

Understanding Process-Structure
Relationship For Site-Specific
Microstructure Control in Electron
Beam Powder Bed Additive
Manufacturing Process Using Numerical
Modeling

A Dissertation Presented for the
Doctor of Philosophy
Degree

The University of Tennessee, Knoxville

Narendran Raghavan

December 2017

© by Narendran Raghavan, 2017
All Rights Reserved.

To my parents,

Anandi and Raghavan

my sister and brother-in-law,

Siva and Muthu

my nephew and niece,

Vishwa and Mithra.

Acknowledgements

I would like to express my sincere gratitude to my advisor **Prof.Sudarsanam Suresh Babu** for his continuous mentoring and support through the years. His scientific insights, guidance and constructive criticisms have been immensely helpful in shaping up my thought process towards efficient way of conducting a research work. This work wouldn't have been possible without his mentoring.

It's been a pleasure to work with Dr.Ryan Dehoff at Oak Ridge National Laboratory. His experience, innovative ideas and inputs helped me with successful experiments at Manufacturing Demonstration Facility. I have learnt a great extent from Dr.Srdjan Simunovic, Dr.John Turner and Dr.Sreekanth Pannala at Oak Ridge National Laboratory. I would like to thank them for their mentorship and input in helping me with the numerical modeling framework. Dr.Neil Carlson at Los Alamos National Laboratory has been immensely patient in answering my novice questions on his code which I used in my work. I would like to acknowledge the support from the committee members Dr.Hahn Choo and Dr.William Hamel. I would like thank Prof.Lee Riedinger and Bredesen Center for Interdisciplinary Research and Graduate Education for providing me with the opportunity to join the program. Summer internship at ORNL provided by the program prior to starting the course works gave me head start in understanding the functioning of the laboratory and in choosing the research area of my interest. I have learnt a great deal from the discussions with Dr.Peeyush Nandwana, Dr.Michael Kirka and Dr.Niyanth Sridharan. I would like to thank all the members of Advanced Materials an Manufacturing Group (AMMG) at the University of Tennessee Knoxville for their "Stone-pelting" and discussions during the weekly group meetings. Specifically, I want to thank Jacob Raplee for his help in analyzing

the infrared data. Also, this work would not have been possible without the financial support from Advanced Manufacturing Office (AMO) of Department of Energy (DOE). This research was sponsored by the US Department of Energy, Office of Energy Efficiency and Renewable Energy, Advanced Manufacturing Office, under contract DE- AC05-00OR22725 with UT-Battelle, LLC. Research was also sponsored by the Laboratory Directed Research and Development Program of Oak Ridge National Laboratory, managed by UT-Battelle, LLC.

Finally, I would like to thank my family and my uncles Dr.Paramasivan and Er.Sachidhanantham for their love and encouragement through the years.

*"Scientific research is not about looking for right answers,
but asking right questions"*

Abstract

Additive Manufacturing (AM), the fabrication of 3-D parts from CAD models, is a disruptive technology that is transforming the manufacturing industry. In any manufacturing process, understanding the process-structure-property-performance (PSPP) linkage is crucial to predict the lifespan of the manufactured component. In the PSPP linkage, the solidification microstructure (liquid-solid phase transformation) is one of the important factors affecting the mechanical property and performance of the additively manufactured components. During rapid solidification, the solidification microstructure is dictated by the thermal gradient (G) at the liquid-solid interface and velocity (R) of the liquid-solid interface. In this work, the transient heat transfer and fluid flow within the molten pool is numerically analyzed using Truchas, a solidification code developed at Los Alamos National Laboratory. The spatial and temporal variations of temperature gradient (G) and growth velocity (R) at the liquid-solid interface of the melt pool were calculated using the data from numerical modeling.

1. The obtained transient knowledge from numerical modeling is used to design new melt scan strategies for an electron beam powder bed AM system (Arcam[®]) and achieve the following.
 - Control the grain size of the columnar grains.
 - Control the grain morphology transition (Columnar to Equiaxed).
2. The effect of build geometry on the grain morphology control has been reported. It is found that the scan parameters have to be modified as a function of geometry. The data from in-situ process monitoring (infrared imaging) is also analyzed to distinguish the underlying grain morphology.

3. The effect of fluid flow and surface tension as a function of different surfactant concentration is analyzed numerically and compared with pure heat conduction simulations. It is concluded that the necessity of fluid flow in the model depends on the scan strategy in addition to the alloy dependent surface tension.

Table of Contents

1	Introduction	1
1.1	Introduction to Additive Manufacturing Process	1
1.1.1	Business and Environmental Implications of Additive Manufacturing Technology	2
1.1.2	A Brief Introduction to Arcam [®] process	5
1.1.3	Alloys used in powder bed AM processes	7
1.2	Factors Affecting Mechanical Performance of Engineering Components . . .	8
2	Literature Review and Scientific Gaps	10
2.1	Literature Review	10
2.1.1	Fundamentals of Solidification	10
2.1.2	Evolution of Microstructure in Rapid Solidification Processes	19
2.1.3	Electron Beam Welding	21
2.1.4	Modeling of Rapid Solidification Process	24
2.2	Scientific Gaps and Research Plan	32
2.2.1	Research Gaps	32
2.2.2	Material System	32
2.2.3	Numerical Code	33
2.2.4	Experimental Methodology	35
3	Overview of Numerical Modeling Framework	36
3.1	Truchas	36
3.1.1	Conservation Equations	36

3.1.2	Heat Source Profile	37
3.1.3	Limitations and Assumptions of the model	39
3.1.4	Initial and Boundary Conditions	39
3.1.5	Geometry and Meshing	40
3.1.6	Scaling of the Code	40
3.1.7	Material Properties	42
3.1.8	Calculation of G and R	42
4	Grain size and Primary Dendrite Arm Spacing control	47
4.1	Influence of Grain Size on Mechanical Properties	47
4.2	Melt-Scan Strategy	48
4.2.1	Conventional Melt Sequence	48
4.2.2	Proposed Melt Strategy	49
4.3	Experimental Set up	50
4.4	Results and Discussions	51
4.4.1	Melt pool shape	51
4.4.2	Variation in Primary Dendrite Arm Spacing	54
4.4.3	Variation in Grain Size	61
4.5	Summary	71
5	Evaluation of Columnar to Equiaxed Transition (CET)	72
5.1	Motivation	72
5.2	Qualitative Analysis of Parameters	73
5.3	Quantitative Analysis	76
5.3.1	Theory	76
5.3.2	Calculation of Spatio-temporal Φ	77
5.3.3	ANOVA	78
5.4	Experimental Validation	81
5.4.1	Scan Strategy and Fabrication	81
5.4.2	Insignificance of Beam Diameter	81
5.4.3	Significance of Preheat Temperature	83

5.5	Summary	85
6	Geometry Dependency of CET and Infrared In-situ Monitoring	88
6.1	Effect of Build Geometry on CET	88
6.1.1	Scan Strategy and Parameters	89
6.1.2	Comparison of Texture	91
6.1.3	Analysis of Island Melt Strategy to Overcome Geometry Effect	94
6.2	Infrared Imaging	99
6.2.1	Algorithm for Calibration of IR data	100
6.2.2	Experimental Observation	101
6.3	Summary	105
7	Effect of Fluid Flow and Surface Tension Gradient on Solidification	108
7.1	Effect of Surface Tension on Convection	108
7.2	Conservation Equations	109
7.3	Simulation and Results	110
7.3.1	Preaheat Temperature: 25°C	112
7.3.2	Preaheat Temperature: 1000°C	113
7.3.3	Comparison of Peclet number	114
7.3.4	Effect of fluid flow on microstructure	116
7.3.5	Significance of Scan Strategy on Fluid Flow and Experimental Com- parison of melt pool shape:	117
7.4	Summary	120
8	Conclusion and Path Forward	122
8.1	Summary	122
8.2	Path Forward	123
	Bibliography	128
	Appendices	142

A	Appendix-1	143
A.1	Nucleation Rate as a Function of Cooling Rate	143
B	Appendix-2	145
B.1	CUBIT: Mesh Generation Script Template	145
B.2	Python script to filter G and R at interface	146
Vita		150

List of Tables

2.1	Nominal composition (wt%) of Inconel 718 [79].	33
2.2	Verification and validation of Truchas [81].	34
3.1	Strong Scaling: Wall Time Usage Analysis	42
3.2	Thermophysical properties of IN718 [93, 94]	43
4.1	Values of input parameters of proposed scan strategy	51
4.2	Properties of IN718 used in analytical equations to predict PDAS [106]. . . .	54
5.1	Values of input parameters of proposed scan strategy	78
5.2	2 ⁴ simulation combinations and the corresponding volume fraction of equiaxed grains used in DOX	79
5.3	Analysis of Variance (ANOVA) of the design	80
6.1	Spot melt parameter list to study the effect of geometry on CET	90
6.2	Spot melt parameter list to study the effect of geometry on CET	98
7.1	Comparison of Φ value with and without fluid flow in the model	116
7.2	Experimental comparison of width and depth of the melt pool with and without fluid flow in the model	120

List of Figures

1.1	Classification of metal AM systems	2
1.2	Energy Usage Comparison between AM and IM [3]	4
1.3	Production scale additively manufactured fuel nozzle from GE Aviation [5] .	6
1.4	Processing steps in Arcam [®]	6
1.5	Schematic of an Arcam [®] machine and corresponding picture of the build chamber [10]	7
2.1	Free energy change during homogeneous nucleation of Aluminum [27]	12
2.2	Heterogeneous nucleation on an existing wall or substrate [27]	13
2.3	Qualitative Comparison of free energy change required for homogeneous, heterogeneous nucleation and epitaxial growth. [25]	15
2.4	Variation of solidification morphology as a function of G and R	16
2.5	(a) Phase Diagram with respect to solute concentration (b) Solute enrichment at the interface	17
2.6	(a) Solute enrichment (b) No constitutional undercooling at the interface (c) Presence of constitutional undercooling	18
2.7	(a) Breakdown of liquid-solid interface due to solute rejection as a function of time (b) Change in morphology by introducing impurity [28]	19
2.8	Epitaxial grain growth at (a) High velocity (b) Low velocity (c) Oscillation[28]	20
2.9	EBSD of (a) unstirred melt pool vs (b) with magnetic stirring at 7Hz. [31] .	21
2.10	Velocity of electrons as a function of accelerating voltage.[35]	22
2.11	Dissimilar metal welding using electron beam. [39]	24

2.12	(a) Hump formation during welding as a function of welding speed [50] (b) Process map of humping as a function of welding speed and power [50] and (c) Melt ball formation in additive manufacturing [49].	26
2.13	Comparison of Langmuir's model and Mundra et al's model. [67]	28
2.14	Key-hole mode in AM: Normalized melt pool depth as a function of normalized enthalpy. [70]	29
2.15	EBSD of Arcam [®] fabricated IN718 (a) XY plane and (b) XZ plane [75]. . .	30
2.16	EBSD of plane along the build direction (XZ) of Arcam [®] fabricated IN718 with hatch spacing and velocity of (a) 150 μm , 2.2 m/s and (b) 37.5 μm , 8.8 m/s [77].	31
2.17	Crystallographic Texture Engineering by Modification of Melt Pattern [78] .	31
3.1	(a) Speed up ratio and (b) Scaling Efficiency of Truchas (Heat transfer and phase change)	43
3.2	Transient Liquid-Solid interface tracking of melt pool (a) at the beginning of solidification 10ms (b) 20 ms, (c) 26 ms, and (d) end of solidification 30 ms.	45
3.3	Overlap of Thermal Gradient vector and Liquidus isotherm (1610 K) along the XZ plane.	45
3.4	Variation of G and R as a function of solidification time	46
4.1	Types of raster melt-scan (a) unidirectional (b) bidirectional.	48
4.2	Proposed Melt Scan Strategy (a) Partitioning of melt area into subdomains (b) Distribution of spots within one subdomain.	50
4.3	Top view of fabricated samples with internal point offset of (1)200 μm (2)400 μm (3)600 μm (4)800 μm	52
4.4	The top surface of fabricated samples showing the variation in met-pool shape as a function of internal point offset (1)200 μm (2)400 μm (3)600 μm (4)800 μm	52
4.5	Size and shape of the top surface of the simulated met-pool as a function of internal point offset (a)200 μm (b)400 μm (c)600 μm (d)800 μm	53
4.6	Experimentally Measured Temperature Profile of the bottom of the substrate during the build.	56

4.7	Experimentally Measured Temperature Profile during melting of the first layer.	57
4.8	Visible dendritic morphology on top layers ($\sim 500\text{ }\mu\text{m}$) of the sample with internal point offset of $200\text{ }\mu\text{m}$.	58
4.9	Variation (along XZ plane of melt pool) of PDAS predicted by the (a) Trivedi and (b) Kurz-Fisher model.	59
4.10	Variation of PDAS (Kurz-Fisher model) as a function of G and R on the reference solidification map for the internal point offset of $200\text{ }\mu\text{m}$.	60
4.11	Comparison of numerically predicted and experimentally measured PDAS.	61
4.12	Variation in grain size and corresponding qualitative texture plot as a function of internal point offset.	64
4.13	Variation of experimentally measured grain size and area as a function of internal point offset in the proposed melt strategy.	65
4.14	Distribution of dendritic growth direction of the bottom $50\text{ }\mu\text{m}$ of the melt pool.	66
4.15	Variation of cooling rate with respect to internal point offset.	67
4.16	Evidence for heterogeneous nucleation by changing the scan strategy along the build direction.	69
5.1	G vs. R plotted on reference solidification map [97] for the simulation with beam current of 20 mA and ON time of 1 ms .	74
5.2	Variation of temperature gradient (G) and liquid-solid interface velocity (R) of spot melt profile as a function of (a) preheat temperature, (b) spot ON time, (c) beam diameter, and (d) spot beam current.	75
5.3	Change in volume fraction of equiaxed grains (Φ) or solidification morphology with respect to different input parameters.	78
5.4	Qualitative comparison of (a) raster scan and (b) spot melting strategy	82
5.5	(a) Top view of samples fabricated using spot melting strategy with varying beam current and beam ON time (b) Energy deposited (kJ) per layer of samples	82

5.6	EBSD: Inverse pole figure of samples (XZ plane) fabricated with beam current of 20 mA, beam ON time of 1 ms and focus offset coil current of (a) 0 mA (b) 20 mA. Grain aspect ratio of sample built with focus offset coil current of (c) 0 mA and (d) 20 mA	84
5.7	Melt pool of samples fabricated with beam current of (a) 10 mA (b) 20 mA with beam ON time of 0.25 ms	85
5.8	EBSD: (a) Inverse pole figure of samples (XZ plane) fabricated with beam current of 10 mA and (b) 20 mA (c) Grain aspect ratio of sample built with 10 mA and (d) 20 mA with beam ON time of 0.25 ms	86
6.1	Qualitative spot melting strategy.	89
6.2	(a) Schematic of the build showing part geometry and corresponding energy density (MJ/sq.m) (b) Top view of the fabricated component	90
6.3	Top surface of the parts showing the melt pools and corresponding grain morphology along the build direction.	92
6.4	EBSD and corresponding texture plot of (a) part 5 and (b) part 6 along the plane perpendicular (XY plane) to the build direction.	93
6.5	A Demonstration component with hybrid microstructure	94
6.6	Schematic and sequence of tile melt strategy.	95
6.7	Top view of fabricated samples.	96
6.8	EBSD of samples	97
6.9	Flowchart describing the algorithm to identify the state of a pixel and calculate spatiotemporal G & R data (recreated based on Raplee et al. [121].)	102
6.10	Regional slope of (a) unmelted and (b) melted pixel. [121].	103
6.11	G and R calculated from IR data for (a) sample 1 (b) sample 2.	103
6.12	Surface topology of (a) sample 1 and (b) sample 2.	104
6.13	G and R overlay on reference solidification map [97] of IN718 for (a) sample 1 and (b) sample 2.	105
6.14	EBSD and aspect ratio map of (a) top region and (b) bottom region of sample 2.	106

7.1	(a) Variation of surface tension with temperature (b) Top surface flow (c) Side view of the flow pattern	110
7.2	Surface Tension as a function of temperature for IN718 with different surfactant concentration [133]	111
7.3	Thermal gradient G (a) Pure Heat transfer (b) Fluid flow (Low Surfactant) (c) Fluid flow (High Surfactant)	112
7.4	Comparison of fluid flow pattern for low and high surfactant concentration at (a) 3 ms (b) 3.5 ms (c) 4.5 ms	113
7.5	Thermal gradient G (a) Pure Heat transfer (b) Fluid flow (Low Surfactant) (c) Fluid flow (High Surfactant)	113
7.6	Comparison of fluid flow pattern for low and high surfactant concentration at (a) 4 ms (b) 5 ms (c) 8 ms	114
7.7	Comparison of Peclet number for low and high surfactant concentration with preheat of 25°C at (a) 3 ms (b) 3.5 ms (c) 4.5 ms	115
7.8	Comparison of Peclet for low and high surfactant concentration with preheat of 1000°C at (a) 4 ms (b) 5 ms (c) 8 ms	116
7.9	Comparison of shape of simulated melt pool with internal point offset of 200 μm (a) Pure heat transfer - top surface (XY plane) (b) Pure heat transfer - build direction (XZ plane) (c) heat transfer plus fluid flow (high surfactant) - top surface (XY plane) (d) heat transfer plus fluid flow (high surfactant) - build direction (XZ plane)	117
7.10	Melt pool with internal point offset of 200 μm (a) Top surface (XY plane) (b) Build direction (XZ plane)	118
7.11	Comparison of shape of simulated melt pool with internal point offset of 800 μm (a) Pure heat transfer - top surface (XY plane) (b) Pure heat transfer - build direction (XZ plane) (c) heat transfer plus fluid flow (high surfactant) - top surface (XY plane) (d) heat transfer plus fluid flow (high surfactant) - build direction (XZ plane)	119
7.12	Melt pool with internal point offset of 800 μm (a) Top surface (XY plane) (b) Build direction (XZ plane)	119

8.1	Sequence of fill in an airfoil (a) Raster fill (b) Dehoff fill or spot fill.	124
8.2	G vs R for the raster fill and "Dehoff" fill	125
8.3	(a) Variation in transient nature as a function of build height (b) Matrix format of complex geometry (triangle) without overhang (c) with overhang.	127
8.4	Integrated Computational and Machine Learning Framework to predict process-structure-property in AM process.	127

Chapter 1

Introduction

1.1 Introduction to Additive Manufacturing Process

Additive manufacturing (AM), commonly referred to as rapid prototyping, is the fabrication of 3-D parts by additively fusing one layer of raw material over the previously fused layers. An important advantage of AM over conventional processes is that it enables fabrication of complex structures which are either impossible or cost prohibitive to manufacture through traditional methods like milling and machining. New additive manufacturing (AM) technologies are revolutionizing the manufacturing sector with a significant reduction in manufacturing lead time, material wastage, and energy consumption. They are being adapted to fabricate critical components in aerospace, automobile, defense and medical industries[1]. In aerospace sector, fabrication of functional parts using AM dramatically reduces the buy-to-fly ratio compared to conventional subtractive manufacturing processes like machining, where the desired shape and size of the part is obtained by removing the excess material. Metal AM systems can be classified based on the type of raw material and energy source used in the process to melt the raw material. The majority of metal AM systems fall into two main classifications; powder bed fusion and directed energy deposition. For powder bed fusion systems the starting material is metal powder in contrast to directed energy technologies where the starting material may be either powder or wire. Lasers and electron beams are the two most commonly used heat sources to selectively melt the metal powder particles in the bed and fuse them to the underlying layers. Thin metal sheets

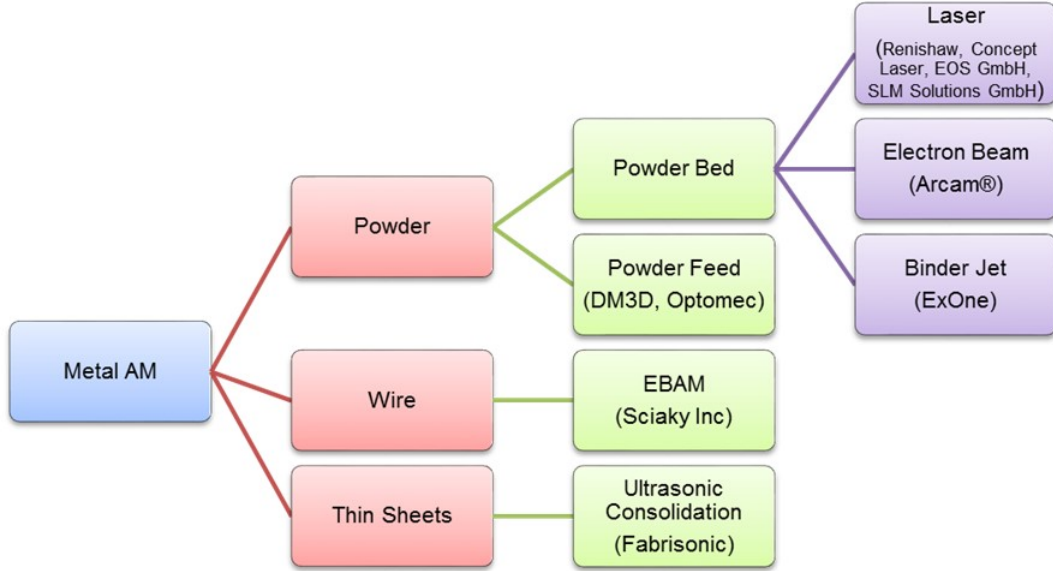


Figure 1.1: Classification of metal AM systems

are also used as raw materials and are bonded layer by layer using fast moving sonotrode. Classification of metal AM systems is shown in Figure 1.1. An electron beam additive process (Arcam[®]) is the focus of this research work.

1.1.1 Business and Environmental Implications of Additive Manufacturing Technology

Conventional manufacturing processes generally involve multiple steps to manufacture a part and number of steps may significantly increase with increase in the complexity of the design of the part. The complexity of the part is also limited by the ability of the conventional manufacturing processes. On the other hand, additive manufacturing is a single step process to manufacture a part however complex it may be. Additive manufacturing eliminates multiple steps involved in the conventional manufacturing process like machining. In the

global market for manufacturing sector, it is important to remain competitive in terms of quality and cost. If wisely chosen, additive manufacturing will help in the reduction of overall product cost by reducing the material wastage, processing cost, energy cost, inventory cost and transportation cost involved during various stages of conventional manufacturing.

Looking at the environmental perspective, AM helps in reducing the carbon footprint of a manufactured part. A case study [2] on a real world application of a titanium part in aerospace industry compared the amount of greenhouse gas emissions due to manufacturing of the part by AM and conventional manufacturing through milling. Using AM technology to manufacture that particular component, GHG emissions was estimated to be 49% less compared to conventional manufacturing process. Once the engineering and metallurgical challenges in various additive manufacturing processes are addressed, AM is claimed to play vital role in reducing greenhouse emissions of next generation smart industries. Figure 1.2 shows the results of detailed investigation [3] on comparison of energy consumption during various stages (raw material preparation phase and part fabrication phase) by AM and injection molding (IM) of 500,000 units of a part. It can be observed that AM uses comparatively less energy during raw material phase and significantly more energy during production phase. It was concluded that for the given case, there is a threshold number of production units below which AM is advantageous and above which conventional IM is profitable in terms of energy and economy. Apart from addressing current engineering challenges in the field additive manufacturing, it is important to study the various other factors involved including market demand situation, inventory costs and other related factors in the entire production chain of a particular component in order for successful production scale implementation of AM to manufacture that component.

During the early stages, additive manufacturing was used to make prototypes, models and for tooling applications. In recent years, it has found the way into large scale manufacturing sector, particularly in aerospace and medical implant industries. Aerospace and medical implant industries are showing great interest in additive manufacturing due to the advantage of reduction in material wastage of expensive raw materials, reduction in manufacturing lead-time and design freedom. In aerospace industries, reduction in material wastage of

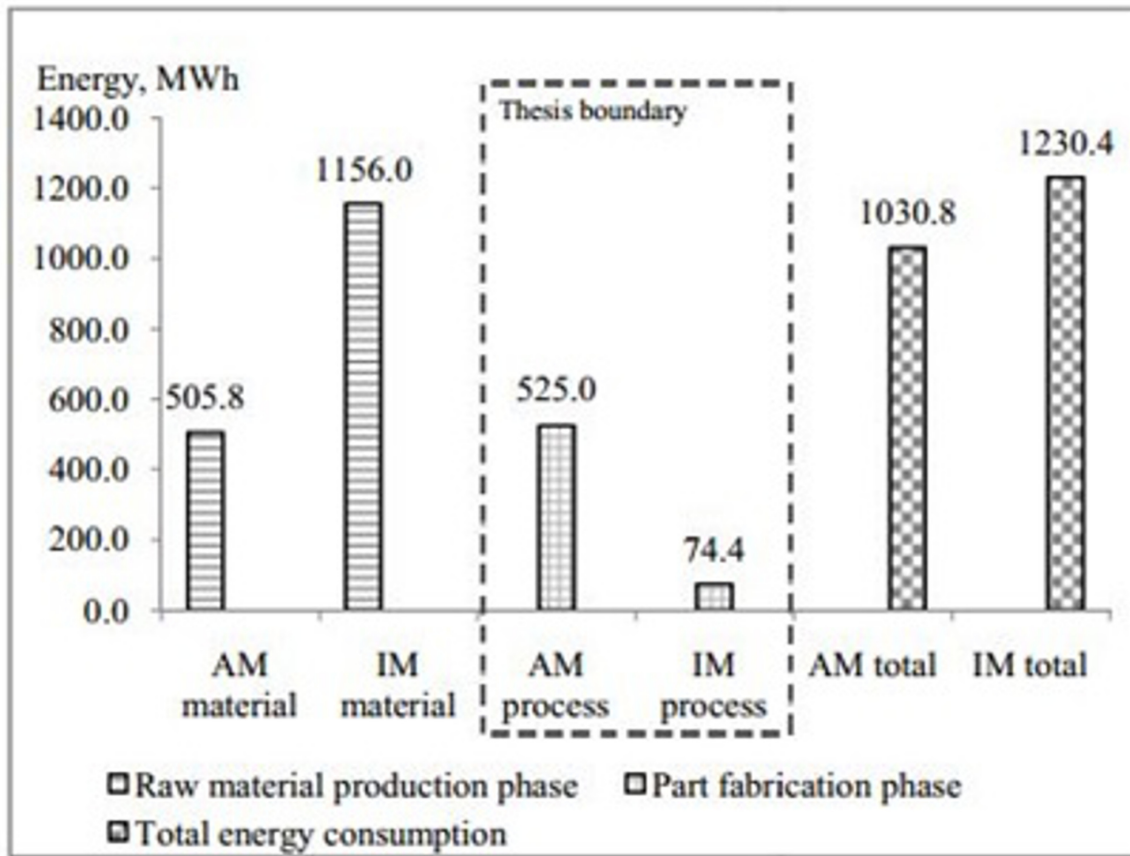


Figure 1.2: Energy Usage Comparison between AM and IM [3]

expensive raw materials like titanium and nickel-base alloys will reduce the buy-to-fly ratio of the components. Design freedom allows the aerospace industry to replace the heavy weight conventional design of the parts with low weight optimized design.

Industrialization of Metal AM

GE Aviation is one of the pioneers in transforming additive manufacturing from prototype level process to pilot-line production scale process. GE Aviation is considering utilizing the laser sintering process to additively manufacture turbine blades with titanium aluminide for next generation engine GE9X for the new Boeing 777X. According to GE, GE9X is the most advanced fuel-efficient commercial aircraft engine ever built. Currently, GE Aviation is additively manufacturing fuel nozzle using direct metal laser melting (DMLM) process for its upcoming LEAP engine with each engine requiring 19 fuel nozzles in its combustion section. With more than 6700 orders till now from 20 countries, adding up to nearly \$96 billion (U.S. list price), the LEAP is GE Aviation's best-selling engine in its history. According to a recent report from GE [4], benefits of the new additively manufactured fuel nozzle compared to the previous generation are

- Consolidation of 20 parts into one additively manufactured component
- 5x life improvement of overall fuel delivery system
- 25% reduction in weight of the part

Figure 1.3 shows the above mentioned fuel nozzle and also the world's first additively manufactured component to fly in a commercial jet engine.

1.1.2 A Brief Introduction to Arcam[®] process

In an electron beam additive system, fine metal powders (30 μm -100 μm) stored in the powder hopper are raked over the start plate using a metallic brush. The layer thickness is usually in the range of 50 μm to 100 μm . The entire layer of powder is preheated and sintered using diffused electron beam to avoid surface charge accumulation on the powder particles and 'smoking'[6] during melting. The powder particles are then melted using the electron beam



Figure 1.3: Production scale additively manufactured fuel nozzle from GE Aviation [5]

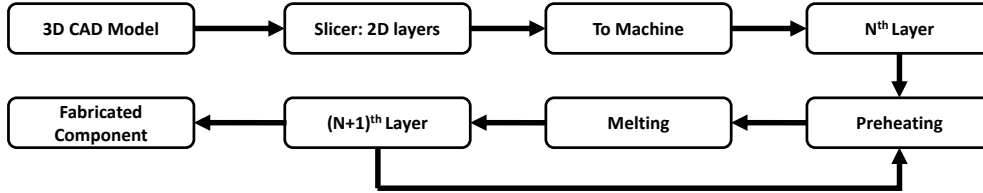


Figure 1.4: Processing steps in Arcam®

according to the 2D design of the particular layer and fused with the previous layers. The build platform is then lowered and new layer of powder is raked and melted. This process continues until melting of all the layers of the part is complete. The component is then taken out of the build chamber and powder recovery system is used to recover the unmelted powder particles. Figure 1.4 shows the processing steps involved in the Arcam® process.

Electron beam based processes have significantly higher power density [7] than laser based processes, and also, the electron beam based Arcam® process has comparatively faster part fabrication rate than laser based processes. The difference in part fabrication rate is attributed to difference in beam deflection mechanisms. Mechanical inertia experienced by the deflecting mirrors limits the travel speed of the laser beam while electron beam can travel much faster (8000 m/sec)[8] owing to the use of electromagnetic deflection coils. Parts fabricated using the electron beam process also tend to have significantly lower residual

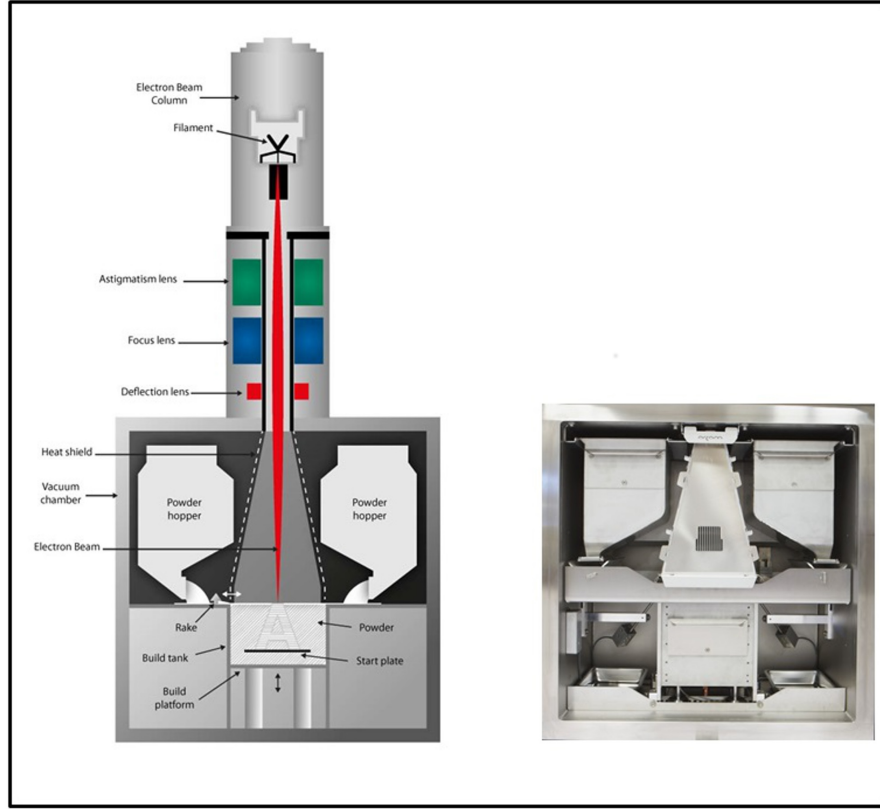


Figure 1.5: Schematic of an Arcam[®] machine and corresponding picture of the build chamber [10]

stresses [9] compared to parts manufactured using laser based fabrication processes. Even though AM is a layer by layer fabrication technique, the underlying physics is the same as welding, but for the complex boundary conditions and type of raw material typical to each AM process. Figure 1.5 shows the schematic of an Arcam[®] machine.

1.1.3 Alloys used in powder bed AM processes

Aerospace and medical implant industries are couple of the major sectors adapting additive manufacturing for production scale. In addition to design freedom, to extract the economical advantage of AM process through reduction of the buy-to-fly ratio, the raw material used for fabrication have to expensive. Couple of the most commonly used alloys that are expensive in aerospace and medical implant industries are nickel-base superalloys and titanium alloys.

In general, nickel-base superalloys constitute about 40-50% of the the total weight of the

aircraft engine [11]. In addition to corrosion resistance at high temperature, they exhibit high strength, toughness, and creep resistance at high temperatures. Some of the nickel alloys can withstand temperatures upto 1200°C [11] ($\sim 90\%$ of the melting point). Due to this, the nickel alloys are used in hot section of a turbine engine. The high temperature properties are owed to precipitation strengthening (γ' - Ni_3Al and γ'' - Ni_3Nb) of the alloy system. Inconel 625 (solid solution strengthened) and Inconel 718 (γ'' strengthened) are couple of the most commonly and successfully used nickel-based superalloys in AM processes. AM fabrication of high γ' nickel-alloys are challenging because of it's non-weldability owing to their sensitivity to cracking. Numerous research works [12, 13] are trying to address the challenges for successful fabrication of non-weldable nickel-base alloys using AM.

On the other hand, due to their high strength to weight ratio, titanium alloys are used in cold section of turbine engine (low pressure compressor section) and for structural applications in replacing low alloy steel and aluminum alloys [14]. Due to it's excellent bio-compatibility, titanium alloys are extensively used in dental and orthopedic implants [15, 16, 17]. Ability of fabricating patient specific designs and complex mesh structures has enabled the foray of AM in this field. Among titanium alloys, Ti6Al4V is the most commonly used in metal AM processes. There has been considerable research and development activities on replacing some heavier nickel-base superalloy components with lighter titanium aluminide alloy (γ -Ti-Al-based alloy) for high temperature applications [18, 19]. Some of the recent studies [20, 21] have evaluated the feasibility of fabricating γ -Ti-Al-based alloys using additive manufacturing.

1.2 Factors Affecting Mechanical Performance of Engineering Components

Lifetime and performance of any fabricated component depend on its mechanical properties. In general, the mechanical properties (fatigue, yield strength, ultimate tensile strength, ductility, and creep) of an engineering component are primarily dictated by (a) design geometry, (b) surface roughness, and (c) microstructure of the component. Usually, the

geometry and surface roughness play a role during crack initiation, while crack propagation is predominantly dictated by the underlying microstructure. In this process-structure-property-performance (PSPP) linkage, understanding the process-structure relationship is important to obtain desired mechanical performance of the part fabricated through AM.

The microstructure of the fabricated component can be controlled by understanding the (a) liquid-solid and (b) solid-solid phase transformations of the alloy system with respect to complex thermal histories, which is in turn affected by the processing conditions. In the electron beam additive process (Arcam[®]) discussed in this work, depending on the material system and processing conditions, both types of phase transformations can occur simultaneously in different locations depending upon spatiotemporal variations of temperature. Depending on the alloying elements and processing conditions, an alloy can have different crystal structures (FCC, BCC, HCP, etc.,) and multiple crystal structures can coexist at the final stage which dictates the ductility, tensile and other mechanical properties of the alloy system. Formation of precipitates is also dictated during the solid-solid phase transition. Understanding this phenomenon helps in pinning the dislocation movement thereby increasing the lifetime of the material. This phenomenon is well understood and Time-Temperature-Transformation (TTT) diagram is available in literature [22, 23, 24] for most commonly used alloy systems. In addition to JMAK and STK models, software like Thermo-Calc can be used to understand the solid-solid phase transitions with respect to the alloy compositions.

The primary focus of this research work is to understand the process-structure relationship by analyzing the transient behavior of liquid-solid phase transformation (solidification) as a function of process parameters.

Chapter 2

Literature Review and Scientific Gaps

2.1 Literature Review

2.1.1 Fundamentals of Solidification

To study the feasibility of grain size and grain morphology control in metal additive manufacturing, it is vital to understand the fundamental concepts of the solidification phenomenon. Two key aspects of the solidification process to be understood are

- Nucleation phenomenon
- Growth mechanism

Some of the fundamental concepts of the solidification process discussed in this section can be referred to the literature [25] and books by Kurz-Fisher [26] and Dantzig-Rappaz [27].

Nucleation

There are 2 types of nucleation that will happen within the liquid.

- Homogeneous nucleation
- Heterogeneous nucleation

Thermodynamics of homogeneous nucleation: Homogeneous nucleation occurs within the liquid melt without the help of any inclusions, impurities or surfaces like substrates

and walls during casting. The Gibbs free energy of the solid phase (G_s^m) is higher than the free energy of liquid phase (G_l^m) above the melting point ($T > T_m$). According to thermodynamics, low energy phase is favored over the higher energy phase. Hence it is not energetically favorable for the solid phase to exist above melting point. In melt at higher temperatures, the atoms are disordered with high mobility. As the temperature decreases below the equilibrium melting point (T_m), the atoms join together form clusters forming short range order over few atomic radii. As the temperature is further decreased, some of the clusters can overcome the energy barrier to form a nuclei of structured solid phase within the liquid phase. Other clusters which can not overcome the energy barrier dissolve back into liquid phase. To quantify it, free energy change of the cluster has to be calculated. Let us assume the geometry of the clusters to be spherical with radius R . When a cluster is formed, there are 2 energies associated with it (a) volumetric energy and (b) interfacial surface energy between the solid cluster and liquid. Hence the total free energy change is the sum of the change in volumetric free energy term and surface energy term. Hence change in free energy of the cluster is given by Equation (2.1).

$$\Delta G = V * \Delta g_v + A * \gamma_{sl} \quad (2.1)$$

where ΔG is the free energy change, V is the volume of the cluster, Δg_v is the volumetric free energy change per unit volume, A is the surface area of the cluster and γ_{sl} is the interfacial surface energy per unit area of the cluster. Since the cluster is assumed to be spherical, the surface area and volume of the cluster can be calculated as a function of it's radius. For $T < T_m$, when the clusters begin to form in the liquid, the difference in temperature ($\Delta T = T - T_m$) is termed as undercooling. The volumetric free energy can be expressed as a function of entropy change ($\Delta g_v = \rho * \Delta s * \Delta T$). Hence, the total free energy change of the system in Equation (2.1) can be derived as

$$\Delta G = -\frac{4}{3}\pi R^3 \rho \Delta s_f \Delta T + 4\pi R^2 \gamma_{sl} \quad (2.2)$$

From Equation (2.2), it can be observed that the volumetric energy term ($\propto R^3$) reduces the overall free energy of the system and the surface energy term ($\propto R^2$) increases the overall

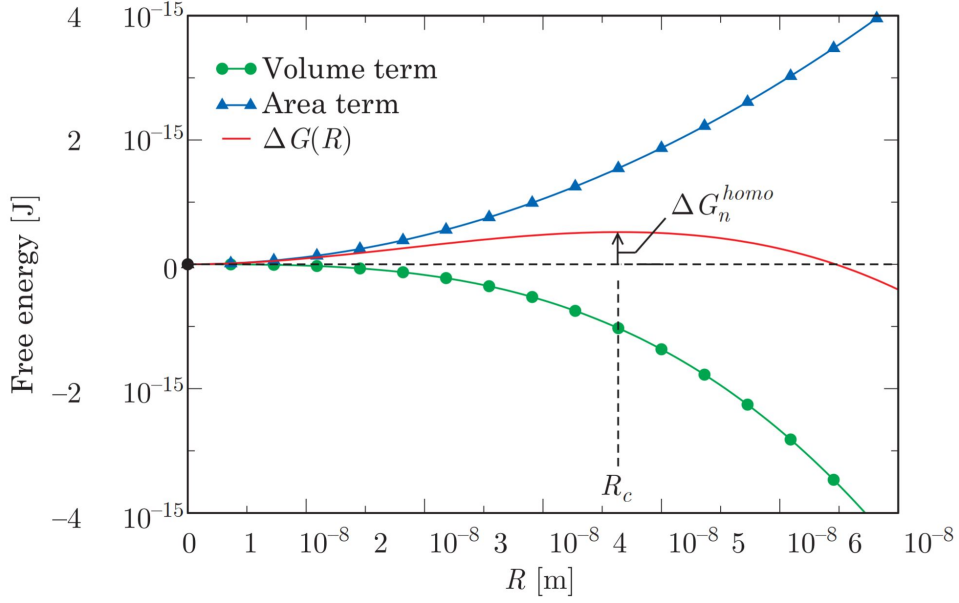


Figure 2.1: Free energy change during homogeneous nucleation of Aluminum [27]

energy. Hence, depending on the radius of the cluster, the dominance of the terms changes. Hence, the radius required to overcome the energy barrier has to be calculated by taking the derivative of Equation (2.2) with respect to the radius and equating it to zero. The critical radius ($R > R_{critical}^{hom}$) for the cluster to avoid remelting and form a homogeneous nucleus is defined as

$$R_{critical}^{hom} = \frac{2\gamma_{sl}}{\rho\Delta s\Delta T} \quad (2.3)$$

The term $\gamma_{sl}/\rho\Delta s$ is called as the Gibbs-Thompson co-efficient Γ_{sl} . From Equation (2.3) and Equation (2.2), the critical free energy change required for heterogeneous nucleation can be defined as

$$\Delta G_{critical}^{hom} = \frac{16\pi\gamma_{sl}^3}{3\rho^2\Delta s^2\Delta T^2} \quad (2.4)$$

Figure 2.1 shows the change in volumetric free energy term, surface energy term and total free energy term as a function of radius of the cluster. Critical radius (R_c) of the cluster and homogeneous nucleation barrier (ΔG_n^{homo}) is also pointed out in Figure 2.1

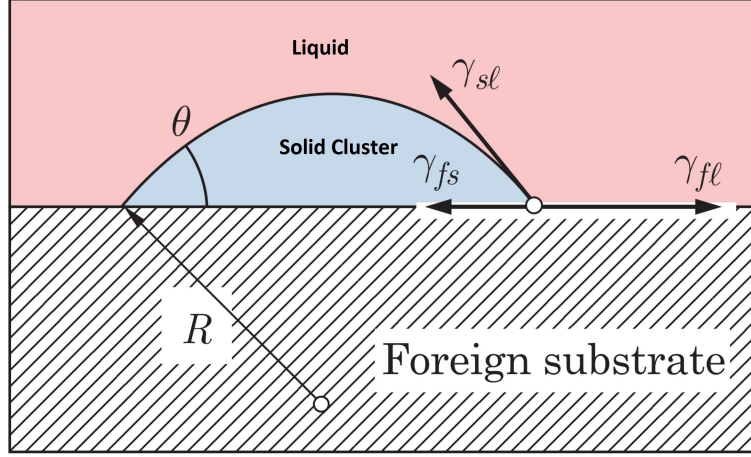


Figure 2.2: Heterogeneous nucleation on an existing wall or substrate [27]

Homogeneous nucleation is an ideal scenario and can be observed in pure metals when extreme care is taken to avoid any impurities.

Thermodynamics of heterogeneous nucleation: Heterogeneous nucleation happens on surfaces of the containers or intentionally added inclusions or unintentional impurities within the liquid melt. For rapid solidification conditions during welding/additive manufacturing and solidification of multicomponent alloy systems, the nucleation is almost always heterogeneous in nature.

Figure 2.2 shows the formation of cluster on an existing foreign body (substrate) and corresponding surface energy term associated with different interfaces (γ_{sl} : solid & liquid, γ_{fl} : foreign body & liquid, γ_{fs} : foreign body & solid). Θ is termed as the wetting angle.

Because of the presence of additional interface, the Gibbs free energy change for heterogeneous nucleation can be formulated similar to Equation (2.1).

$$\Delta G = V * \Delta g_v + A_{sl} * \gamma_{sl} + A_{fs} * (\gamma_{fs} - \gamma_{fl}) \quad (2.5)$$

The area and volume of the spherical cap can be formulated as a function of wetting angle (Θ) and the radius (R). Simplifying the Equation (2.5) by substituting the volume and area terms, the equation of free energy change of heterogeneous nucleation is given by

Equation (2.6)

$$\Delta G = \left(-\frac{4}{3}\pi R^3 \rho \Delta s_f \Delta T + 4\pi R^2 \gamma_{sl} \right) f(\Theta) \quad (2.6)$$

Similar to Equation (2.3) and Equation (2.4), the critical radius and critical free energy change for heterogeneous nucleation can be defined as

$$R_{critical}^{het} = \frac{2\gamma_{sl}}{\rho \Delta s \Delta T} \quad (2.7)$$

$$\Delta G_{critical}^{het} = \left(\frac{16\pi \gamma_{sl}^3}{3\rho^2 \Delta s^2 \Delta T^2} \right) f(\Theta) \quad (2.8)$$

where,

$$f(\Theta) = \frac{(2 + \cos \Theta)(1 - \cos \Theta)^2}{4} \quad (2.9)$$

Equation (2.8) and Equation (2.4), it can be noted that when $f(\Theta) = 1$, i.e, the spherical cap is completely spherical ($\Theta = 180$), the energy barrier for heterogeneous nucleation is equal to the energy barrier for homogeneous nucleation. When $f(\Theta) = 0$ or complete wetting ($\Theta = 0$), the energy barrier for heterogeneous nucleation is zero. Heterogeneous nucleation energy barrier is always less than or equal to the homogeneous nucleation energy barrier. Figure 2.3 compares the energy barrier for homogeneous, heterogeneous nucleation and epitaxial growth. It can be observed that the epitaxial growth has no energy barrier to grow from the existing substrate of the same material (example: growth from fragmented dendrites).

