

Monitoring the Metal Additive Manufacturing Process through
Thermographic Data Analysis

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Dedication

This thesis is dedicated to my mother and father, Scott and Margee Raplee; siblings Joshua and Sarah and to my girlfriend Rachel whom all supported me through my graduate studies and research.

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Abstract

Metal Additive Manufacturing (AM) is the formation of a solid metal part through the layer-wise melting of metal powder, wire, or thin sheets using various heat sources or other bonding methods. This manufacturing method provides nearly limitless complexity with decreased waste, energy needs, and lead time. However, the process faces challenges in part consistency and validation especially for high precision fields such as aerospace and defense. Research has sought to implement robust process monitoring techniques to increase consistency and the reliability of the AM process and detect vital information about the part such as microstructural development, and porosity formation so that eventually the proper control systems can be created to control the desired outcome. The research performed in this thesis seeks to utilize one of the more promising monitoring techniques for the metal PBF processes (selective laser sintering and electron beam melting), infrared (IR) thermography. However, little research has been performed using the technology, and therefore few monitoring applications for AM have been developed. The methods and results in this research will show two potential applications for the use of IR thermography to monitor AM materials: microstructural monitoring and porosity detection. The research will also discuss an algorithmic method for calibrating IR signals for in-situ emissivity change of the material to obtain a more accurate temperature history of a part during the build and direction for future work that needs to be addressed to advance the technology further.

Table of Contents

Chapter One : Introduction.....	1
Chapter Two : Literature Review and Background	3
2.1 The powder bed fusion additive manufacturing process.....	3
2.2 Principles of infrared (IR) thermography and emissivity.....	4
2.2.1 Infrared thermographic technology	4
2.2.2 Emissivity and its effects in the monitoring of AM processes.....	6
2.3 Current state of in-situ process monitoring of powder bed fusion (PBF) processes	7
2.3.1 In-situ process monitoring of selective laser sintering (SLS)	7
2.3.2 Monitoring of the EBM process	11
2.4 Overview of microstructural development within AM Inconel 718 and the effects on part performance.	13
2.4.1 Formation of columnar and equiaxed grains and in-situ control of microstructure	13
2.4.2 Effects of grain structure on part performance	15
2.5 Porosity formation and effects on strength and fatigue life of AM materials	18
2.6 Objectives of Current Research.....	19
Chapter Three : Materials and Methods	21
3.1 Thermographic Camera Setup for In-Situ Process monitoring	21
3.1.1 Camera Setup for the EBM Process on the ARCAM S12	21
3.1.2 Camera Setup for the SLS Process on the Renishaw AM250.....	23
3.2 Build setup, parameters, and description for microstructure monitoring of the EBM process and porosity monitoring of SLS	25
3.2.1 Experimental Setup: microstructure monitoring.....	25
3.2.2 Experimental Setup: porosity monitoring	27
3.3 Specimen preparation for microscopic analysis and electron backscatter diffraction (EBSD).....	27

3.4 Microscopic imaging, electron backscatter diffraction, and confocal microscopy	30
3.4.1 Optical Microscopy Imaging.....	30
3.4.2 Electron backscatter diffraction (EBSD) imaging	30
3.4.3 Surface topology measurement via confocal microscopy	30
Chapter Four : Correcting In-Situ Temperature Measurements for Emittance Change in the EBM Process	31
4.1 Calibrating the data to acquire the initial temperature curves	32
4.2 Filtering the data to remove false positives due to signal noise	32
4.3 Locating the powder to as-printed transition.....	35
4.4 Methods for determining the occurrence of melting	37
4.4.1 Determining melting through calculation	37
4.4.2 Setting the melt region using image registration.....	39
4.5 Results	41
4.6 Limitations and Future Work	44
Chapter Five : Microstructural Monitoring of EBM AM materials.....	51
5.1 Data analysis for microstructural monitoring	51
5.2 Results of the thermal gradient and solidification velocity calculations	53
5.3 Microstructural observations and correlation to thermal data.....	58
5.4 Limitations and further developments	63
Chapter Six : Porosity Monitoring of SLS AM Materials.....	65
6.1 Monitoring bulk build porosity	65
6.1.1 Data analysis.....	65
6.1.2 Analysis results and discussion.....	66
6.2 Monitoring site-specific porosity	83
6.2.1 Data Analysis	83
6.2.2 Analysis results and discussion.....	85

6.3 Sources of error, limitations, and future work	96
Chapter Seven : Conclusions and Future Work.....	99
7.1 Conclusions.....	99
7.2 Current Limitations and future work of thermographic data analysis in AM	100
7.2.1 Current Limitations and Concerns	100
7.2.2 Future Work	103
References.....	107
Appendices.....	113
Appendix A: Tables	114
Vita	120

List of Tables

Table A- 1 Table displaying relative advantages and disadvantages of the EBM and SLM processes.....	114
Table A- 2 Camera setups used.....	115
Table A-3: Build parameters for 25 cube matrix experiment*.....	116
Table A-4: List of Engineered Defects within the build and their grid coordinates.....	117
Table A-5: Weighting factors and frame numbers for use in the Savitzky-Golay filter calculations. The method of using this table is to sum the values obtained by multiplying the frames listed in column one with their respective weighting factor in column two and dividing that sum by the overall weighting factor given at the bottom of the table.	118
Table A-6: Table comparing the calculated volumetric heat input and the cube densities from both of the observed cube builds on the Renishaw AM250.....	119

List of Figures

Figure 2-1 Schematic representations of a line melt (left) and point melt (right) scan strategies.[52].....	16
Figure 2-2 Image showing microstructural control of Inconel 718 from Dehoff et al.[49] By modifying the localized scan strategies the authors were able to create an image within the material from the resultant microstructure.	16
Figure 2-3 Comparison of equiaxed (PHS) and columnar (Raster) material properties at (a) 20°C and (b) 650°C showing the relative isotropy and anisotropy of the materials respectively from Kirka et al. [50].....	17
Figure 2-4 Micrograph showing an example of lack of fusion porosity within SLS produced Ti-6Al-4V. Note the trapped powder particles that appear in the pores at the bottom of the image.	20
Figure 2-5 Micrograph of an SLS produced Ti-6Al-4V that exhibiting small pores that are possibly keyhole or gas porosity produced by excess energy density.....	20
Figure 3-1 A schematic of the IR camera setup used for the EBM process. Another schematic of the shutterless Kapton film canister can be seen as well (left).....	22
Figure 3-2 Image showing leaded glass that has been coated in metal vapor produced during the EBM	24
Figure 3-3 Schematic of the camera setup used to monitor the builds produced in the Renishaw AM250.	24
Figure 3-4 Schematic of the build described in section 3.2.1. The regions in green underwent a spot melt scan strategy in an attempt to induce equiaxed grain structures.....	26
Figure 3-5 The top images show the arrangement of the test cubes for the first set of SLS experiments (left) and the pin supports that were under the build (right and bottom).	28
Figure 3-6 Image taken of the completed cylindrical defect build.....	28
Figure 3-7 Schematic of the defects placed within the cylindrical build the array of defects consists of 20 pores that varied from 200µm to 1mm in diameter and 50µm to 250µm.....	29
Figure 4-1 Intensity-to-temperature calibration curves for Inconel 718 produced with the setup described in this research and their corresponding equation. Both curves show non-linearity with a rapid increase in the lower temperature regions. [52].....	33
Figure 4-2 Powder and as-printed material temperature calibrations for regions that underwent contour melting (top), line scan melting (middle), and point melting (bottom)	

regions. In each respective case, the melted material is significantly higher as expected. [52]	34
Figure 4-3 Representations of the two filtered temperature curves for the as-printed and powder material for a single pixel. The reduction in the peaks is evident in the preheat scanning at the beginning of the build. [52]	36
Figure 4-4 Representation of the preheat, melt, post heat regions of the temperature profiles. Of note is the location of the material transition which rapidly drops off due to the emissivity change of the material. [52]	36
Figure 4-5 The two graphs seen here show the thermal signature of a point that is reheated by a second pass (left) and the slope values calculated for the point at each frame(right). By selecting the images shown on the bottom show this location in the build for the frame in which the actual melting occurs and the incorrect frame where the transition was determined without the threshold value. [52]	38
Figure 4-6 Image showing the representative slope of a region that does not undergo melting (left) and does undergo melting (right). In the melted material there is a significant change in the material emissivity which causes the material to remain at a much lower apparent temperature than a region that does not melt. [52]	38
Figure 4-7 IR image collected from the preheat process in the EBM process. The dark regions within the part could be inadequate powder coverage or unintentional melting of the powder due to overheating.	40
Figure 4-8 Several images of the representative layer geometries given for several different layers in the build. Combined with image registration these can determine where melting is supposed to occur within a part.	40
Figure 4-9 An example of the image registration process. (Left to Right) The process begins with two images to attempt to register to each other. Then it attempts to create a transformation of one image onto the other, and will iteratively attempt to achieve the set tolerance. Finally, after reaching the correct tolerance, the images are considered fully registered to each other, and the layer geometry can be used as a logical operator.	42
Figure 4-10 Final part temperature in a build where the image registration did not accurately apply the layer geometry image to the part (regions highlighted in white).	42
Figure 4-11 Outputs for the original as-printed and powder calibrations (left) and the newly reconstructed output (right). The new thermal signature shows a temperature profile that is	

more consistent with what is expected of the rapid heating and cooling of the AM process. [52]	42
Figure 4-12 Comparison of the original IR video interpretation (left) and the newly calibrated temperature data (right). As seen in the new data the material that is melted has a much higher temperature than the surrounding unmelted powder which is expected. [52]	43
Figure 4-13 Graph showing the change in emissivity of Inconel 718 at regular temperature intervals, as the material transitions from solid to liquid the material drops by about 30% showing which would significantly alter the IR intensity that observed by the camera. Adapted from data presented in Romano et al. [69]	45
Figure 4-14 Image taken of a segment of the Kapton film from the EBM process during the first build. The dark circle in the center of the film is a region where metalized vapor would have been deposited while the film was stuck in the machine.	47
Figure 4-15 Image of the last frame within a layer which should after ample time has passed for the material to come to a uniform temperature. The two regions that seem to have higher temperature are areas that had undergone spot melting	49
Figure 4-16 Image of a surface that had undergone a point melt scan strategy (left) and IR video of another region that had also undergone the same scan strategy (bottom right). The 12x magnification surface height image taken of an individual surface spot reveals a small depression in the surface that could act as a radiation intensifier (top right).	49
Figure 5-1 Maps of the calculated solidification velocity (left) and thermal gradients (right) of a single layer within a build. The square regions that had undergone spot melting show lower thermal gradient values and high solidification velocity when compared to rest of the part. These plots show a high amount of noise and any large regions coincide with regions where issues with the temperature calibration would occur, and the small more random noise occurs due to the lack of resolution and frame rate for the imaging.	54
Figure 5-2 Map of G/R measured for the layer shown in Figure 5-1. This value gives a single representative value that can be used to determine columnar or equiaxed microstructure within the material.	54
Figure 5-3 Histogram showing the distribution of G/R values for the different scan strategies used within a layer of the build. The two histograms show a clear difference between the two with each showing a bimodal distribution.	56

Figure 5-4 Compiled G/R data for the entire monitored build. The regions in red are expected to exhibit equiaxed microstructures, and the blue is expected to exhibit columnar. The noise in the data has carried over to the data collected showing that continued refinement is needed, but the technique shows promise.	56
Figure 5-5 Thermal gradients and solidification velocity maps for the second observed build. Both cubes follow what was expected except for the regions on the closer point-to-point region where that seem to show a drift toward high thermal gradients and low solidification velocity. Also, of note is the powder bed showing through on the outer edges of the build, these appear because the calibration tool was unable to detect the melt regions accurately, and subsequently needs adjustment.	57
Figure 5-6 Image showing the thermal gradients of the closer point-to-point space cube (left) and a surface height map taken at 25x magnification of the cube following the build (right). As seen in the image, the region where the gradients deviated are located where the highest location swollen region and the slope that would be sloping away from the camera.....	57
Figure 5-7 Image showing the representative change in IR signal created by the swollen surface of the material. This swelling is the assumed cause of the change in thermal gradient seen in Figure 5-6	59
Figure 5-8 50x (top) and 1000x (bottom) micrographs of the two observed scan strategy regions. The point melt region (right) shows no discernable dendritic structure, and the line scan region (left) shows coarse dendritic spacing which is characteristic with columnar grain formation. [52]	61
Figure 5-9 Electron Backscatter Diffraction (EBSD) imaging reveals the columnar (left) and equiaxed (right) microstructures for the line melt and point melt regions. [52].....	61
Figure 5-10 EBSD images collected from the bottom of the build showing that although the thermography data collected during the build has a harder time differentiating between columnar or equiaxed microstructure in the material, the intended transition did occur.	62
Figure 5-11 EBSD data collected for the build shows the change from equiaxed to columnar material at the upper edge of the build, confirming the resultant data collected for the build. This work was completed in conjunction with the work provided in Raghavan 2017. [72]....	64
Figure 5-12 Resolution limits of a camera operating at 10Hz with an IFOV of 370 μ m like that seen in the first experiment (Chapter 4). Also in the graph is G and R data collected for the	

actual IR data and a semi-analytical heat transfer model. The data in blue was obtained using the semi-analytical heat transfer model shown in Plotkowski et al. [73].	64
Figure 6-1 Results of a curve fit for the material showing the poor fit. However, it can show the general trend of the curve.	67
Figure 6-2 Integrated intensity maps of cube surfaces over the course of a build for a single cube. The high-value regions at the lower layer numbers are where the build occurred directly over support material which has a different conductivity than the rest of the build.....	68
Figure 6-3 Maximum intensity maps of cube surfaces over the course of a build for a single cube.....	69
Figure 6-4 Decay rate, t_0 , maps of cube surfaces over the course of a build for a single cube.	70
Figure 6-5 Average integrated area as a function of layer height. The dotted lines drawn represent the area that bounds the regions where cubes exhibit full density. The uncharacteristic drops in the data are regions where there are drops in the surface values due to the video not recording the entire melt for the layer.	71
Figure 6-6 Average maximum intensity of cube surface for all of the cubes as a function of layer height. Also of note are three occasions where a cube's value does not necessarily follow the trend of the other layers. Upon review, it was seen that the video files collected for these layers did not record all of the information for these layers, leaving off the time in which substantial regions of these cubes would have been melted.....	72
Figure 6-7 Standard deviation value of the intensity of the layers within the build. Again, the regions that show uncharacteristic peaks and valleys are related to cubes that were not correctly recorded in the video.	73
Figure 6-8 Average maximum intensity value for various layers. The additional images near the peaks and valleys of the data show that representative layer's scan pattern and the regions that seem to reach much higher values.	74
Figure 6-9 Comparison of the average integrated intensity for all cubes produced under both powder bed conductivity conditions. The second higher conductivity build shows a lower average integrated intensity for the material that has a slight decline as the build continues compared to the first build which remains relatively stable throughout the build.	76
Figure 6-10 Comparisons of the integrated intensity (left) and decay rate t_0 (right) for all cubes within the build.....	77

Figure 6-11 Image showing the relative intensity signals for the material in a cube. Note that the signal seen for the longer scan region rapidly decays after two initial passes the material rapidly decays and for the other material the rapidly passing beam maintains intensity for much longer.....	78
Figure 6-12 Images of layers within the cubes where regions of increase integrated intensity of the IR signal are shown. These regions could be locations of voids within the material or a location where spatter landed on the surface, however, further investigation of the cause is needed.....	78
Figure 6-13 Series of images depicting the cubes ranked from least dense (top left) to densest (bottom right). As expected the best parameter sets were among the densest with the extremes of the variations ranking among the lowest.....	80
Figure 6-14 Comparison between the average integrated area for all of the cubes and their relative densities.	81
Figure 6-15 Images of cubes from the second build where the porosity shows a gradient in the material from some porosity to more porous (left) and porous to mostly solid (right). It is believed that this is due to the changes in the powder bed conductivity.....	82
Figure 6-16 Cube porosity (right) compared to the average integrated intensity per layer(left). The graph of the average integrated intensity has been rotated so and scaled so that the data seen in the image coincides with the image. Note that the layer in which the material's average integrated intensity drops below a particular value, the material begins to exhibit higher density.	82
Figure 6-17 IR intensity signal for both the region over a 1mm void and over the solid material.	84
Figure 6-18 Comparison of the first pass of the beam for the material over a 1mm void and the solid material. Note the drastically different decay rate of the two materials.	84
Figure 6-19 IR image taken from the build process during the melt, note each of the voids within the layer are brighter than the surrounding material due to the difference in emissivity.	86
Figure 6-20 Comparison of the area under the curve for material located over a 1mm diameter defect and the solid part. Note the drastically different decay rates of each beam pass as well as the longer overall decay time of the material that is deposited over a void.....	86

Figure 6-21 Images of the integrated area values of the material's surface for a ten-layer defect set. Note how in the case show the defects can be seen both in-layer and underneath the surface of the material when the laser passes over the material.....	87
Figure 6-22 Images of the threshold images that are used to identify potential defect locations within the material.....	88
Figure 6-23 3D representation of the observed build with potential defect locations highlighted in red. The edges show potential defects due to the increase of beam scanning frequency.....	89
Figure 6-24 Flowchart describing the data analysis used for locating pores within the material.	90
Figure 6-25 Series of graphs showing the relationship between the height and diameter of the defects in the material and the integrated intensity values collected while the material was within the layer (negative depth below the surface) or below the surface.	92
Figure 6-26 X-Ray CT image of the defects present in the part. Note the lack of the defects that are small ($<400\mu\text{m}$ in diameter and $<150\mu\text{m}$ in height) due to apparent remelting.....	93
Figure 6-27 Image showing the increased integrated area for the leading and trailing regions of the surface's scan strategy.	95
Figure 6-28 Representative intensity curves(left) for the material at the beginning, end, and middle of the parts melt, and the frequency of the same regions (right). Note the offset bias of the end melt.....	95
Figure 6-29 Surface maps for the integrated area of a cube for all layers (left) shows a dramatic decrease in the measurement for layer 195. Upon reviewing the IR video for the layer, it was discovered that the video ended early and the cube had not been melted in the video yet....	97
Figure 7-1 Conceptual examples of the error in cooling rate calculations brought on by missing the peak intensity of the curve by the equivalent of one frame (right) or by underestimating the slope of the curve due to time resolution (left).	101
Figure 7-2 Image of a spot scan strategy taken in a build far after the end of melting for the current layer. The high-intensity spots were analyzed at 12x magnification using high-resolution confocal microscopy and show the surface topology for these regions.....	104

List of Attachments

Attachment 1: CorrectedTEMP.mp4: Video file that shows IR data collected both before the correction for emissivity is applied and the corrected temperature profile after undergoing the correction process.

Attachment 2: Bracket.mp4: Video file showing a three-dimensional representation of a parts microstructure monitored using IR data analysis.

Attachment 3: EngineeredDefectsbuild.mp4: Video file indicating the presence of defects within a build monitored in-situ.

Chapter One: Introduction

Metal additive manufacturing (AM) is the fabrication of computer-aided design geometry through the layer-wise melting or bonding of metal powders, wires, or sheets. This innovative process seeks to allow for highly sophisticated designs that can remove some of the limitations posed by traditional manufacturing methods [1]. Meanwhile, AM processes can reduce waste, decrease lead time or time to market, and lower the energy needed for the production of a part when compared to conventional methods. There are several different methods in which to bond these materials including sheet lamination, binder jetting, directed energy deposition (DED), or powder bed fusion such as selective laser sintering or electron beam melting [2]. Each of these methods has their advantages and disadvantages in regards to creating parts at the near-net shape, that is, obtaining a geometry that needs only light secondary processing to achieve their final state. Although these methods show many benefits over traditional manufacturing methods for metal parts and assemblies, they still must overcome numerous challenges to increase industry acceptance and confidence. These challenges include: developing robust feedback control systems, increasing print accuracy, improving surface finish, developing methods to validate and qualify part performance, and improving build reliability [1]. Currently, most methods for qualification require destructive testing techniques such as metallographic imaging, fatigue testing, or tensile testing that require the waste of a build for analysis, and there are few techniques presented for in-situ qualification and measurement that could face these obstacles. High-end process monitoring and measurement techniques could allow for a method to overcome these issues without the need for destructive testing. Attempts to develop process monitoring for the AM process such as high-speed optical imaging, beam-line imaging using photodiodes and pyrometry, and infrared thermography using thermal cameras exist, each with their advantages and disadvantages. However, the amount of research in the field is limited when compared to other manufacturing processes. Due to its ability to determine temperatures and larger imaging area than some of the other methods infrared thermography is one of the more promising techniques presented.

Infrared (IR) thermography is a non-contact method for determining the temperature of a material by converting the radiation that is given off by the surface of a material as it is heated up into an electrical signal through the use of specialized sensors that convert infrared

radiation from a material into electrical signals[3]. This technology could be valuable for measuring and determining many different phenomena within an AM build and quantify the presence of defects or consistency of part microstructure, and with the proper amount of research, utilized as a feedback control system. However, there is limited research using the technology for AM process with few fully developed applications and the need for correcting material emissivity change and more robust analysis techniques for detecting relevant qualification metrics is evident.

The primary objective of this research is to advance process monitoring capabilities for AM through the use of IR thermography and develop useful data analysis tools that can address monitoring needs for the process. The methods shown will address in-situ calibration for change in material emissivity, microstructural development, and defect detection in AM materials. With the use of these techniques, the hope is to increase reliability and throughput of the AM process and increase its feasibility for more industrial applications. The work presented in Chapter Two will discuss the relevant background and related literature that will give context to the methods and techniques used and describe the state of the art for this research. Then a review of the materials and methods used to produce the relevant results will be discussed in Chapter Three to allow for repeatability. Chapter Four presents the algorithmic approach and results of a method for navigating emissivity change during the EBM process to produce a more accurate temperature curve. Chapter Five discusses the use of thermographic data to help monitor the development of columnar and equiaxed microstructure in electron beam produced Inconel 718. Then Chapter Six will present a discussion of the methods and results used to track bulk porosity development and intentional defects within the selective laser sintering process. Finally, conclusions and recommendations for future work will be given in Chapter seven to provide direction for research to advance the technology.

Chapter Two: Literature Review and Background

This chapter will review the previous work that has been presented in the literature and describes the relevant information needed to understand the body of the studies provided in this thesis. Section 2.1 will discuss powder bed fusion (PBF) methods of additive manufacturing processes. An explanation infrared (IR) thermography basics are reviewed in Section 2.2 with a focus on emissivity and how it affects IR data in additive manufacturing. Section 2.3 will discuss the current state of the art for process monitoring techniques in the additive process. In Section 2.4 a brief review of the microstructural development of Inconel 718 and the columnar to equiaxed transition in additive materials will be discussed. Finally, the effects of defects such as porosity within the material on mechanical properties will be discussed in Section 2.5 before a brief discussion of the goals of the current research is given in Section 2.6. With a firm understanding of the context for this research obtained the final section of this chapter will discuss the goals of the current research and the proposed approach to accomplishing them.

2.1 The powder bed fusion additive manufacturing process.

Although there are many AM processes, this research will discuss only both the electron beam melting and selective laser sintering processes which both fall within the category of powder bed fusion (PBF). This methodology utilizes the repeated melting of thin layers of powdered metals to a specified geometry given by a computer-aided design (CAD) to create a part at near-net-shape. Both processes involve the same basic steps for a layer of the build with a few small changes [4]:

1. The machine applies a thin layer of powder across the current substrate by a powder distribution mechanism.
2. After the application of the powder, the melting process begins:
 - For selective laser sintering (SLS): The geometry of the layer is melted by a laser beam into the specified shape consolidating the distributed powder.
 - For electron beam melting: The process is more involved, utilizing a preheat scan of the material with a highly diffused electron beam to sinter the powder together lightly. Then fully consolidating the material with a focused beam to the geometry of the current layer, and then heating the material again after the

layer has been melted to attempt to maintain a relatively uniform temperature of the material in the powder bed.

3. After the melting of the layer, the process is repeated for every layer of the CAD geometry to form a near-net shape part. The remaining powder is removed from the powder bed, and the part surfaces and then recycled for use during the next build.

The main differences between these two processes are the change in the energy source used and a change in chamber atmosphere, with the EBM process taking place at near-vacuum and SLS in an inert gas. Each process has its advantages and disadvantages (**Table A- 1**), but both processes can achieve highly complex geometries that cannot be traditionally fabricated using machining or forging. However, one of the critical challenges for these new technologies is their utter complexity and the need to increase reliability and improve as-built part quality to reduce any subsequent processing of the material.

