employed in SLM with the raster directions alternating between two orthogonal directions of every layer [52]. These factors introduced the formation of large elongated grains with their orientation favored by the heat flux.
Figure 4.8. EBSD analysis of SLM IN718 sample (2.1Y2), a larger scanning area: (a) IPF colored map, (b) grain map, (c) PF maps, and (d) grain size plot; and an enlarged area: (e) IPF colored map, (f) grain boundary map, and (g) misorientation angle plot corresponding to (f).
Based on this scanned area of sample 2.1Y2, the statistical results for the grain size and the area fraction are shown in Figure 4.8d. There is no obvious dominated grain size in the Y-plane. The majority of the grains are below 100 um with some larger grains might over 200 um. Comparing to the Z-plane, the Y-plane generally have a higher grain size, which can be seen by comparing the grain size plot maps in Figure 4.7 and Figure 4.8. In addition, the Y-plane also have a higher fraction of high-angle grain boundaries than the Z-plane. As shown in the boundary rotation angle map (Figure 4.8g), the majority of boundary rotation angles are in high level, which resulted in the instability of the microstructure due to the high energy accumulated along the grain boundaries [20].

(b) Samples without further thermal cycles

Figure 4.9 displays the comparison of the EBSD results for three samples close to the top surface at different build heights. They all have identical dimensions of 1.25 mm by 0.75 mm for the scanning area. For the Y-plane (Figure 4.9a-c), the elongated grains present a strong favored orientation of $<1 0 1>$. By comparing the IPF at different heights, it is found that the bottom sample (3.1Y1) has the weakest texture and the middle sample of the part have the strongest texture, which are the combined effects of the thermal gradient, nucleation, epitaxy and competitive growth. This leads to the dendrites having no obvious preferred orientations at the initial stage, and then showing a stronger orientation.

In addition, the occurrence of heterogeneous nucleation and their subsequent growth dominated the solidification in the first tens of layers, which means there is no obvious preferred orientation for the dendrites. Then, the epitaxial nucleation gradually took the position and there was a favored crystal growth direction during the competitive growth of the grains. This led to
the increase of the intensity of the texture, as can be seen from the IPF colored maps and the corresponding pole figures in Figure 4.9. The same change trend can be found in the Z-plane, as can be seen from Figure 4.9d-e, in which the top-end sample (1.0Z1) shows a stronger texture intensity than the bottom sample (3.1Z1).

Moreover, the higher cooling rate at the initial manufacturing stage leads to a smaller grain size. Based on the statistical results for the distribution of the grains, the fraction of larger grains increases with the build-up of the part. For example, the grains over 100 um in the sample 3.1Y1, 2.1Y1 and 1.0Y3 are 0.305, 0.334 and 0.361, respectively.
Figure 4.9. EBSD results of samples from various build height without further thermal cycles: (a) 3.1Y1, (b) 2.1Y1, (c) 1.0Y3, (d) 3.1Z1, and (e) 1.0Y3
(c) Samples with different periods of thermal cycles

Figure 4.10 presents the EBSD results for the samples with various durations of thermal cycles. It is obvious that most of the grains in the sample have a major preferred orientation of \(<1\ 0\ 1>\) with respect to the Z axis. By comparing the samples in same height (Figure 4.10a-c, Figure 4.10d-e), it is found that the maximum intensity of the texture will increase with elongation of the thermal cycles. This was attributed to the thermal heat effects from the subsequent reheating thermal cycles, which aligned the grains along the gain build direction and this further led to the increase of the intensity of the texture.

In addition, the fraction of the grains with a smaller diameter increased with effects from the subsequent thermal cycles, as can be seen from the statistic results of the area fraction of the grain size. However, the fraction of the high misorientation angles (>15°) increased with the continuation of the build process. For example, the high misorientation angles in the sample 3.1Y1, 3.1Y3 and 1.2Y1 are 0.202, 0.268 and 0.363, respectively.
Figure 4.10. EBSD results of samples from various build height with further thermal cycles: (a) 3.1Y1, (b) 3.1Y3, (c) 1.2Y1, (d) 2.1Y1, and (e) 2.1Y2
(d) Samples at same location with various periods of thermal cycles

The EBSD results for the samples at the same location in the part with different build heights are shown in Figure 4.11. The Y-Plane of the part presents a strong texture of \{1 0 1\} along the building direction, and the maximum intensity of the texture increased with the build-up of the part and then decreased at the end period of the SLM process. The grains over 100 um in the sample 1.2Y1, 1.1Y1, and 1.0Y3 are 0.331, 0.445 and 0.361, respectively. This means with the increase of the build height, the grain size increased first and then decrease a little bit at the end of the manufacturing process. This is coordinated with our former studies [27]. The sample from the top region of the part has a smaller grain size than the samples below and larger than the samples at the bottom of the parts.

Generally, the fraction of the high misorientation angles (>15°) increased with the subsequent building of the part as can be seen that the fraction of the high boundary rotation angles in 3.1Y1, 2.1Y1 and 1.0Y3 are 0.202, 0.234 and 0.370, respectively. This indicates that more and more energy accumulated at the grain boundary with the continuation of the re-melting process in SLM.
4.3.4 Porosity in the samples

There are mainly two kinds of pores in the polished longitudinal section of the specimens, small spherical pores (A1) and big pores with spherical or irregular shape (A2), as shown in Figure 4.12. These pores affect the mechanical properties of the material [53],
especially the irregular pores, stress concentration while under loading. The circular small pores are which easier caused entrapped gas bubbles in the melted bead which could not rise and escape to the top surface before solidification [39] and gradually merge into big pores. In addition, irregular pores or open pores are affected by the manufacturing parameters, such as the beam scanning speed [21].

As the SLM process involves consecutive thermal cycles during the whole process, the porosities of the samples at different positions in the part were measured and the statistic results are shown in Figure 4.12b. In general, the average values of the porosity are no more than 0.2% of the total area, and there is no significant change trend along the build height of the part.

The effects of the thermal cycles on the porosity are shown in Figure 4.12c. Even samples at the location of 1.2 have the most subsequent thermal cycles during the manufacturing process, there is no significant change in the porosity in the part considering both Y-plane and X-plane. Therefore, the thermal cycles do not have a significant influence on the porosity in the part. Figure 4.12d presents the porosity along the build height of the part. Based on the statistic results, there is no remarkable change in the build height.
Figure 4.12. IN718 samples, 1.1Y1: (a) pores under optical microscope at the location close to the boundary of the part, A1-small spherical pores, and A2-big pores and irregular pores, (b-d) locations of the samples and the statistic values of the porosity fraction.
4.4 Conclusions

In this study, the effect of the thermal cycles on the microstructures of IN718 samples manufactured by SLM was investigated. The specimens were cut from the as-fabricated IN718 staircase part with wire-electrode and then prepared for microstructure characterization. According to the results, equiaxial grains were observed in the Z-plane, which has a preferred $<001>$ orientation with respect to the Z-axis and a distinct $\{001\}$ texture, while columnar structures presented in the Y-plane. Within the elongated grains are colonies of cellular dendrites with a cell spacing of $0.511 \sim 0.845 \mu m$. The majority of them have a preferred orientation of $<101>$ with respect to the Y-axis and a strong $\{101\}$ texture. In addition, Laves phase in the morphology of irregular white shape were observed in the intercellular zones. Under the effects of the subsequent thermal cycles, the fraction of the Laves phase showed a significant drop with its morphology changing from coarse and interconnected particles to discrete Laves phase. In terms of the width of the cellular dendrites, the longer the thermal cycle period is, the coarser of the elongated grains are. Moreover, the maximum intensity of the texture increased with the effects of the subsequent thermal cycles, same for fraction of larger grains and the high misorientation angles. Furthermore, the area faction of the porosity is below $0.2\%$, and there is no remarkable effects found from the thermal cycles and the build height on the distribution of the porosity.
References


CHAPTER 5

BUILD HEIGHT EFFECTS ON THE FINAL PARTS

5.1 Introduction

As a promising powder-bed AM technology, selective laser melting (SLM) could melt powder selectively layer by the action of a high-energy laser beam that performs a complete fusion of one layer to another to produce complex components with high dimensional precision and good surface integrity precisely [1–4]. It has critical applications for aerospace materials, such as titanium alloys, aluminide, and nickel-based alloys [5–8]. IN718 alloy is a precipitation-hardened solid solution nickel–chromium alloy. However, it is difficult to manufacture the IN718 material by conventional machining methods at room temperature due to excessive tool wear and low material removal rates [9,10]. Moreover, Inconel 718 parts with complex structures, high dimension precision, and further elevated mechanical properties are in higher demand [11]. Therefore, the application of the novel non-traditional processing technology is necessary for the net shape production of Inconel 718 parts with complex configurations and high performance.

Experimental studies regarding microstructures of SLM processed Inconel 718 components have been carried out. Wang et al. [11] found that a regular microstructure with good metallurgical bonding, minimal defects, and fine dendritic grains is formed by SLM. Amato et al. [12] observed that the fabricated components exhibited a more pronounced [13] columnar γ" phase precipitate architecture parallel to the build direction (spaced at ~0.8 μm). Since most the
SLM Inconel 718 components are applied to the structural components, different mechanical properties have been investigated. Wang et al. [11] studied the tensile properties of SLM built Inconel 718 specimens and found that the yield strength and tensile strength are 903 MPa and 1143 MPa, respectively. Amato et al. [12] found the tensile strength and microhardness are about 1120 MPa, and 3.9 GPa, respectively. It demonstrates that the mechanical properties of SLM fabricated specimens are comparable to those of cast or wrought specimens. The mechanical properties of SLM Inconel 718 are excellent enough to meet the expectations in the applications [14], and the detail of the tests can be seen in former studies [15].

The characterizations of the SLM Inconel 718 also vary with the process conditions, such as the manufacturing process parameters and the shape features. Therefore, it is important to understand the effects of the process conditions during the building process. Through various aspects in SLM Inconel 718 have been investigated, the height of the build part also called build height, which may affect the microstructure and mechanical properties of the final part, has not been studied yet. In this study, the objective is to investigate the effect of the build height on the mechanical properties and microstructure of an Inconel 718 part fabricated by SLM using nanoindentation test, optical microscopy (OM) and scanning electron microscopy (SEM). The relationship between SLM build height and the characterization will be established.

5.2 Experimental Procedure

A Concept Laser M2 Laser Cusing System at NASA’s Marshall Space Flight Center (Huntsville, AL) was used to fabricate the solid block modeled from CAD software with a dimension of 40 mm by 40 mm by 6 mm. The M2 system has a maximum build plate size of 250 mm 250 mm and a buildable height of 300 mm, as shown in Figure 5.1a. It utilizes a computer-
controlled continuous wave fiber laser with a fixed laser spot focus diameter of 54 μm and a maximum output of 200 W. It is directed to the powder bed using servo controlled reflectors, which allow the laser spot to scan the surface of the bed at a maximum of 7000 mm/s. Besides, it can be manipulated by scanner system to form multiple laser beams with scanning velocity up to 7 ms-1 and to give a small spot size of 70 ~ 200 μm.

Fine pre-alloyed Inconel 718 powder was used as rough material to fabricate the block. During building each layer of the block, the laser beam moves across the powder-layer surface tracing the model cross-section boundary and then raster-scans thoroughly the inside of the contour using island scan strategy/pattern. The area inside of the contour was divided into 5mm squares named as islands. These islands were selectively melted in a random order with vectors in the adjacent islands perpendicular to each other. The whole pattern is rotated 45 degrees with respect to the substrate plate to reduce any possible interference between the recoated blade and the straight boundary of each individual island [16]. For each island, simple alternating scan vectors with speed of 600 mm/s have been used with scan spacing is 105 μm. The island pattern is shifted by 1 mm in both the X and Y direction for each subsequent layer. The detail of the parameters used in this study is listed in Table 5.1. The as-fabricated part with stress relieved (Heat treatment) is shown in Figure 5.1c.

The standard metallographic procedures with vibratory finishing were used to prepare the eight Inconel 718 samples cut from the part along the Z axis, in which S1 (Sample 1) to S4 were used to investigate the Y-plane and S5 to S8 were used to study the Z-plane. Then, the EBSD and nanoindentation tests were conducted on the surfaces of the well-prepared samples first. After that, they were etched to reveal the microstructures with an acid-based solution made of 20 ml hydrochloric acid (37 wt. %), 20 ml (68 wt. %) nitric acid and 1g copper chloride. The etched
metallographic samples were observed using a Leitz optical microscope (OM) and JEOL 7000 FE Scanning Electron Microscope (SEM). Nanoindentation tests were performed using a Triboindenter from Hysitron Inc. and a radius of 100 nm Berkovich tip. The details setting are same as in former study [17]. The indent pattern used in all the experiments is a matrix of $3 \times 4$ with a spacing of $5 \mu m$ between any two horizontal or vertical adjacent points.

Figure 5.1 (a) Laser Concept M2 Cusing System, (b) island scanning pattern and (c) as-deposited part with stress relieved

Table 5.1 Manufacturing parameters used in this study

<table>
<thead>
<tr>
<th>System</th>
<th>Laser Type</th>
<th>Spot size, $\mu m$</th>
<th>Power, W</th>
<th>Layer thickness, $\mu m$</th>
<th>Hatch spacing, $\mu m$</th>
<th>Scanning speed, mm/s</th>
<th>Scanning Pattern</th>
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<tr>
<td>Concept Laser M2</td>
<td>CW Fiber</td>
<td>54</td>
<td>180</td>
<td>30</td>
<td>105</td>
<td>600</td>
<td>Island, 5mm</td>
</tr>
</tbody>
</table>

5.3 Results and Discussions

5.3.1 Typical Microstructure

The typical optical microstructure on of the stress relieved samples is shown in Figure 5.2. The island scanning pattern with the antiparallel raster hatching traces disappeared, and only $100 \mu m$ squares with fine equiaxed microstructure shown in the Z-plane. This implied that the microstructure had evolved during the heat treatment for stress relief, and the squares of pattern
grew out of the standard alternating x/y raster strategy in addition to the shifting of the island pattern for each subsequent layer. In addition, columnar microstructures were revealed in the Y-plane, and the average width of them are about 150 µm which is just about the length of the diagonal of the square pattern in the Z-plane. The equiaxed microstructure was found within the columnar structure and fine vertical elongated microstructure was observed at the inter-regions of the columnar structures which resulted from the overlap of the melting tracks. Therefore, the SLM Inconel 718 samples after stress relief have a rod-shaped equiaxed microstructure, which is much more homogeneous than the as-fabricated specimens, also can be verified under SEM shown in Figure 5.3.

Figure 5.2 Optical micrographs of as-fabricated SLM Inconel 718 alloy with heat treatment for stress relief, (a) Z-plane (b) Y-plane

Figure 5.3 presents the SEM micrographs of the microstructure of the SLM Inconel 718 alloy. It was found that the scanning direction of the beam tracks has significant effects on the growth direction of the columnar cellular dendrites (Figure 5.3a and Figure 5.3b). The dark color region was revealed to be the basic γ phase and the write color areas located at the interdendrites, grain boundaries, and layer interfaces were determined as Laves phase (Figure 5.3b). The
formation of Laves phase resulted from the dendritic-cellular growth of grains and susceptibility of Nb and Mo to microsegregation at high cooling rates [14]. The average width of the cellular dendrites is about 0.68 µm.

![Figure 5.3 Microstructures on Y-Plane of Inconel 718 under SEM (a, b) as-fabricated and (c-d) as-fabricated and stress relieved](image)

It is known that at proper heat treatment, a homogeneous microstructure can be obtained which has a stable γ phase strengthened with coherent and dispersive precipitates of the phases γ' and γ'' as Inconel 718 parts [10]. After the heat treatment for stress relief, the microstructure became more homogenous as Laves phase in the melt pool was partly solved into the basic γ
matrix with their morphology changing from coarse and interconnected particles to discrete Laves phase, as clearly demonstrated by Figure 5.3b and Figure 5.3d. Some regions are dominated by black color which indicates a very low fraction of the Laves phase (Figure 5.3d). In addition, δ phase and carbides were observed in the stress relieved samples, which is caused by the condition of the heat treatment [14].

5.3.2 Build Height Effects

The optical examination of the samples reveals that columnar structures present on the Y-plane along the build direction and perpendicular to the melted powder layers. This is caused by the vertical heat flux related to heat transfer into the substrate. The Z-plane shows equiaxed grains, which results from the horizontal heat flux related to the movement of the heat source, as shown in Figure 5.4. The effect of the build height on the width of the columnar structure is shown in Table 5.2. Generally, the average width of the columnar structures increases with the increase of the building height of the part until a stable stage reached. The bottom sample shows narrow and uniformly distributed columnar dendrites, which is caused by the larger cooling rate due to the higher thermal conductivity between the bottom layers and their direct connecting build substrate plate. The substrate plate has a lower temperature (about 300 °C) compared to the melt pool of Inconel 718, as shown in Figure 5.4c. Increasing the cooling rate will form smaller columnar grains [18] because more nuclei could be generated at a higher cooling rate. This will further form finer grains in long and narrow columnar morphology.

In addition, the cooling rate changes with the build height of the part during the manufacturing process. This leads to the transition of the microstructure and further results in the larger standard deviation for the width of the columnar structures in the top and middle-bottom
samples, as listed in Table 5.2. Same for the top sample, as shown in Figure 5.4a, it presents clear transition of the columnar microstructure at the very top area where the width value decrease from \(~147\ \mu m\) to \(~75\ \mu m\). This is because the top layers are close to the end of the manufacturing process, which means they are directly touching the environment temperature in the chamber. This leads to a higher cooling rate compared to that in the middle of the part. There are some pores on the surface of the samples. The measured results of the porosity fraction of the samples from the solid part are shown in Figure 5.5. Based on the statistic results, the samples have a low percentage of porosity and the X-plane has a higher porosity than the Z-plane.

Figure 5.4 Optical micrograph showing etched microstructure in the Y-plane (a) S1, (b) S2, (c) S4 and Z-plane (d) S8
Table 5.2 Measured characteristic sizes SLM samples with build height

<table>
<thead>
<tr>
<th>Sample</th>
<th>Columnar structure width (µm)</th>
<th>Columnar structure width (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Average</td>
<td>Standard deviation</td>
</tr>
<tr>
<td>Top (S1)</td>
<td>112.45</td>
<td>38.02</td>
</tr>
<tr>
<td>Middle-Top (S2)</td>
<td>146.77</td>
<td>11.04</td>
</tr>
<tr>
<td>Middle-Bottom (S3)</td>
<td>111.40</td>
<td>38.33</td>
</tr>
<tr>
<td>Bottom (S4)</td>
<td>74.92</td>
<td>9.39</td>
</tr>
</tbody>
</table>

Figure 5.5 Statistic results of porosity in the Inconel 718 samples: (a) X-plane and (b) Y-plane

5.3.3 Texture analysis

The orientation maps from the Y-plane (#1-4) are shown in Figure 5.6a-d. The grain features of laser melting are shown clearly, such as the configuration of the grains and columnar architecture throughout the Y-plane. This geometry was determined by the building strategy, such as the laser scanning pattern, hatch spacing, and layer thickness. The cut ends of the grains in the form of a series of arcs on the Y-plane are induced by the Gauss energy distribution of laser in the melt pool [11]. At the intersection of the columnar structures, there are many
elongated grains growing through several layers, which were caused by the re-melting process due to the overlap of the scanning lines.

The pole figures corresponding to the orientation maps are shown in Figure 5.6e-h. Generally, the degree of the prominent orientation decreased from the top to the bottom of the samples. The existence of a fiber texture in samples is because the grain dendrites oriented parallel to the heat flow direction. The main heat transfer path is from the melt pool to the substrate, which has a maximum temperature gradient at Z-direction. Solidification occurs by the formation of nuclei and/or growth of crystals, and either of them may be predominant depending upon solidification conditions. As shown in the orientation maps, the growth of the columnar-grain texture has been interrupted by grains of random orientation formed at the solidification front. This implies that nucleation of new grains is more effective in accommodating the undercooling at the solidification front than the growth and branching of the dendrites in the existing columnar grains. The fastest growth direction of the basic $\gamma$ phase (fcc) is the $<1 0 0>$ direction. Thus, the grains were generally grown in the Z-direction with the orientation of $<1 0 0>$ along with the build of the part. According to the maximum intensity of the texture in the four samples, #4 sample has the weakest texture (4.76) comparing to the other three samples. This is because the first melt layers were in direct contact with the substrate stage, which resulted in a larger cooling rate without a particular direction and this further promoted the generation of nuclei and grew in a random way. After that, the nucleation of new grains occurred in some region at the solid–liquid interface between the melt pool and former layers. The grains, having a $<1 0 0>$ direction nearly parallel to the heat flow direction, grew rapidly while the growth of other grains would cease. Therefore, columnar grains formed with the majority of them having a
<1 0 0> crystallographic orientation. As mentioned above, the top layers have a higher cooling rate, which leads to a slightly strong grain orientation, as shown in Figure 5.6e.

Figure 5.6 (a-d) are orientation maps from Y-plane of the samples #1, #2, #3 and #4, respectively, and (e-h) are pole figures corresponding to (a-d), sequentially
Figure 5.7 (a-b) are orientation maps from Z-plane of the samples #5 and #8, respectively, and (c-d) are pole figures corresponding to (a-b).

The orientation maps of Z-plane from the top and bottom samples are shown in Figure 5.7a-b. The scanning pattern with a width around 100 μm is shown and there are numerous small-sized grains at the interface of the scanning paths. During the manufacturing process, the main heat transfer way on the scanning surface was from the inside to the boundary of the layers. This led to a fast growth for some of the grains with the <1 0 0> orientation parallel to the heat flow direction. Then, solidification occurred by the epitaxial growth of crystals from the melt pool. The pole figures from the Z-plane reveal a weak edge-on cube texture component presenting in the Inconel 718 sample, as shown in Figure 5.7c-d. This is a common texture in face centered cubic metals subjected to static recrystallization. This texture results in the
anisotropy in the samples, which also has been mentioned by Tayon et al. [21]. Compared to the Y-plane, the Z-plane was generally found to have a weaker texture as the maximum intensity if only about 5.1, although the overall texture of the entire build appeared to be a complex composite cluster of textures. Crystallographic texture affects the mechanical properties as it results in anisotropy in the elastic constants, etc. In the present case, there will be no significant various mechanical properties as the extent of the crystallographic texture presented is not very strong.

Figure 5.8 (a, b) are the typical misorientation angle map from Y-plane (#2) and Z-plane (#8), and (c, d) are the statistics results corresponding to (a, b)
Figure 5.8 presents the typical grain boundary maps, in which different colors represent different boundary rotation angles. The statistics results are shown in Figure 5.8c-d. The majority boundary angles are above 15°, which are called high-angle grain boundaries. This will lead to the instability of the microstructure because the high energy accumulated in the form of grain boundaries and, to a lesser degree, of dislocations. To obtain the microstructure composing of a stable γ phase strengthened with coherent and dispersive precipitates of the phases, γ’ and γ”, post heat treatment is needed [22]. The presence of high-angle grain boundaries also could strongly affect both the density and distribution of the cracks in the SLM material [16] which is more susceptible to ductility dip cracking (DDC) at high-angle grains boundaries.