Undercooling: The term ΔT in Equation (2.8) and Equation (2.4) is termed as undercooling or supercooling. For a give composition of an alloy system, amount of undercooling plays crucial role for nucleation. Higher the undercooling, easier the nucleation. It is the sum of 4 major types of undercooling $\Delta T = \Delta T_C + \Delta T_{th} + \Delta T_k + \Delta T_{curv}$.

- **Thermal Undercooling (ΔT_{th}) :** Undercooling the liquid bath below the melting point for nucleation. This type of undercooling plays vital role only when nucleation due to other means are difficult. For rapid solidification conditions, this is negligible.

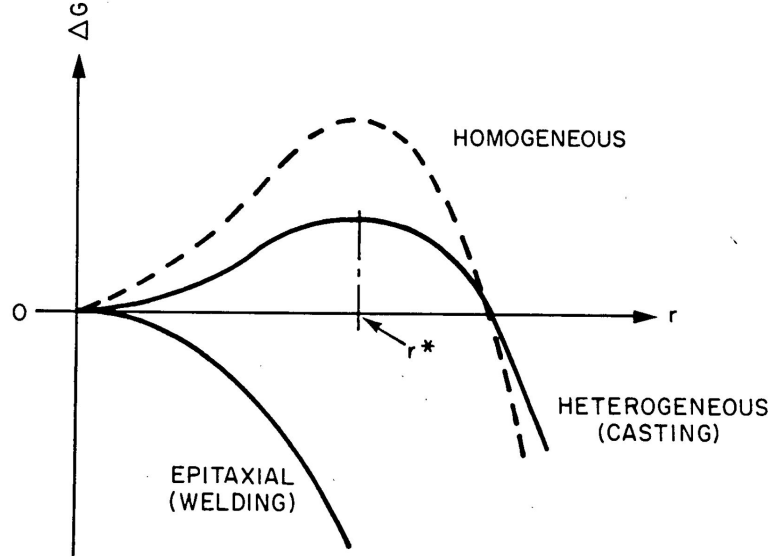


Figure 2.3: Qualitative Comparison of free energy change required for homogeneous, heterogeneous nucleation and epitaxial growth. [25]

- Kinetic Undercooling (ΔT_k) : This is directly proportional to the rate of attachment of atoms to form nucleus. For all metal solidification processes, this value is comparatively smaller ($<1K$).
- Curvature Undercooling (ΔT_{curv}) : When there is curvature at the liquid-solid interface, the surface energy term in the energy barrier changes with respect to the curvature. The undercooling associated with that is called curvature undercooling.
- Constitutional Undercooling (ΔT_C) : During solidification, rejection of solutes at the liquid-solid interface reduces the freezing point of the enriched region due to difference in composition. This is termed as constitutional supercooling. This plays significant role in determining the morphology during rapid solidification of alloy systems and is explained in the following section.

Growth

Growth is followed by nucleation. Depending on different solidification conditions, the growth will result in different grain morphology . Figure 2.4 shows a schematic of different solidification morphology as a function of processing conditions namely

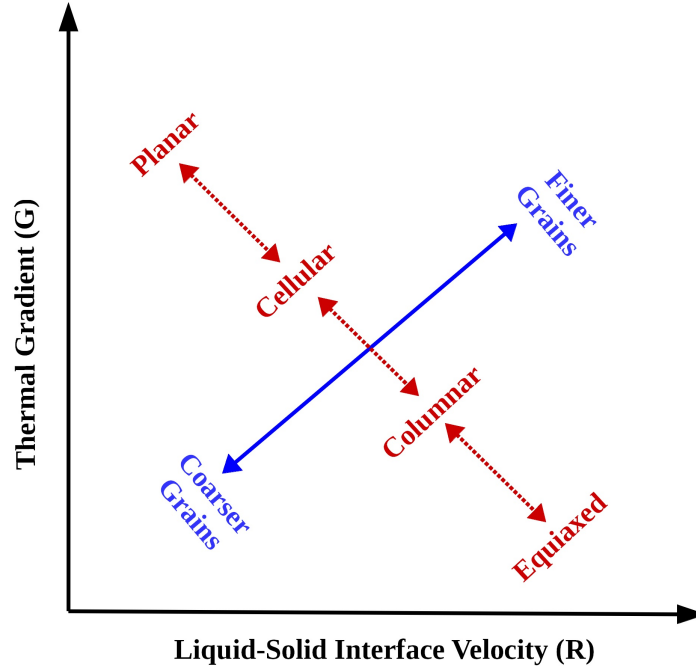


Figure 2.4: Variation of solidification morphology as a function of G and R

- Thermal Gradient at the liquid-solid interface : G
- Velocity of the liquid-solid interface : R

Effect of constitutional supercooling is significant in determining the stability of the interface and nucleation ahead of the interface. These two factors (stability and nucleation) are significant in dictating the solidification grain morphology.

To understand constitutional supercooling, non-equilibrium (no diffusion in solid) solidification aspect of the phase diagram has to be understood. Rapid solidification during welding and additive manufacturing is non-equilibrium in nature. Figure 2.5 (a) shows the schematic of a phase diagram of an alloy with equilibrium solute concentration of C_o . As the temperature drops below the liquidus, the absence of diffusion in solid results in rejection of solute to the liquid at the interface. Increase in concentration of the solute at the interface is schematically shown in Figure 2.5 (b). From Figure 2.5 (a), it can be observed that increasing the concentration of the solute reduces the liquidus temperature locally.

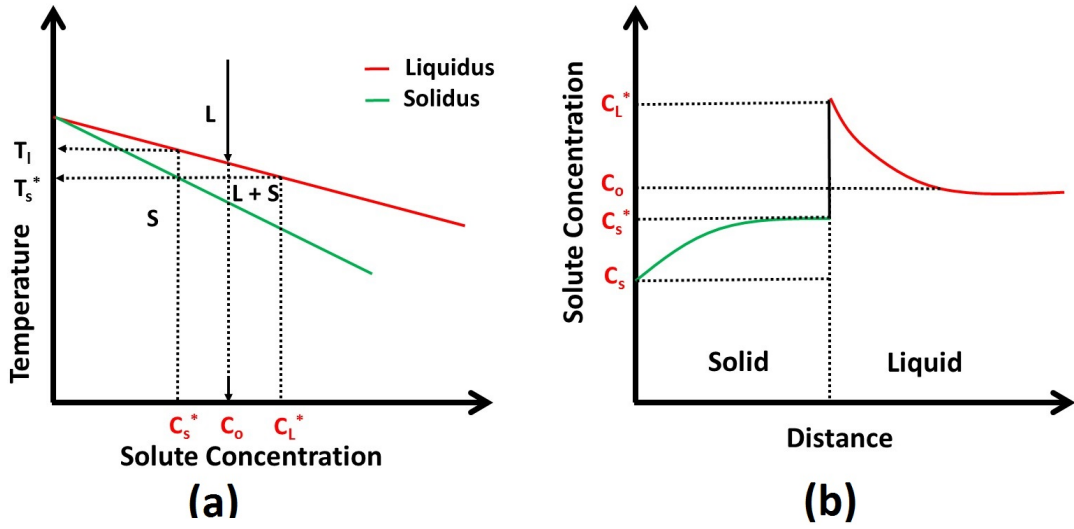


Figure 2.5: (a) Phase Diagram with respect to solute concentration
(b) Solute enrichment at the interface

Figure 2.6 (b) shows the schematic of change in liquidus temperature as a function of distance from the liquid-solid interface. The slope of the curve is the thermal gradient at the interface. If the processing thermal gradient (actual thermal gradient) is higher than the theoretical thermal gradient, then there will be no constitutional supercooling. If the processing thermal gradient is controlled and reduced, there will be undercooled region ahead of the solidification front. The region can be measured by the area of the curve overlapping with the processing thermal gradient.

Based on processing conditions, the condition for constitutional super cooling can be derived with respect to alloy system. The criteria for constitutional supercooling to occur is

$$\frac{G}{R} < \frac{T_l - T_s}{D_L} \quad (2.10)$$

where G is thermal gradient at the interface, R is the velocity of the interface, T_l and T_s is the liquidus and solidus of the alloy system, D_L is the diffusion co-efficient of the the solute in the liquid.

There are 2 ways in which constitutional supercooling can be introduced in the melt

- By changing the processing conditions affecting solidification.

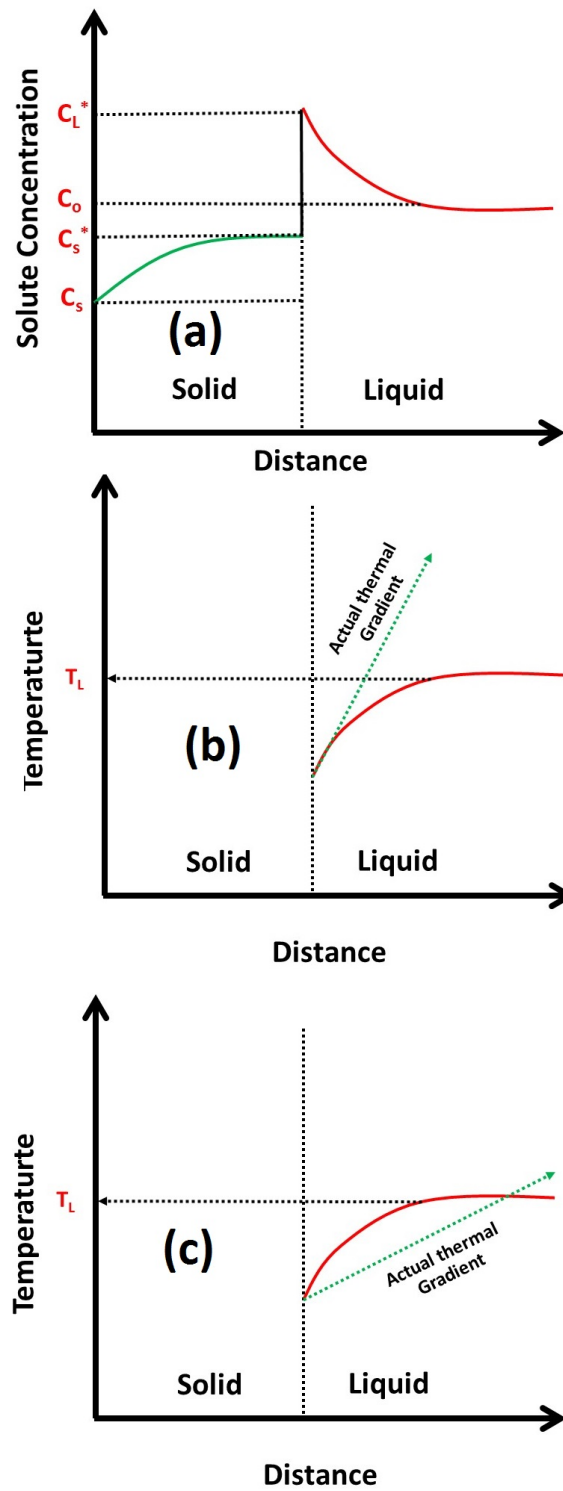


Figure 2.6: (a) Solute enrichment (b) No constitutional undercooling at the interface (c) Presence of constitutional undercooling

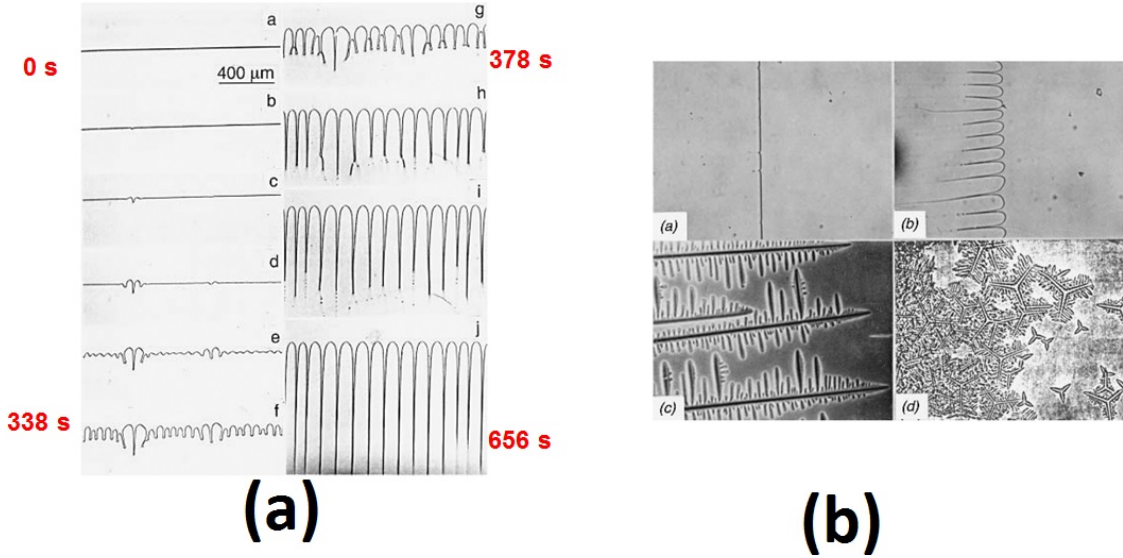


Figure 2.7: (a) Breakdown of liquid-solid interface due to solute rejection as a function of time (b) Change in morphology by introducing impurity [28]

- By changing the composition of the alloy.

Figure 2.7 (a) shows the breakdown of solid-liquid interface (from planar to cellular due to solute segregation) of a metal analog system (pivolic acid) as a function of time. Figure 2.7 (b) shows the variation in solidification morphology during solidification of carbon tetrabromide through increase of constitutional supercooling by increasing the amount of impurity in the melt.

2.1.2 Evolution of Microstructure in Rapid Solidification Processes

Lessons from Welding Literature

The grains grow normal to the interface of the melt pool which is the direction of resultant thermal gradient. Hence, shape of the melt pool is crucial for the resultant microstructure. Shape of the melt pool is determined by the velocity of the welding heat source and thermal diffusivity of the material. The shape will be circular if the welding velocity is very small [28]. As the velocity is increased the shape becomes elliptical and on further increasing the velocity, it becomes tear drop shaped. In electron beam AM process, the velocity of the heat source for melting can be in the range of 4-10 m/s. In such case, it is possible for the

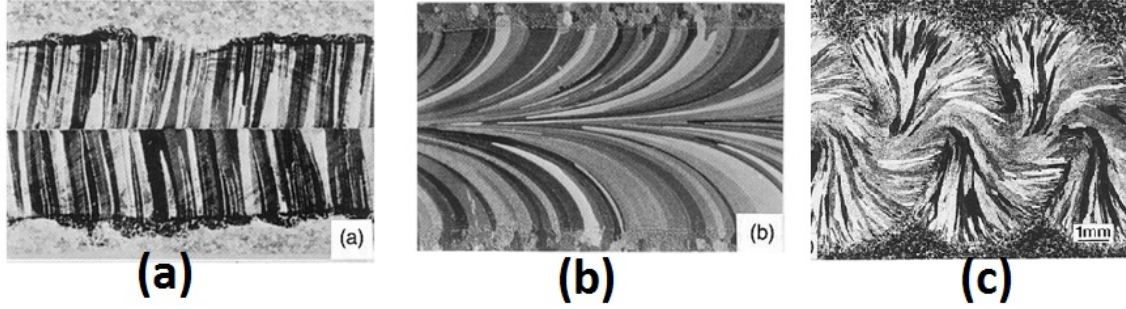


Figure 2.8: Epitaxial grain growth at (a) High velocity (b) Low velocity (c) Oscillation[28]

melt pool to be elongated such that it becomes a linear heat source. When the melt pool is elongated with high aspect ratio, the grains grow perpendicular to the direction of the melt pool. Effect of oscillation of heat source on microstructure has also been reported. Figure 2.8 shows the effect microstructure variation with respect to change in the linear speed of the welding torch and also due to oscillation of the welding torch.

To promote finer grains during weld through nucleation, different engineering approaches have been made. Kou et al [29, 28] proposed multiple strategies including dendritic fragmentation and promoting nucleation by blowing cooling gas at the top surface. Heterogeneous nucleation by adding TiB_2 particles in aluminum welds [30] is also reported. Lim et al. [31] used magnetic stirring to break down the long columnar grains. Figure 2.9 shows comparison of the EBSD data of solidification of gas tungsten arc welding (GTAW) of a nickel alloy without (Figure 2.9 (a)) and with magnetic stirring (Figure 2.9 (b)). The evaluation was done over a range of stirring frequencies (1.5 - 50 Hz). The maximum amount of misorientated grain growth was reported to be at 7 Hz. In addition to dendrite fragmentation, this might also be attributed to the spatiotemporal variation of solidification velocity and thermal gradient promoting nucleation ahead of solidification front due to constitutional supercooling. These results prove the significance of weld path or scan strategy in additive manufacturing to control the solidification microstructure.

Few of the most relevant works to this research are the study of repair of single crystal blades. Gäumann et al. [32, 33] and Vitek [34] studied the formation of stray grains during the welding of single crystal turbine blade as a function of weld input parameters. It

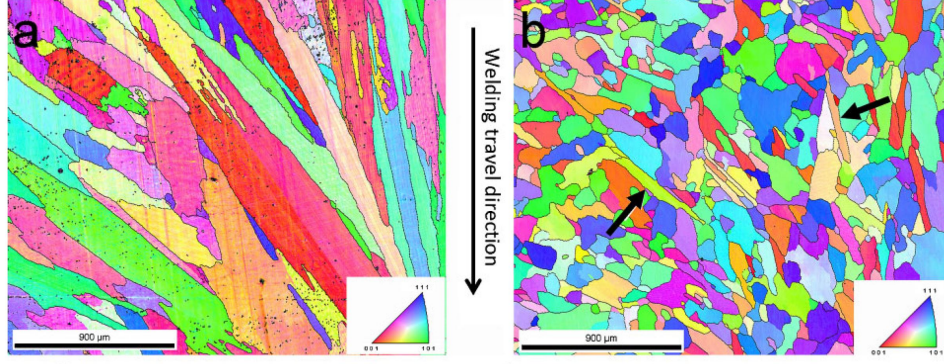


Figure 2.9: EBSD of (a) unstirred melt pool vs (b) with magnetic stirring at 7Hz. [31]

was shown that the formation of stray grains can be controlled by changing the process parameters (including weld velocity and beam diameter) which in turn controls the local solidification parameters of $\mathbf{G}$ and $\mathbf{R}$. Theoretical formulation of the relationship between the stray grain formation and solidification parameters ($\mathbf{G}, \mathbf{R}$) is given in Chapter 5.

The fundamental understanding of the correlation between the process parameters and solidification microstructure in welding can be translated to AM process.

2.1.3 Electron Beam Welding

History of usage of electron beam as heat source dates back to 1905 when Marcello von Pirani designed an electron beam furnace to melt tantalum powder [35]. In 1958, first successful electron beam welding was done on Zircaloy plates (5mm thick) [36]. One of the significant advantages of electron beam heat source compared to other heat sources is the power density. Power density of current electron beam systems is around $10^{11} - 10^{12} \text{W/m}^2$ compared to 10^9W/m^2 for arc welding processes [35]. Due to such high energy density, the electron beam welding exhibits high depth to width ratio of the melt pool and is extensively used in joining of thick plates. The power of the most powerful electron beam welder developed till now is 300kW with an accelerating voltage of 60kV which is capable of penetrating into 12 inches of steel [37].

Primary energy transfer mechanism in electron beam melting is the transfer of kinetic energy ($\frac{1}{2}mv^2$) from the beam to the substrate or material to be welded. The mass of the impinging

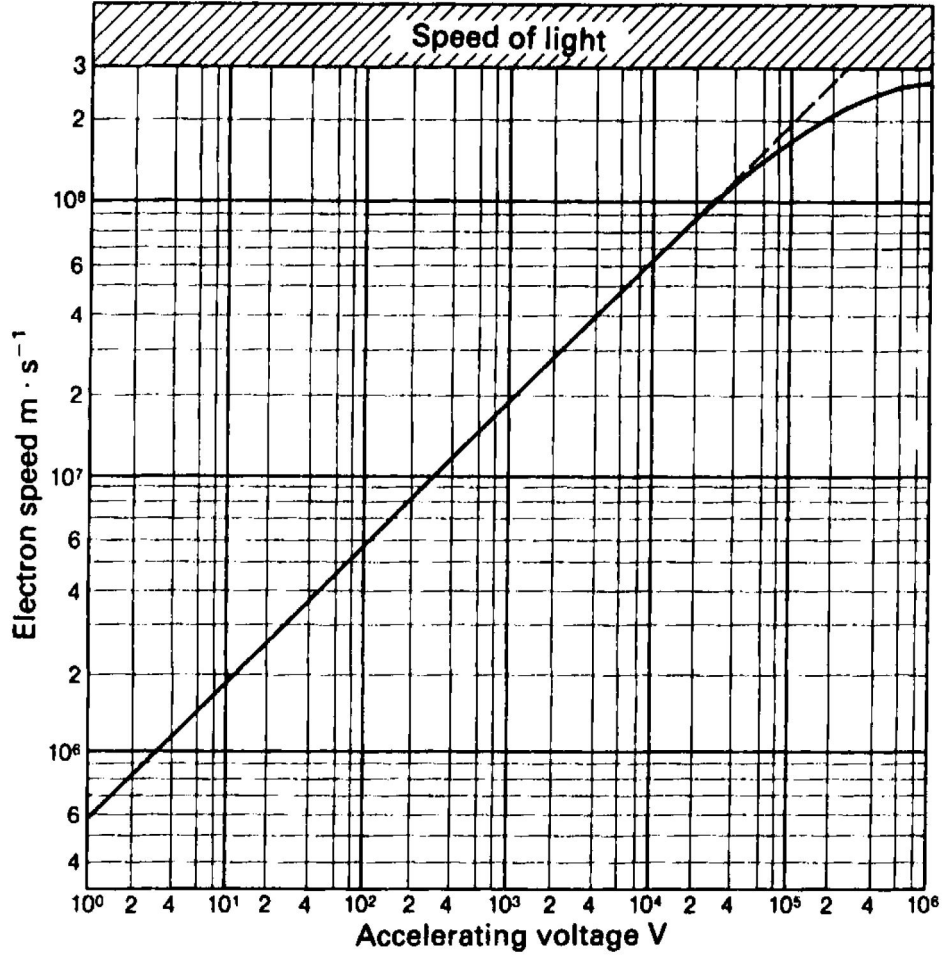


Figure 2.10: Velocity of electrons as a function of accelerating voltage.[35]

beam is decided by the current (amount of electrons) and the velocity is dictated by the accelerating voltage. At an accelerating voltage of 60kV , the electrons travel at a speed of $1.5 \times 10^8 \text{ m/s}$ [35]. At such high speeds, the mass of an electron increases by 35%. Figure 2.10 shows the variation of velocity of the electrons with respect to the accelerating voltage.

There are 4 important components in any electron beam melting systems.

- Cathode to generate electrons - made up of high melting point elements (tungsten or tantalum).
- Anode to accelerate the generated electrons.
- Focusing coils to focus the accelerated electrons.
- Electromagnetic beam deflection coils to deflect the beam to required co-ordinates.

During the impingement of electrons on the substrate, the few of the electrons undergo elastic reflection by the electrons in the substrate and are called primary electrons. Further, some conduction electrons from the substrate are ejected by the collision with the incoming electrons. the ejected electrons are termed as secondary electrons. Primary and secondary electrons are called back scattered electrons. Scattered electrons contribute to reduction of energy transfer efficiency. But, in general, the energy transfer efficiency is high (~ 0.9) in electron beam processes compared to other heat source interaction like arc and laser [38]. In addition, X-rays are emitted due to the loss in energy of the reflected primary electrons. The penetration depth of the electrons is dictated by the accelerating voltage and the density of the substrate. Comprehensive details on the application of electron beam welding in aerospace, automobile, construction and medical device fabrication is given in chapter 10 of the book "Electron Beam Welding" by Schultz [35].

Electron beam welding has been extensively used in dissimilar metal weldings as well. A review article by Sun et al. [39] describes the successful application and challenges of electron beam for dissimilar welding. Due to high energy density, electron beam welding is used to join refractory elements (Nb, Mo) with high melting point. For refractory metals like Niobium and Molybdenum, the ductile to brittle transition temperature (DBTT) is increased above the room temperature because of recrystallization and grain growth [39]. Due to fine beam radius and high cooling rate, the heat affected zone is smaller. Hence, by using electron beam welding, the brittle cracking can be avoided by reducing or avoiding the amount of recrystallization in the heat affected zone. In addition, since electron beam welding is done in vacuum or inert atmosphere, it is also used to weld reactive metals (Al, Ti alloys). Figure 2.11 shows the successful dissimilar metal welding done using electron beam heat source. Kim and Kawamura [40] also reported successful dissimilar electron beam welding of Zr based bulk metallic glass and Titanium.

Rappaz et al. [41] and David et al. [42] studied the effect of orientation of single crystal substrate (Fe-15Ni-15Cr) on melt pool shape and grain growth during electron beam welding.

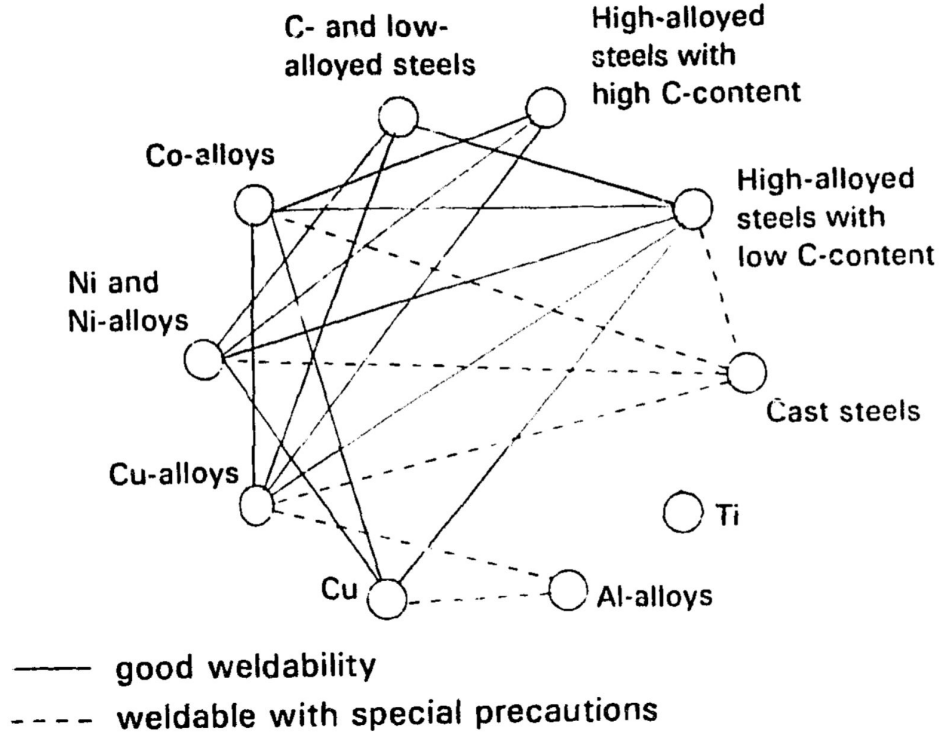


Figure 2.11: Dissimilar metal welding using electron beam. [39]

In addition, Su et al. [43] studied welding of light weight metals including pure Mg and Mg-Al alloys using electron beam and evaluated the variation in melt pool shape and texture as a function of input power.

2.1.4 Modeling of Rapid Solidification Process

It is evident that the thermal management in and around the melt pool is important to control the solidification microstructure. Experimental measurement of the solidification parameters (G and R) is challenging. Hence, to reduce the experimental expense in terms of money and time, modeling tools can be employed apriori to understand the effect of process parameters on the spatio-temporal variation of temperature.

Analytical Models

Analytical models are mathematical models derived as closed form solutions. Since the output is expressed as a function of input parameters, analytical models are not

computationally expensive compared to numerical models. To understand the temperature distribution in welding applications, one of the most commonly used analytical solution is the Rosenthal equation [44]. Rosenthal derived the analytical equation for a moving heat source by solving the heat flow equation with appropriate boundary conditions. The 3D analytical formulation for a point heat source moving with velocity $\mathbf{V}$ in x-direction is given in Equation (2.11).

$$T = T_o + \left(\frac{\eta Q}{2\pi k \sqrt[3]{x^2 + y^2 + z^2}} \right) * \exp \left(\frac{-V \left(\sqrt[3]{x^2 + y^2 + z^2} - x \right)}{2\alpha} \right) \quad (2.11)$$

where T_o is the temperature of the substrate, η is the efficiency, Q is the beam power, k is the thermal conductivity, α is the thermal diffusivity. This analytical solution does not account for latent heat of fusion. Several analytical models listed in Grong [45] have been derived for different boundary conditions based on Rosenthal solution. Few other studies [46, 47] built on the initial Rosenthal (analytical) model to include the effect of phase change and convection due to fluid flow. However, since additive manufacturing is highly transient in nature with complex boundary conditions, it becomes difficult to capture the accurate thermal signatures using analytical models. Numerical modeling tools can be used to address this issue.

In addition to the prediction of thermal field through analytical model, theoretical prediction of "humping" phenomenon has been investigated [48] using Plateau-Rayleigh instability criterion with the assumption that the weld pool is partially bounded cylinder. Width to length ratio of the weld pool was found to be the significant factor in dictating the onset of the "humping" phenomenon. It was concluded that the surface tension affects the kinetics of "humping" phenomenon but not the onset. Lower the width-to-length ratio, higher the instability and vice versa. Figure 2.12(a) shows the humping phenomenon in welding as a function of weld speed and Figure 2.12(b) shows the corresponding process map as a function of power and weld speed. This phenomenon in welding is analogous to the "balling effect" Figure 2.12(c) in electron beam additive manufacturing. Zah et al. [49] did a parametric study and developed a process map to avoid the balling effect. By increasing

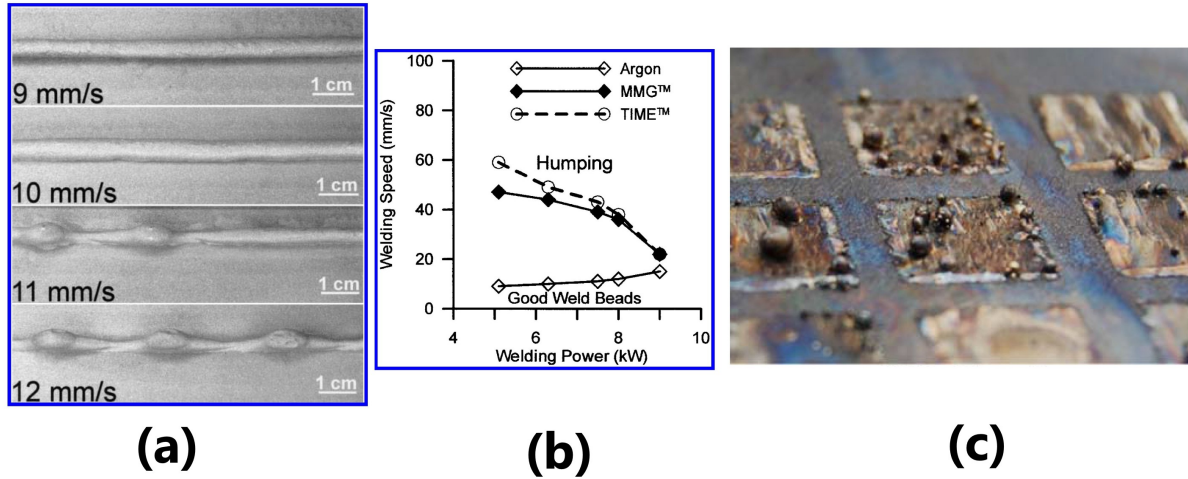


Figure 2.12: (a) Hump formation during welding as a function of welding speed [50] (b) Process map of humping as a function of welding speed and power [50] and (c) Melt ball formation in additive manufacturing [49].

the power and reducing the velocity, the width-to-length ratio of melt pool increased thereby increasing the stability of the molten pool. This prevented the "balling" during fabrication.

Numerical Models

In numerical modeling, the physical process is defined as a partial differential equation. The differential equations are then discretized into set of polynomial equations using different discretization techniques (finite difference, finite element, finite volume). The set of polynomial equations are then solved using iterative solvers. Since the solution is obtained in an iterative manner, numerical models are computationally expensive than analytical models.

The heat transfer and fluid flow during welding, can be captured by solving 3 conservation equations of mass, momentum and energy. Comprehensive explanation of the conservation equation are given in Chapter 3 and Chapter 7. The numerical modeling of welding phenomenon has been pioneered by Zacharia et al. [51] and Debroy et al. [52, 53, 54]. Extensive work has been done by Debroy et al.[55, 56, 53, 54] on understanding the effect of marangoni flow and weld process parameters of spot welding, repair welding and friction stir welding on the solidification parameters of G and R.

Some studies [57, 58, 59, 60] employed thermal imaging techniques to measure the surface temperature distribution of the melt pool. The validity of understanding the melt pool behavior by analyzing the surface temperature data using thermal cameras is questioned using heat transfer and fluid flow modeling by Raghavan et al.[61]. It was proved that the molten pools with similar surface can have significantly different subsurface shapes resulting in different cooling rates at different locations across the melt pool.

In addition, study on evaporation kinetics of weld pool [62, 63] begun using Langmuir's equation (Equation (2.12)).

$$J = \frac{p_i}{\sqrt{2 * \pi * MRT}} \quad (2.12)$$

where $\mathbf{J}$ is the evaporation flux, p_i is the vapor pressure of i^{th} element in the alloy system, $\mathbf{M}$ is the molecular weight, $\mathbf{R}$ is the gas constant, $\mathbf{T}$ is the temperature. Experimental evidences [64] showed that the Langmuir's equation holds good at very low pressure environments but tends to over-predict (5-10 times) the amount of vaporization at normal welding conditions and welding at atmospheric pressure [65]. Even at pressure as low as 200 μm of Hg, the expression over-predicted the evaporation rate [66]. To overcome this drawback and improve the accuracy, in addition to solving equation of mass, momentum and energy, Mundra and Debroy[67] added the equation of translational kinetic energy of vapor near the weld pool surface. Interaction of vapors between the various layers (Knudsen layer, Shielding gas layer) near the weld pool surface was taken into account in the numerical model. He et al. [68, 69] used the results from numerical modeling to understand the extent of alloying element vaporization during spot welding of steel. They also calculated the mass loss due to ejection of powder particles by calculating the recoil force as a function of pressure difference.

Duringn welding, key-holing occurs when the recoil force in the molten pool is greater than the surface tension force holding the molten metal together to the substrate. This causes void in the melt pool thereby increasing the internal reflections of laser. This increases the laser energy deposition desnity on the substrate or weld metal. Rai et al. [56] reported the numerical modeling of key-hole welding of Ti-6Al-4V and 304 stainless steel.In addition,

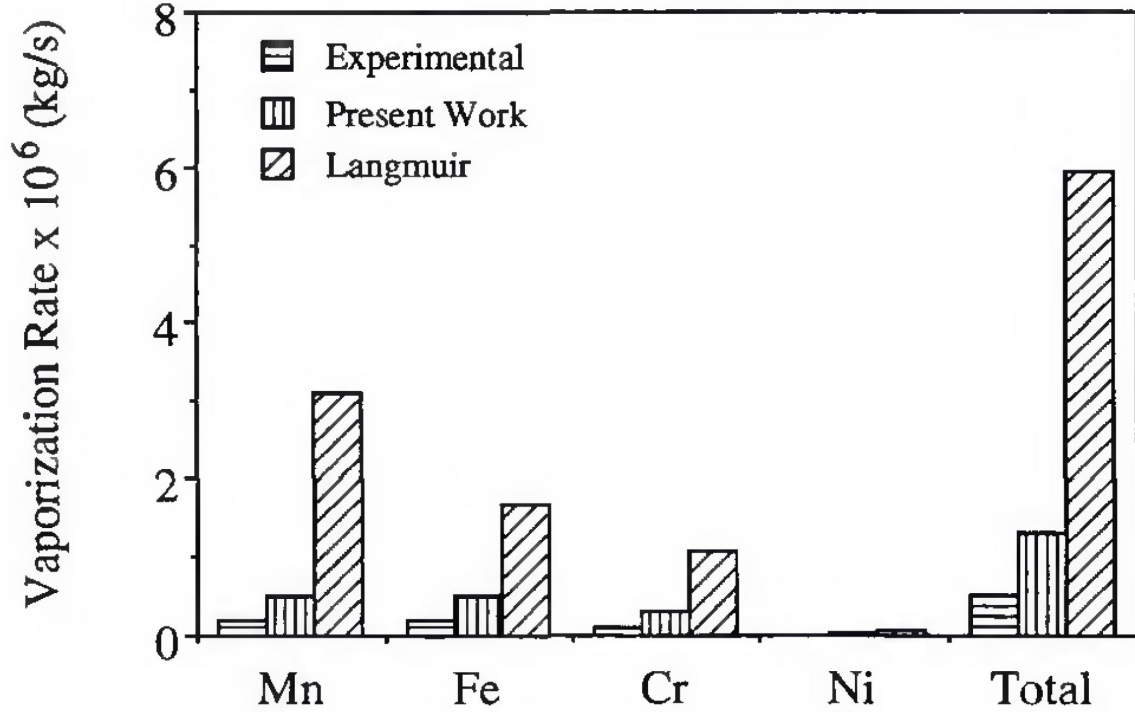


Figure 2.13: Comparison of Langmuir's model and Mundra et al's model. [67]

"key-hole" mode melting in laser additive manufacturing was reported by King et al.[70]. A parametric study of single track melting was done in a powder bed laser AM machine (Concept Laser) using 316L stainless steel. This combined experimental and modeling work showed that the ratio of absorbed energy density to melting enthalpy is the key factor determining the transition from conduction mode to key hole mode. Ratio of melt pool depth normalized to the beam diameter as a function of normalized enthalpy is shown in Figure 2.14. Transition from conduction mode to key-hole mode can be observed for different values of power.

Feasibility of Site-Specific Microstructure Control in AM

In general, during the rapid solidification in additive manufacturing processes, the grains grow epitaxially from the previously solidified layers. Formation of highly textured columnar grains along the build direction is a well known phenomenon [71, 72, 73, 74]. Sames [75] reported the formation of textured columnar grains in a electron beam fabricated Inconel 718 (IN718). Figure 2.15 shows the electron back scattered diffraction data (EBSD) of

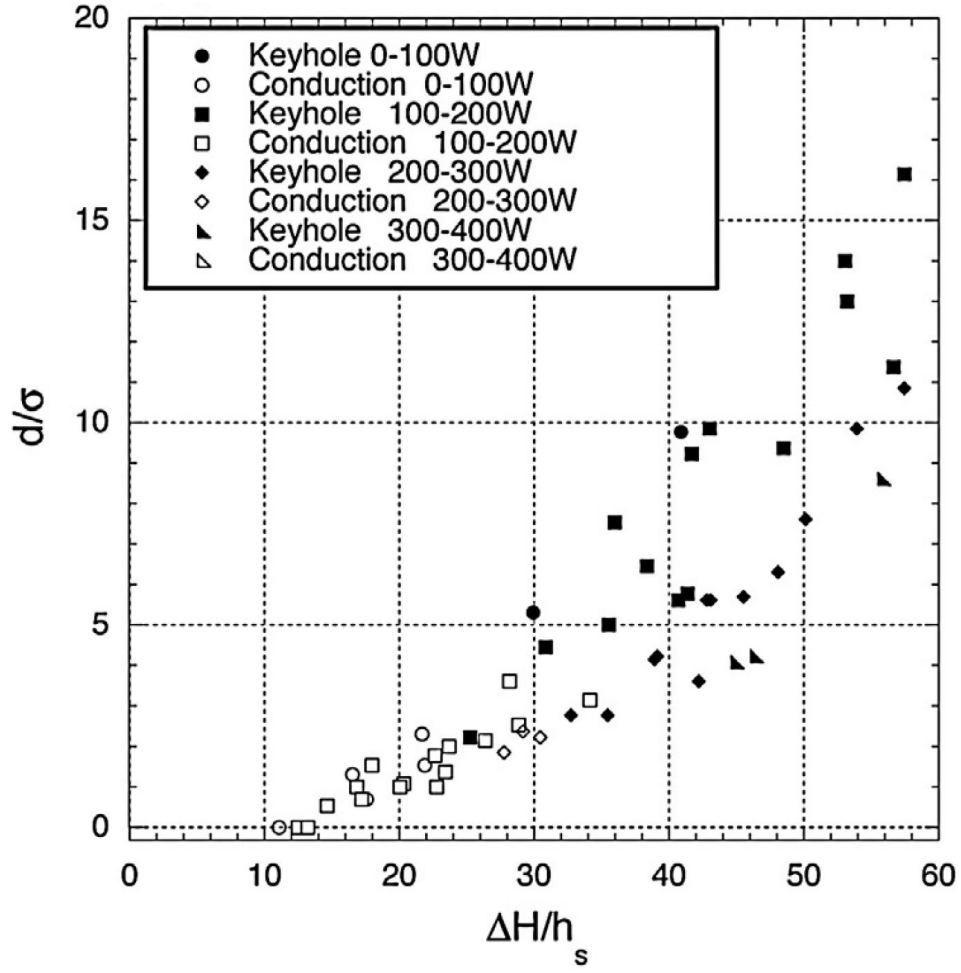


Figure 2.14: Key-hole mode in AM: Normalized melt pool depth as a function of normalized enthalpy. [70]

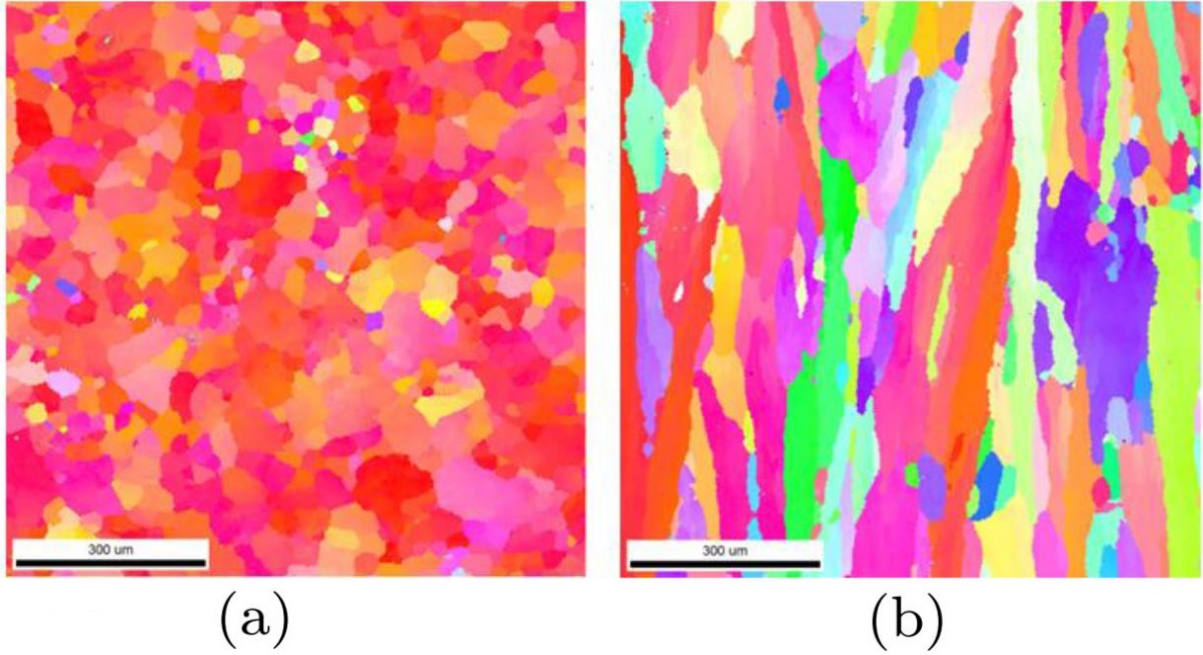


Figure 2.15: EBSD of Arcam[®] fabricated IN718 (a) XY plane and (b) XZ plane [75].

planes parallel to build direction (Figure 2.15(b)) and perpendicular (Figure 2.15(a)) to the build direction. This will lead to anisotropic mechanical response of the additively manufactured component. Depending on the slip systems and critically resolved shear stresses, the directional behavior of mechanical response varies for different crystal structures.

Bontha et al. [76] employed combination of analytical and numerical model to analyze the effect of beam velocity and power on the temperature gradient and growth rate at the liquid solid interface of the melt pool to understand the columnar to equiaxed transition in Ti-6Al-4V. Only two (beam velocity and power) of the many available process parameters were considered and the process map generated by the study predicts a range of velocity (2-10mm/s) and beam power (300W-30,000W) which is significantly unusual compared to nominal range of values of beam power (100-2000W). Experimental validation was not reported by Bontha et al. [76] and there are potential practical difficulties in fabrication of the parts with such high values of energy density.