2.2 Principles of infrared (IR) thermography and emissivity

The research in this thesis focuses solely on the use of IR thermography as a means for monitoring powder bed fusion of additively manufactured metals. As such, this section will discuss the basic principles of IR thermography and the effects of emissivity, or the efficiency of a materials ability to give off radiation, as they apply to AM. It will first give a brief overview of thermographic technology as presented for thermal camera systems and then explain how emissivity changes within the process.

2.2.1 Infrared thermographic technology

As objects heat up to a finite temperature, the material will begin to emit energy in the form of electromagnetic waves [5]. The Stefan-Boltzmann law governs the amount of this radiation, which states that for a real surface the material will produce an amount of energy represented by Equation 2.1.

$$E = \varepsilon \sigma T_s^4 \quad 2.1$$

In the equation, σ refers to the Stefan-Boltzmann constant ($5.67 \times 10^{-8} \text{ W/m}^2\cdot\text{K}^4$), T_s denotes to the surface temperature of the material, and, most important for real surfaces, ε is the emissivity which will be described further in Section 2.1.2. Infrared thermography utilizes these principles of material radiation to determine the temperature of the material observed. The way the technology can detect this change in the temperature is through the use of

specialized camera sensors that collect the incident radiation produced by a material and then convert it into an electrical charge [3]. Circuitry within the camera then filters and amplifies this signal so that it can calculate the temperature based on the given emissivity value of the material [3]. This measurement is governed by Equation 2.2 which states the spectral radiance of the material at a given wavelength and temperature, and the amount of this radiance collected by the camera sensor respectively[3].

$$L_{\lambda}(\lambda, T) = \varepsilon \frac{2hc^2}{\lambda \left(e^{\frac{hc}{\lambda \sigma T_s}} - 1 \right)} \quad 2.2$$

In Equation 2.2, T_s , σ , and ε are the surface temperature (K), Stefan-Boltzmann constant ($5.67 \times 10^{-8} \text{ W/m}^2 \cdot \text{K}^4$), and emissivity as previously described, h denotes Planck's constant ($6.626076 \times 10^{-34} \text{ J} \cdot \text{s}$), c is the speed of light ($1.381 \times 10^8 \text{ m} \cdot \text{s}^{-1}$) [3]. The array of sensors within the camera gathers this radiation over a specified unit of time, referred to in this work as the integration time, and converts it into a small voltage signal which is amplified and filtered before being sent to a computer for analysis. The software used to interpret the data then arranges the individual sensor readings into an array of values that represent the pixels in the resultant image. The software can finally extract a temperature with proper calibration. Thermal imaging technology has been employed in numerous fields for monitoring and qualification of various conditions or processes such as monitoring infill during casting processes, monitoring welding temperature and penetration depth in a material, as well as the construction, defense, arts and medical fields [3], [6], [7].

Thermal camera systems work just like any typical camera system by using optical components such as lenses to direct this radiation toward the sensors much like a traditional camera will direct visible light toward its film, and the resolution of the data collected is significantly affected by the size of the lens used. The resolution of the data is crucial in determining the effectiveness of the data collected both influencing the speed of the data collection (the frame rate (Hz)) and the smallest feature distinguished by the detector (instantaneous field of view or IFOV). These measures of data resolution work in conjunction with the Nyquist-Shannon criteria for spatial or temporal resolution, which states that to accurately measure or quantify any phenomenon it needs to be at least twice the value of the smallest resolvable features in time or space[8]. For instance, to detect and quantify any

phenomena that are 1µm in size and occur within 1ms, the spatial and temporal resolution will need to be .5µm and .5ms respectively. All of these factors can significantly influence the results of the data collected through IR thermography, but in this research, the emissivity of the material is the most crucial factor to understand.

2.2.2 Emissivity and its effects in the monitoring of AM processes

Emissivity is the measure of the amount of radiation that a surface produces at a given temperature to the amount of energy generated by a blackbody surface, which emits the theoretical maximum amount of energy that is possible for a given surface[3]. The literature shows that the emissivity of a given material is directly related to several different factors including the composition of the material, the surface roughness, corrosion and oxidation of the surface, and the angle between the camera and the object [3], [9]–[13]. In additive manufacturing, it has been documented by the literature that emissivity poses a problem in the determination of the temperature of the material because the emissivity of the material changes as the process is performed due to the transition from highly emissive powder to less emissive solid material [14].

In the powdered state, the material will exhibit high emissivity, near blackbody for some materials, when compared to that of the as-printed material as shown by Dinwiddie et al. [15]. Sih et al. related the high emittance of the material to the presence of numerous small cavities within the powder bed as a result of the particle packing density[16]. In this work, the authors state that when a cavity forms within a material, the emissivity of that void can be described using a simple function, seen in Equation 2.3.

$$\varepsilon_h = \frac{\varepsilon_s}{\varepsilon_s + f(1 - \varepsilon_s)} \quad 2.3$$

This equation states, the emissivity of a cavity or hole, ε_h , is directly related to the emissivity of the solid ε_s and the ratio of the cavities opening to the total surface area of the cavity, f . The factor, f , is the most important, with smaller values of f causing ε_h to approach the emissivity of a blackbody. The authors then chose a value that represented the characteristic cavity morphology for the powder bed and applied it to Equation 2.3 and using the new value of ε_h in Equation 2.4 to approximate the emissivity for a given powder bed.

$$\varepsilon_p = \varepsilon_h A_h + (1 - A_h) \varepsilon_s \quad 2.4$$

In the equation, the authors show that with an increase in the area of a given surface covered in these characteristic voids, A_h , will increase the overall emissivity of the surface ϵ_p until the entire surface is at the emissivity of the cavity emissivity which is not possible. When the part undergoes melting the material will begin to consolidate, and the value of A_h will decrease to near zero, and the emissivity will drop to that of solid material, which is much lower than the near blackbody emissivity of the powder. The change in emissivity makes the use of IR thermography for the measurement of temperature, and therefore temperature affected phenomena, complicated as it requires extra processing and calibration. As of right now, there has been no proposed method for mitigating this change which affects the usefulness of IR data as a means of monitoring PBF additive processes.

2.3 Current state of in-situ process monitoring of powder bed fusion (PBF) processes

Research on the topic of in-situ process monitoring of PBF additive manufacturing processes is limited in scope within the current research landscape. However, lately, the topic has gained increased interest in researchers attempting to understand methods to detect microstructure development, defects, geometric conformity and other phenomena that occur within the build for both the SLS and EBM processes with some success [17]–[20]. This section will discuss the current landscape of in-situ monitoring efforts for each of these processes with emphasis given to thermographic technologies.

2.3.1 In-situ process monitoring of selective laser sintering (SLS)

Of the two powder bed fusion processes described, in-situ process monitoring in laser powder bed systems is more readily available with multiple research efforts utilizing in-situ monitoring due to fewer limitations from machine interactions [17]. Initial ventures, into process monitoring the SLS process mainly focused on the use of high-speed optical or near infrared (NIR) cameras in conjunction with photodiodes to collect data about the melt pool as it traversed the surface of a part. The technique involves the use of sophisticated mirror systems that move in synchronous with the mechanical galvanometer directing the laser allowing for in-line viewing of the melt pool. Various experiments used this method within the literature and were able to obtain highly useful information about the process [21]–[25]. Kruth et al. performed one of the earliest attempts to utilize this approach for monitoring the SLS process in 2007 [21]. In the work, the authors were able to monitor changing melt pool

conditions that occurred as the laser's scan speed was changed, noting splitting of the weld pool at very high scan speeds. When this splitting occurs regions that occur in overhang regions tend to sag causing an error in the geometry of the part, and thus the authors were able to create a rudimentary control system that would utilize the photodiode signal as the control input. By setting a specific threshold value of the diode signal and then proportionally lowering the laser power as the signal increases over this value they were able to reduce the sagging within a test sample dramatically producing a much better part. Berumen et al. were able to use this methodology to determine a correlation between voids generated during the process to changes in the photodiode values and the shape of the melt pool that represents drifting from the correct processing conditions of the material [22]. Expanding upon that research, Craeghs et al. monitored the process to gain an understanding of how overhangs, adjacent scans, and corners affect the melt pool and change the processing conditions of the material [23]. This work showed that these geometrical factors influence the state of the melt pool either by changing the size and shape, causing overheating in sharp corners and small hatch distances. The authors were also able to detect separation of the melt pool to multiple parts in the overhang regions which would ruin the resulting surface finish just as Kruth had observed. The authors then continued this research, developing methods to process and map this data onto an x-y representation of the layer, giving a better understanding of the entire part surface [24]. Clijsters et al. utilized the same principles to map regions of porosity that were very comparable to collected computed tomography (CT) scans of the material [26]. Along with the inline camera and photodiode approach, offline high-speed optical cameras were used to determine useful information on the SLS process as well. Furumoto et al. used this method to monitor the consolidation of metal powder during the melting process and correlate the consolidation effectiveness to the powder layer thickness[27]. All of these techniques show promise for the ability to monitor and detect processing flaws and eventually control the process in real time, but the small field of view and limited time frame of observation dramatically limits the obtained information and does not allow for capture of other information such as temperature.

Another technology explored for the monitoring of the SLS process is pyrometry, a thermography technique that utilizes incident radiation to determine temperature based on the emissivity of the material (Section 2.2). SLS processes employ this technology, similar to the photodiode setup discussed earlier where the pyrometer views down the laser optic's path

through the use of a mirror system. One of the earliest attempts to utilize a pyrometer for SLS was the work performed by Bayle and Doubenskaia where the authors made an effort to resolve the effects of process parameters (namely powder layer thickness and hatch distance) on the brightness temperature measurements collected by an inline pyrometer [28]. The authors determined that the perceived temperature measurements of the material would increase as the powder layer thickness increased as well as the hatch spacing increased. Similar work was completed by Pavlov et al., where the authors monitored variation to the hatch distance at various powder layer thicknesses, and different scan strategies to collect relevant information about the material; however, they only used the signal rather than temperature calculations [29]. Chivel et al. were able to utilize actual temperature measurements of the additive manufacturing process utilizing a combination of a high-speed two wavelength pyrometer, creating a pathway for temperature measurements to be taken in-situ [30]. Islam et al. were able to utilize actual temperature measurements through the use of a pyrometer as well and observing a larger area of coverage compared to previous work obtaining temperature measurements over an area that is 15mm in diameter [31]. In the work, the authors were able to use this temperature data to understand the appearance of balling, where the material melts but does not adequately flow over the surface, during the SLS process and determine optimum melt conditions to prevent this from occurring during the build process. Despite these advances in the ability to detect various phenomena during the SLS process using pyrometry, there have been no efforts for automatic detection or feedback control of the process. Also, as previously discussed for photodiode sensing, collected data only encompasses the area directly surrounding the current location of the melt pool for a very brief time frame rendering some temporal analysis techniques impossible.

To analyze the entire surface of the material during the build process, the use of IR thermography could provide a means of analysis, due to its ability to detect temperature or thermal data across a large area. There is limited use of the technology in the literature, but it could allow for enhanced knowledge of the process. Bayle and Doubenskaia performed one of the first uses of IR thermography in the literature in an attempt to understand what kind of information is obtainable from the collected data [28]. In the experiment, the authors mounted a camera within an SLS system but only observed a small region of about 13mm x 6mm using a camera setup that allowed for 2031-3556Hz sampling rates. The authors also utilized a specialized neutral density filter, a specialized material that attenuates radiation across all

wavelengths within a given range, allowing for the camera to detect much higher intensity values than it usually would be capable of achieving [28], [32]. The authors stated that by using IR thermography, they were able to observe phenomena that occurred during the build, specifically, transience within the melt pool as a result of changing thermal conditions and information about the development and movement of melt pool ejecta. The authors were able to measure the speed and size of the ejecta particles and determined that typically the ejecta will move in the direction of the highest thermal gradient of the material with few cases moving against the thermal gradient. As for the transients within the melt pool, the authors were able to observe that as the beam moves along the surface, the heat affected zone will increase creating a “tail” of high-temperature material that follows behind the beam. The authors also note that there is a need for a calibration method to gain the correct temperature information however at the time it was practically impossible to do so because they could not achieve accurate enough calibration techniques. Krauss et al. also utilized an IR camera for thermographic data collection, in this case, a microbolometer which has a much slower integration time than other IR detectors such as the one used in Bayle and Doubenskaia, with an integration time of about 8ms [33]. In this work, the authors externally mounted the microbolometer camera to an SLS chamber peering through a small observation window to view a build with a small void purposely inserted within the material. Due to the large viewing angle that this camera setup has with the observed built, the imaging could only correctly view a small section of the build in focus as a result of the camera’s limited depth of field. Despite these limitations, the authors were able to discern the size and circularity of the heat affected zone as a result of varying parameters (scan speed, hatch spacing, laser power, and scan lengths), and also the sensitivity of measurements to artificial flaws. The results collected showed that the heat affected zone would change size with variances in build parameters as expected, with increases in scan length and laser power leading to an increase in the area of the heat affected zone, and increased scan speed and hatch spacing causing a drop in the size of heat affected zone. As for the artificial flaw sensitivity, the author was able to discern that subsurface flaws could be detected by a thermal camera to some degree with larger pore sizes ($\geq 200\mu\text{m}$ diameter and $\geq 100\mu\text{m}$ height). When scanning above these flaws, the maximum IR intensity in the location would increase about 100 or 200 units higher than a reference signal, and they would take longer to decay, but these difference where very small and could be hard to differentiate from noise within the data for other techniques, and the integration time of the

camera was very long at about 8ms. These results show promise for the use of thermal cameras in SLS processes. However, as previously stated, the integration time and lack of full layer monitoring, severely limits the effectiveness of such a technique as a robust monitoring tool.

2.3.2 Monitoring of the EBM process

While there have been attempts to monitor the SLS process using pyrometry, optical cameras, and photodiode detectors in line with the laser system, no such methods can be utilized with the EBM process. Because the process requires large electromagnetic coils to direct the electron beam, there is inherent interference with the electronics of these sensors as well as a lack of the mechanical systems used for beam direction which allowed for these sensors to follow the melt pool [17]. Due to these limitations, the EBM process can only be observed by externally mounted detectors with a large enough viewing area to monitor the build surface and quick enough response times to resolve beam movement to some degree; leaving high-speed optical, NIR, and IR cameras as the only viable option. Due to the limitation of sensor applications in the EBM process, there is limited literature involving in-situ monitoring. However, the few applications of in-situ monitoring for EBM show great promise for the use of process monitoring techniques to help understand more about the process as well as being used as a feedback control tool [34]–[38].

One of the first monitoring methods of the EBM process was performed by Scharowsky et al. who utilized high-speed optical imaging to monitor the melting of Ti-6Al-4V powder to confirm numerical simulations that they had performed[39]. In the work, the authors were able to determine just how long the melt pool would remain liquid, about 10ms. Another one of the earliest successes for the use of monitoring within the EBM process was performed by Rodriguez et al., who were able to use an infrared camera within a feedback control system[14]. The work showed that the authors were able to calibrate an IR camera through the utilization of a calibration target that comprised of a large cube of Ti-6Al-4V that contained a spherical cavity in the center and a small hole on the top surface that when viewed by the camera acted as a blackbody cavity. By placing a thermocouple within this spherical cavity and reading the temperature, the authors were then able to utilize the measurements taken to approximate the emissivity of the material and allow for a calibration to determine the relative temperature of the material. By using the calibrated relative temperature of the build, the authors were able to create a rudimentary control system that could adjust the power and scan speed of the beam

based on measured thermographic values on a layer-by-layer basis and succeeded in maintaining more consistent temperature throughout the build. The experimental control system could reduce the change in temperature across multiple parts within the build area by adjusting the scan parameters based on a single image taken at the end of each layer. Although the results seem promising, the authors mention that the temperature values are not representative of the real temperatures of the material, stating that the newly melted material has a lower apparent temperature than the powder which is not representative of the newly melted solid. They mention that a method to correct for this error and calibrate for the material change in real time is needed, but has not been created.

Also, other researchers began to develop monitoring tools for the process that could allow for in-situ qualification. Ridwan et al. were able to use an IR camera to determine the geometric accuracy of parts produced using EBM [34]. The authors determined that the parts geometric accuracy varied in accuracy from different locations in the build, showing that the parts measured differed by about 5-17% from their designed values. The works of both Schwerdtfeger et al. and Dinwiddie et al. were able to detect surface voids within the material by finding regions of increased IR intensity which is indicative of unmelted powder or surface porosity [38], [40]. Both works mention that needs for performing automatic detection is necessary. Dinwiddie et al. also noted that a need for proper calibration is required to fully utilize IR thermography as a means of process monitoring and control and developed a method to prevent metal vapor from harming the accuracy of the build using a scrolling layer of Kapton film [40]. Along with these defect detection techniques, the work of Mireles et al. was also able to find in layer vacancies. After detection, the authors were able to successfully correct these defects in-situ by rescanning over the defects and melting the material with a second pass within the same layer [36]. Following this work, Mireles et al. were also able to use the IR signal intensity within the material as a means to detect the coarsening of grains within printed Ti-6Al-4V [35]. The authors were able to use this information to create a control system that could change the parameters of the melting process through simulated mouse clicks. However, this does not make for a very robust control system as the changes do not take effect instantaneously, but rather in the following layer.

As mentioned throughout the literature, one of the most pressing issues in the use of thermography for EBM processes is the need for proper calibration techniques for the

temperature of both the powder and solid material. As previously mentioned, Rodriguez et al. were able to achieve a relative calibration of the temperature of the material through the use of a calibration target that utilized an artificial blackbody [14]. Dinwiddie et al. used a similar methodology that included a method for gaining a calibration for both the solid material and powder through the use of another calibration target design [15]. Rodriguez et al. also went on to approximate the emissivity and surface temperature of both the powder and as-printed material as well [41]. To achieve this goal, the authors calculated the transmission of the optics, the incident radiation between the walls of the build chamber and the build plate, the absorption and generation of IR radiation from the surrounding atmosphere, and so forth. By taking all of these factors into consideration, they then adjusted the camera settings to account for all of these effects and verified that the collected signal matched the expected temperature through the use of a thermocouple placed within the material with accurate results ($<1\%$). All of these techniques show relative success in determining a calibration for the temperatures of the solid material or the solid material and the unmelted powder. However, there is no reliable methodology to apply these calibrations in-situ as the material transitions from one state to another. In order to develop a robust monitoring methodology using thermography, a system would have to be aware of the relative surface topology, material state, and emissivity simultaneously at any given time in order to achieve near perfect temperature accuracy as well as high-speed, high-resolution data to ensure that no data is left out of the analysis.

2.4 Overview of microstructural development within AM Inconel 718 and the effects on part performance.

For the results and discussion in Chapter 5 of this research, an understanding of the microstructural texture of Inconel 718 as it pertains to AM materials is needed. In this section, a brief explanation of this microstructural development, specifically the columnar-to-equiaxed transition of the material as produced in the EBM process, is given as well as its effects on the mechanical properties of the part. Also discussed are current research efforts to achieve desired microstructural outcomes in the AM process.

2.4.1 Formation of columnar and equiaxed grains and in-situ control of microstructure

The literature states that solidification parameters of the melting processes, namely the solidification velocity and thermal gradient of the material, impact the formation of

columnar and equiaxed grains in AM material [42], [43]. The understanding demonstrated in work for the welding processes transitions well to the solidification parameters of the metal AM process, due to its similarity. Typically, as the beam moves across the surface of the part, epitaxial grain growth is favored in the fast growth direction ($\langle 0\ 0\ 1 \rangle$) which is in the direction of the maximum thermal gradient [44]. However, when there are decreased thermal gradients in the material, undercooling due to changing composition of a solute-rich region of the liquid allows for spontaneous nucleation to occur ahead of the solidification front [38]. When this occurs, dendrite growth will take place radially around these new nucleation sites and can potentially disrupt the growth of columnar grains, and since these newly nucleated materials do not form in the same direction each comprises a small grain that has a much different orientation than the surrounding grains. With a basic understanding of these solidification effects, researchers have been able to develop numerical models that allow for better prediction of material microstructures based on the solidification velocity and thermal gradients within the material [45], [46]. Gaumann et al. were one of the first to determine phenomenological equations that utilize the understanding of material properties, undercooling, and processing parameters in the laser melting process to understand the formation of columnar and equiaxed grades and develop processing parameters to form these grains [45]. Following this research, Raghavan et al. developed simulated melts in the material to determine further how beam diameter, current, on time, and preheat temperatures affect the formation of equiaxed grains [46]. The authors were able to distill the equations provided by Gaumann et al. into simple calculations that could measure the sensitivity of the formation of equiaxed grains to these process parameters. Both showed that with proper modeling of the process, the microstructure could be predicted for a given material.

With an understanding of the effects of these solidification parameters on the resultant microstructural development, researchers have been successfully able to control the microstructure in-situ. Korner et al. were able to influence microstructural change within a build, as a result of the changes made to the power, beam speed, and distance between subsequent raster lines [47]. The authors noted that, when using slow scanning speeds and greater distances between subsequent melt lines, the material followed the characteristic columnar grain structure that is typically produced by AM processes. However, with increased the speed of the melt pool and decreased the offset distance between the line passes they achieved a relatively equiaxed grain structure. Dehoff et al. were able also able to create

location-specific microstructures through the use of various processing parameters. However, instead of changing only the beam speed and the spacing between line scans in a typical raster pattern, the authors changed the scan strategy itself [48], [49]. The authors determined that instead of using a typical line scan that moves across the surface, that utilization of a point scan strategy where the beam creates a series of melt pools in a grid-like formation they could achieve the correct processing conditions to produce variation in the grain texture. **Figure 2-1** shows a schematic of both of the scan strategies. By utilizing this point melt scan strategy, the thermal gradients within the material become much lower than those of the typical line scan strategy which will allow for the proper undercooling to take effect, letting nucleation sites occur ahead of the solidification front. The authors were able to use versions of both of these scan strategies within the same part and were able to create an image that had an equiaxed grain structure outlining a columnar microstructure within the material (**Figure 2-2**). Kirka et al. were able to create similar results on a bulk scale by modifying the point scan strategy and building tensile test specimens to understand the performance capabilities of this microstructure in AM materials [50]. All of these works show that through careful understanding of the processing parameters and the involved solidification parameters of the material, control over the microstructure of Inconel 718 is possible within a part.