There are some micro-segregations within the grains or at the interfaces of them, as shown clearly in Figure 5.7a-b. Through the phase analysis, it is the γ’ phases because inside of the grain is a face-centered cubic phase containing Aluminum. The segregations at the boundaries should contain Laves phase as there is Fe element. During solidification, dendrites formed and this would cause Nb and other alloy elements to segregate at the interdendritic regions. This leads to the formation of Laves phase, which are irregularly shaped phases with a typical composition of (Ni, Fe, Cr)2(Mo, Nb, Ti). They are detrimental to mechanical properties and can be dissolved in the matrix by proper heat treatments [19]. Thus, the as-fabricated builds have to be improved by performing heat treatment as they generally have poor mechanical properties due to these defects.

5.3.4 Mechanical Properties

Nanoindentation has been used to measure the elastic modulus and hardness of the samples. The Young’s modulus of the Inconel 718 sample, E, could be calculated by,
\[
\frac{1}{E_r} = \frac{1 - \nu_i^2}{E_i} + \frac{1 - \nu_s^2}{E_s} \quad (4.1)
\]

where \(\nu_i\) is the Poisson’s ratio of indenter, 0.07, \(E_i\) is the Young’s modulus of the indenter, 1140 GPa, \(\nu_s\) is the Poisson’s ratio of Inconel 718 samples, 0.294 and \(E_r\) is the Young’s modulus of the Inconel 718 samples directly obtained from the tests. The hardness is calculated by following equation,

\[
H = \frac{P_{max}}{A_r} \quad (4.2)
\]

where \(P_{max}\) is the maximum load applied and \(A_r\) is the residual area left by the indenter, which is evaluated from the shape function of the indenter and the maximum indent displacement. Two typical test curves obtained from the nanoindentation test are shown in Figure 5.9. The Young’s modulus of them are 177.3 GPa and 219.0 GPa, respectively. The results from the nanoindentation tests are concluded in Figure 5.10 and Figure 5.11.

Generally, it can be inferred from the results that there is no significant changing trend for the elastic modulus along the build height. Even though the samples from the middle portion of the part have a higher elastic modulus on the Y-plane, there is no significant difference on the Z-plane. Both the values are comparable with or superior to the literature values and wrought material [19]. The average hardness values on the Y-plane shows a higher value on the bottom sample, then decreases a little with the increase in the build height for the middle-bottom sample, and increases against the end of the manufacturing process for the top sample. This is caused by the cooling rate during the solidification from the melting pool, same as that of electron beam additive manufacturing (EBAM) [17,23]. The different microstructure distribution results in the variation of changing trend of the values. The repeated heating may result in the softening of the already deposited layers, and such influence to the middle layers is most evident.
Figure 5.9 Load vs. displacement curve from the nanoindentation test

Figure 5.10 Young’s Modulus for the SLM-processed Inconel 718 alloy

Figure 5.11 Nano-hardness of SLM-processed Inconel 718 alloy
The highest hardness values are achieved from the bottom and top layers of the parts, which results from the manufacturing process since the mechanical properties of materials are strongly dependent on the microstructures. From the comparison between the width of the columnar microstructure of different layers, it is known that the bottom and top layers achieve the smallest columnar structure width [23]. According to Hall-Petch relation [24,25], the finer grain size strengthens the materials. From this point of view, that is why the middle-top sample 2 and sample 6 both have a relatively lower hardness as they have the widest columnar structures. Most of the hardness values fall in the range of 6.08 – 7.20 GPa, which is 50% higher than the microhardness values obtained from Vickers’ indentation [26], around 3.9 – 4.3 GPa. Overall, the hardness values are superior to the literature results due to the higher cooling rate in solidification during the manufacturing process [19]. In addition, there is some difference between the Y-plane and the Z-plane for the mechanical properties, which may be caused by the different orientation of the grains.

5.4 Conclusions

In this study, the effect of the build height on the mechanical properties and microstructure of Inconel 718 manufactured by selective laser melting (SLM) technology was investigated. Eight samples were cut from the Inconel 718 parts with wire-electrode and then prepared for microstructure observation and nanoindentation test. The main findings are list below.

(1) With heat treatment for the stress relief, the microstructure becomes relatively homogeneous than that in the as-fabricated samples. Laves phase was partly solved into the basic
\( \gamma \) matrix with their morphology changing from coarse and interconnected particles to discrete particles.

(2) The Y-plane (side surface) exhibits columnar microstructures with an average width of 75 to 150 \( \mu \)m. The columnar grains at the bottom layers appear narrower possibly because of a higher cooling rate. The Z-plane (scanning surface) shows equiaxed grains with a feature of patch patterns (each about 100 \( \mu \)m by 100 \( \mu \)m) resulted from the scanning strategy.

(3) There appears only weak crystallographic texture in both the Y-plane and Z-plane surfaces of the SLM Inconel 718 parts, implying no significant anisotropic traits in part mechanical properties, which has been verified by the nanoindentation test.

(4) The SLM Inconel 718 microstructures are composed of high-angle boundaries with a larger fraction of subgrain boundaries, which may result in the instability of the microstructures and require heat treatments after the SLM process.

(5) The Young’s modulus and hardness are comparable with or superior to that in traditional methods or literature values. There is no obvious changing trend along the build height of the parts, even though the side surface of the specimen from the bottom of the part presents narrower columnar grains.
References


CHAPTER 6

RESIDUAL STRESS EVALUATION IN POWDER-BED ADDITIVE MANUFACTURING PROCESSES

6.1 Introduction

Powder-bed fusion additive manufacturing processes (PBFAM) have attracted increasing attention from industries due to their capability of producing functional metal components with excellent mechanical properties [1–3], which are comparable with parts fabricated by traditional manufacturing techniques [4,5]. Two typical PBAM processes are electron beam additive manufacturing (EBAM) and selective laser melting (SLM) [6]. In general, EBAM produce parts in a vacuum chamber, which is suit for processing high melting point and reactive metallic materials, such as Ti-6Al-4V alloy [7], and SLM can fabricate geometrically complex components with high dimensional precision and good surface integrity [8] which is applicable to various raw materials include copper, aluminum, stainless steel, titanium, and nickel alloys [9,10]. During the manufacturing processes in EBAM and SLM, the repeated rapid heating and cooling processes will also result in the residual thermal stresses in the parts, which has a remarkable influence on the finish machining and the geometric resolution of the parts. Therefore, post heat treatment is usually needed to homogenize the microstructure and relieve the residual stresses to achieve the desired microstructure and mechanical properties. Residual stresses not only have a remarkable influence on the geometric resolution of the parts, but also play a crucial role in mechanical performance, integrity, and lifetime of the components [11,12].
Various techniques were developed to measure the residual stress, such as neutron diffraction [13,14], and X-ray [15,16]. Most methods are complicated, expensive or time-consuming [17,18]. However, the instrumented indentation technique (IIT) has attracted extensive interest due to its simplicity, convenience, and applicability at various scales. Based on the experimental correlation between indentation characteristic parameters and residual stresses, Carlsson et al. [19] proposed a model based on Vickers indentation which is an appropriate method for the rapid evaluation of residual stresses in microstructural scale [20,21]. To date, the distribution of residual thermal stresses in EBAM and SLM processes has been studied by many researchers [22–26]. Cao et al. [23] found that the overlap area had higher residual stresses than the inner-pass area, and the variation range of the residual stresses expanded with the increase of the overlap rate. Liu et al. [25] reported that there was an alternative distribution between high residual stress regions and low residual stress regions within a single deposited layer. After investigating the effect of the island size on the residual stress of the fabricated components, Lu et al. [26] revealed that the smaller the island size is, the higher the residual stresses are. However, due to the dramatic variation of the thermal gradient caused by the complicated physical and chemical processes within the melt pool due to the non-equilibrium processing technique of the energy beams, a thorough study is still needed to further understanding the residual stress in the metal components fabricated by power-bed fusion technologies.

In the present study, the residual stress distribution of two typical PBAM processes fabricated components were investigated by Vickers micro-indentation test method. A staircase Inconel 718 part was fabricated to study the influence of the thermal cycles and build height, and two block parts were produced to investigate the effect of the heat treatment for stress relief. The microstructure of the Inconel 718 alloy was characterized and the distribution of residual stress
was investigated by Vickers micro-indentation test method. In addition, the difference between the X-Z side surface (Y-plane) and Y-Z side surface (X-plane) was studied and the effect of thermal cycles, build height, and the post heat treatment for stress relief was also compared and discussed. Four Ti-6AL-4V alloy parts were fabricated by EBAM process to investigate beam scanning speed effect on the residual stress in the part.

6.2 Methodology

Under the assumptions that the deformation resulted from indentation takes place under quasi-static and isothermal conditions and there is no friction contact between the indenter and the half-space, Carlsson and Larsson [18,19] indicated that equi-biaxial residual strain fields can be accurately correlated with the hardness value and the equi-biaxial residual stresses can be related to the size of the contact area. In the Vickers test, it is assumed that elastic recovery does not occur once the load is removed. However, elastic recovery does occur, and sometimes its influence is quite pronounced. As shown in Figure 6.1, the impression shape is distorted due to elastic recovery, which is very common in anisotropic materials.

Figure 6.1. Schematic of the geometry of the Vickers indentation tests [18], and (b) Schematic of the nominal projected contact area $A_{nom}$
The hardness value is accurately given by the formula

\[ H = C\sigma_Y \]  \hspace{1cm} (6.1)

where \( C \) is a constant, depending on the geometry of the sharp indenter only, and \( \sigma_Y \) is the yield strength of the material. For a strain-hardening material \( \sigma_Y \) is replaced by the flow stress at a representative value of plastic strain, \( \varepsilon_{repr} \).

\[ H = C\sigma(\varepsilon_{repr}) \]  \hspace{1cm} (6.2)

It could be expected that the change of the hardness value due to a residual plastic strain can be accurately described by the equation above if the representative strain in those expressions is replaced by the sum of the representative strain and the residual strain. It can then be rewritten as

\[ H = C\sigma(\varepsilon_{repr} + \varepsilon_{res}) \]  \hspace{1cm} (6.3)

\( H \) is the micro-hardness of the tested point, and here

\[ H = \frac{P}{A} \]  \hspace{1cm} (6.4)

\((P \text{ is the testing load used, } N \text{ and } A \text{ is the real projected contact area}), \text{ which is different with the definition of micro-Vickers hardness, where } \frac{HV}{P} = \frac{1}{A_{surf}} \text{ kgf/mm}^2 \text{ (} A_{surf} \text{ is the surface area of the sharp indenter, mm}^2\text{)}; \) \( C \) is a constant, which depends on the geometry of the sharp indenter only.

In order to establish a correlation between the equiaxial residual stress/strain fields and the contact area/indentation hardness, a new indentation parameter \( c^2 \) was introduced in their model to calculate the residual stress for a sharp indentation,

\[ c^2 = \frac{A_{real}}{A_{nom}} \]  \hspace{1cm} (6.5)

where \( A \) represents the real contact area of a sample showing sink-in or pile-up, and \( A_{nom} \) is the nominal contact area directly calculated from the indentation depth \( h_{max} \) without consideration of sink-in or pile up along the contact boundary.
$A_{\text{nom}} = \left(\frac{2h_{\text{max}}}{\tan 22^\circ}\right)^2 = 24.5h_{\text{max}}^2$ \quad (6.6)

Carlsson et al.'s model can be expressed as the following form,

$$c^2 = c_0^2 - 0.32 \ln \left(1 + \frac{\sigma_{\text{res}}}{\sigma(\varepsilon_{\text{res}})}\right)$$ \quad (6.7)

where $\varepsilon_{\text{res}}$ is the residual plastic strain that can be deduced from the changes of hardness according to Tabor's equation [19], $\sigma(\varepsilon_{\text{res}})$ is the flow stress when the effective plastic strain equals to $\varepsilon_{\text{res}}$. $c^2$ and $c_0^2$ are the area ratios for the case with both residual stresses and residual strains and original material, respectively.

In this study, the nominal projected contact area was calculated by the length of the two diagonal lines of the indentation,

$$A_{\text{nom}} = \frac{1}{2} \left(\frac{L_1 + L_2}{2}\right)^2$$ \quad (6.8)

Take Inconel 718 as example, the stress–strain curve obtained by Liu et al. [18] is used in this study. His tensile test result was fitted approximately by a power-law function as

$$\sigma(\varepsilon_{\text{res}}) = \sigma_0 \varepsilon_{\text{res}}^n = 1181\varepsilon_{\text{res}}^{0.1754}$$ \quad (6.9)

Thus, with the formula below,

$$\varepsilon_{\text{res}} = \left(\frac{P}{C\sigma_0 * A_{\text{nom}}}\right)^{\frac{1}{n}} - \varepsilon_{\text{rep}}$$ \quad (6.10)

$$\sigma_{\text{res}} = \sigma(\varepsilon_{\text{res}}) * \left[ e^{\left(\frac{c_0^2 - c^2}{0.32}\right)} - 1 \right]$$ \quad (6.11)

the residual stresses can be calculated with

$$\sigma_{\text{res}} = \sigma_0 \left( \left[ \frac{8 * P}{C\sigma_0 * (L_1 + L_2)^2} \right]^{\frac{1}{n}} - \varepsilon_{\text{rep}} \right)^n * \left\{ e^{\left[ \frac{c_0^2 - c^2}{0.32} \right]} - 1 \right\}$$ \quad (6.12)
When a Vickers indenter is used, \( C = 3 \), \( \varepsilon_{\text{repr}} = 0.08 \) and \( c_0^2 = 1 \) within a certain accuracy standard [13],

\[
\sigma_{\text{res}} = 1181 \left\{ \frac{8 \cdot P}{3 \cdot 1181 \cdot (L_1 + L_2)^2} \right\}^{0.1754} - 0.08 \times e^{\left\{ \frac{1 - 8 \cdot A_{\text{real}}}{(L_1 + L_2)^2} \right\}^{0.32}} - 1 \]  \hspace{1cm} (6.13)

Then, the residual stress can be determined with the formula developed above. For Ti-6Al-4V samples, the stress–strain curve is obtained by Lalit et al. [20]. The power-law function fitted from the tensile results is,

\[
\sigma(\varepsilon_{\text{res}}) = \sigma_0 \varepsilon_{\text{res}}^n = 858 \varepsilon_{\text{res}}^{0.049} \]  \hspace{1cm} (6.14)

Therefore, the residual stress in the EBAM-ed parts could be evaluated using the equation,

\[
\sigma_{\text{res}} = 858 \left\{ \frac{8 \cdot P}{3 \cdot 858 \cdot (L_1 + L_2)^2} \right\}^{0.049} - 0.08 \times e^{\left\{ \frac{1 - 8 \cdot A_{\text{real}}}{(L_1 + L_2)^2} \right\}^{0.32}} - 1 \]  \hspace{1cm} (6.15)

6.3 Experiments

6.3.1 Manufacturing of Ti-6Al-4V samples

A Concept Laser M2 Laser Cusing System at NASA’s Marshall Space Flight Center (Huntsville, AL) was used to fabricate Inconel 718 samples using fine pre-alloyed Inconel 718 powder. Two solid blocks with a dimension of 40 mm by 40 mm by 6 mm were in stress-relieved condition, as can be seen from their black-burned color in Figure 6.2a. They are very different with the as-fabricated brown staircase Inconel 718 part presented in Figure 6.2d. The square island scan strategy/pattern was used during raster-scans thoroughly the inside of the contour of the scanning surface, which is visible to the naked eyes (Figure 6.2d). The size of the
island was set as 5 mm which was determined based on former studies [20]. These islands were selectively melted in a random order with vectors in the adjacent islands perpendicular to each other. The whole pattern was rotated 45 degrees with respect to the substrate plate, which can not only reduce any possible interference between the recoated blade and the straight boundary of each individual island [39] but also reduce the residual stress generated during the manufacturing process [40]. For each island, simple alternating scan vectors with speed of 600 mm/s were used with scan spacing of 54 μm. The island pattern was shifted by 1 mm in both the X and Y direction for each subsequent layer.

To examine the anisotropic conditions in microstructure, eight specimens were cut from the stress-relieved part along the build height, in which S1 (Sample 1) to S4 were used to investigate the side surface (Y plane), and S5 to S8 were used to study the scanning surface (Z plane), as shown in Figure 6.2b. In addition, two other samples were cut from the middle of the second stress relieved part at the same build height from X-plane (sample 1) and Y plane (sample 2) to study the difference between X plane and Y plane, as illustrated in Figure 6.2c. All the samples were prepared by standard metallographic procedures. To reduce the effect of the cutting on the residual stresses, all the samples were removed 2 mm thickness during the grinding process and then polished down to 1 μm.
6.3.2 Fabrication of Inconel 718 samples

An Arcam S12 EBAM machine located at NASA’s Marshall Space Flight Center (Huntsville, AL) was used to fabricate the Ti-6Al-4V parts from the fine pre-alloyed Ti-6Al-4V powder with a diameter between 45 and 100 µm. The parts, having a length of 60 mm, a width of 5.5 mm and a height of 25 mm, were built layer by layer with a layer thickness of 70 µm. For this system, the speed function (SF) is a process parameter setting related to the actual beam scanning speed [41], and four-speed functions (SFx) (SF20, SF36, SF50, and SF65), were used to fabricate the samples to study the beam speed effect on the build parts. The final finishing part built with SF50 is shown in Figure 6.2f. The Ti-6Al-4V samples used in this study were cut from the as-fabricated parts.
6.3.3 Microindentation test

The Vickers indentation tests were performed on each surface of specimens using a model from Buehler Inc. During the test, the sample was put on the horizontal adjustment stage, as shown in Figure 6.3a, which can guarantee the test surface of the sample vertical to the indenter. Because the polished surface might not be parallel to its back surface induced by the sample preparation, and a more than 1° tilting surface from perpendicular can result in nonsymmetrical impressions and can produce lateral movement between specimen and indenter. The minimum spacing (center to the edge of adjacent indent) is 5 times of the Vickers diagonal, the force applied was 500gf at the loading and unloading time of 15 s.

![Image of Vickers indentation test stage and typical residual profiles](image)

Figure 6.3. (a) Vickers indentation test stage, and (b-d) typical residual profiles of Vickers indentation test on the surface of as-fabricated Inconel 718 samples

The typical residual profiles of the Vickers indentation tests on Inconel 718 build height samples are shown in Figure 6.3. Even though it is assumed that elastic recovery does not occur
once the load is removed. However, elastic recovery does occur, and sometimes its influence is quite pronounced. As displayed in Figure 6.3b, the total area of the residual contact area is smaller than its real contact area, while Figure 6.3c is bigger than its real contact area. The sides of the indentation in Figure 6.3d were tortured under the action of force, which is very common in anisotropic materials.

6.4 Results and Discussions

6.4.1 Comparison of residual stresses in X and Y planes

The hardness values and residual stresses on the X-plane and Y-plane of Inconel 718 samples are plotted in Figure 6.4. Based on the average value of the test results, X-plane and Y-plane almost have identical characteristics in hardness and residual stresses, which attributes to the similar microstructures that induced by the identical manufacturing parameters (beam scanning pattern and the beam parameters) in both X-plane and Y-plane. They both have an average Vickers hardness around 4.2 GPa, which is comparable with literature results [33]. This attributes to the optimized manufacturing processes, such as the beam scanning strategy [26] and the beam scanning parameters. In addition, no significant pattern was found for the residual stresses and they are just unevenly distributed in the parts. They both have a compressive residual stress with the maximum absolute value around 320 MPa, which is about 27% of the yield strength of Inconel 718.
6.4.2 Thermal cycle effects

The statistic results of the Vickers hardness for the as-fabricated staircase Inconel 718 part are shown in Figure 6.5. According to the test results, they are in the range of 3.17 Gpa to 3.77 Gpa, which is superior than the results (2.91 Gpa ~ 3.16 GPa) of Chlebus et al. [34] and comparable with the value (3.85 GPa) obtained by Amato et al. [33] and Jia et al. [35] (3.25 Gpa ~ 3.88Gpa). In addition, the specimens from the bottom of the part have higher hardness values, as can be seen from Figure 6.5a-c, which mainly attributes to the finest microstructure as measured above in addition to the difference fraction of phases. As shown in Figure 6.5, sample S3H59 and S3H29 have the lowest and highest hardness values, respectively, which are results from the fraction of Laves phase that is detrimental to the mechanical properties of Inconel 718. As verified by Figure 6.5d-e, the lower volume fraction of Laves phase in S3H59 leads to its high hardness values. Moreover, by comparing the samples with different time duration of the thermal cycles in Figure 6.5a-c, the thermal cycles had effects on the hardness of the part as the
thermal cycles can decrease the fraction of the Laves phase in the sample. Therefore, the hardness values are more likely combination effects of manufacturing process parameters and thermal cycles.

Figure 6.5 Y-plane of as-fabricated SLM Inconel 718 alloy: (a-c) Vickers hardness, and SEM images of (d) S2H29 and (d) S3H29
Figure 6.6 listed the residual stresses of as-fabricated samples that calculated from the Vickers indentation tests. Without stress relief, the samples have a much larger standard deviation. Some test points have tensile residual stresses (Maximum, 145.1 MPa), and some other test points have very big compressive stresses with a maximum absolute value of 378.4 MPa, which is about 32 percent of the yield strength (~1181.2 MPa) of Inconel 718. This was caused by the characteristic of the layer by layer manufacturing process. Generally, the samples have a compressive stress in the Y-plane, as shown in Figure 6.6. The big standard deviation was caused by the inhomogeneity properties of the microstructure in the part. This was also mentioned by former studies [23,25,28], it is said that the big standard deviation of the calculated residual stresses resulted from the real variation of residual stresses from point to point due to the nonhomogeneity of the microstructure.

Figure 6.6 Y-plane of as-barbicated SLM Inconel 718 alloy: (a-c) Residual stresses
Comparing to the residual stress in the stress relieved part, the as-fabricated samples have a higher compressive residual stress in average value and maximum values, which attributes the evolution of the microstructure during the heat treatment for the stress relieving process. According to the results obtained, the thermal cycles have no remarkable effects on the residual stresses in the part. Moreover, if comparing the changing trend of the hardness and the residual stresses in Figure 6.5b-c and Figure 6.6b-c, respectively, the residual stresses have influences on the hardness. However, there is no correlation in the changing trend of hardness and residual stresses in Figure 6.5a and Figure 6.6a. Therefore, the hardness values are more likely to be the consequence of joint contributions of several factors, including phase fraction, thermal cycles, and residual stresses.