Körner et al. [77] achieved near equiaxed grain growth by using the raster melt pattern. They reduced the hatch spacing from 150 µm to 37.5 µm and increased the velocity from 2.2 m/s to 8.8 m/s to break down the columnar grains. Figure 2.16 shows the EBSD of

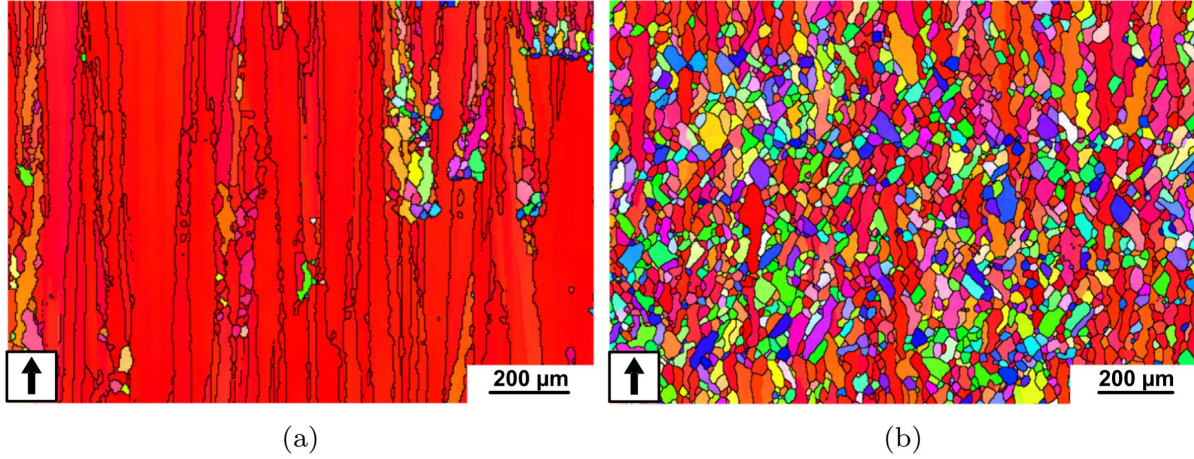


Figure 2.16: EBSD of plane along the build direction (XZ) of Arcam[®] fabricated IN718 with hatch spacing and velocity of (a) 150 μm , 2.2 m/s and (b) 37.5 μm , 8.8 m/s [77].

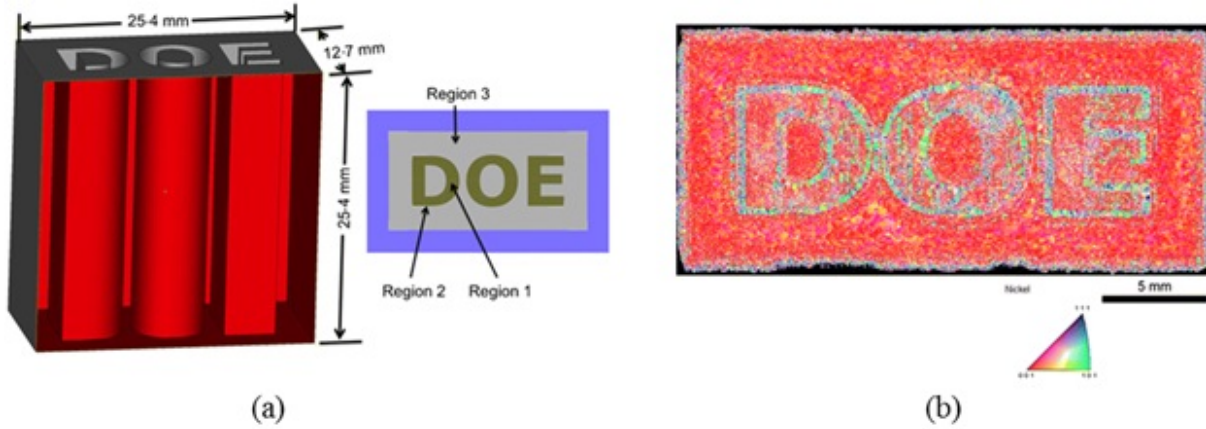


Figure 2.17: Crystallographic Texture Engineering by Modification of Melt Pattern [78]

the plane of the samples parallel to the build direction. By reducing the hatch spacing and increasing the velocity of the beam, they were able to breakdown the columnar grains. However, the effect of scan strategy on nucleation and constitutional supercooling was not comprehensively rationalized.

Dehoff et al.[78] demonstrated the columnar to equiaxed transition (CET) in the same layer by introducing pulsed point heat source that resulted in highly misoriented equiaxed grains and columnar grains within the same layer. Figure 2.17 shows the combination of melt patterns used and corresponding crystallographic orientation map (XY plane).

From Figure 2.17(b), it can be noted in that the grains are oriented along preferred $\langle 001 \rangle$ direction in the region 3 where the raster melt pattern is used and misoriented

grains in region 2 and combination of oriented and misoriented grains in region 1. There is no prior work on the numerical modeling of heat transfer phenomenon in the application of point heat source scan strategy in AM which quantifies the effect all the process parameters that assists in controlling the grain orientation as demonstrated by Dehoff et al. [78].

2.2 Scientific Gaps and Research Plan

2.2.1 Research Gaps

However, none of the aforementioned studies have been extended to design a new melt strategy for site-specific control over grain morphology and grain size in metal additive manufacturing by predicting the spatial and temporal variation of thermal gradient (G) and liquid-solid interface velocity (R). The crux of this work is to introduce new melt scan strategies for electron beam additive manufacturing system and predict the transient solidification conditions *a-priori* using a heat transfer and fluid flow modeling tool. To be precise, the primary research questions to be addressed are

- Is it possible to predict spatio-temporal variation of solidification conditions using numerical model and use the data in already developed theoretical models developed for rapid solidification in welding?
- Is it possible to control the columnar grains size in bulk sample fabricated through electron beam AM system?
- Is it possible to change the bulk grain morphology of additively manufactured samples from columnar to equiaxed?

2.2.2 Material System

Inconel 718 has been used as the processing material in this research work. Inconel 718 (*IN718*) is a precipitation strengthened nickel-base superalloy that has excellent mechanical properties at elevated temperatures primarily used in gas turbine engines in aircraft and power generation industries. The matrix (γ) has face centered cubic (FCC) crystal structure.

Table 2.1: Nominal composition (wt%) of Inconel 718 [79].

	<i>Ni</i>	<i>Cr</i>	<i>Nb</i>	<i>Mo</i>	<i>Ti</i>	<i>Al</i>	<i>Co</i>	<i>C</i>	<i>Mn</i>	<i>Si</i>	<i>P</i>	<i>S</i>	<i>B</i>	<i>Cu</i>	<i>Fe</i>
Min	50	17	4.7	2.8	0.65	0.2	1	0.08	0.35	0.35	0.015	0.015	0.006	0.3	<i>bal</i>
Max	55	21	5.5	3.3	1.15	0.8	<i>max</i>	<i>max</i>	<i>max</i>	<i>max</i>	<i>max</i>	<i>max</i>	<i>max</i>	<i>max</i>	<i>bal</i>

Primary strengthening precipitate (γ'' - Ni_3Nb) has Body Centered Tetragonal (BCT -DO22) crystal structure. Contribution of gamma prime (γ' - $Ni_3(Al, Ti)$, LI2 crystal structure) to strengthening is significantly lesser due to lower concentration of Al and Ti compared to Nb. These phases (γ' , γ'') are coherent with the γ matrix with similar lattice parameters. IN718 is a highly weldable nickel alloy compared to other high γ' nickel alloys. MX carbides, laves and delta are the other precipitates that can form inside the gamma matrix. These three precipitates have significantly larger lattice parameters making them incoherent with the matrix. Formation of these precipitates are detrimental to the strength of the material. These carbides, laves and delta phases consume Nb from the matrix reducing the amount of γ'' precipitation thereby reducing the strength of the material. Nominal chemical composition of IN718 is given in Table 2.1.

2.2.3 Numerical Code

Three dimensional numerical simulations were performed using the code Truchas[80]. Truchas was developed under the Advanced Simulation and Computing (ASC) program at Los Alamos National Laboratory (LANL) for simulation of metal casting processes. It is an open-source, continuum scale, multi-physics simulation tool designed to solve large problems on parallel high-performance computing (HPC) platforms. Truchas employs physical models for alloy solidification with heat transfer and phase change algorithms accounting for non-isothermal phase change in mushy zones, which is a typical characteristic of complex alloys like IN718. Appropriate user subroutines are written to define the scan strategy and other beam input parameters including the profile of the heat source distribution. A comprehensive explanation of the numerical modeling tool is provided in Chapter 3.

Table 2.2: Verification and validation of Truchas [81].

	Truchas	Debroy et al.[69]	Experiment
Weld pool radius (mm)	0.241	0.254	0.313
%difference vs experiment	-23%	-19%	
Weld pool depth (mm)	0.199	0.202	0.190
%difference vs experiment	+4.7%	+6.0%	
Radius/depth ratio	1.21	1.26	1.65
%difference vs experiment	-27%	-24%	

Why Truchas?

There are many commercial numerical codes like Abaqus, Comsol, Flow3D available to analyze the rapid solidification behavior. One of the main reasons for choosing Truchas is because it is an open source code and has been tested for welding and casting applications [81, 82, 80, 83]. Parietti and Lim [81] validated multiple solidification physics of Truchas by comparing the results of solidification during welding (with and without phase change, with and without convection) with ABAQUS. The code was also validated against experimental results and modeling results by Debroy et al. [69]. Table 2.2 shows the quantitative comparison of the codes in terms of melt pool width and depth. The results were found in good agreement for both the cases.

The code can also be employed on a high performance computing platform. Comparatively, ease of access to developers of the code at Los Alamos National Laboratory was also taken into consideration before choosing this code for this research work. Similar open source software with larger user community like OpenFOAM [84] can also be used incorporate the physics and evaluate this research work. At the time of beginning of this work, Truchas was the known, parallel capable and tested open-source code with the solidification physics built-in and hence, it was used in this research work.

2.2.4 Experimental Methodology

Part Fabrication

For experimental validation, the samples were fabricated using Arcam[®] S12 and A2 machines located at the Manufacturing Demonstration Facility (MDF) in Oak Ridge National Laboratory (ORNL). The *stl* files were sliced into 2-D layers using Arcam[®] build assembler software. An in-house code was developed and used to create a new file defining the proposed melt-scan strategy. The generated file was fed back into the Arcam[®] build assembler to create the Arcam build file (*.abf) which was then loaded into the machine for fabrication.

Characterization

The fabricated samples have been cut along the planes parallel (XY plane) and perpendicular (XZ plane) to the build direction. The samples are then mounted and well-polished ($<1\text{ }\mu\text{m}$) for characterization. Optical microscopy has been used to study the grain structure and analyze the primary dendrite arm spacing of the etched sample. To reveal the dendrites, the samples were etched using a mixture of nitric acid, acetic acid and hydrochloric acid in equal proportion (1:1:1). *Leica DM4000 M* instrument has been used to perform optical microscopy. Further, to understand the variation in grain morphology, crystallographic orientation, and texture variation, a scanning electron microscope (SEM - *Leica DM4000 M*) equipped with electron back scattered diffraction (EBSD) detector has been used.

Chapter 3

Overview of Numerical Modeling Framework

3.1 Truchas

Truchas [80] is an open-source, highly parallel, multi-physics continuum scale code developed as a part of Advanced Simulation and Computing (ASC) project (Telluride) at Los Alamos National Laboratory (LANL). Primary motivation for the development of the code was to understand the transient metal casting process for nuclear application. Truchas is primarily written in Fortran with a small fraction of the software written in C and Python. Appropriate user-subroutines have been developed to design and implement melt scan strategies (moving heat source) in Truchas to simulate the transient rapid solidification occurring during metal powder bed additive manufacturing processes.

3.1.1 Conservation Equations

Multi-physics aspect of Truchas includes heat transfer and phase change, fluid dynamics including Marangoni flow due to surface tension gradient, electromagnetics and solid mechanics. The simulations and results in Chapter 4 and Chapter 5 considers only the physics of heat conduction and phase change. Conservation equation for heat conduction and phase change is given by Equation (3.1).

$$\frac{\partial(\rho h)}{\partial t} = \nabla \cdot (k \nabla T(h)) + \dot{S} \quad (3.1)$$

where ρ is the density, k is the thermal conductivity, h is the specific enthalpy and $\dot{S}$ is the source term. Source term includes both positive and negative fluxes. Some of the source terms considered for the additive manufacturing problem are

- Heat source used to melt the powder particles (electron beam or laser).
- Latent heat evolution during phase change.
- Radiative and convective heat fluxes at the boundaries.

Equation (3.1) contains the diffusive flux, the advective flux is described in Chapter 7. The partial differential equation is then discretized by mimetic finite difference method. The resulting set of non-linear equations are then solved using one of Jacobian Free Newton Krylov (JFNK) or Accelerated Inexact Newton(AIN) methods.

The effect of fluid mechanics on solidification is explained in Chapter 7. The physics involved, conservation equation and solution procedure are discussed in Chapter 7.

3.1.2 Heat Source Profile

To improve the accuracy of the prediction of the numerical simulations, it is important to understand the effect of interaction of the heat source on the material being fabricated. One of the important parameters of the interaction is the distribution profile of heat source. An electron beam additive manufacturing system (Arcam[®]) is the focus of this research. In general, laser interacts only with the surface of the material while electron beam penetrates into the impinging material. In addition to the surface heat source profile, there will be a heat source profile along the depth of the material as well. The surface profile is given by Gaussian distribution as given in Equation (3.2).

$$I_{xy} = \frac{1}{2 * \pi * \sigma^2} \exp\left(\frac{-(x^2 + y^2)}{2 * \sigma^2}\right) \quad (3.2)$$

where I_{xy} is the surface intensity profile, σ is the standard deviation, x and y are the distance from the center of the beam in X and Y axis respectively. Beam diameter is directly related to σ .

The vertical intensity profile is given by an equation derived from experimental measurement. Absorbed power per unit volume and penetration depth has been approximated and the quadratic approximation of the vertical intensity profile is given in Equation (3.3)[49]

$$I_z = -3\left(\frac{z^2}{z_e^2}\right) + 2\frac{z}{z_e} + 1 \quad (3.3)$$

where I_z is the heat source intensity profile along z-direction, z is the distance (depth) from the surface of the sample, z_e is the absolute penetration depth. Absolute penetration depth is defined as the depth at which 99% of the electron beam power is absorbed. Absolute penetration depth is given by Equation (3.4) [85, 86]

$$z_e = 21 * \left(\frac{V_e^2}{\rho}\right) \quad (3.4)$$

where z_e is the penetration depth in μm , V_e is the accelerating voltage of the electron beam in kV , ρ is the density of the material in $\frac{kg}{m^3}$.

Volumetric profile of the electron beam heat source is given by

$$\dot{Q}(x, y, z) = -\eta_e * \eta_b * Q_{max} * \frac{I_{xy} * I_z}{z_e} \quad (3.5)$$

where

$$Q_{max} = I_e * V_e \quad (3.6)$$

where I_e is the electron beam current, V_e is the electron beam voltage, η_e is the surface energy conversion and η_b is the beam control efficiency. In general, beam control efficiency and surface energy conversion are high (~ 0.9) for most of the materials interacting with

electron beam systems [38]. Combining Equation (3.2), Equation (3.3), Equation (3.5), following heat source profile can be defined.

$$\dot{Q}(x, y, z) = -\eta_e * \eta_b * I_e * V_e * \frac{4 * \ln(0.1)}{\pi * d^2 * z_e} \exp\left(\frac{4 \ln(0.1) * (x^2 + y^2)}{d^2}\right) * \left(-3 \left(\frac{z^2}{z_e^2}\right) + 2 \frac{z}{z_e} + 1\right) \quad (3.7)$$

where d is the electron beam diameter. In Arcam[®], the beam voltage (V_e) is constant (60 kV) and beam current (I_e) is variable.

3.1.3 Limitations and Assumptions of the model

The simulations are completely continuum scale. The interaction between the powder particles and electron beam is ignored. Preheating (sintering) phase of the process is ignored. Selective evaporation of elements in the melt pool has been reported [87] in AM process. Energy loss due to evaporation cooling is not considered in this model. Expansion and shrinkage of the material due to the changes in temperature during melting and solidification are not accounted for in these simulations. The mass is conserved within each cell, and cell volumes are kept constant before, during and after the phase change. Hence, the density of the material is assumed to be constant for all the phases of materials used in the simulation.

3.1.4 Initial and Boundary Conditions

Local preheat temperature of the substrate, one of the indirect control parameters in Arcam[®] process, was used as the initial condition of the spatial domain, and was considered a variable in the simulations. The top surface of the build radiates heat to the surroundings. Part fabrication in the Arcam[®] process takes place in a low pressure environment (10^{-3} mbar partial pressure of helium) [88], therefore the heat transfer due to convection on the top surface of the domain is negligible and was assumed to be zero in the simulations. Hence the boundary condition at the top surface (γ) was formulated by applying energy conservation Equation (3.8)

$$-k\Delta T = \epsilon * \sigma * (T_x^4 - T_a^4) \quad x \in \gamma \quad (3.8)$$

where T_a is the ambient temperature ($25^\circ C$), k is the thermal conductivity of the material Table 3.2, ϵ is the emissivity (0.4 [89]) and σ is the Stefan-Boltzmann constant ($5.67 \times 10^{-8} W.m.K^{-4}$).

During the fabrication process, the part is encapsulated by fine metal powder particles of varying diameters ($20 \mu m - 100 \mu m$). Alkahari et al [90] experimentally measured and theoretically verified the thermal conductivity of metal powders of varying diameters ($10 \mu m - 100 \mu m$) used in selective laser melting. The thermal conductivity of metal powders was found to be between 0.33–1.5% of the thermal conductivity of bulk metals. The thermal conductivity of metal powders is so low such that it can be considered an insulator for the short duration (ms) of simulations. Hence the boundary conditions on the remaining five faces were assumed to be adiabatic. That is, a Neumann boundary condition with zero flux across the surfaces.

3.1.5 Geometry and Meshing

Since the focus of this research is to understand the transient conditions in bulk geometry of the printed part, the physical domain is assumed to be a thick plate. The maximum size of the domain simulated was $5 \times 5 \times 2$ mm. The geometry is created and meshed using CUBIT[91]. Depending on the beam diameter, the mesh size was changed between $5 \mu m$ and $20 \mu m$. Template script for generating the mesh using CUBIT command line can be found in Appendix B.1.

3.1.6 Scaling of the Code

Truchas is developed to run on high performance clusters with distributed memory systems. Systems in which the CPU's have their own memory is called distributed memory architecture opposed to shared memory architecture where a common memory is shared by all

the CPUs. Truchas uses MPI library (Message Passing Interface) along with PGSLib to communicate between the processors. Depending on the size of the problem, the maximum number of spatial domains used in the simulations is about 3 million. Truchas is a parallel code capable of employed in HPC clusters for efficient simulations. Depending on the number of processors used, the spatial domain (mesh) is discretized into numerous sub-domains during the initializing phase of the simulation using Chaco partition library.

Scalability analysis was done to understand the effectiveness of the code in clusters. 2 types of scaling analysis can be done (a) weak scaling and (b) strong scaling. During weak scaling analysis, load per core or processor remains constant (number of nodes/equations in the mesh allocated per core/processor is constant) but number of workers/processors is increased. In strong scaling, the total load (number of nodes/equations in the mesh allocated to the cluster is constant) is kept constant but the number of processors is increased.

Strong scaling analysis is done for pure heat transfer and phase change problem in Truchas. Total number of elements in the mesh is kept constant at 2 million. Total number of time steps in the simulation is constant at 2068. Number of cores used to solve the problem has been increased from 2^3 to 2^{10} . This analysis was done on a Cray XK7 machine with 16 core AMD interlagos and 32GB of RAM per node (cluster name: CADES, location: ORNL).

Absolute strong scaling efficiency of a problem size N on P processors is defined in Equation (3.9) [92].

$$\eta(N, P) = \frac{1}{P} \frac{T(N, 1)}{T(N, P)} \quad (3.9)$$

where $T(N, 1)$ is the total wall time of the simulation of size N using 1 processor and $T(N, P)$ is total wall time of the simulation using P processors. Since the current problem size (2 million elements) is too large for 1 processor, minimum number of processors used in this case is 16. Hence, the relative scaling efficiency of scaling from Q processors to P processors is defined in Equation (3.10) [92].

$$\eta(N, P|Q) = \frac{Q}{P} \frac{T(N, Q)}{T(N, P)} \quad (3.10)$$

Speed up ratio is another quantity to measure the performance of the code. Speed up of the code is defined as the ratio of time taken for the code to run on 1 core to time taken on N cores. Table 3.1 shows the detailed usage of wall clock time by each part of the code during execution for the specified fixed problem size (strong scaling).

Table 3.1: Strong Scaling: Wall Time Usage Analysis

Total Number of Cores	Elements assigned per core	Total Wall Time (s)	Reading Input (s)	Initialization(s)	Diffusion Solver (s)	Writing Output (s)
16	125,000	15640	0.104	1934	13060	449
32	62,500	8510	0.140	1551	6599	265.7
64	31,250	4915	0.145	1394	3305	171.8
128	15625	3261	0.150	1400	1732	117.6
256	7813	2420	0.161	1401	906.8	106.2
512	3906	1955	0.1548	1377	472.3	101.4
1024	1953	1816	0.2947	1460	276	77.26

Figure 3.1(a) shows the speed up ratio and Figure 3.1(b) shows the comparison of relative scaling efficiency with data from 16 processors as the baseline calculated from the data in Table 3.1. From Figure 3.1 (b), it can be concluded that for this physics and defined problem size, increasing the number of processors to 1024 is essentially a waste of the computational resource. It can also be concluded that for efficient use of the computational resource, the load assigned should be between 30k and 15k elements per processor approximately. Care should be taken that this scaling analysis will not be valid if new physics is added to the model.

3.1.7 Material Properties

Thermophysical properties of IN718 used in the simulations are given in Table 3.2

3.1.8 Calculation of G and R

As explained in Chapter 2, the solidification microstructure is primarily dictated by thermal gradient (G) at the liquid-solid interface and velocity (R) of the liquid-solid interface. The

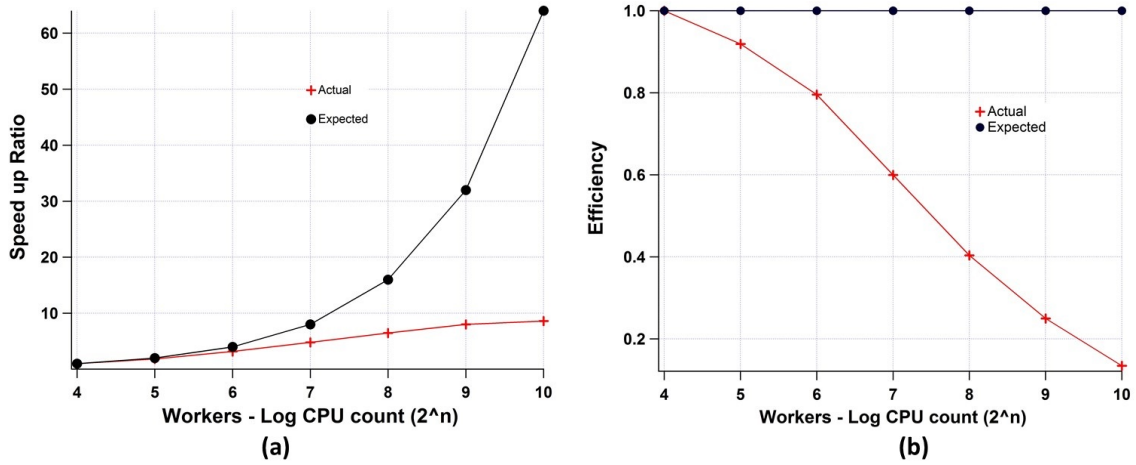


Figure 3.1: (a) Speed up ratio and (b) Scaling Efficiency of Truchas (Heat transfer and phase change)

Table 3.2: Thermophysical properties of IN718 [93, 94]

Property	Value	Unit
Density	7451	$kg.m^{-3}$
Solidus Temperature	1528	K
Liquidus Temperature	1610	K
Latent Heat of Fusion	227000	$J.kg^{-1}$
Specific heat capacity of solid	600	$J.kg^{-1}.K^{-1}$
Specific heat capacity of liquid	775	$J.kg^{-1}.K^{-1}$
Thermal conductivity of solid @ 1300 K	26.6	$W.m^{-1}.K^{-1}$
Thermal conductivity of liquid @ 1850 K	29	$W.m^{-1}.K^{-1}$

output of Truchas is spatio-temporal variation of temperature. The values of resultant G is extracted from the temperature data based on Equation (3.11).

$$G = \sqrt[2]{G_x^2 + G_y^2 + G_z^2} \quad (3.11)$$

where G_x , G_y and G_z are thermal gradient along X, Y and Z directions respectively as shown in Equation (3.12).

$$G_x = \frac{dT}{dx}; G_y = \frac{dT}{dy}; G_z = \frac{dT}{dz} \quad (3.12)$$

The cooling rate is calculated according to Equation (3.13).

$$CoolingRate = \left| \frac{dT}{dt} \right| \quad (3.13)$$

The cooling rate is negative during melting and positive during solidification. This will help in identifying whether particular mesh element is melting or solidifying.

The liquid-solid interface velocity can now be calculated as the ratio of cooling rate and resultant thermal gradient as shown in Equation (3.14)[95].

$$R = \frac{\left| \frac{dT}{dt} \right|}{\sqrt[2]{G_x^2 + G_y^2 + G_z^2}} \quad (3.14)$$

To demonstrate the calculation of G and R and their distribution, a single spot simulation is performed. Figure 3.2 shows the liquid-solid interface tracking along XZ symmetrical plane (side view) of the melt pool solidification with plots of liquidus (1610K) isotherm of the melt pool at different time of a simulation with beam current of 20 mA turned ON for 1 ms with a preheat temperature of 1528 K (solidus). From Figure 3.2, it is evident that the melting and expansion of the melt pool continues even after the beam is turned off at 1 ms. In this case, the melt pool continues to expand and solidification begins only at 10 ms as shown in Figure 3.2 (a) and completely solidifies at 30 ms as shown in Figure 3.2 (d).

Figure 3.3 shows the overlap of Figure 3.2 (a), i.e. the liquidus isotherm at the beginning of the solidification, and temperature gradient vector of the entire domain. The temperature

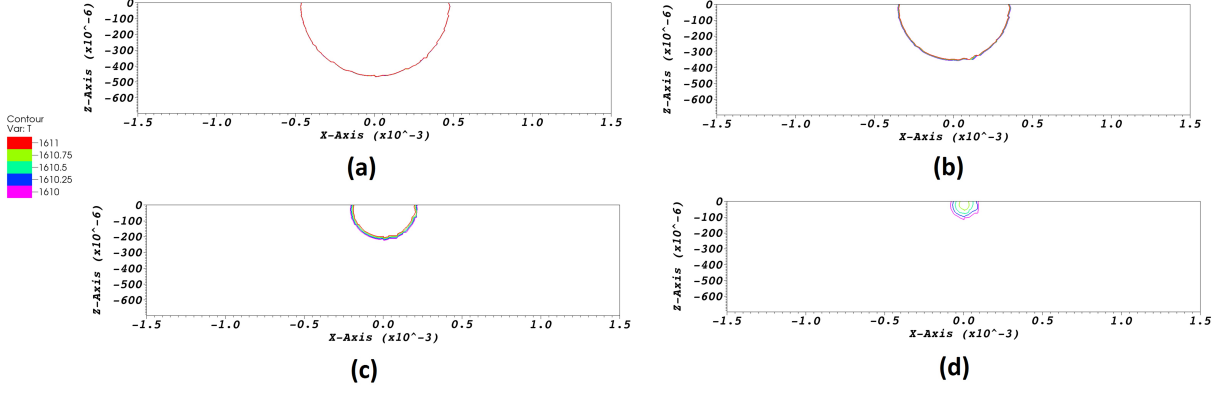


Figure 3.2: Transient Liquid-Solid interface tracking of melt pool (a) at the beginning of solidification 10ms (b) 20 ms, (c) 26 ms, and (d) end of solidification 30 ms.

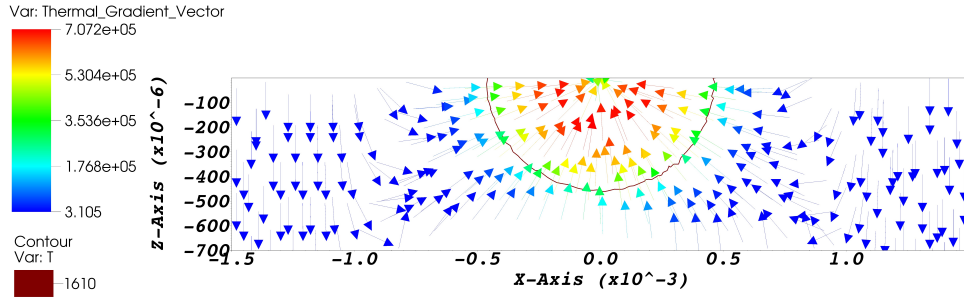


Figure 3.3: Overlap of Thermal Gradient vector and Liquidus isotherm (1610 K) along the XZ plane.

gradient vectors in the melt pool point towards the center of the heat source where the temperature is higher. The direction of the thermal gradient vector is normal to the liquid-solid interface along the liquidus isotherm which essentially dictates the direction of crystal growth at the liquid-solid interface.

Spatio-temporal distribution of G and R are then calculated according to Equation (3.11) and Equation (3.14). The values are calculated at all of the elements in the spatial domain of the simulation. The values have to be filtered only along the elements at the liquid-solid interface. This extraction is automated and performed using a python script coupled with an open-source, parallel, data visualization software VisIt [96]. The script is given in Appendix B.2. The values along the liquidus isotherm (1610 K) of the melt pool are used to obtain spatial and temporal distributions of the G and R only at the liquid-solid interface as the melt pool solidifies. These distributions are then used to predict the transition from

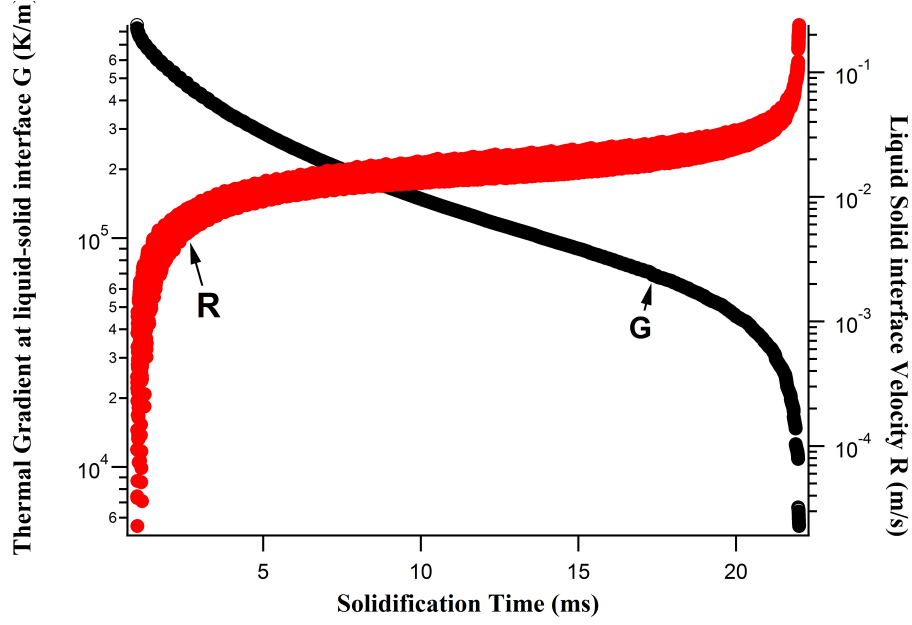


Figure 3.4: Variation of G and R as a function of solidification time

columnar to equiaxed microstructure. The extracted G and R values are plotted on the reference solidification map [97] for IN718.

Figure 3.4 shows the variation of temperature gradient (G) and liquid-solid interface velocity (R) as the melt pool collapses. From Figure 3.4, it is evident that the temperature gradient is high and the liquid-solid interface velocity is low as the melt pool begins to solidify. As the liquid front advances and melt pool shrinks, the temperature gradient decreases and the liquid-solid interface velocity increases rapidly. This behavior of G and R with respect to solidification time agrees with the results for spot welding by Debroy et al. [54, 98, 53, 52]. Quantitative and qualitative analysis of this spatio-temporal variation and its effect on solidification microstructure in electron beam additive manufacturing is explained in the following chapters.

Chapter 4

Grain size and Primary Dendrite Arm Spacing control

4.1 Influence of Grain Size on Mechanical Properties

In general, for room temperature applications, the yield strength of a metallic component is inversely proportional to the square root of grain size (Hall-Petch relationship). Grain boundaries pin the movement of dislocations by creating lattice mismatch thereby increasing the yield strength [99]. In structural components used in the environment at or below the homologous temperature of the material, the mechanical properties (yield strength, fracture toughness, fatigue life) tend to increase with increase in grain boundary area per unit volume (grain size refinement). On the other hand, for components used in high-temperature applications, the reduction in grain size or the increase in grain boundary area per unit volume is detrimental to the creep and stress rupture properties. This is because, a decrease in grain size increases the diffusion rate, thereby increasing the creep rate of the component and thereby inducing shorter service life. The effect of grain size on creep behavior of Nickel-base superalloys has been reported in the literature [100, 101]. In addition to grain size, primary dendrite arm spacing (PDAS) is also reported to have significant impact on the high cycle fatigue life of components fabricated using Nickel alloys [102]. This chapter examines the feasibility of grain size and PDAS control by changing the melt strategy.

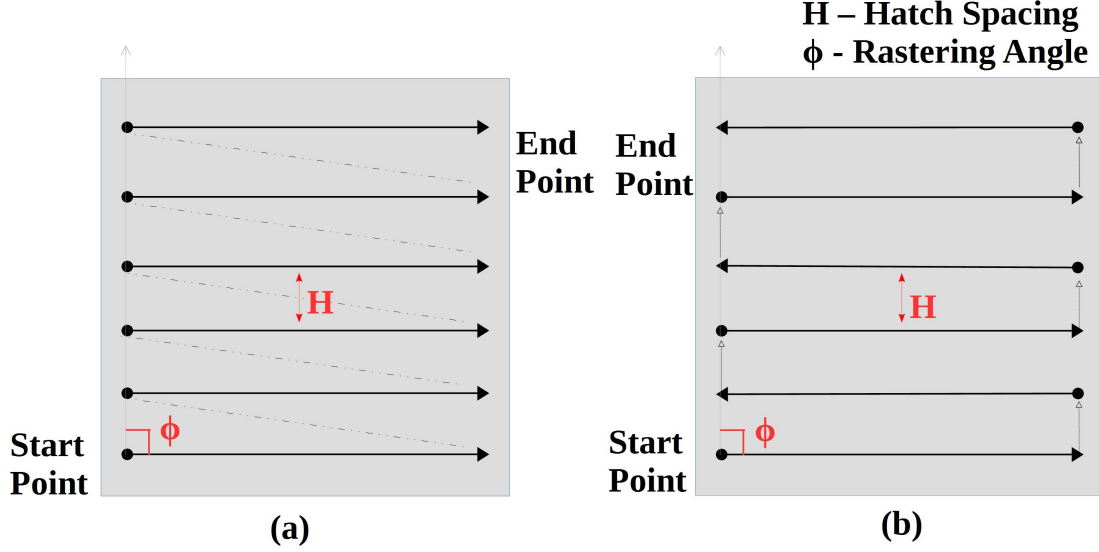


Figure 4.1: Types of raster melt-scan (a) unidirectional (b) bidirectional.

4.2 Melt-Scan Strategy

4.2.1 Conventional Melt Sequence

The most widely used melt-scan strategy (Figure 4.1) in the current AM processes is similar to multi-pass welding. The heat source moves linearly from one end to the other with temporally varying current and velocity. The distance between adjacent raster lines (H -hatch spacing) dictates the overlap of the melt pools. As shown in Figure 4.1, the scanning can be unidirectional or bidirectional. The angle (ϕ -rastering angle) between the scan direction and a reference axis can also be changed from one layer to the next. Epitaxial solidification of columnar grains within the melt pool and along the build direction is a well-known phenomenon for raster melt-scans[2,27]. The length of the raster scan depends on the geometry of the part being fabricated. Therefore, the local thermal history and solidification conditions are significantly affected by changes in geometry and it is a complex challenge to obtain robust site-specific control over the solidification microstructure using the raster melt-scan strategy for arbitrary geometries.

4.2.2 Proposed Melt Strategy

As explained in the Section 2.1 in Chapter 2, during rapid solidification, the solidification microstructure is primarily dictated by the thermal gradient (G) at the liquid-solid interface and velocity of the liquid-solid interface (R). The motivation behind the design of new melt-scan strategy is to control the local G and R during solidification. Localized solidification control leads to consistent and similar solidification microstructure within the whole component.

In this study, continuous melt scan has been replaced by pulsed spot melt scan strategy to achieve localized control over solidification condition. Since electron beam in Arcam[®] can move at the high velocities ($\sim 4 \text{ km/s}$), the ratio of energy deposition rate to the heat removal or diffusion rate can be so high such that, depending on velocity and power, the raster scan can be made to have a linear energy profile in 1D or areal energy profile in 2D. This complexity makes it challenging to use raster melt strategy to control solidification microstructure for complex geometry. Since point or spot melt has zero velocity, it can be considered as 0-D and it becomes relatively easier to control the solidification phenomenon in complex geometries. Thereby, using spot scan pattern as the design variable is to decouple the process parameters from the geometry of the part being fabricated. To decouple the geometry and achieve localized solidification conditions throughout the area of a particular layer, the 2-D slice of the layer is spatially discretized into smaller sub-domains. Figure 4.2(a) shows a schematic of a square area to be melted, which is divided into numerous smaller sub-domains or 'tiles'. The numbers within each sub-domain indicate the sequence in which each of sub-domains will be melted. The horizontal or vertical distance between a sub-domain and the next in the sequence along X and Y axis is termed as external offset (X_1 and Y_1 respectively). External offset is employed to ensure that the melting and solidification of the sub-domains in sequence are independent of each other (i.e. solidification in domain 1 is independent of domain 2). Further, as shown in Figure 4.2(b), each sub-domain contains 5 spots at which the electron beam energy is deposited. The 5 spots within one sub-domain can be moved closer or away from each other. The horizontal (X_o) or vertical (Y_o) distance between the center spot and one of the fringe spots within the sub-domain is termed as

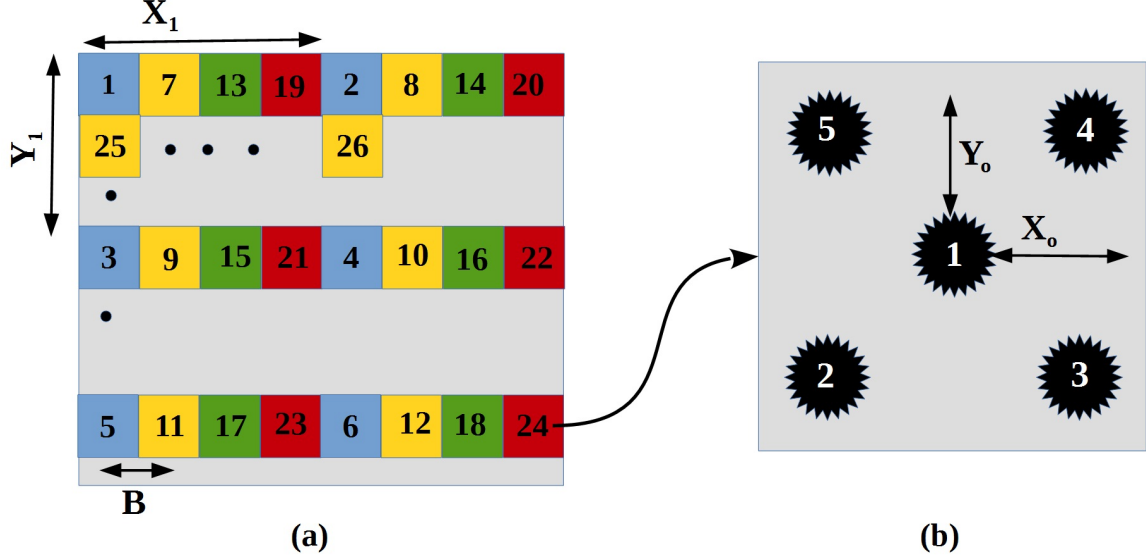


Figure 4.2: Proposed Melt Scan Strategy (a) Partitioning of melt area into subdomains (b) Distribution of spots within one subdomain.

internal point offset. The rationale behind moving the spots closer or farther from each other is to gain control over the solidification parameters (thermal gradient G , liquid-solid interface velocity R and cooling rate $G \cdot R$) which is vital to control the solidification grain size. This spot melting (Figure 3) pattern is repeated layer-by-layer depending on the geometry of the slice of the particular layer. Since a cubic geometry is chosen for this investigation, the 2-D cross section of the geometry remains constant for all the layers. In this chapter, the experiment was done in advance and then the results were analyzed and rationalized using the results from numerical modeling.

4.3 Experimental Set up

Samples were fabricated using IN718 powder in an Arcam[®] S12 machine with a layer thickness of $50 \mu\text{m}$ and a bed temperature of 1050°C . The dimension of the samples was $2 \times 2 \times 2 \text{ cm}$. A layer of $2 \times 2 \text{ cm}$ is divided into 40×40 overlapping sub-domains on the XY plane. The sub-domains overlap laterally ($500 \mu\text{m}$ from one sub-domain to the adjacent) to avoid lack of fusion porosity through the build. At each spot, the electron beam is turned on for a constant amount of time (0.25 ms). The accelerating voltage (60 kV) of the Arcam[®] machine cannot be varied. Beam current is a variable, but for this study, uniform beam

Table 4.1: Values of input parameters of proposed scan strategy

Case	Internal Point Offset (μm)	Beam Current (mA)	Beam On Time (ms)
1	200	20	0.25
2	400	20	0.25
3	600	20	0.25
4	800	20	0.25

current of 20 mA is used at each spot. The external offset value ($X_1=Y_1=3\text{mm}$) was chosen based on the predicted heat affected zone during solidification of a sub-domain and is kept constant for all the samples. There are 1600 sub-domains and 8000 spots per layer per sample. Because the number of spots per layer was kept constant, energy deposition per layer (2.4 kJ) remained constant for all the samples. Variation of solidification microstructure between the samples at constant energy density is also to show the significance of the melt strategy on solidification. Table 4.1 shows the values of beam input parameters and internal point offset variable of the proposed scan strategy to be analyzed. The size of the sample and corresponding process parameter window including the number of spots and energy density per layer were chosen based on the previous experience for successful fabrication. Focus coil current of 0 mA was used for fabrication. The focus coil current controls the beam diameter. Analyzing the quantitative relationship between the focus coil current and beam diameter is beyond the scope of this work.

4.4 Results and Discussions

4.4.1 Melt pool shape

Figure 4.3 shows the image of the fabricated samples on the substrate with varying internal point offset as mentioned in Table 4.1. On closer observation, variation in surface topology can be noticed. To further investigate this variation, the samples were examined under the optical microscope.

Figure 4.4 shows the differences in shape of top surface of the melt pool formed within one sub-domain. All the melt pools were concave in nature with respect to the surface. From

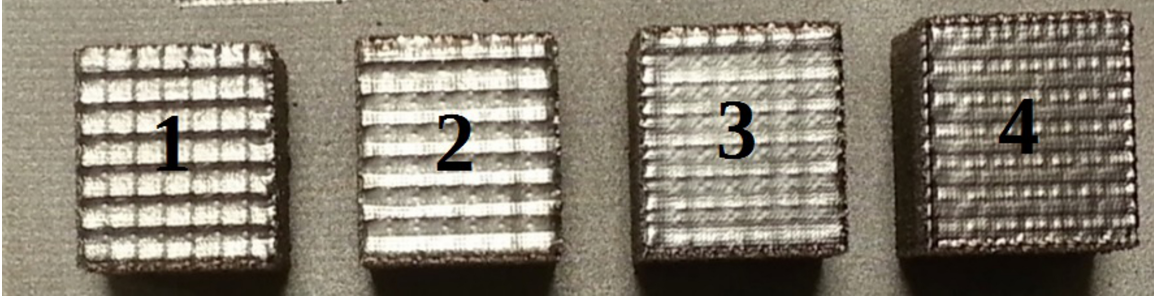


Figure 4.3: Top view of fabricated samples with internal point offset of (1)200 μm (2)400 μm (3)600 μm (4)800 μm

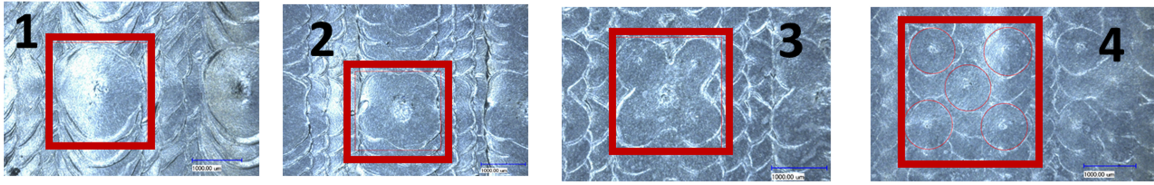


Figure 4.4: The top surface of fabricated samples showing the variation in met-pool shape as a function of internal point offset (1)200 μm (2)400 μm (3)600 μm (4)800 μm

Figure 4.4 (1) and (2), it can be observed that for samples with internal point offset of 200 μm and 400 μm respectively, the individual melt pools of all the 5 spots merge together to form a single melt pool. While looking at Figure 4.4 (3), it can be observed that the melt pools begin to pull away from each other forming 'butterfly' like shape and by further increasing the internal point offset to 800 μm , all the 5 melt pools are completely independent of each other (Figure 4.4 (4)).