2.4.2 Effects of grain structure on part performance

The grain structure of a material plays a significant role in the mechanical properties of the material. The literature states that materials produced with columnar grain structures have much longer high-temperature creep life when compared to the materials produced with equiaxed grain structures and that the materials display anisotropic and isotropic elastic and plastic properties respectively [51]. Also, increased creep life has been shown in directionally solidified materials with columnar structures compared to those with equiaxed grain structures at high temperatures [51]. As for the anisotropy and isotropy of strength in the two microstructures, Kirka et al. were able to show that this is the case in their work on the mechanical properties of EBM fabricated parts [50]. As previously mentioned, the authors produced columnar and equiaxed grain structures in mechanical test specimens that then underwent tensile testing. In the results of this testing, the authors were able to show the anisotropy and isotropy of these different material microstructures. The equiaxed material exhibited equal strength and ductility in both the longitudinal and transverse directions of the build for both high and low-temperature applications (**Figure 2-3**). In contrast, the columnar

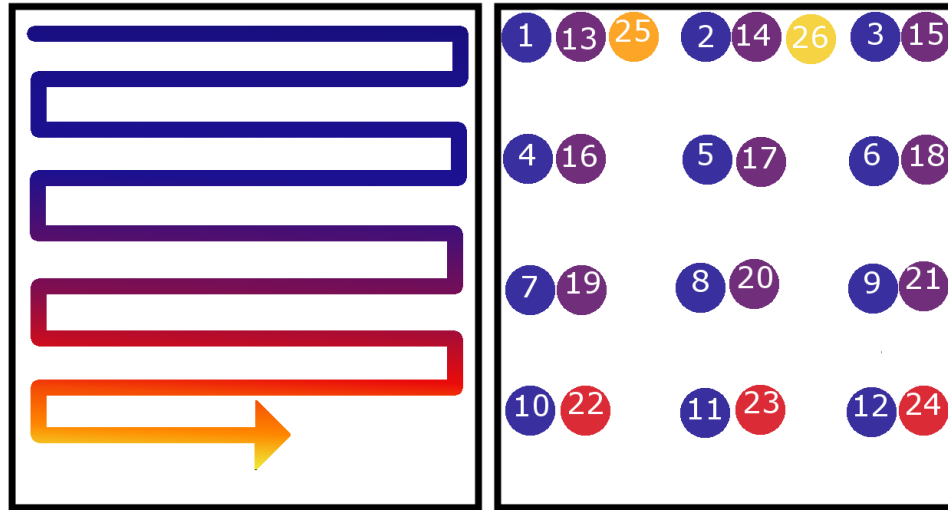


Figure 2-1 Schematic representations of a line melt (left) and point melt (right) scan strategies.[52]

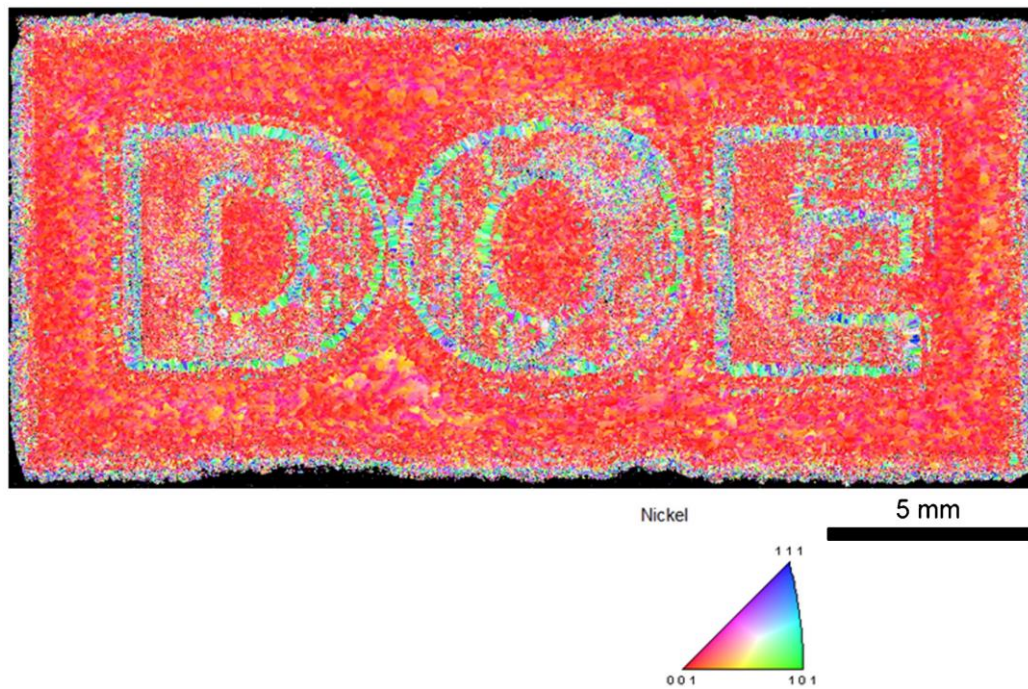


Figure 2-2 Image showing microstructural control of Inconel 718 from Dehoff et al.[49] By modifying the localized scan strategies the authors were able to create an image within the material from the resultant microstructure.

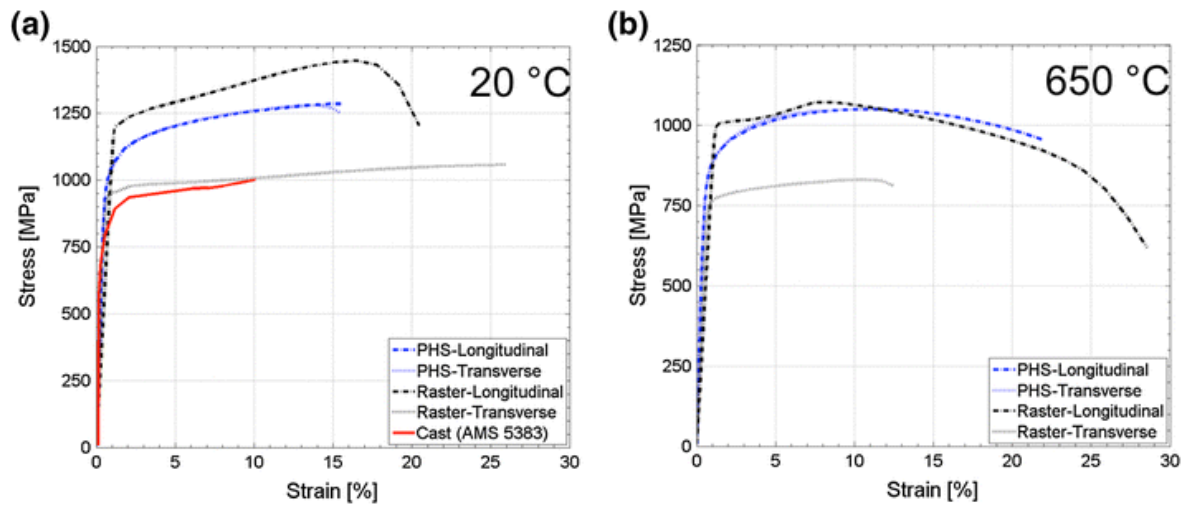


Figure 2-3 Comparison of equiaxed (PHS) and columnar (Raster) material properties at (a) 20°C and (b) 650°C showing the relative isotropy and anisotropy of the materials respectively from Kirka et al. [50]

microstructures produced exhibited much higher longitudinal strength and ductility when compared to the transverse, with an increase of about 30% in the yield and ultimate tensile stress (**Figure 2-3**).

The effects of the microstructure on the fatigue life and strength of the material can prove to be crucial for high-performance applications. The ability to obtain location-specific control the formation of these microstructures in-situ gives AM processes a distinct advantage over traditional processing techniques. However, at present, the ability to produce these microstructures requires a priori knowledge and careful planning of the processing conditions of the material. As research determines more robust methods for in-situ microstructure determination, proper control systems can be created to control the microstructural consistency of the material.

2.5 Porosity formation and effects on strength and fatigue life of AM materials

In any material, the presence of voids can present severe effects for the strength and fatigue life. These detrimental effects to the mechanical properties of a material occur due to stress concentrations that surround the void in the material. When a discontinuity is present within the material, the stress in the material adjacent to the void is much higher than the average stress of the material [53]. This increase in material stress allows for crack propagation in the material stemming from this void in the material, therefore, reducing the cross-sectional area and further increasing the stress in the material. Eventually, this leads to complete part failure due to a through thickness fracture much sooner than for a sufficiently dense material.

Several works within the literature list the formation of porosity as one of the causes for early part failure in AM materials and state that methods are needed to prevent their formation to increase reliability [54]–[59]. In this research, two different types of process induced porosity seem to be most prevalent: gas porosity or keyhole porosity, and lack of fusion within the material. Gas porosity can occur as a result of capturing the trapped gas within the powder by the rapidly solidifying material, and cannot be readily prevented in-situ [59]. Processing parameters and energy deposition within the material can cause increases in the lack of fusion or keyhole porosity within the part. When too little energy is deposited into the material, adequate melting may not take place to make leaving large irregularly shaped

pores that can contain powder particles (**Figure 2-4**) [58]. Traditionally seen in welding, keyhole porosity forms when the energy source penetrates deep into the material due to high power causing instability within the melt pool between the surface tension of the surrounding material and the recoil pressure at the center [60]–[62]. When this occurs, the surrounding material could potentially collapse in on itself, and any remaining gas is trapped below this collapsed material as it solidifies, leaving small spherical or slightly elliptical voids within the material (**Figure 2-5**). In additive manufacturing, keyhole porosity can be formed and may be located along the beam path of the material as shown by King et al. in their research [61].

The formation of porosity poses a fundamental challenge for the AM process. As shown in Section 2.3, this had led to an increase in efforts to attempt to locate and correct for these pores in-situ however very few methods for doing so exist. Research needs to be expanded on this subject, and greater understanding of its causes and mitigation is required to address this concern and improve the AM process.

2.6 Objectives of Current Research

As shown in the literature review, methods of in-situ qualification and detection of detrimental factors within the build need to be developed to increase the acceptance of additive manufacturing within industry. The primary purpose of this research is to identify and develop thermographic data analysis tools that can be used to monitor PBF additive manufacturing processes that can achieve these goals. Also, this research seeks to increase confidence in thermographic monitoring technology as a means of process monitoring and qualification. This research will attempt to do so, first, by focusing on a method for correcting for the emissivity change of the material as it melts during the build process to increase the accuracy of thermographic data. Then, showing a method to calculate the solidification parameters to determine the formation of columnar or equiaxed microstructures. Finally, this work hopes to identify a way to detect both bulk porosity and site-specific porosity within an AM process through the use of thermographic data analysis. All of these goals seek to help improve the AM process and allow for increased use in high precision industries.

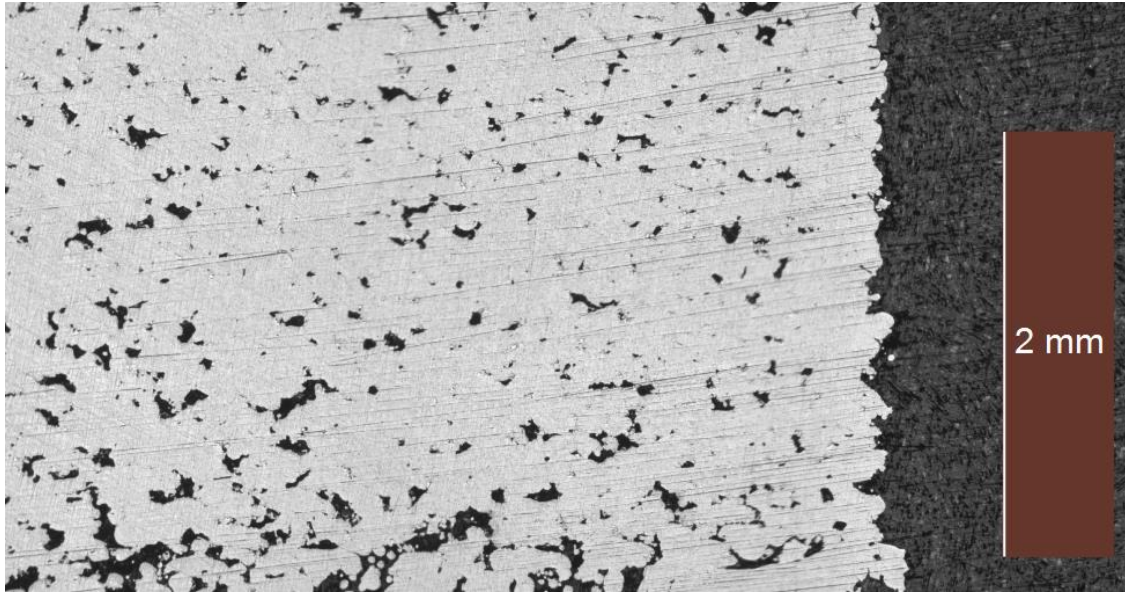


Figure 2-4 Micrograph showing an example of lack of fusion porosity within SLS produced Ti-6Al-4V. Note the trapped powder particles that appear in the pores at the bottom of the image.

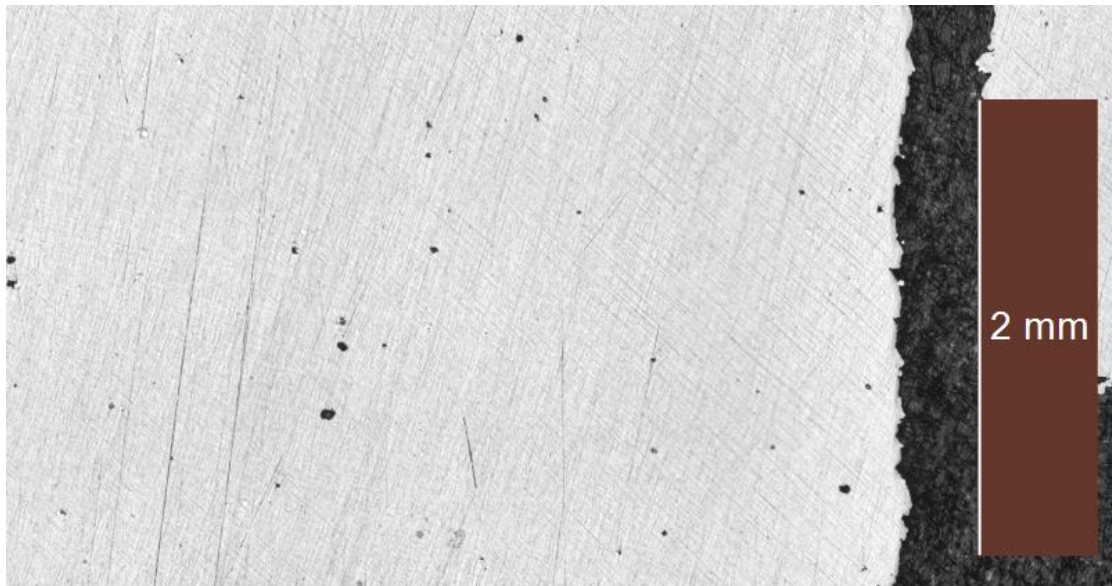


Figure 2-5 Micrograph of an SLS produced Ti-6Al-4V that exhibiting small pores that are possibly keyhole or gas porosity produced by excess energy density.

Chapter Three: Materials and Methods

This chapter will address the experimental setup used to obtain the data analyzed in chapters 4-6. The discussed materials will include IR camera setup for the EBM and SLS processes (Section 3.1), build parameters and part descriptions used in the experiments (Section 3.2), specimen preparation for use in microscopy and electron backscatter diffraction (EBSD) (Section 3.4), and the method for taking surface topology measurements through confocal microscopy (Section 3.5).

3.1 Thermographic Camera Setup for In-Situ Process monitoring

Due to the differences in the SLS and EBM processes, each requires a unique camera set up to ensure that the data collected is as accurate and reliable as possible. This section will cover the essential setup materials and methods needed for each process and discuss their importance.

3.1.1 Camera Setup for the EBM Process on the ARCAM S12

As discussed in Section 2.1, during the EBM process thermographic and optical monitoring equipment must be mounted externally to avoid damage to the camera and its optics and because of the space limitations of the machine itself. For the current research, two builds were monitored using these methods, and the same general setup applies to both. In each case, the camera was mounted externally on an attached metal structure that allows for the camera to be tilted and moved to achieve the best positioning for viewing the build within the machine. For both builds, a FLIR SC7600 camera was mounted on the metal framework and angled at an approximate 22-degree angle to the build plate; **Table A- 2** lists some of the specifications for the SC7600 as well as the camera used during the SLS experiments. To protect the camera's optics and sensor a shutter-less Kapton film canister was mounted on a pre-existing viewport on the ARCAM S12 system as described by Dinwiddie et al. (schematically shown in **Figure 3-1**) [15], [37]. The canister consists of a metal housing containing a roll of 60 μ m thick Kapton film mounted on a spindle, a viewing window made of leaded glass, and an external belt system to control the scrolling of the Kapton film. This continuous scrolling of the Kapton film is vital for protecting the accuracy of the IR data collected during the build by collecting and subsequently removing any metal vapor that is created by the process from the path of the camera's view. Without the film, this vapor will accumulate on the leaded glass window consequently, creating a highly reflective and opaque

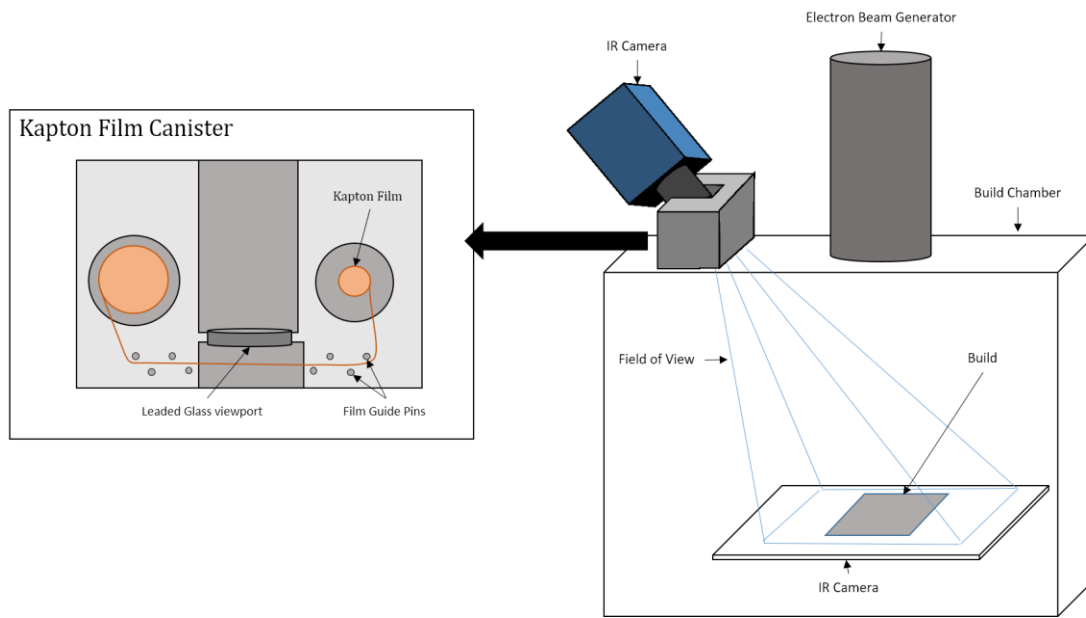


Figure 3-1 A schematic of the IR camera setup used for the EBM process. Another schematic of the shutterless Kapton film canister can be seen as well (left).

surface like that seen in **Figure 3-2**. This coating on the window will significantly reduce the amount of IR radiation that the camera can collect thus impacting the accuracy of the data. The leaded glass window within the canister that acts as the new viewport into the build chamber protects the camera sensor and optics from x-rays produced during the build which could be potentially damaging to the sensitive electronics used to acquire the IR signal as well as the machine operators. However, the use of these materials dramatically lowers the transmissivity and therefore the amount of radiation that the camera captures. In this case, the Kapton film used has only allowed for 78.7% transmission while the leaded glass only allows 1.08% [15], [37]. Due to this reduced transmission, the dynamic range of the camera (the difference between the highest and lowest detectable values) is reduced as well, but correct temperature calibration can allow the user to ensure the accuracy of the thermographic results. Once the camera was mounted, a 25mm lens was attached to the camera without any other image filters. For the first build on the ARCAM S12, the camera's frame rate was 10Hz and the spatial resolution of 380 by 480 pixels yielding pixel an instantaneous field of view of about 350 μ m in the x-direction and 370 μ m in the y-direction (recorded with all other setup parameters in **Table A- 2** for visibility). The reduced frame rate was chosen to condense the amount of data to be stored due to the size of the build, in this case, the amount of data collected was approximately 328 GB of data for the entire build. The second experiment used the same mounting procedure, camera, and camera settings. However, with an increased frame rate of 100 Hz and the overall resolution was increased and utilized a 50mm giving a slightly increased instantaneous field of view at about 320 by 350 μ m(**Table A- 2**).

3.1.2 Camera Setup for the SLS Process on the Renishaw AM250

Due to the differences between the SLS and EBM processes the only limitation on the camera setup was the amount of space that the Renishaw AM250 could provide for the camera and equipment. To effectively monitor the SLS process, the camera was mounted in the machine utilizing a mirror system to view the build plate; **Figure 3-3** shows a schematic of this setup. Using a mirror system allows for the camera to observe the entirety of the build plate and allows for better utilization of the limited space that is available inside the machine. The camera and optics used for the SLS process were different from those employed in the EBM process. The camera used for monitoring the SLS process was a FLIR SC8200 which allows for higher resolution (up to 1024 x 1024 pixels) at much higher frame rates (124Hz at full resolution) when compared to the SC7600 utilized in the EBM

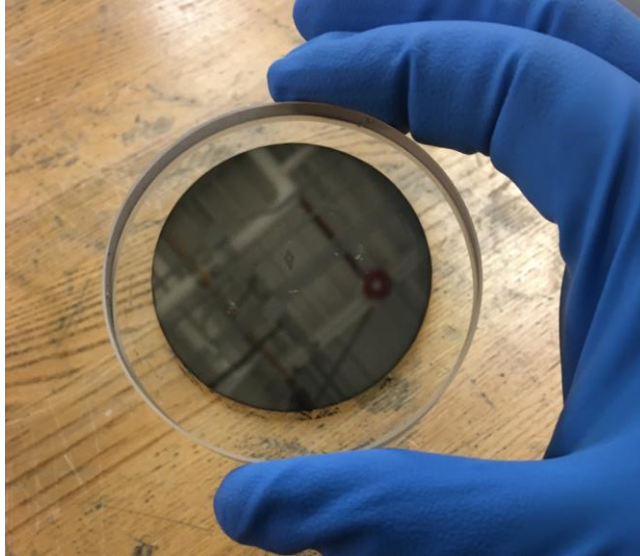


Figure 3-2 Image showing leaded glass that has been coated in metal vapor produced during the EBM

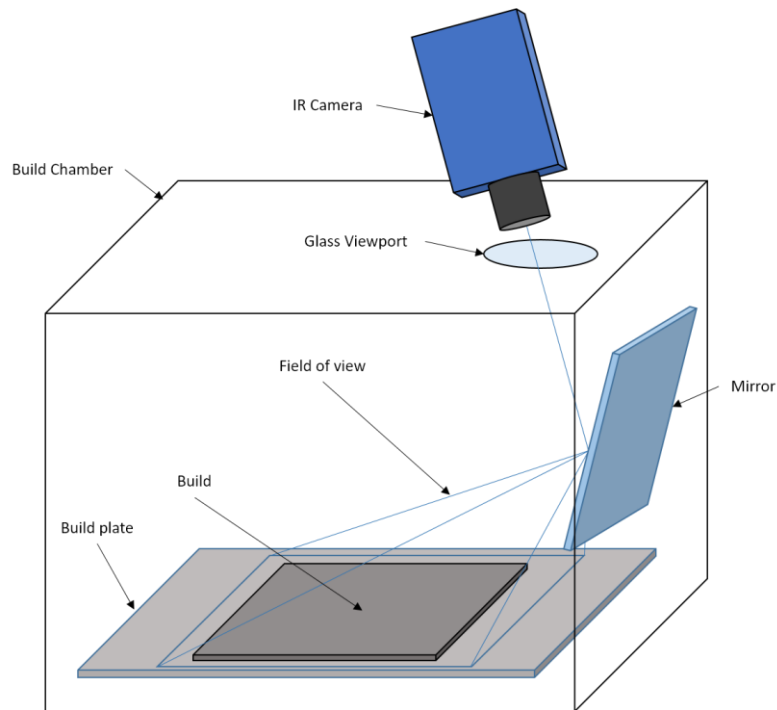


Figure 3-3 Schematic of the camera setup used to monitor the builds produced in the Renishaw AM250.

experiments[63], [64]. For the first two SLS experiments, a 25mm lens was used (same as for the EBM experiment). Also, a neutral density (ND) filter was used to reduce the intensity of the signal seen in the SLS process. The ND filter enabled the camera to monitor the higher temperature portions of the melt pool that would have otherwise been unable to be accurately determined due to oversaturation of the camera's sensor. For the third SLS experiment, a 50mm focal length lens was used to increase the spatial resolution and decrease the instantaneous field of view allowing for the monitoring of smaller features. For these experiments, the resolution and frame rates were modified from those seen in the experiment with the frame rates set much higher than the EBM experiments. For the first two SLS experiments, the frame rate was set to 742Hz (1.347ms), and the image resolution was 320 by 270 (with a pixel measurement of 350 μ m by 350 μ m) which was the maximum resolution that would allow imaging of the entire build plate. In the third monitored SLS build, the frame rate was reduced to 500Hz (2ms) to reduce data storage size, and the field of view measured 256 by 176 with a pixel measurement of 200 μ m by 167 μ m (**Table A- 2**) in an attempt to detect the smallest voids possible.

3.2 Build setup, parameters, and description for microstructure monitoring of the EBM process and porosity monitoring of SLS

3.2.1 Experimental Setup: microstructure monitoring

As previously mentioned, two builds were monitored to attempt to follow the microstructural development of the material during the build. The first build, produced in an ARCAM S12 machine, included a part made of Inconel 718 constructed to simulate structural bracket designs commonly developed within the aerospace industry. This bracket had a continuously changing cross-sectional geometry and utilized two different scan strategies to obtain site-specific control of the microstructure within the build (**Figure 3-4**).

The second build was a grid of nine 20x20mm Inconel 718 cubes divided into two different scan strategies. In this build, all of the cubes utilized a spot-melt pattern described in the literature as a method for acquiring equiaxed microstructure within the build [46], [49], [50]. One set of cubes employed closely grouped melts whereas the other utilized wider point-to-point spacing the hope was to understand the effects that this change has on the resultant microstructure.