6.4.3 Build height effects

Figure 6.7 and Figure 6.8 include the Vickers hardness and the residual stress results changing with the build height of the stress relieved SLM Inconel 718 block. After stress relief, the Vickers hardness values of the samples increased to 3.93 Gpa to 4.31 Gpa, which indicates that there is a 19% average increase in the hardness. This attributes to the homogenized microstructure in the samples as the microstructure had evolved during the heat treatment for the stress relief, as demonstrated in Figure 6.7c and Figure 6.7d. The majority phase in S1 sample is basic γ phase and it has a more homogeneous microstructure that the S4 which still has vast regions that contain a larger fraction of Laves phase, and this accounts for the higher hardness values in S1. It has been reported that the hardness of the as-fabricated sample could have a 48% increase when the microstructure was fully homogenized under proper heat treatments (solutions and double aging) [36,34].
The average value of the residual stresses in the Y-plane and Z-plane are compressive and tensile residual stresses, respectively, as shown in Figure 6.7a and Figure 6.8a. It can be seen that the residual stresses in the block only increased slightly along the direction of the Z direction. By comparing the residual stress in the as-fabricated samples, it was found that there was no significant drop in the average absolute values, but the maximum absolute values did decrease, which is around 320 MPa. In addition, the residual stresses almost do not have influences on the micro-hardness of the Inconel 718, and the Y-plane has a higher Vickers hardness than the Z-plane.

Figure 6.7 Y-plane of stress relieved SLM Inconel 718 alloy: (a) Residual stress, (b) Vickers hardness, and SEM images of (c) S1 and (d) S4
6.4.4 Beam scanning speed effects

Generally, the residual stresses are unevenly distributed in the parts based on the results calculated. For the as-deposited Ti-6AL-4V specimens, some test points almost have none residual stresses, while some other test points have very big compressive stresses and the maximum absolute value is around 181 MPa, which is about 21 percent of the yield strength (858 MPa) of Ti-6Al-4V. The average value of the residual stresses in the Z plane and Y plane both are compressive stress, as shown in Figure 6.10.

For the Ti-6Al-4V parts, the Vickers hardness increases with the increase in electron beam scanning speed, but this changing trend is not very significant if incorporating the standard deviation of the hardness. Generally, these test results are in accordance with our former results [47]. Besides, the EBAM samples also show higher micro-hardness than the cast or wrought specimens, which was also mentioned by Koike et al. [48]. He said it was probably attributed to finer α/β lamellar microstructure. They are comparable/superior to the cast/wrought parts, as shown in Figure 6.9.
6.4.5 Comparison of the Residual Stresses in EBAM and SLM

There is another thing that needs to mention that the EBAM process produced parts have smaller residual stresses than the parts made with SLM process, which can be seen by comparing the Figure 6.10 and Figure 6.6. One of the reasons is the EBM process takes place in a vacuum chamber at high temperature. Thus, the EBM process has a slower cool down rate than SLM process, which results in stress relieved components. The other one is the electron beam preheats the base plate or the entire powder to an optimal ambient temperature before the melting process of the powder in the build [34]. As a result, the parts produced with the EBAM process have smaller stresses than the laser beam produced samples.

Figure 6.9. Hardness of Ti-6Al-4V specimens built with different SFs

Figure 6.10. Residual stresses in the Ti-6Al-4V samples
6.5 Conclusions

In this study, the build height, thermal cycles, and beam scanning effects on the distribution of the residual stresses in metal parts fabricated by powder-bed fusion additive manufacturing technologies were investigated using micro-indentation method. To examine the anisotropic conditions in the fabricated parts, specimens from the scanning surface (Z plane) and the side surface (Y plane, X plane) were prepared. According to the test results, the following conclusions have been found.

(1) The residual stresses are unevenly distributed in the parts. For the Ti-6Al-4V parts, the average residual stress in both Z plane and Y plane is compressive stress, while the Inconel 718 SLM parts show tensile stresses and compressive stresses in the Z plane and Y plane, respectively.

(2) There is no notable difference between the X plane and Y plane of SLM Inconel 718 samples in both hardness and residual stresses.

(3) By comparing the residual stress results, the Ti-6Al-4V parts have lower absolute residual stresses than the Inconel 718 parts, which attributes to the characteristics of the EBAM process, such as the high-temperature vacuum chamber and the preheating process.

(4) The maximum absolute value compressive stress in EBAM Ti-6Al-4V sample is around 181 MPa, which is about 21 percent of the yield strength (858 MPa). For the Inconel 718 parts, the maximum absolute compressive residual stress dropped from 378.4 MPa to 321 MPa after heat treatment for stress relieving, which are 32% and 27% of its yield strength, respectively.
(5) The residual stresses do not change significantly along the building direction of the Inconel 718 part fabricated by selective laser melting, and beam scanning speed and thermal cycles do not show remarkable influences on the distribution of the stress in the parts.

(6) The Vickers hardness values of the SLM Inconel 718 part and EBAM Ti-6Al-4V components are comparable to the literature data. Y plane surface has a higher average hardness value than the Z plane surface. The residual stresses almost do not have influences on the micro-hardness of the Ti-6Al-4V and Inconel 718 parts.
References


[33] Amato, K. N., Gaytan, S. M., Murr, L. E., Martinez, E., Shindo, P. W., Hernandez, J.,


CHAPTER 7
EFFECT OF SUPPORT STRUCTURE ON THE FINAL PARTS

7.1 Introduction

Electron beam additive manufacturing (EBAM) is a powder-bed additive manufacturing (AM) technology [1], which uses an electron beam to selectively melt a metallic powder-bed layer by layer to fabricate metal products. Compared with other AM technologies [2–5], EBAM offers several unique advantages such as high energy efficiency, fast scanning speed and less residual stress in the final parts [6,7]. Therefore, EBAM has attracted ever-increasing interests from aerospace, military and biomedical industries [8–10]. Titanium alloys have rapidly emerging applications in industries due to their high melting temperature, high strength to density ratio, and excellent properties and corrosion resistance [11–13], which makes them having comparative advantages over other competing metallic materials, such as high-strength magnesium alloys and steel. Ti-6Al-4V, as one of the most common titanium alloys, is the workhorse for many aerospace applications (airframes and turbine engines) and it accounts for more than 80% of the total US market usage [14,15].

Various studies have been carried out to characterize the EBAM-produced Ti-6Al-4V components [1,16]. Al-Bermani et al. [17] observed the columnar prior β grains in as-deposited EBAM Ti-6Al-4V sample which was resulted from thermal characteristics of the EBAM process, such as small melt pools and rapid cooling rates. Lu et al. [7] found ultrafine lamellar α and β phases in the massive grains that transformed from prior β phase, which is not available in
conventional components. Murr et al. [18] measured the thickness of the α-lath in varied EBAM processed samples and the value was around 1.4-2.1 µm, which could be caused by the high cooling rates during the solidification. In addition, α′-martensitic platelets were observed in EBAM parts [19], and the EBAM-fabricated Ti-6Al-4V parts had comparable mechanical properties with that of the conventional wrought parts [20,21]. Formanoir et al. [1] reported that horizontally build samples show higher tensile properties which are also affected by the surface finish and the heat treatment. Vickers microhardness values have been reported as varied from 345 to 350 HV [22]. The improvement in mechanical properties of EBAM-fabricated Ti-6Al-4V is probably due to the fine α/β microstructure according to Hall-Petch relation, as mentioned by Gong et al. [19]. In summary, EBAM-processed Ti-6Al-4V specimens present an ultrafine α, α′ and β microstructure and comparable tensile properties of conventional samples.

Even though the EBAM process has various advantages in producing Ti-6Al-4V parts, there are still several challenges, such as the control over microstructural morphology, porosity, shape accuracy, and surface finish [23]. In general, the powder-bed additive manufacturing produced parts do not require support structures for overhang geometry as they can be supported by their surrounding raw powder [24]. However, in practice, the overhang area indeed needs to be supported, otherwise, defects would occur frequently, such as staircase effect, dross formation and warp [25]. When beam scans the surface of the paved powder layer, the heat conduction rate is high at the solid-supported zones and it becomes much lower at powder-supported zones which are often common to be seen in the fabrication process of the parts with overhanging features [26]. This results in a much higher absorbed energy input at the powder-supported zones. The heat would cause unintentional sintering of the powder particles below and result in unsatisfactory downside skin finishes [27]. In addition, this may lead to a large melt pool to sink
into the powder underneath due to a high gravity and capillary forces. Consequently, dross forms easily and dimensional accuracy is not well controlled during fabrication of overhanging surfaces [28]. In addition, the rapid solidification during powder-bed AM processes also induces thermal stresses, which may lead to warping defect when it exceeds the strength of the material [29,30]. The support structure is an essential part in the AM processes as overhanging features cannot always be eliminated through reorientation [27,31]. According to the study of Thomas and Bibb [32], the SLM process is only capable of building unsupported overhanging features inclined up to a maximum of 45° and below which support structures are required. These structures function as anchors for initially floating objects [33], dissipate melt pool heat and prevent thermal warping of the parts [34].

There are seldom studies on the effect of the supporting structures of the part on the microstructure and mechanical properties of the EBAM printed components. This work thus aims to advance the adoption of this technology by studying the microstructure of the final part in the solid region and the overhang region. In addition, the evolution of the microstructure and the relationship between microstructure and mechanical properties were revealed. Moreover, the residual stress distribution in the Y-plane of the parts, and the changing trend from the solid were also investigated.

7.2 Experimental Procedure

Two types of support structures were explored to investigate the practical supports effects. They were molded using CAD software with the specific dimensions shown in Figure 7.1. The first type is named as thin wall tooth contact (TC) support, as shown in Figure 7.1a, in which only the top surface of the thin-wall tooth (1 mm × 1 mm) are in intact with the overhang
to supply the support, and all other places are filled with metal powder during manufacturing process. The other design displayed in Figure 7.1b is called solid-gap (SG) support, in which a support surface is put underneath an overhang with a gap filled in with un-melted metallic powder during manufacturing process. The support structure acts as a heat sink to enhance heat transfer.

![Figure 7.1. Schematic of Ti-6Al-4V overhang parts (a) tooth contact (TC), (b) solid-gap (SG), and (c) locations of the samples (S1-4) cut from the part](image)

Table 7.1 Compositions of Ti-6Al-4V powder [35]

<table>
<thead>
<tr>
<th>Composition</th>
<th>Al</th>
<th>V</th>
<th>C</th>
<th>Fe</th>
<th>O</th>
<th>N</th>
<th>H</th>
<th>Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti6Al4V (wt. %)</td>
<td>6</td>
<td>4</td>
<td>0.03</td>
<td>0.1</td>
<td>0.15</td>
<td>0.01</td>
<td>0.003</td>
<td>Balance</td>
</tr>
</tbody>
</table>

The two overhang components were fabricated by an Arcam S12 EBAM machine at NASA’s Marshall Space Flight Center (Huntsville, AL). During the fabrication process, the beam size was 0.5 mm and the upper chamber was kept at $7.5 \times 10^{-7}$ Torr (1 Torr = 0.0013157895 atm) to keep the quality of the electron beam, and the fabrication chamber was maintained at a pressure of $7.5 \times 10^{-5}$ Torr to avoid oxidation of titanium. It was built layer by layer with a layer thickness of 70 µm through melting the fine pre-alloyed Ti-6Al-4V powder. The majority diameter of the powder falls in 45 and 100 µm, and their compositions are listed in
Table 7.1. The completely manufacturing process involves 4 stages, including powder, spreading, pre-heating, contour melting and hatch melting. In melting stages, an electron beam would be first melt the contour of the powder layer surface by tracing the model cross-section boundary and then raster scanned thoroughly the inside of the contour.

The samples used in this study were cut from the as-deposited parts by the TechCut 4TM precision low speed saw with the location of them shown in Figure 7.1. Four samples (S1-S4) were cut along X-axis to reveal Y-plane from the as-built part. Except S2 containing a transition zone from solid area to overhang area, S1 and S3-4 are located at the solid area and overhang area, respectively. Then, they were hot-mounted in PolyFast thermoplastic resin from Struers with the standard metallographic procedure followed to prepare the samples for microstructure analysis. The final step was conducted using a vibratory polisher with 0.05 μm diamond suspension. For microstructural analysis, the final polished specimens were etched with the Kroll’s Reagent to reveal the microstructures. The etched metallographic samples were examined using an optical microscope (OM) and a JOEL F7000 scanning electron microscope (SEM). Electron backscatter detection (EBSD) was also conducted using a JEOL 7000 scanning electron microscope equipped with an EBSD camera to analysis the texture in the specimens with a scanning size of 500 μm by 500 μm and a step size of 1.5 μm.

Vickers indentation tests were conducted to obtain the Vickers hardness and calculate the residual stresses in the samples. A pattern of 18 × 3 was used for the 4 samples with the tests points almost uniformly distributed on the surface of the part. The load of 1 kgf was used for all the tests with a loading time of 10s. In addition, Carlsson and Larsson [36] model was used in this study to evaluate the residual stress in the part considering of its simplicity and convenience [37].
7.3 Results and Discussions

7.3.1 Microstructure characteristics

The microstructure of the as-fabricated EBAM Ti64 samples under an optical microscope are listed in Figure 7.2 and Figure 7.3. Columnar prior β structures were revealed in S1, which is a typical feature commonly observed in metals subjected to rapid cooling processes, such as laser cladding and selective laser melting [40,41]. During the initial rapid solidification, the nucleation and growth of columnar grains of prior β occurred when the temperature was above the β-transus temperature. They grew along the Z-axis (build direction) and across tens of layers in the specimen. This is significantly affected by the heat flux during solidification. The rapid heat loss via conduction into the stainless steel base plate was dominant at the initial stage of manufacturing process.

With the increase of the build height, the heat loss from the melt pool through the surrounding powder also occurred [42]. It is obvious that the main direction of the prior β grains is in Z-axis at the center position of the S1, as shown in Figure 7.2b. The prior β grains near the contour section are inclined to grow in a direction toward to the center of the specimen, as shown in Figure 7.2a. The angles between the growing directions with the Z-axis are determined by the heat flux during the solidification. This also was observed in the corner area of S2, as illustrated in Figure 7.2c.
For the specimens from the overhang section, equiaxed grains were observed in the Y-plane at the area near the bottom surface, as presented in Figure 7.3a and Figure 7.3c. During the manufacturing process, the bottom of the overhang was surrounded by the raw powder. The β grains initially nucleated with random orientation at the first-melt layers, as there was no obvious thermal gradient due to the ineffective heat loss through the metal powder. The conduction of the powder is much lower than the stainless steel base stage. With the increase of the height, the heat loss from the melt pool to the pre-solidified layers would be gradually dominant during the
solidification. Then, the β phase (cubic crystals) in the chill zone grew dendritically in <100> direction, which is close to the direction of heat flow. They grew fastest and were able to outgrow less favorably oriented neighbors. This leads to the formation of the columnar grains all with <100> almost parallel to the Z- axis. The transition area can be seen clearly in Figure 7.3c.

The lower cooling rate caused by the ineffective heat loss at the overhang area also lead to wider columnar structures, as shown in Figure 7.3b and Figure 7.3c. It is known that a higher cooling rate and the thermal gradient will form smaller columnar β grains because more nuclei can be generated at a higher cooling rate and form finer grains in a long and narrow columnar morphology [43]. The width of the columnar β structure close to the top surface of the overhang was characterized by a statistical method, and the measuring example is shown in Figure 7.3b. According to the statistical values listed in Figure 7.3e, the overhang region has a width of 176.3 – 194.4 µm while the solid region only shows a width of 65.9- 70.2 µm, especially the contour scanning region which only shows a width of 46.5 µm. This attributes to the high cooling rate at the contour region, as they were in direct- contact with the metal powder without further heat on the other side. Some of the heat was passed to the metal powder in addition to the previous fabricated solid, and this results in a larger cooling rate in the contour scan tracks than the inner hatch scan beads. This is consistent with literature reports that the width of the columnar structure falls in 20 to 200 µm [17,44].
Figure 7.3. EBAM-fabricated Ti-6Al-4V TC sample: optical microstructure of (a, b) S3, and (c, d) S4, and (e) width of columnar structures changing along the X axis at the height close to the top end surface.
The microstructures from the Y-plane of the SG samples are shown in Figure 7.4. The microstructure of SG part presents some similar characteristics with the TC part. For example, columnar microstructure was revealed in the contour scanned region, which has an angle with the Z-axis that determined by the heat loss through the metal powder and the previously solidified layers. The columnar structure at the middle of the solid regions in the part grows along the Z-axis, which has a narrower width than that in the overhang region, as can be seen from Figure 7.4a and Figure 7.4b. In addition, more defects were revealed around the corner of the interface, including pores and un-melt metal powder.

However, the SG part shows a slow changing trend in the width of the columnar structure, while TC part presents a sharp increase/drop at the interface from the solid to the overhang and the right tip end of the overhang feature. This was mainly caused by the tooth support, which leads to a fast cooling rate. However, due to the larger distance from the middle top surface of the sample to the cuboid tooth top surface, there is no bigger difference in the width of the columnar structure for the overhang region. As shown in Figure 7.3se and Figure 7.4e, the SG part only presents a slight wider columnar structure than the TC part. This indicates a slight lower cooling rate at the centerline along the X-axis which attributes to the thick loose metal powder underneath of the overhang region. On the other hand, this implying that the solid part in the SG sample did act as a heat sink to enhance heat transfer during the manufacturing process which increased the thermal gradient at the overhang region.
Figure 7.4. EBAM-processed Ti-6Al-4V SG sample: optical microstructure of (a) S1, (b) S3, (c) S2, and (d) S4, and (e) width of columnar structures changing along the X axis at the height close to the top end surface.
Another significant difference is the surface roughness. As displayed in Figure 7.5, the TC part was full of bumps and hollows with a lot of un-melt metal powder attached to the bottom surface TC part, while the SG part has an almost clean flat surface. In addition, the SG part has a much smooth bottom surface than the TC part. This indicates that sink phenomena occurred during the manufacturing process of the TC part, and the serious location was shown in the enlarged area in Figure 7.5a, in which the melted metal was totally separated from the main body. During the manufacturing process, the loss metal powder under the overhang region can not supply enough strength support for the melted metal which has a higher density than the powder. This leads to the occurrence of the roughness of the surface and the sink effects. From this comparison, the results did state that the SG support design could supply some support for melt metal. The height of the as-fabricated overhang feature was measured based on the optical images with the measuring examples shown in Figure 7.5a-b. The statistic results were plotted in Figure 7.5c. It can be seen that there is no significant difference in the height of the overhang region, however, the TC part does present a much larger standard deviation, which is about 4 times of that from SG part.
Figure 7.5. Compare of the equiaxed grains and the transition zone from equiaxed to columnar grains in (a) TC part (H1/H2: height of the overhang feature, T1/T2: height of the transition zone, P: metal powder) and (b) SG part, (E1/E2: thickness of the equiaxed grains), and (c) the statistical results.

In addition, only one or two layers of equiaxed grains were observed at the bottom of the overhang area in TC part while three or four layers were revealed in the SG part, as illustrated in
Figure 7.5. The characterized results of the thickness of the equiaxed grains for the TC and SG parts are 0.559 mm and 0.893 mm, respectively. However, the TC part presents a fast transition from the equiaxed grains to the columnar grains, as displayed by transition grains highlighted by the disconnected lines in Figure 7.5a-b. These phenomena were caused by the variation of the cooling rate during the manufacturing process. As the boundaries of the overhang region for the TC part was supported by the thin wall tooth structure, the main heat loss path for the overhang would be from the melt pool to the tooth contact support structure due to the much higher thermal conductivity of the solid than the powder. This definitely lead to a higher cooling rate and a faster changing rate than that in the SG part, which further resulted in the fast transition from the equiaxed grains to the columnar structure.

7.3.2 Advanced analysis of microstructure

The SEM morphology of Ti-6Al-4V alloy from S4 is shown in Figure 7.6. Fine Widmanstätten structure ((\(\alpha+\beta\)), were identified inside of equiaxed/columnar grains. However, the bottom area shows a fine fully equiaxed type \(\alpha/\beta\) microstructure (Figure 7.6d) while the top area presents a fully lamellar type \(\alpha/\beta\) microstructure (Figure 7.6b), which indicates a varied cooling rate in the EBAM overhang region. During manufacturing, the main heat flux path is from the melt pool to the powder at the initial layers and it will gradually change to the path through the former solidified layers, which is especially at the overhang area. In addition, it is difficult to keep the electron beam scanning speed at a constant, which will change along with the build height according to our former study [19]. The thermal history has significant effects on the microstructural evolution [3,45]. Upon continuous cooling from the \(\beta\)-transus temperature, the initial \(\alpha\)-phase nucleates at \(\beta\)-grain boundaries. The cooling rate during the allotropic
transformation from the bcc $\beta$-phase to the hcp $\alpha$-phase is the most important parameter controlling the final microstructure. Depending on thermal processing conditions, the $\alpha$ particles develop into equiaxed or acicular lath structures [46]. In the columnar $\beta$ field, $\alpha$-phase eventually grows as parallel lamellae with the same crystal orientation within the same $\beta$ grain until it meets other colonies nucleated at other grain boundaries. There is no preferred direction for the $\alpha$-phase within the equiaxed $\beta$ grains. However, all of the individual $\alpha$ lamellae are separated by a very small amount of $\beta$ in $\alpha$ boundaries [47].

In addition, there is a significant changing in the $\alpha$ lath grow direction. As in Figure 7.6a, lots of $\alpha$ laths growing almost parallel to the beam scanning plane, which indicates that there is a higher thermal gradient in the plane and a lot of heat loss horizontally. Considering the distribution of the support teeth, it is not difficult to understand that the vast of heat loss path was lost from the melt pool to the tooth contact support structure located at the boundary of the overhang district in addition to the previous solidified solid, which leads to this morphology of $\alpha$ laths.
Figure 7.6. SEM images of the microstructures of Ti-6Al-4V samples from TC part

The variation of the thermal process that caused by the changing of the external morphology of the part also has a significant influence on the microstructural evolution during the EBAM process, including the fraction of the phases. At the area close to the top surface of the specimen, martensitic phase, $\alpha'$, in shape of the plate was also observed [48]. The $\alpha'$ is transformed from the $\beta$ phase due to a very high cooling rate. The high-magnified images for regions A1 to A3 in Figure 7.4d under SEM are shown in Figure 7.7, which shows clearly that with the increase of the build height, the proportion of $\alpha$ phase decreases along with the increase of martensitic phase, $\alpha'$. This also can be seen intuitively in Figure 7.3d and Figure 7.4d. in
which the differences between them can be distinguished by the brightness under the optical microscope that looks like there are four layers.

Figure 7.7. Microstructure of EBAM-processed Ti-6Al-4V SG samples under SEM (A1-bottom, A2-middle, and A3-top)

The volume fraction of the β phase in the specimens, which is usually located at the grain boundaries and triple points with a white color under SEM, has been characterized by estimating the fraction of the white area using ImageJ. The final statistical results are shown in Figure 7.8 and the measuring examples are shown in Figure 7.8e-f. The fraction of the β phase in the TC part at the height around the bottom surface of the overhang region increases from the solid region (7.8%) to the overhang area (13.9%). For the area in the Y-plane close to the top surface of the samples, the β-phase fraction is higher than the area close to the bottom surface, and the overhang region shows higher statistic values.