All 4 processing conditions were simulated using Truchas (Chapter 3) in conduction only mode to verify the solidification behavior. Beam diameter of 200 μm was used in the simulations. Figure 4.5 shows the shape and size of the simulated melt pools captured at the simulation time just before the melt pools start to collapse. The results agree with the experimental results shown in Figure 4.4.

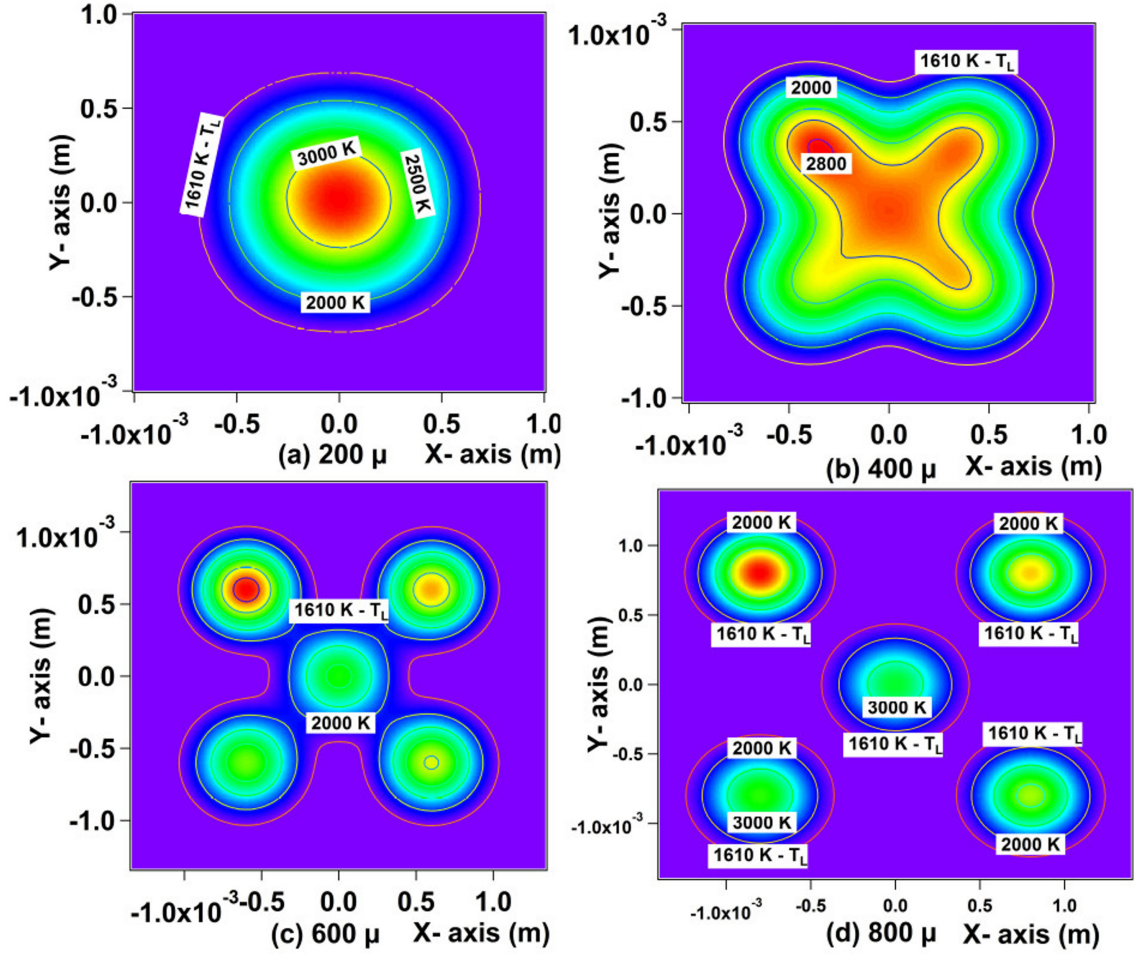


Figure 4.5: Size and shape of the top surface of the simulated met-pool as a function of internal point offset (a)200 μ m (b)400 μ m (c)600 μ m (d)800 μ m

Table 4.2: Properties of IN718 used in analytical equations to predict PDAS [106].

Parameter	Value	Unit
L	28	-
Partition Co-efficient k_p (Nb)	0.48	-
Diffusivity D	$3E-9$	m^2s^{-1}
Gibbs Thompson Co-efficient Γ	$1E-7$	-
Equilibrium Solidification Range ΔT_o	82	K
Non-Equilibrium Solidification Range ΔT	200	K

4.4.2 Variation in Primary Dendrite Arm Spacing

Theory

Hunt [28] derived an analytical formulation for the calculation of PDAS as a function of solidification conditions and alloy properties with an assumption that the dendrite tip can be approximated as a sphere. Trivedi [103] extended the model derived by Hunt [28] as shown in Equation (4.1). Kurz-Fisher et al. [104] derived a similar analytical formulation based on the Langer and Muller-Krumbhaar stability criterion [105] which assumes that the dendrite tip radius is equal to the wavelength of the instability at the liquid-solid interface. The simplified relationship derived by Kurz-Fisher et al. [104] is given in Equation (4.2).

$$\lambda = 2.83 * \left(\frac{1}{G}\right)^{0.5} * \left(\frac{\Delta T_o * L * k_p * D * \Gamma}{R}\right)^{0.25} \quad (4.1)$$

$$\lambda = 4.3 * \left(\frac{\Delta T}{G}\right)^{0.5} * \left(\frac{D * \Gamma}{R * k_p * \Delta T_o}\right)^{0.25} \quad (4.2)$$

where λ is the primary dendrite arm spacing, G is the thermal gradient at the liquid-solid interface, R is the velocity of the liquid-solid interface, ΔT_o is the temperature difference between equilibrium liquidus and solidus temperature of the alloy, ΔT is the non-equilibrium solidification range of the alloy, Γ is the Gibbs-Thompson co-efficient, D is the diffusion co-efficient, k_p is the partition co-efficient, L is the harmonic function of the perturbation. The material dependent properties in the above equations are listed in Table 4.2.

By applying known values for the variables in Equation (4.1) and Equation (4.2), the PDAS can be expressed as a function of just G and R . For IN718, the Trivedi and Kurz-Fisher models can be simplified to Equation (4.3) and Equation (4.4) respectively.

$$\lambda = 2145.952 * G^{-0.5} * R^{-0.25} \quad (4.3)$$

$$\lambda = 3195.214 * G^{-0.5} * R^{-0.25} \quad (4.4)$$

These analytical equations have been used by Lee et al. [106] to predict the PDAS in laser deposited IN718 and the experimental results were found to compare reasonably well with the theoretical prediction. The comparison of the predicted values from the theoretical models and experimentally measured values is discussed in the following section.

Experimental Observation

For IN718 in the Arcam[®] process, immediately after the solidification, the columnar dendritic structures formed with microsegregated elements (Mo, Nb) [107] between the dendrites. However, because the build chamber was held at an elevated temperature (1000°C) during the build, the segregated elements diffuse from the interdendritic region into dendrites [108, 107]. Immediately after the melting of the final layer completes, the chamber begins to cool. Figure 4.6 shows the experimentally measured temperature profile at the bottom of the substrate during fabrication of the samples.

Since rapid cooling commences instantaneously after melting the last layer, segregated elements in the interdendritic region of the melt pool do not have enough time to diffuse and homogenize. The diffusion kinetics of additively fabricated nickel-base superalloy (IN625) has been studied by Zhang et al.[108] using synchrotron. At 870°C, the segregated elements diffused completely in 900 seconds. Since the build for this work was done at 1050°C, the homogenization kinetics should be equal to or faster than the kinetics reported by Zhang et al.[108]. The time taken for fabrication of single layer was about 100 seconds and hence, microsegregation and dendritic structures was observed only in the top few layers (<10) of the samples. Accordingly, after etching, columnar dendrites were observed in the top layers

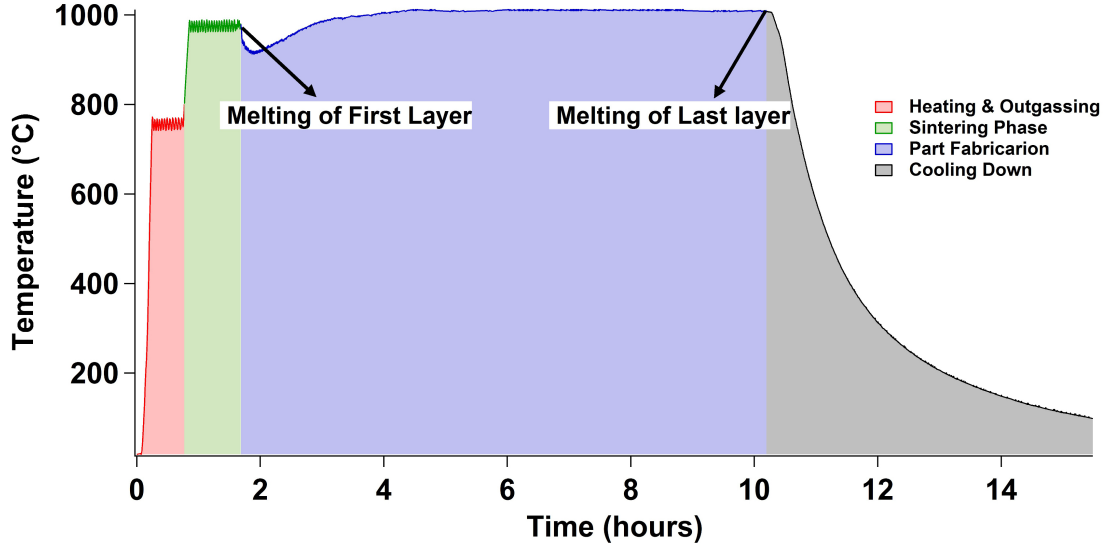


Figure 4.6: Experimentally Measured Temperature Profile of the bottom of the substrate during the build.

($\sim 500 \mu\text{m}$) and a homogenized, columnar microstructure in the rest of the layers of the fabricated sample.

Figure 4.6 shows the experimentally measured temperature profile at the bottom of the substrate during fabrication of the samples. Color coded regions shows the different stages of the Arcam[®] process. Figure 4.7 shows the same data points zoomed during the transition from sintering to melting of the first layer of the samples. The drop in temperature during initial few layers of the build can be attributed to radiative heat loss from the substrate since it is exposed to the ambiance. After few layers, most of the substrate is covered with sintered powder which has low thermal diffusivity and hence the radiative energy loss is minimized to a significant extent. Therefore, measured temperature at the bottom of the substrate stabilizes after initial few layers of the build.

Since rapid cooling commences immediately after melting of the last layer, segregated elements in the interdendritic region do not have enough time to diffuse and homogenize. Hence, the characteristics of segregation and dendritic structures can be observed only in the top layers of the samples. Figure 4.8 shows an example of variation in microstructure as a function of build height. Columnar dendrites were observed in the top layers ($\sim 500 \mu\text{m}$) and a homogenized, bulk columnar microstructure in the rest of the layers of the fabricated

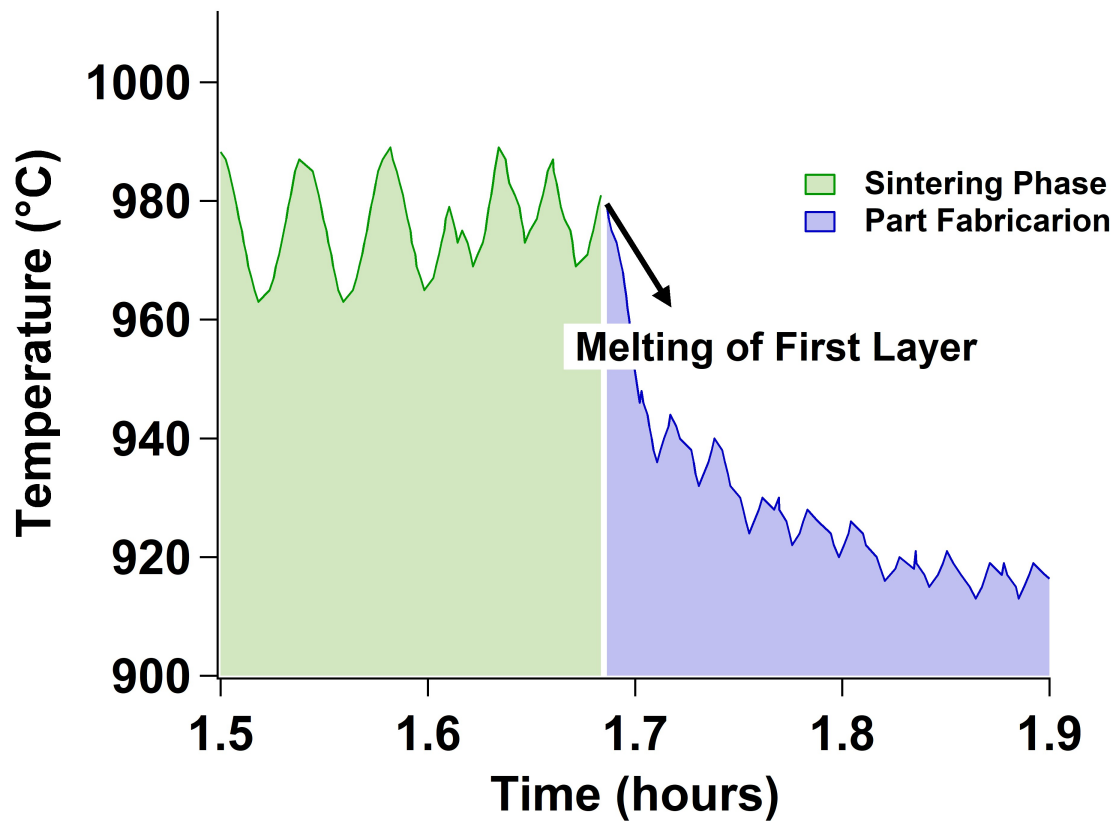


Figure 4.7: Experimentally Measured Temperature Profile during melting of the first layer.

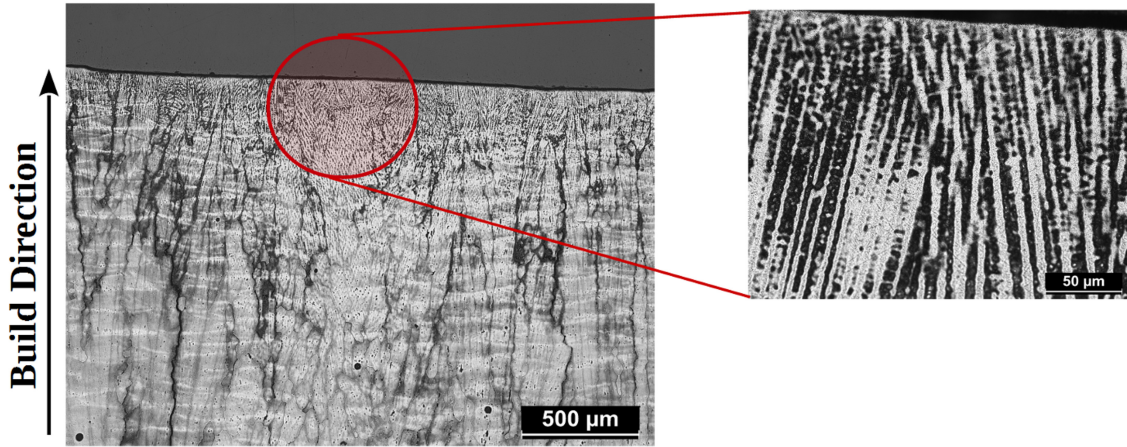


Figure 4.8: Visible dendritic morphology on top layers ($\sim 500 \mu\text{m}$) of the sample with internal point offset of $200 \mu\text{m}$.

sample. This phenomenon was observed in all the fabricated samples. The visible dendrites at the top most layers were used to measure the primary dendrite arm spacing (PDAS) of the samples fabricated with the proposed scan strategy. From Figure 4.8, it can also be observed that groups of primary dendrites are parallel to each other. The change in the dendritic growth direction between the groups can be attributed to change in the direction of the thermal gradient vector during solidification of overlapping sub-domains in the melt-scan strategy.

Numerical Prediction

From the simulated data, the liquid-solid interface was tracked during solidification and the spatiotemporal values of G and R were extracted for each of the 4 cases specified in Table 4.1. The PDAS was calculated based on Equation (4.3) and Equation (4.4). Figure 4.9 shows the spatial variation of calculated PDAS within the melt pool for the simulation with $200 \mu\text{m}$ internal point offset. The depth of the melt-pool ($\sim 450 \mu\text{m}$) can also be inferred from Figure 4.9. The thickness of the powder layer used in the process is $50 \mu\text{m}$. When the i^{th} layer is melted, the previously solidified melt pools of $(i-8)$ layers are re-melted. Hence, the simulation data from bottom $50 \mu\text{m}$ of the melt pool was used in all the upcoming calculations to provide the statistics of the solidification microstructure since they are not

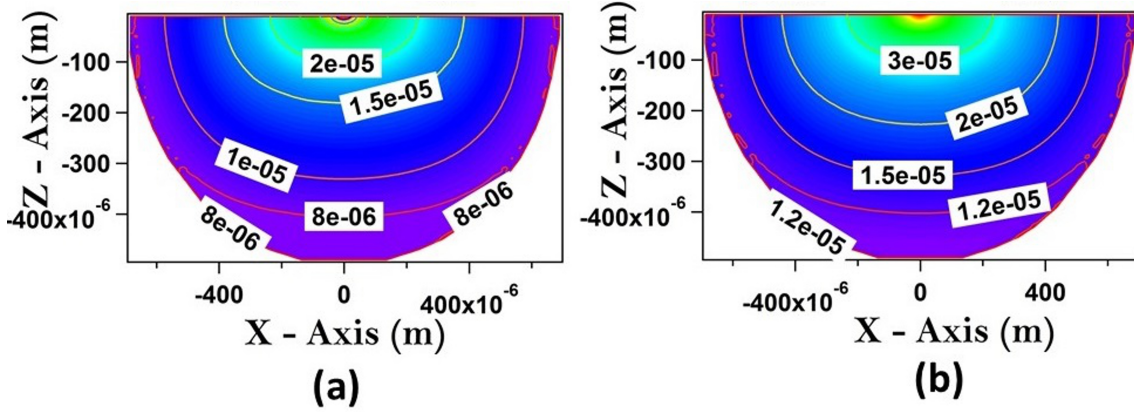


Figure 4.9: Variation (along XZ plane of melt pool) of PDAS predicted by the (a) Trivedi and (b) Kurz-Fisher model.

re-melted during subsequent layers. From Figure 4.9 (a),(b) it can be inferred that the PDAS predicted by the Trivedi and Kurz-Fisher models are about $8\text{ }\mu\text{m}$ and $12\text{ }\mu\text{m}$ respectively.

Figure 4.10 shows a colored scatter plot of the variation in PDAS as a function of G and R on a reference solidification map [97] for the columnar-to-equiaxed transition of IN718. Data used in Figure 4.9(b) is plotted as a function of G and R in Figure 4.10. Comparing the distribution of PDAS as a function of melt pool depth (Figure 4.9(b)) and solidification parameters (G & R Figure 4.10), it can be observed that the G and R of the bottom region of the metlpool lies within the $12\text{ }\mu\text{m}$ region in the scatter plot which is consistent with a columnar grain structure. Figure 3.4 shows the variation of G and R as a function of solidification time. Following the logic, the area of the map (Figure 4.10) in the equiaxed region corresponds to the top region in the melt pool which will be remelted by the subsequent layers. Hence, the model prediction is consistent with the observation of a columnar microstructure through the entire height of the sample.

Similarly, the PDAS was predicted for all 4 cases using the coupled numerical-theoretical model. Figure 4.11 shows the comparison of mean PDAS (experimental vs predicted) as a function of internal point offset. It can be observed that the PDAS varies from $10\text{ }\mu\text{m}$ to $4\text{ }\mu\text{m}$ when the internal point offset is varied from $200\text{ }\mu\text{m}$ to $800\text{ }\mu\text{m}$, respectively. The Trivedi model overestimates and the Kurz-Fisher model underestimates the PDAS prediction, but the trends are consistent with the experimental observations. The experimentally measured

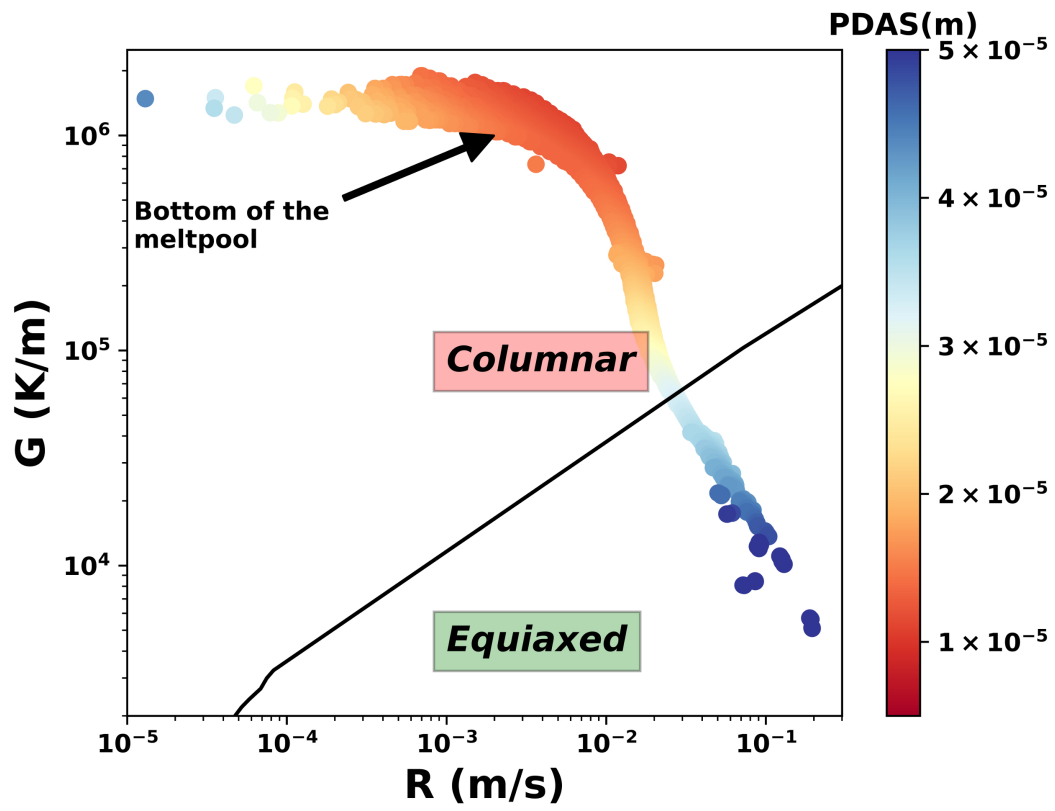


Figure 4.10: Variation of PDAS (Kurz-Fisher model) as a function of G and R on the reference solidification map for the internal point offset of $200 \mu\text{m}$.

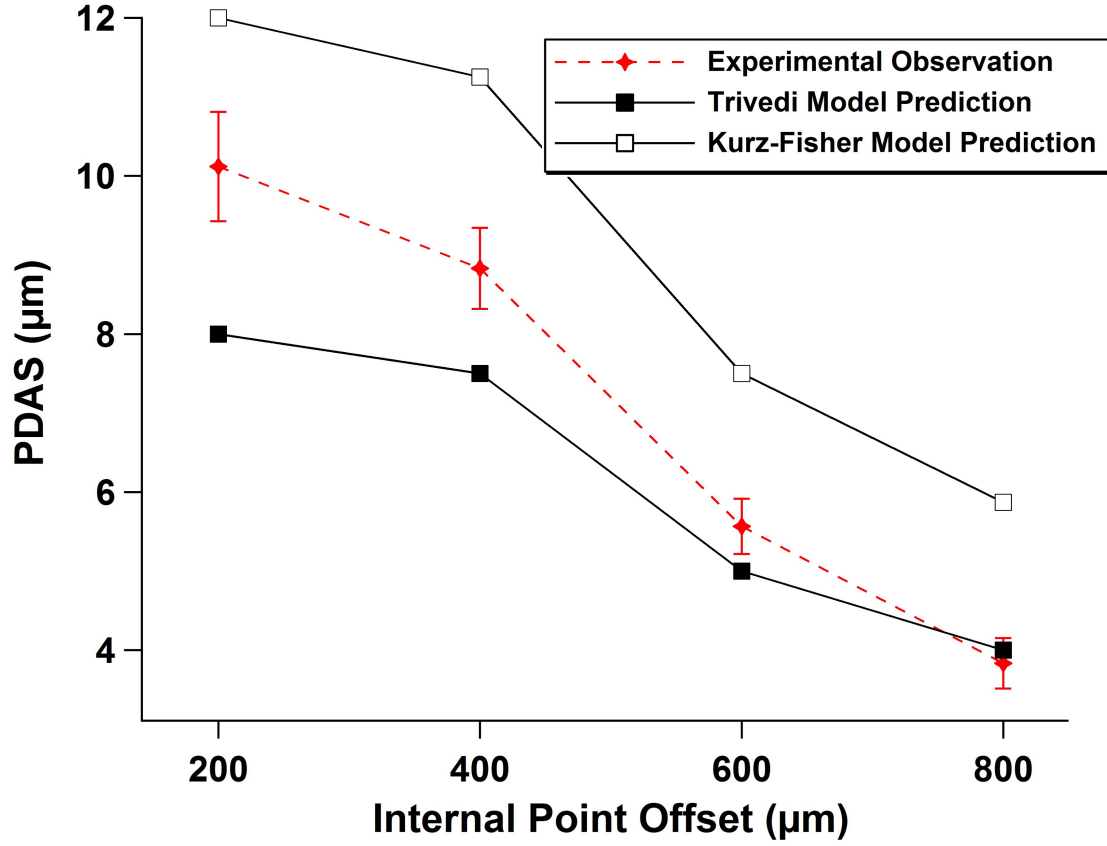


Figure 4.11: Comparison of numerically predicted and experimentally measured PDAS.

PDAS data is sandwiched between the two theoretical models but closer to the predictions of the Kurz-Fisher model.

4.4.3 Variation in Grain Size

Theory

During rapid solidification conditions in AM, the two most probable solidification mechanisms are (a) epitaxial growth and (b) heterogeneous nucleation and growth. During epitaxial growth, the shape of the melt pool plays a crucial role in determining the dendritic growth direction. This phenomenon may be analogous to formation of misoriented grains during the casting of single crystal or directionally solidified alloys. During solidification of single crystals, thermosolutal convection and fragmentation of grains causes the deviation of the primary dendrite growth direction with respect to the direction of the thermal gradient,

resulting in the formation of misoriented grains [109, 11, 110]. In this study, the deviation of primary dendrites from the build direction has been correlated to the shape of the melt pool. The shape of the melt pool is quantified based on the spatiotemporal distribution of the thermal gradient vector normal to the solid-liquid interface during solidification.

On the other hand, grain size may also be determined by the nucleation density within the liquid melt pool. In a temporal scale, nucleation density controls the nucleation rate. It is valid to assume that nucleation rate dictates the solidification grain size. Nucleation can be either (a) homogeneous or (b) heterogeneous. Heterogeneous nucleation during rapid solidification is primarily due to constitutional supercooling. Compositional gradient at the liquid-solid interface due to segregation affects the local equilibrium liquidus temperature (T_L) near the interface. Depending on the compositional gradient at the liquid-solid interface, G , R , and the amount of constitutional supercooling varies. For a steep concentration gradient at the interface, T_L at the interface is lowered. When the actual temperature gradient is lower than the temperature gradient at the interface due to spatial variation of T_L , constitutional supercooling occurs at the interface and promotes nucleation ahead of the solidification front. The volume rate of heterogeneous nucleation is defined by Equation (4.5) [111].

$$N_{het} = f * C * \exp\left(\frac{-\Delta G_{het}^*}{k * T}\right) \quad (4.5)$$

where N_{het} is the heterogeneous nucleation rate per unit volume, f a function of the surface area of the critical nuclei, vibrational frequency of atoms and the activation energy for diffusion in the liquid, C the number of atoms in contact with the nucleation sites per unit volume of the melt pool, and G_{het}^* the activation energy required to overcome the barrier for heterogeneous nucleation, which is a function of undercooling. Higher undercooling makes it easier to overcome the energy barrier for nucleation.

An empirical study by Uttormark et al. [112] analyzed the relationship between undercooling and nucleation rate in a molten spherical aluminum droplet. The nucleation rate of the aluminum droplet was observed to increase with an increase in undercooling. Yang et al. [113] extended the study by developing fast scanning calorimetry to understand the effect of cooling rate on the undercooling of a molten tin droplet. The observation proved that an increase in cooling rate increased the undercooling within the molten droplet. Hence, an increase in the cooling rate of the melt pool increases the undercooling which in turn increases the nucleation density resulting in finer grains. Equation (4.6) can be derived (see Appendix A) from Equation (4.5) to show the analytical relationship between undercooling and cooling rate $\left(\dot{T}\right)$ with an assumption that the melt pool is a spherical droplet.

$$\frac{C_1}{\dot{T}} \int_0^{\Delta T} \frac{1}{T^2} * \exp\left(-\frac{C_2}{(T_m - T) T^2}\right) dT = 1 \quad (4.6)$$

where C_1 and C_2 are material constants and T_m is the melting point.

Hence, the nucleation rate can be controlled by the controlling the cooling rate. The cooling rate of the melt pools in the proposed melt strategy can be predicted. Variation in the predicted cooling rates and corresponding experimental grain size variation are discussed in the next section.

Experimental Observation

EBSD characterization was used to understand the size and morphology of the grains. Figure 4.12 shows the EBSD data depicting the variation in the size of the columnar grains along the build direction for each melt-scan strategy.

The grains are coarser in sample 1 when the 5 spots merge together to form a single melt pool. The grains become finer as the melt pools move away from each other, i.e. as the internal point offset distance is increased from 200 μm to 800 μm . The grains are not only wider in sample 1 but also longer compared to sample 4, effectively reducing the grain boundary area per unit volume of the sample, which may influence the mechanical behavior of the component.

Figure 4.13 shows the quantitative variation in grain size with respect to the internal point

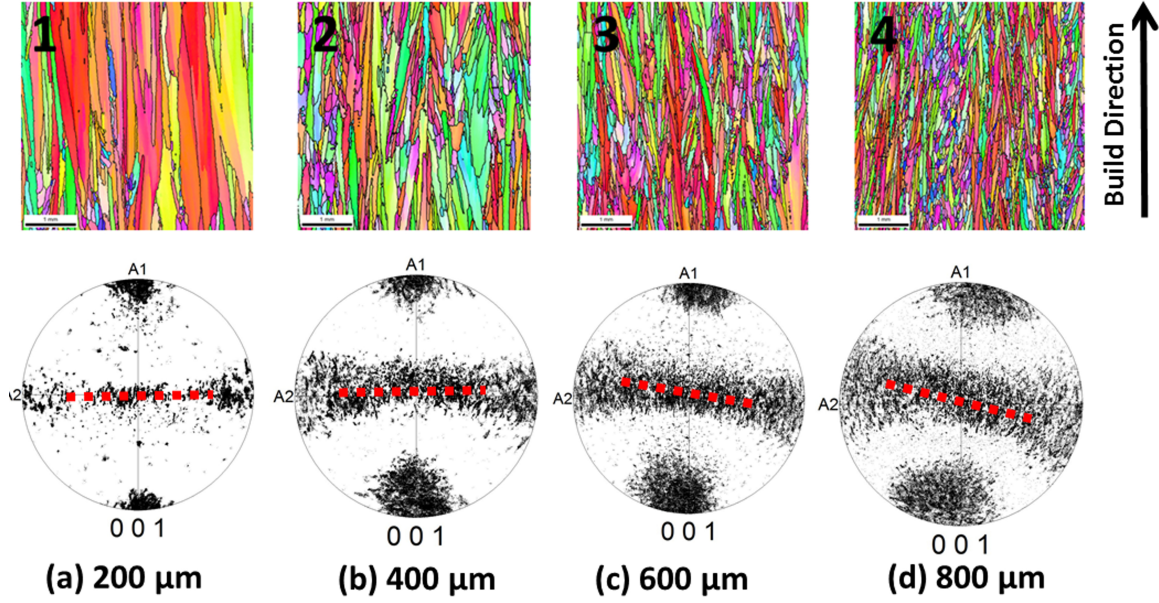


Figure 4.12: Variation in grain size and corresponding qualitative texture plot as a function of internal point offset.

offset. Even though the shape and size of the top surface of the melt-pools from samples 1 and 2 look similar (Figure 4.4), the resultant microstructure (Figure 4.12) is significantly different. With the help of numerical modeling, Debroy et al. [98] showed that the subsurface solidification behavior of two similarly shaped melt pools can be significantly different even though their surfaces look similar. Current results reiterate the above argument and calls for further investigation using modeling. The pole figures in Figure 4.12 also indicate that the tilt of the grains with respect to build direction increases as the internal point offset increases, which may be due to the change in the resultant melt pool shape (3-D). As mentioned earlier, the grain size variation is rationalized considering two solidification phenomenon (a) epitaxial growth (b) heterogeneous nucleation and growth ahead of the solidification front due to undercooling.

Epitaxial Growth

Considering epitaxial growth, the variation in grain size is attributed to changes in the dendritic growth direction. The dendritic growth direction was quantified using the

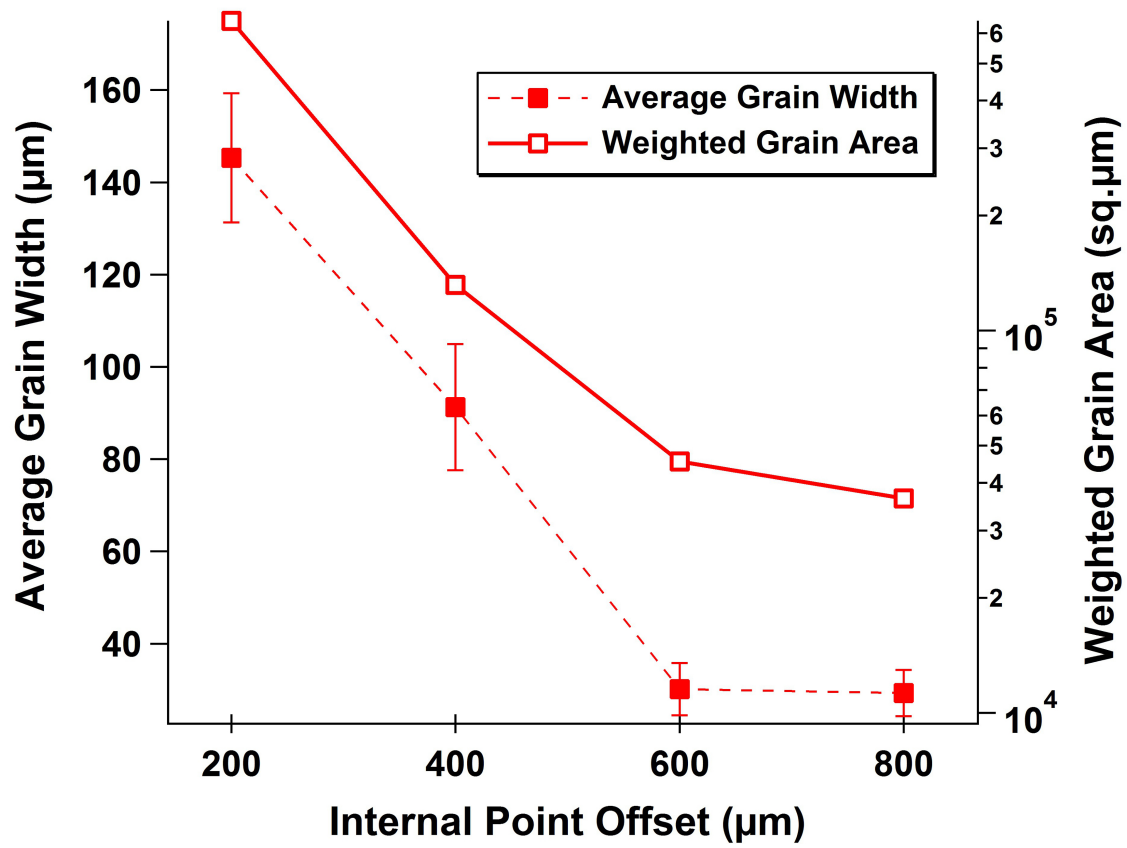


Figure 4.13: Variation of experimentally measured grain size and area as a function of internal point offset in the proposed melt strategy.

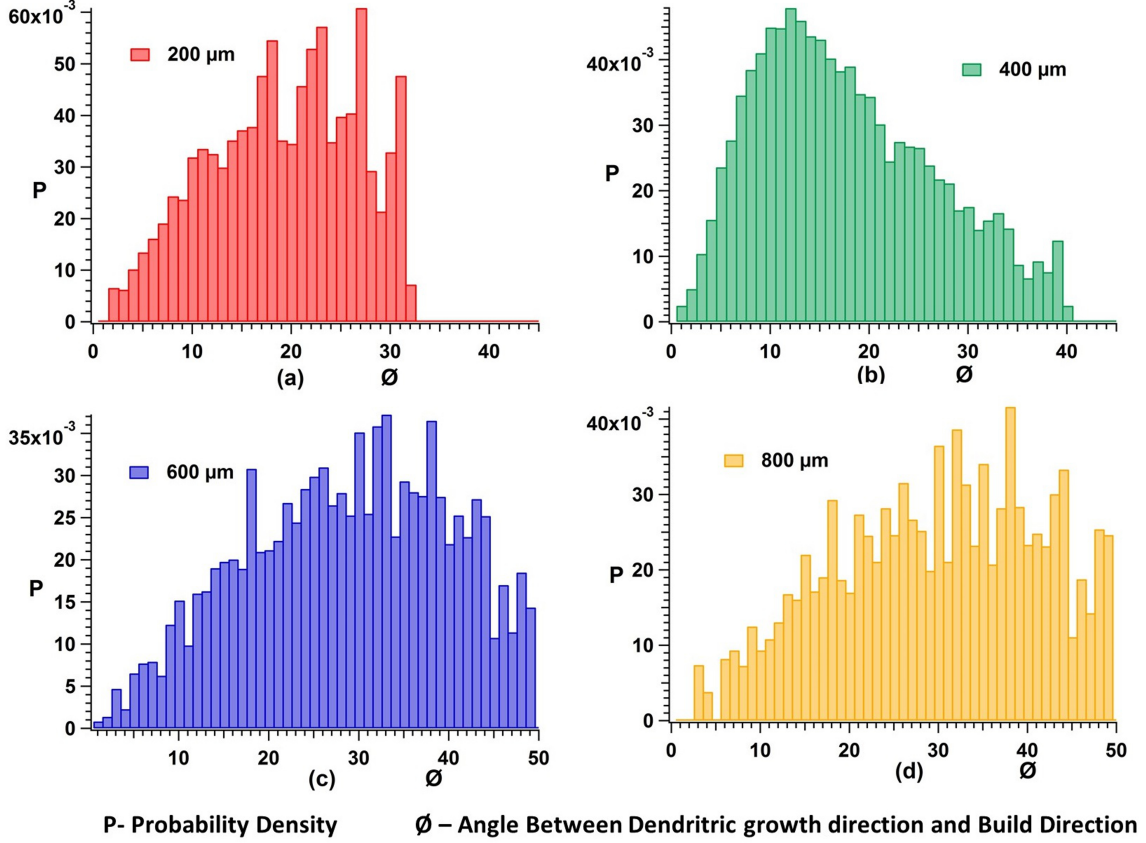


Figure 4.14: Distribution of dendritic growth direction of the bottom 50 µm of the melt pool.

spatiotemporal variation of direction of temperature gradient at the liquid-solid interface during solidification. The growth direction was calculated based on Equation (4.7).

$$\theta = \cos^{-1} \left(\frac{G_z}{G_{resultant}} \right) \quad (4.7)$$

where θ is the angle between the resultant thermal gradient vector ($G_{resultant}$) at the solid-liquid interface and the z-component of the thermal gradient vector (G_z) which is aligned with the build direction. Like the PDAS calculations, the data was filtered to retain only the bottom 50 µm of the melt pool. Histograms of the distributions of calculated solid-liquid interface growth directions relative to the build direction as calculated from the simulations are shown in Figure 4.14.

The curvature at the bottom 50 µm of the simulated melt pools is quantified in Figure 4.14 and it can be observed that for the simulation with internal point offset of 200 µm (coarser

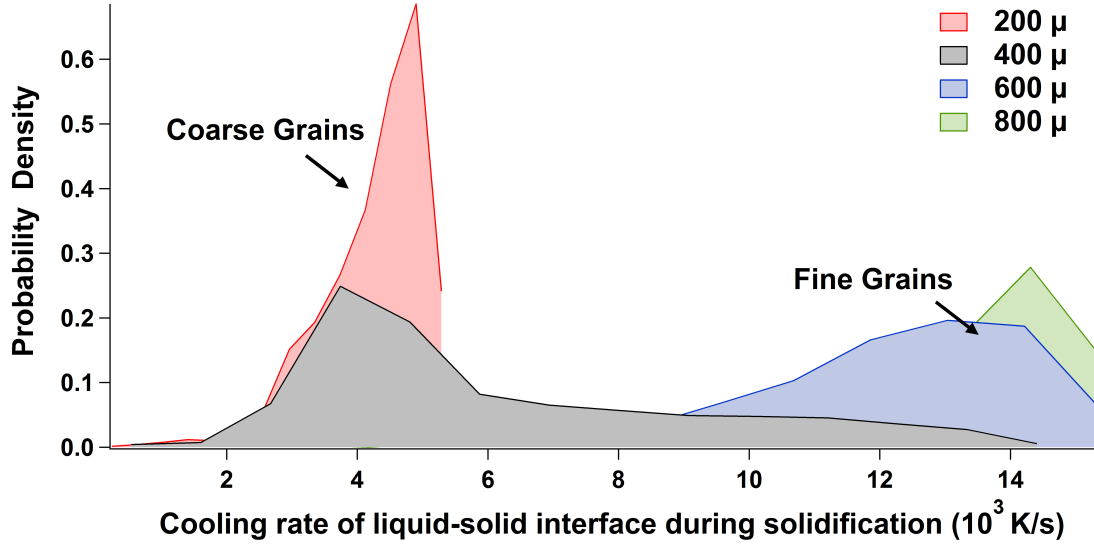


Figure 4.15: Variation of cooling rate with respect to internal point offset.

grains - 1 merged melt pool), the curvature is smaller and the dendrites are well-aligned with the build direction compared to the case with 800 μm internal point offset (finer grains - 5 independent melt pools). Hence, introducing the curvature in the resultant melt pool promotes larger mismatch between the resultant thermal gradient and the fast-growth $\langle 001 \rangle$ orientations of the pre-existing grains that solidified during the production of previous layers.

Heterogeneous Nucleation and Growth

Changes to the internal point offset also corresponds to significant changes in the cooling rate during solidification. The spatiotemporal variation in cooling rate for different conditions was calculated from the simulations and the probability density distribution is plotted in Figure 15. It can be observed from Figure 4.15 that the cooling rate of the merged melt pool (point offset: 200 μm) is smaller than the 5 independent melt pools (point offset: 800 μm). The cooling rate increases from $2 * 10^3 K/s$ to $15 * 10^3 K/s$ if the internal point offset is increased from 200 μm to 800 μm respectively. The cooling rate of the extreme cases differs by a factor of 3-4.