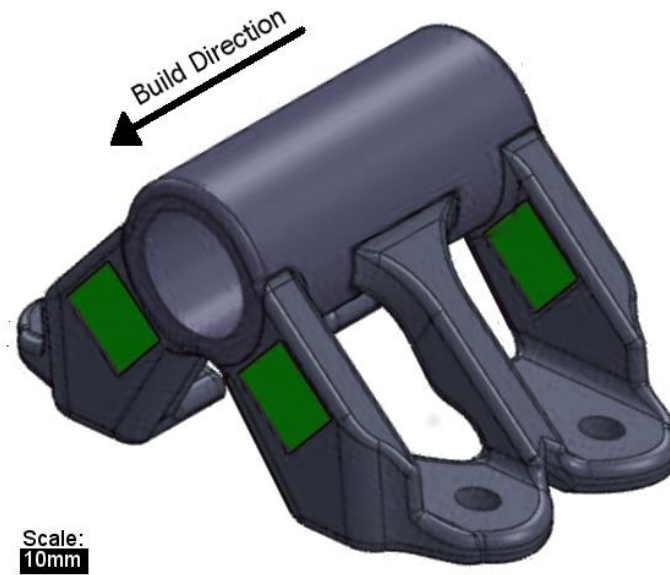


Figure 3-4 Schematic of the build described in section 3.2.1. The regions in green underwent a spot melt scan strategy in an attempt to induce equiaxed grain structures.

3.2.2 Experimental Setup: porosity monitoring

For the first two monitored SLS builds discussed in this research, a matrix of twenty-five, 1cm, Ti-6Al-4V cubes were built on 1.5mm tall pin supports (**Figure 3-5**). For both builds, the hatch spacing, point spacing, and exposure time were varied to show the effects of volumetric heat density on the porosity of the build. Of the 25 cubes, there eleven cubes were replicated using a “best practice” energy density of 58 J/mm³. The remaining thirteen cubes varied from 32-111 J/mm³ to induce either lack of fusion or keyhole porosity within the build. **Table A-3** displays all of the parameters used for the first two builds. Although the build parameters for the cube arrays for both builds were identical, variations on the processing conditions were made in an attempt to change the conductivity of the powder bed. The means by which the thermal conductivity was modified is considered business proprietary and cannot be discussed in this document.

The second experiment performed using the Renishaw AM250 was an attempt to locate engineered defects within the 316L stainless steel. In this case, a grid of cylindrical defects in repeating ten-layer sequences throughout a 25mm diameter cylindrical build (**Figure 3-6**). The defects ranged in diameter from 200µm to 1mm at 200µm intervals and the height from 20µm to 100µm followed by solid layers of material for the imaging. Each of these sets of voids were then separated by another ten layers of solid material to isolate the effects of each set of defects from the following set. A schematic of these defects can be seen in **Figure 3-7** and are their measurements are recorded in **Table A-4**.

3.3 Specimen preparation for microscopic analysis and electron backscatter diffraction (EBSD)

All samples were section mounted in conductive phenolic resin and mechanically ground and polished. Samples used in electron microscopy were polished to a final polish using a 0.5µm colloidal silica. Specimens that underwent optical microscopy to reveal dendrite spacing were later etched using an equal mixture of HCl, acetic acid, and HNO₃ to expose the material microstructure.

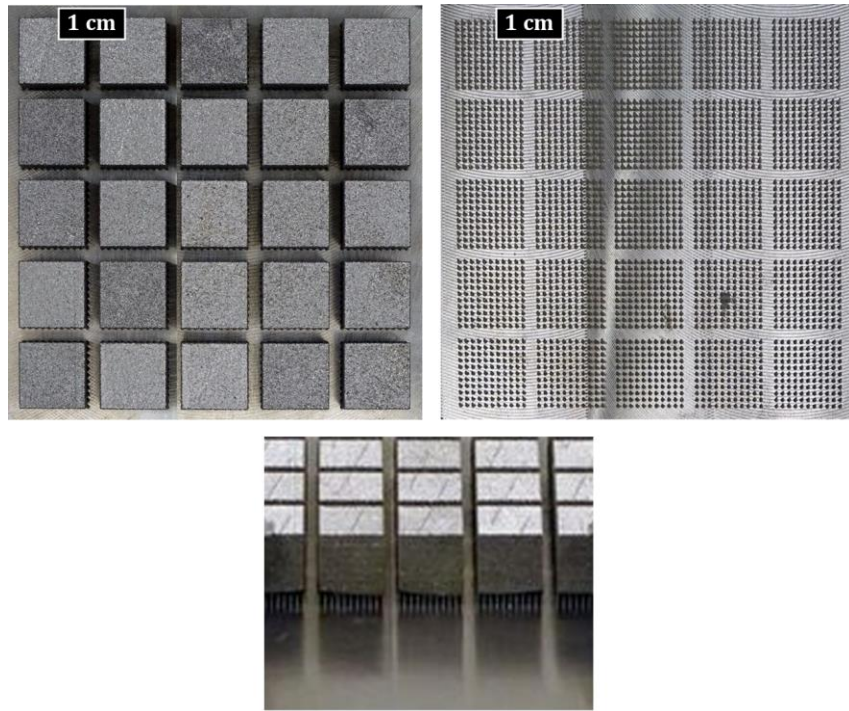


Figure 3-5 The top images show the arrangement of the test cubes for the first set of SLS experiments (left) and the pin supports that were under the build (right and bottom).

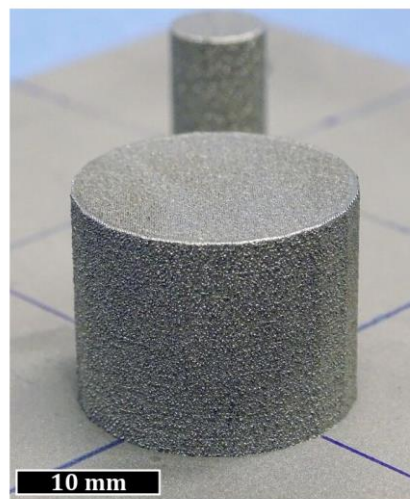


Figure 3-6 Image taken of the completed cylindrical defect build.

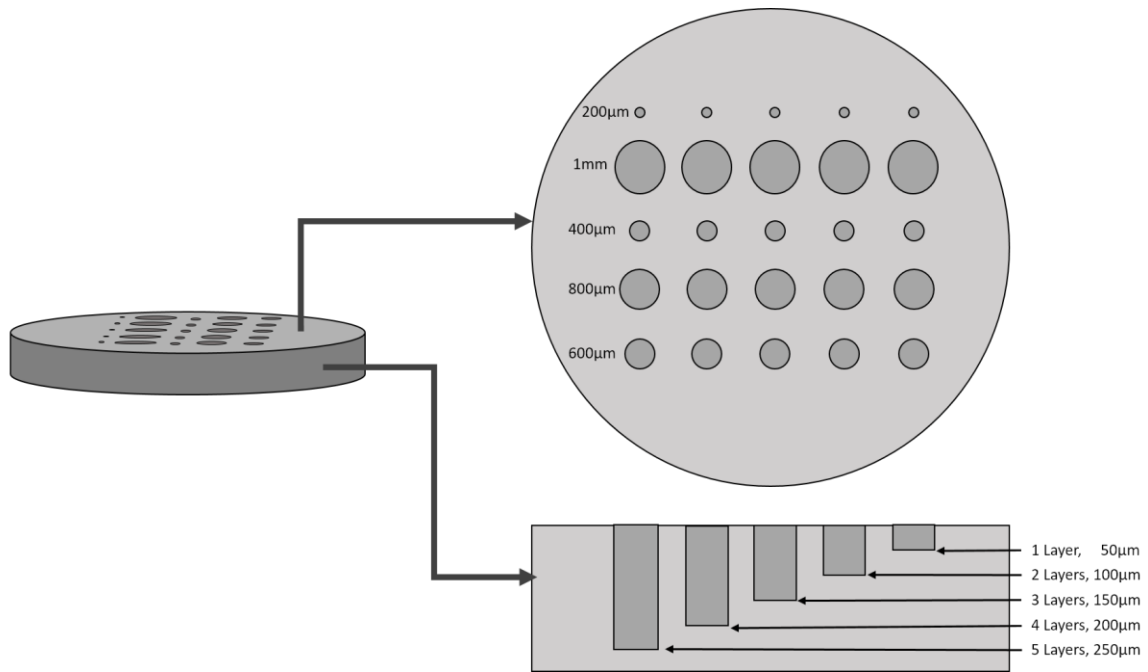


Figure 3-7 Schematic of the defects placed within the cylindrical build the array of defects consists of 20 pores that varied from 200µm to 1mm in diameter and 50µm to 250µm.

3.4 Microscopic imaging, electron backscatter diffraction, and confocal microscopy

3.4.1 Optical Microscopy Imaging

For monitoring the microstructural development of the materials collected from the EBM process and the porosity of the material built in the SLS system the parts were imaged using the LEICA DM4000 MLED. All images used for viewing porosity and bulk microstructure were imaged at 50x, and all images taken to reveal dendrites were imaged at 1000x.

3.4.2 Electron backscatter diffraction (EBSD) imaging

All EBSD data used in this research was performed using a JEOL 6500 field emission scanning electron microscope in conjunction with an EDAX Hikari EBSD camera. Imaging was taken using an accelerating voltage of 20kV and a current of 4nA. The resultant imaging and data produced treated the material as a standard FCC nickel crystal structure with a step size of 11 μ m.

3.4.3 Surface topology measurement via confocal microscopy

All surface topology measurements shown in this research were collected using the Keyence VR system. Imaging was taken at 12x (23.6 μ m/pixel) and 25x (11.8 μ m/pixel) zoom across the surface of the parts and stitched together using the associated Keyence software. All measurements were corrected for any surface tilt to remove any bias in the measurements taken and preserve the surface features. The resulting measurements were collected and then overlaid on the collected images for easy comparison.

Chapter Four: Correcting In-Situ Temperature Measurements for Emittance Change in the EBM Process

The work included in this chapter has been published in the Scientific Reports article “Thermographic Microstructure Monitoring in Electron Beam Additive Manufacturing” produced by Raplee et al. [65]. Some of the figures, results, and discussion showed here were presented in the article. All work presented has been modified and revised to expand the discussion of the material presented in the published work and introduce some new concepts. My primary contribution as the first author for this work included: (i) development of the problem and reviewing all relevant scholarly works, (ii) developing the methods discussed, (iii) analyzing and discussing the data collected, and (iv) writing the article. As the first author, I retain the rights to reproduce the contribution as part of my thesis per the ownership of copyright given by Nature Publishing Group.

During the metal AM process as the part undergoes melting, the material melts transitions from high emittance powder to relatively low emittance solid material, leading to a drastic change in surface emissivity. This change in emissivity occurs because when the material is in powder form the porous surface of the material allows for large amounts of internal radiative reflections from the various powder particles. As these particles undergo melting this porous surface melts together and creates a solid relatively flat surface thus reducing these pseudo-blackbody cavities that cover the surface [16]. This change in emissivity causes the camera to interpret the IR signal as a drop in temperature during the melt rather than the expected increase due to rapid heating. To navigate this issue and method for precise location of the time in which the material transitions from powder to the as-printed material must be determined for the material and then a corrected temperature profile reconstructed. As stated in the literature, it is critical to the value of thermographic monitoring to understand this change in emittance, but no methods for navigating this issue have been proposed. In this chapter, a detailed explanation of the data analysis algorithm used to correct for emittance change within a layer will be discussed in full. It will include the necessary steps needed to calibrate the temperature curves for the two different material states (4.1). Then describe the subsequent filtering (4.2) and analysis (4.3-4.4) that are needed to locate the transition between the two temperature calculations, to obtain a more accurate temperature measurement.

4.1 Calibrating the data to acquire the initial temperature curves

To accurately acquire the temperature curves to acquire the true temperature of both the powder and as-printed material, a pair of signal intensity temperature calibrations is needed. A calibration target built using the material observed, Inconel 718, was utilized to obtain these temperature curves for the material. The specifications of this target are similar to the target described in the work of Dinwiddie et al. with a simulated blackbody, pocket to collect loose powder, and a series of thermocouple mounting locations that allow for comparative calibration[15]. The block was then placed in the powder bed of an ARCAM S12, fitted with four type K thermocouples, and heated using the machine's preheating scan strategy to slowly heat the part from room temperature until the operating limits of the thermocouples, 1200°C. During this heating, the target was monitored using the IR camera, and the signal intensity and thermocouple readings were recorded at regular intervals. The temperature of the surface is determined by linear extrapolation from the temperature values collected by the four thermocouples. Using the calculated surface temperature and signal intensity values collected from the material, a simple polynomial curve fit could be performed to obtain a mathematical relationship between the two. **Figure 4-1** shows these curves and their related equations used for calculating the metal powder and as-printed part temperatures. Once the signal intensity-to-temperature curves were determined for the material, the curve equations could then be applied to every pixel within the image throughout the length of the video to obtain two different temperature curves for the material at each state, powder and as-printed. **Figure 4-2** gives examples of these temperature curves for pixels at different locations in the build with different scan strategies. These initial temperature curves will then be used for the subsequent reconstruction of the material's temperature during the layer's melting process, being used as the pre and post-melt temperature curves depending on the state of the material.

4.2 Filtering the data to remove false positives due to signal noise

When analyzing the data to detect the change in emission for a part undergoing EBM melting, several factors influence the success of determining the transition from metal powder to the as-printed state, specifically the pre-heating and post-heating processes. During these processes, the electron beam is unfocused and rapidly passed across the surface which can create significant negative slopes which register as the transition rather than the actual transition point. Also, small signal peaks within the data that only last for a single frame which

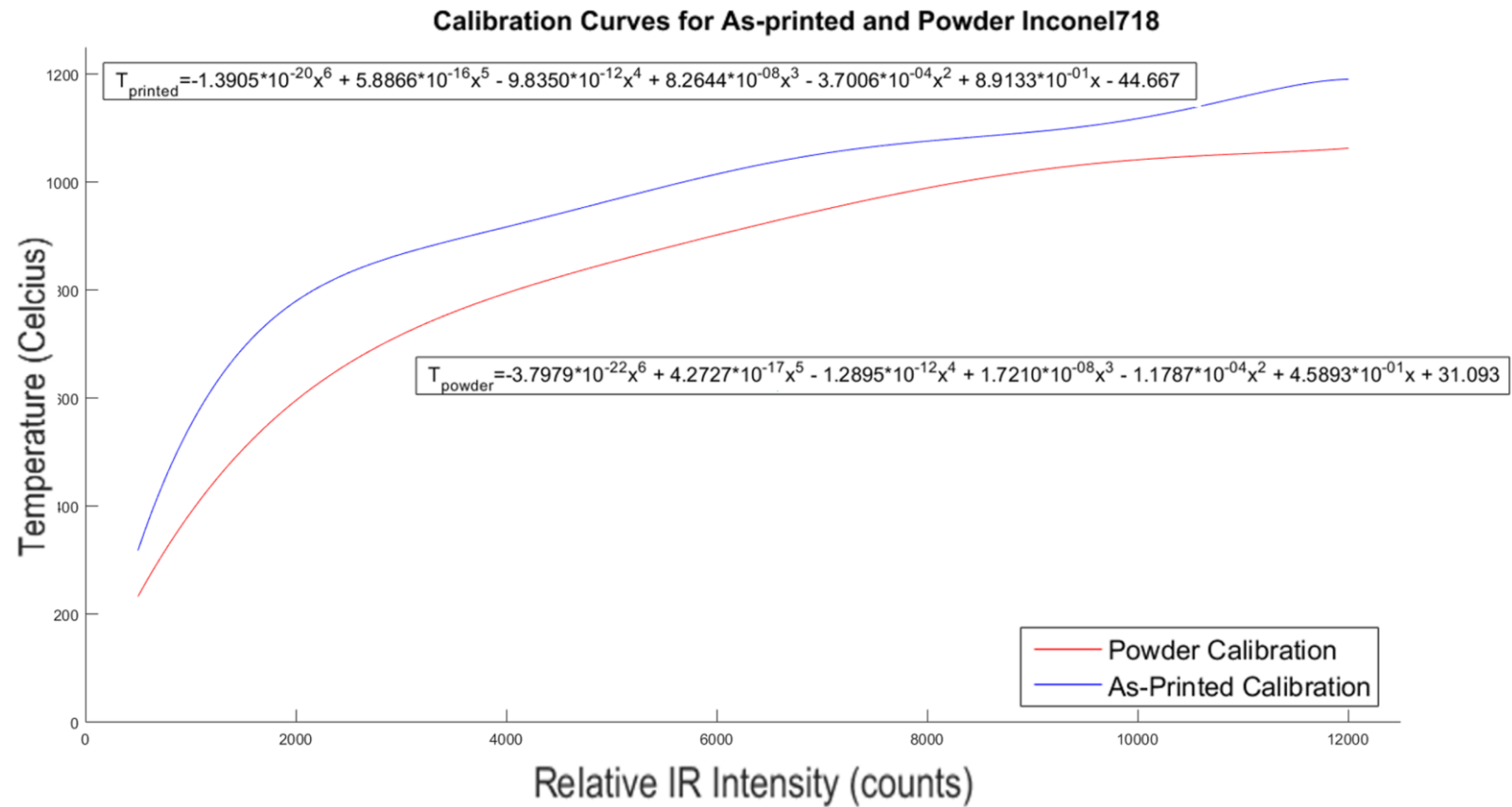


Figure 4-1 Intensity-to-temperature calibration curves for Inconel 718 produced with the setup described in this research and their corresponding equation. Both curves show non-linearity with a rapid increase in the lower temperature regions. [52]

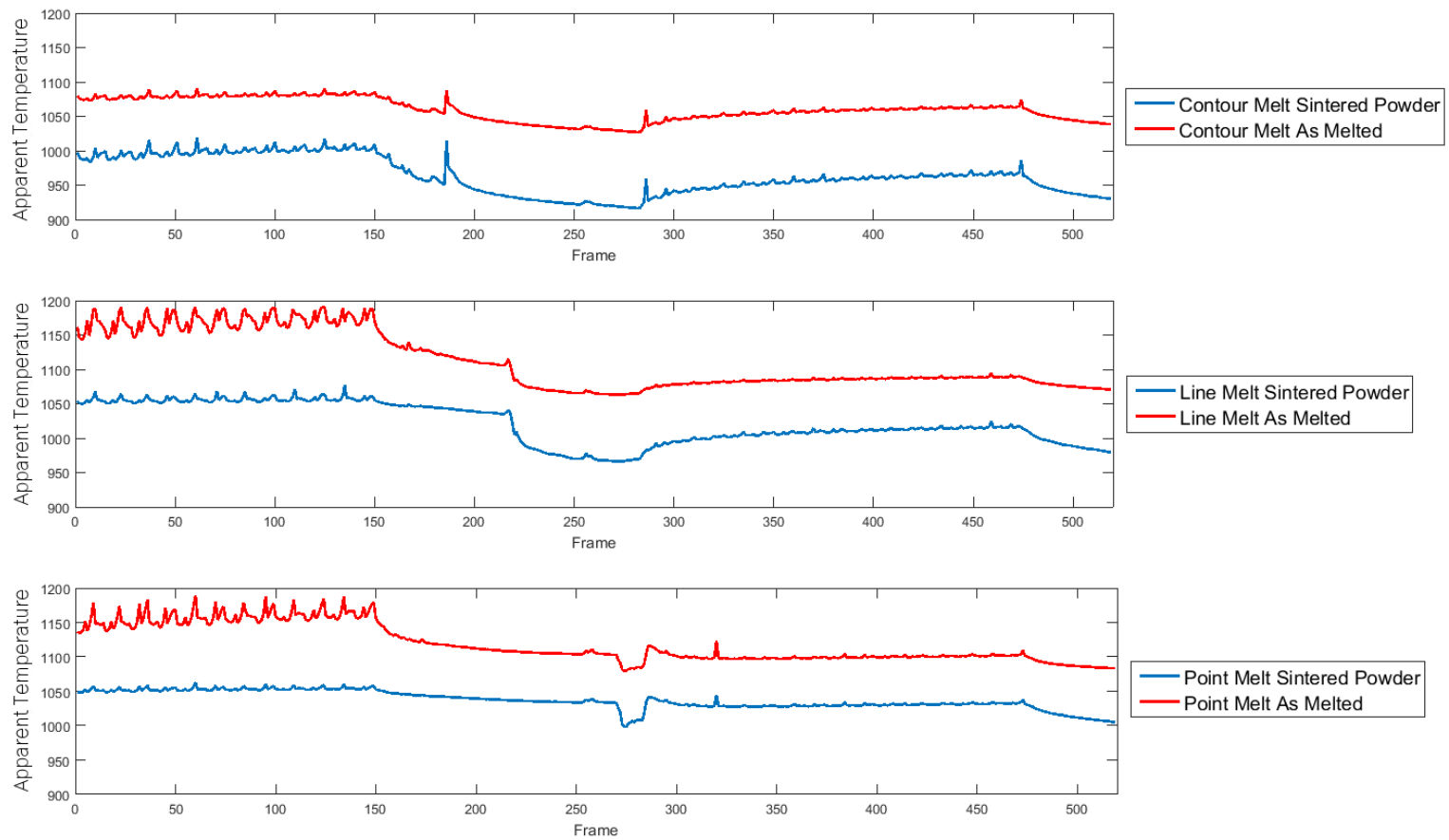


Figure 4-2 Powder and as-printed material temperature calibrations for regions that underwent contour melting (top), line scan melting (middle), and point melting (bottom) regions. In each respective case, the melted material is significantly higher as expected. [52]

Can also be misinterpreted by the computer as a false transition point. Selectively trimming and filtering the data helps to remove these false positives and combat this interference. First, the data is trimmed to focus on strictly the timeframe in which the melting of the layer geometry occurs to avoid interference from the pre-heat and post-heat processes. Then to reduce the noise within the signal, it is filtered by mathematically processing the data using the methods described by Savitzky and Golay for smoothing using a simplified least squares procedure [66]. This method requires the use of polynomial equations of varying numbers of data points and weighting coefficients, smoothing to the data series more as the size of the polynomial and number of data points used increases. **Table A-5** contains the weighting values utilized for this research, which includes seventeen frames, eight preceding the current data point, the current data point, and the eight frames following the current data point. **Figure 4-3** shows the results of this filtering, which shows a considerable reduction in the noise within the data, giving a much smoother temperature history curve. This smoothed data can then be used to locate the material transition point and aid in accurately reconstructing the correct temperature profile of the part.

4.3 Locating the powder to as-printed transition

With the newly filtered and trimmed temperature data correctly calibrated, the location of the exact frame where the transition from powder to as-printed material occurs needs to be located. As previously mentioned, this transition is characterized by a substantial decrease in temperature that follows melting (**Figure 4-4**). One of the easiest ways to identify these rapid declines in intensity is to determine the regional slope of the temperature curve for each frame recorded. This slope data is collected by taking the value of the pixel's temperature five frames before and after the current frame and then a simple slope calculation between the two points. Then, to determine the transition point, the region with the most significant negative slope is selected. However, in areas where a second pass reheats the material, the material can see a sizeable secondary peak in the IR signal that behaves much like the actual melting of the material. In some cases, the algorithm may treat this secondary heating as if it is the real melt point which can cause error in the resulting data.