Moreover, the variation of the thermal process also affects the characteristic size of the phases. Figure 7.8b lists the statistic results of the α-lath width in the samples, which was

164
characterized using SEM images. Based on the quantized values, the width of the α-lath increases from the solid region to the overhang area. It increases from 0.60 μm to 0.75 μm, and from 0.79 μm to 1.79 μm, for the bottom and the top area, respectively. Furthermore, at the location close to the top surface, the α-lath has a higher value than the location close to the bottom surface of the overhang region, as clearly shown in Figure 7.8b.

The SG part also presents a similar changing trend with the β phase, as listed in Figure 7.8d. One of the significant difference in β fraction is that SG part shows higher values than the TC part at the area close to the top surface. This is because a lot α′ phases transformed from prior β phase has been included in the statistic results as the α′ phase also has a shallow white color. In addition, the overhang region of the SG part has a finer α-lath than the TC part. This is because the solid part underneath of the SG overhang part acted as a heat sink to enhance heat transfer, which leads to a higher thermal gradient and further led to a coarser α lath. However, the tip end of the overhang region in TC shows a much finer α lath than the SG part, which attributes to the higher thermal gradient results from the tooth support at the contour area.

There are some defects in the overhang part. First, S4 has a curved bottom surface, which could be caused by the residual stresses. Based on former studies [49,50], as the Y-plane of the as-deposited part has compressive residual stresses which may lead to the tip of the part curved toward the positive Z-axis. Second, unmelt powder and pores were observed in the part [51,52], as shown in Figure 7.9a, in which area A1 stands for the big spherical pores and A2 represents the irregular pores. In addition, some of the porous regions are also accompanied with un-melted particles and unconsolidated materials, as displayed in Figure 7.5c and Figure 7.9c. Pores are noticeable in all the specimens, and the statistical data collected are plotted in Figure 7.9b, which indicates that the overhang area has a higher porosity than the solid area.
Figure 7.8. Statistic results of fraction of $\beta$ phase width of the $\alpha$ lath for (a, b) TC samples and (c, d) SG samples, and (e, f) examples of the measurements.

Figure 7.9c illustrates the defects presented at the corner of the overhang part. There are two big holes with a length of about 500 $\mu m$. In some holes, there are some un-melted particles accompanied with segregation. In addition, there is a white area with a crack at the center of it. Through EDX analysis of this region, it reveals the presence of Cr, Fe, and Ni that all act to stabilize the $\beta$ phase. For the stuff filled around the unmelt powder, it is segregation as it has a higher content of oxygen.
Figure 7.9. Y-plane of the SG samples: (a) porosity, (b) statistic results of porosity, and (c-d) EDS analysis of the microstructures.

<table>
<thead>
<tr>
<th>Precipitates</th>
<th>Composition (Wt %)</th>
<th>Powder</th>
<th>Carbide</th>
<th>β + Carbide</th>
<th>α phase</th>
<th>Carbide</th>
<th>β phase (V)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti</td>
<td>90.0</td>
<td>57.9</td>
<td>68.6</td>
<td>89.6</td>
<td>23.1</td>
<td>91.0</td>
<td></td>
</tr>
<tr>
<td>Al</td>
<td>6.0</td>
<td>2.5</td>
<td>4.3</td>
<td>5.5</td>
<td>0.8</td>
<td>1.8</td>
<td></td>
</tr>
<tr>
<td>V</td>
<td>4.0</td>
<td>0.9</td>
<td>6.1</td>
<td>3.3</td>
<td>1.8</td>
<td>6.6</td>
<td></td>
</tr>
<tr>
<td>Fe</td>
<td>&lt;=0.25</td>
<td>4.8</td>
<td>2.0</td>
<td>0.2</td>
<td>1.9</td>
<td>0.5</td>
<td></td>
</tr>
<tr>
<td>O</td>
<td>&lt;=0.2</td>
<td>2.6</td>
<td>8.3</td>
<td>0.9</td>
<td>30.2</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ni</td>
<td></td>
<td>25.0</td>
<td>6.3</td>
<td>0.8</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Si</td>
<td></td>
<td>0.2</td>
<td></td>
<td></td>
<td></td>
<td>1.4</td>
<td></td>
</tr>
<tr>
<td>F</td>
<td></td>
<td>4.7</td>
<td>4.3</td>
<td></td>
<td></td>
<td>3.2</td>
<td></td>
</tr>
<tr>
<td>Cr</td>
<td></td>
<td>1.5</td>
<td>0.7</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Na</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>8.7</td>
<td></td>
</tr>
</tbody>
</table>

Note: The table shows the composition of precipitates in different phases and phases (V) for different powder carbide compositions.
7.3.3 Texture analysis

The EBSD results were displayed in Figure 7.10, in which the columnar and equiaxed microstructures were revealed clearly in the orientation maps with the boundaries of the β' microstructures marked with white lines in the image quality (IQ) maps (Figure 7.10b,e). Some narrower columnar structures were observed in the wider ones, and α phases within them almost share the same orientation, as marked by the thin white lines in Figure 7.10b. This attributes to the preferential growth of the α phase parallel to the (110) family of crystallographic planes of the β phase during β → α/α' phase transformation [53]. Some minor α phases present different variants, but they are inherited from the same parent β grain. Generally, the IQ maps have a good overall quality. Some dark areas indicate a poor relative image quality which is caused by the higher internal strains associated with the α' formation that would degrade the quality of the EBSD image obtained at these analysis points [54].

The EBSD pole figures for the α phase and β phase show a strong texture (Figure 7.10g,h). The columnar β phase in Figure 7.10a presents a strong intensity with its max value about 20, which is much like rolled sheet counterparts, and the equiaxed β phase shows a strong cube like texture (Figure 7.10g). During the phase transformation stage, the prior directional solidified β phase transferred into α/α' phase following the Burgers relationship, \{0001\}_α \parallel \{110\}_β (parallel planes), and \{1120\}_α \parallel \{111\}_β (parallel directions). Correspondingly, the texture in β' phase will be transmitted to a texture in the α/α' phase, as shown in the \{1 1 2 0\} α pole figure, Figure 7.10c, where the \{1 1 2 0\} α pole figure looks similar to \{1 1 0\} β pole figure [53,55]. As shown in Figure 7.10g, α phase have strong textures of \{0 0 0 1\} and \{1 1 2 0\} parallel to the z-axis that transferred from the β’ phase. However, the intensities of the α phase
texture are much lower than those of the β’ phase, which attributes to the 12 distinct variants of α orientations can be formed from one parent β grain.

Figure 7.10. Ti-6Al-4V SG part: (a, d) Orientation maps from top and bottom of S3, (b, d) Image qualify maps, and (c, f) Pole figures of α and β phases corresponding to (a, d) respectively
EBSD results for S4 are shown in Figure 7.10c and Figure 7.10f. Comparing to S3, there is no remarkable change in the texture maximum intensity at the top area while a sharp increase at the bottom region. This attributes to the narrower columnar structures at the tip of the overhang section present strong textures with higher intensities in both β phase and α phase, as also can be seen from the columnar grains in the grain map displayed in Figure 7.10i.

7.3.4 Microhardness

The Vickers hardness of the TC and SG parts changing with the distance from the left solid side along the x-axis were plotted in Figure 7.11 and Figure 7.12. Generally, the solid region of the part shows a slightly higher average value (3.41/3.67 GPa) than the overhang section (3.29/3.66 GPa), as can be seen in Figure 7.11. This attributes to the differences of the microstructure size. As mentioned above, both the width of prior β structure and α lath have a larger size at the overhang region than the solid area. At the right end of the overhang region, the TC part shows a slightly increasing trend while the SG part presents a continued decreasing trend. This corresponds to the changing trend of the microstructure size that resulted from the difference of the support structure. The SG part also presents higher hardness values than the TC part, as illustrated in Figure 7.11. This mainly caused by the phases and grain size. As mentioned above, the SG part has a higher fraction of α′ phase and finer microstructure, which leads to the higher microhardness values. The average values are in the range of 3.16 GPa to 3.50 GPa for the TC part and 3.58 GPa to 3.78 GPa for the SG part.

The hardness values changing vertically along the build height are shown in Figure 7.12. The top of the TC and SG parts present slight decreased average values at the overhang region, which was caused by the increase of the β phase in addition to the effects of grain size. Even
though the bottom of the SG part has an increase in the hardness, it is not remarkable once considering its largest standard deviation, as indicated in Figure 7.12b. The changing trend for the hardness at the solid section agrees well with the former study that the hardness values would have a slight increase at the end of the manufacturing process [51, 52].

Figure 7.11. Average Vickers Hardness, $H_v(1kgf)$, of EBAM Ti-6Al-4V parts changing with the distance from the solid side along the X axis: (a) TC part and (b) SG part
7.3.5 Residual stress

The average value of the residual stress at the centerline of the TC and SG parts changing along the X-axis with the distance from the solid side were plotted in Figure 7.13. In general, the EBAM fabricated parts have a compressive residual stress randomly distributed in the Y-plane along the center line. As shown in Figure 7.13a, the TC part has a compressive residual stress with some area presenting tensile residual stress at the X-axis of 16-17 mm. The maximum absolute value of the residual stress is about 271 MPa, which is ~31.6% of its yield strength (~858 MPa) [53]. The average value of the compressive residual stress for the TC part is about 200 MPa in the solid area and 77 MPa in the overhang region, which results from the lower temperature gradient during the manufacturing process. However, there is no significant change in the absolute values of the residual stress for the SG part, as shown in Figure 7.13b, which also indicated that the solid part beneath the overhang feature did increase the thermal gradient during the manufacturing process. On the other hand, the larger residual stress led to the warping of the
overhang sample at the tip of the S4, as can be in Figure 7.3d, as there are no constraints at the right-end terminal of the overhang region in the SG part.

In addition, there is a sharp increase in the absolute residual stress at the transition region. The average compressive residual stress in the TC part increased from 197 MPa to 238 MPa and then decreased to 87 MPa. This is even more obvious in the SG part as the residual stress increased from (164 MPa) to the overhang features (233 MPa), and then decreases to 156 MPa. By examining the hardness values changing vertically along the build height, the BTM values for the SG part increased from 86 MPa to 260 MPa and then decrease to 159 MPa, as presented in Figure 7.14b. This is brought about by the changing of the macroscopic geometry from the left solid region to the right overhang section, where forming a right angle interface between them and this would cause the stress concentration at the corner, same as the finding through simulation from Bobbio et al. [54].

Moreover, compared to the SG part, the TC part showed a larger increase in compressive residual stress at the right end of overhang region, especially the bottom area. This is caused by the higher temperature gradient at the right end of the TC part the heat can be passed away through the tooth support. By comparing the changing trends in the residual stress and the hardness shown in Figure 7.14 and Figure 7.12, the residual stresses have effects on the hardness of the Ti-6Al-4V part, but no significant effects can be observed.
Figure 7.13. Residual stress of EBAM Ti-6Al-4V parts at various locations changing with the distance from the solid side along the X axis: (a) TC part and (b) SG part
Figure 7.14. Residual stress of EBAM Ti-6Al-4V parts at various locations changing along with the distance from the solid side along the X axis: (a) TC part and (b) SG part

7.4 Conclusions

In this study, the effects of support structures (solid-gap vs. tooth contact) on the microstructures and mechanical properties of the Ti-6Al-4V parts fabricated by EBAM were experimentally investigated. Based on the results, the major findings are summarized as follows.

(a) Generally, the Y-plane has a typical β columnar structure with fine (α+β) within it. Martensitic phase, α’, was also observed in the specimens with its content increased from the bottom to the top of the part.

(b) The equiaxed microstructure was observed at the area near the bottom surface of the overhang section and gradually grew into wider columnar structures, around 176.3 – 202.5 µm, in contrast to about 65.9 – 70.2 µm from the solid substrate section. The width of the α-lath increases from the solid substrate (0.60 µm – 0.75 µm) to the overhang area (0.79 µm to 1.79 µm).
(c) The overhang section presents more defects, including porosity and un-melted particles, compared to the solid section. The largest pores, with a diameter of about 500, were observed at the interface of the solid substrate and the overhang.

(d) There is a strong texture presenting in the $\beta'$ phase, and the texture will transfer to the texture of phase during the phase transformation from $\beta'$ to $\beta$ and $\alpha'$, which have strong textures of and parallel to the z-axis.

(e) Generally, the solid substrate region has slightly higher average values of hardness than the overhang which mainly attributes to the morphology of microstructure, such as the composition of the phases and the size of the microstructure. The Vickers hardness of the SG part is in the range of 3.58 to 3.78 GPa while the Vickers hardness for the TC part is in the range of 3.16 to 3.50 GPa. This is caused by the difference of the support structures as a lower fraction of $\alpha'$ phase and coarser microstructure was observed in the TC part.

(f) Compressive residual stresses were measured in the parts with some locations showing minor tensile residual stresses. The maximum absolute value is around 271 MPa, which is about 31.6% of its yield strength and there is a sharp increase in the average residual stress magnitude at the interface corner between the solid substrate and the overhang because of possibly the stress concentration at the corner due to the change of the geometry.

(g) The solid part beneath the overhang region in the SG part may function as a heat sink to enhance heat dissipation during the manufacturing process, which alters the process thermal characteristics, and thus, overhang microstructures.
References


8.1 Introduction

During the selective laser melting (SLM) process, metallic powder particles are selectively melted by a laser beam, and then rapidly cool and solidify [1,2]. Fine microstructure commonly forms during the solidification process [3,4], which benefits to the properties of the fabricated part. Multiple physical phenomena occur during the microstructural evolution process, which is affected by both processing parameters and material parameters [5–7]. In order to control the microstructure and achieve the desired properties, fundamental knowledge about the mechanism of microstructural evolution process in SLM is required because the dendritic growth behavior directly determines the microstructure evolution [8–10]. Many experimental and theoretical studies have been carried out to characterize the fabricated materials [11–14]. Nowadays, the advance of the computing capacity enables the prediction of the dendritic and cell morphology in the molten pool during solidification using the emerged simulation techniques. Two of the most used simulation techniques are phase field method and cellular automata method [15]. As the most powerful numerical scheme, phase field method is more popular than cellular automata method due to its diffuse interface description [16,17]. However, it still requires considerable computation time and can only simulate very small domains with a few dendrites [18].
There are few studies about the simulation of dendritic morphology growth during the rapid solidification process [19–21]. Yin and Felicelli [17] simulated the dendritic structure during solidification of a Fe–C alloy in the molten pool of the LENS laser deposition process using a combined FE-CA technique. Tan et al. [15] developed a combined CA–PF model which utilized the phrase field method for growth kinetics and cellular automata method to track solid/liquid interface. Fallah et al. [6] developed a quantitative phase field model for the laser powder deposition of Ti–Nb alloy to simulate the dendrite growth under local steady-state thermal conditions which were obtained from the previously developed thermal model. Farzadi et al. [22] investigated the evolution of solidification microstructure in the weld pool of gas-tungsten arc welding using phase field method, in which the solidification parameters, the temperature gradient and solidification growth rate, were extracted from a macroscopic heat transfer and fluid flow model. Wang et al. [23] studied dendrite growth under forced flow conditions in an Al-Cu Gas Tungsten Arc (GTA) welding molten pool using phase-field model. The dendrite morphology and solute distributions of the Al–4 wt-% Cu alloy in Gas Tungsten Arc Welding (GTAW) welding molten pool under transient conditions were also investigated [24]. Gong et al. [19] studied the microstructural evolution of Ti-6Al-4V alloy in the electron beam melting process and reported that undercooling affected the columnar growth significantly.

To date, no comprehensive study of the microstructural evolution of the rapid solidification of Inconel 718 alloy in SLM has been reported yet.

The objective of this numerical study is to better understand the microstructural evolution, in terms of phase transformation and solute distribution, in SLM built Inconel 718. A phase field model was applied to model the dendritic formation in SLM. The intent is to predict the microstructure evolution during the solidification process in SLM.
8.2 Mathematical formulation

8.2.1 Thermal analysis model

Finite element method (FEM) has been used to simulate the transient temperature field during the deposition of two consecutive layers of Inconel 718. Figure 8.1 is the schematic of the geometry for the model, which contains two basic domains, the two layers of metallic powder on the top (Domain A) and the solid substrate (Domain B). Domain A includes two thin powder layers on the top of the domain B. Two sequential layers are simulated for the multi-layer manufacturing process. The first scan which moves from left to right on the first powder layer, and then right to left during the second scan on the second powder layer. In this study, the distance for a single scan is 8 mm and the layer thickness is 0.1 mm, and the substrate thickness is 8.8 mm.

![Figure 8.1 Schematic of the simplified simulation domain](image)

The microstructure evolution during SLM is determined by the thermal history of the materials, which is the result of energy absorption by the materials, heat conduction within the built part, and heat losses. With the assumption of negligible molten flow during the solidification process, the governing equation of heat transport during the SLM process becomes thermal conduction based [25],
\[ \nabla (\kappa \nabla T) + \bar{Q} = \frac{\partial \left( \rho c_p \cdot T \right)}{\partial t} + v \frac{\partial \left( \rho c_p \cdot T \right)}{\partial x} \]  
\[ (8.1) \]

where \( \kappa \) is thermal conductivity, \( T \) is temperature, \( \rho \) is density, \( \bar{Q} \) is the absorbed heat flux, \( c \) is specific heat capacity, and \( v \) is the constant speed of the moving heat source on the scanning surface.

The latent heat of fusion, \( L_f \), was considered in this model to track the solid/liquid interface of the molten pool. When the temperature drops between the liquidus and solidus temperatures, \( T_L \) and \( T_S \), respectively, the latent heat of fusion is modeled as an additional term of the internal thermal energy per unit mass, \( dU \). Hence, the enthalpy is defined as:

\[ H(T) = \int C_p dT + L_f f_L \]  
\[ (8.2) \]

where \( L_f \) is the volumetric liquid fraction, which is defined as

\[ f_L = \begin{cases} 
0 & \text{if } T < T_S \\
\frac{T - T_S}{T_L - T_S} & \text{if } T_S \leq T \leq T_L \\
0 & \text{if } T < T_L 
\end{cases} \]  
\[ (8.3) \]

In this study, the intensity of the heat source for the 2D model is described as,

\[ S(x, y) = f(y) \frac{8\eta U I_b}{\pi \Phi^2} e^{-\frac{8(x-x_c)^2}{\Phi^2}}, \quad y < h \]  
\[ (8.4) \]

With \( f(y) = \frac{2}{h_p} \left(1 - \frac{y}{h_p}\right) \)  
\[ (8.5) \]

where the parameters are: efficiency coefficient \( \eta \), voltage \( U \), current \( I_b \), penetration \( h_p \), beam diameter \( \Phi \). This subroutine of DFLUX can read the simulation time and determine the beam center position and raster direction before each calculation so that the domain of the volumetric heat flux can be determined. The magnitude of the heat flux at each node will be interpolated with the heat source equation [25].
The geometric boundary conditions are shown in the model configuration (Figure 8.1). The bottom of the substrate is set as stable. Therefore, only the top section of the actual substrate is simulated; all the mechanical degrees of freedom are confined at the bottom of the substrate due to the bottom being bound by the solid bulk material, which is not included in this study. The contact between the solid bulk material and powder is assumed to be as same as the solid.

There are two cooling steps: the first cooling stage, which lasts a few seconds, occurs during a pause between the first and the second powder laying scans; the second cooling state to room temperature commences after both powder layers have been scanned. The time for the first cooling step is set at 3 s, which approximates the break for the new powder spreading in SLM machine. The thermal boundary conditions are the same as those in the melting step. The second cooling step is defined in the same way as the final cooling step in the single straight scan simulation, which is 50 s in this simulation. The final temperature of the model is assumed to be room temperature (20 °C). The parameters used in the thermal simulation are listed in Table 8.1.

<table>
<thead>
<tr>
<th>Thermal-physical parameters</th>
<th>Values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Beam power, $P$ (W)</td>
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</tr>
<tr>
<td>Beam diameter, $D_b$ (mm)</td>
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</tr>
<tr>
<td>Absorption efficiency, $\eta$</td>
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<tr>
<td>Power layer thickness, $T_{Layer}$, mm</td>
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<tr>
<td>Powder porosity, $\theta$</td>
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<td>Beam penetration depth, $P_D$ (mm)</td>
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<td>Scanning speed, $mm/s$</td>
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<td>Thermal initial condition, $T_{Initial}$ (°C)</td>
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<td>Emissivity, $\epsilon$</td>
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<tr>
<td>Convection coefficient, $h_v (W/(m^2K))$</td>
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<tr>
<td>Solidus Temperature, $T_S$ (K)</td>
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</tr>
<tr>
<td>Liquidus Temperature, $T_L$ (K)</td>
<td>1633</td>
</tr>
</tbody>
</table>

8.2.2 Phase Field Model

(a) Sharp interface problem

Consider the solidification of a dilute binary alloy made of substances $A$ and $B$, with an idealized phase diagram that consists of straight liquidus and solidus lines of slopes $m$ and $m/k$, respectively, where $k$ is the partition coefficient [26]. The interface between the solid and liquid is considered by smooth but rapid change in phase field variable. The assumption that solute diffusivity in the solid is very small compared to liquid diffusivity, and the solid/liquid interface is considered to be in local equilibrium. The interface is supposed to be in local equilibrium [27],

$$c_s = kc_l$$

(8.6)

where $C_s$ and $C_l$ are the concentrations (in molar fractions) of impurities $B$ at the solid and liquid side of the interface, respectively. The interface temperature satisfies the generalized Gibbs Thomson relation [26],

$$T = T_m + |m|c_l - \Gamma K - \frac{V_n}{\mu_k}$$

(8.7)

where $T_m$ is the melting temperature of pure $A$, $m$ is the slop of the liquidus line, $\Gamma = \frac{\gamma T_m}{L_f}$ is the Gibbs-Thomson constant, $\gamma$ is the surface tension, $L_f$ is the latent heat of fusion per unit volume, $K$ is the interface curvature, $V_n$ is its normal velocity, and $\mu_k$ is the linear kinetic coefficient.

Heat is supposed to diffuse much faster than impurities, so that the temperature field can be taken as fixed by external conditions, in spite of the rejection of latent heat during
solidification. Then, Eq. (8.7) yields a boundary condition for the solute concentration at the 
interface. A good approximation for alloy solidification is a one-sided model of solidification, 
which assumes zero diffusivity in the solid, in which the solute diffusivity in the solid may be 
several orders of magnitude lower than in the liquid.

For isothermal solidification at a fixed temperature $T_0 < T_m$, the concentration obeys the 
set of sharp-interface equations [6],

$$\partial_t c = D \nabla^2 c$$  \hspace{1cm} (8.8)

$$c_l(1 - k)V_n = -D(\partial_n c)|_l$$  \hspace{1cm} (8.9)

$$\frac{c}{c_l^0} = 1 - (1 - k)d_0 \chi - (1 - k)\beta V_n$$  \hspace{1cm} (8.10)

where $D$ is the solute diffusivity in the liquid, $(\partial_n c)|_l$ is the derivative of the concentration field 
normal to the interface, taken on the liquid side of the interface,

$$c_l^0 = \frac{T_m - T_0}{|m|}$$  \hspace{1cm} (8.11)

is the equilibrium concentration of the liquid at $T_0$, and $d_0 = \frac{r}{\Delta T_0}$ is the chemical capillary length,

where

$$\Delta T_0 = |m|(1 - k)c_l^0$$  \hspace{1cm} (8.12)

is the freezing range, and $\beta = \frac{1}{\mu k \Delta T_0}$.