The higher cooling rates for individual melt pools (600 μm and 800 μm spacing) shown in Figure 4.15 will tend to increase curvature undercooling at the dendrite tips, producing an increased driving force for heterogeneous nucleation [113, 112, 25]. However, this alone

does not explain the production of highly textured, fine-grained columnar structures as shown in Figure 4.12, because if it was the case that heterogeneous nucleation is favored under these conditions, then a weakly textured equiaxed structure would be expected. To rationalize these results, it is necessary to also consider the effect of the distribution of angles between the resultant thermal gradient and build direction as shown in Figure 4.14. Consider a simple case in which the grains from the previous layer have their $\langle 001 \rangle$ fast-growth directions perfectly aligned with the build direction. In this case, the distributions of the angles between the thermal gradients and the build direction (Figure 4.14) is also a description of the misorientation between the thermal gradient vector and the dendrite growth direction. For epitaxial growth to continue under these conditions, the component of the dendrite tip velocity aligned with the thermal gradient must equal the velocity of the liquidus isotherm. The dendrite tip velocity in the $\langle 001 \rangle$ direction may then be expressed as a function of the solid-liquid interface velocity (R) and the angle between the $\langle 001 \rangle$ direction and the thermal gradient [114].

$$R_{\langle 001 \rangle} = \frac{R}{\cos \theta} \quad (4.8)$$

Due to this relationship between the thermal gradient vector and the $\langle 001 \rangle$ crystallographic direction, larger misorientations result in higher dendrite tip velocities. The undercooling of the dendrite tips increases with the tip velocity, and consequently, with this misorientation angle, creating a region with an increased driving force for heterogeneous nucleation. If indeed nucleation takes place under these conditions, it is expected that the newly nucleated grains would undergo a brief period of competition, resulting in the selection of those grains that have a $\langle 001 \rangle$ type crystallographic direction most closely aligned with the thermal gradient vector. These grains will therefore grow with a smaller amount of undercooling, reducing the thermodynamic driving force available for heterogeneous nucleation. The consequence of this mechanism is therefore the nucleation of a small number of equiaxed grains at the edge of the re-melted region, followed by a transition back to columnar growth more closely aligned with the thermal gradient vector. Larger misorientations and higher cooling rates will result in a higher heterogeneous nucleation rate,

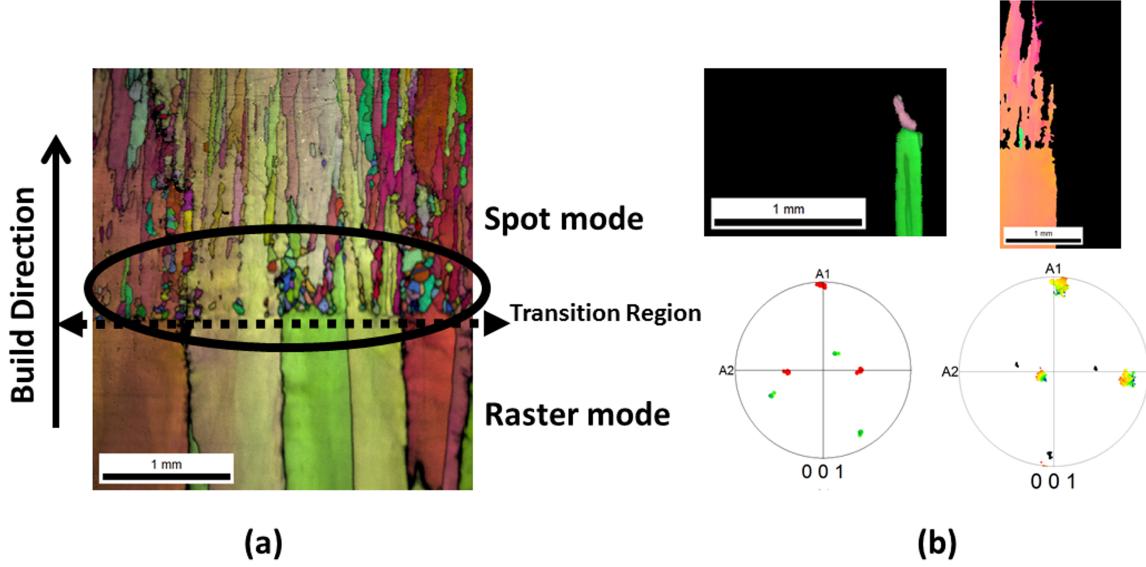


Figure 4.16: Evidence for heterogeneous nucleation by changing the scan strategy along the build direction.

and therefore, a smaller grain size, consistent with the observations in textured, fine-grained columnar structures as shown in Figure 4.12 and the simulated distribution of thermal gradient angles in textured, fine-grained columnar structures as shown in Figure 4.14.

To test this theory, an experiment was designed in which the melt strategy was changed along the height of the build. A sample of 20 mm was fabricated with the bottom half (10 mm) of the sample using a raster scan strategy (0.1 mm hatch spacing) and the top half (10 mm) of the sample fabricated using a spot melt strategy (20 mA, 0.25 ms). From previous experience, the raster scan strategy was expected to result in comparatively coarse columnar grains. The occurrence of coarser columnar grains ($600 \sim 700 \mu\text{m}$) may be due to comparatively flatter bottom surface of the melt pool and therefore, thermal gradients that are more uniformly aligned with the build direction. Alternatively, the spot melt-scan strategy was expected to produce fine columnar grains due to large deviations in the direction of the thermal gradients from the build direction. This experiment was designed to investigate the possibility of heterogeneous nucleation at the transition region due to changes in the thermal gradient relative to the $\langle 001 \rangle$ direction of the pre-existing columnar grains.

Figure 4.16(a) shows the inverse pole figure of the fabricated sample along the build direction. At the transition region from raster to spot mode, evidence for grain refinement

can be observed. To obtain evidence for heterogeneous nucleation in the constitutionally cooled region, which can be challenging, pairs of grains were chosen at the transition region and checked for any common $\langle 001 \rangle$ poles. Figure 4.16(b) shows that there are no common $\langle 001 \rangle$ poles, eliminating the possibility of epitaxial growth in these cases. It appears that at the transition point, heterogeneous nucleation occurred, creating a small number of equiaxed grains, which then competed to return to columnar growth. The pole figures in Figure 4.16(b) show that pairs of grains observed at the transition region of the sample did not have common $\langle 001 \rangle$ poles, supporting the conclusion that these grains nucleated heterogeneously. The results in Figure 4.16 support the theory that mismatch between the direction of the thermal gradient and the $\langle 001 \rangle$ growth direction of the previously existing grains requires a large enough undercooling to enable heterogeneous nucleation to take place. Even though the result in Figure 4.16 supports the argument, it may not be the conclusive proof since the grains may have grown from out of plane as well. This theory is also consistent with results observed by Helmer et al. [77] in which periodic changes in the direction of the raster pattern produce highly textured columnar grains of different lengths due to changes in the orientation of the resultant thermal gradient.

The evidence presented in Figure 4.16(b) is only a simplified example of the phenomenon discussed above. In this case, the raster pattern used for the bottom 10mm of the build produced a coarse columnar structure with grains whose $\langle 001 \rangle$ directions are highly oriented with the build direction. However, when solidifying new layers on existing grains with more textural variation (Figure 4.12) the relationship between the thermal gradient and the epitaxial growth directions is significantly more complex. Modeling of the resultant thermal gradients across several subsequent layers is necessary to fully understand these effects and may necessitate the use of microscale numerical models for grain nucleation and growth [115].

4.5 Summary

- A new scan strategy was developed to control the size of the columnar grains and the primary dendrite arm spacing. The experimental observations were rationalized using the numerical modeling tool.
- To rationalize the variation in grain size, two of the most probable solidification mechanisms (epitaxial growth and heterogeneous nucleation) were considered. The thermodynamic driving force available for heterogeneous nucleation was correlated to the local solidification conditions as well as the shape of the resultant melt pool by calculating the dendritic growth direction with respect to build direction. The key factor deciding the grain size was inferred to be the curvature of the resultant melt pool. Increasing the curvature of the melt pool promotes finer grains by increasing the undercooling available for heterogeneous nucleation due to misorientation between the resultant thermal gradient and pre-existing $\langle 001 \rangle$ directions of grains from previous layers. The grain size variation is attributed to the nucleation rate which is a function of both the cooling rate and melt pool curvature through undercooling. Hence, controlling the geometry of the resultant melt pool of a layer plays an important role in the development of the grain structure.
- A simple experiment was designed and analyzed to show the evidence of heterogeneous nucleation and examine the effect of mismatch between the thermal gradient and epitaxial growth direction. The experimental results support a mechanism based on heterogeneous nucleation followed by grain growth competition and the establishment of new columnar grains with $\langle 001 \rangle$ crystallographic directions that are highly oriented with the thermal gradient.
- One of the advantages of the proposed melt-scan strategy is that it is localized and allows for consistent solidification microstructure across the build. Localized solidification control partially decouples the effect of geometry and scan strategy on the solidification microstructure. Hence, the same parameters may be used across geometries, provided the geometry is larger than the size of the melt pool.

Chapter 5

Evaluation of Columnar to Equiaxed Transition (CET)

5.1 Motivation

This chapter investigates the feasibility of columnar to equiaxed transition during solidification of Inconel 718 in electron beam AM process. Components fabricated in metal AM process tend to have highly textured columnar grains along the build direction [71, 72, 73, 74]. Feasibility of controlling the solidification grain morphology adds another dimensional freedom to the AM process. As discussed in Chapter 3, the grain morphology of metals during rapid solidification processes is dictated by the thermal gradient (G) and the velocity or growth rate (R) at the liquid-solid interface of the melt pool. The G and R can be controlled by controlling the process parameters during fabrication. In general, for any powder bed metal AM process, multiple input parameters affect the solidification behavior. The availability of numerous controllable input parameters (power, velocity, scan strategy, layer thickness, etc) in EBM process means that the control strategy becomes enormous. Hence, experimental verification and validation of multiple input parameters becomes an expensive process in terms of cost and time. To reduce the experimental expense, in this chapter, the numerical modeling and statistical analysis is done apriori to the experimental investigations. Results in this chapter are published in the journal [116].

5.2 Qualitative Analysis of Parameters

Based on the prior published literature by Dehoff et al.[78], spot melt scan strategy was chosen in place of raster melt scan strategy to investigate the bulk columnar to equiaxed transition. Four process parameters of the spot melt scan strategy were selected for the comprehensive numerical experimentation. The chosen controllable input process parameters were

- Electron beam diameter
- Electron beam current
- Spot ON time
- Preheat temperature

The chosen process parameters are applicable to each of the spots in the melting strategy. Since electron beam accelerating voltage is constant (60 kV), the electron beam current was used as the variable to change the power deposited at a spot. Spot ON time is the dwell time of the electron beam at a particular spot. The preheat temperature is the temperature of the substrate or region nearby to that particular spot. This factor cannot be explicitly controlled like electron beam ON time and diameter but can be controlled by coupling it with the scan strategy. This is explained in the later part of this chapter.

As explained in Section 3.1.8, Truchas was used to simulate a single spot with beam ON time of 1 ms, preheat temperature of 1528 K (solidus) and beam current of 20 mA. The G and R data were extracted at each of the nodal elements in the spatial domain as explained in Section 3.1.8. The extracted data in Figure 3.4 were the plotted on a reference solidification map of IN718. Figure 5.1 shows the G vs R plot on a reference solidification map [97] of IN718. From Figure 5.1, it is evident that grain morphology is columnar as the melt pool begins to solidify and it moves towards mixed region during solidification and results in an equiaxed region at the end of solidification.

To do a qualitative analysis of the effect of the process variables on the distribution of G and R, simulations have been performed for different values of the input variables which were

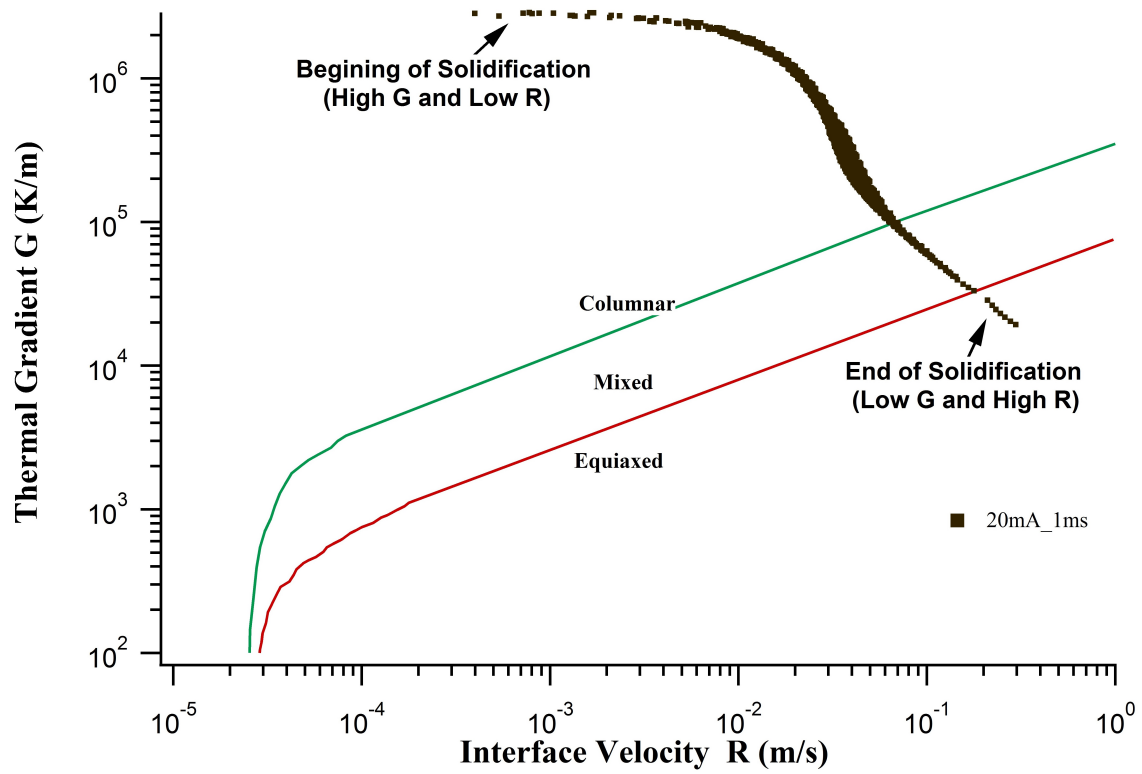


Figure 5.1: G vs. R plotted on reference solidification map [97] for the simulation with beam current of 20 mA and ON time of 1 ms.

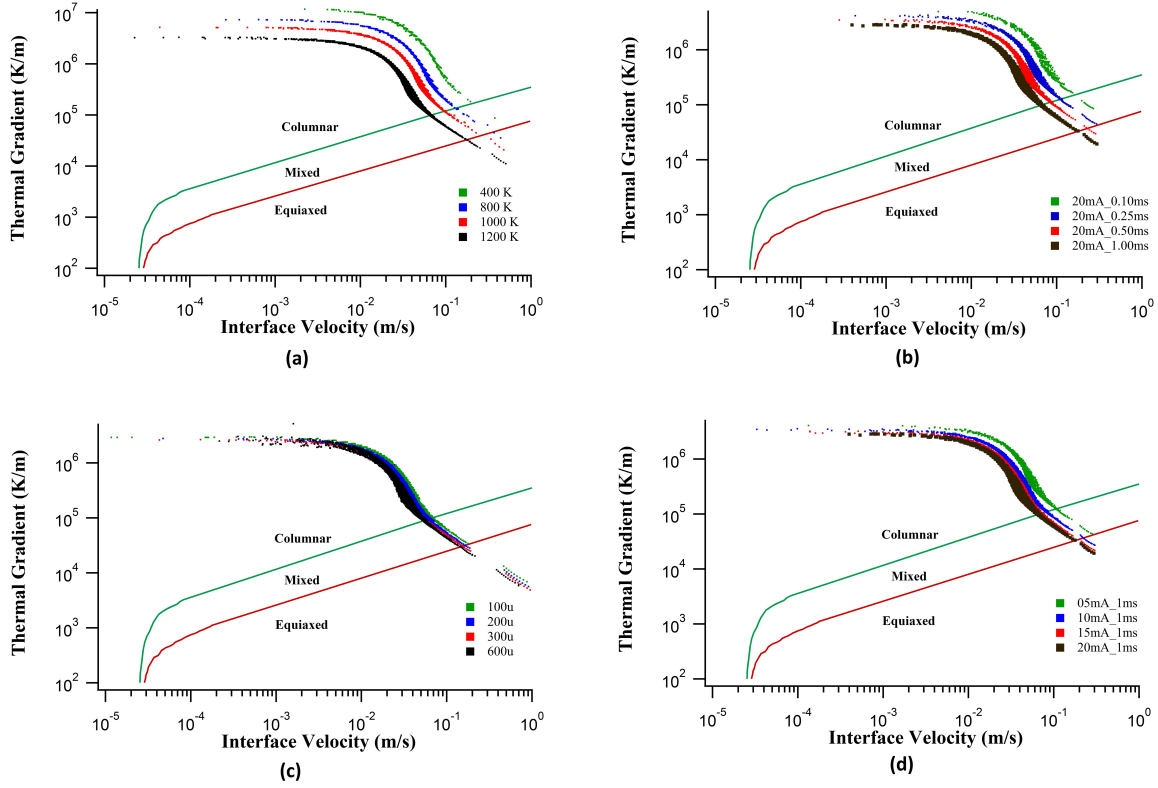


Figure 5.2: Variation of temperature gradient (G) and liquid-solid interface velocity (R) of spot melt profile as a function of (a) preheat temperature, (b) spot ON time, (c) beam diameter, and (d) spot beam current.

changed one by one while the value of other input variables were kept constant. For example, 4 different preheat conditions have been simulated while keeping beam current, diameter and ON time constant for all 4 simulations. In a similar manner, 12 more simulations have been performed for rest of the variables. Values of the variables used in the simulations are

- Electron beam diameter - 200 μm , 400 μm , 600 μm , 800 μm
- Electron beam current - 5 mA, 10 mA, 15 mA, 20 mA
- Spot ON time - 0.1 ms, 0.25 ms, 0.5 ms, 1 ms
- Preheat temperature - 400 K, 800 K, 1000 K, 1200 K

Figure 5.2 shows the spatio-temporal variation of G and R as a function of different values of 4 different beam input parameters.

From Figure 5.2, it can be concluded that increasing the preheat temperature, beam current, spot ON time and beam diameter moves the microstructure map towards the equiaxed region from the columnar region by reducing the temperature gradient. Increasing the energy input by increasing the beam power and ON time effectively moves the grain morphology towards the equiaxed region, which is in accordance with the results shown by Bontha et al. [76]. The sensitivity of the temperature gradient (G) and the liquid-solid interface velocity (R) with respect to different parameters is shown in Figure 5.2. In order for robust understanding of the columnar to equiaxed transition (CET) during the solidification of the melt pool, it is important to quantify the influence of the beam input parameters on G and R .

5.3 Quantitative Analysis

5.3.1 Theory

An analytical model for the columnar to equiaxed transition in casting processes was developed by Hunt [117] which provided relationship between the thermal gradient (G), interface velocity (R) and volume fraction of equiaxed grains (Φ) formed during solidification. Gäumann et al. [32] extended the theory using Kurz-Giovanola-Trivedi (KGT) model for rapid solidification processes by neglecting the nucleation undercooling (ΔT_n) at high thermal gradients ($\sim 10^6 K/m$). The underlying thermodynamics and kinetics of the columnar to equiaxed transition were simplified by Gäumann et al. [32] as formulated in Equation (5.1).

$$\left(\frac{G^n}{R}\right) = a \left(\left(\frac{-4 * \pi * N_o}{3 \ln(1 - \Phi)} \right)^{\frac{1}{3}} \frac{1}{n + 1} \right)^n \quad (5.1)$$

where $\mathbf{G}$ is the temperature gradient, $\mathbf{R}$ is the velocity of the liquid-solid interface, N_o is the nucleation density, Φ is the volume fraction of equiaxed grains or probability of stray grain formation, $\mathbf{n}$ and $\mathbf{a}$ are the alloy constants. Nucleation density (N_o) depends on composition of the alloy and undercooling. Higher the value of N_o , higher the probability of formation of equiaxed grains during the solidification. Analytical calculation of the value of N_o is complex and beyond the scope of this research work. In the literature [34, 118, 32], the value of N_o

was experimentally calibrated and optimized. Different values are being reported for the same alloy system [34, 118, 32]. Values of a , n and N_o assumed in the results section of this paper are 1.25×10^6 , 3.4 and 2×10^{15} respectively as reported by Gäumann et al. [32] and Vitek [34] for a similar nickel-base superalloy. By rearranging Equation (5.1) and applying the value for constants, the ratio G^n/R can be directly correlated to the volume fraction of equiaxed or stray grains (Φ) formed during the solidification of the melt pool [119].

$$\Phi = 1 - \exp\left(-2.36E19 * \frac{R^{3/3.4}}{G^3}\right) \quad (5.2)$$

From Equation (5.2), it can be noted that, for a given material system, the value of Φ depends only on the values of G and R .

5.3.2 Calculation of Spatio-temporal Φ

Quantitative analysis can be done by calculating the volume fraction of equiaxed grains formed during solidification for different cases of simulated melt pool. The volume fraction of equiaxed grains, or the probability of stray grain formation (Φ), can be calculated at every time step and at every nodal element of spatial domain of the liquid-solid interface using Equation (5.2). The histogram plot in Figure 5.3 shows the non-dimensional volume (frequency of nodal elements) vs. the probability of stray grain formation (Φ). It can be observed from Figure 5.3 that the volume fraction of equiaxed grains in the solidified melt pool can be changed from less than 10% (Figure 5.3(a)) to more than 90% (Figure 5.3(b)) by changing the beam input parameters.

For quantitative analysis of the effect of input parameters on the volume fraction of stray grains, design and analysis of experiments (DOX) [120] approach was used. A full factorial model was developed with all the 4 input parameters with 2 levels for each of the factors ($2^k = 2^4$ simulations). To do the analysis, the response variable has to be a single value. So, volume weighted average was used to calculate the weighted average probability of stray grain formation in the entire melt pool. The volume fraction of equiaxed grains (Φ) was used as the response variable in the design. Formulation to calculate the response variable is given in Equation (5.3).

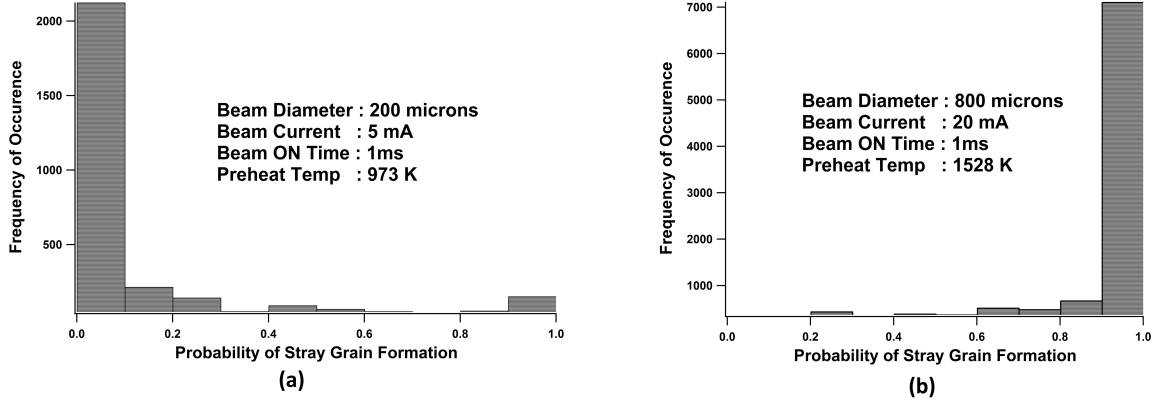


Figure 5.3: Change in volume fraction of equiaxed grains (Φ) or solidification morphology with respect to different input parameters.

$$\Phi = \frac{\sum v_i \phi_i}{\sum v_i} \quad (5.3)$$

where v_i is the volume of the discretized nodal element in the spatial domain ($dx*dy*dz$) and ϕ_i is the probability of stray grain formation at the corresponding nodal element calculated using Equation (5.2).

5.3.3 ANOVA

The input variables and the parameter window within the practical limitations are listed in Table 5.1

Table 5.1: Values of input parameters of proposed scan strategy

Parameter	Minimum	Maximum
Electron Beam Diameter (μm)	200	800
Electron Beam Current (mA)	5	20
Spot ON time (ms)	0.1	1
Preheat Temperature (K)	973	1528

Simulations were performed for all 16 possible combinations of parameters and weighted volume average of Φ was calculated for each of the simulations. The response variable (Φ) calculated from the simulations for the statistical design is given in Table 5.2.

Table 5.2: 2^4 simulation combinations and the corresponding volume fraction of equiaxed grains used in DOX

Case	A Beam Diameter (μm)	B Beam Current (mA)	C Beam On Time (ms)	D Preheat Temperature (K)	Response Variable Φ (%)
1	200	5	0.1	973	13.7
2	200	5	0.1	1528	57.5
3	200	5	1	973	15.9
4	200	5	1	1528	75.3
5	200	20	0.1	973	15.8
6	200	20	0.1	1528	67.8
7	200	20	1	973	20.6
8	200	20	1	1528	86.0
9	800	5	0.1	973	14.1
10	800	5	0.1	1528	58.8
11	800	5	1	973	20.4
12	800	5	1	1528	76.9
13	800	20	0.1	973	17.4
14	800	20	0.1	1528	68.2
15	800	20	1	973	22.9
16	800	20	1	1528	88.1

Analysis of variance (ANOVA) approach was used to quantify the effect of each input parameter on the response variable (Φ). The correlation between the input parameters and the response variable was studied in detail.

Statistical significance of the individual parameters on the response variable was understood by analyzing the variance (ANOVA) of the design as shown in Table 5.3. The statistical validity of the model was confirmed with the adjusted R-squared value of the design greater than 98%. The significance of each of the input parameters can be explained by their corresponding F-values listed in Table 5.3. The higher the F-value of the parameter, the greater its influence on the response variable (Φ). Factors with F-value less than 4 are deemed statistically insignificant. The F-values of the parameters indicate that the beam diameter is insignificant (F-value=2.12) and that preheat temperature (F-value = 2012.99) is the most significant in affecting the volume fraction of equiaxed grains (Φ) formed during

Table 5.3: Analysis of Variance (ANOVA) of the design

Source	Sum of Squares	Degrees of Freedom	Mean Square	F-Value	p-value Probability > F
Model	12904.19	5	2580.84	433.68	< 0.0001
A-Beam Diameter	12.60	1	12.60	2.12	0.1763
B-Beam Current	183.6	1	183.6	30.85	0.0002
C-Beam ON Time	538.4	1	538.4	90.45	< 0.0001
D-Preheat Temperature	11979.30	1	11979.30	2012.99	< 0.0001
C*D	190.44	1	190.44	32.00	0.0002

the solidification. Quantitative relationship between the input parameters and the output response variable (volume fraction of equiaxed grains) was obtained using DOX approach which is formulated in Equation (5.4).

$$\Phi (\%) = -72.07 + 0.4517 * B - 21.6595 * C + 0.083408 * D + 0.027628 * CD \quad (5.4)$$

where **B** is beam current (mA), **C** is beam ON time (ms) and **D** is the preheat temperature (K). Local preheat temperatures can be maintained high by modifying melt strategies and keeping the entire layer close to the solidus temperature of the alloy. The design of scan strategy and experimental validation of simulation results is reported in the following section.

5.4 Experimental Validation

5.4.1 Scan Strategy and Fabrication

To validate the modeling results, bulk samples of dimension 2 cm x 2 cm x 2 cm were fabricated with IN718 powder using Arcam[®] S12 machine. As mentioned earlier, a novel spot melting strategy was developed and used instead of a standard raster melt pattern. The qualitative difference between spot and raster melting is explained in Figure 5.4. Figure 5.4(a) depicts a standard raster melt pattern in which the electron beam moves linearly to fill the space, as shown by the lines and arrows. Figure 5.4(b) depicts the spot melting pattern used to fabricate the samples to validate the model. In spot melting, the electron beam is turned on at a point with a specified current for a period of time (beam ON time), as indicated in Table 5.1. Once the time period exceeds the defined beam ON time, the beam is moved to a new spot according to the sequence shown in Figure 5.4(b). Once the entire layer is filled with the independent spots (1-9), the next spot (10) is placed, overlapping the first spot, and the 11th spot is placed beside the 2nd spot, making it independent of the 10th spot, and so on. The spot filling pattern shown in Figure 5.4 (b) is purely qualitative. The number of spots per unit length and number of spots to skip in X and Y direction changes with respect to the fabricated samples. The spot filling continues in both the horizontal and vertical directions until the entire 2D layer (2 cm x 2 cm) is completely melted. Subsequently, the build platform is lowered and the melt pattern continues for the next 2D layer. Each layer is 50 microns thick and the samples consist of 400 layers (2 cm) in total.

A total of 16 samples (Figure 5.5(a)) were fabricated with varying beam current (5-20 mA) and beam ON time (0.05-0.25 ms). Figure 5.5(b) shows the corresponding energy deposited (kJ) per layer of the samples.

5.4.2 Insignificance of Beam Diameter

The significance of beam diameter on grain morphology was experimentally analyzed by fabricating two samples using spot scan strategy (Figure 5.4(b)) with same beam current (20 mA) and beam ON time (1 ms) but different beam diameters. The electron beam in

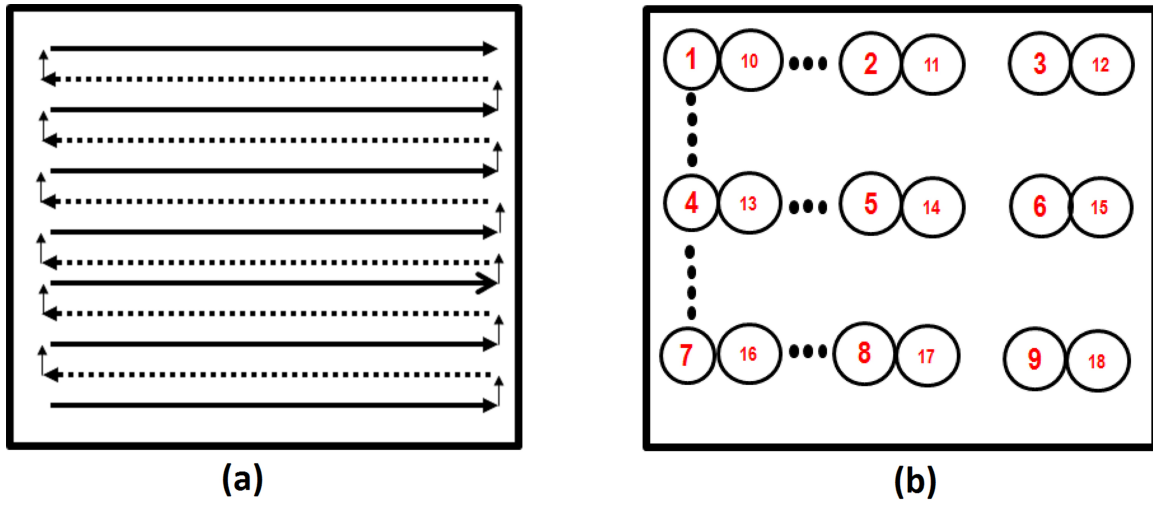


Figure 5.4: Qualitative comparison of (a) raster scan and (b) spot melting strategy

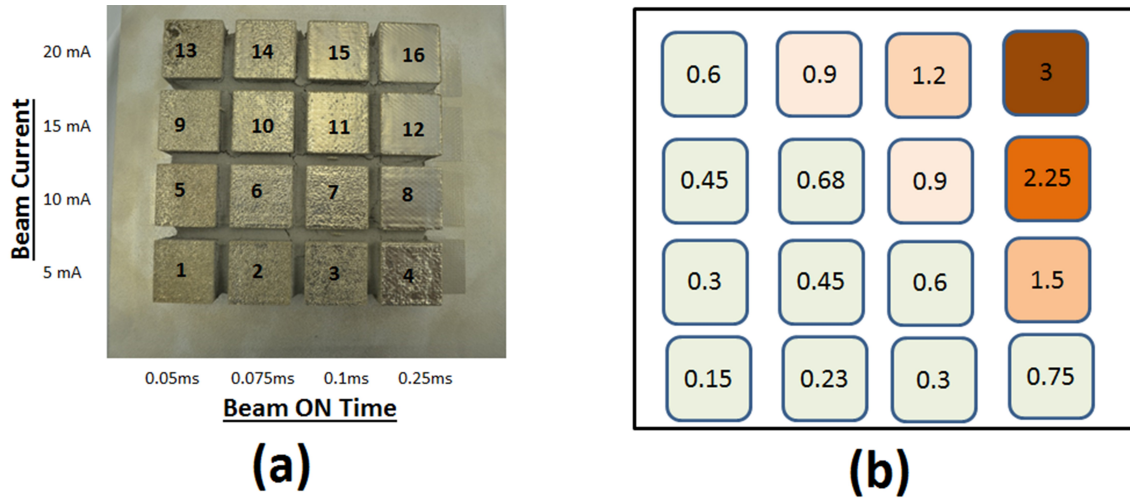


Figure 5.5: (a) Top view of samples fabricated using spot melting strategy with varying beam current and beam ON time (b) Energy deposited (kJ) per layer of samples

Arcam[®] is focused using electromagnetic coils, and changing the focus coil current controls the diameter of the beam. Even though the quantitative relationship between focus coil current and beam diameter is unknown, it can be qualitatively determined that larger coil current defocuses the beam, resulting in a larger beam diameter. Samples were fabricated with focus coil currents of 0 mA (highly focused - small beam diameter) and 20 mA (defocused - large beam diameter) within the practical limitation of the process. The samples were fabricated with lower energy deposition (1.9 kJ) per layer in order to decouple the effect of beam diameter from the preheat temperature. Energy deposition was controlled by reducing the number of spots in a layer. Samples were cut along the build direction (XZ plane) and the orientation of the grains were analyzed by studying the EBSD data using a scanning electron microscope. Figure 5.6 shows the inverse pole figure along with the corresponding grain aspect ratio of the two samples. In agreement with the results from numerical simulations, no significant change in grain aspect ratio was observed between the samples.

5.4.3 Significance of Preheat Temperature

Figure 5.7 shows the top surface (XY plane) of samples 8 and 16 (in Figure 5.5(a)), that are fabricated using beam currents of 10 mA and 20 mA, respectively. The beam ON time of 0.25 ms was kept the same for each spot. In the sample fabricated with the beam current of 10 mA (Figure 5.7 (a)), it is important to note that the adjacent melt pools are distinct and their solidification are independent of each other. It can be logically inferred that the local preheat temperature of the substrate when electron beam hits spot # 10 (see Figure 5.4(b)) is less than the solidus temperature of IN718 (1528 K). But in Figure 5.7 (b), the adjacent melt pools are indistinguishable, which means melt pool # 1 does not solidify completely before the electron beam hits spot # 10 (see Figure 5.4(b)). This shows that the local preheat temperature of the substrate when electron beam hits spot # 10 is greater than or equal to the solidus temperature of IN718 (1528 K). Local preheat temperature of the substrate in the sample 16 is maintained high by depositing more energy per layer (Figure 5.5(b)) which keeps the entire layer in molten state before the solidification begins.

The samples 8 and 16 (Figure 5.5) were cut along the build direction (XZ plane) and electron back scattered diffraction (EBSD) imaging was done to determine the

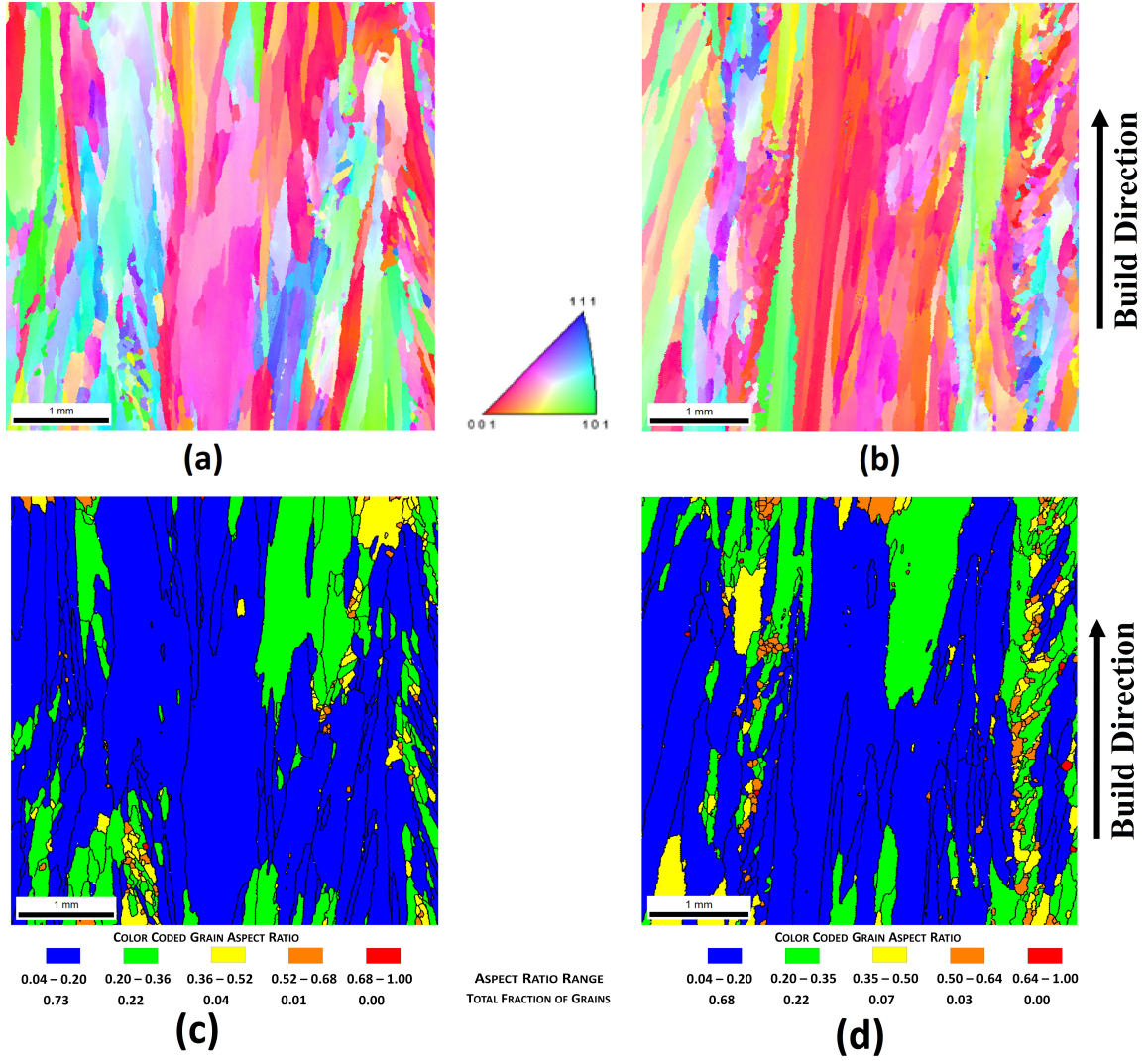


Figure 5.6: EBSD: Inverse pole figure of samples (XZ plane) fabricated with beam current of 20 mA, beam ON time of 1 ms and focus offset coil current of (a) 0 mA (b) 20 mA. Grain aspect ratio of sample built with focus offset coil current of (c) 0 mA and (d) 20 mA

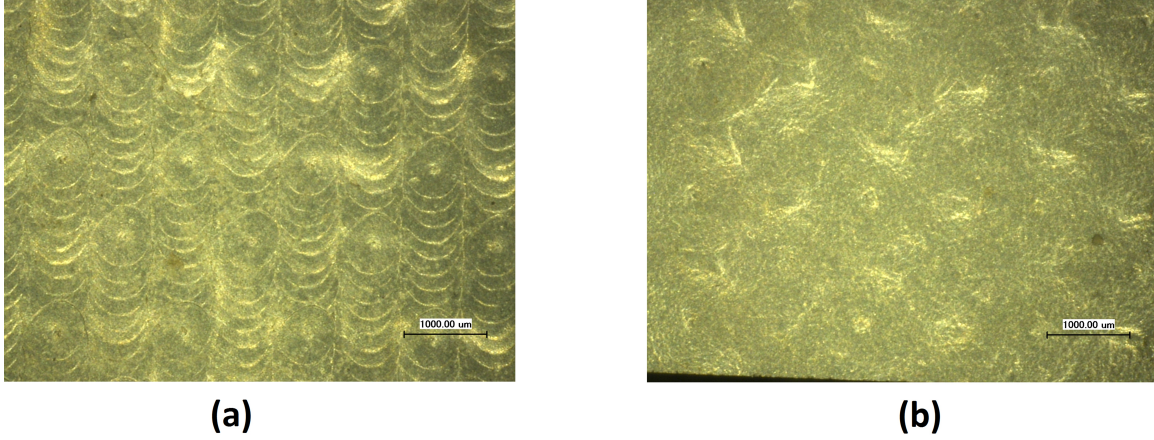


Figure 5.7: Melt pool of samples fabricated with beam current of (a) 10 mA (b) 20 mA with beam ON time of 0.25 ms

crystallographic orientation of the grains. Figure 5.8(a) and (b) shows the inverse pole figures of samples 8 and 16 respectively. Figure 5.8(c) and (d) shows the corresponding grain aspect ratio map. Directional (columnar) grain growth along the build direction was observed in the sample 8 with distinct melt pools (Figure 5.7(a)). Equiaxed grain growth was observed in the sample 16 with indistinguishable melt pools (Figure 5.7(b)).

These experimental results validate the modeling results that local preheat temperature is the most significant factor in columnar to equiaxed transition (CET) during the solidification in the electron beam AM process. Statistical evidence is provided by corresponding grain aspect ratio maps shown in Figure 5.8 8(c) and (d). The sample fabricated with beam current of 10 mA has 60% of grains with aspect ratio less than 0.25 while the sample fabricated with beam current of 20 mA has only 6% of grains with aspect ratio less than 0.25.

The results in Figure 5.8 and Figure 5.6 experimentally verify that changing beam diameter has an insignificant effect compared to changing the local preheat temperature of the substrate.

5.5 Summary

- Solidification texture of IN718 alloy was controlled during electron beam additive manufacturing by developing a novel melt scanning strategy optimized with a 3-D

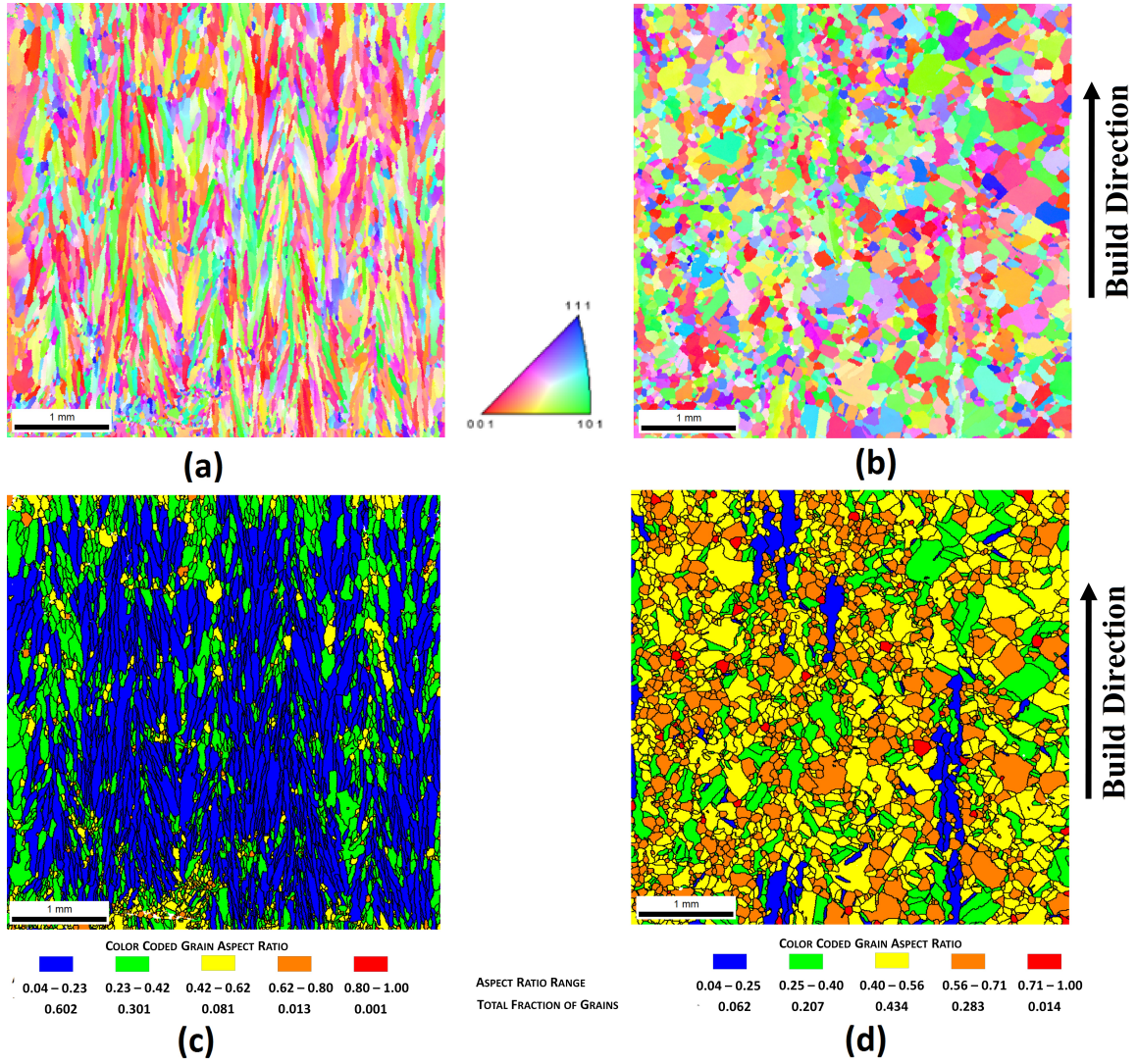


Figure 5.8: EBSD: (a) Inverse pole figure of samples (XZ plane) fabricated with beam current of 10mA and (b) 20mA (c) Grain aspect ratio of sample built with 10mA and (d) 20mA with beam ON time of 0.25 ms

numerical model capable of predicting the fraction of equiaxed grain formation as a function of beam diameter, beam current, beam ON time, and preheat temperature. The model relies on the spatial and temporal calculation of thermal gradient (G) and liquid-solid interface velocity (R). The results of the numerical calculations were then coupled with theoretical model for columnar to equiaxed transition during solidification.

- Using design of experiments principles, numerical evaluations showed that the volume fraction of equiaxed grains (Φ) increased, with an increase in beam-diameter, current and ON time, as well as, preheat temperature. The analysis of variance (ANOVA) approach was used to quantify the effect of all the input parameters on the volume fraction of equiaxed grains formed during the solidification of the simulated melt pool. A quantitative relationship was derived between the input parameters and the volume fraction of equiaxed grains formed. Preheat temperature is found to contribute the most in altering the volume fraction of equiaxed grains formed, and beam diameter is found to be the least significant among all the parameters considered. The influence of each of the beam input parameters on the columnar to equiaxed transition during the solidification was statistically explained.
- The obtained results not only depend on the parameters of the new scan strategy but also rely on the geometry of the part being fabricated. The effect of build geometry on columnar to equiaxed transition is described in next chapter Chapter 6.