A way to fix this error is to employ a threshold value that selects the first instance in which the slope is significant enough to indicate the transition from powder to as-printed material as a fraction of the maximum intensity seen within the data. This threshold value has

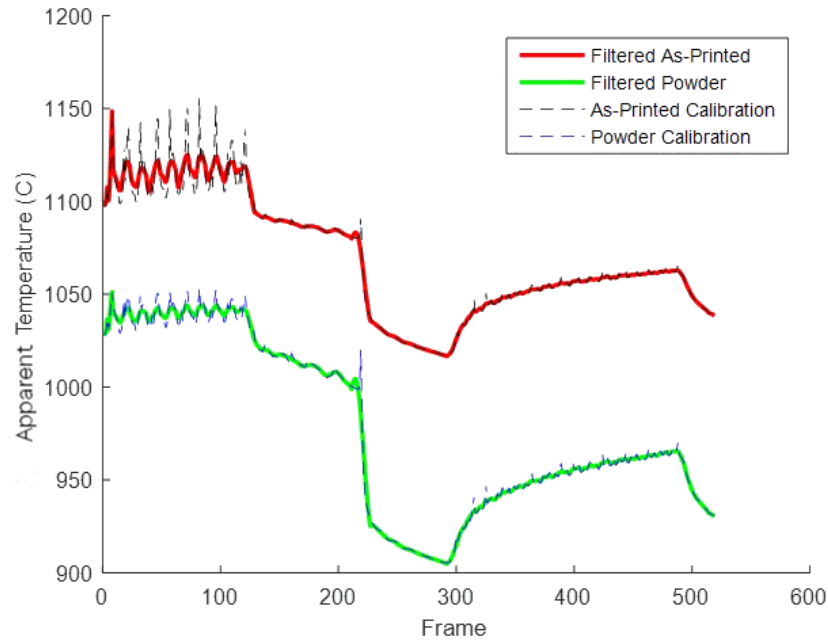


Figure 4-3 Representations of the two filtered temperature curves for the as-printed and powder material for a single pixel. The reduction in the peaks is evident in the preheat scanning at the beginning of the build. [52]

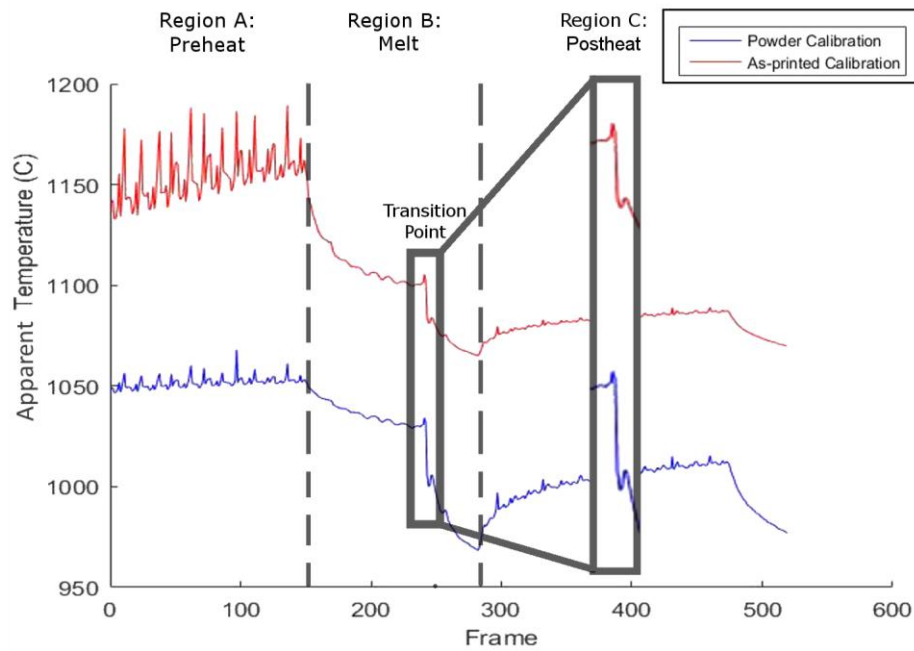


Figure 4-4 Representation of the preheat, melt, post heat regions of the temperature profiles. Of note is the location of the material transition which rapidly drops off due to the emissivity change of the material. [52]

to be determined by the user observing the effect seen in the resulting output and the initially collected IR data. In the results displayed in this research, the selected threshold value for this research was 70% of the largest negative slope. By setting this threshold value, the detection of the correct slope can be determined as seen in **Figure 4-5**.

Once the proper transition region of the build is selected using the threshold value, the final step to ensure that the data behaves correctly is to select the maximum value within the transition region. Ensuring that the temperature curve flows smoothly from one temperature to another instead of reaching a peak and then dropping before transitioning to the higher temperature calibration.

4.4 Methods for determining the occurrence of melting

With the transition from powder to as-printed material identified for all of the pixels within the data, a method for determining whether or not the material had melted is needed to ensure that the temperature of the unmelted regions within the part and powder bed adhere to the correct temperature calibration curve. There are two methods of determining the no melt condition, each with their advantages and disadvantages; determining melt conditions through calculation and determining melt conditions using the existing known part geometry.

4.4.1 Determining melting through calculation

The first method used to determine whether melting had occurred during the current layer was to calculate if a significant enough drop in the IR signal intensity took place in the data which would indicate that the material had changed from powder to solid and underwent a drop in emissivity. To determine whether this decline in intensity and therefore melting had occurred, two data points were selected along the filtered temperature curve, the first being one of the first frames directly after the end of the preheating procedure and the second being a point within the post-heating procedure in which the material begins to reach the predetermined post-heat temperature. Then by taking the slope between these two data points over time, a value is given to every point within the build. When melting occurs within the observed layer, the slope calculated is a much steeper negative slope, than the relatively flat or sometimes positive slope calculated for regions in which no melting occurred (**Figure 4-6**). After collecting the slope values for the material, a threshold slope value is selected as a logical operator in conjunction with the previously determined material transition point as a means of reconstructing the temperature curve.

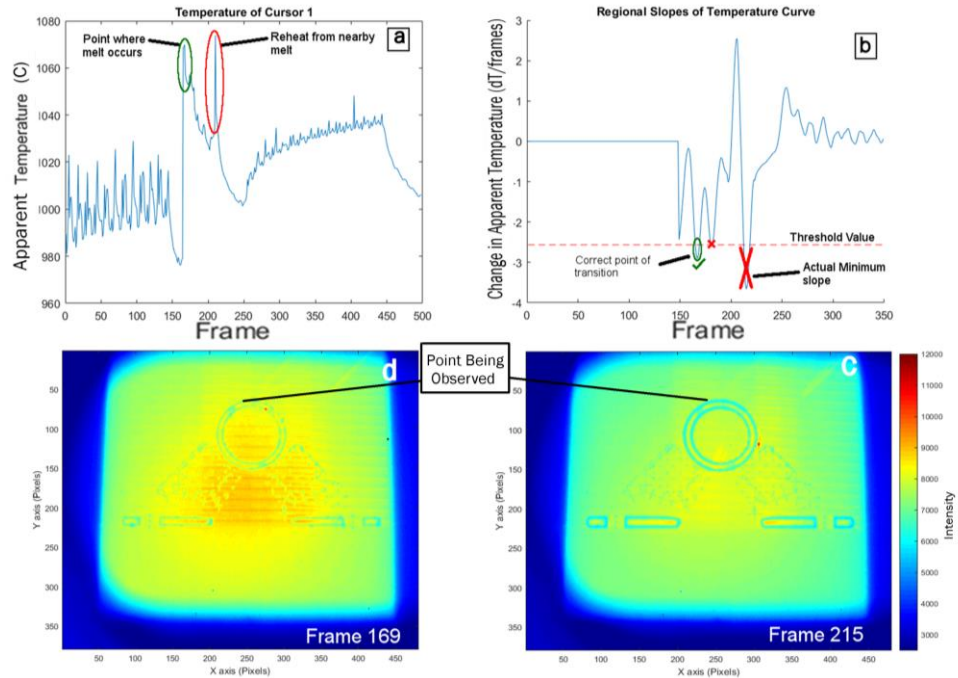


Figure 4-5 The two graphs seen here show the thermal signature of a point that is reheated by a second pass (left) and the slope values calculated for the point at each frame(right). By selecting the images shown on the bottom show this location in the build for the frame in which the actual melting occurs and the incorrect frame where the transition was determined without the threshold value. [52]

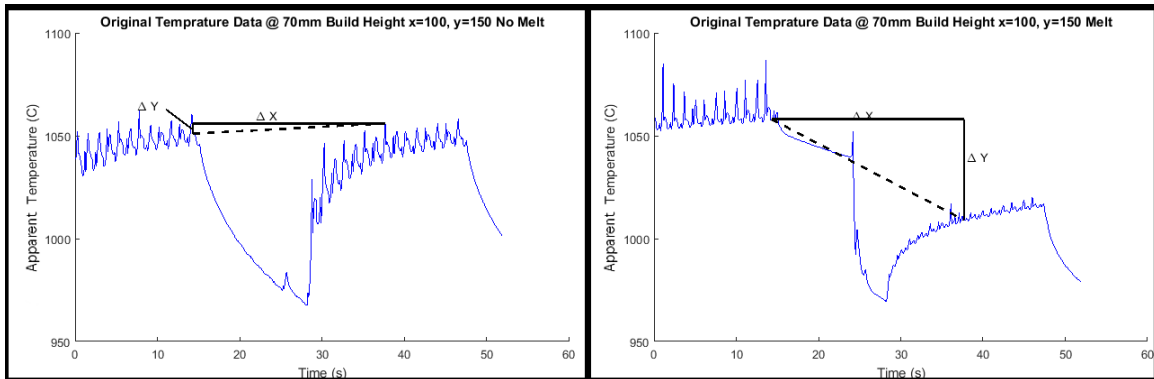


Figure 4-6 Image showing the representative slope of a region that does not undergo melting (left) and does undergo melting (right). In the melted material there is a significant change in the material emissivity which causes the material to remain at a much lower apparent temperature that a region that does not melt. [52]

This method provides a quick and efficient method for determining whether or not melting occurred within the imaged layer. However, this approach is susceptible to errors that occur involving material swelling or inconsistent powder distribution during the raking process. When swelling occurs on the material surface, the signal received by the camera is reduced due to the change in the direction of the material's emitting surface. This reduction in the signal received by the camera decreases the amount of the perceived temperature drop that occurs, and the algorithm may determine that no melting occurred within the region. Similarly, when there is inconsistent coverage during powder application, the low emissivity material underneath could show through, or interpret the increased layer thickness as a lower starting signal intensity. When there is improper coverage, it will also cause the algorithm to incorrectly determine regions where melting did not occur even though the material in that region had melted. It has also been seen in the literature that powder can begin to melt during the preheat process if the preheat parameters are sufficiently high which will cause the material to drop in intensity and may also be a cause of this error [37]. **Figure 4-7** shows a few examples of these problematic areas and the correlation to the images taken directly after the application of the powder. However, these errors could also give the user an indication of when swelling or poor powder application is occurring on the surface leading to issues such as build failure due to damage of the powder distribution rake and could be a monitoring tool themselves despite its detrimental effect on the accuracy of the temperature correction.

4.4.2 Setting the melt region using image registration.

In an attempt to reduce the computation time of the algorithm, another method utilizing the governing cross-sectional geometry for a layer in conjunction with the data was used to determine whether or not melting occurred. When a design file is prepared for use in the machine a series of image files are created to instruct the machine where the geometries lie on the substrate (**Figure 4-8**). These files could act as a useful way to determine where in the IR imaging melting occurs as well and can be leveraged to determine regions where melting occurs by fitting the computer-generated files to the geometry that is imaged by the IR camera. This technique of fitting one image to another is known as image registration and involves the correlation of two images by detecting outstanding features within the two images [67]. Then the algorithm iteratively attempts to match them based on the grayscale intensity of the images, determining the transformation needed to align one image to match the other designated as the reference [67]. In this case, the IR image was selected as the reference image

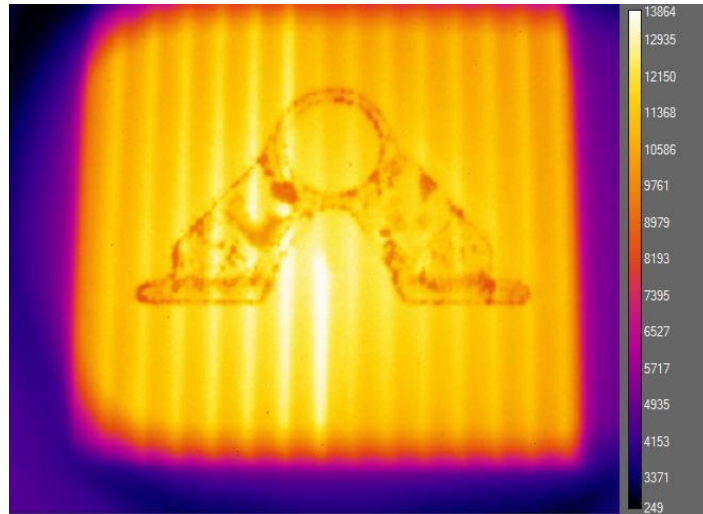


Figure 4-7 IR image collected from the preheat process in the EBM process. The dark regions within the part could be inadequate powder coverage or unintentional melting of the powder due to overheating.

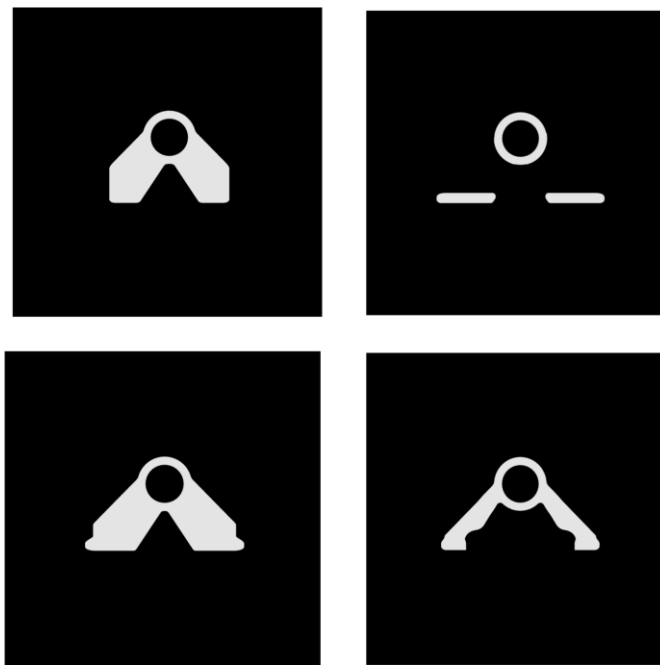


Figure 4-8 Several images of the representative layer geometries given for several different layers in the build. Combined with image registration these can determine where melting is supposed to occur within a part.

so that the computer-generated image, when properly aligned, would act as a logical operator to tell the algorithm that melting occurred (**Figure 4-9**). By applying image registration rather than calculation to determine the melt within the imaging file, the process was much faster with the proper settings, and it removed the errors that occurred due to inconsistency within the powder bed and surface swelling. However, this also means that the algorithm is blind to regions that go unmelted due to faults in the build process. Another issue that can occur due to the iterative nature of the automatic registration is that the process can become computationally taxing if too many iterations are needed to resolve the proper transformation. Also, the algorithm can fail to determine the exact transformation to perfectly align the images, especially when trying to align complex geometries. When this misalignment occurs the image can cause areas of the build to seem as though they had melted even though they had not, typically in the regions surrounding the part perimeter (**Figure 4-10**). These errors are rare but they can dramatically affect the results, so careful observation of the outcomes is needed to verify if the correct regions are selected.

4.5 Results

Once the algorithm has calculated the powder and as-printed temperature profiles, located the instance where the material transition occurs, and determined whether a melt occurred for that region, a newly corrected temperature history for the part can be determined. With all of the requisite information, the algorithm begins to form the temperature curve using the temperature calibration for the material in the powder state until the instance in which the transition is determined. At the determined transition point, the algorithm then switches to the as-printed calibration for the material and continues with this new temperature calibration until the end of the data collected for a layer. **Figure 4-11**, displays an example of the output created by a single point in the build, which shows the original calibrated powder and as-printed temperature curves for the material and then the recreated temperature profile after the algorithm is applied. By systematically applying this correction approach for every pixel within the collected data, a corrected IR video can be readily created (**Figure 4-12** and Attachment 1), allowing for a better visualization of the temperature of the surface during the melting process. This correction can allow for much more useful build information such as thermal gradients within the build or solidification parameters that govern the material's performance and microstructure.

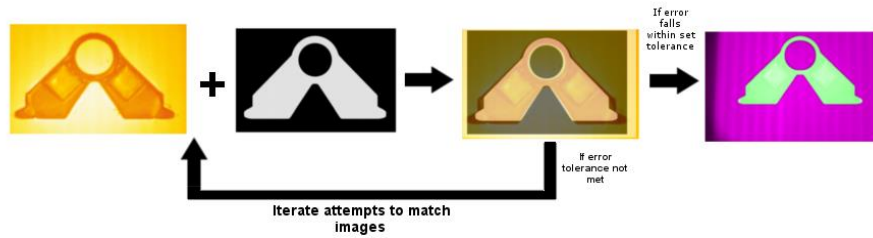


Figure 4-9 An example of the image registration process. (Left to Right) The process begins with two images to attempt to register to each other. Then it attempts to create a transformation of one image onto the other, and will iteratively attempt to achieve the set tolerance. Finally, after reaching the correct tolerance, the images are considered fully registered to each other, and the layer geometry can be used as a logical operator.

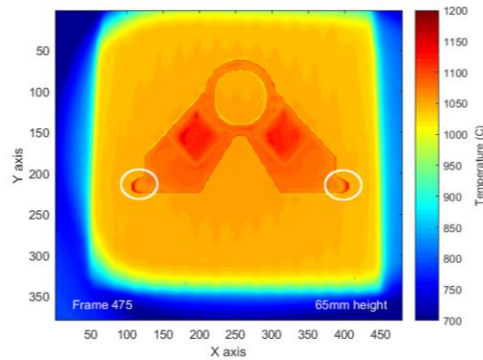


Figure 4-10 Final part temperature in a build where the image registration did not accurately apply the layer geometry image to the part (regions highlighted in white).

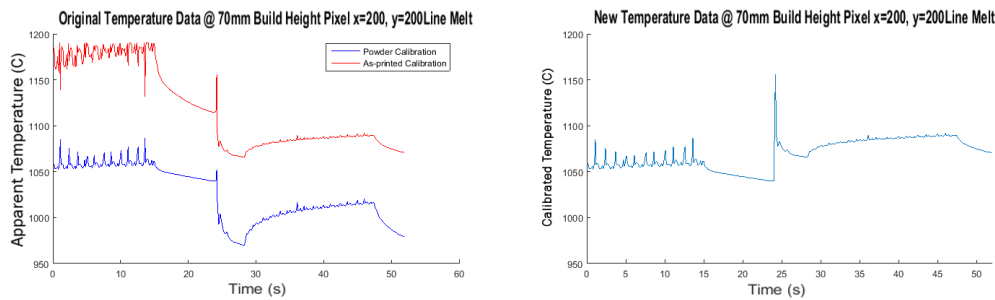


Figure 4-11 Outputs for the original as-printed and powder calibrations (left) and the newly reconstructed output (right). The new thermal signature shows a temperature profile that is more consistent with what is expected of the rapid heating and cooling of the AM process. [52]

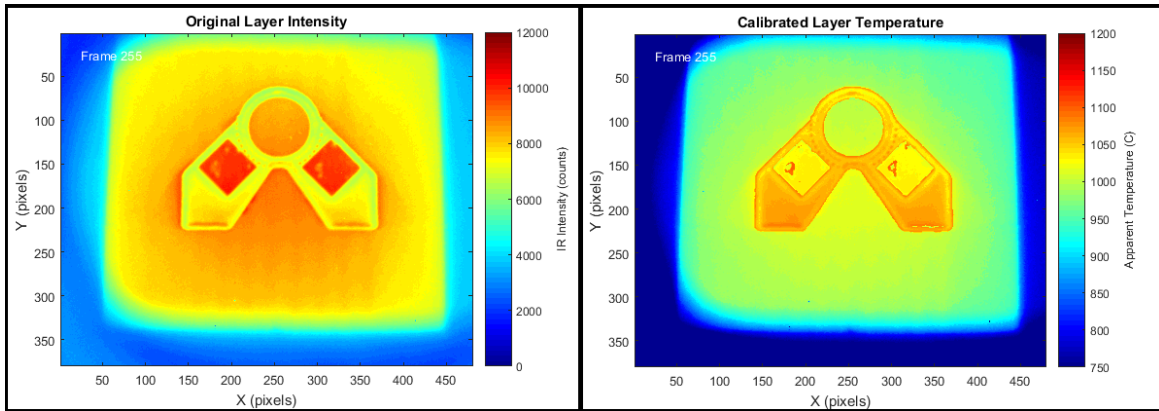


Figure 4-12 Comparison of the original IR video interpretation (left) and the newly calibrated temperature data (right). As seen in the new data the material that is melted has a much higher temperature than the surrounding unmelted powder which is expected. [52]

4.6 Limitations and Future Work

Although the technique described in this chapter can generate a corrected temperature curve that gives a more accurate description of the material's thermal history, there are still many limitations to the technique's use and sources of error that still need solutions.

The most prominent issue that needs addressing is that the time and length scales that could be detected by the camera are not adequate to resolve both the size and movement of an electron or laser beam reliably. For example, in the data shown the spatial resolution was measured at about $350\mu\text{m}$ which is significantly larger than the $100\text{-}200\mu\text{m}$ size of the electron beam [68]. Which means that if the beam were to land within a pixel during an image, the data within that pixel would represent the average temperature of the beam and the space surrounding it within the pixel, which reduces the temperature values collected and decreases the accuracy. Compounding this error is the lack of temporal resolution that the cameras can discern as well. For instance, in the data shown, the framerate was set at $10\text{-}100\text{ Hz}$ to reduce the amount of storage needed to handle the data. Meaning that if the beam is capable of moving at 5m/s , the beam could pass through upwards of 200 pixels between frames before collecting the next frame, giving less accurate results for these regions. Data should be collected with high-speed, high-resolution imaging to determine if increasing the temperature profile accuracy is possible. However, this would add to the amount of data storage and processing that would be needed as well which could prevent real-time processing. Also, because of the resolution and frame rate for this data is relatively low, the camera is also unable to detect the liquidus region of the temperature curve both spatially and temporally, which will also affect the resulting temperature profile's accuracy as well.

Even though increasing the camera resolution could help to enhance the accuracy of the corrected temperature profile and allow for resolution of the liquidus region, a calibration for the liquidus regions of the build has not been determined. As the material reaches its liquidus temperature, the emittance of the now liquid material drops yet again, as seen in **Figure 4-13** which shows the emissivity of Inconel 718 for various temperature values [69], [70]. Since the calibration method used to determine the calibration curves for the powder and as-printed materials rely on the use of Type-K thermocouples, whose maximum operating temperature is 1200°C , it is not possible to determine the emissivity and calibration for the 1300°C and higher liquidus region of Inconel 718. This lack of a liquidus calibration means that

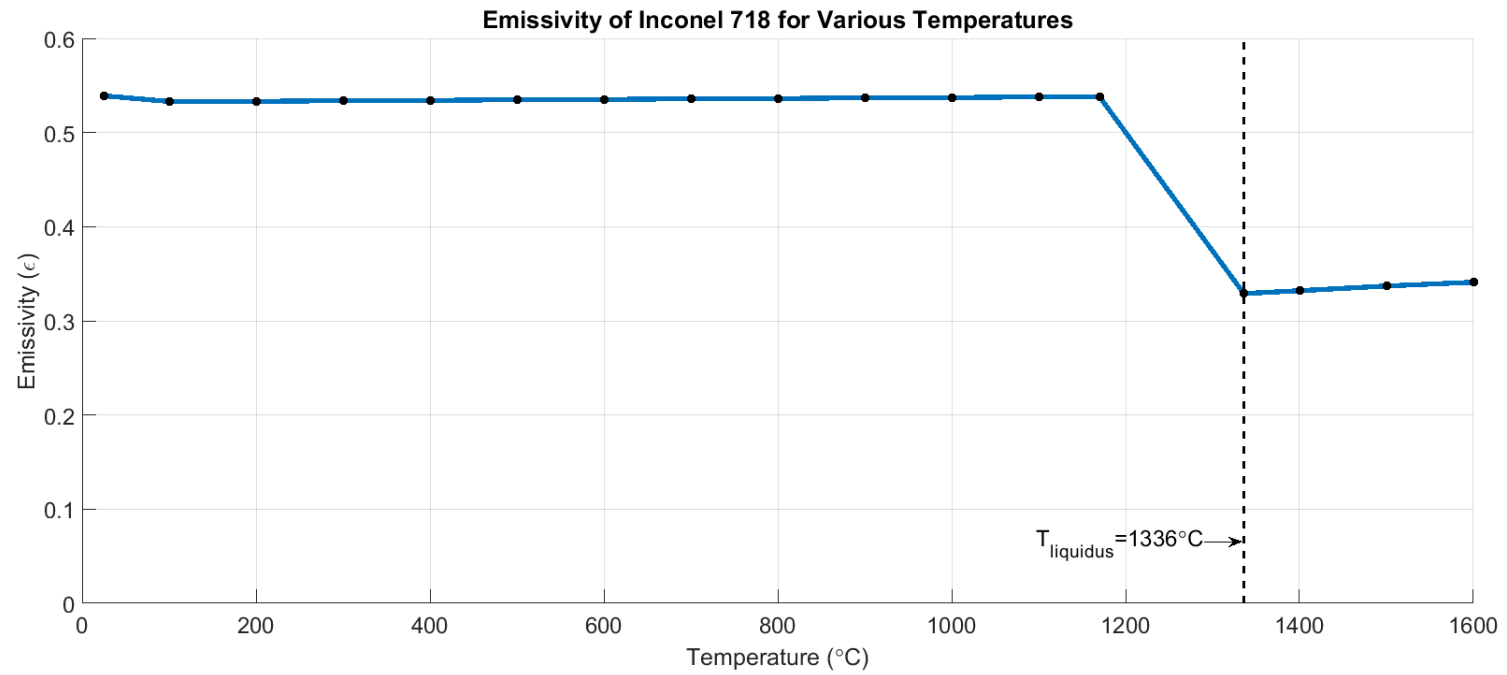


Figure 4-13 Graph showing the change in emissivity of Inconel 718 at regular temperature intervals, as the material transitions from solid to liquid the material drops by about 30% showing which would significantly alter the IR intensity that observed by the camera. Adapted from data presented in Romano et al. [69].

even if the resolution of the IR camera allowed for the detection the liquidus region, both temporally and spatially, the data would drastically depart from the actual temperature as the part reached the liquidus and the emittance of the material changed once again. If high-speed studies are performed, and the real temperature of the material is to be determined, this calibration is needed to be able to obtain useful data as well as another data analysis method for detecting and correcting this second change in material emittance.