For directional solidification, we use the frozen temperature approximation, in which the 
temperature field for solidification with speed $V_p$ in a temperature gradient of magnitude $G$ 
directed along the $z$ axis is taken as

$$T(z) = T_0 + G(z - V_p t)$$  \hspace{1cm} (8.13)
Now $T_0$ is given by inverting Eq. (8.12), and $c_i^0 = c_{\infty}/k$, where $c_{\infty} \equiv c(z = +\infty)$ is the global sample composition. Thus, $c_i^0$ is the solute concentration on the liquid side of a steady-state planar interface. Then
\[ c_i/c_i^0 = 1 - (1 - k)d_0 \kappa - (1 - k)\beta V_n - (1 - k)(z - V_p t)/l_T \quad (8.14) \]

Where
\[ l_T = \frac{|m(1-k)c_i^0|}{g} \quad (8.15) \]
is the thermal length. To relate the sharp-interface and phase-field models later, the dimensionless variables $U$ and $\theta$ were defined as
\[ \theta = \frac{T - T_m - mC_{\infty}}{L_f/C_p} \quad (8.16) \]
\[ U = \frac{c - c_i^0}{(1 - k)c_i^0} \quad (8.17) \]
where $C_{\infty}$ is the value of $c$ far from the interface that equals the initial concentration of the alloy. According to the above definitions, $U$ and $\theta$ are dimensionless measures of the concentration (supersaturation) and the undercooling, respectively. In terms of these variables, Eqs. (8.9), (8.10), and (8.17) in terms of the local supersaturation with respect to the point $(c_i^0, T_0)$,
\[ \frac{\partial U}{\partial t} = D\nabla^2 U \quad (8.18) \]
\[ [1 + (1 - k)U^+]V_n = -D\partial_n U^+ \quad (8.19) \]
\[ U^+ = -d_0 \kappa - \beta V_n - \frac{z - V_p t}{l_T} \quad (8.20) \]

Note that, for $k = 1$, we recover the constant miscibility gap model. Furthermore, if we reinterpret $U$ as a dimensionless temperature and drop the directional solidification term $(z - V_p t)/l_T$, we obtain a one-sided version of the pure substance model.
(b) Phase field models

In a phase-field model, \( \varnothing \) is introduced to distinguish between solid \((\varnothing = +1)\) and liquid \((\varnothing = -1)\). The two-phase system is usually described by a phenomenological free-energy functional [26],

\[
F(\varnothing, c, T) = \int_{dV} \left[ \frac{\varnothing^2}{2} |\nabla \varnothing|^2 + f(\varnothing, T_m) + f_{AB}(\varnothing, c, T) \right] dV
\]

Where \( V \) is the volume of the system, \( f(\varnothing, T_m) = H \left(-\frac{\varnothing^2}{2} + \frac{\varnothing^4}{4}\right) \) is the standard form of a double-well potential providing the stability of the two phases \((\varnothing = \mp 1)\) with a barrier height \( H \), \( f_{AB}(\varnothing, c, T) \) stands for the bulk free energies which changes the relative stability of substances \( A \) and \( B \) in the alloy as a function of the position in a \( T - c \) phase diagram, and the term in \( \sigma \) provides a penalty for phase gradients which ensures a finite interface thickness. \( H \) has dimensions of energy per unit volume, and \( \sigma \) of energy per unit length.

In a variational formulation, the equations of motion for all fields can be derived from that functional,

\[
\frac{\partial \varnothing}{\partial t} = -K_\varnothing \frac{\delta F}{\delta \varnothing}
\]

\[
\frac{\partial c}{\partial t} = \nabla \cdot \left( M(\varnothing, c) \nabla \frac{\delta F}{\delta c} \right)
\]

where \( K_\varnothing(T) \) is a kinetic constant that can generally be temperature-dependent. The second equation is a statement of mass conservation since it can be rewritten as

\[
\frac{\partial c}{\partial t} + \nabla \cdot \vec{j}_c = 0
\]

where

\[
\vec{j}_c = -M\nabla \mu
\]

is the solute current density,
\[
\mu \equiv \frac{\delta F}{\delta t} \quad (8.26)
\]
is the chemical potential, and \( M(\emptyset, c) \) is the mobility of solute atoms or molecules, which we choose to be
\[
M(\emptyset, c) = \frac{v_0}{RT_m} D \tilde{q}(\emptyset)c \quad (8.27)
\]
in order to later obtain Fick’s law of diffusion in the liquid. Here, \( v_0 \) is the molar volume of \( A \), \( R \) is the gas constant, and \( \tilde{q}(\emptyset) \) is a dimensionless function that interpolates between 0 in the solid and 1 in the liquid, and hence dictates how the solute diffusivity varies through the diffuse interface.

An important step is the construction of the function \( f_{AB} \) that interpolates between the free-energy densities of the bulk phases (solid and liquid). While these bulk free energies should reduce to the curves that can be obtained from thermodynamic databases, the dependence of \( f_{AB} \) on \( \emptyset \) influences only the interfacial region, and this freedom can be used to construct a particularly simple phase field model. For a dilute alloy, the free energies of solid and liquid \( f_v(c, T) \), where the subscript \( v \) refers to either the solid or the liquid, can be written as the sum of the free energy of pure \( A \), \( f_v^A(T) \), and the contributions due to solute addition which includes the dilute form of the mixing entropy, \( \frac{RT}{v_0} (c \ln c - c) \), and the change of the internal energy density \( \varepsilon_v c \).

\[
f_v(c, T) = f_v^A(T) + \frac{RT}{v_0} (c \ln c - c) + \varepsilon_v c, \quad v = l, s \quad (8.28)
\]

Expand this expression to first order in \( T - T_m \) to recover the straight liquidus and solidus lines,
\[
f_v(c, T) = f^A(T_m) - s_v(T - T_m) + \frac{RT_m}{v_0} (c \ln c - c) + \varepsilon_v c \quad (8.29)
\]
where \( s_v = \frac{\partial f_A}{\partial T} \) are the entropy densities of the solid and the liquid at \( T_m \), and we have used that both phases have equal free energies for pure \( A \) at \( T_m \), \( f_s^A(T) = f_l^A(T_m) = f^A(T_m) \). By using \( T_m \) instead of \( T \) in the mixing entropy, we have neglected terms of order \((T - T_m)c\) which are second-order for dilute alloys. The phase diagram is determined by the standard common tangent construction, which is equivalent to requiring that the chemical potential and the grand potential \( \omega \) be equal in the solid and liquid. The corresponding equilibrium concentrations \( c_s(T) \) and \( c_l(T) \) are the solutions of

\[
\frac{\partial f_s(c, T)}{\partial c}_{|c=c_s} = \frac{\partial f_l(c, T)}{\partial c}_{|c=c_l} = \mu_E(T) \tag{8.30}
\]
\[
f_s(c_s, T) - \mu_E c_s = f_l(c_l, T) - \mu_E c_l = \omega_E(T) \tag{8.31}
\]

The first equality yields the partition relation \( c_s = k c_l \) with a partition coefficient \( k = e^{\frac{v_0 \Delta s}{RT_m}} \), where we have defined \( \Delta s = s_l - s_s \). Combining this result with Eq. (8.26) yields

\[
c_l = \frac{L_f v_0}{T^2 R (1 - k)} (T_m - T) \tag{8.32}
\]

where the latent heat per unit volume is \( L_f = T_m (s_l - s_s) \). From Eq. (8.27), we identify the liquidus slope to be

\[
m = \frac{T^2 R (1 - k)}{L_f v_0} \tag{8.33}
\]

the van 't Hoff relation for dilute binary alloys. The two bulk free energies are interpolated with the help of a single function of the phase field \( \phi \). Here, it is advantageous to use two different interpolation functions for the entropy and the internal energy terms,

\[
f_{AB}(\phi, c, T) = f^A(T_m) - (T - T_m)s(\phi) + \frac{RT_m}{v_0} (c \ln c - c) + \phi(\phi) c \tag{8.34}
\]

with
\[ \varepsilon(\emptyset) = \bar{\varepsilon} + \bar{g}(\emptyset) \frac{\Delta \varepsilon}{2} \]  
(8.35)

\[ s(\emptyset) = \frac{s_s - s_l}{2} - \bar{g}(\emptyset) \frac{L_f}{2T_m} \]  
(8.36)

where \( \bar{\varepsilon} = \frac{\varepsilon_s + \varepsilon_l}{2} \) and \( L_f = T_m(s_l - s_s) \). \( \bar{g}(\mp 1) = \bar{g}(\mp 1) = \mp 1 \), and we further require \( \bar{g}'(\mp 1) = \mp 1 \) to remain bulk equilibrium solutions for any value of \( c \) and \( T \). This completes the model specification, except for the interpolation functions \( \bar{g}(\emptyset) \) and \( \bar{g}(\emptyset) \). In order to choose them appropriately, it is important to consider the equilibrium properties of the model, which follow from the conditions

\[ \frac{\delta F}{\delta c} = \mu_E \]  
(8.37)

\[ \frac{\delta F}{\delta \emptyset} = 0 \]  
(8.38)

where \( \mu_E \) is the spatially uniform equilibrium value of the chemical potential. These two conditions uniquely determine the spatially varying stationary profiles of \( c \) and \( \emptyset \) in the diffuse interface region. Since the phase field interpolates between the two bulk free energies, the limiting values of the concentrations and the equilibrium chemical potential are the ones determined by the common tangent construction above. From Eq. (8.37),

\[ \frac{RT_M}{v_0} \ln c_0 + \bar{\varepsilon} + \bar{g}(\emptyset_0) \frac{\Delta \varepsilon}{2} = \mu_E \]  
(8.39)

from which we obtain the expression for the equilibrium concentration profile using the solution of Eq. (8.30) and Eq. (8.31),

\[ c_0(x) = c_l e^{\left( \frac{1}{2} k \right) \left( \ln \frac{k}{\varepsilon_0(x)} \right)} = c_l k^{\frac{1+g(\emptyset_0(x))}{2}} \]  
(8.40)

From the equilibrium condition for \( \emptyset \), Eq. (8.38), we obtain
\[
\sigma \frac{d^2 \phi_0}{dx^2} + H(\phi_0 - \phi_0^2) = \frac{\tilde{g}'(\phi_0) T - T_m}{2 T_m} L_f + \frac{\tilde{g}'(\phi_0) \Delta \varepsilon_0}{2} \quad \text{(8.41)}
\]

Based on Eq. (8.32) and Eq. (8.33), Eq. (8.39) can be rewritten as

\[
\sigma \frac{d^2 \phi_0}{dx^2} + H(\phi_0 - \phi_0^2) = -\frac{RT_m(T - T_m)}{2v_0 m} \left[ (1 - k)\tilde{g}'(\phi_0) + \ln k \frac{c_0(x)}{c_l} \tilde{g}'(\phi_0) \right] \quad \text{(8.42)}
\]

For a generic choice of the functions \( \tilde{g} \) and \( \overline{g} \), and in particular for the “standard” choice \( \tilde{g} = \overline{g} \), no analytic solution for \( \phi \) is known. Furthermore, the equilibrium solution and its properties, in particular, its surface tension, depend on the various coefficients that appear on the right-hand side. This can be avoided if the right-hand side vanishes \[ \partial \phi \mathcal{F}(\phi, c, T) = 0 \]. With the help of Eq. (8.42), we obtain the condition on the interpolation functions,

\[
(1 - k) \frac{\tilde{g}'(\phi)}{2} + \ln k \frac{\overline{g}'(\phi)}{2} e^{\frac{\ln k (1 + \overline{g}(\phi))}{2}} = 0 \quad \text{(8.43)}
\]

It can be used to eliminate one of them in terms of the other. Taking into account the requirement \( \tilde{g}(\mp 1) = \overline{g}(\mp 1) = \mp 1 \), we find

\[
\tilde{g}(\phi) = \frac{1 + k - 2e^{\frac{\ln k (1 + \overline{g}(\phi))}{2}}}{1 - k} = \frac{1 + k - 2k[1 + \overline{g}(\phi)]}{1 - k} \quad \text{(8.44)}
\]

\[
\overline{g}(\phi) = \frac{2}{\ln k} \ln \left( \frac{1 + k - (1 - k)\tilde{g}(\phi)}{2} \right) - 1 \quad \text{(8.45)}
\]

Using the latter relation, the equilibrium concentration profile can also be rewritten as

\[
c_0(\phi) = c_l \frac{1 + k - (1 - k)\tilde{g}(\phi)}{2} = c_s + c_l + \tilde{g}(\phi) \frac{c_s - c_l}{2} \quad \text{(8.46)}
\]

The physical meaning of the two interpolation functions is hence completely transparent:

\( \overline{g} \) interpolates the internal energy [Eq. (8.35)] and consequently the chemical potentials [Eqs. (8.39) and (8.40)], whereas \( \tilde{g} \) interpolates the entropy density [Eq. (8.37)] and, because of Eq. (8.43), the concentration [Eq. (8.46)]. If Eq. (8.43) is satisfied, the right-hand side of Eq. (8.42) vanishes and the solution for the equilibrium profile of \( \phi \) is the usual hyperbolic tangent [28].
$\phi_0(x) = -\tanh \frac{x}{\sqrt{2W}} \quad (8.47)$

where $W = \frac{\sigma}{H}$ measures the width of the diffuse interface. Furthermore, the surface tension is defined as the excess of the grand potential $\omega = f - \mu c$, integrated through the interface, that is, $\gamma = \int [\omega(x) - \omega_E] dx$. Because condition (8.43) is equivalent to requiring $\partial_\phi f_{AB}(\emptyset, c, T) = 0$, under this condition $f_{AB}(\emptyset, c, T)$ is independent of $x$ and equals its bulk phase values $f_\nu(c_\nu, T)$.

Since the latter enter the expression for the equilibrium grand potential $\omega_E$ as given by Eq. (8.30), the contribution of $f_{AB}$ to $\omega(x) - \omega_E$ is zero.

Thus, only the two other interface terms in Eq. (8.23) contribute. Taking into account that both contribute the same amount (equipartition relation), we have $\omega(x) - \omega_E = H \frac{(1 - \phi_0(x))^2}{2}$, and hence the surface tension is

$\gamma = IWH \quad (8.43)$

with $I = \frac{2\sqrt{2}}{3}$. As in the sharp-interface model, $\gamma$ is independent of solute concentration and temperature. This property is only achieved if condition (8.43) is satisfied. Otherwise, Eq. (8.35) is replaced by a more complicated expression which contains the impurity concentration, and which needs in general to be calculated numerically. A drawback of this more complicated expression is that the dependence of $\gamma$ on concentration along the interface cannot be chosen independently of the value of $W$. This feature leads to an unphysically large variation of $\gamma$ with concentration for computationally tractable mesoscopic values of $W$. Equation (8.48) yields a concentration independent expression for $\gamma$ that is free of this limitation. Moreover, the fact that the equilibrium profile remains a hyperbolic tangent for arbitrary values of the concentration makes the relationship between phase-field and sharp interface parameters obtained from the
thin-interface analysis independent of the value of the local concentration. This, in turn, avoids spurious kinetic corrections that are present otherwise.

Once we have found a convenient relation between $\bar{g}(\emptyset)$ and $\bar{g}(\emptyset)$, we come back to the complete dynamical model. The relations we have found in equilibrium can now be used to obtain two particularly simple forms of the phase-field equation out of equilibrium. For the first, we remark that Eq. (8.43) implies that $\bar{g}'(\emptyset_0)c_i(1 - k) = g'(\emptyset_0)\ln kc_0$, and therefore the function $\bar{g}$ can be eliminated in favour of the phase dependent equilibrium concentration $c_0(\emptyset, T)$ and the function $\bar{g}$. Dividing Eq. (8.25) by $H$, we obtain

$$\tau \frac{\partial \emptyset}{\partial t} = W^2\nabla^2 \emptyset + \emptyset - \emptyset^3 + \frac{RTm(T - T_m)}{2v_0Hm} \bar{g}'(\emptyset) \ln k \left[ \frac{c - c_0(\emptyset, T)}{c_i(T)} \right]$$  \hspace{1cm} \text{(8.49)}$$

with $\tau = \frac{1}{k_0(T)\mu}$. The driving force is the local supersaturation.

The second possibility is to rewrite the phase-field equation in terms of the dimensionless variable,

$$u = \frac{v_0}{RT_M}(\mu - \mu_E) = \ln \frac{c}{c_i^0} - \frac{\ln k}{2} [1 + \bar{g}(\emptyset)] = \ln \left( \frac{2c}{c_i^0 [1 + k - (1 - k)\bar{g}(\emptyset)]} \right)$$  \hspace{1cm} \text{(8.50)}$$

which measures the departure of the chemical potential from its value $\mu_E(T_0)$ for a flat interface at the equilibrium liquidus temperature $T_0$ [and liquid concentration $c_i^0 = c_i(T_0)$]. Then, it is preferable to eliminate $\bar{g}(\emptyset)$ in favour of $\bar{g}(\emptyset)$.

$$\tau \frac{\partial \emptyset}{\partial t} = W^2\nabla^2 \emptyset + \emptyset - \emptyset^3 + \frac{\lambda}{1 - k} \bar{g}'(\emptyset) \left[ e^u - 1 - \frac{T - T_m}{m c_i^0} \right]$$  \hspace{1cm} \text{(8.51)}$$

where we have defined the constant

$$\lambda = \frac{RT_m(1 - k)^2 c_i^0}{2v_0H} = \frac{L_f \Delta T_0}{2HT_m}$$  \hspace{1cm} \text{(8.52)}$$
where $\Delta T_0 = |m|(1 - k)c_0^l$ is called the freezing range. Note that the parameter $H$ can be expressed in terms of the surface tension, $H = \frac{\gamma}{lW}$. Then, we have

$$\tilde{\lambda} = \frac{I \Delta T_0 W}{2\Gamma}$$

(8.53)

where $\Gamma$ is the Gibbs-Thomson constant. Therefore, up to numerical constants, $\tilde{\lambda}$ is the dimensionless ratio of interface thickness times freezing range and the Gibbs-Thomson constant.

It is immediately clear that a variation of the interface thickness corresponds to a change in $\tilde{\lambda}$.

Though $h(\phi)$ and $\bar{q}(\phi)$ are completely free functions which purely need to interpolate from +1 to -1 and from 0 to 1, respectively, this does not yet provide enough freedom to cancel the three spurious effects mentioned in the Introduction. To achieve this goal, we add an extra term in the model equations to specifically cancel one of them. The extra interpolation function contained in this new term provides the necessary third degree of freedom to make all three effects vanish. We specifically target the solute trapping effect. This occurs when solute atoms or molecules cannot escape the advancing solidification front fast enough to maintain local equilibrium at the interface. The characteristic interface velocities where solute trapping becomes important can be estimated by comparing the time of advance by one interface thickness, $W/V$, and the time it takes for the solute to diffuse through the interface, $W^2/D$. The result is $V \sim D/W$, and hence the critical speed depends on the interface thickness. Since we ultimately want to simulate solidification with diffuse interfaces that are orders of magnitude larger than the real solid-liquid interfaces, solute trapping sets in for much lower speeds than in reality.

To eliminate this interface-thickness effect, we introduce a supplementary current in the equation for the solute concentration, the antistripping current. Its purpose is to transport solute atoms from the solid to the liquid. Therefore, it has to fulfill a number of properties. First, it must be proportional to the speed of the interface, and hence to $\partial_t \phi$. Next, it must be directed from the
solid to the liquid, that is, along with the unit normal vector \( \vec{n} \), which in terms of the phase field can be expressed as \( \vec{n} = -\frac{\nabla \phi}{|\nabla \phi|} \). Furthermore, it must be proportional to the interface thickness \( W \), and to the local concentration difference between solid and liquid. In contrast, we do not know a priori the profile of the current function through the interface. The time derivative of the phase field \( \partial_t \phi \) is sensibly different from zero only in the interface regions and induces a certain anti-trapping current profile. Additional freedom may be gained by allowing for a shape function \( a(\phi) \) that must be appropriately chosen in order to obtain the correct thin-interface limit. As we shall see, choosing \( a(\phi) \) constant suffices to eliminate all spurious effects for the simplest choice of the functions \( h(\phi) \) and \( \tilde{q}(\phi) \). In summary, we write

\[
\vec{J}_{at} = a(\phi)W(1-k)c_i^0e^u \frac{\partial \phi}{\partial t} \vec{n} = -a(\phi)W(1-k)c_i^0e^u \frac{\partial \phi}{\partial t} \frac{\nabla \phi}{|\nabla \phi|}
\]

and the equation for the concentration becomes

\[
\frac{\partial c}{\partial t} = \nabla \cdot (Dq(\phi)c\nabla u - \vec{J}_{at}) = \nabla \cdot \left( Dq(\phi)c\nabla u + a(\phi)W(1-k)c_i^0e^u \frac{\partial \phi}{\partial t} \frac{\nabla \phi}{|\nabla \phi|} \right)
\]

Note that the latter no longer derives from a functional \( F \), even if such a functional is allowed to be different from that giving rise to the equation of motion for \( \phi \). To this end, we introduce the diffuse-interface extension \( U(\phi) \) of the dimensionless supersaturation \( U \) in Eq. (8.19), now defined in the whole system,

\[
U = \frac{e^u - 1}{1-k} = \frac{1}{1-k} \left( \frac{c}{c_i^0} \frac{1}{1-\phi} - \frac{k(1+\phi)}{2} - 1 \right)
\]

Furthermore, we fix now the interpolation function \( \tilde{g} \) to be

\[
\tilde{g}(\phi) = \frac{15}{8} \left( \phi - \frac{2}{3} \phi^3 + \frac{1}{5} \phi^5 \right)
\]
define new interpolation functions

\[ q(\phi) = \tilde{q}(\phi) \frac{1 + k - (1 - k)h(\phi)}{2} \quad (8.58) \]

\[ g(\phi) = \frac{8}{15} \tilde{g}(\phi) = \phi - \frac{2}{3} \phi^3 + \frac{1}{5} \phi^5 \quad (8.59) \]

and transform the equation for \( c \) into one for \( U \). Taking into account that \( T(z) = T_0 + G(z - V_p t) \) and the temperature dependent relaxation time \( \tau = \tau_0 [1 - (1 - k)(z - V_p t)/l_T] \).