Chapter 6

Geometry Dependency of CET and Infrared In-situ Monitoring

6.1 Effect of Build Geometry on CET

The obtained result from previous chapter is extended to study the effect of the process parameters of the scan strategy on build geometry. The scan strategy was explained qualitatively in the previous chapter Figure 5.4 (b).

Initially, the 3D CAD geometry is sliced into numerous thin layers with respect to the layer thickness. Then, the corresponding images of all the 2D layers are pixelated. Each pixel corresponds to a spot to be melted. The linear pixel density (number of pixels per unit length) is one of the variables during this step. The pixel density is kept constant for a given part. Each pixel corresponds to one melt spot during fabrication as shown in Figure 6.1. Another parameter in the spot melt strategy is the number of pixels to skip between two consecutive spot melts (pixels skipped between #1 and #2 in Figure 6.1). Hence, energy deposition per layer per part can be varied by varying (a) pixel density and/or (b) beam ON time at a spot and/or (c) beam current.

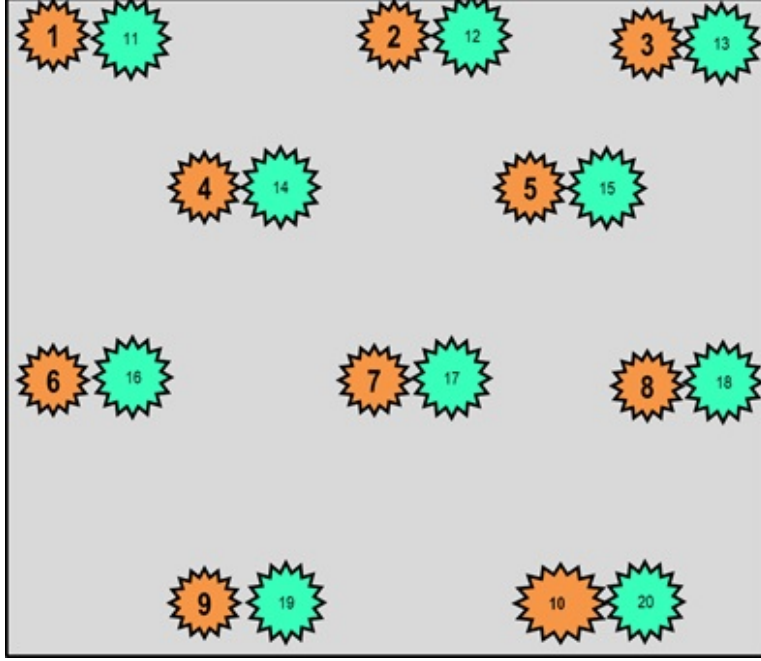


Figure 6.1: Qualitative spot melting strategy.

6.1.1 Scan Strategy and Parameters

In this experiment, 6 samples were fabricated with the spot melting using an Arcam[®] machine. From Figure 5.5 and Figure 5.7, it can be observed that increasing the energy density resulted in the transition from columnar to equiaxed morphology. In this study, the beam ON time 0.25 ms and beam current 20 mA were kept constant for all the samples. The energy deposited per layer was changed from one part to the other by varying the linear pixel density. Table 6.1 lists the dimensions of the parts, linear pixel density and associated energy density. Energy density is calculated as per Equation (6.1).

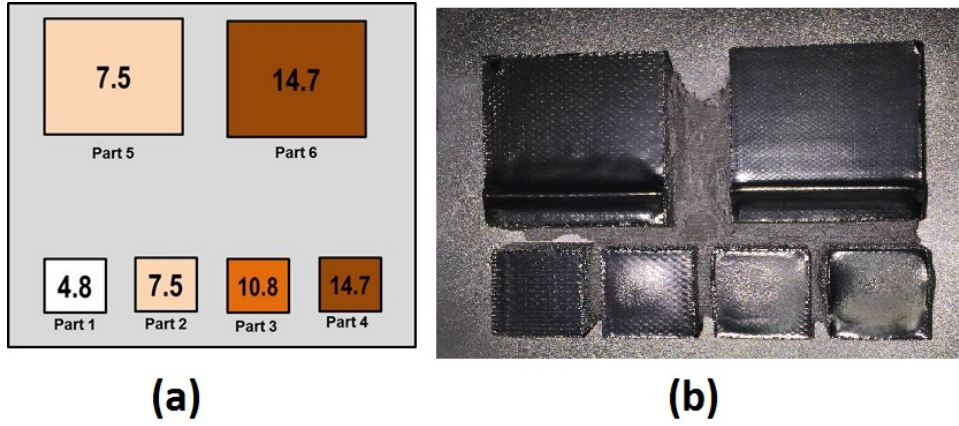
$$EnergyDensity = \frac{N_p * I * V}{A} \quad (6.1)$$

where N_p is the number of pixels per part per layer, I is the beam current, V is the beam voltage, A is the cross sectional area of part.

All the parts are 20 mm tall. 4 parts were fabricated with cross section of 20x20mm and 2 parts were fabricated with a cross section of 40x40mm. The linear pixel density was increased from 4px/mm to 7 px/mm from part 1 to part 4. It is important to note that part

Table 6.1: Spot melt parameter list to study the effect of geometry on CET

Part #	Dimensions (L x W x H)(mm)	Linear pixel Density (pixels per mm)	Total pixels per layer	Energy Density (MJ/m ²)
1	20x20x20	4	6400	4.8
2	20x20x20	5	10000	7.5
3	20x20x20	6	14400	10.8
4	20x20x20	7	19600	14.7
5	40x40x20	5	40000	7.5
6	40x40x20	7	78400	14.7

**Figure 6.2:** (a) Schematic of the build showing part geometry and corresponding energy density (MJ/sq.m) (b) Top view of the fabricated component

2 and part 5 has same pixel density (energy density) but different cross section. Similarly, part 4 and part 6 has the same energy density but different cross section.

Figure 6.2(a) shows the part geometry and the energy density associated. Figure 6.2(b) shows the fabricated samples.

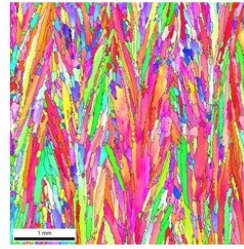
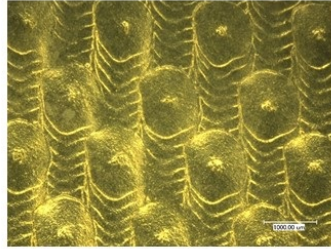
Figure 6.3 shows the top surface (melt pool) of the parts and corresponding grain morphology along the build direction (XZ plane). The results agree subjectively with the results explained in Figure 5.7 and Figure 5.8. Whenever distinct melt pools are visible on the top surface of the part, it results in columnar grains and whenever entire area is molten, it results in equiaxed grain structure. This essentially reiterates the numerical modeling results in Table 5.3 which showed that preheat temperature of the substrate is crucial for equiaxed

grain formation. It is important to note that even though part 2 and part 5 have same spot scan parameters (pixel density and energy density), the resulting grain morphology is different. This is attributed to the change in geometry. Increasing the cross sectional area of the parts without increasing the energy density will result in different return time of the electron beam because of increase in number of pixels per layer. Using Figure 6.1, the return time is subjectively defined as the time difference between the beginning of melt 1 and beginning of melt 11. By increasing the cross sectional area by 4 times from $400sq.mm$, to $1600sq.mm$, the return time is increased by 4 fold. Because of this increase in return time in part 5, the energy deposited in the melt spot 1 is diffused through conduction and radiation. Melt spot 1 is already solidified before melt spot 11 hits (Figure 6.1). This results in lower local preheat temperature for melt spot 11. This initial condition with lower local preheat temperature applies to all the pixels in part 5. Absence of sufficient preheat temperature prevent any nucleation ahead of the solidification front thereby resulting in columnar morphology. Hence, increasing the energy density from part 5 to part 6, there is enough energy to preheat the substrate locally and maintain the area melt resulting in equiaxed grains. This shows that the columnar to equiaxed transition (CET) is highly geometry dependent and comprehensive tool path optimization is required to translate this result to a complex geometry.

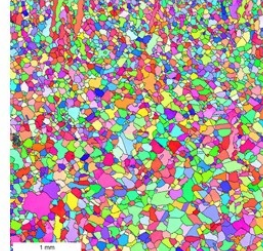
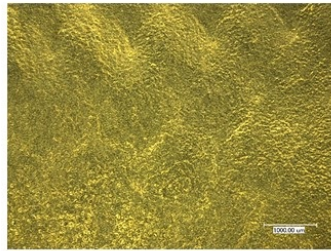
6.1.2 Comparison of Texture

To further quantify the grain structure, EBSD of the plane perpendicular (XY) to build direction was studied. Figure 6.4 (a) and Figure 6.4 (b) show the EBSD and texture of XY plane of part 5 and part 6 respectively. The EBSD data of XY plane reported in Figure 6.4 can be compared with the EBSD of XY plane of sample fabricated through raster melt strategy reported in literature(Figure 2.15 (a)). There is no surprise in the texture of part 5 as it is highly textured along the build direction. But for part 6, it is interesting to note that even though the grain morphology is equiaxed, it exhibits texture along the build direction. The texture has not been completely removed but weakened to an extent. Similar texture

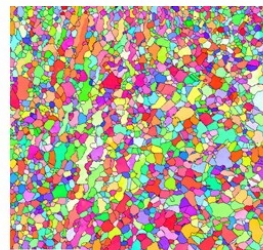
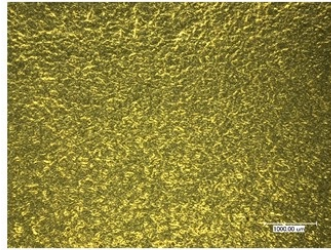
**Part 1:
Distinct Meltpool
Columnar**



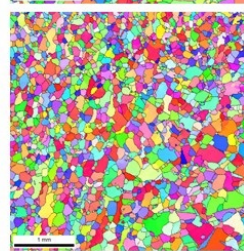
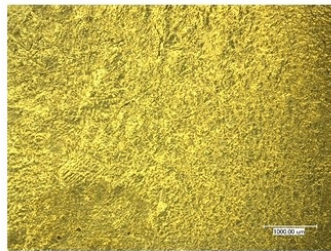
**Part 2:
Area Melt
Equiaxed**



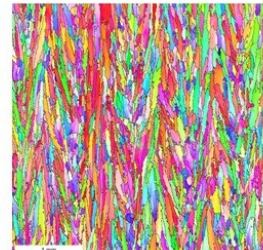
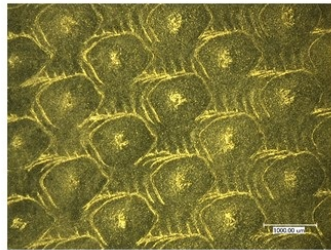
**Part 3:
Area Melt
Equiaxed**



**Part 4:
Area Melt
Equiaxed**



**Part 5:
Distinct Meltpool
Columnar**



**Part 6:
Area Melt
Equiaxed**

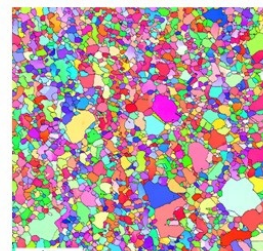
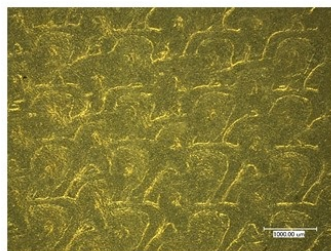


Figure 6.3: Top surface of the parts showing the melt pools and corresponding grain morphology along the build direction.

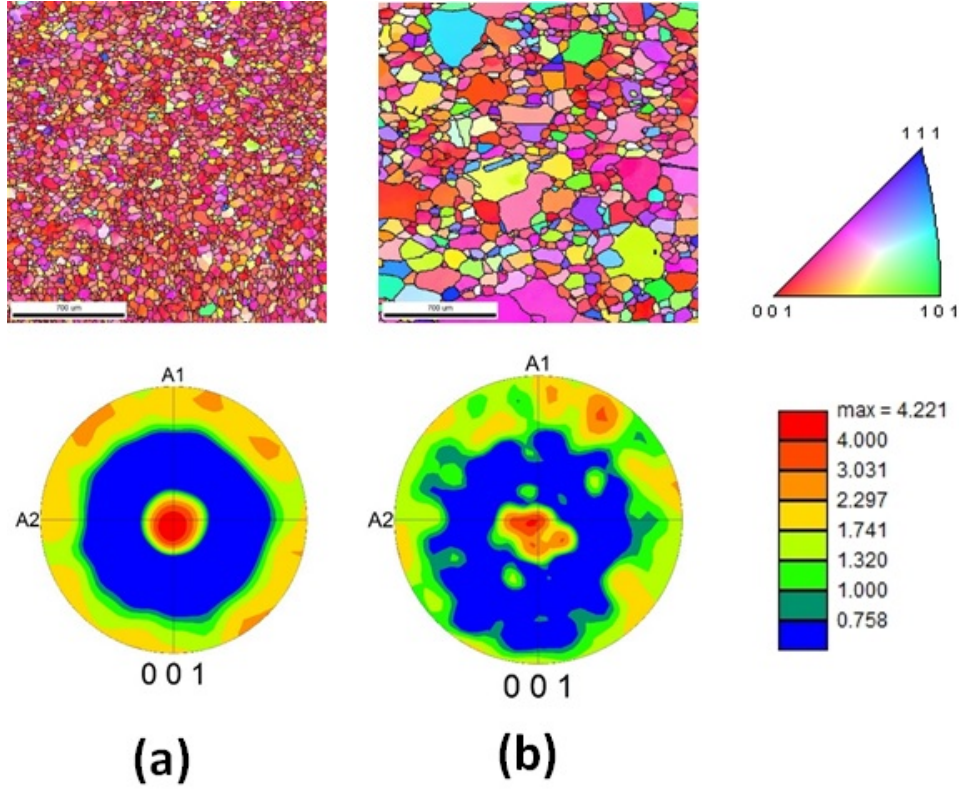


Figure 6.4: EBSD and corresponding texture plot of (a) part 5 and (b) part 6 along the plane perpendicular (XY plane) to the build direction.

is observed in part 2, 3 and 4 with equiaxed morphology. It can be concluded that even though there is nucleation ahead of the solidification front, the majority of the direction of thermal gradient is still along the build direction.

Demonstration build with hybrid microstructure: Based on these results, a demonstration component (Figure 6.5) was fabricated using IN718 with hybrid grain morphology (columnar + equiaxed in same component). In the arms where equiaxed data is shown, a part with 20x20mm cross section was loaded separately and spot parameters corresponding to part 2 in Table 6.1 was used. Rest of the part was built using normal raster scan strategy.

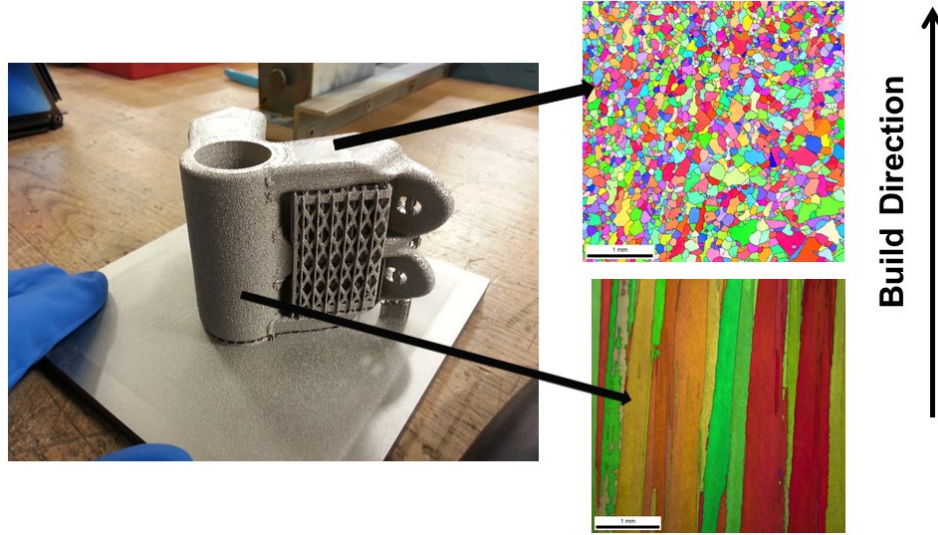


Figure 6.5: A Demonstration component with hybrid microstructure

6.1.3 Analysis of Island Melt Strategy to Overcome Geometry Effect

To decouple the effect of geometry and melt pattern, a new localized "island" or "checkerboard" or "tile" melt scan strategy was designed. In this melt strategy, the layer to be melted was divided into smaller squares of 5 mm each. Each square is termed as a "tile". For example, Figure 6.6 shows a 20x20 mm geometry discretized into 16 5x5 mm tiles. Numbers within each tile dictates the sequence in which they will be melted. Skipping the tile is to maintain thermal isolation in such a way that melting of tile 2 doesn't have significant thermal effect on the melting of tile 1 and so on. The hypothesis behind the tile melt sequence is that, if area melting can be induced within a tile (similar to Figure 6.3), equiaxed grain can be obtained in the area of the tile irrespective of the geometry.

Within each tile, spot melt strategy as shown in Figure 6.1 was implemented. Number of pixels or spots within a tile can be varied along with beam current and dwell time. In addition, number of pixels to skip along X and Y direction (Figure 6.1) can also be varied. This affects the return time of the adjacent spot. A total of 16 samples were fabricated. Table 6.2 shows the variables used to fabricate the samples.

Two sets of 8 samples were built. One with 100 pixels per tile and other with 144 pixels

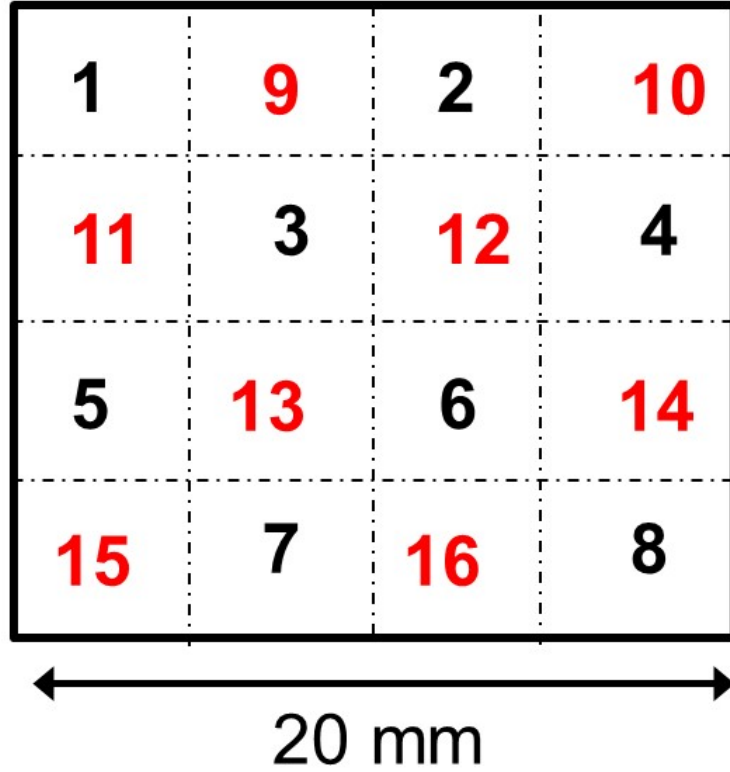


Figure 6.6: Schematic and sequence of tile melt strategy.

per tile essentially changing the amount of energy deposited per tile. Within one set of 8 samples, 4 were built with beam current of 10 mA and ON time of 1 ms and rest were built with beam current of 5 mA and ON time of 2 ms. Similar variation is performed for the other set of 8 samples with 144 pixels per tile. Figure 6.7 shows the picture of as-built samples.

The fabricated samples were then cut, mounted, polished and EBSD data was collected on the plane along the build direction (XZ plane). Figure 6.8 shows the sample number and corresponding EBSD data along the build direction. Figure 6.8(a) corresponds to samples fabricated with beam current of 10 mA and ON time of 1 ms and Figure 6.8(b) corresponds to samples fabricated with beam current of 5 mA and ON time of 2 ms.

From, Figure 6.7, it can be seen that all the samples have complete area melt within all the tiles. According to previous conclusion in Figure 6.3, area melting should result in equiaxed grain structure. Earlier in Chapter 5 (Table 5.3), it has been concluded that preheat temperature of the substrate is the most important factor in increasing the volume fraction of equiaxed grains within the melt pool. But, from Figure 6.8, it can be observed that most

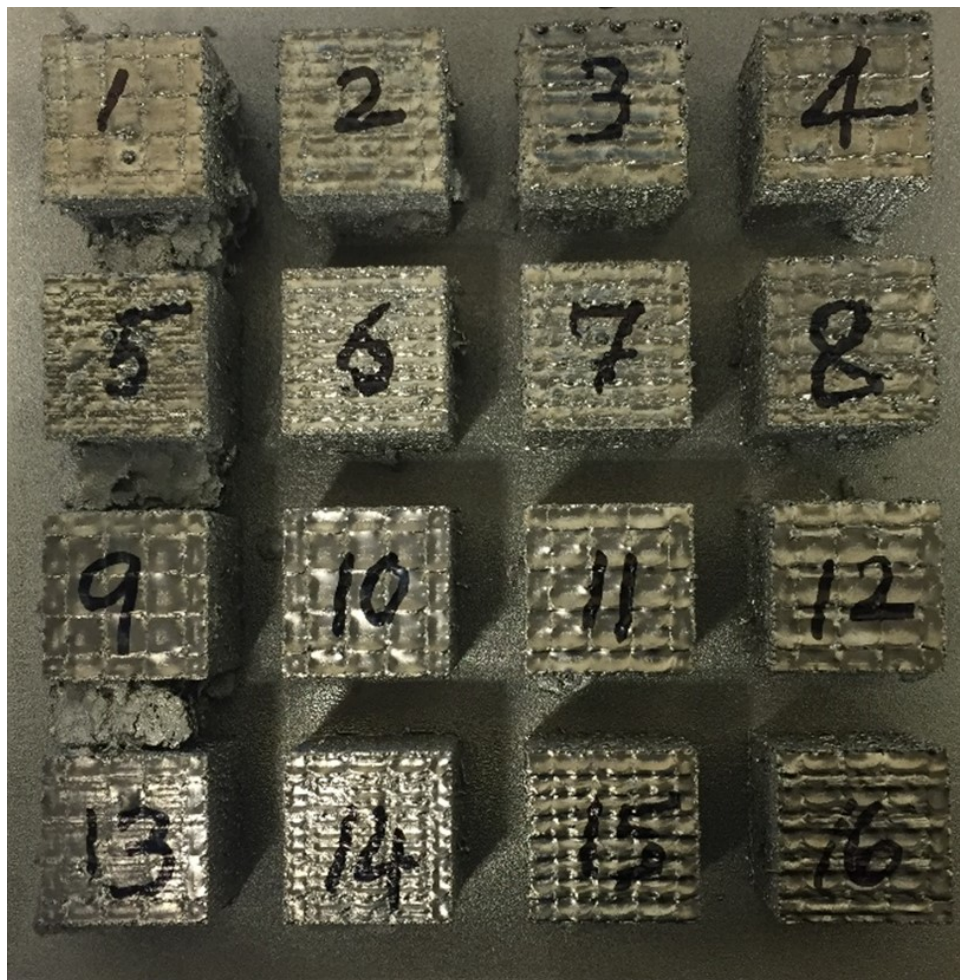


Figure 6.7: Top view of fabricated samples.

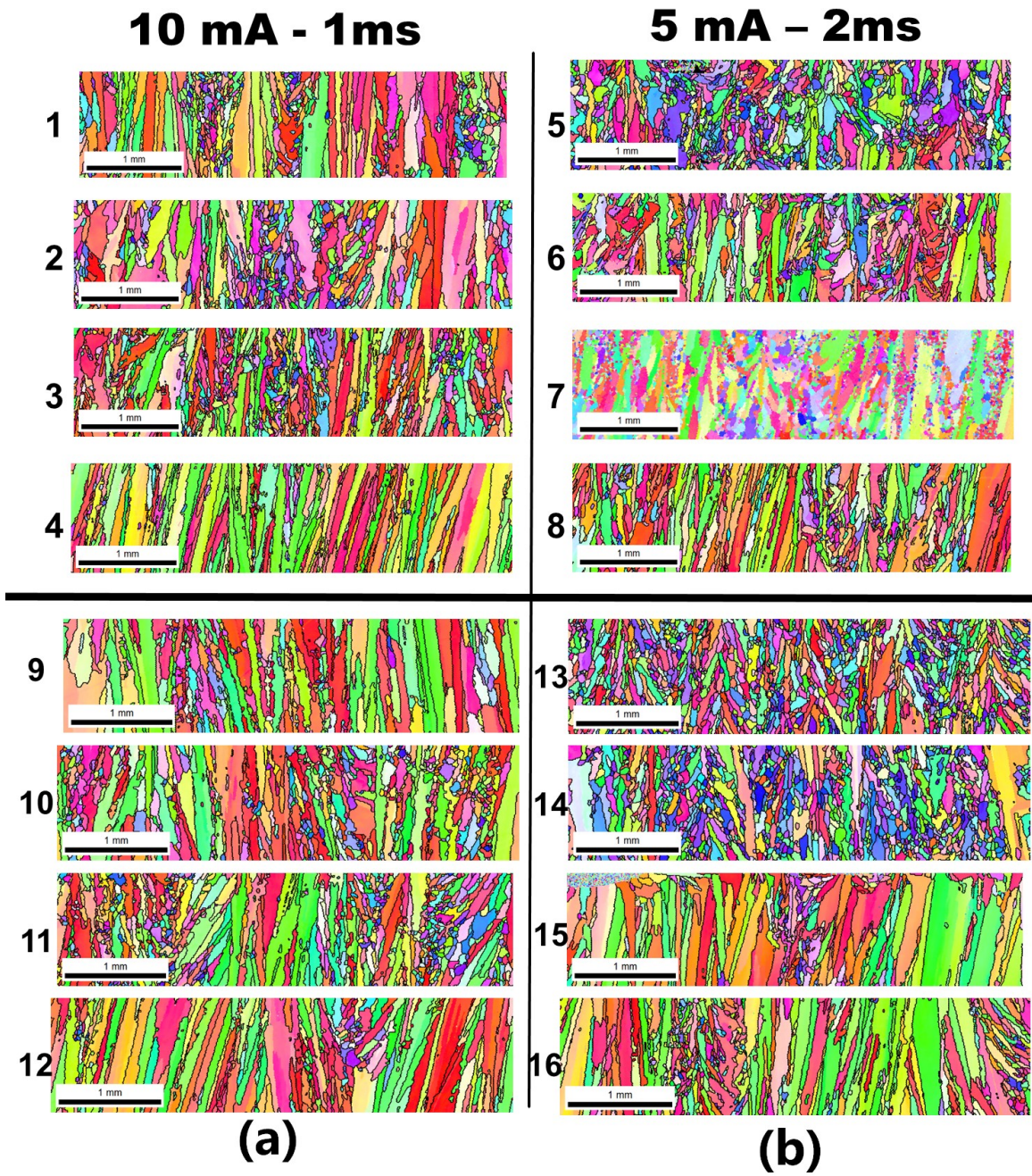


Figure 6.8: EBSD of samples

Table 6.2: Spot melt parameter list to study the effect of geometry on CET

Part #	Linear pixel count per tile	Pixel skip	Total pixels per tile	Beam Current (mA)	Beam ON Time (ms)	Energy per tile (J)
1	10	2	100	10	1	60
2	10	4	100	10	1	60
3	10	6	100	10	1	60
4	10	8	100	10	1	60
5	10	2	100	5	2	60
6	10	4	100	5	2	60
7	10	6	100	5	2	60
8	10	8	100	5	2	60
9	12	2	144	10	1	86.4
10	12	4	144	10	1	86.4
11	12	6	144	10	1	86.4
12	12	8	144	10	1	86.4
13	12	2	144	5	2	86.4
14	12	4	144	5	2	86.4
15	12	6	144	5	2	86.4
16	12	8	144	5	2	86.4

of samples resulted in columnar grains even though the tile had area melt. But in samples 5 and 13, the columnar grains were broken with misoriented grains were observed. Only difference between sample 1 and 5 is the reduced current and increased ON time (Table 6.2). The difference can be attributed to the competition between the energy input rate and the energy removal rate due to thermal diffusion and radiation. Even though same amount of energy is deposited per tile for sample 1 and 5, by increasing the beam ON time by twice and reducing the current by twice, the ratio of energy input rate per tile is reduced. In sample 1, it took 100 ms to deposit 60 J of energy in one tile while in sample 2, it took 200 ms to deposit same amount of energy. There is more time for sample 2 to diffuse heat into the substrate compared to sample 1. Hence, it is fair to conclude that in sample 1, the energy deposition rate is high such that the substrate is not hot enough to reduce the thermal gradient at the liquid-solid interface and promote nucleation ahead of the solidification front. But in

sample 2, the energy deposition rate is slow enough to promote some nucleation ahead of the interface. The results of samples 9 and 13 can be explained based on the similar argument.

6.2 Infrared Imaging

Numerical modeling tools are computationally expensive to capture the variation in thermal signatures and corresponding solidification characteristics over an entire layer of the build. Hence, for efficient detection of solidification structure over larger time and length scales, a non-contact, in situ process monitoring technique has been employed. The method used in this section employs the algorithm and tool developed by Raplee et al. [121].

Infrared thermal imaging is used to measure the solidification parameters and detect the microstructure variation in the build. In this experiment, a FLIR 7600 camera was focused onto the substrate inside the build chamber of an Arcam[®] S12 through a circular window. The interaction of electron beam with the powder particles produces X-rays. To avoid damage due to X-rays, a lead glass is placed over the window. Since the electron beam energy source is highly dense, the liquid metal vaporizes and gets deposited on the circular window including the surface inside the chamber [87]. To reduce the metalization on the window, a rolling kapton film set up was used. The frame rate was set at 100Hz. The physical set up of the system is comprehensively discussed by Dinwiddie et al. [122].

Infrared radiation is emitted by all the bodies except blackbody. IR camera has a sensor which records the intensity of the radiation. Intensity of the radiation depends on the (a) emissivity, (b) surface roughness or topology and (c) thermal energy of the emitting body. Higher the thermal energy higher the intensity of radiation recorded. In electron beam process, during melting, both preheated sintered powder and printed surface co-exist. Since the surface topologies of the sintered powder and printed surface are significantly different, each state of the system must be calibrated to convert the intensity to temperature of the respective regions. Calibration polynomials of the camera for as printed surface and sintered powder (IN718) was taken from Dinwiddie et al.[123].

6.2.1 Algorithm for Calibration of IR data

The raw infrared data is in the form of 3D matrix. At a given instant, the data is 2D and the third dimension is the time. At a given time, a given pixel in the raw IR data, can either be at sintered powder state or at printed solid state or at liquid metal state. Each of the states have their own emissivity. Hence each of the state has to be calibrated to convert the raw data to accurate temperature data. The temperature calibration was done by capturing the raw intensity data at different states and curve fitting the raw intensity to known temperature measure by thermocouple at solid and sintered powder state for the set up. Calibrating for liquid state is challenging and it has not been done. Method of calibration for IN718 and Ti-6Al-4V is comprehensively discussed by Dinwiddie et al. [123]. For IN718, the calibration equation for sintered powder and printed solid surface derived by Dinwiddie et al. [123] is given in Equation (6.2) and Equation (6.3) respectively.

$$\begin{aligned} T_{powder}(C) = & -3.7979 * 10^{-22} I^6 + 4.2727 * 10^{-17} I^5 - 1.2895 * 10^{-12} I^4 \\ & + 1.7210 * 10^{-8} I^3 - 1.1787 * 10^{-4} I^2 + 4.5893 * 10^{-1} I + 31.093 \end{aligned} \quad (6.2)$$

$$\begin{aligned} T_{solid}(C) = & 1.3905 * 10^{-20} I^6 + 5.8866 * 10^{-16} I^5 - 9.8350 * 10^{-12} I^4 \\ & + 8.2644 * 10^{-8} I^3 - 3.7006 * 10^{-4} I^2 + 8.9133 * 10^{-1} I - 44.667 \end{aligned} \quad (6.3)$$

where T_{powder} , T_{solid} are the calibrated temprature of the sintered powder and printed solid respectively and I is the raw intensity count recorded at each pixel.

During the process, a layer of powder is initially raked on the previous layer and is sintered using a preheat current. After that melting process begins. Depending on the 2D layer geometry, at the end of melting of a layer, only certain pixels are melted and converted into solid part and rest remains in the sintered powder state. Initially, the temporal data is calibrated at both the states (sintered powder and printed solid) for each pixel. But, the actual state of the pixel is unknown. The state of the pixel has to be identified to apply

proper calibration equation. Before melting, the sintered powder equation has to be used and after melting as printed equation has to be used. To identify this transition region, Raplee et al. [121] outlined an algorithm by studying the temporal behavior of each of the pixels. The details of the algorithm is given in Figure 6.9 and briefly discussed below.

When the melting occurs, there is an instantaneous drop in the calculated temperature at the pixel due to change in the emittance of the liquid state. To identify the correct frame at which the drop occurs, local slope is calculated and compared with a threshold value. The data is smoothed in advance to avoid noises. In addition, the unmelted pixels cool down due to radiation heat loss while the melting is happening. To distinguish the unmelted pixels, regional slope is calculated. The slope of melted pixel is significantly smaller than the unmelted pixel. Figure 6.10 shows the difference in regional slope between the unmelted and melted pixel.

Once the transition point is identified and correct temperature calibration is applied before and after melting, the thermal gradient along X and Y axis and cooling rate are calculated in a similar manner explained in Section 3.1.8 (Equation (3.11), Equation (3.12) and Equation (3.13)). From resultant thermal gradient and cooling rate, interface velocity can be calculated. At the end of solidification of a layer, spatial (2D surface) and temporal variation of G and R are captured. This can be plotted on a reference map and correlated to corresponding grain morphology.

6.2.2 Experimental Observation

For a fixed geometry, the melt strategy and process parameters have already been identified Table 6.1 to obtain the required solidification microstructure. For this study, 2 samples (20x20x20 mm) were fabricated. For sample 1, the linear pixel density was set at 4 and for sample 2, the linear pixel density was set at 6. From Table 6.1 and Figure 6.3, it can be observed that sample 1 will result in columnar grain and sample 2 will result in equiaxed grain.

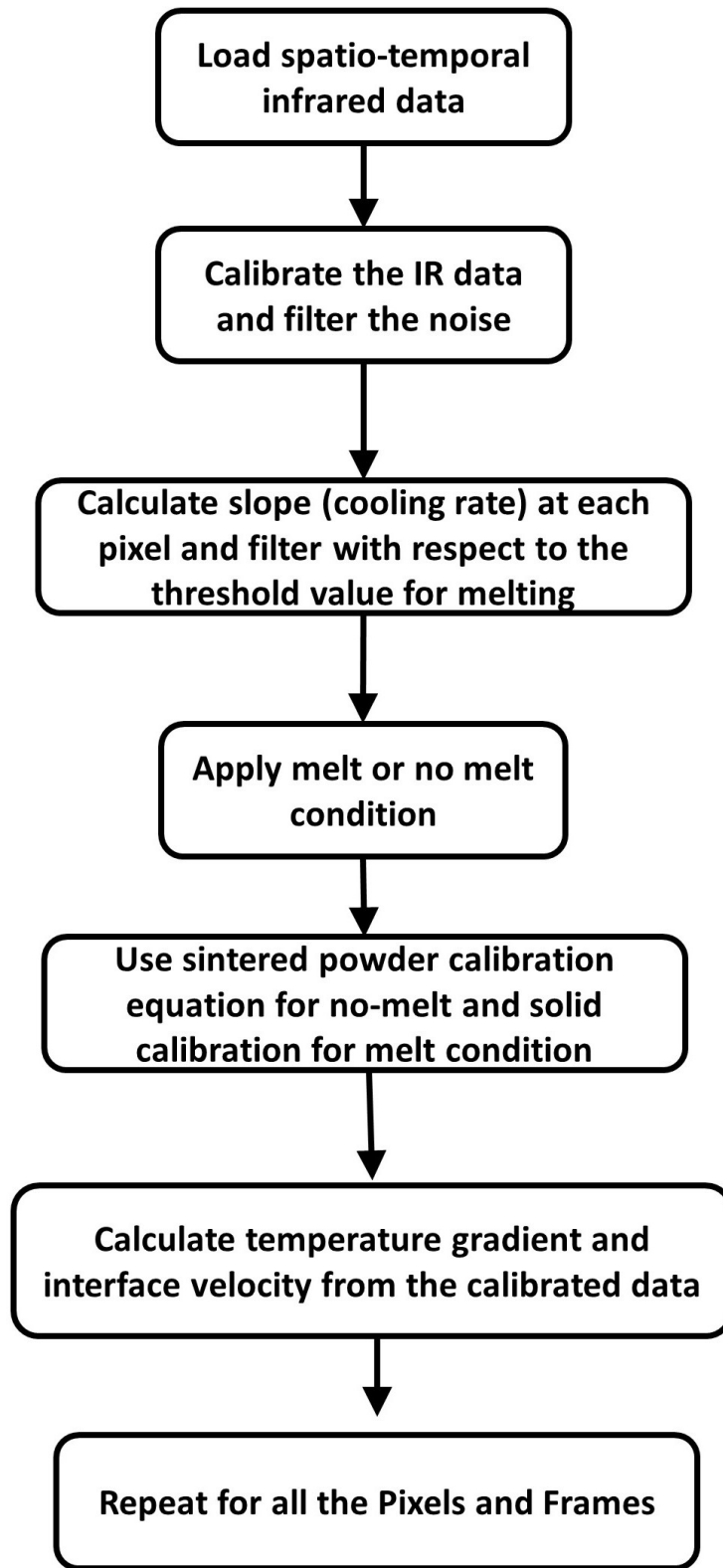


Figure 6.9: Flowchart describing the algorithm to identify the state of a pixel and calculate spatiotemporal G & R data (recreated based on Raplee et al. [121].)

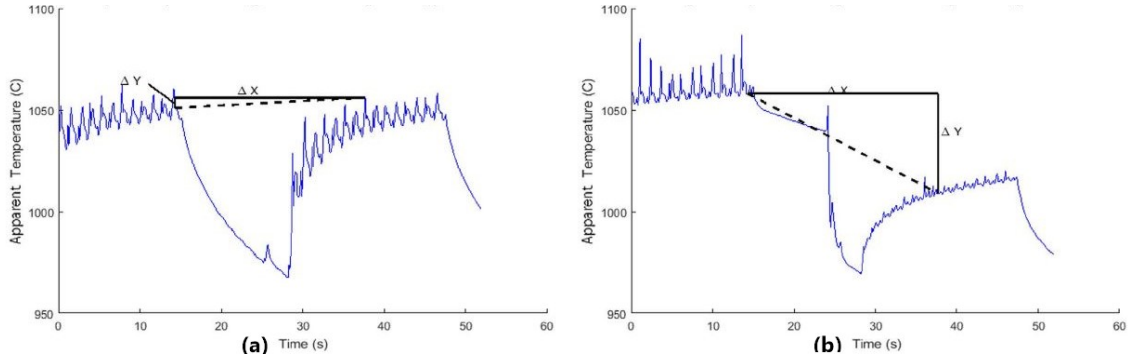


Figure 6.10: Regional slope of (a) unmelted and (b) melted pixel. [121].

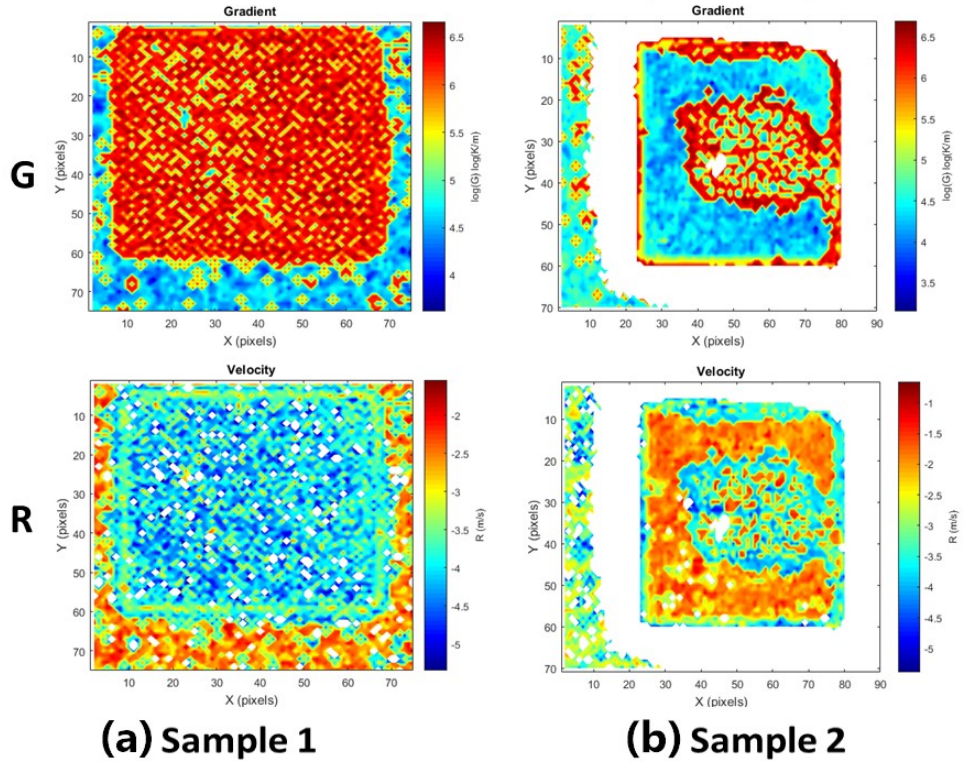


Figure 6.11: G and R calculated from IR data for (a) sample 1 (b) sample 2.

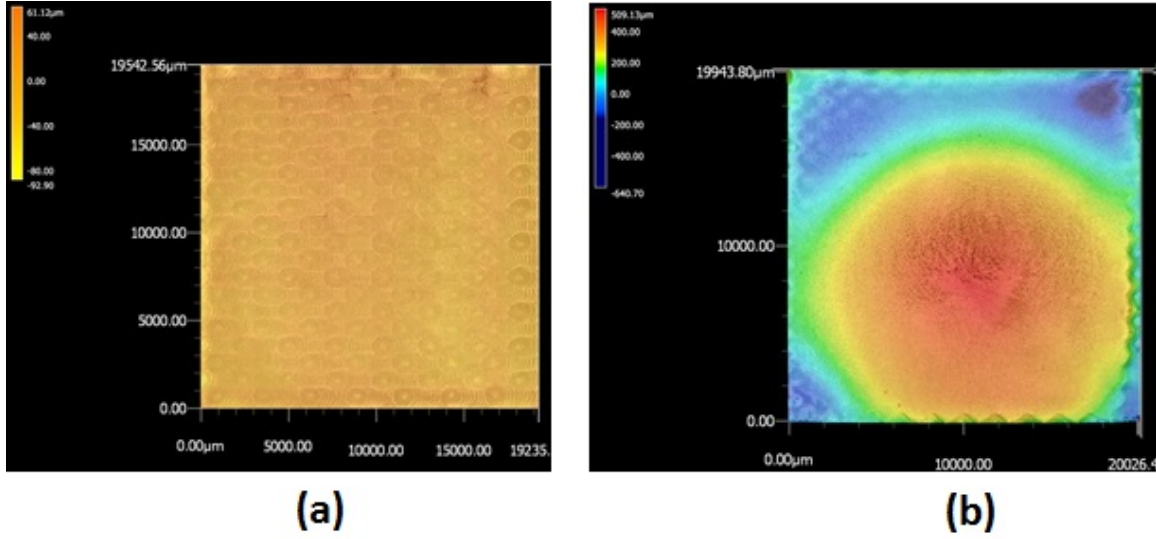


Figure 6.12: Surface topology of (a) sample 1 and (b) sample 2.

Figure 6.11 shows the heat map of temperature gradient and liquid-solid interface velocity calculated from the IR data. The square region in Figure 6.11 (a) corresponds to the surface of the sample 1 and the peripheral noises are due to sintered powder surrounding the sample. Data for sample 2 (Figure 6.11 (b)) is interesting to observe. Significant amount of noise can be observed. Since surface topology can affect the intensity, to rationalize this, the surface topology of the samples were analyzed. From Figure 6.12, it can be observed that surface of sample 1 is almost flat and the variation is approximately 50 μm . For sample 2, the surface is skewed (convex) by about 500 μm . Correlation between the keyence data (Figure 6.12(b)) and the thermal data (Figure 6.11 (b)) can be observed. the effect of concavity and convexity on the collected intensity is discussed by Raplee et al. [121].