Also of note in regards to error are issues that occur with changing surface topology. In the data, the regions that underwent a point melt scan strategy seemed to be at a slightly higher temperature when compared to line scan regions even after ample time had passed to allow for the two regions to reach the same uniform temperature (**Figure 4-15**). Upon analyzing the surface of other parts that underwent a point melt scan strategy, the surface topology is noticeably different from that of the other region (**Figure 4-15**). These point melt regions show a characteristically rippled surface texture that consists of numerous depressions in the surface with small cavities in their centers (**Figure 4-16**). It is assumed that in the data collected these surface recessions and small cavities act as high emissivity locations for the material, and when averaged within single pixels that are much larger than these regions, the resolution cannot detect these cavities and the average pixel intensity increases. This phenomenon needs to be further analyzed, and a correct calibration is necessary to correctly provide the temperature profile of the build for these regions.

Also, a possible source of error within the part is metallization of the Kapton film used in the camera setup. Although the Kapton film was used to remove any error associated with the metallization of the viewport between the camera and the build chamber, the device used does have its flaws. During the build, the operators noticed several instances in which the canister would fail to move the Kapton film, and the film would become stationary for some amount of time. This could have allowed for metallization of the film's surface which could affect the data collected once a significant amount of metal vapor had accumulated. In **Figure 4-14** an image taken of the Kapton film after the end of the build reveals the amount of metal vapor deposited on the film while it was unable to move, in this case causing the film itself to burn. When the film was caught in the device, the accuracy of the data may have been harmed. However, the extent of these effects has not been assessed.



Figure 4-14 Image taken of a segment of the Kapton film from the EBM process during the first build. The dark circle in the center of the film is a region where metalized vapor would have been deposited while the film was stuck in the machine.

One final hurdle that the use of this technique faces are issues with processing speed and data handling. Currently, to correctly determine the temperature profile with this method the entire set of data collected must be known which limits its use for real-time monitoring purposes. However, if there were methods that allowed for the beam location to be known at any given point and then used in conjunction with this methodology; the correction algorithm could inherently know the location of the beam at any given time. This synergy between the machine and the calibration algorithm would remove the need for complex calculations and filter to determine the transition from powder to as-printed material which would allow for real-time processing of the data and promote its use in a process-control architecture. Also, if the need to employ image registration on a layer by layer basis was unnecessary, and a single image could be used for registration before the build process to give a transformation matrix for aligning all of the imaging, it would reduce the processing power and time needed.

By addressing these issues and concerns, the hope is that real-time processing would become more achievable as well as utilize the temperature data for process control which could significantly improve the additive manufacturing process. Along with addressing these issues, there is a need for the creation of a correction algorithm for the laser powder bed process. Due to the high operating temperature of the electron beam process, the change in emittance within the material has a much more profound effect on the intensity received by the camera. Since this effect drastically changes the intensity value at the transition of the material, it allows for easy recognition of the transition between powder and as-printed material. In the laser system, the change in emittance is subtler due to the lower operating temperature, but the effects are still present, and a proper calibration is needed to acquire true temperature data. In laser systems, there is not a drop in intensity that occurs because the chamber temperature does not make the change in emissivity as apparent. Therefore, another method for determining the transition from powder to as-printed materials would need to be determined. Also, another appropriate method to calibrate the camera for the calibrated temperature curves is required to facilitate a correction algorithm, because the differences between this process and the EBM process and the camera setup vary which impacts the resulting calculated temperature values. By developing the correct calibration and detection algorithm, the ability to collect temperature data should be rather straightforward and follow

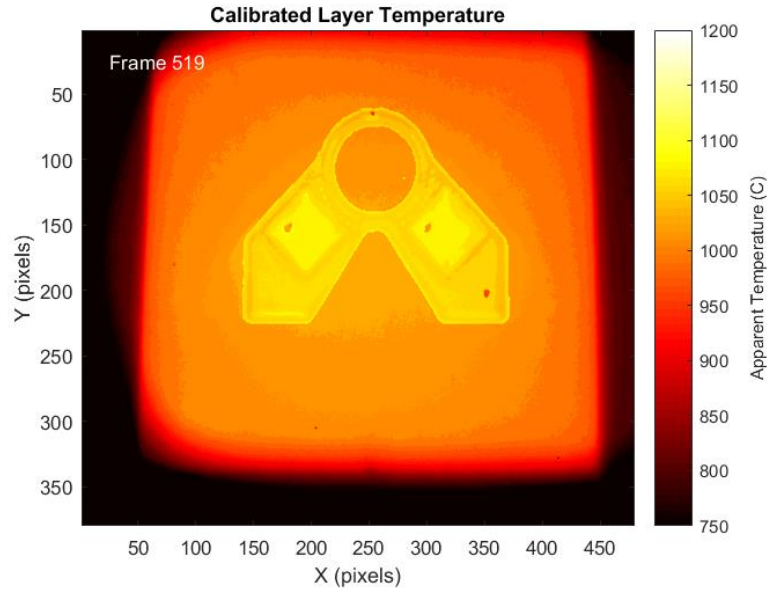


Figure 4-15 Image of the last frame within a layer which should after ample time has passed for the material to come to a uniform temperature. The two regions that seem to have higher temperature are areas that had undergone spot melting.

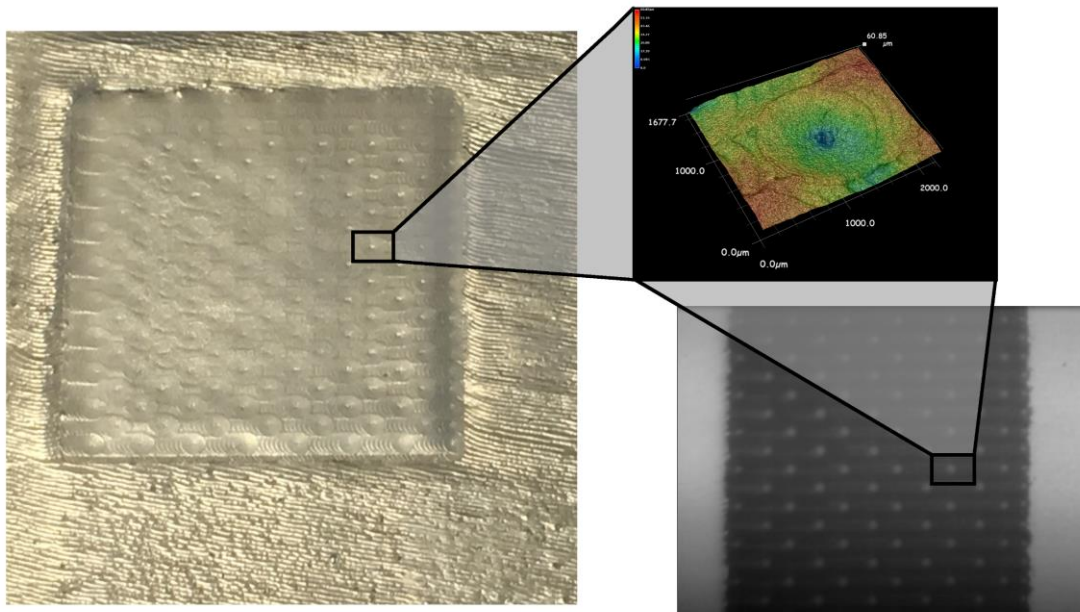


Figure 4-16 Image of a surface that had undergone a point melt scan strategy (left) and IR video of another region that had also undergone the same scan strategy (bottom right). The 12x magnification surface height image taken of an individual surface spot reveals a small depression in the surface that could act as a radiation intensifier (top right).

similarly to the EBM process and help to give insight into the resulting thermal history of SLS developed parts.

Chapter Five: Microstructural Monitoring of EBM AM materials

As stated in the previous chapter, a portion of the following work included in this chapter has been published in the Scientific Reports article “Thermographic Microstructure Monitoring in Electron Beam Additive Manufacturing” produced by Raplee et al. [65]. The work presented in this chapter has been modified and revised to expand on new results collected as well as the results presented in the published work. Therefore some of the figures and results included are taken from that work. My primary contribution as the first author for this work included: (i) development of the problem and reviewing all relevant scholarly works, (ii) developing the methods discussed, (iii) analyzing and discussing the data collected, and (iv) writing the article. As the first author, I retain the rights to reproduce the contribution as part of my thesis per the ownership of copyright given by Nature Publishing Group.

The material in this chapter will discuss the data analysis performed to monitor the development of the underlying microstructure within a material, specifically, the formation of columnar and equiaxed microstructures within parts built using Inconel 718. This chapter will address this topic by describing the measurement and calculation methods (5.2), and the results (5.3) obtained from two different monitored builds and compare these results to the actual corresponding microstructures produced (5.3). Section 5.4 will discuss accuracy effects due to camera resolution, changes in the surface topology and discuss proposed methods for increasing this precision. Again the material covered in this chapter was obtained using analysis of two EBM builds; however, the material referred to in this section is readily applicable to the SLS process because the measurement of thermal data is relevant for all metal AM applications, and transitioning the methodology between the two would require only slight modification.

5.1 Data analysis for microstructural monitoring

The IR data collected must first be corrected to give the most accurate temperature profile of the material (Chapter 4) and all for the proper calculation needed to understand the columnar-to-equiaxed transition of the monitored material. With this information, the thermal gradient and solidification velocity (the rate at which the solidification front progresses along the material) are readily determined with just a few general calculations. These values allow for an understanding of the solidification conditions that govern the development of microstructures within the material. Obtaining a linear approximation of the temperature

differences formed between adjacent pixels will give an approximate measure the thermal gradient of a location within the build. These temperature differences can quickly be achieved for the in-plane directions that are seen in the image using equations 5.1 and 5.2. In these equations x and y represent the current pixel's indexed location within the plane and T_x , T_y , T_{x+1} , T_{x-1} , T_{y-1} , and T_{y+1} refer to the temperatures of the current pixel location and adjacent pixels in each direction during the frame where melting occurs.

$$\Delta T_x = \frac{|T_x - T_{x-1}| + |T_x - T_{x+1}|}{2} \quad 5.1$$

$$\Delta T_y = \frac{|T_y - T_{y-1}| + |T_y - T_{y+1}|}{2} \quad 5.2$$

These equations produce relative temperature differences, but the distance between pixel centroids are needed to obtain the actual thermal gradients within the material. Using a scale derived from a feature of a known dimension in ImageJ digital image analysis software, these distances are easily measured and collected for the build, assuming that the known measurement used was accurately printed within the build [71]. For the results discussed in Section 5.2, these measured values were 350 μ m and 370 μ m per pixel in the x and y directions for the first monitored build and 320 μ m and 350 μ m per pixel in the second discussed build. Also of note is that since the camera is unable to be mounted perpendicular to the build plate due the mounting procedure used, there is a slight variance in the in the pixel to pixel distance as a function of the pixel's location. This variance causes the instantaneous field of view at the extremities of the build to change by about 14% from measurements taken at the center. However, this distortion was not corrected for the presented results, and the amount of error that this would produce is negligible to the error created due to the inability to resolve the beam size and its exact location because the melt pool is much larger than the size of a pixel. With the pixel to pixel distance and the relative temperature differences determined, the estimated thermal gradient for each pixel can be determined using Equation 5.3 from Raghavan et al. where the values of T_x and T_y are the results of Equations 5.1 and 5.2, and the values of Δx and Δy are pixel-to-pixel distances for each respective direction [46].

$$G(K/m) = \sqrt{\left(\frac{\Delta T_x}{\Delta x}\right)^2 + \left(\frac{\Delta T_y}{\Delta y}\right)^2} \quad 5.3$$

Of note is the absence of the temperature gradient in the build direction, z, however since the use of IR thermography is limited to the temperature of the material's surface the thermal gradient in beneath the surface cannot be accurately assessed.

With the thermal gradient calculated for every pixel within the image the resulting values can be used to determine the value of the solid-liquid interface velocity as well by dividing the cooling rate of the temperature curve that occurs directly after the melt, $\frac{dT}{dt}$.

$$R(m/s) = \frac{\left(\frac{dT}{dt}\right)}{G} \quad 5.4$$

In the results presented in this work, the cooling rate of the material was determined by finding the slope of the temperature curve over the three frames following the transition from powder to the as-printed material.

With the solidification parameters calculated for every pixel within the build geometry for every layer, the trends in these values can be used to observe the microstructure development within the build. All of the results for the monitored build were recorded and saved to be used for data visualization.

5.2 Results of the thermal gradient and solidification velocity calculations

The thermal gradient and solidification velocity of the material can be readily discerned for each layer using the previously described calculations. As seen in **Figure 5-1**, the regions of the build that underwent spot melt scan strategies exhibit much lower thermal gradient values and higher solidification velocities when compared to the regions that underwent a line melt scan strategy. By taking the ratio of these values (G/R seen in **Figure 5-2**), it is possible to quantify the existence of columnar or equiaxed microstructures following solidification. Although there is a discernable difference between regions that use different scan strategies, there is a significant amount of noise and inconsistency within the data. To better visualize the difference between these two regions, a histogram of the G/R values (**Figure 5-3**) was created showing the distributions for each scan strategy, which shows a clear

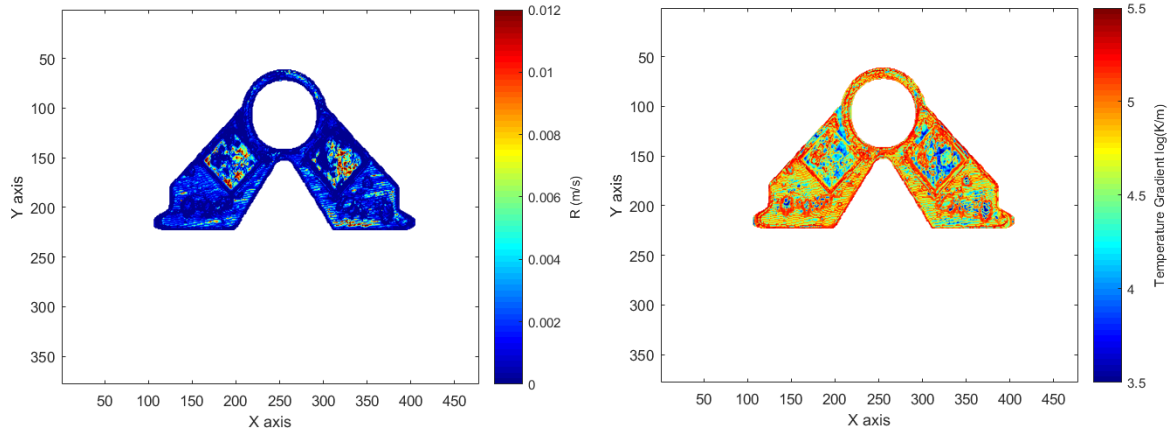


Figure 5-1 Maps of the calculated solidification velocity (left) and thermal gradients (right) of a single layer within a build. The square regions that had undergone spot melting show lower thermal gradient values and high solidification velocity when compared to rest of the part. These plots show a high amount of noise and any large regions coincide with regions where issues with the temperature calibration would occur, and the small more random noise occurs due to the lack of resolution and frame rate for the imaging.

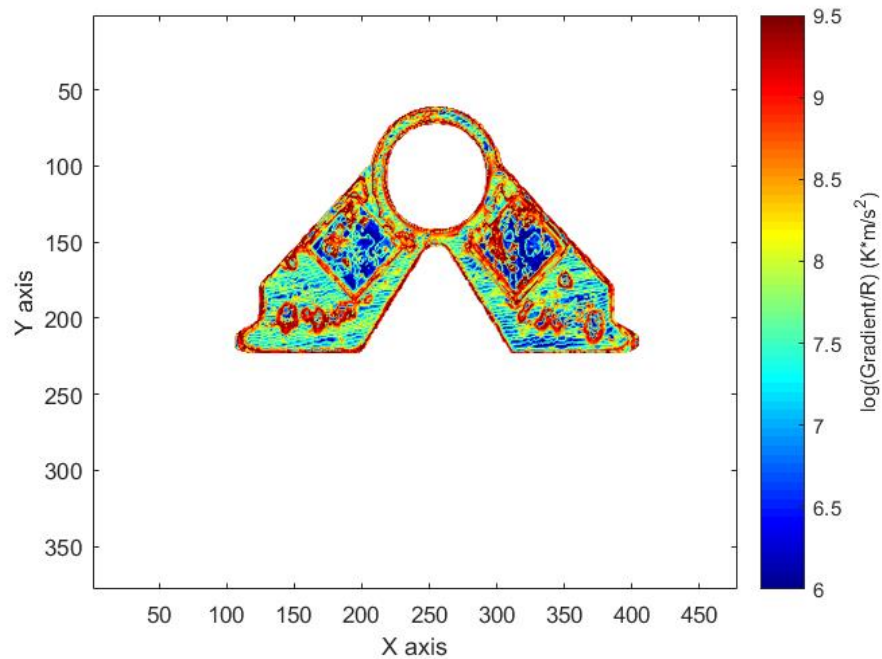


Figure 5-2 Map of G/R measured for the layer shown in Figure 5-1. This value gives a single representative value that can be used to determine columnar or equiaxed microstructure within the material.

difference between the different scan strategy regions with the peak values of the two different regions falling about three orders of magnitude away from each other. The distributions generated for each scan strategy are bimodal therefore there is a significant amount of overlap between them, which is a result of the noise within the data collected. Despite the overlap, there is a clear divide between the majorities of these G/R values; with the majority of the point melt regions having a low G/R representative of a columnar microstructure, and the raster scan region has a much higher G/R value indicating an equiaxed microstructure. These values can be reconstructed in a three-dimensional space through the mapping of the values for every 1mm within the build (**Figure 5-4** and **Attachment 2**) to show the overall development of the microstructure within the build with specific colors representing the approximate microstructure of the part. Further analysis shows that in the regions where the material was deposited on top of the supports, and the powder below the difference between the columnar and the equiaxed microstructure regions is harder to discern. The error in the results could be due to changes in the thermal properties as a consequence of building directly over the low conductivity powder or error produced by issues in the temperature calibration process, and further testing is needed to determine the cause. For most of the build, the data collected is consistent with the expected microstructural results and were later confirmed through electron backscatter diffraction, discussed in Section 5.3.

In the second observed build, the two cubes that underwent data analysis for microstructural development both utilized the point melt scan strategy. The difference between the cubes is that the distances between the points being melted were changed to increase the thermal gradients between adjacent melt pools. The analysis shows that the difference between the two cubes is evident with less noise and error and when compared to the previous build, but there is still noise within the material. In the cube with broader point-to-point distance, the thermal gradients were much higher, and solidification rate was much lower when compared to the cube of lower point-to-point distance as expected. In the closer point-to-point spacing cubes attempting to induce equiaxed grains also follow the expected solidification parameter results, except regions in the center of the cube showing a drastic difference from their surroundings (**Figure 5-5**). Upon inspection of the cube and subsequent confocal microscopy measurements of the build, this region of error happens to coincide with an area where material swelling occurs (**Figure 5-6**). Localized topology changes such as this will change the relative angle between the surface and the camera sensor; this will cause a

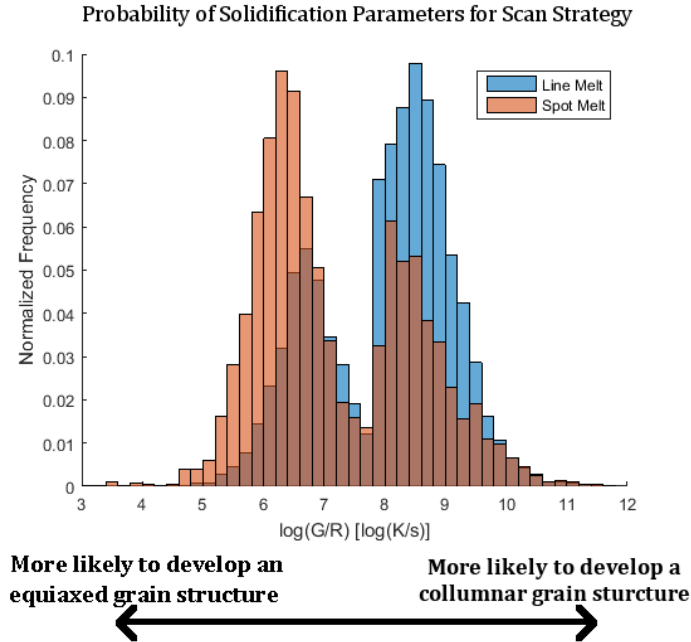


Figure 5-3 Histogram showing the distribution of G/R values for the different scan strategies used within a layer of the build. The two histograms show a clear difference between the two with each showing a bimodal distribution.

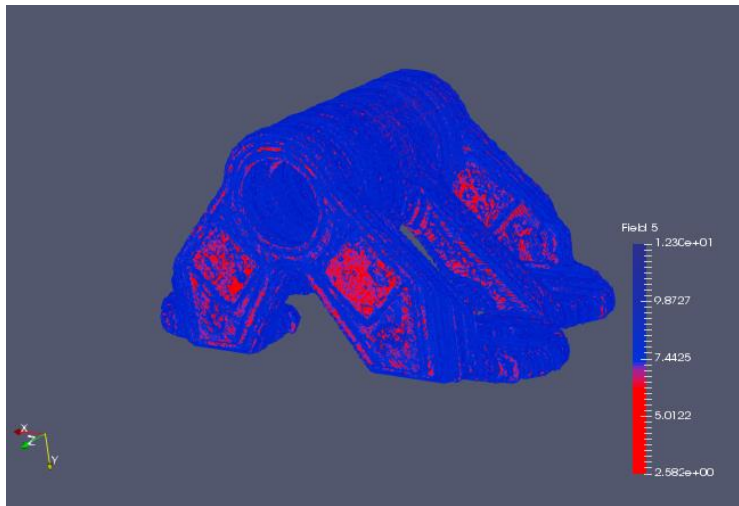


Figure 5-4 Compiled G/R data for the entire monitored build. The regions in red are expected to exhibit equiaxed microstructures, and the blue is expected to exhibit columnar. The noise in the data has carried over to the data collected showing that continued refinement is needed, but the technique shows promise.

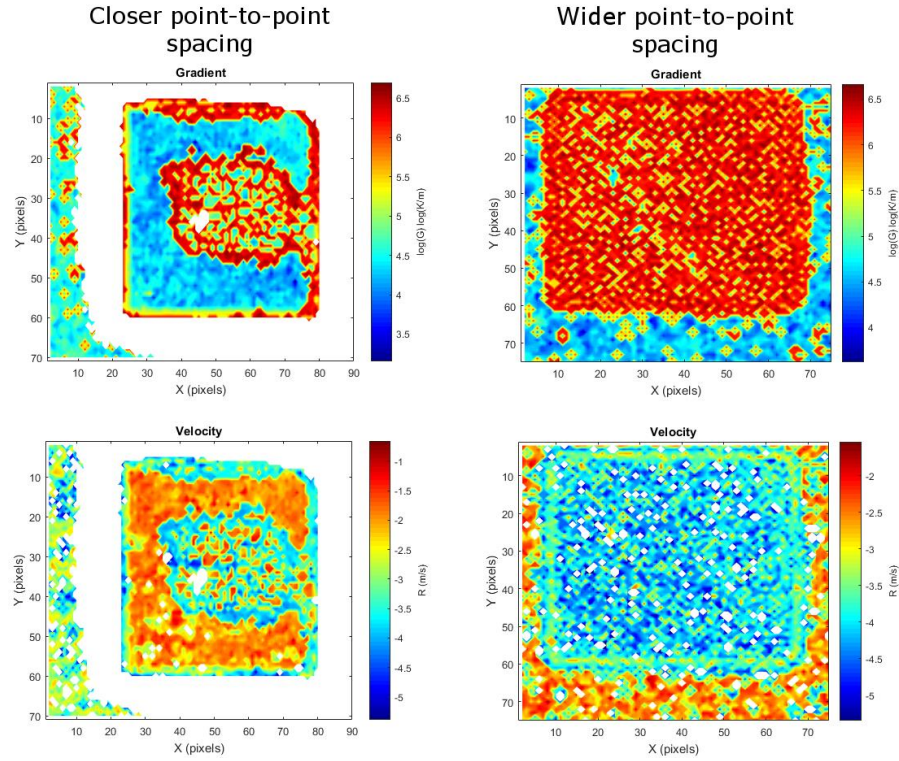


Figure 5-5 Thermal gradients and solidification velocity maps for the second observed build. Both cubes follow what was expected except for the regions on the closer point-to-point region where that seem to show a drift toward high thermal gradients and low solidification velocity. Also, of note is the powder bed showing through on the outer edges of the build, these appear because the calibration tool was unable to detect the melt regions accurately, and subsequently needs adjustment.