The final set of equations is

\[ \tau_0 \left[ 1 - (1 - k) \frac{Z - V_p t}{l_T} \right] \frac{\partial \phi}{\partial t} = W^2 \nabla^2 \phi + \phi - \phi^3 - \lambda g'(\phi) \left( U + \frac{Z - V_p t}{l_T} \right) \quad (8.60) \]

\[ \left( \frac{1 + k}{2} - \frac{1 - k}{2} h(\phi) \right) \frac{\partial U}{\partial t} = \nabla \cdot \left( Dq(\phi) \nabla U + a(\phi) W[1 + (1 - k)U] \times \frac{\partial \phi}{\partial t} \frac{\nabla \phi}{|\nabla \phi|} \right) + \frac{[1 + (1 - k)U] \partial h(\phi)}{2} \frac{\partial t}{\partial t} \quad (8.61) \]

where \( \lambda = \frac{15}{8} \tilde{\lambda} \), \( h(\phi) = \phi, q(\phi) = \frac{1 - \phi}{2}, \tau_0 = \frac{a_s W^2}{D} \) and \( a(\phi) = \frac{1}{2 \sqrt{2}} \). Crystalline anisotropy is included by letting \( W = W_0 a_s(n) \), and

\[ a_s(n) = a_s_0 \left[ 1 + \epsilon' \left[ (n_x)^4 + (n_y)^4 \right] \right] = a_s_0 \left[ 1 + \epsilon' [(\sin \varphi)^4 + (\cos \varphi)^4] \right] \quad (8.62) \]

is a function that describes the anisotropy, where \( \varphi = \tan^{-1} \frac{\theta_y}{\theta_x} \) is the angle between the direction normal to the interface and the \( x \) (horizontal) axis and \( \epsilon' \) is a dimensionless parameter that characterizes the anisotropy strength [29]. The anisotropy strength is characterized by the parameter \( \epsilon_4 \) that can be measured experimentally by examining the deviation of an equilibrium shape from a circle. For small \( \epsilon_4 \), this deviation is given by \( R(\varphi) = R_0 (1 + \epsilon_4 \cos 4\varphi_0) \), where
\( R \) is the radial polar coordinate measured from a fixed origin. In this study, \( \epsilon_4 \) was used as the measure of the anisotropy and it is easy to check that \( a_{s0} \) and \( \epsilon' \) are related to \( \epsilon_4 \) by the expressions

\[
a_{s0} = 1 - 3 \epsilon_4 \quad \text{and} \quad \epsilon' = \frac{4 \epsilon_4}{1 - 3 \epsilon_4}.
\]

Thus,

\[
a_s(\vec{n}) = (1 - 3 \epsilon_4) \left[ 1 + \frac{4 \epsilon_4}{1 - 3 \epsilon_4} \frac{(\partial_x \phi)^4 + (\partial_y \phi)^4}{((\partial_x \phi)^2 + (\partial_y \phi)^2)^2} \right]
\]

(8.63)

The scaled phase-field equations then only depend on \( \epsilon \) through the dimensionless parameters. Then, writing out explicitly all the interpolation functions, and taking into account the contributions of the anisotropic \( W(\vec{n}) \) in the functional derivative, the equations read

\[
\left[ 1 - (1 - k) \frac{z - \bar{V}_p t}{\bar{l}_T} \right] a_s(\vec{n})^2 \frac{\partial \phi}{\partial t}
\]

\[
= \vec{V} \cdot (a_s(\vec{n})^2 \nabla \phi) - \frac{\partial}{\partial x} \left[ a_s(\vec{n}) a_s(\vec{n})' \frac{\partial \phi}{\partial x} \right] + \frac{\partial}{\partial y} \left[ a_s(\vec{n}) a_s(\vec{n})' \frac{\partial \phi}{\partial y} \right] + \phi - \phi^3
\]

\[
- \lambda (1 - \phi^2)^2 \left( U + \frac{z - \bar{V}_p t}{\bar{l}_T} \right)
\]

(8.64)

\[
\left( \frac{1 + k}{2} - \frac{1 - k}{2} \phi \right) \frac{\partial U}{\partial t}
\]

\[
= \vec{V} \cdot \left( \bar{D} \frac{1 - \phi}{2} \nabla U + \frac{1}{2\sqrt{2}} \left[ 1 + (1 - k) U \right] \frac{\partial \phi}{\partial t} \frac{\nabla \phi}{|\nabla \phi|} \right)
\]

\[
+ \left[ 1 + (1 - k) U \right] \frac{\partial \phi}{\partial t}
\]

(8.65)

Where \( \bar{D} = \frac{d_{\tau_0}}{W^2} = a_1 a_2 \epsilon, \bar{V}_p = \frac{V_{p\tau_0}}{W^2} = V_p a_1 a_2 \epsilon^2 \) and \( \bar{l}_T = \frac{l_T}{W} = \frac{1}{\epsilon v} \).

For non-isothermal solidification, considering non-isothermal temperature evolution by allowing the constant temperature \( T(\vec{x}, t) \), where \( t \) is time and \( \vec{x} \) is a position vector. The temperature evolves such that the flux of heat into a volume element lead to a corresponding change in entropy. This is expressed in the form of an entropy production equation [30],

\[
\]
\[ T \frac{\partial S}{\partial t} + \nabla \cdot \vec{J}_e = 0 \quad (8.66) \]

where \( \vec{J}_e \) is the entropy flux. If mass transport and convection are neglected \( \vec{J}_e \approx \vec{J}_0 \). Moreover, with the substitution \( T \, dS = dQ = dH_p \), it becomes

\[ \frac{\partial H_p}{\partial t} = \nabla \cdot (\kappa \nabla T) \quad (8.67) \]

where \( H_p \) denotes the enthalpy at constant pressure. The enthalpy can be written in terms of the order parameter as \( H_p = \rho c_p T - \rho L_f h(\emptyset) \), where \( c_p \) is the specific heat at constant pressure.

The function \( h(\emptyset) \) assumed to be some smooth function with limits \( h(-1) = 0 \) and \( h(1) = 1 \). It has been added to describe the generation of excess heat production if solid (\( \emptyset = 1 \)) phase appears. In the liquid, where \( \emptyset = -1 \), the enthalpy is due only to temperature changes. In the solid, where \( \emptyset = 1 \), the enthalpy is reduced due to latent heat. The variation of \( h(\emptyset) \) for \( 0 - 1 < \emptyset < 1 \) corresponds to the solid liquid interface. In this way, Eq. (8.66) is

\[ \rho c_p \frac{\partial T}{\partial t} - \rho L_f \frac{dh(\emptyset)}{d\emptyset} \frac{\partial \emptyset}{\partial t} = -\nabla \cdot \vec{J}_0 \quad (8.68) \]

If convection effects are ignored the heat flux is \( \vec{J}_0 = -\kappa \nabla T \), where \( \kappa \) is the thermal conductivity of the material and has the form \( \kappa = \frac{RM}{T^2} \). This leads to Fourier’s law of heat conduction, modified for changes of phase through the order parameter \( \emptyset \). The conductivity can make a function of the phase by expressing it as \( \kappa = \kappa L_f q(\emptyset) \), where \( q(\emptyset) \) is an unknown function that interpolates the conductivity across the solid-liquid interface. So the partial differential equation for the evolution of the temperature \( (T) \) is,

\[ \frac{\partial T}{\partial t} = \nabla \cdot \alpha \nabla T + \frac{L_f h'(\emptyset)}{c_p} \frac{\partial \emptyset}{\partial t} \quad (8.69) \]
where $\alpha \equiv \frac{\kappa}{\rho c_p}$, $h'(\emptyset) = \frac{1}{2}$ is the thermal diffusion coefficient and $h'(\emptyset)$ denotes the derivative of $h(\emptyset)$ with respect to $\emptyset$. Replacing the temperature $T$ using the dimensionless variables $\theta = \frac{T-T_m-mc_\infty}{L_f/c_p}$, we obtained [31]

$$\frac{\partial \theta}{\partial t} = \alpha \nabla^2 \theta + \frac{1}{2} \frac{\partial \emptyset}{\partial t} \quad (8.70)$$

In this model, the Inconel 718 alloy is treated as binary and the solute is the combination of Ni and Nb. The thermo-physical parameters used in the phase field modeling are considered as constant, as listed in Table 8.2. It is assumed that the initial temperature and concentration of liquid is uniform in the calculated region.

<table>
<thead>
<tr>
<th>Physical properties</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Partition coefficient, $k$</td>
<td>0.48</td>
</tr>
<tr>
<td>Melting temperature, $T_m$ (K)</td>
<td>1534</td>
</tr>
<tr>
<td>Liquidus Temperature, $T_L$ (K)</td>
<td>1633</td>
</tr>
<tr>
<td>Initial solute concentration, $c_0$ (wt. %)</td>
<td>5</td>
</tr>
<tr>
<td>Initial solute concentration by mole fraction, $(c_0, mol%)$</td>
<td>3.22</td>
</tr>
<tr>
<td>Dimensionless temperature gradient, $\Delta$</td>
<td>0.5 ~ 0.9</td>
</tr>
<tr>
<td>Dimensionless Concentration value, $\Omega$</td>
<td>0.55</td>
</tr>
</tbody>
</table>
8. 3 Results and discussion
8.3.1 Temperature gradient and cooling rate

The solidification mode can vary from one beam scanning path to another in selective laser melting process, it can also vary within a single scanning path from the fusion line to the centerline based on the growth rate $R_m$ and the temperature gradient $G$. To simulate the microstructure evolution, thermal gradient is calculated by tracking the simulated temperature
profile from the thermal simulation of SLM built Inconel 718 alloy. The resulted melt pool at a constant situation is shown in Figure 8.3. The temperature gradient in the molten pool is defined as,

$$G = \frac{\partial T}{\partial Z} = -\frac{Q(Z)}{\kappa}$$  \hspace{1cm} (8.71)

where $Q(Z)$ is the heat flux ($W \cdot m^{-2}$) in the liquid pool, $\kappa$ is the thermal conductivity ($W \cdot m^{-1} \cdot K^{-1}$). Individual components of the temperature gradient, $\vec{G}_x$, $\vec{G}_y$ and $\vec{G}_z$, are obtained by differentiating the temperature profile at the S/L interface of the molten pool with respect to the $x$, $y$ and $z$ coordinates, respectively. The temperature gradient normal to $S/L$ interface, $\vec{G}_n$, is then calculated using the simple expression below:

$$|\vec{G}_n| = \sqrt{\vec{G}_x^2 + \vec{G}_y^2 + \vec{G}_z^2}$$  \hspace{1cm} (8.72)

Figure 8.3 2D view of thermal longitudinal profile of Inconel 718 in SLM process
The solidification rate, $\vec{R}_s$, normal to the $S/L$ interface, can be calculated based on the following geometrical relationship [34],

$$\left| \vec{R}_s \right| = v \cdot \cos \theta = v \cdot \frac{\vec{G}_x}{\vec{G}_n}$$  \hspace{1cm} (8.73)

where $v$ is the travel speed of laser beam which moves along the $X$ direction, and $\theta$ is the angle between the normal direction of local $S/L$ interface and the laser travel direction (i.e., the $X$ direction). The solidification morphology can be evaluated using the ratio of $\left| \vec{G}_n \right|$ to $\left| \vec{R}_s \right|$. A decrease in the $\left| \vec{G}_n \right| \rightarrow \left| \vec{R}_s \right|$ ratio corresponds to a transition of solidification morphology from planar to cellular, columnar dendrite and eventually equiaxed grains. To evaluate the solidification morphology in the molten pool during SLM, the minimums and maximums of solidification parameters are located at the molten pool side edge (“edge”) and its trailing end (“tail”), as shown in Figure 8.4.

![Figure 8.4](image)

Figure 8.4 Schematics showing the edge and tail monitoring locations selected to analyze the solidification conditions: (a) 3D view, and (2) 2D cross-section view [35]

The distance between the maximum pool temperature ($T_{max}$) and the pool boundary ($T_L$) is greater at the centerline than at the fusion line because the melt pool is elongated.
Consequently, the temperature gradient normal to the pool boundary at the centerline, $G_{CL}$, is less than that at the fusion line, $G_{FL}$. Since $G_{CL} < G_{FL}$ and $R_{CL} \gg R_{FL}$,

$$\left( \frac{G}{|R_s|} \right)_{CL} \ll \left( \frac{G}{|R_s|} \right)_{FL}$$ (8.74)

In general, the temperature gradient $G$ is high at the melt-pool side edge where the solidification rate $|R_s|$ is slow. On the other hand, at the molten pool-trailing end, the solidification rate is fast whereas the temperature gradient is small. In other words, those two locations correspond to the minimums and maximums of solidification parameters. In this study reality, the melt pool is a narrow long ellipsoid, as shown in Figure 8.4. In solidification process, the area close to the rear tail will have a higher solidification speed with its direction towards the opposite temperature gradient direction. Considering of the remelting/rescanning processes in AM, some top solidification area will be re-melted. Therefore, the effective region is the area enclosed by the white rectangle. The enclosed area will have a higher solidification speed with its direction towards the opposite temperature gradient direction.

The solidification rate $|R_s^+|$, normal to the $S/L$ interface, and $\alpha$ is the angle between the normal direction of local $S/L$ interface and the laser travel direction, as illustrated in Figure 8.5. This angle $\alpha$ would be affected by the heat flux in the solidification due to the scanning strategy. It also affected by the build height, which will also have a critical influence on the heat flux. Based on the analysis of the solidification process and the re-melting characterizations of AM process, the majority of the grains would have a certain growth direction. The angle, $\alpha$, between the beam scanning direction and the grain growth direction was measured on the $Y$-plane of the samples, as shown in Figure 8.6. The average growth direction of the dendrites is around 45°.
Thus, it is reasonable to use the thermal gradient of the point on the fusion line having an angle of 45° with the beam scanning path.

Phase field simulation of Inconel 718 alloy built fabricated in SLM process is carried out by taking the average thermal gradient, which is used to model the solidification microstructure. For this model, the temperature gradient will be rescaled into dimensionless values. The dimensionless temperature gradient \( U \) is

\[
U = \frac{\Delta T}{L_f C_p} \quad (8.75)
\]

where \( \Delta T \) is the temperature difference between that point A and B, as shown in Figure 8.5. Based on the thermal profile, the equivalent temperature difference is defined as

\[
\Delta T = T_p - T_L = G \cdot r \quad (8.76)
\]

where \( T_{\text{max}} \) is the maximum temperature in the liquid pool, \( T_L \) is the melting temperature and \( r \) is the distance between the location of the point (A) on the melt pool and the point of the intersection of the melt pool surface and the normal direction of the melt pool, as illustrated in Figure 8.5.

![Figure 8.5 Schematic of the melt pool in selective laser melting process](image)

8.3.2 Typical microstructural evolution in SLM process of Inconel 718
According to the experimental results of build height effects on the microstructural evolution, the characteristic width of the columnar structure has the lowest value at the bottom of the part and it would increase to a stable value determined by the manufacturing parameters, and then slight increases at the end of the manufacturing process [11]. This is attributed to the varying of the cooling rate along the build height results from the thermal history of the manufacturing process. In this section, the thermal profiles of the melt pool at different height are extracted from the thermal analysis results obtained by CFD through thermal simulation that implemented with a model at the same manufacturing parameters adopted in the experiments. For the simulation, three layers as Layer 02, Layer 44, and Layer 79 at the first pass have been taken out for further analysis, in which Layer 02 is close to the bottom of the modeled part, Layer 44 is at about the middle of the part, and Layer 79 is close to the end of the part. The thermal profile of melt pool at the first pass layer 44 is shown in Figure 8.3, in which the laser beam traveling from the left to the right at a speed of 600 mm/s.

Take the case of layer 44 as an example, the thermal gradient values along the fusion boundary of the melt pool are shown in Figure 8.6, which was obtained from thermal analysis of SLM process that conducted by CFD Inc.. The two critical variables in determining the solidification microstructure of alloys are temperature gradient $G$ and solidification growth rate $R_m$ [36]. The ratio $G/R_m$ determines the mode of solidification while the product $GR_m$ governs the size of the solidification structure as $GR_m (°C/mm \times mm/s)$ is the cooling rate ($°C/s$). It is known that the higher the cooling rate, the finer the cell dendrites become. The calculated values of cooling rate are shown in Table 8.6.

It can be seen that among the sample points, the region around point D has the highest cooling rate, about $1.04 \times 10^6 \ K/s$, with a relatively high growth rate $0.237 \ m/s$. The angle
between the thermal gradient direction and the opposite direction of the beam scanning direction is around 66.8°, which agrees well with the experimental results as can be seen in Figure 8.7. It is in the range of our predicted values in the former section. Based on the thermal results, the dimensionless values of the thermal gradient at the three points of C, D, and E are calculated using the Eq. (8.75), as shown in Table 8.3. The cooling rates calculated here are in the range of the statistic values estimated from the experiments based on the empirical equations. According to the solidification map [37] for Inconel 718, the solidification structure has a columnar cellular morphology, as illustrated in Figure 8.8. At point D, the dimensionless thermal gradient is 0.498, which is corresponding to the equivalent thermal gradient of $4.38 \times 10^6$ °C/mm.

Figure 8.6 Inconel 718: (a) Longitudinal section, and (b) Thermal gradient at the fusion boundary, of the melt pool at the first pass layer 44 in SLM process

Table 8.3 Temperature gradient, growth rate and cooling rate at the fusion boundary of the melt pool during SLM process of Inconel 718 at the first pass, layer 44

<table>
<thead>
<tr>
<th>Point</th>
<th>$G$, °C/m</th>
<th>$\alpha$, °</th>
<th>$R_m$, m/s</th>
<th>$\dot{\beta}$, °C/s</th>
<th>$G/R_m$, °C · s/m²</th>
<th>$U$</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>$1.46 \times 10^6$</td>
<td>43.6</td>
<td>0.435</td>
<td>$6.36 \times 10^5$</td>
<td>$3.37 \times 10^6$</td>
<td>-</td>
</tr>
<tr>
<td>B</td>
<td>$2.49 \times 10^6$</td>
<td>61.6</td>
<td>0.285</td>
<td>$7.11 \times 10^5$</td>
<td>$8.72 \times 10^6$</td>
<td>-</td>
</tr>
<tr>
<td>C</td>
<td>$3.11 \times 10^6$</td>
<td>62.9</td>
<td>0.273</td>
<td>$8.51 \times 10^5$</td>
<td>$1.14 \times 10^7$</td>
<td>0.295</td>
</tr>
</tbody>
</table>

209
Figure 8.7 Example of measuring the angle between the dendrites growing direction and the beam scanning direction.
The typical phase profiles of Inconel 718 alloy during solidification in SLM are shown in Figure 8.9. The computational domain is initialized with a small solid nucleus, which was located at the bottom of the domain with a diameter of 1 μm. It can be seen that the nucleus began to grow and formed the dendrites perpendicular to the solid–liquid interface at time \( t = 0.1 \) ms. With the increase of solidification time, the dendritic arms grew and finally developed into the columnar structure.
Figure 8.9 Phase field profile of Inconel 718 at the average dimensionless thermal gradient of 0.498 (Temperature gradient = $1.04 \times 10^6 \, ^\circ\text{C/mm}$)

Figure 8.10 shows the corresponding concentration profile. From the simulation results, it is found that the liquid concentration decreases with increase in solidification time as the columnar dendrites growing within the domain. The liquid concentration is higher near the dendritic tip, where it decreases more steeply and reaches to $c_\infty$. This is because of the interaction between the dendrites and the solute fields. In the solid, the concentrations are relatively uniform since the diffusivity of solid-state Inconel 718 is almost equals zero.
For the validation of the model, the second dendritic arm spacing (SDAS) of the columnar structure was measured and compared with the experimental and analytical values. The analytical data was calculated through the analytical model proposed by Burden and Hunt [39,40]. According to Hunts model, a quantitative relation between the solidification parameters may be established, which includes temperature gradient ($G$), growth rate ($R_m$), and the dendritic arm spacing for columnar dendritic growth, by applying a mass balance and the minimum undercooling

$$
\lambda = 2.83(k\Delta T_0 D \Gamma)^{\frac{1}{4}} R_m^{-\frac{1}{4}} G^{-\frac{1}{2}}
$$

(8.77)

where $\lambda$= dendritic arm spacing ($\mu m$), $k$= partition coefficient, $D$= diffusion coefficient ($mm^2/s$), $\Gamma$= Gibb-Thomson coefficient ($K \cdot mm$), $R_m$ = Solidification velocity ($mm/s$), $G$ = temperature gradient ($^\circ C/mm$) and $\Delta T_0 = |m|C_{\infty}(1 - k)/k$. Based on the results of simulation obtained, the second dendritic arm spacing is calculated. The average value SDAS for the
experimental, analytical and simulation values are $0.621 \pm 0.108 \mu m$, $0.252 \mu m$ and $0.809 \pm 0.122 \mu m$, respectively. The measuring examples are shown in Figure 8.11. In general, the simulation results are very close to the experimental and analytical results. As there are some other factors may affect the SDAS in experiments, such as the complex reheating process during the manufacturing process.

Figure 8.11 Example of measuring second dendrites arm spacing (a) experimental test, (b) microstructure simulation

8.3.3 Beam scanning speed effects

(a) Thermal parameters analysis

Figure 8.12 presents the longitudinal section of the melt pool for the 4 cases during SLM process with their detail dimensions listed in Table 8.4. Based on the simulated results, it is found that with increase of the beam scanning speed, the length of the melt pool would increase while its depth would decrease, which might result from the variant linear electron beam energy density, which is defined as the energy input per unit length with respect to the layer [41]. Given a beam energy power, the linear electron beam energy density would decrease with the increase
of the beam scanning speed, and this would lead to this changing trend of the melt pool. In addition, the fraction of the melt pool trail would increase remarkably from 48.8% to 72.5% with the increase of the beam scanning speed, as can be seen from Table 8.4.

![Figure 8.12](image.png)

Table 8.4 Changing trend of melt pool during SLM process of Inconel 718

<table>
<thead>
<tr>
<th>Beam speed</th>
<th>Depth</th>
<th>Source left</th>
<th>Source right</th>
<th>Total length</th>
<th>Trail, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>200</td>
<td>-46.9</td>
<td>-120.0</td>
<td>126.0</td>
<td>246.0</td>
<td>48.8</td>
</tr>
<tr>
<td>600</td>
<td>-44.9</td>
<td>-157.2</td>
<td>115.2</td>
<td>272.4</td>
<td>57.7</td>
</tr>
<tr>
<td>1000</td>
<td>-41.5</td>
<td>-190.0</td>
<td>98.0</td>
<td>288.0</td>
<td>66.0</td>
</tr>
<tr>
<td>1500</td>
<td>-36.7</td>
<td>-210.0</td>
<td>79.5</td>
<td>289.5</td>
<td>72.5</td>
</tr>
</tbody>
</table>
The thermal gradient values along the fusion boundary of the melt pool during SLM process at beam scanning speed of 200 mm/s, 600 mm/s, 1000 mm/s, and 1500 mm/s are shown in Figure 8.13, Figure 8.14, Figure 8.15, and Figure 8.16, respectively, and the details of the calculated thermal parameters are listed in Table 8.5, Table 8.6, Table 8.7, and Table 8.8, respectively. The points with the maximum cooling rate at the fusion boundary of the melt pool during SLM process have been used as the input for the microstructure simulation as it dominates the morphology and size of the microstructure. The dimensionless value of temperature gradients for 200 mm/s, 600 mm/s, 1000 mm/s, and 1500 mm/s are 1.027, 1.187, 1.442, and 1.068, respectively.