Another important thing to note in the thermal data of sample 2 (Figure 6.11 (b)) is that, the top most region has high thermal gradient and low velocity corresponding to the columnar regime. Keyence data of the same region is not skewed and rightly so, the effect of topology can be ruled out. It can be correlated to the spot melting sequence described in Figure 6.1. It should be noted that the bands of spots moves from top left to the bottom right, hence the top most region will not have interaction with the spots in upcoming sequences once the top few bands of pixels are melted. G and R are plotted on the reference solidification map. Figure 6.13 (a) and (b) show the distribution of G and R for sample 1 and sample 2

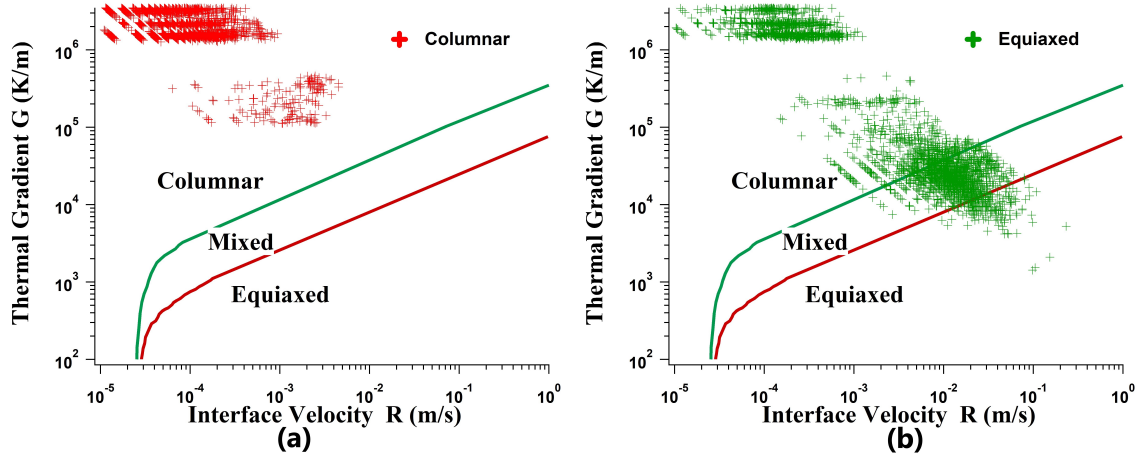


Figure 6.13: G and R overlay on reference solidification map [97] of IN718 for (a) sample 1 and (b) sample 2.

respectively. It can be observed that the data for sample 1 lies completely in the columnar regime but sample 2 is widely distributed across. As mentioned earlier, the variation can be attributed to the noise due to non-flat surface topology but can also be attributed to the top most region where the gradient is higher.

To validate it experimentally, EBSD was done at top and bottom region of the sample 2 as pointed out in Figure 6.14. Figure 6.14 (a) and (b) shows the EBSD and grain aspect ratio of the edge (XZ plane) where the thermal gradient is high and low respectively. It can be observed that, at one edge of the sample the grains grow in columnar orientation and at the other edge the grains are equiaxed. This result correlates with the data shown in Figure 6.11 (b) and Figure 6.13 (b).

6.3 Summary

Three topics have been discussed in this chapter.

- Effect of build geometry on columnar to equiaxed transition has been analyzed. It has been concluded that same melt strategy and process parameters that resulted in equiaxed grains for a geometry can not be used for a different geometry to induce equiaxed grains. Appropriate process parameter modifications have to be made as a function of geometry.

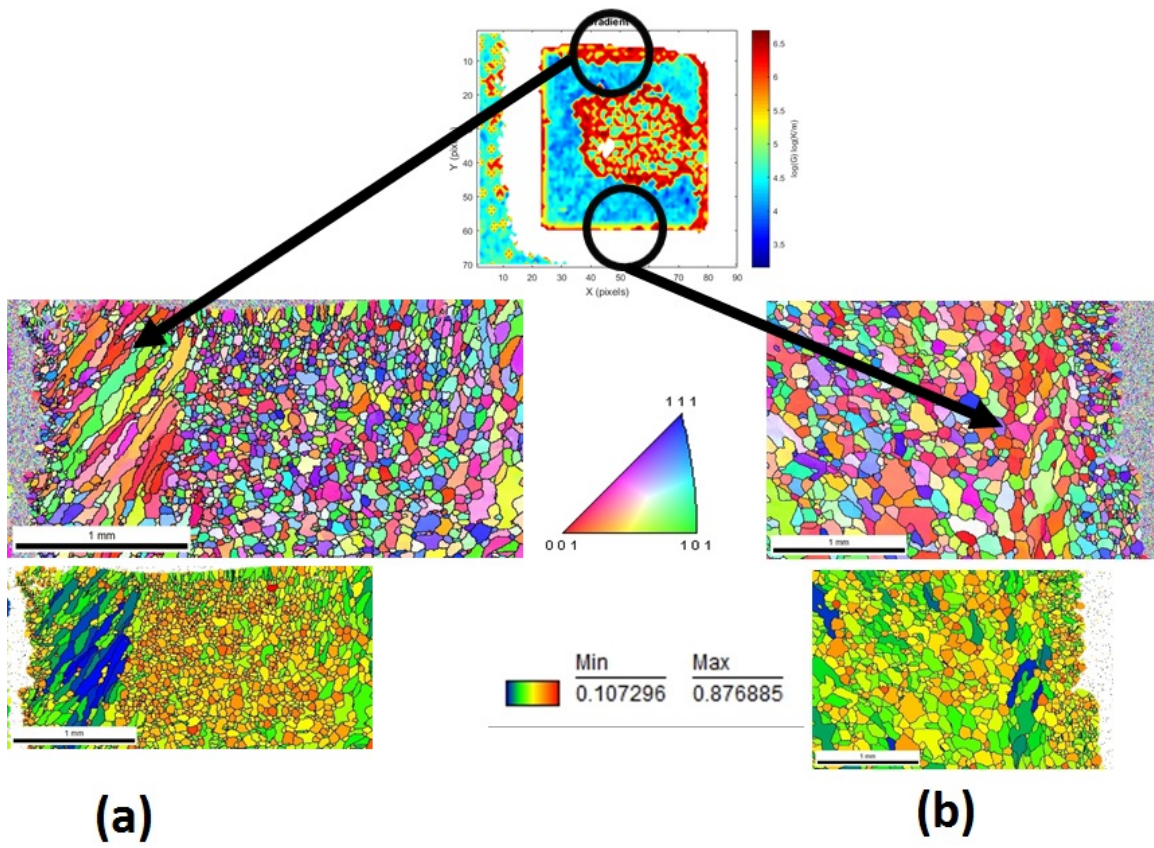


Figure 6.14: EBSD and aspect ratio map of (a) top region and (b) bottom region of sample 2.

- A demonstration component was built with both columnar and equiaxed grains within the same component.
- A localized "island" melt scan strategy has been evaluated. The importance of the competition between energy deposition rate and diffusion rate has been discussed. Further modeling and experimental work is necessary to understand and analyze the thermal behavior of the proposed scan strategy.
- Usage of in-situ process monitoring technique of infrared imaging to detect the grain morphology has been discussed based on a published algorithm. Correlation of surface topology and noise in the IR data has been reported.

Chapter 7

Effect of Fluid Flow and Surface Tension Gradient on Solidification

The numerical modeling results in Chapter 4 and Chapter 5 considers only the physics of heat transfer and phase change. Effect of convection within the melt pool is discussed in this chapter.

7.1 Effect of Surface Tension on Convection

Oreper et al. [124] and Kou et al. [125] pioneered the development of 2D and 3D numerical tools respectively to study the convection within the melt pool during welding. Effect of convection within the molten pool has been extensively studied using numerical modeling by Zacharia et al. [51] and Debroy et al. [52, 53, 54]. Depending on the forces acting on the liquid within the molten pool, the convection might have significant effect on the solidification microstructure and mechanical properties. Heiple [126, 127, 128, 129, 130] reported various experimental investigations on variation in shape of the fusion zone during welding of stainless steel and other alloys. The variation was correlated to variation in elemental fraction certain elements and impurities in the base metal. During electron beam welding, 4 major forces affect the fluid flow including buoyancy, electromagnetic, drag and surface tension or thermocapillary force. In general, among all the forces, surface tension force is found to be the most dominant during electron beam welding [127]. Surface tension is a function of

temperature. For pure metals, the variation of surface tension with respect to temperature ($d\gamma/dT$) is negative. But addition of certain surface active elements over a threshold level is found to alter the sign of surface tension gradient with respect to temperature from negative to positive. For example, in Fe and Ni alloys, when concentration of Sulfur and Oxygen is increased above 10 ppm, the value ($d\gamma/dT$) becomes positive at 1600°C [131]. The resultant flow due to surface tension gradient is termed as Thermocapillary or Marangoni convection. Sahoo et al. [131] reported theoretical analysis of the effect of surface active elements (Oxygen, Sulfur, Selenium and Tellurium) on surface tension gradient. These elements alter the surface tension gradient thereby altering the direction of the flow within the melt pool. Theoretical relationship derived by Sahoo et al. [131, 132] for calculation of surface tension as a function of temperature and solute is given in Equation (7.1).

$$\gamma = \gamma^o - A(T - T_m) - RT\Gamma_s \ln(1 + ka * \exp(-\Delta H_o/RT)) \quad (7.1)$$

where γ is the surface tension of metal plus solute, γ^o is the surface tension of pure metal, T is the temperature, T_m is the melting point, R is gas constant, Γ_s is surface excess at saturation, k is a constant related to entropy of segregation and ΔH_o heat of adsorption.

The direction of fluid flow is always from low tension to high tension region. Figure 7.1 shows the the variation of flow pattern with respect to change in surface gradient with respect to temperature. Negative surface tension gradient

7.2 Conservation Equations

In addition to diffusive energy transfer, convection also plays vital role within the melt pool. Hence, to increase the accuracy of the prediction, the fluid dynamics in the molten pool has to be accounted for in the model. To account for fluid dynamics, an advection term has to be added to the energy conservation equation (Equation (3.1)). In addition to solving conservation of energy, mass and momentum equations have to solved simultaneously. The 3 conservation equations are given by Equation (7.2), Equation (7.3) and Equation (7.4) respectively.

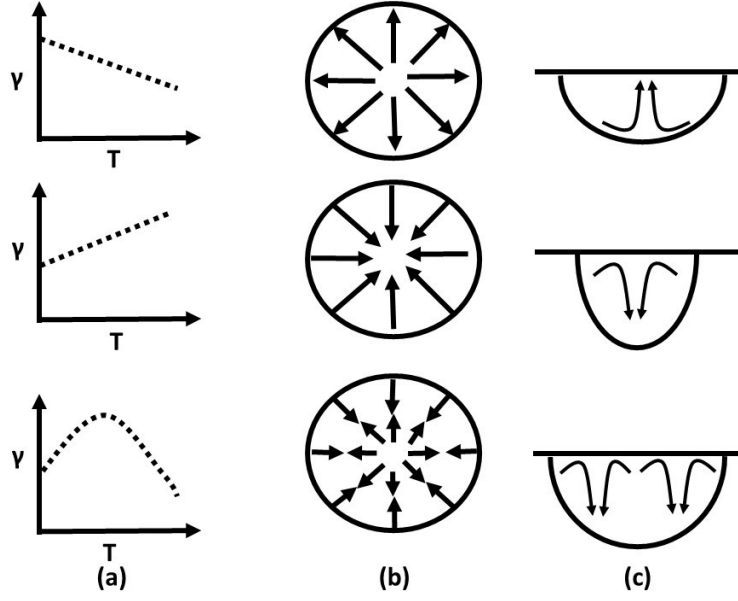


Figure 7.1: (a) Variation of surface tension with temperature (b) Top surface flow (c) Side view of the flow pattern

$$\frac{\partial(\rho h)}{\partial t} + \nabla \cdot (\rho^L h^L f^L u) - \nabla \cdot (k \nabla T(h)) = \dot{S} \quad (7.2)$$

where f^L, h^L and u are volume fraction of liquid in the node and specific enthalpy of the liquid and velocity respectively.

$$\frac{\partial \rho}{\partial t} + \nabla \cdot (\rho u) = 0 \quad (7.3)$$

$$\frac{\partial \rho u}{\partial t} + \nabla \cdot (\rho u u) = -\nabla p + \nabla \cdot \tilde{\tau} + S \quad (7.4)$$

where ρ is density, p is pressure, $\tilde{\tau}$ is the shear stress tensor and S is the source term. Source term can contain body forces, surface tension forces, drag force etc.

7.3 Simulation and Results

A single spot is simulated with beam current of 20 mA and on time of 1 ms. Since in Chapter 5, it was figured out that preheat temperature plays crucial role in stray grain formation, in this section two extreme cases of initial conditions were considered (25°C and

1000°C). In addition, effect of different levels of surfactant concentration on the fluid flow pattern has been studied. A total of 4 simulations with different combinations of preheat temperature and surfactant concentration have been done. Thermophysical properties used in the simulations are given in Table 3.2. Surface tension gradient for low (6 ppm sulfur, < 10 ppm Oxygen) and high (20 ppm sulfur, 8 ppm Oxygen) surfactant concentration used in the simulations is given in Figure 7.2. Surface tension (Nm^{-1}) and surface tension

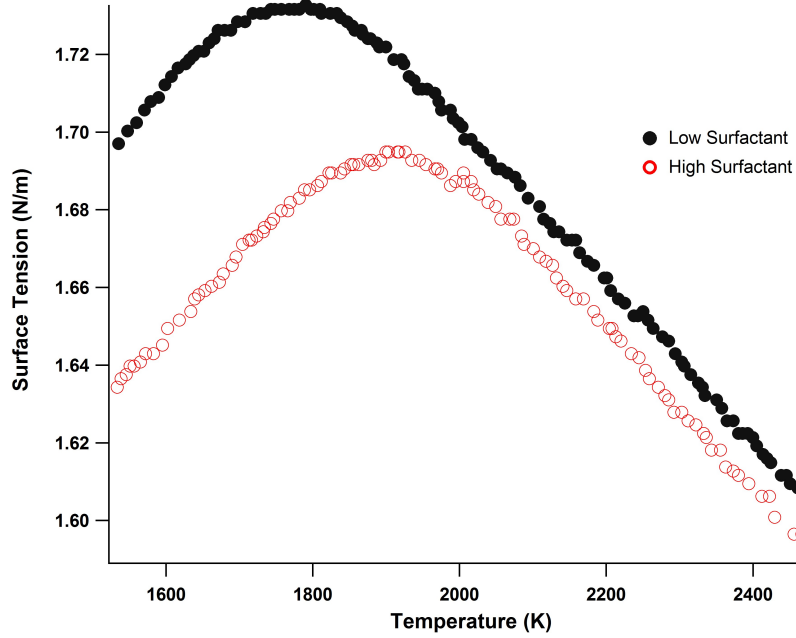


Figure 7.2: Surface Tension as a function of temperature for IN718 with different surfactant concentration [133]

gradient ($Nm^{-1}K^{-1}$) as a function of temperature for low surfactant concentration are given by Equation (7.5) and Equation (7.6) respectively. The transition temperature where the surface tension gradient flips the direction is 1740 K.

$$\gamma = (1.02308E - 10) T^3 - (0.84788E - 6) T^2 + (2.02088E - 3) T + 0.236238505 \quad (7.5)$$

$$\frac{d\gamma}{dT} = (13.06924E - 10) T^2 - (1.6958E - 6) T + 2.02088E - 3 \quad (7.6)$$

Surface tension (Nm^{-1}) and surface tension gradient ($Nm^{-1}K^{-1}$) as a function of temperature for high surfactant concentration are given by Equation (7.7) and Equation (7.8) respectively. The transition temperature where the surface tension gradient flips the direction

is 1895 K.

$$\gamma = (1.4849E - 10) T^3 - (1.2436E - 6) T^2 + (3.1122E - 3) T + 0.7539 \quad (7.7)$$

$$\frac{d\gamma}{dT} = (4.45455E - 10) T^2 - (2.48721E - 6) T + 3.1122E - 3 \quad (7.8)$$

7.3.1 Preaheat Temperature: 25°C

Since the melt pool is a single spot, four fold symmetry is applied and quarter of the melt pool has been simulated for all the cases. As described in Section 3.1.8, the G and R data is calculated for these simulations. Figure 7.3 compares the spatially varying G data along the depth of the melt pool (XZ plane) for pure heat transfer simulation and fluid flow simulations with low and high surfactant concentration for preheat of 25°C. Figure 7.3 also provides comparison of melt pool shapes for different cases. From Figure 7.3, it can be concluded that there is no significant difference in the sub-surface shape of the melt pool.

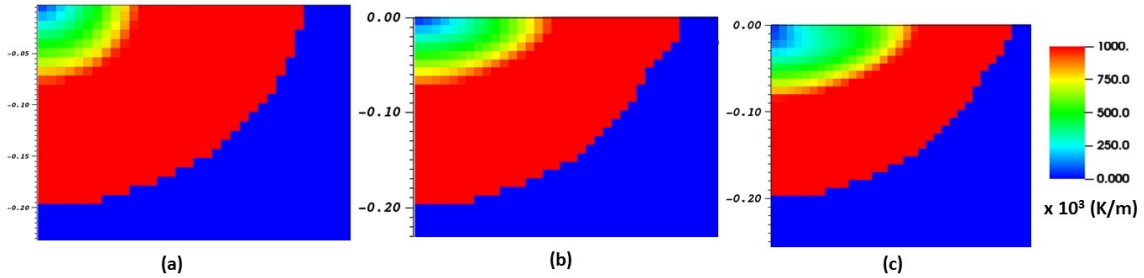


Figure 7.3: Thermal gradient G (a) Pure Heat transfer (b) Fluid flow (Low Surfactant) (c) Fluid flow (High Surfactant)

Figure 7.4 shows the comparison of fluid flow pattern at different time of the simulation. Images in top row corresponds to low surfactant concentration and images in bottom row corresponds to high surfactant concentration. The heat source was turned ON for 1 ms. From Figure 7.4(a) at 3 ms (3ms after the the beam is turned on), it can be seen that for both the cases, the direction of flow is in the same direction (outwards flow). At 3.5 ms, low surfactant concentration maintains the direction of flow but the high surfactant concentration starts to flip the direction of the flow at te edge of the melt pool with both inward and outward flow co-existing. At 4.5 ms, the direction of flow for high surfactant concentration case is

completely inwards while the low surfactant concentration just begins the flip the direction with both flows co-existing.

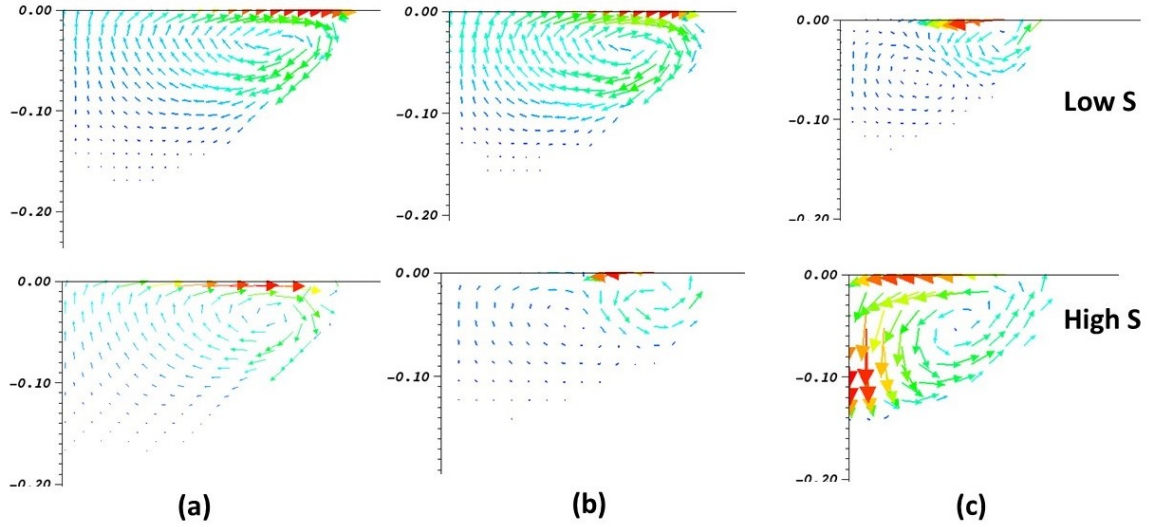


Figure 7.4: Comparison of fluid flow pattern for low and high surfactant concentration at (a) 3 ms (b) 3.5 ms (c) 4.5 ms

7.3.2 Preaheat Temperature: 1000°C

Figure 7.5 compares the spatially varying G data along the depth of the melt pool (XZ plane) for pure heat transfer simulation and fluid flows simulations with low and high surfactant concentration for preheat of 1000°C. Figure 7.5 also provides comparison of melt pool shapes for different cases. Unlike the simulations at low preheat, from Figure 7.5, difference in the sub-surface shape of the melt pool can be observed due to 'lipping' effect near the top edge of the melt pool. This can be attributed to the flow pattern within the molten pool.

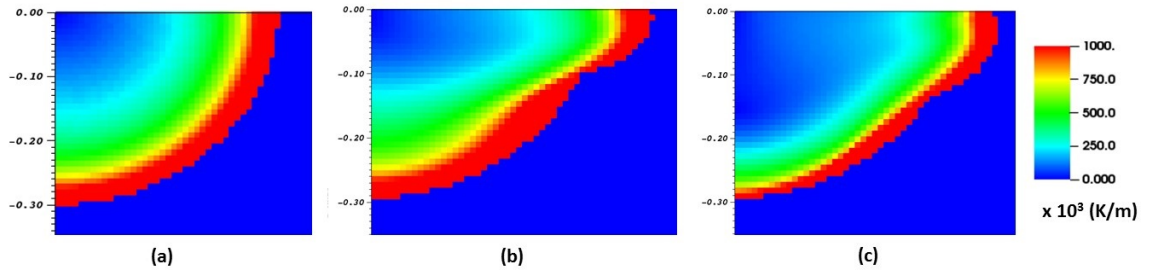


Figure 7.5: Thermal gradient G (a) Pure Heat transfer (b) Fluid flow (Low Surfactant) (c) Fluid flow (High Surfactant)

Figure 7.6 shows the comparison of fluid flow pattern at different time of the simulation. Images in top row corresponds to low surfactant concentration and images in bottom row corresponds to high surfactant concentration. In this case also, the beam was turned ON for 1 ms. From Figure 7.6(a) at 4 ms, it can be seen that for low surfactant cases, the direction of flow is outward and for the high surfactant case, the majority of the flow is in outwards direction but, at the edge of the molten pool, the direction begins to flip. At 5 ms, low surfactant concentration maintains the direction of flow and start to flip the direction at the edge but the direction of flow for high surfactant concentration is completely reversed (inward). At 8 ms, for both the cases, the direction of flow is inward but the maximum velocity of flow for high surfactant case is found to be higher at $0.3m/s$ compared to $0.13m/s$ for low surfactant case. Non-dimensional analysis of the modes of heat transfer is reported in the next section.

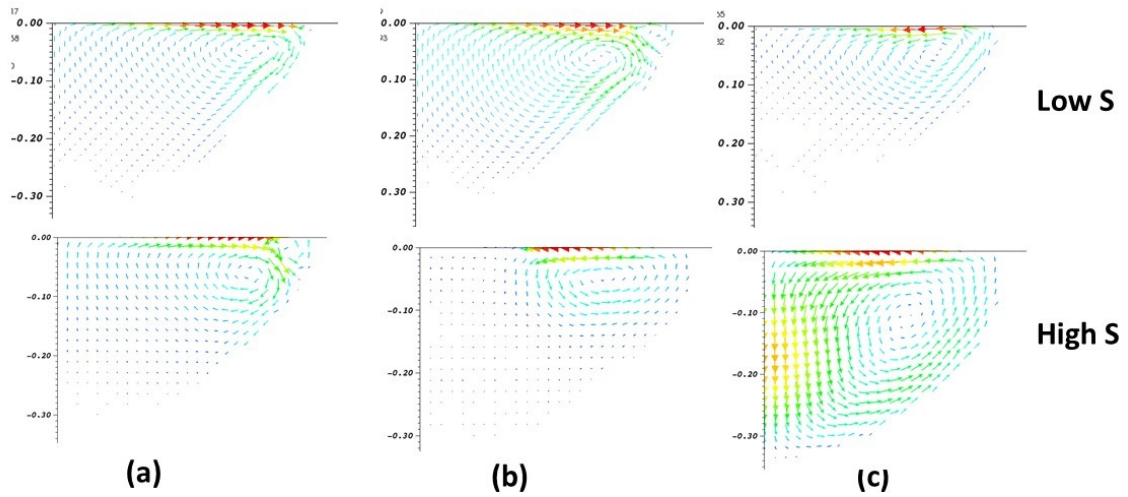


Figure 7.6: Comparison of fluid flow pattern for low and high surfactant concentration at (a) 4 ms (b) 5 ms (c) 8 ms

7.3.3 Comparison of Peclet number

Peclet number is a dimensionless number and is defined as the ratio of convective heat transfer to the diffusive heat transfer. In a melt pool, calculation of Peclet number describes the dominating heat transfer mechanism during melting and solidification. Peclet number is given by Equation (7.9).

$$Pe = \frac{\text{ConvectiveHeatTransferRate}}{\text{DiffusiveHeatTransferRate}} Pe = \frac{L.U}{\alpha} \quad (7.9)$$

where U is the velocity, L is the characteristic length and α is the thermal diffusivity of the material. In this case spatially varying grid Peclet number is calculated by using the velocity data at each of the discretized elements in the melt pool and L is taken as the size of the element (δx) [134]. Thermal diffusivity is calculated from the specific heat capacity and thermal conductivity values listed in Table 3.2. Figure 7.7 and Figure 7.8 shows the Peclet number calculated for 25°C and 1000°C respectively with the maximum Peclet number mentioned for each data.

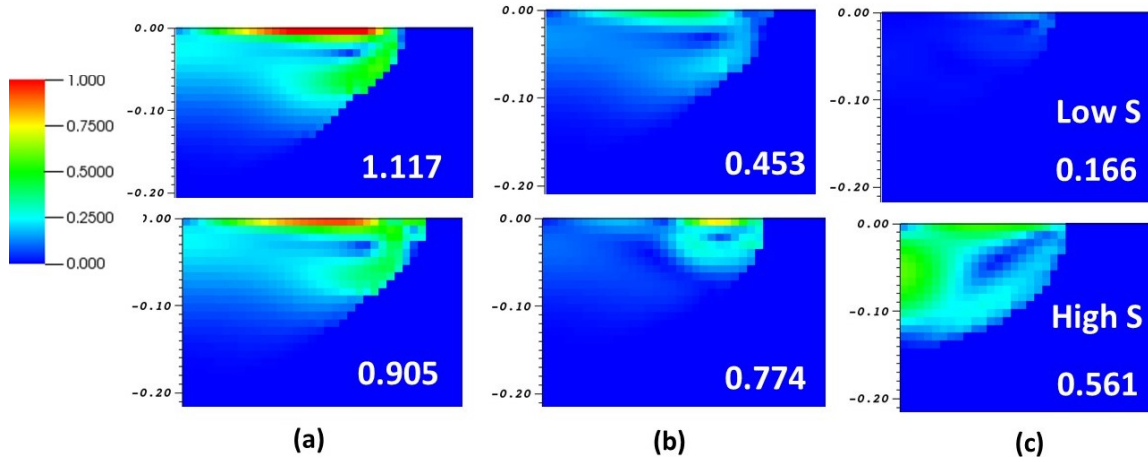


Figure 7.7: Comparison of Peclet number for low and high surfactant concentration with preheat of 25°C at (a) 3 ms (b) 3.5 ms (c) 4.5 ms

If the Pe is equal to 1, both convection and conduction play equal role. In general, Pe is high at the surface of the melt pool. In both the cases, the conductive heat transfer begin to dominate as the time proceeds. But, convective heat transfer is much more prominent for the alloy with high surfactant concentration compared to low surfactant concentration. For example, in Figure 7.7 (c), for high S content, the maximum Pe is 0.56 compared to 0.16 for low S alloy. This difference in Pe between different S concentration might alter the volume fraction of stray grains formed in melt pool.

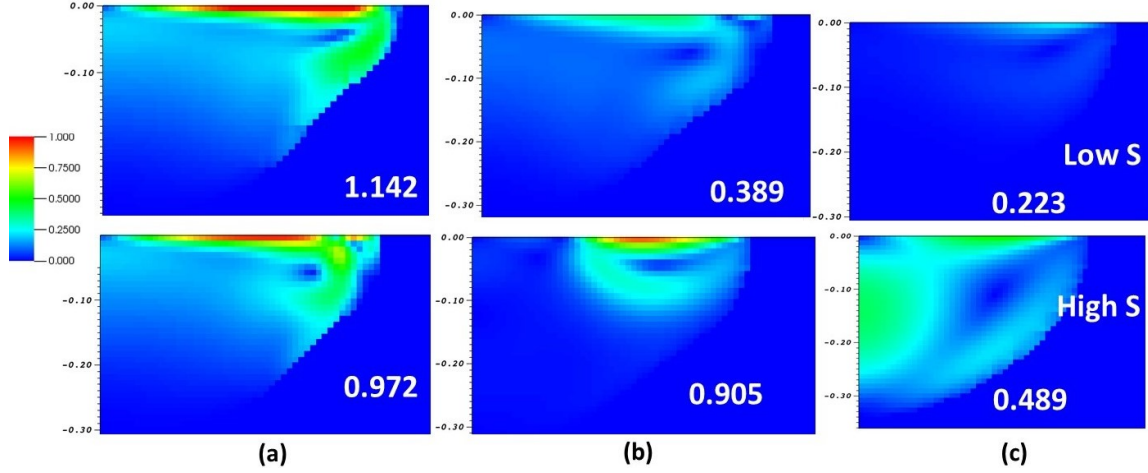


Figure 7.8: Comparison of Peclet for low and high surfactant concentration with preheat of 1000°C at (a) 4 ms (b) 5 ms (c) 8 ms

7.3.4 Effect of fluid flow on microstructure

In order to check the influence of fluid flow and surfactant concentration on the solidification microstructure, the predicted G and R data is then compared with pure heat conduction simulation for the same parameters. Weighted average of volume fraction of stray grains was calculated for each of the cases and is reported in Table 7.1.

Table 7.1: Comparison of Φ value with and without fluid flow in the model

Preheat Temperature	Physics	Volume fraction of equiaxed grains Φ %
25	HT	15.46
25	HT + FF (Low Surfactant)	24.3
25	HT + FF (High Surfactant)	29.7
1000	HT	74.84
1000	HT + FF (Low Surfactant)	78.01
1000	HT + FF (High Surfactant)	84.44

It can be observed that presence of high surfactant concentration increases the probability of stray grain formation.

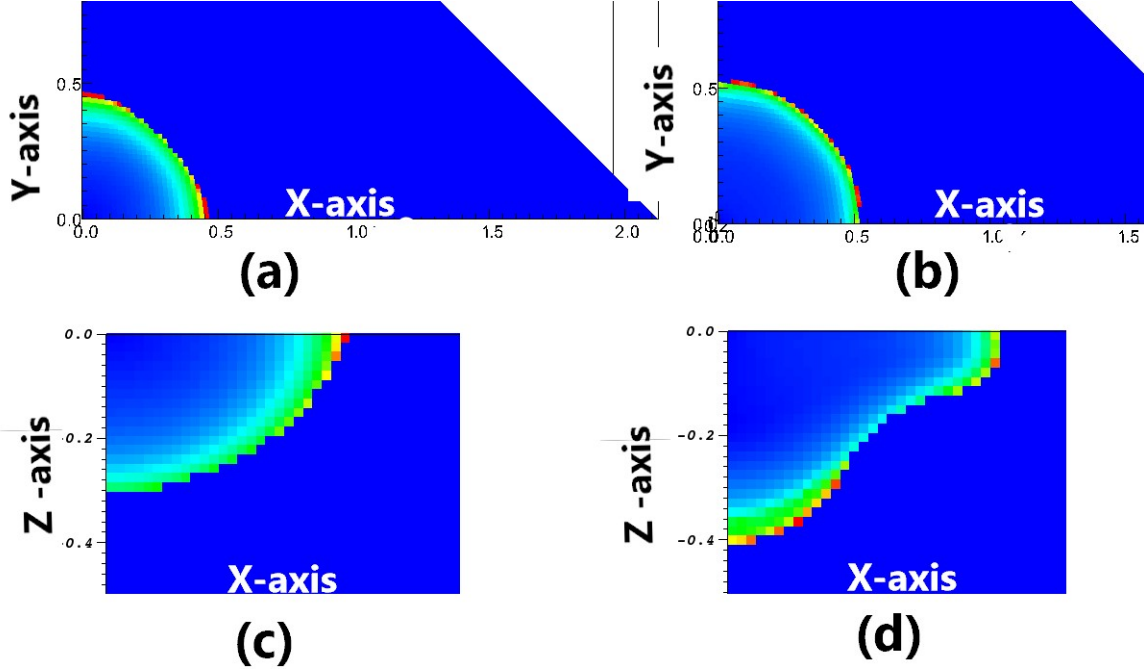


Figure 7.9: Comparison of shape of simulated melt pool with internal point offset of $200\text{ }\mu\text{m}$ (a) Pure heat transfer - top surface (XY plane) (b) Pure heat transfer - build direction (XZ plane) (c) heat transfer plus fluid flow (high surfactant) - top surface (XY plane) (d) heat transfer plus fluid flow (high surfactant) - build direction (XZ plane)

7.3.5 Significance of Scan Strategy on Fluid Flow and Experimental Comparison of melt pool shape:

To further understand the effect of fluid flow on the shape of the melt pool as a function of complex scan pattern, "5-spot" pattern described in Chapter 4 (Figure 4.2) and by Raghavan et al.[135] is used in the simulations. At each spot, the electron beam was turned ON for 0.25 ms with beam current of 20 mA . Two extreme cases with internal point offset (ref: Figure 4.2(b)) of $200\text{ }\mu\text{m}$ and $800\text{ }\mu\text{m}$ are used to analyze the effect of fluid flow on the size of the melt pool. In case 1 (internal point offset: $200\text{ }\mu\text{m}$), 5 melt pools combine to form single melt pool and in case 2, 5 independent melt pools are formed (Figure 4.3). Two simulations of each case has been performed (pure heat transfer and fluid flow with high surfactant concentration). For, the fluid flow cases, the surface tension gradient described in Equation (7.8) is applied. Since the "5-spot" scan pattern exhibits four fold symmetry, one quadrant of the scan strategy is simulated and compared with the experimental data.

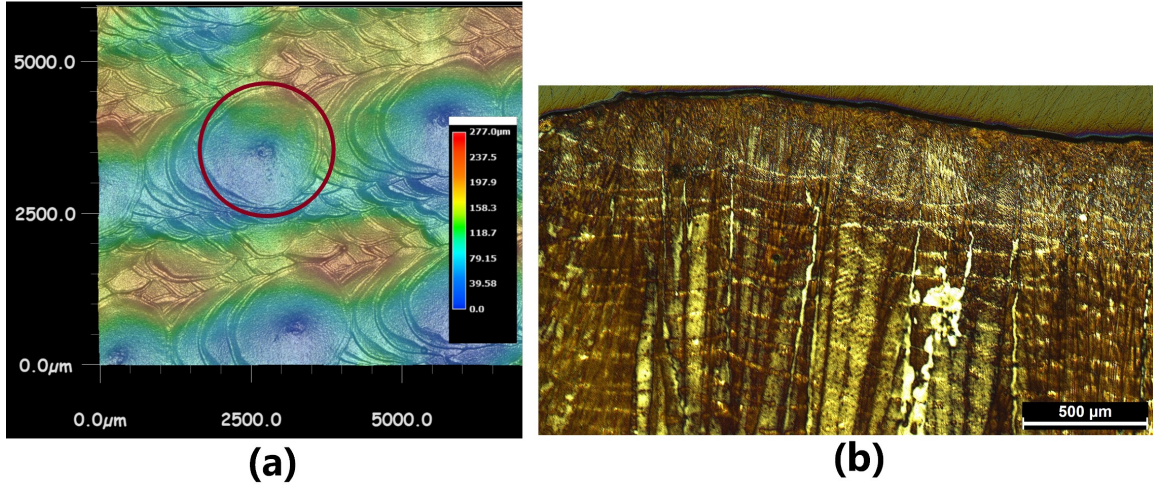


Figure 7.10: Melt pool with internal point offset of 200 μm (a) Top surface (XY plane) (b) Build direction (XZ plane)

Case 1: Figure 7.9 shows the comparison of the shape of the top surface of one quadrant simulated melt pool (Internal point offset: 200 μm) of heat transfer only (Figure 7.9 (a)) and with fluid flow (Figure 7.9 (b)). In this case, 5 spots within a domain of the scan strategy (Figure 4.2) merges to form single melt pool. Figure 7.9 (c) and (d) shows the shape of the melt pool along the build direction without and with fluid flow respectively. The simulation data is compared with the experimental data. Figure 7.10 (a) shows the surface topology of the melt pool depicting it's width. Figure 7.10 (b) is the optical image of the etched sample showing the depth of the melt pool traces along the build direction. The measured depth of the melt pool will not be accurate as the 3-D data of the depth was not characterized. Serial sectioning of the sample has to be done to improve the accuracy of the data.

Case 2: Similarly, simulations has been done for the case 2 (Internal point offset: 800 μm). In this case, 5 independent molten pools are formed within a domain of the scan strategy (Figure 4.2). Figure 7.11 shows the comparison of the shape of the top surface of one quadrant simulated melt pool (Internal point offset: 800 μm) of heat transfer only (Figure 7.11 (a)) and with fluid flow (Figure 7.11 (b)). Figure 7.11 (c) and (d) shows the shape of the melt pool along the build direction without and with fluid flow respectively. Figure 7.12 (a) shows the top surface of the melt pool depicting it's width. Figure 7.12 (b) is the optical image of the sample showing the depth of the melt pool traces along the build direction.

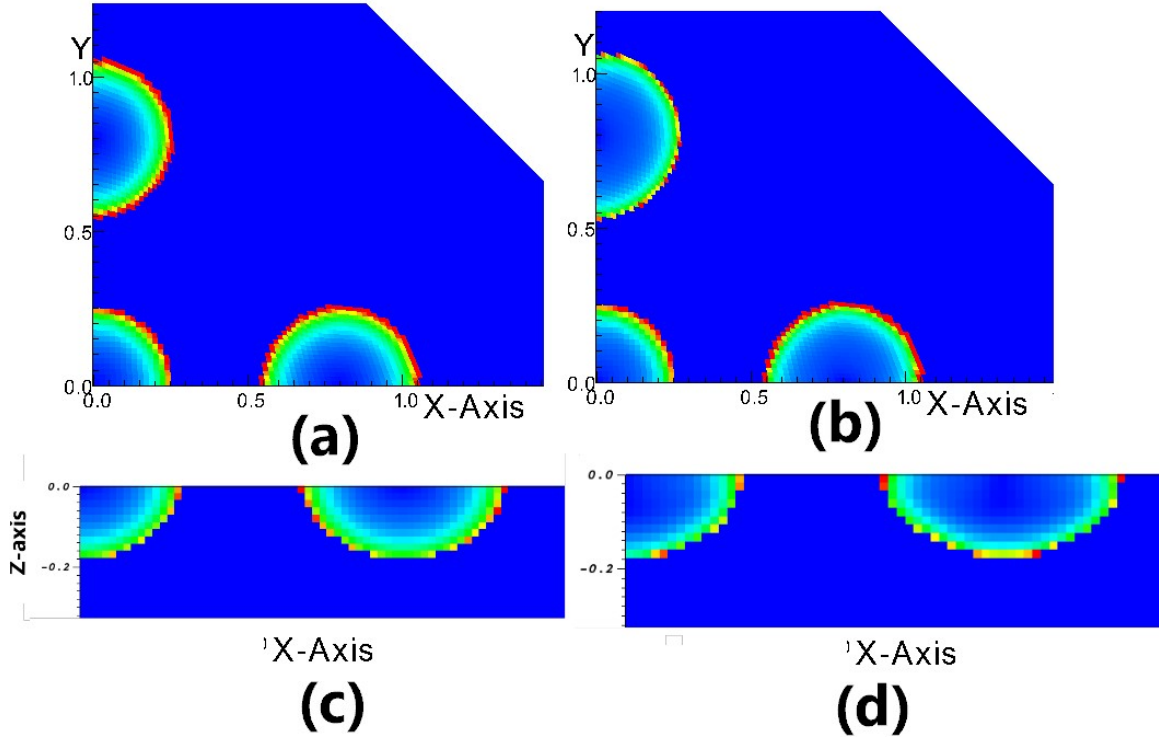


Figure 7.11: Comparison of shape of simulated melt pool with internal point offset of 800 μm (a) Pure heat transfer - top surface (XY plane) (b) Pure heat transfer - build direction (XZ plane) (c) heat transfer plus fluid flow (high surfactant) - top surface (XY plane) (d) heat transfer plus fluid flow (high surfactant) - build direction (XZ plane)

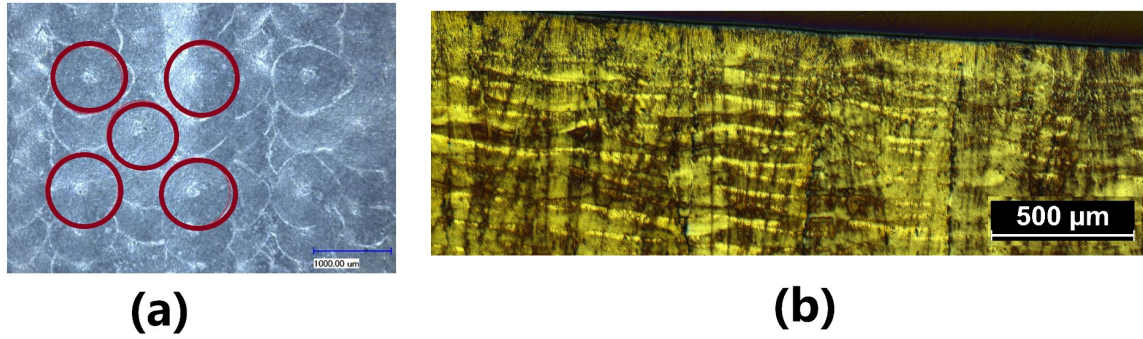


Figure 7.12: Melt pool with internal point offset of 800 μm (a) Top surface (XY plane) (b) Build direction (XZ plane)

To analyze the data quantitatively, the width and depth of the melt pool were measured for different simulation cases and the experiment. The data was quantified using ImageJ software [136]. Measured data are listed in Table 7.2. It can be observed that for the case with 800 μm internal point offset, there is no significant difference between the depth and

width of the simulation data between the heat transfer only (HT) model and the model with the fluid flow (HT+FF). But, significant difference is observed between the 2 models for the case with 200 μm internal point offset. This shows that the scan pattern has significant impact on the influence on the necessity of fluid flow in the model. Also, for both cases, the simulation tend to under predict the width of the melt pool. This can be attributed to the uncertainty in the surface tension data used in the simulations.

Table 7.2: Experimental comparison of width and depth of the melt pool with and without fluid flow in the model

Internal Point Offset (μm)	Data Type	Depth (μm)	Width (μm)
800	Experiment	193*	823
800	HT	178 (-7.7%)	606 (-26.4%)
800	HT + FF	184 (-4.7%)	620 (-24.6%)
200	Experiment	390*	1439
200	HT	300 (-23.1%)	910 (-36.7 %)
200	HT + FF	415 (+6.02%)	1124 (-21.8 %)

7.4 Summary

Effect of fluid flow on a single spot melting has been studied in this chapter. In particular,

- Effect of different concentration of surfactents on the fluid flow pattern and shape has been analyzed for extreme cases of preheat temperatures of 25°C and 1000°C. The results are compared with the simulations without fluid flow.
- Competition between convective heat transport and conductive heat transport is analyzed by calculating spatially varying non-dimensional Peclet number.
- Volume fraction of stray grain was calculated and compared for low S, high S cases with pure heat transfer simulation. A maximum of 10% difference was observed for the case with 1000°C while a maximum of 15% was observed for the case with 25°C preheat.

- Effect of fluid flow on the shape and size of the melt pool was compared with the experimental data. In the "5-spot" melting strategy, for the scan strategy with smaller areal energy density, there is no significant difference in the shape and size of the melt pool between the heat transfer only model and the model with fluid flow turned ON. Significant difference was observed with higher areal density. It was concluded that in addition to the material dependent surface tension, the scan strategy also has significant impact on the necessity of the fluid flow in the model to improve the accuracy of the prediction of shape of the melt pool. Higher the energy density, more important the fluid flow is and vice versa.

Chapter 8

Conclusion and Path Forward

8.1 Summary

- Significance of tool path design to control the solidification microstructure in metal additive manufacturing process has been reported.
- A parallel numerical modeling tool has been used to predict the spatiotemporal variation of the solidification parameters (a) Thermal gradient at the liquid-solid interface (**G**) (b) Velocity of the liquid-solid interface (**R**).
- A new scan strategy has been introduced for on-demand control of solidification grain size and primary dendrite arm spacing. Numerical tool was used to rationalize the effect of the scan strategy on the grain size by quantifying the shape of the molten pool. Increase in curvature of the melt pool resulted in finer grains. In addition to epitaxial solidification, possibility of heterogeneous nucleation has been analyzed based on the variation in cooling rate predicted using the simulations. Conventional raster pattern resulted in columnar grain size of about 700 μm , while using the modified scan strategy, the grain size was reduced to about 30 μm .
- Predicted spatio-temporal G and R data was coupled with theoretical model to quantify the volume fraction of equiaxed grains formed within a melt pool during solidification. Relative significance of different beam input parameters on equiaxed

grain formation was statistically analyzed through design of experiments approach. Preheat temperature was found to be the most significant factor in contributing to the increase in stray grain formation. Appropriate scan strategy was designed and bulk columnar to equiaxed transition has been experimentally verified.

- In addition to the effect of scan strategy, geometry was also found to play a crucial role in columnar to equiaxed grain morphology transition. The parameters of the scan strategy has to be changed appropriately with respect to theh geometry to obtain the desired microstructure. A localized "tile" melt strategy was introduced to overcome the effect of geometry and the competition between the energy deposition rate and energy diffusion rate has been analyzed.
- In-situ process monitoring tool (IR imaging) was employed to capture the spatial variation of the solidification parameters (G and R). Spatially varying G,R was correlated to spatially varying microstructure (grain morphology).
- Effect of surface tension on melt pool shape and volume fraction of stray grain formation was studied. Flow patterns for low and high surfactant concentration has been compared. Competition between convective heat transfer rate and conductive heat transfer rate was analyzed by calculating the Peclet number within the melt pool.
- In addition, the importance of fluid flow with respect to the scan strategy (energy density) has been discussed by comparing the simulation data with the experimental data (width and depth of melt pool). Fluid flow was found to play a key role in altering the shape of the melt pool when the energy deposition density is higher and vice versa.