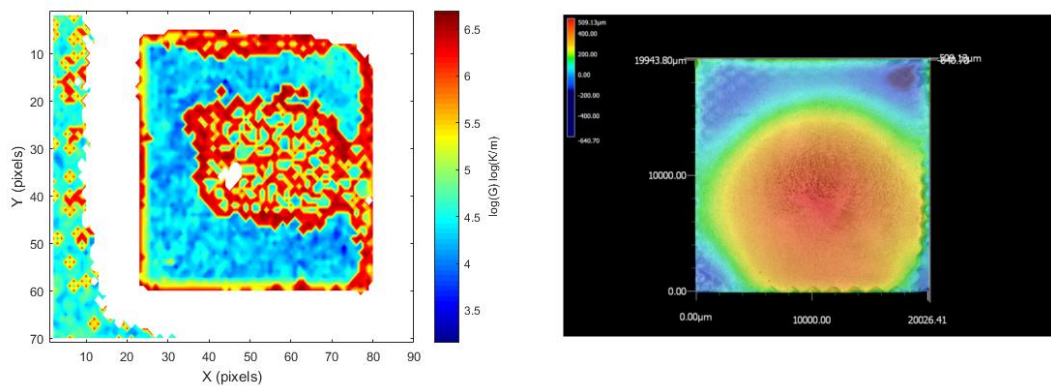


Figure 5-6 Image showing the thermal gradients of the closer point-to-point space cube (left) and a surface height map taken at 25x magnification of the cube following the build (right). As seen in the image, the region where the gradients deviated are located where the highest location swollen region and the slope that would be sloping away from the camera.

change in the relative intensity even though the material should be at a uniform temperature (**Figure 5-7**). This relative change could cause increases in the thermal gradient values, especially surrounding the swollen region where the change is most dramatic. Also, as mentioned in Chapter 4, these regions will cause issues to occur within the temperature correction algorithm used to obtain the correct temperature of the material which may cause the calculated gradients to utilize the wrong time frame which could increase the relative thermal gradients as well. To correct this issue, a separate correction method that can adjust for these localized changes in material emittance to further correct the temperature would be needed to ensure that the outcome is as accurate as possible. Also, the data collected along the upper edge of the cube shows parameters that signify columnar grain growth. This location is where the scan strategy begins the melt and is surrounded by more insulative material. The presence of these solidification parameters was later determined to be accurate to the underlying microstructure and will be further discussed in Section 5.3.

Overall, the results collected show that, for the most part, there is a quantifiable difference within the thermal histories of regions that are attempting to achieve columnar or equiaxed microstructural development, and this difference is detectable using IR imaging. However, a connection to the material's actual microstructures needs to be understood as well before any conclusions can be drawn about the efficacy of the measurement tool and the effects of other build dependent phenomena such as support structure placement and geometric changes need to be understood as well.

5.3 Microstructural observations and correlation to thermal data

As mentioned previously, the established understanding of competition between columnar and equiaxed grain growth is that, when thermal gradients are low, and solidification velocity is high, undercooling allows for crystal nucleation to occur in front of the solidification front permitting the formation of equiaxed grains [43]. When the opposite is true, the material will form columnar dendritic growth along the preferential growth direction ($\langle 0\ 0\ 1 \rangle$), the build direction within PBF processes, resulting in highly textured columnar structures reminiscent of directionally solidified castings.

Microscopy and EBSD imaging were performed on several sample specimens that were collected from the monitored EBM builds to validate that the thermographic data collected for the material correctly monitored the microstructure development of the grain structure. In

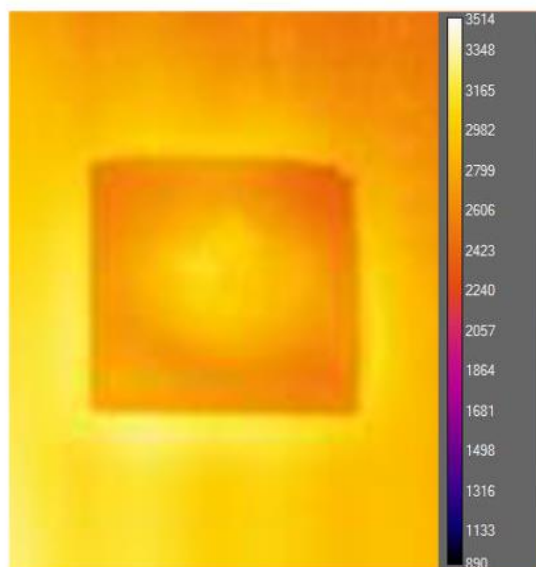


Figure 5-7 Image showing the representative change in IR signal created by the swollen surface of the material. This swelling is the assumed cause of the change in thermal gradient seen in **Figure 5-6**.

data gathered from the bracket, a distinct difference in the microstructures can be seen in the regions that employed a spot scan strategy rather than the traditional line scan methods (**Figure 5-8** and **Figure 5-9**). In the micrographs seen in **Figure 5-8**, the regions that undergo line scan strategies that create large thermal gradients and slow solidification velocities display coarser dendritic spacing. EBSD imaging (**Figure 5-9**) also shows large (~1mm), highly-textured grains aligned with the preferred growth direction along the build height as expected. In the regions that undergo the point melt scan strategy, the microstructure is dramatically different with dendritic growth unable to be easily identified and small, randomly oriented grains seen in the EBSD data. These observations are consistent with what is seen in the literature for these scan strategies, showing that utilizing this methodology to quantify the microstructure of a build is viable and that with further development of thermographic could be a handy quantification tool used to validate a build's viability in-situ [48], [49]. As mentioned in the previous section, the material does show a change in the values for the calculated thermal gradient and solidification velocities. However, this change is not seen in the EBSD results in **Figure 5-10**, which shows an equiaxed grain structure from the region that underwent point melting that transitions to a columnar structure within the line scan regions. These measurements confirm that further error analysis needs to be performed to understand the cause and develop methods to increase the accuracy of the data collection.

For the second observed build that compared differing point-to-point spacing for a point scan strategy, the material observed showed differing thermal conditions that were indicative of changes in columnar or equiaxed microstructure as well. The material that had increased point to point spacing which showed increased thermal gradients and low solidification velocity, the material exhibits the same columnar structure seen in materials that are produced using the line scan strategy due to the increase of thermal gradients within the material. The cubes produced using a reduced point to point scan strategy exhibits the small randomly oriented grains that are expected from the spot melt scan strategy seen before from the literature due to the formation of a large melt pool decreasing the thermal gradient and increasing the solid-liquid interface velocity [49]. However, as previously mentioned the data collected for this strategy shows deviation at top face and right face of the material that indicates columnar grain growth. The EBSD data gathered for this material also shows the diversion in the microstructure from equiaxed grains to columnar grains at the upper edge of the part due to the change in the thermal conditions affecting the thermal gradient measure as

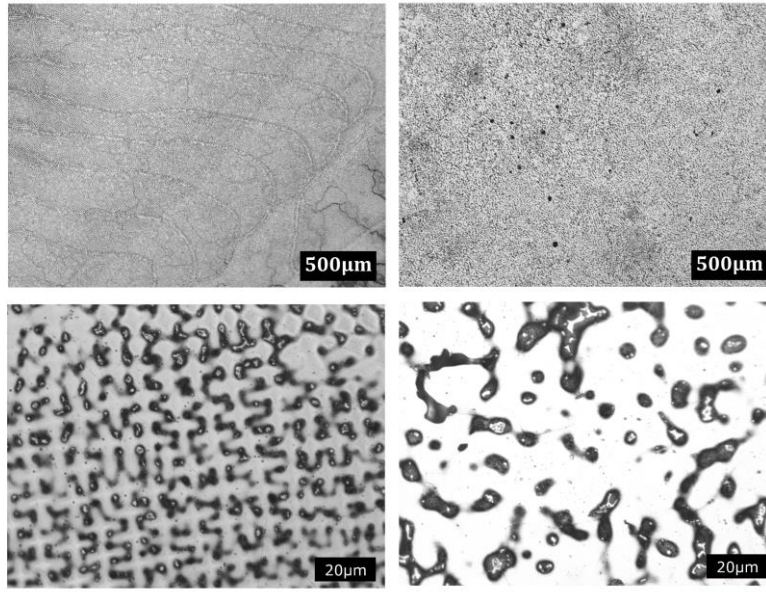


Figure 5-8 50x (top) and 1000x (bottom) micrographs of the two observed scan strategy regions. The point melt region (right) shows no discernable dendritic structure, and the line scan region (left) shows coarse dendritic spacing which is characteristic with columnar grain formation. [52]

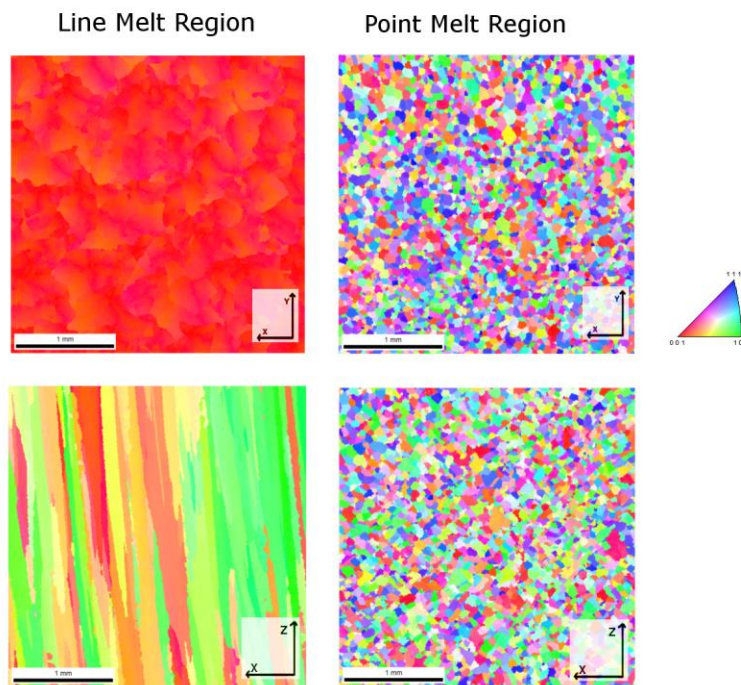


Figure 5-9 Electron Backscatter Diffraction (EBSD) imaging reveals the columnar (left) and equiaxed (right) microstructures for the line melt and point melt regions. [52]

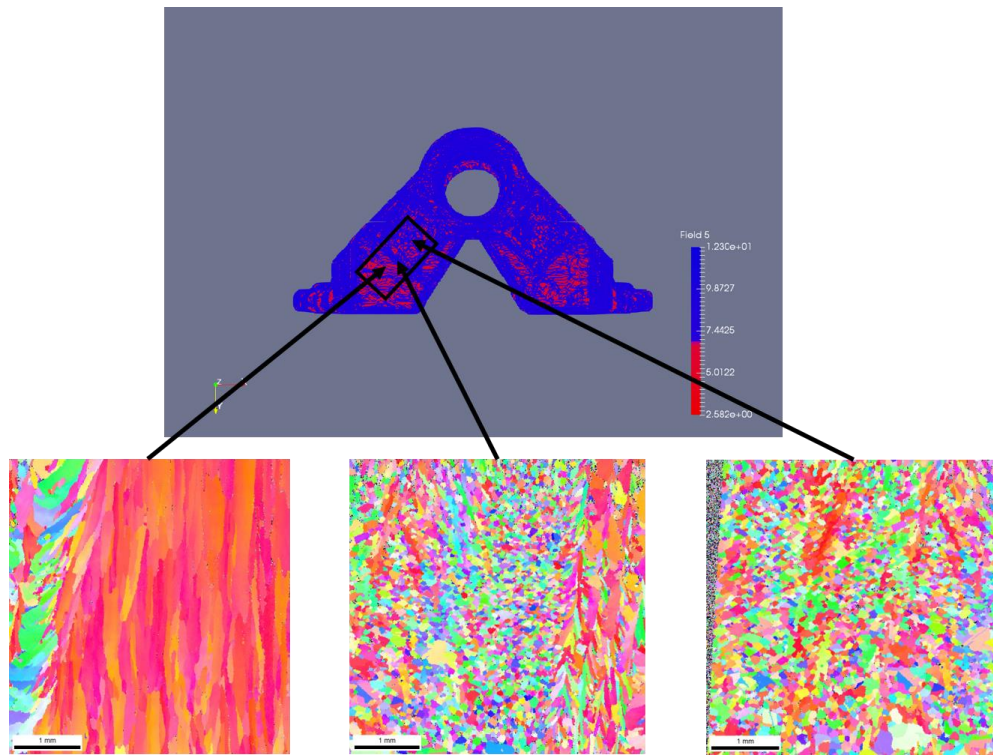


Figure 5-10 EBSD images collected from the bottom of the build showing that although the thermography data collected during the build has a harder time differentiating between columnar or equiaxed microstructure in the material, the intended transition did occur.

well (**Figure 5-11**). These results show the sensitivity and accuracy of the measurement process and its ability to measure the thermal conditions that control the microstructure, but further work is needed to increase its accuracy and capabilities moving forward.

5.4 Limitations and further developments

As mentioned before this technique is highly sensitive to and limited by the relative spatial and temporal resolution of the camera setup used to monitor the build. Much like the temperature correction algorithm described in Chapter 4, the lack of the ability to determine the beam size and movement across the surface of the material can affect the resultant calculations of the cooling rate, thermal gradient, and solidification velocity. These errors could dramatically affect the calculations given (**Figure 5-12**) due to the inability to detect the actual thermal gradient and solid-liquid interface velocity and thus are assumed to be the cause of the noise and error generated in the resulting data, often causing the values to be much lower than they should be. Also, as mentioned in the previous chapter, the lack of a liquidus temperature calibration means that the data collected here is still relative temperature data despite having a quantified calibration applied. High-resolution data needs to be collected to detect this region and then adequately allow for a method to correct for this new change in emissivity needs to be determined, and the necessary calculations need to be modified to ensure higher accuracy.

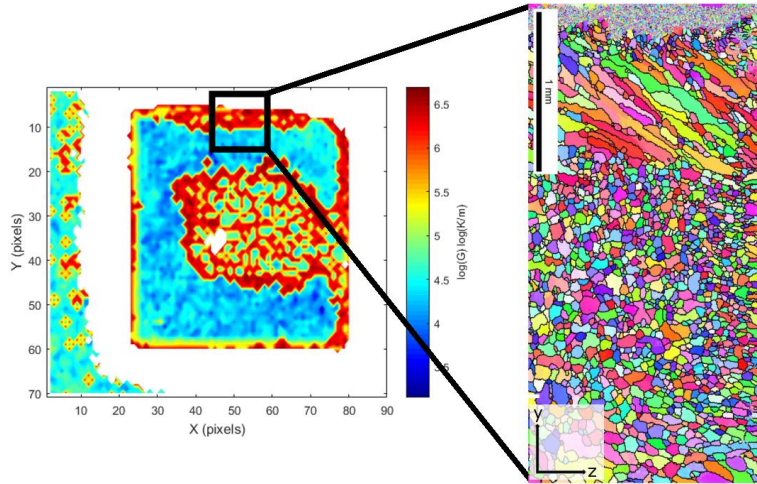


Figure 5-11 EBSD data collected for the build shows the change from equiaxed to columnar material at the upper edge of the build, confirming the resultant data collected for the build. This work was completed in conjunction with the work provided in Raghavan 2017. [72]

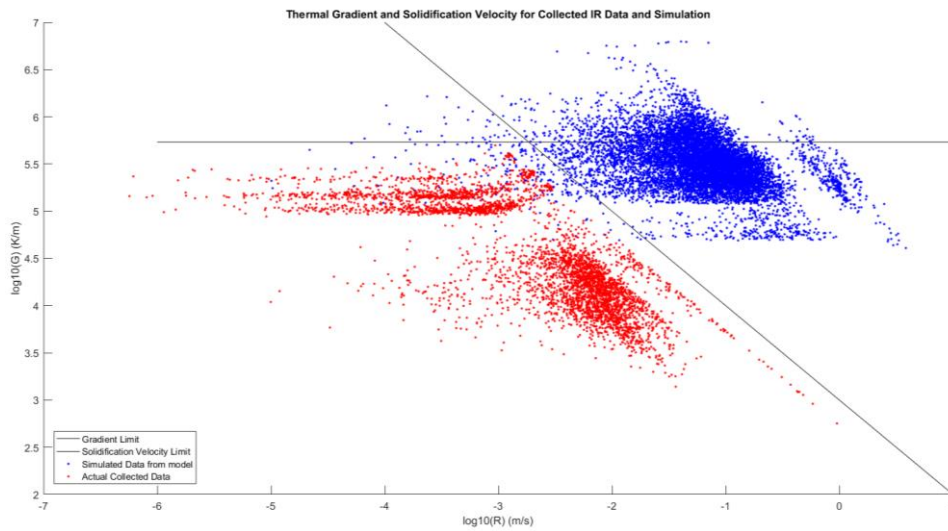


Figure 5-12 Resolution limits of a camera operating at 10Hz with an IFOV of $370\mu\text{m}$ like that seen in the first experiment (Chapter 4). Also in the graph is G and R data collected for the actual IR data and a semi-analytical heat transfer model. The data in blue was obtained using the semi-analytical heat transfer model shown in Plotkowski et al. [73].

Chapter Six: Porosity Monitoring of SLS AM Materials

This chapter will discuss the application of IR thermography as an in-situ method for determining porosity during the build process. This chapter will discuss two different experimental approaches taken towards developing a method to detect porosity both in the bulk material and through location-specific detection along with their respective results and limitations

6.1 Monitoring bulk build porosity

The first section of this chapter will discuss the first build produced in order to understand a method for characterizing a material's bulk porosity resulting from changes in process parameters. The builds created were large matrices of small cubes that were built using varying processing parameters attempting to intentionally induce porosity, as well as changing the build conditions to increase the powder bed conductivity and measure the effects, as described in Chapter 3. This experiment's intent was to determine a relationship between changing build conditions and the resultant IR data collected as a means of understanding the formation of porosity within a build.

6.1.1 Data analysis

In order to determine porosity characteristics within a build, high-speed (742 Hz) data was collected for two series of Ti-6Al-4V cubes built under varying processing conditions. Once all of the data was collected, it was processed and truncated to remove the data from the powder bed and the edges of the cubes, resulting in twenty-five 25x25 pixel region dataset for each of the 33 layers monitored for the build. By removing the unnecessary data, these small pixel regions could more easily be processed. Each pixel within these data sets was processed to obtain statistical and numerical values, including:

- The standard deviation of the intensity data collected for the pixel
- The minimum and maximum intensity values
- The average intensity value of the IR signal
- An exponential curve fit (Equation 6.1) to the data that occurs after the melt to attempt to determine signal decay rates, t_0 .

$$I = Ae^{t/t_0} + I_0 \quad 6.1$$

- Numerically integrating the area under the Intensity curve from about 1.5 seconds before to 1.5 seconds after the melt to give a single representative value of the entire melting process. Henceforth referred to as integrated intensity.

The curve fits created were not very robust, (R^2 values ranged from about .6 to .9) but the intent was to get a qualitative understanding of how a specific parameter set compared to others regarding signal intensity and decay rate (**Figure 6-1**). The intention was to draw correlations between changing process conditions, whether controlled or not, to porosity within the build. Data maps of each cube for every monitored layer were created for the maximum intensity, decay rate, and the integrated intensity (**Figure 6-2- Figure 6-4**) each showing minute differences within the cube surfaces. By taking the average value of each layer within these maps and plotting over the height of the build, general trends of these values for the cubes can be seen throughout the build (**Figure 6-5- Figure 6-7**). The data could then be further analyzed and compared to the porosity measurements collected from optical microscopy for the builds to determine correlations between the IR data and the development of potentially critical defects.

6.1.2 Analysis results and discussion

Upon performing analysis, several interesting trends became apparent. As can be seen in (**Figure 6-5 - Figure 6-7**) the general trend was that for the first few monitored layers of the build (fabricated over support material and powder), the material would retain a significant amount of heat. This retention of heat would result in substantial values for the integrated intensity curve (>20% greater than the average surface values) and extended decay rates (up to 5x longer). As the build progressed, these values begin to decrease. In the first build, all of the data collected seems to decrease to an asymptotic value except for the maximum intensity, which has several peaks in the data as seen in **Figure 6-6**. This increase in maximum intensity values correlates to layers that used a scan strategy that included small melt regions where the intensity will increase significantly due to apparent overheating. In these regions, the shorter distance of the raster paths will cause these areas to obtain much higher intensity values than they would typically achieve with a longer raster path, increasing the average value of the layer (**Figure 6-8**). In the second observed build, the data followed a

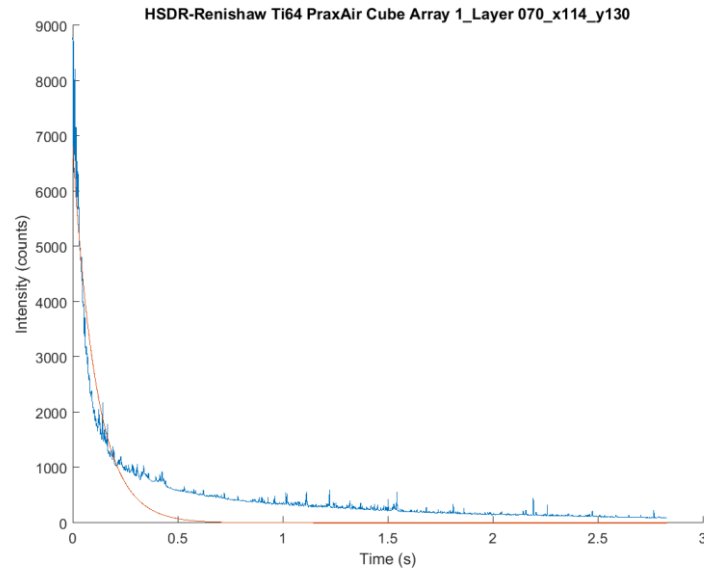


Figure 6-1 Results of a curve fit for the material showing the poor fit. However, it can show the general trend of the curve.