The comparison of the thermal parameters for the 4 cases with different beam scanning speeds is shown in Table 8.9. Generally, with the increase of the beam scanning speed, the dominated thermal gradient will increase. However, at the beam scanning speed of 1500 mm/s, the thermal gradient has a slight drop, which also has been reflected in the dimensionless value of the thermal gradient and the cooling rate. This is consistent with our former studies [42,43], and it might result from the insufficient melt and other defects caused by the high beam scanning speed. In addition, the angle between the thermal gradient direction and the opposite direction of the beam scanning direction will increase from 45.5° to 74.7° with the increase of the beam scanning speed. This means the direction of the maximum thermal gradient will become more and more parallel to the part build direction. In other words, the vertical heat loss path from the melt pool to the bottom substrate become more and more dominated.
Figure 8.13 Thermal gradient at the fusion boundary of the melt pool during SLM process of Inconel 718 at the beam scanning speed of 200 mm/s

Table 8.5 Temperature gradient, growth rate and cooling rate at the fusion boundary of the melt pool during SLM process of Inconel 718 at beam scanning speed of 200 mm/s

<table>
<thead>
<tr>
<th>Point</th>
<th>( G, ^\circ \text{C}/\text{m} )</th>
<th>( \alpha, ^\circ )</th>
<th>( R_m, \text{m/s} )</th>
<th>( \dot{T}, ^\circ \text{C}/\text{s} )</th>
<th>( G/R_m, ^\circ \text{C} \cdot \text{s}/\text{m}^2 )</th>
<th>( U )</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>( 8.61 \times 10^6 )</td>
<td>16.77</td>
<td>0.191</td>
<td>( 1.65 \times 10^6 )</td>
<td>( 4.50 \times 10^7 )</td>
<td>-</td>
</tr>
<tr>
<td>B</td>
<td>( 1.05 \times 10^7 )</td>
<td>39.19</td>
<td>0.155</td>
<td>( 1.63 \times 10^6 )</td>
<td>( 6.78 \times 10^7 )</td>
<td>0.814</td>
</tr>
<tr>
<td>C</td>
<td>( 1.21 \times 10^7 )</td>
<td>45.54</td>
<td>0.140</td>
<td>( 1.69 \times 10^6 )</td>
<td>( 8.64 \times 10^7 )</td>
<td>1.027</td>
</tr>
<tr>
<td>D</td>
<td>( 1.37 \times 10^7 )</td>
<td>52.12</td>
<td>0.123</td>
<td>( 1.69 \times 10^6 )</td>
<td>( 1.12 \times 10^8 )</td>
<td>1.281</td>
</tr>
<tr>
<td>E</td>
<td>( 1.48 \times 10^7 )</td>
<td>57.26</td>
<td>0.108</td>
<td>( 1.60 \times 10^6 )</td>
<td>( 1.37 \times 10^8 )</td>
<td>-</td>
</tr>
<tr>
<td>F</td>
<td>( 2.17 \times 10^7 )</td>
<td>74.46</td>
<td>0.054</td>
<td>( 1.16 \times 10^6 )</td>
<td>( 4.05 \times 10^8 )</td>
<td>-</td>
</tr>
</tbody>
</table>

Figure 8.14 Thermal gradient at the fusion boundary of the melt pool during SLM process of Inconel 718 at the beam scanning speed of 600 mm/s
Table 8.6 Temperature gradient, growth rate and cooling rate at the fusion boundary of the melt pool during SLM process of Inconel 718 at beam scanning speed of 600 mm/s

<table>
<thead>
<tr>
<th>Point</th>
<th>$G$, °C/m</th>
<th>$\alpha$, °</th>
<th>$R_m$, m/s</th>
<th>$\dot{T}$, °C/s</th>
<th>$G/R_m$, °C·s/m²</th>
<th>$U$</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>$6.20 \times 10^6$</td>
<td>30.0</td>
<td>0.519</td>
<td>$3.22 \times 10^6$</td>
<td>$1.19 \times 10^7$</td>
<td>-</td>
</tr>
<tr>
<td>B</td>
<td>$8.72 \times 10^6$</td>
<td>49.74</td>
<td>0.388</td>
<td>$3.38 \times 10^6$</td>
<td>$2.25 \times 10^7$</td>
<td>-</td>
</tr>
<tr>
<td>C</td>
<td>$1.23 \times 10^7$</td>
<td>61.68</td>
<td>0.285</td>
<td>$3.49 \times 10^6$</td>
<td>$4.31 \times 10^7$</td>
<td>0.812</td>
</tr>
<tr>
<td>D</td>
<td>$1.50 \times 10^7$</td>
<td>66.22</td>
<td>0.242</td>
<td>$3.62 \times 10^6$</td>
<td>$6.19 \times 10^7$</td>
<td>1.187</td>
</tr>
<tr>
<td>E</td>
<td>$2.16 \times 10^7$</td>
<td>73.89</td>
<td>0.166</td>
<td>$3.60 \times 10^6$</td>
<td>$1.30 \times 10^8$</td>
<td>1.907</td>
</tr>
<tr>
<td>F</td>
<td>$2.88 \times 10^7$</td>
<td>78.23</td>
<td>0.122</td>
<td>$3.53 \times 10^6$</td>
<td>$2.36 \times 10^8$</td>
<td>-</td>
</tr>
</tbody>
</table>

Figure 8.15 SLM process of Inconel 718 at beam scanning speed of 1000 mm/s

Table 8.7 Temperature gradient, growth rate and cooling rate at the fusion boundary of the melt pool during SLM process of Inconel 718 at beam scanning speed of 1000 mm/s

<table>
<thead>
<tr>
<th>Point</th>
<th>$G$, °C/m</th>
<th>$\alpha$, °</th>
<th>$R_m$, m/s</th>
<th>$\dot{T}$, °C/s</th>
<th>$G/R_m$, °C·s/m²</th>
<th>$U$</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>$8.13 \times 10^6$</td>
<td>59.5</td>
<td>0.507</td>
<td>$4.12 \times 10^6$</td>
<td>$1.60 \times 10^7$</td>
<td>-</td>
</tr>
<tr>
<td>B</td>
<td>$1.21 \times 10^7$</td>
<td>68.9</td>
<td>0.359</td>
<td>$4.34 \times 10^6$</td>
<td>$3.36 \times 10^7$</td>
<td>-</td>
</tr>
<tr>
<td>C</td>
<td>$1.56 \times 10^7$</td>
<td>73.7</td>
<td>0.281</td>
<td>$4.38 \times 10^6$</td>
<td>$5.54 \times 10^7$</td>
<td>1.213</td>
</tr>
<tr>
<td>D</td>
<td>$1.74 \times 10^7$</td>
<td>74.1</td>
<td>0.274</td>
<td>$4.76 \times 10^6$</td>
<td>$6.36 \times 10^7$</td>
<td>1.442</td>
</tr>
<tr>
<td>E</td>
<td>$1.87 \times 10^7$</td>
<td>75.7</td>
<td>0.247</td>
<td>$4.62 \times 10^6$</td>
<td>$7.57 \times 10^7$</td>
<td>1.658</td>
</tr>
</tbody>
</table>
Figure 8.16 Temperature gradient at the fusion boundary of the melt pool during SLM process of Inconel 718 at beam scanning speed of 1500 mm/s

Table 8.8 Temperature gradient, growth rate and cooling rate at the fusion boundary of the melt pool during SLM process of Inconel 718 at beam scanning speed of 1500 mm/s

<table>
<thead>
<tr>
<th>Point</th>
<th>G, °C/m</th>
<th>α, °</th>
<th>Rm, m/s</th>
<th>Ṫ, °C/s</th>
<th>G/Rm, °C·s/m²</th>
<th>U</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>3.49 × 10⁶</td>
<td>33.6</td>
<td>1.250</td>
<td>4.36 × 10⁶</td>
<td>2.79 × 10⁶</td>
<td>-</td>
</tr>
<tr>
<td>B</td>
<td>7.11 × 10⁶</td>
<td>62.5</td>
<td>0.693</td>
<td>4.93 × 10⁶</td>
<td>1.03 × 10⁷</td>
<td>-</td>
</tr>
<tr>
<td>C</td>
<td>1.23 × 10⁷</td>
<td>72.2</td>
<td>0.458</td>
<td>5.62 × 10⁶</td>
<td>2.68 × 10⁷</td>
<td>0.623</td>
</tr>
<tr>
<td>D</td>
<td>1.63 × 10⁷</td>
<td>74.7</td>
<td>0.395</td>
<td>6.44 × 10⁶</td>
<td>4.14 × 10⁷</td>
<td>1.068</td>
</tr>
<tr>
<td>E</td>
<td>2.87 × 10⁷</td>
<td>80.2</td>
<td>0.255</td>
<td>4.76 × 10⁶</td>
<td>7.35 × 10⁷</td>
<td>-</td>
</tr>
<tr>
<td>F</td>
<td>2.17 × 10⁷</td>
<td>80.3</td>
<td>0.253</td>
<td>5.49 × 10⁶</td>
<td>8.54 × 10⁷</td>
<td>-</td>
</tr>
</tbody>
</table>

Table 8.9 Temperature gradient, growth rate and cooling rate at the fusion boundary of the melt pool during SLM process of Inconel 718 at different beam scanning speed

<table>
<thead>
<tr>
<th>Scanning speed (mm/s)</th>
<th>G, °C/m</th>
<th>α, °</th>
<th>Rm, m/s</th>
<th>Ṫ, °C/s</th>
<th>G/Rm, °C·s/m²</th>
<th>U</th>
</tr>
</thead>
<tbody>
<tr>
<td>200</td>
<td>1.21 × 10⁷</td>
<td>45.5</td>
<td>0.140</td>
<td>1.69 × 10⁶</td>
<td>8.64 × 10⁷</td>
<td>1.027</td>
</tr>
<tr>
<td>600</td>
<td>1.50 × 10⁷</td>
<td>66.2</td>
<td>0.242</td>
<td>3.62 × 10⁶</td>
<td>6.19 × 10⁷</td>
<td>1.187</td>
</tr>
</tbody>
</table>
(b) Simulation results

The simulated results are shown in Figure 8.17, Figure 8.19, and Figure 8.20, respectively. It can be seen that the nuclei grew and formed cellular dendritic, which were determined by the step size and the finite element mesh that were decided according to the cost of the computing time. The width of the dendrites also has been characterized, and the statistic results of the secondary dendritic arm spacing (SDAS) from simulation with the trend line are shown in Figure 8.21a. In general, the simulated microstructure follows the rules that the higher the cooling rate, the finer of the dendrites in solidification. In addition, the growth rate the dendrites also has been characterized, as can be seen in Figure 8.21b. With the increase of the beam scanning speed, the growth rate of the dendrites will increase until a certain point and then it will decrease. According to the trend line, the maximum growth rate of the dendrites will be found between the beam scanning speed of 600 and 1500 mms.
Figure 8.17 Microstructure evolution of SLM Inconel 718 at $t = 1$ ms with the laser beam scanning speed of 200 mm/s (Dimensionless thermal gradient $U = 1.027$)

Figure 8.18 Microstructure evolution of SLM Inconel 718 at $t = 1$ ms with the laser beam scanning speed of 600 mm/s (Dimensionless thermal gradient $U = 1.187$)
Figure 8.19 Microstructure evolution of SLM Inconel 718 at $t = 1 \, \text{ms}$ with the laser beam scanning speed of $1000 \, \text{mm/s}$ (Dimensionless thermal gradient $U = 1.442$)

Figure 8.20 Microstructure evolution of SLM Inconel 718 at $t = 1 \, \text{ms}$ with the laser beam scanning speed of $1500 \, \text{mm/s}$ (Dimensionless thermal gradient $U = 1.068$)
8.3.4 Build height effects

(a) Thermal parameters analysis

Longitudinal section of melt pools during SLM Inconel 718 at different heights are shown in Figure 8.22. The size of the melt pool also has been calculated, and the results for the three cases are listed in Table 8.10. It can be seen that there is the almost difference in the size of the melt pool for layer 44 and layer 79 in the first pass. This indicates that the laser beam has arrived a stable state during the manufacturing process, which also can be seen from the section of the trial melt pool that almost has a constant value of 78%. For layer 2, the melt pool has a flat bottom fusion line as its depth is limited by the layer thickness, which is only 60 \( \mu m \) for 2 layers, and this would account for the lower melt size.
The longitudinal section and thermal profile of the melt pool for first pass layer 2 are shown in Figure 8.23. According to the simulated results of the thermal gradient ($G$) and the angle ($\alpha$) between it and the reverse direction of the beam scanning direction along the trail fusion boundary of the melt pool, the growth rate ($R_m$), cooling rate ($\dot{T}$) and dimensionless value of the thermal gradient can be obtained, as listed in Table 8.11. Based on the calculated values,
the maximum cooling rate at the first pass layer 2 is around point D with a value of $2.17 \times 10^6 \, (^\circ C/s)$, and the angle between the thermal gradient direction and the opposite direction of the beam scanning direction is around $62.5^\circ$. The growth of the microstructure at the region around point D along the fusion line will dominate the morphology and size of the microstructure, as shown in Figure 8.23. The dimensionless values of the thermal gradient at the three points of C, D, and E are calculated using the Eq. (1). The dimensionless value of temperature gradient at point D is around 0.907.

![Beam Direction](image)

**Figure 8.23 Inconel 718: (a) Longitudinal section, and (b) Thermal gradient at the fusion boundary, of the melt pool at the first pass layer 2 in SLM process**

<table>
<thead>
<tr>
<th>Point</th>
<th>$G, , ^\circ C/m$</th>
<th>$\alpha, ^\circ$</th>
<th>$R_m, m/s$</th>
<th>$\dot{T}, ^\circ C/s$</th>
<th>$G/R_m, ^\circ C \cdot s/m^2$</th>
<th>$U$</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>$4.02 \times 10^6$</td>
<td>31.6</td>
<td>0.511</td>
<td>$2.05 \times 10^6$</td>
<td>$7.86 \times 10^6$</td>
<td>-</td>
</tr>
<tr>
<td>B</td>
<td>$5.22 \times 10^6$</td>
<td>53.2</td>
<td>0.360</td>
<td>$1.88 \times 10^6$</td>
<td>$1.45 \times 10^7$</td>
<td>-</td>
</tr>
<tr>
<td>C</td>
<td>$6.15 \times 10^6$</td>
<td>55.0</td>
<td>0.344</td>
<td>$2.11 \times 10^6$</td>
<td>$1.79 \times 10^7$</td>
<td>0.664</td>
</tr>
<tr>
<td>D</td>
<td>$7.83 \times 10^6$</td>
<td>62.5</td>
<td>0.277</td>
<td>$2.17 \times 10^6$</td>
<td>$2.83 \times 10^7$</td>
<td>0.907</td>
</tr>
<tr>
<td>E</td>
<td>$8.53 \times 10^6$</td>
<td>66.1</td>
<td>0.243</td>
<td>$2.07 \times 10^6$</td>
<td>$3.51 \times 10^7$</td>
<td>1.118</td>
</tr>
<tr>
<td>F</td>
<td>$1.02 \times 10^7$</td>
<td>72.6</td>
<td>0.179</td>
<td>$1.83 \times 10^6$</td>
<td>$5.69 \times 10^7$</td>
<td>-</td>
</tr>
</tbody>
</table>
Using the same method, the thermal parameters for layer 44 was also calculated, as shown in Figure 8.24. The details of the values are listed in Table 8.12. The comparison of the results is listed in Table 8.13. It can be seen that with the increase of the height, the thermal gradient would have a slight decrease and then increase at the end of the manufacturing process, same variation trend can be seen in all other parameters, including $\alpha$, $R$, $\dot{T}$, $G/R$, and $U$. This consists with experimental results [11].

![Figure 8.24 Inconel 718: (a) Longitudinal section, and (b) Thermal gradient at the fusion boundary, of the melt pool at the first pass layer 79 in SLM process](image)

Table 8.12 Temperature gradient, growth rate and cooling rate at the fusion boundary of the melt pool during SLM process of Inconel 718 at the first pass, layer 79

<table>
<thead>
<tr>
<th>Point</th>
<th>$G$, °C/m</th>
<th>$\alpha$, °</th>
<th>$R$, m/s</th>
<th>$\dot{T}$, °C/s</th>
<th>$G/R$, °C·s/m²</th>
<th>$U$</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>$1.60 \times 10^6$</td>
<td>43.2</td>
<td>0.437</td>
<td>$6.99 \times 10^5$</td>
<td>$3.66 \times 10^6$</td>
<td>-</td>
</tr>
<tr>
<td>B</td>
<td>$2.79 \times 10^6$</td>
<td>62.0</td>
<td>0.281</td>
<td>$7.85 \times 10^5$</td>
<td>$9.92 \times 10^6$</td>
<td>-</td>
</tr>
<tr>
<td>C</td>
<td>$2.90 \times 10^6$</td>
<td>62.2</td>
<td>0.280</td>
<td>$8.13 \times 10^5$</td>
<td>$1.04 \times 10^7$</td>
<td>0.293</td>
</tr>
<tr>
<td>D</td>
<td>$4.49 \times 10^6$</td>
<td>66.6</td>
<td>0.238</td>
<td>$1.07 \times 10^6$</td>
<td>$1.88 \times 10^7$</td>
<td>0.529</td>
</tr>
<tr>
<td>E</td>
<td>$5.19 \times 10^6$</td>
<td>73.2</td>
<td>0.173</td>
<td>$8.99 \times 10^5$</td>
<td>$3.00 \times 10^7$</td>
<td>0.666</td>
</tr>
<tr>
<td>F</td>
<td>$7.41 \times 10^6$</td>
<td>74.4</td>
<td>0.161</td>
<td>$1.20 \times 10^6$</td>
<td>$4.59 \times 10^7$</td>
<td>-</td>
</tr>
</tbody>
</table>
Table 8.13 Temperature gradient, growth rate and cooling rate at the fusion boundary of the melt pool during first pass SLM process of Inconel 718 at different heights

<table>
<thead>
<tr>
<th>layer #</th>
<th>$G$, °C/m</th>
<th>$\alpha$, °</th>
<th>$R_m$, m/s</th>
<th>$\dot{T}$, °C/s</th>
<th>$G/R_m$, °C·s/m²</th>
<th>$U$</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>$7.83 \times 10^6$</td>
<td>62.5</td>
<td>0.277</td>
<td>$2.17 \times 10^6$</td>
<td>$2.83 \times 10^7$</td>
<td>0.907</td>
</tr>
<tr>
<td>44</td>
<td>$4.38 \times 10^6$</td>
<td>66.8</td>
<td>0.237</td>
<td>$1.04 \times 10^6$</td>
<td>$1.85 \times 10^7$</td>
<td>0.498</td>
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<tr>
<td>79</td>
<td>$4.49 \times 10^6$</td>
<td>66.6</td>
<td>0.238</td>
<td>$1.07 \times 10^6$</td>
<td>$1.88 \times 10^7$</td>
<td>0.529</td>
</tr>
</tbody>
</table>

(b) Simulation of microstructure

The phase field profile and concentration profile of microstructure of SLM Inconel 718 at 1ms for layer 2 and layer 79 are shown in Figure 8.25 and Figure 8.26, respectively. It can be seen that layer 2 shows a fast growth rate than the other two layers, which attributes to its largest cooling rate in these three cases as it is in direct contact with the stainless steel sub-plate. With the increase of the build height, the main heat losing approach is from the melt pool down to the sustain plate, and the thermal process would arrive at a stable state. This can be seen from Figure 8.10. As closing to the end of the manufacturing process, there will be a slight increase in the thermal gradient as there will be no further thermal heating cycles. Then, the dendrites would have a little bit higher growth rate than those in the stable state, as reported in Table 8.13. This has been reflected visually in Figure 8.26.

The width of the dendrites and the growth rate of the dendrites also have been characterized, as shown in Figure 8.27. It is known that the higher the cooling rate, the finer of the dendrites in solidification, and the simulated results consistent with this rule. In addition, they are in the range of 0.581 μm - 0.809 μm, which has a very well agreement with our experimental results, of 0.511 μm to 0.845 μm. Based on the growth rate of the dendrites, it can be seen that at the initial state of the manufacturing process, the dendrites have a higher growth rate, and it tend to be constant along with the increase of the build height.
Figure 8.25 Microstructure evolution of SLM Inconel 718 for layer 2 at $t = 1\, \text{ms}$ and dimensionless thermal gradient $U = 0.907$

Figure 8.26 Microstructure evolution of SLM Inconel 718 for layer 79 at $t = 1\, \text{ms}$ and dimensionless thermal gradient $U = 0.529$
8.4 Conclusions

A phase field model was developed for microstructural modeling of SLM-processed Inconel 718 alloy. FORTRAN code was used to solve the phase field equations. The temperature gradient and solidification velocity were extracted from the thermal model, which will be used as the input to the phase field model. Effects of beam scanning speed and the build height have been investigated and evaluated. Based on the results, the main findings are as follows.

(a) The phase-field model can be used to predict the morphology and size of the microstructure in solidification as the columnar dendritic arm spacing values estimated from the phase field simulation match the experimental and analytical results.

(b) With the increase of the build height, the dominated values of thermal gradient and cooling rate would increase first and then slightly increase at the top end of the manufacturing process. The microstructure evolution along the build height agrees well the test.

(c) The manufacturing parameters play an important role in the formation of microstructure in SLM process. Generally, with the increase of the beam scanning speed, the dominated thermal gradient will increase until a certain value and then decrease and the direction of the maximum thermal gradient will become more and more parallel to the part build direction.
(d) The temperature gradient is shown to affect the dendrite growth significantly, and a greater temperature gradient will result in a higher growth speed.

(e) Most of the cellular dendrites have a preferred growth direction with the angle is around $45 - 72^\circ$.

(f) This study demonstrates that phase field method can be a powerful tool for quantitative simulation of microstructural evolution in SLM process.
References


Dendrite Growth under Forced Flow Conditions in an Al–Cu Welding Molten Pool,”

Morphology and Solute Distributions under Transient Conditions in an Al–Cu Welding


Trans Tech House, 4711, Aedermannsdorf, Switzerland, 1986. 244.

246101.


[30] Provatas, N., and Elder, K., 2011, Phase-Field Methods in Materials Science and
Engineering, John Wiley & Sons.

Modeling of Binary Alloy Solidification with Coupled Heat and Solute Diffusion,” Phys.


[34] Liu, Z., and Qi, H., 2015, “Effects of Processing Parameters on Crystal Growth and

Solidification Microstructure of Nickel-Base Superalloy Fabricated by Laser Powder Bed
Fusion,” Addit. Manuf.