8.2 Path Forward

There are numerous pathways to improvise the reported modeling and experimental framework.

Other AM processes and Materials: Is it possible to translate the results of this work across different AM processes and material systems? What is the significance of scan strategy

in laser powder bed system, laser powder blown system and wire based AM systems? Few other interesting improvisations and questions to be addressed are listed below.

Complex Geometry: So far in this work, effect of scan strategy in complex geometry has not been studied. Figure 8.1 shows the different scan strategy implemented on an airfoil. The airfoil layer is discretized into 115 pixels or spots. Color scale corresponds to the time stamp or the sequence of each spot with respect to the scan pattern. Figure 8.1 (a) is the normal raster pattern and Figure 8.1 (b) is the "Dehoff" fill which is same as the spot fill pattern mentioned in Figure 6.1 but for complex geometry.

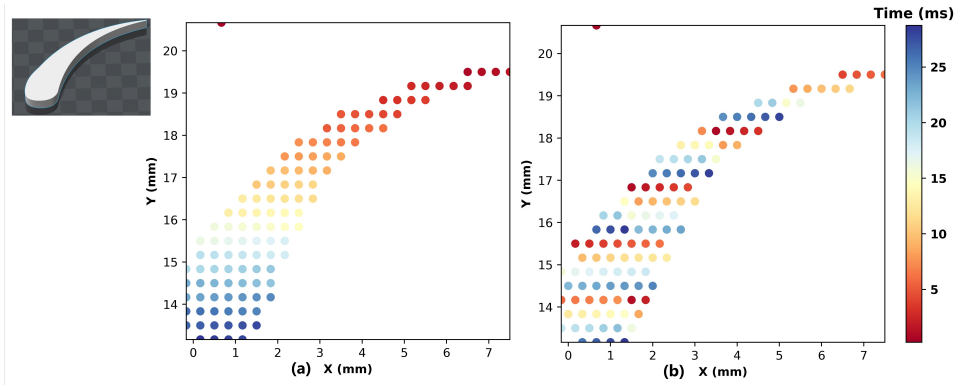


Figure 8.1: Sequence of fill in an airfoil (a) Raster fill (b) Dehoff fill or spot fill.

Corresponding G and R plot is given in Figure 8.2. There is no significant difference between the 2 cases. Effect of scan pattern on complex geometry was discussed briefly in this section and further investigation is necessary. Innovative scan strategies have to be taken into consideration to achieve on-demand solidification microstructure control for complex geometries. This is just a proof of concept that the framework and proposed approach can be used for complex geometries. Detailed investigation can be made to design the scan strategy for complex geometry.

Fidelity Analysis and Uncertainty Quantification: Through the knowledge gained from this work, it would be interesting to analyze the effect of the fidelity of the numerical models on solidification microstructure. Is it vital to include the beam-powder interaction in the model? Is free surface modeling important to understand the solidification microstructure? Models across scales have to be coupled with uncertainty quantification tools like Dakota [137] and PUQ [138] to check for the sensitivity of the output as a function

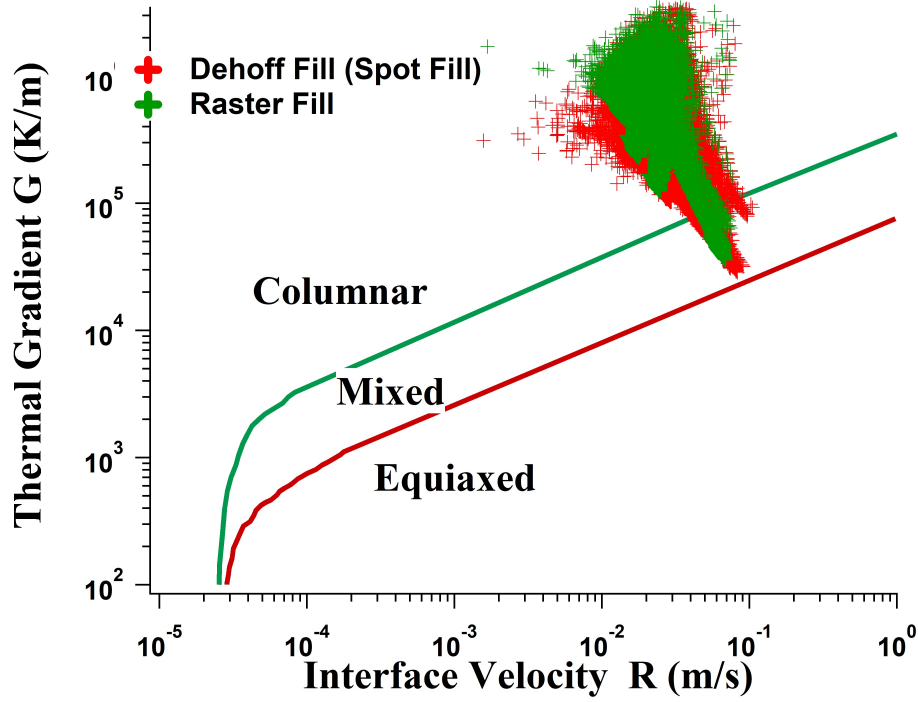


Figure 8.2: G vs R for the raster fill and "Dehoff" fill

of input material properties.

Alloy Design for AM: Most of the current researches in AM are focussed on processing existing commercial alloys which are processed through casting. Since AM is highly transient in nature, the solidification phenomenon including segregation, undercooling and solid state precipitation will be different compared to casting processes. Alloy design/modifications in specific for AM is an important topic to be discussed. Is it possible to modify alloy compositions and/or add inoculants/nucleating agents which can promote equiaxed grains for complex geometries but at the same time maintain the columnar growth when necessary within the processing window? Recent work by Martin et al. [139] showed the possibility of mitigating cracking of additively fabricated high strength Aluminum alloys by adding inoculants in the form of Zr nanoparticles and producing equiaxed grains. However the thermodynamic reasoning behind the choice of nucleant was not discussed comprehensively. Modification of alloy chemistry based on interface response function theory for columnar to equiaxed transition is an important research topic to be addressed.

Integration With Machine Learning Algorithms: One of the significant unanswered

questions in this work of controlling grain morphology is the design of scan strategy for complex geometries. As described in Chapter 5 (Table 5.3), for any given geometry, the grain morphology control is dictated by the local preheat temperature of the substrate. Local preheat temperature is dictated by the competition between the energy deposition rate and energy dissipation rate. Energy deposition rate is controlled by the frequency of the adjacent spots in the scan strategy. Frequency analysis has to be done to control the energy deposition rate. Since complex geometries will have complex boundary conditions (overhangs vs no-overhangs - powder vs solid), the energy dissipation rate will vary with respect to the geometry. Energy dissipation rate can be studied as a function of geometry by coupling the numerical modeling with machine learning algorithms. But simulating the entire part will be computationally expensive. A brief suggestion that can be developed into a comprehensive research work is detailed below. In any geometry, highly transient heat transfer effect can be observed only in the top few layers Figure 8.3 (a). The rest of the previously built layers will be at steady state (comparatively). Hence, it is sufficient to understand the effect of processing parameters in the transient region to control the solidification microstructure. The transient region can then be spatially discretized into a binary matrix format of 1's and 0's. 1's represent solid (bulk) part and 0's represent powder region. This essentially represents a complex geometry with complex boundary conditions. Figure 8.3 (b) shows the matrix format of a triangle built without any overhangs and Figure 8.3 (c) shows the triangle with overhangs. Numerical simulations can be performed for different frequencies of the spots and different boundary conditions. The results can be analyzed by using sophisticated machine learning algorithms and a surrogate model can be created. The surrogate model can be used to reduce the computational expense and optimize the scan strategy as a function of geometry.

Integrated Computational Framework: Looking from a larger perspective, the numerical codes across different time and length scales can be coupled along with machine learning algorithms to develop an integrated computational framework to predict the process-structure-property linkage. Schematic of the proposed approach is given in Figure 8.4. Using this framework, the lead time and experimental expense in designing new alloy systems and time taken to optimize the process parameters can be reduced significantly.

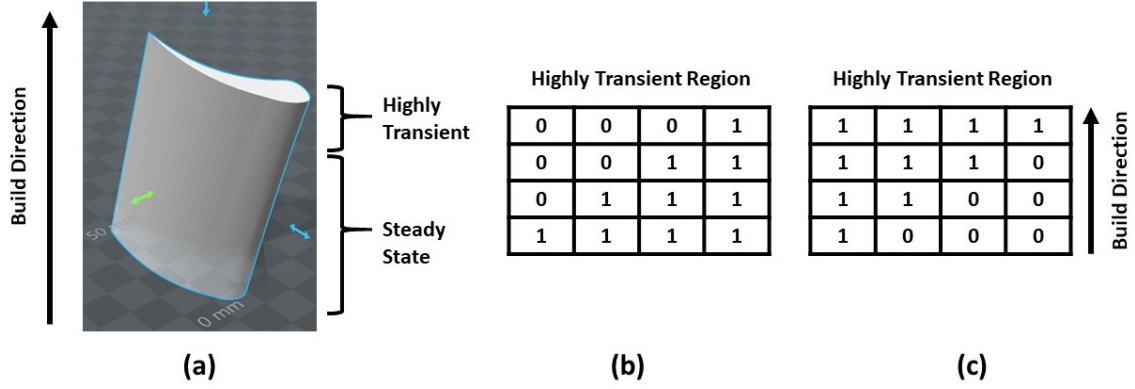


Figure 8.3: (a) Variation in transient nature as a function of build height (b) Matrix format of complex geometry (triangle) without overhang (c) with overhang.

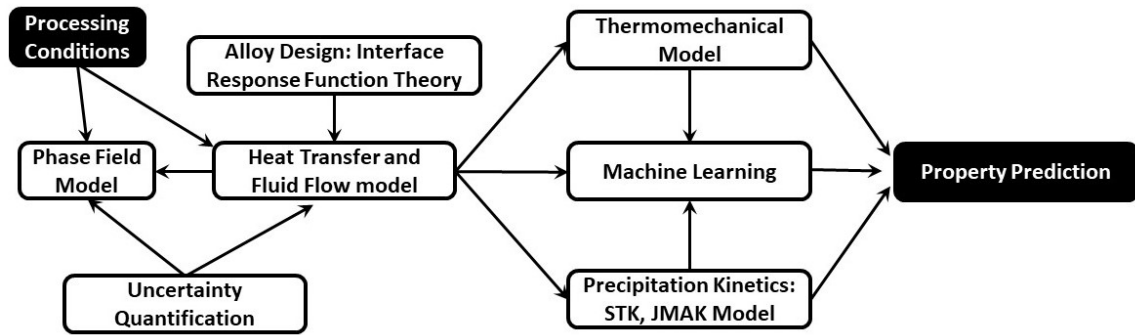


Figure 8.4: Integrated Computational and Machine Learning Framework to predict process-structure-property in AM process.

Bibliography

- [1] Kaufui V Wong and Aldo Hernandez. “A review of additive manufacturing”. In: *ISRN Mechanical Engineering* 2012 (2012).
- [2] Jianing Guo. “Feature based cost and carbon emission modelling for wire and arc additive manufacturing”. In: (2012).
- [3] Nanond Nopparat and Babak Kianian. *Resource Consumption of Additive Manufacturing Technology*. 2012.
- [4] Todd Rockstroh. *Additive manufacturing at GE Aviation - Lessons learned from development cycle*. 2013.
- [5] Tomas Kellner. *The FAA Cleared The First 3D Printed Part To Fly In A Commercial Jet Engine From GE*. <http://www.gereports.com/post/116402870270/the-faa-cleared-the-first-3d-printed-part-to-fly/>. [Online; accessed 2-July-2017]. 2015.
- [6] Zachary C Cordero et al. “Powder bed charging during electron-beam additive manufacturing”. In: *Acta Materialia* 124 (2017), pp. 437–445.
- [7] Satya S Gajapathi, Sushanta K Mitra, and Patricio F Mendez. “Controlling heat transfer in micro electron beam welding using volumetric heating”. In: *International Journal of Heat and Mass Transfer* 54.25 (2011), pp. 5545–5553.
- [8] Arcam. *Arcam Q10 Datasheet*.
- [9] LM Sochalski-Kolbus et al. “Comparison of residual stresses in Inconel 718 simple parts made by electron beam melting and direct laser metal sintering”. In: *Metallurgical and Materials Transactions A* 46.3 (2015), pp. 1419–1432.
- [10] Arcam. *EBM Hardware*. <http://www.arcam.com/technology/electron-beam-melting/hardware/>. [Online; accessed 2-July-2017]. 2017.
- [11] Tresa M Pollock and Sammy Tin. “Nickel-based superalloys for advanced turbine engines: chemistry, microstructure, and properties”. In: *Journal of propulsion and power* 22.2 (2006), pp. 361–374.

- [12] Markus Ramsperger, Robert F Singer, and Carolin Körner. “Microstructure of the Nickel-Base superalloy CMSX-4 fabricated by selective electron beam melting”. In: *Metallurgical and Materials Transactions* 47.3 (2016), p. 1469.
- [13] Luke N Carter et al. “The influence of the laser scan strategy on grain structure and cracking behaviour in SLM powder-bed fabricated nickel superalloy”. In: *Journal of Alloys and Compounds* 615 (2014), pp. 338–347.
- [14] RR Boyer. “An overview on the use of titanium in the aerospace industry”. In: *Materials Science and Engineering: A* 213.1-2 (1996), pp. 103–114.
- [15] Donald M Brunette et al. *Titanium in medicine: material science, surface science, engineering, biological responses and medical applications*. Springer Science & Business Media, 2012.
- [16] André Luiz Jardini et al. “Cranial reconstruction: 3D biomodel and custom-built implant created using additive manufacturing”. In: *Journal of Cranio-Maxillofacial Surgery* 42.8 (2014), pp. 1877–1884.
- [17] LE Murr et al. “Next-generation biomedical implants using additive manufacturing of complex, cellular and functional mesh arrays”. In: *Philosophical Transactions of the Royal Society of London A: Mathematical, Physical and Engineering Sciences* 368.1917 (2010), pp. 1999–2032.
- [18] DM Dimiduk. “Gamma titanium aluminide alloys an assessment within the competition of aerospace structural materials”. In: *Materials Science and Engineering: A* 263.2 (1999), pp. 281–288.
- [19] Young-Won Kim. “Intermetallic alloys based on gamma titanium aluminide”. In: *JOM Journal of the Minerals, Metals and Materials Society* 41.7 (1989), pp. 24–30.
- [20] Lawrence E Murr et al. “Characterization of titanium aluminide alloy components fabricated by additive manufacturing using electron beam melting”. In: *Acta Materialia* 58.5 (2010), pp. 1887–1894.

- [21] HP Tang et al. “Additive manufacturing of a high niobium-containing titanium aluminide alloy by selective electron beam melting”. In: *Materials Science and Engineering: A* 636 (2015), pp. 103–107.
- [22] T Ahmed and HJ Rack. “Phase transformations during cooling in $\alpha + \beta$ titanium alloys”. In: *Materials Science and Engineering: A* 243.1 (1998), pp. 206–211.
- [23] Xishan Xie et al. “TTT Diagram of a Newly Developed Nickel Base Superalloy Allvac® 718Plus”. In: *Sixth International Special Emphasis Symposium on Superalloys 718, 625, 706 and Derivatives*. 2005, pp. 193–202.
- [24] Armida Oradei-Basile and John F Radavich. “A current TTT diagram for wrought alloy 718”. In: *Superalloys 718.625* (1991), pp. 325–335.
- [25] SA David and JM Vitek. “Correlation between solidification parameters and weld microstructures”. In: *International Materials Reviews* 34.1 (1989), pp. 213–245.
- [26] W Kurz and DJ Fisher. *Fundamentals of solidification, 1986*. Trans Tech Publications, Switzerland.
- [27] Jonathan A Dantzig and Michel Rappaz. *Solidification: -Revised & Expanded*. EPFL press, 2016.
- [28] Sindo Kou. *Welding metallurgy*. John Wiley & Sons, 2003.
- [29] S Kou and Y Le. “Nucleation mechanism and grain refining of weld metal”. In: *Welding Journal* 65.12 (1986), 305s–313s.
- [30] Mark Easton and David StJohn. “Grain refinement of aluminum alloys: Part II. Confirmation of, and a mechanism for, the solute paradigm”. In: *Metallurgical and Materials Transactions A* 30.6 (1999), pp. 1625–1633.
- [31] YC Lim et al. “Effect of magnetic stirring on grain structure refinement: Part 1—Autogenous nickel alloy welds”. In: *Science and Technology of Welding and Joining* 15.7 (2010), pp. 583–589.
- [32] M Gäumann et al. “Single-crystal laser deposition of superalloys: processing—microstructure maps”. In: *Acta Materialia* 49.6 (2001), pp. 1051–1062.

- [33] M Gäumann, R Trivedi, and W Kurz. “Nucleation ahead of the advancing interface in directional solidification”. In: *Materials Science and Engineering: A* 226 (1997), pp. 763–769.
- [34] JM Vitek. “The effect of welding conditions on stray grain formation in single crystal welds—theoretical analysis”. In: *Acta materialia* 53.1 (2005), pp. 53–67.
- [35] Helmut Schultz. *Electron beam welding*. Elsevier, 1994.
- [36] Steigerwald. “Jahre Elektronenstrahl-tiefschweißen. Schw. Schn”. In: (1983), pp. 589–592.
- [37] J Elmer et al. *Introduction to High Energy Density Electron and Laser Beam Welding*. Tech. rep. Lawrence Livermore National Laboratory (LLNL), Livermore, CA, 2009.
- [38] Robert W Messler Jr. *Principles of welding: processes, physics, chemistry, and metallurgy*. John Wiley & Sons, 2008.
- [39] Z Sun and R Karppi. “The application of electron beam welding for the joining of dissimilar metals: an overview”. In: *Journal of Materials Processing Technology* 59.3 (1996), pp. 257–267.
- [40] Jonghyun Kim and Y Kawamura. “Electron beam welding of the dissimilar Zr-based bulk metallic glass and Ti metal”. In: *Scripta Materialia* 56.8 (2007), pp. 709–712.
- [41] M Rappaz et al. “Analysis of solidification microstructures in Fe-Ni-Cr single-crystal welds”. In: *Metallurgical Transactions A* 21.6 (1990), pp. 1767–1782.
- [42] SA David et al. “Microstructure of stainless steel single-crystal electron beam welds”. In: *Metallurgical Transactions A* 21.6 (1990), pp. 1753–1766.
- [43] SF Su et al. “Electron-beam welding behavior in Mg-Al-based alloys”. In: *Metallurgical and Materials Transactions A* 33.5 (2002), pp. 1461–1473.
- [44] Rosenthal D. “Nucleation ahead of the advancing interface in directional solidification”. In: *Weld J* 20 (1941), p. 220.
- [45] Oystein Grong. “Metallurgical Modelling of Welding.” In: *Institute of Materials, 1 Carlton House Terrace, London, SW 1 Y 5 DB, UK, 1997. 605* (1997).

- [46] AC Nunes. “An extended Rosenthal weld model”. In: *Welding journal* 62.6 (1983), 165s–170s.
- [47] Nunsian Tsai. “Heat distribution and weld bead geometry in arc welding”. PhD thesis. Massachusetts Institute of Technology, 1983.
- [48] U Gratzke et al. “Theoretical approach to the humping phenomenon in welding processes”. In: *Journal of Physics D: Applied Physics* 25.11 (1992), p. 1640.
- [49] Michael F Zäh and S Lutzmann. “Modelling and simulation of electron beam melting”. In: *Production Engineering* 4.1 (2010), pp. 15–23.
- [50] TC Nguyen et al. “The humping phenomenon during high speed gas metal arc welding”. In: *Science and Technology of Welding and Joining* 10.4 (2005), pp. 447–459.
- [51] T Zacharia et al. “Three-dimensional transient model for arc welding process”. In: *Metallurgical and Materials Transactions B* 20.5 (1989), pp. 645–659.
- [52] W Zhang et al. “Modeling of heat transfer and fluid flow during gas tungsten arc spot welding of low carbon steel”. In: *Journal of Applied Physics* 93.5 (2003), pp. 3022–3033.
- [53] R Rai et al. “Heat transfer and fluid flow during electron beam welding of 21Cr–6Ni–9Mn steel and Ti–6Al–4V alloy”. In: *Journal of Physics D: Applied Physics* 42.2 (2008), p. 025503.
- [54] X He, PW Fuerschbach, and T DebRoy. “Heat transfer and fluid flow during laser spot welding of 304 stainless steel”. In: *Journal of Physics D: Applied Physics* 36.12 (2003), p. 1388.
- [55] T Zacharia et al. “Modeling of fundamental phenomena in welds”. In: *Modelling and Simulation in Materials Science and Engineering* 3.2 (1995), p. 265.
- [56] R Rai et al. “Heat transfer and fluid flow during keyhole mode laser welding of tantalum, Ti–6Al–4V, 304L stainless steel and vanadium”. In: *Journal of physics D: Applied physics* 40.18 (2007), p. 5753.

- [57] Steven Price, Ken Cooper, and Kevin Chou. “Evaluations of temperature measurements by near-infrared thermography in powder-based electron-beam additive manufacturing”. In: *Proceedings of the Solid Freeform Fabrication Symposium*. University of Texas, Austin, TX. 2012, pp. 761–773.
- [58] Steven Price et al. “Experimental temperature analysis of powder-based electron beam additive manufacturing”. In: *Proceedings of the Solid Freeform Fabrication Symposium*. 2013, pp. 162–173.
- [59] Jorge Mireles et al. “Closed-loop automatic feedback control in electron beam melting.” In: *International Journal of Advanced Manufacturing Technology* 78 (2015).
- [60] Gustavo Tapia and Alaa Elwany. “A review on process monitoring and control in metal-based additive manufacturing”. In: *Journal of Manufacturing Science and Engineering* 136.6 (2014), p. 060801.
- [61] A Raghavan et al. “Heat transfer and fluid flow in additive manufacturing”. In: *Journal of Laser Applications* 25.5 (2013), p. 052006.
- [62] Adam Clayton Powell IV. “Transport phenomena in electron beam melting and evaporation”. PhD thesis. Massachusetts Institute of Technology, 1997.
- [63] Tl DebRoy and SA David. “Physical processes in fusion welding”. In: *Reviews of modern physics* 67.1 (1995), p. 85.
- [64] PAA Khan and T DebRoy. “Alloying element vaporization and weld pool temperature during laser welding of AISI 202 stainless steel”. In: *Metallurgical Transactions B* 15.4 (1984), pp. 641–644.
- [65] EA Metzbower. “Keyhole formation”. In: *Metallurgical and Materials Transactions B* 24.5 (1993), pp. 875–880.
- [66] P Sahoo, MM Collur, and T DebRoy. “Effects of oxygen and sulfur on alloying element vaporization rates during laser welding”. In: *Metallurgical and Materials Transactions B* 19.6 (1988), pp. 967–972.

- [67] K Mundra and T DebRoy. “Toward understanding alloying element vaporization during laser beam welding of stainless steel”. In: *WELDING JOURNAL-NEW YORK*- 72 (1993), 1–s.
- [68] X He, T DebRoy, and PW Fuerschbach. “Alloying element vaporization during laser spot welding of stainless steel”. In: *Journal of Physics D: Applied Physics* 36.23 (2003), p. 3079.
- [69] X He, T DebRoy, and PW Fuerschbach. “Probing temperature during laser spot welding from vapor composition and modeling”. In: *Journal of Applied Physics* 94.10 (2003), pp. 6949–6958.
- [70] Wayne E King et al. “Observation of keyhole-mode laser melting in laser powder-bed fusion additive manufacturing”. In: *Journal of Materials Processing Technology* 214.12 (2014), pp. 2915–2925.
- [71] Fencheng Liu et al. “The effect of laser scanning path on microstructures and mechanical properties of laser solid formed nickel-base superalloy Inconel 718”. In: *Journal of Alloys and Compounds* 509.13 (2011), pp. 4505–4509.
- [72] Alphons Anandaraj Antonysamy. “Microstructure, texture and mechanical property evolution during additive manufacturing of Ti6Al4V alloy for aerospace applications”. In: (2012).
- [73] Qingbo Jia and Dongdong Gu. “Selective laser melting additive manufacturing of Inconel 718 superalloy parts: Densification, microstructure and properties”. In: *Journal of Alloys and Compounds* 585 (2014), pp. 713–721.
- [74] Lawrence E Murr et al. “Metal fabrication by additive manufacturing using laser and electron beam melting technologies”. In: *Journal of Materials Science & Technology* 28.1 (2012), pp. 1–14.
- [75] William Sames. “Additive manufacturing of Inconel 718 using electron beam melting: Processing, post-processing, & Mechanical properties”. PhD thesis. 2015.

- [76] Srikanth Bontha et al. “Effects of process variables and size-scale on solidification microstructure in beam-based fabrication of bulky 3D structures”. In: *Materials Science and Engineering: A* 513 (2009), pp. 311–318.
- [77] H Helmer et al. “Grain structure evolution in Inconel 718 during selective electron beam melting”. In: *Materials Science and Engineering: A* 668 (2016), pp. 180–187.
- [78] RR Dehoff et al. “Site specific control of crystallographic grain orientation through electron beam additive manufacturing”. In: *Materials Science and Technology* 31.8 (2015), pp. 931–938.
- [79] Special Metals. *Composition of Inconel 718*. http://www.specialmetals.com/assets/smc/documents/inconel_alloy_718.pdf. [Online; accessed 25-July-2017]. 2017.
- [80] DA Korzekwa. “Truchas—a multi-physics tool for casting simulation”. In: *International Journal of Cast Metals Research* 22.1-4 (2009), pp. 187–191.
- [81] L Parietti and K Lam. “Validation of a multiphysics software package for simulations of pulsed laser welding at Los Alamos National Laboratory”. In: *Technical Report LA-UR-06 7622* (2006).
- [82] Marianne M Francois, James M Sicilian, and Douglas B Kothe. “Modeling of thermocapillary forces within a volume tracking algorithm”. In: *Charles-André Gandin, Bellet, Michel, editos. Modeling of casting, welding and advanced solidification processes—XI, TMS (the minerals, metals & materials society)* (2006), pp. 935–42.
- [83] Marianne M François. “Recent numerical and algorithmic advances within the volume tracking framework for modeling interfacial flows”. In: *Procedia IUTAM* 15 (2015), pp. 270–277.
- [84] Henry G Weller et al. “A tensorial approach to computational continuum mechanics using object-oriented techniques”. In: *Computers in physics* 12.6 (1998), pp. 620–631.
- [85] Siegfried Schiller, Ullrich Heisig, and Siegfried Panzer. *Electron beam technology*. John Wiley & Sons, 1982.

- [86] Ying Qin et al. “Temperature–stress fields and related phenomena induced by a high current pulsed electron beam”. In: *Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms* 225.4 (2004), pp. 544–554.
- [87] Peeyush Nandwana et al. “Recyclability study on Inconel 718 and Ti-6Al-4V powders for use in electron beam melting”. In: *Metallurgical and Materials Transactions* 47.1 (2016), p. 754.
- [88] Arcam. *Q10 Datasheet*. URL: <http://www.arcam.com/wp-content/uploads/Arcam-Q10.pdf> (visited on 06/27/2017).
- [89] Benjamin P Keller et al. “Total hemispherical emissivity of Inconel 718”. In: *Nuclear Engineering and Design* 287 (2015), pp. 11–18.
- [90] Mohd Rizal Alkahari et al. “Thermal conductivity of metal powder and consolidated material fabricated via selective laser melting”. In: *Key Engineering Materials*. Vol. 523. Trans Tech Publ. 2012, pp. 244–249.
- [91] Ted D Blacker, William J Bohnhoff, and Tony L Edwards. *CUBIT mesh generation environment. Volume 1: Users manual*. Tech. rep. Sandia National Labs., Albuquerque, NM (United States), 1994.
- [92] DE Keyes. “How scalable is domain decomposition in practice”. In: *Proceedings of the 11th International Conference on Domain Decomposition Methods*. 1998, pp. 286–297.
- [93] S Kamnis, Sai Gu, and N Zeoli. “Mathematical modelling of Inconel 718 particles in HVOF thermal spraying”. In: *Surface and Coatings Technology* 202.12 (2008), pp. 2715–2724.
- [94] G Pottlacher et al. “Thermophysical properties of solid and liquid Inconel 718 Alloy”. In: *Scandinavian Journal of Metallurgy* 31.3 (2002), pp. 161–168.
- [95] M Rappaz. “Modelling of microstructure formation in solidification processes”. In: *International Materials Reviews* 34.1 (1989), pp. 93–124.
- [96] VISIT Team et al. “VISIT: Software the Delivers Parallel Interactive Visualization”. In: *Lawrence Livermore National Laboratory*, <http://www.llnl.gov/visit> (2003).

- [97] L Nastac et al. “Advances in the Solidification of IN718 and RS5 Alloys”. In: *Proceedings of Superalloys 718, 625, 706 and Various Derivatives* (2001).
- [98] X He, JW Elmer, and T DebRoy. “Heat transfer and fluid flow in laser microwelding”. In: *Journal of Applied Physics* 97.8 (2005), p. 084909.
- [99] George Ellwood Dieter and David J Bacon. *Mechanical metallurgy*. Vol. 3. McGraw-Hill New York, 1986.
- [100] Stolof. “Microstructure and Properties of Materials: (Volume 1)”. In: ed. by JCM Li. 1996. Chap. 2, pp. 51–107.
- [101] J Drapier. “High temperature alloys for gas turbines”. In: *Applied Science, London* (1978), p. 701.
- [102] M Lamm and RF Singer. “The effect of casting conditions on the high-cycle fatigue properties of the single-crystal nickel-base superalloy PWA 1483”. In: *Metallurgical and Materials Transactions A* 38.6 (2007), pp. 1177–1183.
- [103] Rohit Trivedi. “Interdendritic spacing: Part II. A comparison of theory and experiment”. In: *Metallurgical Transactions A* 15.6 (1984), pp. 977–982.
- [104] W Kurz and DJ Fisher. “Dendrite growth at the limit of stability: tip radius and spacing”. In: *Acta Metallurgica* 29.1 (1981), pp. 11–20.
- [105] JS Langer and J Müller-Krumbhaar. “Stability effects in dendritic crystal growth”. In: *Journal of Crystal Growth* 42 (1977), pp. 11–14.
- [106] Yousub Lee et al. “Effect of fluid convection on dendrite arm spacing in laser deposition”. In: *Metallurgical and Materials Transactions B* 45.4 (2014), pp. 1520–1529.
- [107] MM Kirka et al. “Microstructure Development in Electron Beam-Melted Inconel 718 and Associated Tensile Properties”. In: *JOM* 68.3 (2016), pp. 1012–1020.
- [108] Fan Zhang et al. “Homogenization kinetics of a nickel-based superalloy produced by powder bed fusion laser sintering”. In: *Scripta Materialia* 131 (2017), pp. 98–102.
- [109] C Beckermann, JP Gu, and William J Boettinger. “Development of a freckle predictor via Rayleigh number method for single-crystal nickel-base superalloy castings”. In: *Metallurgical and materials transactions A* 31.10 (2000), pp. 2545–2557.

- [110] JW Aveson et al. “Origins of misorientation defects in single crystal castings: A time resolved in situ synchrotron X-ray radiography study”. In: *MATEC Web of Conferences*. Vol. 14. EDP Sciences. 2014, p. 05003.
- [111] David A Porter, Kenneth E Easterling, and Mohamed Sherif. *Phase Transformations in Metals and Alloys, (Revised Reprint)*. CRC press, 2009.
- [112] MJ Uttormark, JW Zanter, and JH Perepezko. “Repeated nucleation in an under-cooled aluminum droplet”. In: *Journal of crystal growth* 177.3-4 (1997), pp. 258–264.
- [113] Bin Yang et al. “Cooling rate dependence of undercooling of pure Sn single drop by fast scanning calorimetry”. In: *Applied Physics A: Materials Science & Processing* 104.1 (2011), pp. 189–196.
- [114] Michel Rappaz and Jonathan A Dantzig. *Solidification*. EPFL press, 2009.
- [115] HL Wei, JW Elmer, and T DebRoy. “Three-dimensional modeling of grain structure evolution during welding of an aluminum alloy”. In: *Acta Materialia* (2017).
- [116] Narendran Raghavan et al. “Numerical modeling of heat-transfer and the influence of process parameters on tailoring the grain morphology of IN718 in electron beam additive manufacturing”. In: *Acta Materialia* 112 (2016), pp. 303–314.
- [117] JD Hunt. “Steady state columnar and equiaxed growth of dendrites and eutectic”. In: *Materials science and engineering* 65.1 (1984), pp. 75–83.
- [118] TD Anderson, JN DuPont, and T DebRoy. “Origin of stray grain formation in single-crystal superalloy weld pools from heat transfer and fluid flow modeling”. In: *Acta Materialia* 58.4 (2010), pp. 1441–1454.
- [119] Narendran Raghavan et al. “Corrigendum to Numerical modeling of heat-transfer and the influence of process parameters on tailoring the grain morphology of IN718 in electron beam additive manufacturing *Acta Materialia* 112C (2016) 303-314”. In: *Acta Materialia* 140 (2017), p. 472.
- [120] Douglas C Montgomery. *Design and analysis of experiments*. John Wiley & Sons, 2017.

- [121] J Raplee et al. “Thermographic Microstructure Monitoring in Electron Beam Additive Manufacturing”. In: *Scientific Reports* 7 (2017), p. 43554.
- [122] Ralph B Dinwiddie et al. “Thermographic in-situ process monitoring of the electron-beam melting technology used in additive manufacturing”. In: *Proc. SPIE K* 87050 (2013), 87050K–87059K.
- [123] RB Dinwiddie et al. “Calibrating IR cameras for in-situ temperature measurement during the electron beam melt processing of Inconel 718 and Ti-Al6-V4”. In: *SPIE Commercial+ Scientific Sensing and Imaging*. International Society for Optics and Photonics. 2016, pp. 986107–986107.
- [124] GM Oreper, TW Eagar, and J Szekely. “Convection in arc weld pools”. In: *Welding journal* 62.11 (1983), pp. 307–312.
- [125] S Kou and YH Wang. “Weld pool convection and its effect”. In: *Weld. J* 65.3 (1986), 63s–70s.
- [126] CR Heiple and JR Roper. “Effect of selenium on GTAW fusion zone geometry”. In: *Welding journal* 60.8 (1981), p. 143.
- [127] CA Heiple. “Mechanism for minor element effect on GTA fusion zone geometry”. In: *Welding journal* 61 (1982), pp. 975–1025.
- [128] CR Heiple and JR Roper. *Effects of minor elements on GTAW fusion-zone shape*. Tech. rep. Rockwell International Corp., Golden, CO (USA). Rocky Flats Plant, 1981.
- [129] CR Heiple et al. “Surface active element effects on the shape of GTA, laser and electron beam welds”. In: *Weld. J.* 62.3 (1983), p. 72.
- [130] CR Heiple and P Burgardt. “Effects of SO₂ shielding gas additions on GTA weld shape”. In: *Welding journal* 64.6 (1985), pp. 159–162.
- [131] P Sahoo, T DebRoy, and MJ McNallan. “Surface tension of binary metal surface active solute systems under conditions relevant to welding metallurgy”. In: *Metallurgical and Materials Transactions B* 19.3 (1988), pp. 483–491.
- [132] KC Mills and BJ Keene. “Factors affecting variable weld penetration”. In: *International Materials Reviews* 35.1 (1990), pp. 185–216.

- [133] PD Lee, PN Quested, and M McLean. “Modelling of Marangoni effects in electron beam melting”. In: *PHILOSOPHICAL TRANSACTIONS-ROYAL SOCIETY OF LONDON SERIES A MATHEMATICAL PHYSICAL AND ENGINEERING SCIENCES* (1998), pp. 1027–1044.
- [134] Brian P Leonard. “A stable and accurate convective modelling procedure based on quadratic upstream interpolation”. In: *Computer methods in applied mechanics and engineering* 19.1 (1979), pp. 59–98.
- [135] Narendran Raghavan et al. “Localized melt-scan strategy for site specific control of grain size and primary dendrite arm spacing in electron beam additive manufacturing”. In: *Acta Materialia* 140 (2017), pp. 375–387.
- [136] Caroline A Schneider, Wayne S Rasband, and Kevin W Eliceiri. “NIH Image to ImageJ: 25 years of image analysis”. In: *Nature methods* 9.7 (2012), pp. 671–675.
- [137] Brian M Adams et al. “DAKOTA, a multilevel parallel object oriented framework for design optimization, parameter estimation, uncertainty quantification, and sensitivity analysis version 5.0 users manual”. In: *Sandia National Laboratories, Tech. Rep. SAND 2010-2183* (2009).
- [138] Martin Hunt et al. “PUQ: A code for non intrusive uncertainty propagation in computer simulations”. In: *Computer Physics Communications* 194 (2015), pp. 97–107.
- [139] John H Martin et al. “3D printing of high-strength aluminium alloys”. In: *Nature* 549.7672 (2017), pp. 365–369.
- [140] D Turnbull. “Formation of crystal nuclei in liquid metals”. In: *Journal of Applied Physics* 21.10 (1950), pp. 1022–1028.

Appendices

Appendix A

Appendix-1

A.1 Nucleation Rate as a Function of Cooling Rate

Cooling rate is defined by

$$CoolingRate = \frac{dT}{dt} \quad (A.1)$$

Number of nuclei formed in a given liquid melt at a given time can be formulated as

$$n(t) = \int (N_s * S + N_v * V) dt \quad (A.2)$$

where, $n(t)$ is the number of nuclei a time t , N_s is the nucleation rate per unit surface area of the liquid melt, S is the surface area of the liquid melt, N_v is the nucleation rate per unit volume of the liquid melt, V is the volume of the liquid melt.

When the first critical nucleus is formed, the integral of the Equation (A.2) from the liquidus temperature (T_l) to nucleation temperature (T_n) should be equal to unity. From Equation (A.1) and Equation (A.2), following equation can be derived.

$$\frac{1}{CoolingRate} \int_{T_l}^{T_n} (N_s * S + N_v * V) dT = 1 \quad (A.3)$$

Undercooling is be defined by

$$\Delta T = T_l - T_n \quad (A.4)$$

From classical nucleation theory, analytical equation was derived [112, 113, 140] to predict the surface nucleation rate for spherical liquid droplet.

$$N_s = \frac{n_s * \Phi * (1 - \cos \theta)}{\Delta T} * \exp \left(\frac{-\Psi * F_s}{(T_m - \Delta T) \Delta T^2} \right) \quad (\text{A.5})$$

where,

$$\Phi = \frac{8\pi * \sigma^2 * D_l * T_m^2}{a_o^4 \Delta H^2} \quad (\text{A.6})$$

$$\Psi = \frac{16\pi * \sigma^3 * T_m^2}{3 * k * \Delta H^2} \quad (\text{A.7})$$

$$n_s = \frac{c_s}{a_o^2} \quad (\text{A.8})$$

$$F_s = F_o * f(\theta) \quad (\text{A.9})$$

$$f(\theta) = \frac{(2 + \cos \theta)(1 - \cos \theta)^2}{4} \quad (\text{A.10})$$

where $f(\theta)$ is the contact angle factor between the liquid melt surface and the catalytic heterogeneous surface. In this case the value of θ is close to 90. Hence $f(\theta) = 0.5$. ΔH is the melting enthalpy, c_s is the surface impurity concentration, T_m is the melting point, σ is the solid-liquid interfacial energy, D_l is the diffusivity in liquid metal, a_o is the atomic spacing, k is the Boltzmann constant. These equations can be simplified to obtain Equation (4.6) providing relationship between the cooling rate and undercooling.

Appendix B

Appendix-2

B.1 CUBIT: Mesh Generation Script Template

```
# Delete existing mesh
reset

# Specify the XYZ dimensions of the entire domain
create brick x 0.008 y 0.008 z 0.0007

# Move to make z=0 on surface and X and Y appropriately
move volume 1 z -0.0004
move volume 1 x +0.0025
move volume 1 y +0.0025

# Number of spatial discretisation in X
curve 2 4 6 8 interval 266

# Number of spatial discretisation in Y
curve 1 3 5 7 interval 266

# Number of spatial discretization in Z
curve 9 10 11 12 interval 28

# Specify sidesets that will help in mentioning Boundary Conditions while running the
program
sideset 1 surface 1
sideset 2 surface 2
```

```

sideset 3 surface 3
sideset 4 surface 4
sideset 5 surface 5
sideset 6 surface 6
preview mesh volume all
mesh volume all
block 1 volume 1
block 1 element type HEX
nodeset 100 volume all
set large exodus file off
# export the mesh file *.exo to specified path Change the path in the following command
export mesh "/home/n22/block.exo" block 1 overwrite

```

B.2 Python script to filter G and R at interface

```

#!/usr/bin/python

import os
import sys
sys.path.append('/home/n22/softwares/visit/2_10/2.10.2/linux-x86_64/lib/site-packages')
from visit import *
import glob

LaunchNowin()

# Construct Thermal Gradient vector and filter the thermal
  gradient at the liquid solid interface and the liquid solid
  interface velocity with help of cooling rate
OpenDatabase("MAIN-data-*.gmv_database", 0)

```

```

DefineScalarExpression("Cooling_Rate", "<dT/dt>")
DefineVectorExpression("Thermal_Gradient_Vector", "{<dT/dx>,<dT/dy>,<dT/dz>}")
DefineScalarExpression("Thermal_Gradient_Magnitude", "magnitude(Thermal_Gradient_Vector)")
DefineScalarExpression("Thermal_Gradient_Interface", "if(and(and(lt(T,1610),_gt(T,1600)),lt(Cooling_Rate,0)),_abs(Thermal_Gradient_Magnitude),_0)")
DefineScalarExpression("LS_Interface_Velocity", "if(and(and(lt(T,1610),_gt(T,1600)),gt(Thermal_Gradient_Magnitude,1)),_abs(Cooling_Rate/(Thermal_Gradient_Magnitude)),_0)")

#Calculate number of time states
n=TimeSliderGetNStates()-1;
#Check for complete solidification
TS = open("Timestates.txt", "wb+")
TS.write(str(n));
TS.close()
AddPlot("Pseudocolor", "T")
DrawPlots()
SetTimeSliderState(n)
Query("Max")
max_temp = GetQueryOutputValue()
MT = open("max_temp.txt", "wb+")
MT.write(str(max_temp)+"_K"+"_is_the_maximum_temperature_at_the_final_time_step_of_the_simulation");
MT.close()
if max_temp > 1610:
    ER = open("postprocessing_failure.txt", "wb+")

```

```

ER.write("PostProcessing_has_been_terminated_prematurely.\n
        nMelt_pool_has_not_solidified_completely_when_simulation_
        ended.Try_increasing_bounds_of_Output_T_in_the_simulation_
        input_file.\nMaximum_Temperature_at_the_last_time_step_is:_
        "+str(max_temp)+"_K");
ER.close()
sys.exit()

```

#Export Interface Velocity in XYZ(.okc) format

```

DeleteAllPlots()
AddPlot("Pseudocolor","LS_Interface_Velocity")
DrawPlots()
for i in range(TimeSliderGetNStates()):
    SetActiveWindow(1)
    SetTimeSliderState(i)
    ExportDBAtts = ExportDBAttributes()
    ExportDBAtts.db_type = "Xmdv"
    ExportDBAtts.filename = "R%04d" %i
    ExportDBAtts.dirname = "."
    ExportDBAtts.variables = ("LS_Interface_Velocity")
    ExportDBAtts.opts.types = ()
    ExportDatabase(ExportDBAtts)

```

Export Interface Thermal Gradient in XYZ (.okc) format

```

DeleteAllPlots()
AddPlot("Pseudocolor","Thermal_Gradient_Interface")
DrawPlots()
for i in range(TimeSliderGetNStates()):
    SetActiveWindow(1)
    SetTimeSliderState(i)

```

```

ExportDBAtts = ExportDBAttributes()
ExportDBAtts.db_type = "Xmdv"
ExportDBAtts.filename = "G%04d" %i
ExportDBAtts.dirname = "."
ExportDBAtts.variables = ("Thermal_Gradient_Interface")
ExportDBAtts.opts.types = ()
ExportDatabase(ExportDBAtts)

sys.exit()

```

Vita

Narendran was born in TamilNadu, India. He completed his higher secondary education at Shree Vidhyalaya Matriculation Higher Secondary School, Gobichettipalayam in India. He graduated from College of Engineering Guindy, Chennai, India in May 2010 with an undergraduate degree in Electronics and Communication Engineering. Later, he worked for IBM India Pvt. Ltd as an associate system engineer for an year before moving to the United States in Fall 2011. After graduating with masters in renewable and clean energy from University of Dayton, Ohio, he moved to University of Tennessee, Knoxville in June 2013. He was part of Bredesen Center for Interdisciplinary Research and Graduate Education with his doctoral degree in Energy Science and Engineering. As a graduate research assistant, he worked at Manufacturing Demonstration Facility (MDF) at Oak Ridge National Laboratory (ORNL) for his research work. His PhD research work focused on understanding the solidification behavior during powder bed metal additive manufacturing with the help of numerical modeling. His research work resulted in 2 first author publications in the journal of Acta Materialia and 1 other publication where he co-authored in addition to 6 presentations in international conferences. Outside the research work, during spare time, he loves to play soccer and cricket.