CHAPTER 9
CONCLUSIONS AND RECOMMENDATIONS FOR FUTURE RESEARCH

9.1 Conclusions

Powder bed fusion additive manufacturing (AM) technologies have the capability of fabricating functional components directly from 3D-models using metal powder, and selective laser melting (SLM) and electron beam additive manufacturing (EBAM) are two typical powder-bed AM systems which could produce parts having comparable strength with the counterparts from traditional methods. Specifically, SLM process can fabricate parts with complex profiles at a high geometrical accuracy, and EBAM has the capability of creating fully dense parts in a variety of metal alloy applications. These characterizations have made SLM and EBAM into promising processes to produce functional parts for various applications. As the operating parameters and the morphology of the parts could significantly affect the thermal history of the process, which would further affect the microstructure evolution in the final parts and further determines the attainable mechanical properties, so this study was conducted to comprehensively understand the manufacturing process and achieve the desired properties for the finishing parts. The major findings obtained so far are summarized as follows.

(a) Part A: Microstructure and texture

(1) In general, for the SLM/EBAM build parts, the X-Z plane shows a typical columnar structure and Z plane presents an equiaxed microstructure. The as-fabricated Inconel 718 samples
have a distinct \{0 0 1\} texture in the Z plan and a strong \{1 0 1\} texture in the Y-plane. There are strong textures presenting in the \(\beta'\) phase in the EBAM TI-6Al-4V alloy, and the texture would transfer to the texture of \(\alpha\) phase during the phase transformation from \(\beta'\) to \(\beta\) and \(\alpha/\alpha'\), which have strong textures of \(\langle0 0 0 1\rangle\) and \(\langle1 1 2 0\rangle\) parallel to the z-axis.

(2) Fine colonies of cellular dendrites with a cell spacing of 0.511 ~ 0.845 \(\mu m\) were revealed in the grains in SLM Inconel 718, and this indicating the SLM process having a cooling rate of \(1.74 \times 10^6\) K \(\cdot\) s\(^{-1}\) (°C \(\cdot\) s\(^{-1}\)) to \(3.88 \times 10^7\) K \(\cdot\) s\(^{-1}\) (°C \(\cdot\) s\(^{-1}\)).

(3) The temperature gradient has significant effects on the dendrites. With the increase of the build height, the dominated values of thermal gradient and cooling rate would increase first and then slightly increase at the top end of the manufacturing process. The microstructure evolution along the build height agrees well the test.

(4) Laves phase in the morphology of irregular white shape was observed in the intercellular zones, and they would change from coarse and interconnected particles to discrete Laves phase under continued effects of thermal cycles. Moreover, the maximum intensity of the texture will increase with the continuing effects of the thermal cycles.

(5) The overhang feature will induce a lower thermal cooling rate and facilitate the formation of equiaxed microstructure at the bottom of the surface and wider columnar structures with a width of 192.4 – 202.5 \(\mu m\), which is about three times of that from the solid section (66.1 \(\mu m\)). In addition, more defects than the solid section, including porosity and un-melted particles were observed in the overhang region.

(b) Part B: Mechanical properties and residual stress
(1) The mechanical properties of the parts are comparable with or superior to the counterparts made by traditional methods, and heat treatment for stress relief has significant effects on the hardness of the samples.

(2) There are some differences between the X-plane and Z-plane in the mechanical properties, but there are no significant anisotropic characteristics in mechanical properties.

(3) The residual stress is unevenly distributed in the parts, and there is no notable difference between the X plane and Y plane of SLM Inconel 718 samples. The EBAM processed parts have lower absolute values of residual stress than the SLM-built parts. At some areas, the maximum absolute value of the residual stress in the SLM Inconel 718 sample is around 350 MPa, about 30 percent of its yield strength.

(4) The build height and the thermal cycles do not show remarkable influences on the residual stress in the parts, while the support structure has significant effects on the residual stress. Especially, the right angle interface of the geometry would induce a stress concentration.

(5) In general, the area fraction of the porosity is below 2%, and there is no remarkable effects were found from the thermal cycles and the build height on the distribution of the porosity. However, the electron beam scanning speed and the overhang feature indeed have significant influences on the porosity.

(c) Part C: Microstructure simulation

(1) The phase-field model was used to predict the morphology and size of the microstructure in solidification process of Inconel 718 during SLM process, and it was validated by the comparison of the columnar dendritic arm spacing values estimated from the phase field simulation and the experimental results.
(2) The thermal gradient plays an important role in the microstructure evolution of SLM process. With the increase of the beam scanning speed, the dominated thermal gradient will increase until reaching a certain value and then decrease, and the direction of the maximum thermal gradient will become more and more parallel to the part build direction. In addition, a larger temperature gradient will result in a higher growth speed of the dendrites.

(3) The phase field method is a powerful tool for quantitative simulation of microstructural evolution in SLM process to predict the morphology and size of the microstructure in solidification.

9.2 Contributions of this Study

The contributions of this study are summarized below:

(1) Metal components of Ti-6Al-4V and Inconel 718 were designed and fabricated using two typical powder bed additive manufacturing technologies, EBAM and SLM, respectively, to deep understanding the manufacturing process through materials characterization.

(2) The microstructure of the fabricated parts was observed and analyzed by optical microscopy, scanning electron microscopy, energy dispersive X-ray spectroscopy, and electron backscatter diffraction, and the mechanical properties were obtained through nanoindentation and Vickers’ indentation tests.

(3) The distribution of the residual stress in the parts was evaluated through instrumented indentation technique, which was based on the experimental correlation between indentation characteristic parameters and residual stresses. Vickers micro-indentation was used in this study for the rapid evaluation of residual stresses in microstructural scale.
(4) The microstructure evolution, including the morphology and solute concentration, during the solidification process in the SLM process, was simulated by phase field method based on the dominated cooling rate obtained from thermal analysis, which is critical in fundamental understanding and optimizing the manufacturing process.

(5) The effects of the beam scanning speed, build height, thermal cycles, and support structure on the fabricated components were thoroughly studied by characterization of the microstructure and mechanical properties and modeling of the microstructure evolution during the solidification process.

(6) The corrections between the manufacturing process, thermal history, microstructure, texture, defects, residual stress and mechanical properties were established. The results have very great significance in understanding and optimizing the manufacturing processes.

9.3 Recommendations for Future Research

The present study has provided a better understanding of the powder-bed fusion additive manufacturing technologies based on the thorough study of processing, microstructure, and properties of the metallic components fabricated by the two typical powder-bed fusion additive manufacturing technologies, electron beam additive manufacturing, and selective laser melting. In addition, the simulation of the microstructure evolution during solidification process in SLM process could further advance and fast the optimization of the manufacturing process. Future study can be explored in the following three major channels,

(1) In this study, only two mechanical properties, elastic modulus, and hardness, obtained from nanoindentation and Vicker’s indentation tests used to characterize of the fabricated
components. Other properties, such as yield strength, tensile strength, ductility, and fatigue strength, and failure analysis are needed to be further studied.

(2) The effect of beam scanning speed during SLM process on the microstructure evolution of Inconel 718 was studied in this study with the case of 600 mm/s was verified by the experimental results. Further experiments are needed in order to deepen understanding the effect of the beam scanning effects.

(3) The residual stresses induced in the EBAM and SLM processes were characterized by Vickers’s indentation tests. Same material with different manufacturing processes is needed in future study in order to get a good comparison. In addition, using another measuring method to evaluate the residuals stress for comparison of the results would make the conclusion more solid. Moreover, the effects of the procedures that were proposed to reduce the residual stress in the parts are needed to be further studied.

(4) The structural morphology of the fabricated components has significant effects on the microstructure evolution and the properties, and only one overhang feature with two types of support structures was investigated and evaluated in this study. Further study is needed to evaluate the effects of various overhang features with different support structures.
APPENDIX A

PHASE FIELD MODELING: DISCRETIONS OF THE DIFFERENTIAL EQUATIONS AND COMPUTATION PROCEDURES

A.1 Discretion of the Differential Equations

(a) Phase field equation

The mass/heat diffusion equations are examples of flux conserving equations. They have the form,

$$\frac{\partial f}{\partial t} = -\nabla \cdot j_f \quad (A.1)$$

where $j_f$ is a flux of some quantity, which is typically related to the gradient of the field. The flux balance required to conserve solute in the case of two-sided diffusivity. The finite volume method is used to discretize flux conserving equations.

$$\nabla f = \text{grad } f = \left(\frac{\partial f}{\partial x}, \frac{\partial f}{\partial y}\right) = \frac{\partial f}{\partial x} \vec{i} + \frac{\partial f}{\partial y} \vec{j} \quad (A.2)$$

$$\int_{\text{surf}} \frac{\partial f}{\partial t} d\vec{x} d\vec{y} = - \iint_{d\vec{x}d\vec{y}} \nabla \cdot j_f d\vec{x} d\vec{y} = - \iint_{d\vec{x}d\vec{y}} \nabla \cdot j_f d\vec{s} = \left\{ -j_{\text{Right}} \cdot \vec{i} dy + j_{\text{Top}} \cdot \vec{j} dx - j_{\text{Left}} \cdot \vec{i} dy - j_{\text{Bottom}} \cdot \vec{j} dy \right\} \quad (A.3)$$
Figure A.1 Schematic of the element \((i, j)\) and its adjacent elements \(\Delta x = \Delta y\)

\[
\begin{align*}
\left[ 1 - (1 - k) \frac{z - \bar{V}_p t}{\bar{l}_T} \right] a_s(\bar{n})^2 \frac{\partial \phi}{\partial t} &= \bar{V} \cdot (a_s(\bar{n})^2 \bar{V} \phi) - \frac{\partial}{\partial x} \left[ a_s(\bar{n}) a_s(\bar{n})' \frac{\partial \phi}{\partial y} \right] + \frac{\partial}{\partial y} \left[ a_s(\bar{n}) a_s(\bar{n})' \frac{\partial \phi}{\partial x} \right] + \phi - \phi^3 \\
- \lambda (1 - \phi^2)^2 \left( U + \frac{z - \bar{V}_p t}{\bar{l}_T} \right) &= (A.4)
\end{align*}
\]

\[
\nabla \cdot \bar{j} = \bar{V} \cdot (a_s(\bar{n})^2 \bar{V} \phi) - \frac{\partial}{\partial x} \left[ a_s(\bar{n}) a_s(\bar{n})' \frac{\partial \phi}{\partial y} \right] + \frac{\partial}{\partial y} \left[ a_s(\bar{n}) a_s(\bar{n})' \frac{\partial \phi}{\partial x} \right]
\]

\[
= \left\{ \bar{V} \left[ a_s(\bar{n})^2 \frac{\partial \phi}{\partial x}, a_s(\bar{n})^2 \frac{\partial \phi}{\partial y} \right], \left[ a_s(\bar{n}) a_s(\bar{n})' \frac{\partial \phi}{\partial x}, -a_s(\bar{n}) a_s(\bar{n})' \frac{\partial \phi}{\partial y} \right] \right\}
\]

\[
= \left\{ \bar{V} \cdot \left[ \left[ a_s(\bar{n})^2 \frac{\partial \phi}{\partial x} \right], \left[ a_s(\bar{n})^2 \frac{\partial \phi}{\partial y} \right], \left[ -a_s(\bar{n}) a_s(\bar{n})' \frac{\partial \phi}{\partial x} \right], \left[ a_s(\bar{n}) a_s(\bar{n})' \frac{\partial \phi}{\partial y} \right] \right] \right\}
\]

\[
= \bar{j}_{Right} + \bar{j}_{Top} - \bar{j}_{Left} - \bar{j}_{Bottom} \quad (A.5)
\]

Where, \(\bar{j}_{Right} = \bar{j} \cdot \hat{y} \ dy = \left( \bar{j}_{Right} \right)_x = a_s(\bar{n})^2 \frac{\partial \phi}{\partial x} + a_s(\bar{n}) a_s(\bar{n})' \frac{\partial \phi}{\partial y} = JR, \bar{j}_{Top} = \bar{j} \cdot \hat{y} \ dx = \left( \bar{j}_{Top} \right)_y = a_s(\bar{n})^2 \frac{\partial \phi}{\partial y} - a_s(\bar{n}) a_s(\bar{n})' \frac{\partial \phi}{\partial x} = JT, \bar{j}_{Left} = \bar{j} \cdot (-\hat{y}) \ dy = -\left( \bar{j}_{Right} \right)_x = a_s(\bar{n})^2 \frac{\partial \phi}{\partial x} +
\]
\[ a_s(\vec{n})a_s(\vec{n})' \frac{\partial \vec{n}}{\partial y} = jL \text{ and } \mathbf{j}_{\text{Bottom}} = \mathbf{j} \cdot (-j) \, dx = -(\mathbf{j}_{\text{Bottom}})_Y = a_s(\vec{n})^2 \frac{\partial \phi}{\partial y} - \]

\[ a_s(\vec{n})a_s(\vec{n})' \frac{\partial \phi}{\partial x} = JB. \]

Therefore, at time \( t = n\Delta t \), we have,

\[ \phi^{n+1} = \frac{\Delta t}{[1 + (1 - k)\zeta] + a_s(\vec{n})^2} \]

\[ . \left\{ \frac{\vec{v} \cdot \left[ a_s(\vec{n})^2 \frac{\partial \phi^n}{\partial x}, a_s(\vec{n})^2 \frac{\partial \phi^n}{\partial y}, -a_s(\vec{n})a_s(\vec{n})' \frac{\partial \phi^n}{\partial x}, a_s(\vec{n})a_s(\vec{n})' \frac{\partial \phi^n}{\partial y} \right]} {+\phi^n - \phi^{n+1} - \lambda(1 - \phi^n)^2 (U + \zeta) \right\} \]  

\[ (A.6) \]

Where \( a_s(\vec{n})' = \frac{\partial a_s(\phi)}{\partial \phi} = -4\epsilon_4 \sin 4\phi = -\frac{16\epsilon_4 \phi_x \phi_y (\phi_x^2 - \phi_y^2)}{\phi_x^2 + \phi_y^2} \), and \( \zeta = \frac{z - \bar{v}_p t}{l_T} = \frac{T(z,t)-T_0}{\Delta T_0} = \frac{T(z,t)-T_0}{|m(1-k)c_t^0|} \)

\[ (b) \text{ Concentration equation} \]

For the concentration equation, its update can be efficiently done using a finite volume method since it is a flux conserving equation.

\[ \frac{\partial c}{\partial t} = \nabla \cdot \left( D\bar{q}(\phi) c \frac{\bar{v}}{\bar{u}} + a(\phi) W(1 - k)c_t^0 e^n \frac{\partial \phi}{\partial t} \frac{\bar{v}}{\bar{u}} \right) \]  

\[ (A.8) \]

Where \( c = \frac{(1-\phi)c_t + (1+\phi)c_s}{2} \), \( \bar{q}(\phi) = \frac{1-\phi}{1+k-(1-k)\phi} \), \( a(\phi) = \frac{1}{2\sqrt{2}} \), \( u = \ln \left( \frac{2c}{c_t^0[1 + k - (1-k)\phi]} \right) \), \( e^n = \frac{2c}{c_t^0[1 + k - (1-k)\phi]} = 1 + (1 - k)U. \]

Thus,

\[ \nabla \cdot \mathbf{j} = \nabla \cdot \left( D\bar{q}(\phi) c \frac{\bar{v}}{\bar{u}} \mathbf{j}_{\text{at}} \right) = \mathbf{\nabla} \cdot \left( D \frac{1-\phi}{2} c \frac{\bar{v}}{\bar{u}} + \frac{1}{2\sqrt{2}} [1 + (1-k)U] \frac{\partial \phi}{\partial t} \frac{\bar{v}}{\bar{u}} \right) \]  

\[ (A.9) \]

Where \( c \equiv \frac{c_{\text{Real}}}{c_t^0}, D = \frac{D_L \tau}{W_\phi^2}. \)
The discrete update equation for the concentration $c^{n+1}(i,j)$ is given by

$$c^{n+1}(i,j) = c^n(i,j) - \frac{\Delta t}{\Delta x} \left\{ (J^R_R - J^R_L) + (J^T_T - J^T_B) \right\} \quad (A.10)$$

The notation $J^R_R = \vec{j} \cdot \vec{n}$, with $\vec{n}$ being the unit normal of the right edge of the finite volume.

Referring to the right hand edge of the control volume, the quantities that enter $J_R$ are evaluated at $(i + 1/2; j)$ as follows

$$Q\left(\varnothing^n\left(i + \frac{1}{2}; j\right)\right) c^n\left(i + \frac{1}{2}; j\right)$$

$$= Q\left(\frac{\varnothing^n(i + 1,j) + \varnothing^n(i,j)}{2}\right) c^n\left(\frac{c^n(\varnothing(i + 1,j)) + c^n(\varnothing(i,j))}{2}\right) \quad (A.11)$$

$$\vec{v} \cdot \vec{u} \equiv \frac{\partial u^n}{\partial x} \mid_{i + \frac{1}{2}; j} = \frac{EU^n(i + 1, j) - EU^n(i, j)}{\Delta x(EU^n(i + 1, j) + EU^n(i, j))} \quad (A.12)$$

$$\left[ e^u \frac{\partial \varnothing^n}{\partial t} \right]^{n+1}_{(i + \frac{1}{2}; j)}$$

$$= \left( e^u(\varnothing^n(i+1,j), c^n(i+1,j)) + e^u(\varnothing^n(i,j), c^n(i,j)) \right) \left( \frac{\partial \varnothing^{n+1}}{\partial t} \mid_{(i+1;j)} + \frac{\partial \varnothing^{n+1}}{\partial t} \mid_{(i;j)} \right) \quad (A.13)$$

$$\frac{\partial \varnothing^n}{\partial x} \mid_{(i + \frac{1}{2}; j)} = \frac{\varnothing(i + 1,j) - \varnothing(i,j)}{\Delta x} \quad (A.14)$$

$$\frac{\vec{v} \cdot \varnothing}{|\vec{v} \cdot \varnothing|} \mid_x^R = \frac{\left( \frac{\partial \varnothing^n}{\partial x} \right)^n \mid_{i + \frac{1}{2}; j}}{\left\{ \left( \frac{\partial \varnothing^n}{\partial x} \right)^n \mid_{i + \frac{1}{2}; j} + \left( \frac{\partial \varnothing^n}{\partial y} \right)^n \mid_{i + \frac{1}{2}; j} \right\} \frac{1}{2}} \quad (A.15)$$

Therefore,
\[ J_R = \vec{j} \cdot \vec{i} = -DQ \left( \phi^n \left( i + \frac{1}{2}, j \right) \right) c^n \left( i + \frac{1}{2}, j \right) \frac{\partial u^n}{\partial x} |_{i + \frac{1}{2}, j} \]

\[ - \frac{1}{2\sqrt{2}} \left[ e^u \left( \phi \right)^n_{t+1, i+\frac{1}{2}, j} \right] \frac{\nabla \phi}{\nabla R} |_{x} (6.85) \]

The fluxes in the other directions are calculated analogously.

(c) Temperature

\[ \frac{\partial \theta}{\partial t} = \alpha \nabla^2 \theta + \frac{1}{2} \frac{\partial \phi}{\partial t} \quad (A.16) \]

Where, \( \alpha = a_2 \lambda \).

For the Laplacian operator, it is obtained by incorporating information from the next nearest neighbors.

\[ \nabla^2 \theta = \frac{\text{temp}(i+1,j) + \text{temp}(i-1,j) + \text{temp}(i,j+1) + \text{temp}(i,j-1)}{4} \]

\[ + \frac{\text{temp}(i+1,j+1) + \text{temp}(i-1,j+1) + \text{temp}(i+1,j-1) + \text{temp}(i-1,j-1)}{4} \]

\[ - 3 \times \text{temp}(i,j) \]

\[ \times \left( \frac{1}{dx} \right) \quad (A.17) \]

Then a simple forward differencing scheme is used to update the temperature field.

A.2 Initial and Boundary Conditions

(a) Initial condition

Without considering the effect of convection in solidification, the main factors affecting the growth of the nuclei are thermal diffusion and solute diffusion. Assume the radius of the nuclei is \( r \), then we have

\[ (x - a)^2 + (y - b)^2 <= r^2, \ \phi = +1, \ \theta = 0, \ c = c_s \]
\[(x - a)^2 + (y - b)^2 \geq r^2, \ \emptyset = -1, \ \theta = -\Delta, \ c = c_\infty, \ \Delta \text{ is the undercooling}\]

(b) Boundary condition

Zero-Neumann boundary condition has been used in this study, which means there is no heat flux through the boundary.

\[
\frac{\partial \emptyset}{\partial n} = \frac{\partial c}{\partial n} = \frac{\partial T}{\partial n} = 0 \quad (A.18)
\]

A.3 Computational Procedures

The phase-field model is simulated using FEM, with zero-flux boundary conditions in both \(\emptyset\) and \(U\). A FORTRAN code was developed to solve the equations, and its flow chart is shown in Figure 6.4. Parallel computation is utilized to reduce the overall computational time on a supercomputer. The grid spacing is set to \(dx = dy = 0.8 \ \mu m\) in all cases with 1105 by 1105 grid points contained in this grid system. The simulations are initialized with a thin solid layer on the left-hand side of the domain. The domain has dimensions of 50 \(\mu m\) in length and 50 \(\mu m\) in width, which is long enough to always include the liquid part on the right-hand side of the domain in a steady state. Steady state means that the dendrite tips are at constant temperature and move with a constant velocity equal to the solidification velocity. The initial solute profile \(C\) is set to the \(C_\infty\) in the liquid and \(kC_\infty\) in the solid. The other parameters used in the calculation are listed in Table A.1.

### Table A.1 Input in the calculations

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<th>Input</th>
<th>Value</th>
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<td>Calculate Domain, (\mu m)</td>
<td>50 (\times) 50</td>
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</table>
Mesh, \((N_x \& N_y \leq 2000)\)  
\[ 1105 \times 1105 \]

<table>
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<th>Physical properties</th>
<th>Value</th>
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<td>Interface kinetics time, (\tau_0)</td>
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<td>Step size, (\Delta x)</td>
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<tr>
<td>Time step, (\Delta t)</td>
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<td>Coupling constant between potentials, (\lambda)</td>
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<td>Nuclei Radius, (r)</td>
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<td>Anisotropy strength, (\epsilon_4)</td>
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Table A.2 Physical properties of Inconel 718

<table>
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<tr>
<td>Liquid diffusion coefficient, (D_L) ((m^2s^{-1}))</td>
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</tr>
<tr>
<td>Diffusion coefficient in solid, (D_s) ((m^{-2}s^{-1}))</td>
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<td>Gibbs–Thomson coefficient, (\Gamma) ((K \cdot m))</td>
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</tr>
<tr>
<td>Solidus Temperature, (T_S) ((K))</td>
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</tr>
<tr>
<td>Liquidus Temperature, (T_L) ((K))</td>
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</tr>
<tr>
<td>Latent heat of fusion, (L_f) ((J/Kg))</td>
<td>227000</td>
</tr>
<tr>
<td>Specific heat, (C_p) ((J/Kg/K))</td>
<td>600</td>
</tr>
<tr>
<td>Liquidus line of slope, (m)</td>
<td>-10.5</td>
</tr>
</tbody>
</table>
Figure A.2 Flow chart for the FORTRAN code in microstructural simulation