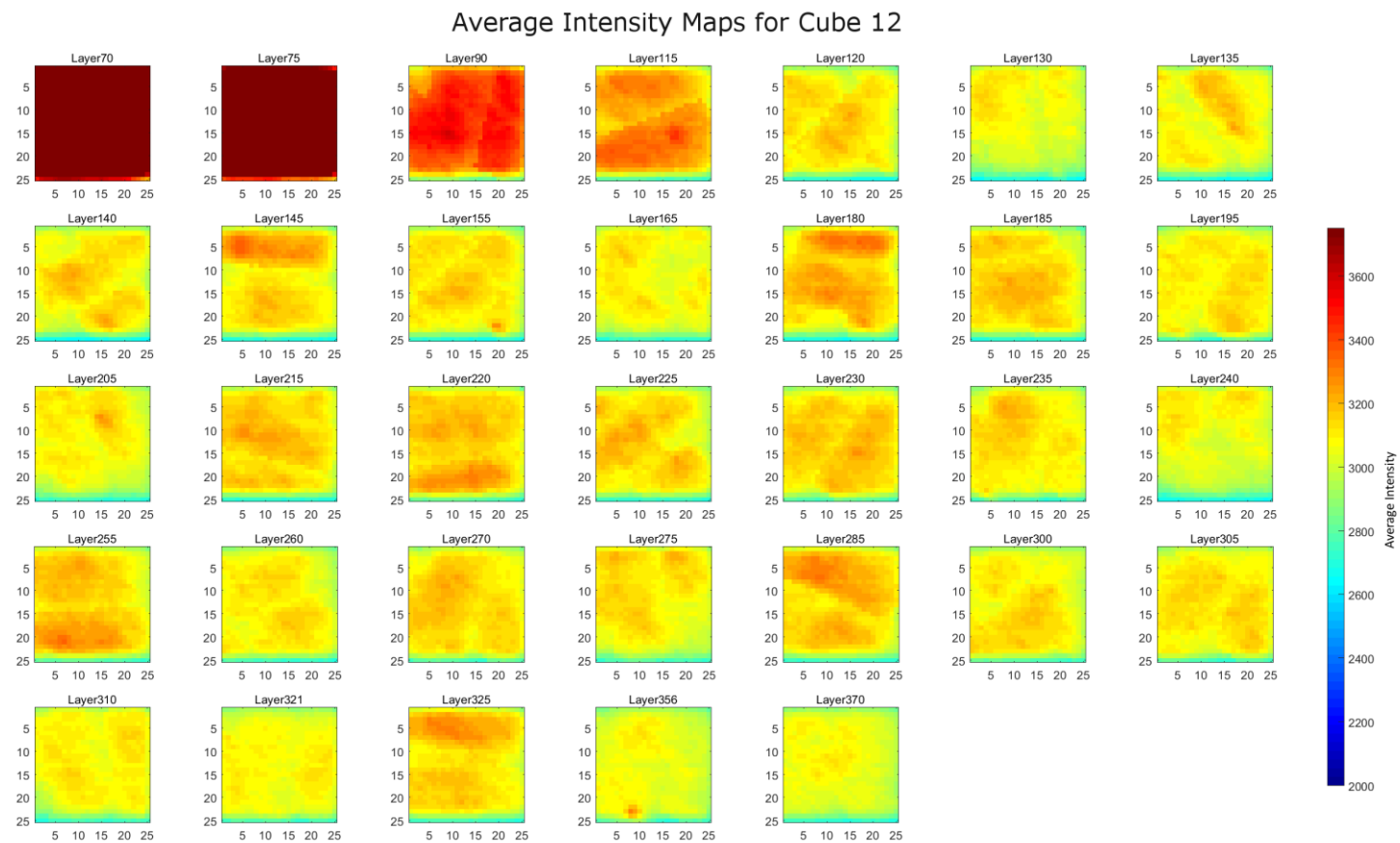


Figure 6-2 Integrated intensity maps of cube surfaces over the course of a build for a single cube. The high-value regions at the lower layer numbers are where the build occurred directly over support material which has a different conductivity than the rest of the build.

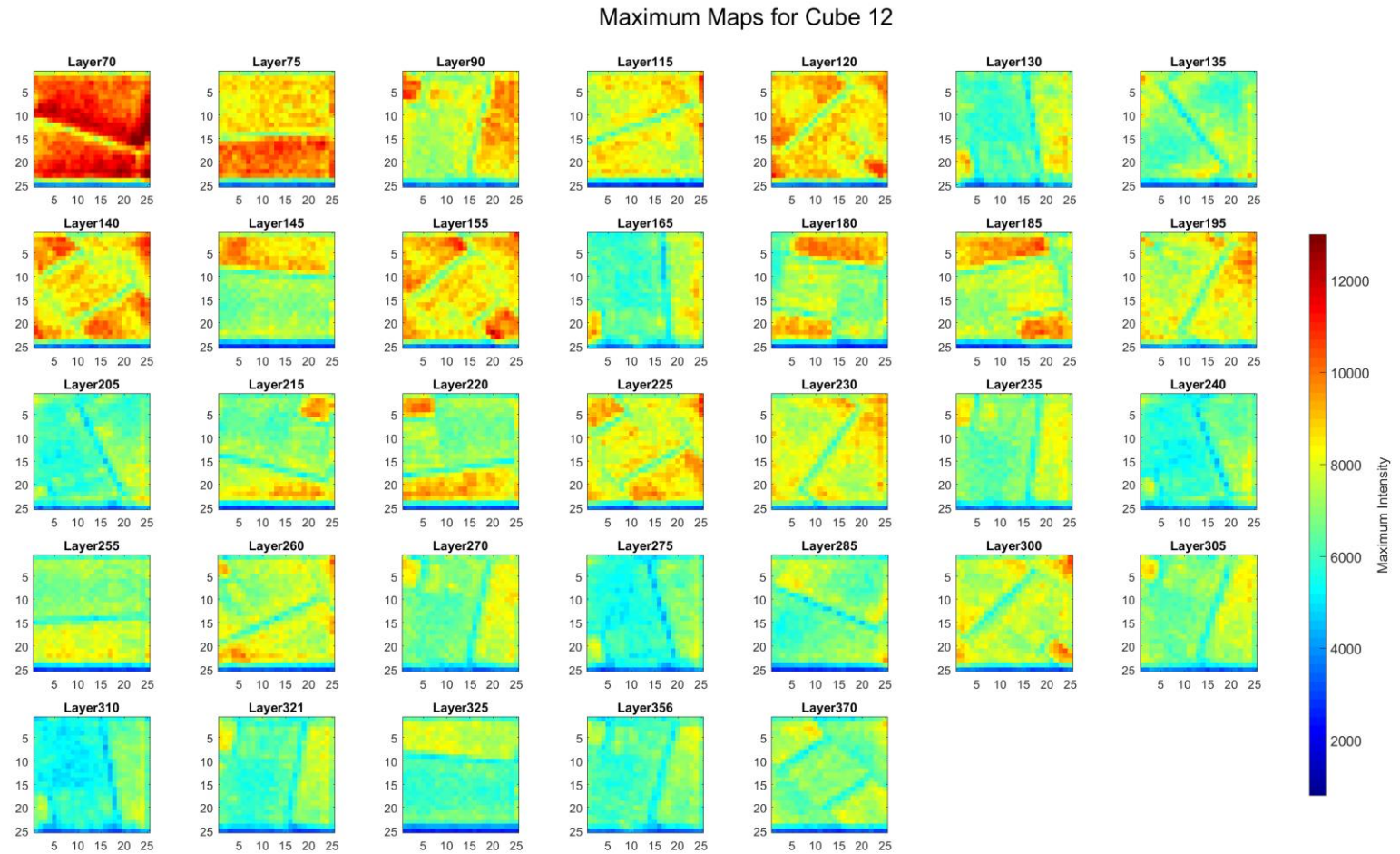


Figure 6-3 Maximum intensity maps of cube surfaces over the course of a build for a single cube.

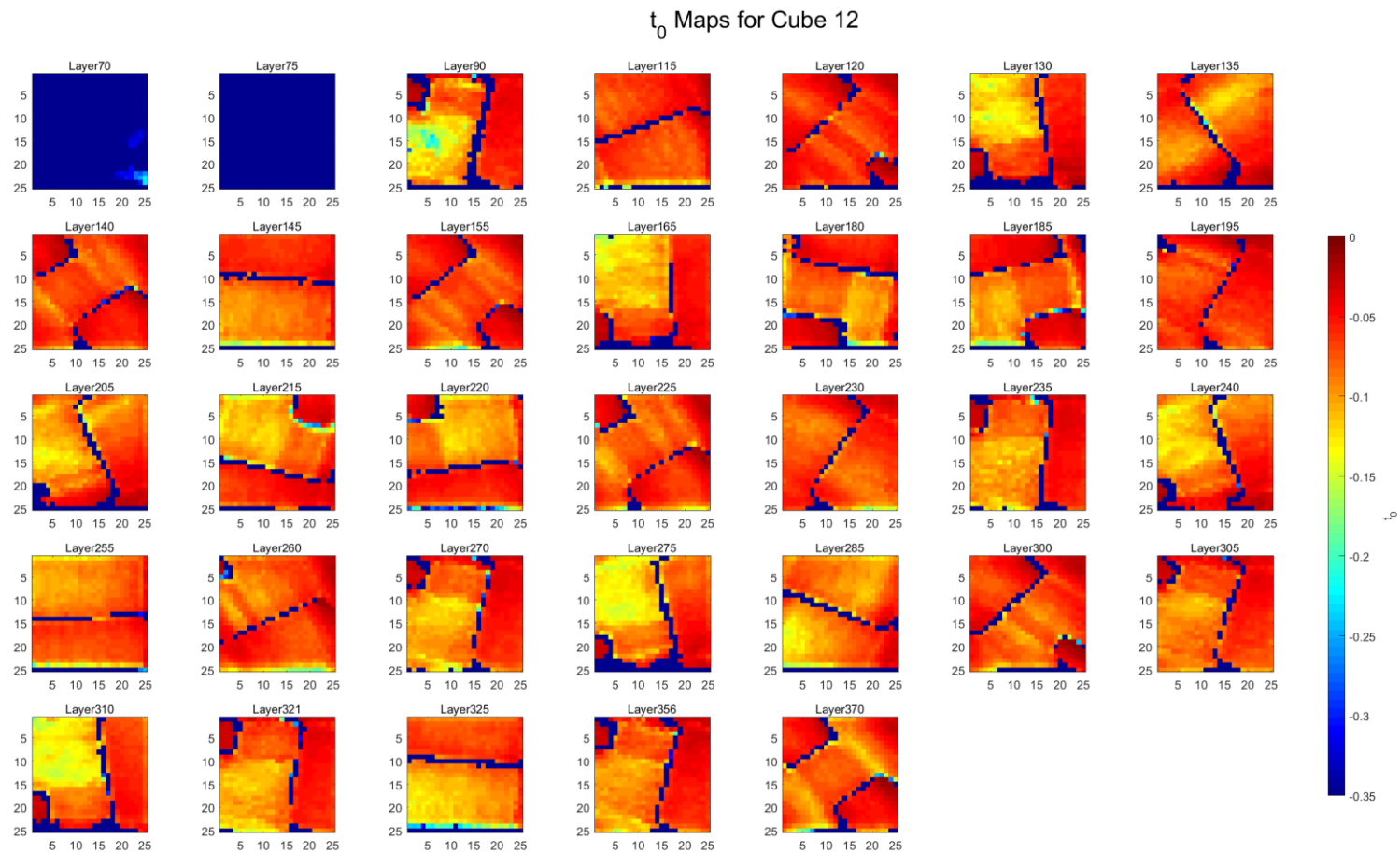


Figure 6-4 Decay rate, t_0 , maps of cube surfaces over the course of a build for a single cube.

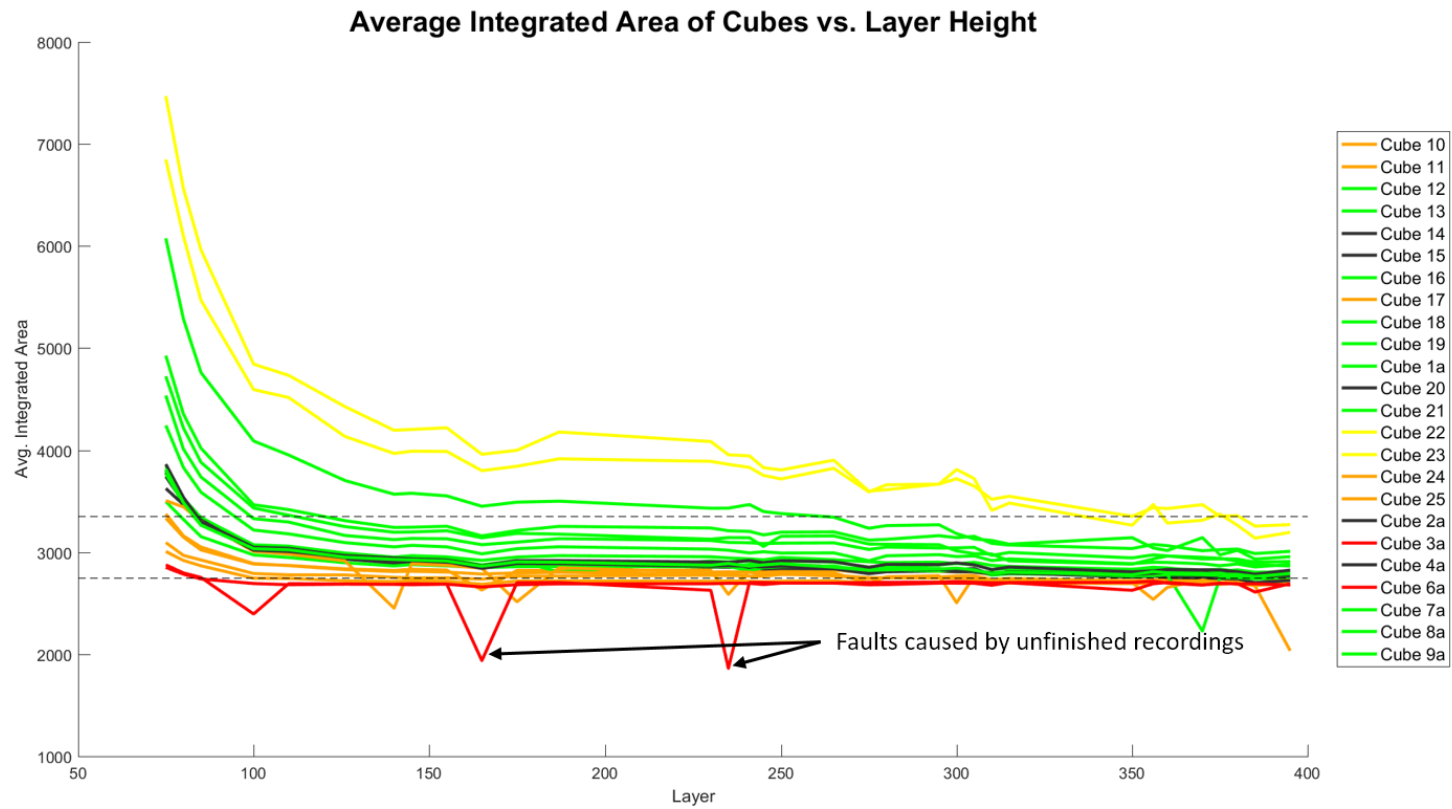


Figure 6-5 Average integrated area as a function of layer height. The dotted lines drawn represent the area that bounds the regions where cubes exhibit full density. The uncharacteristic drops in the data are regions where there are drops in the surface values due to the video not recording the entire melt for the layer.

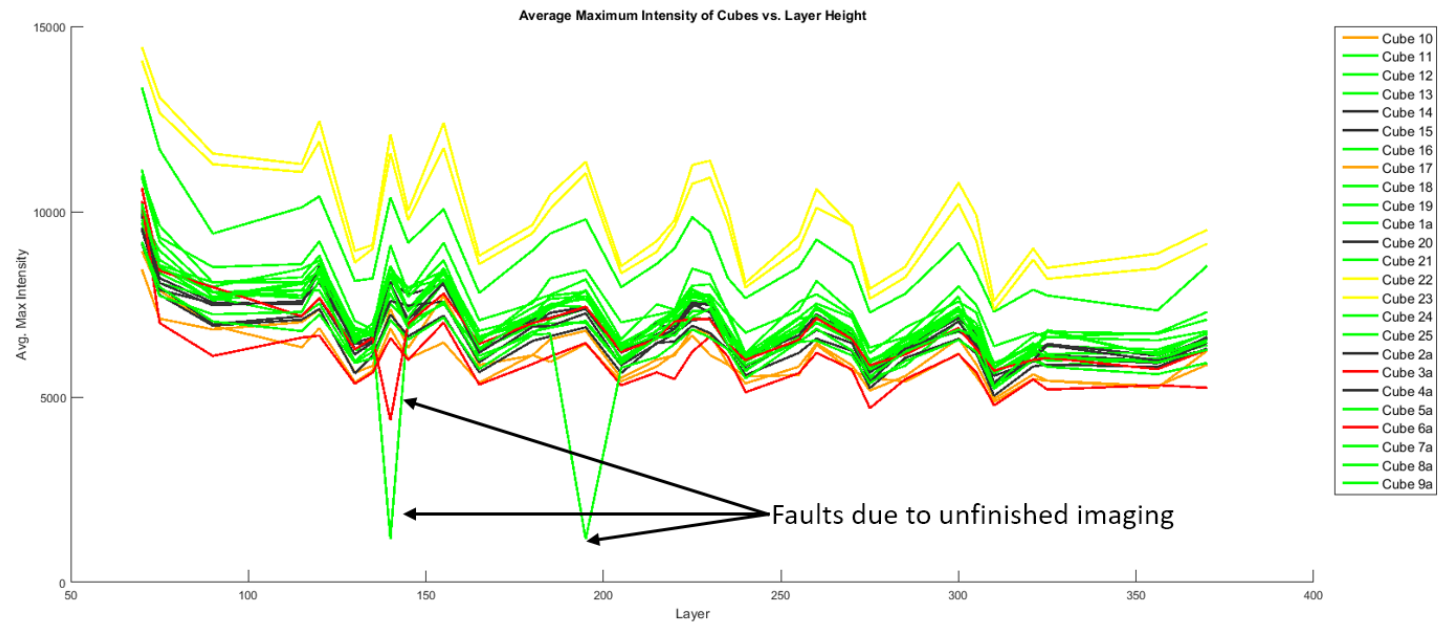


Figure 6-6 Average maximum intensity of cube surface for all of the cubes as a function of layer height. Also of note are three occasions where a cube's value does not necessarily follow the trend of the other layers. Upon review, it was seen that the video files collected for these layers did not record all of the information for these layers, leaving off the time in which substantial regions of these cubes would have been melted.

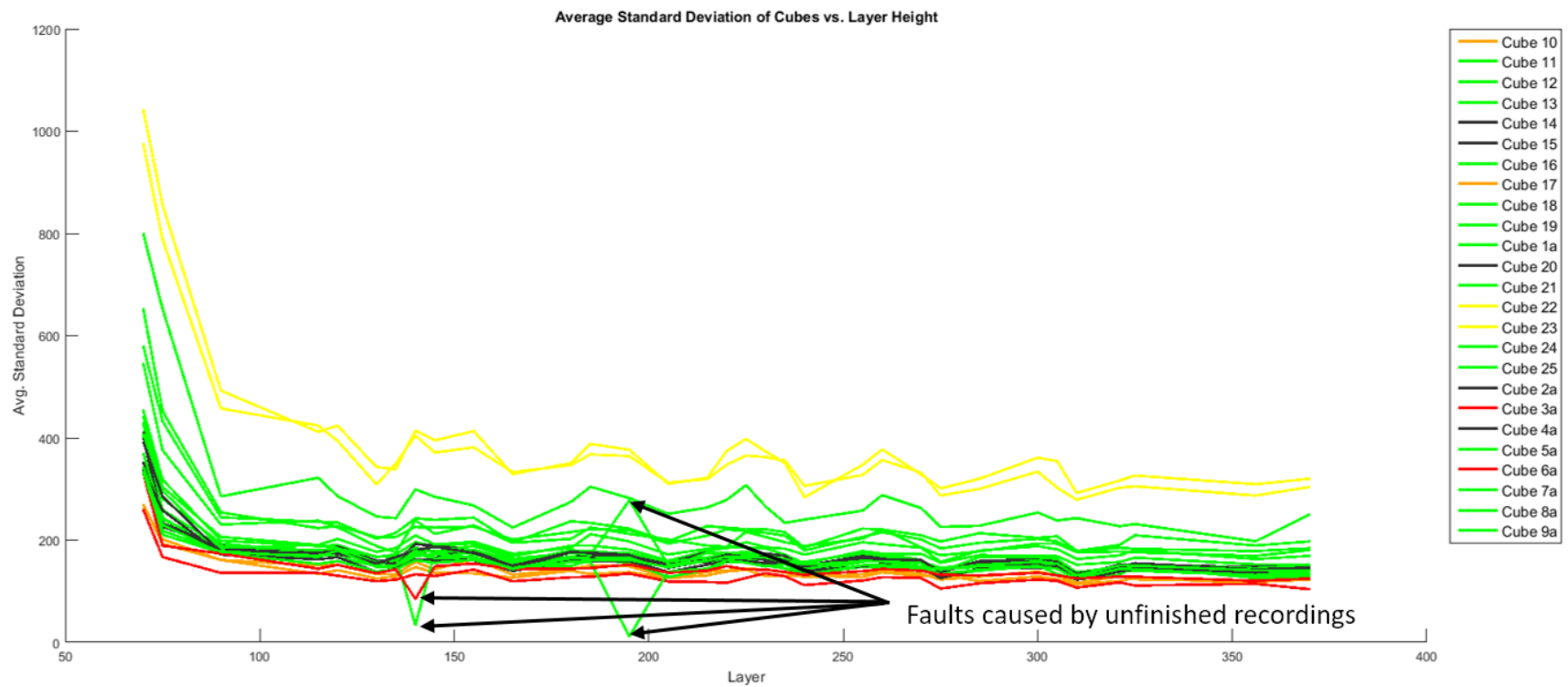


Figure 6-7 Standard deviation value of the intensity of the layers within the build. Again, the regions that show uncharacteristic peaks and valleys are related to cubes that were not correctly recorded in the video.

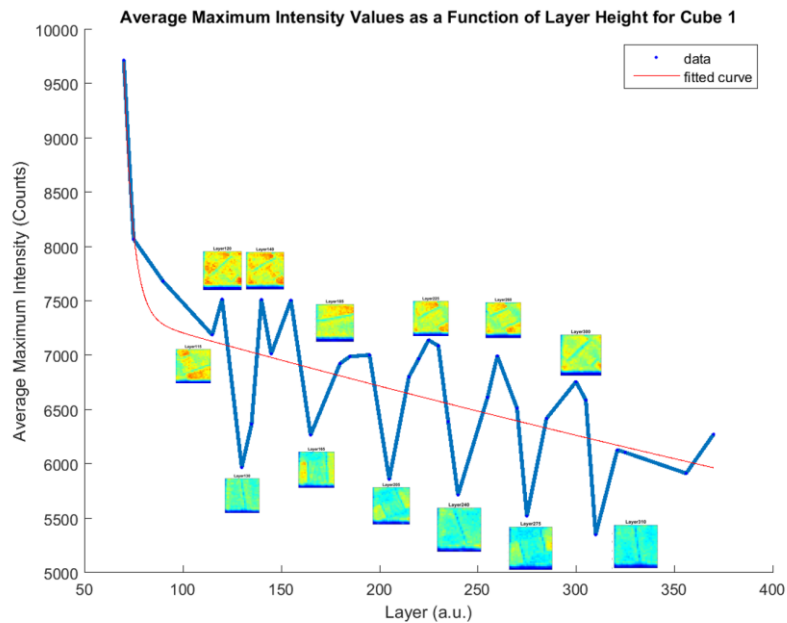


Figure 6-8 Average maximum intensity value for various layers. The additional images near the peaks and valleys of the data show that representative layer's scan pattern and the regions that seem to reach much higher values.

similar pattern but rather than remaining relatively constant; the data continued to decline as the build progressed (**Figure 6-9**). This steady decline in value most likely occurs due to the increased conductivity within the powder bed allowing for heat to leave the material more rapidly as it increases in height when compared to a powder bed of lower conductivity.

Along with these general trends, apparent differences can be seen between the different parameter sets utilized during the build. For every metric given, the values of the cubes that utilized scan strategies that intentionally increased the input energy density were significantly higher than those gathered for the standardized, “best” parameter set (**Figure 6-10**). For the lower energy density builds these values are lower than the standard melt parameters, although the difference between the standardized melt parameters and the lower energy density builds was not as significant as the higher energy density cubes. This difference in the data results shows that the techniques are much more sensitive to high energy density values when compared to the low energy sets of data, and care needs to be taken when interpreting the data.

Other patterns for each measurement metric can be seen in the data maps of the surfaces of the cubes as well. In the maps given for the value of t_0 , the data shows a strong correlation between the line scan length of the beam. Within the same layer of a cube, regions that have longer scan paths show higher decay values, and shorter scan paths show decreased decay rates. Upon inspecting the intensity signal for these regions over time (**Figure 6-11**), it is seen that the longer scan strategies have a lower frequency at which the beam passes, often only registering a one or two peaks before decaying. This change in the frequency causes the decay rate to increase whereas the shorter line scans have higher frequencies which cause the signal to decay slowly. Also of note are the lines of pixels within the maps that have much longer decay rates than the rest of the surface that fall along the boundaries of the striped path that the beam travels along and change direction every layer. The integrated intensity data shows values that tend to be more uniform across the surface with only slight gradation in values in regions where the scan length of the material lengthens. Although the surfaces typically remain consistent, there are a few cubes where there are anomalous areas within a layer that range in size from about one to four pixels wide, where the integrated intensity value is much higher than the rest of the material (**Figure 6-12**). These regions correlate to similar regions in the maximum intensity maps as well. These areas could be related to spatter that had fallen onto

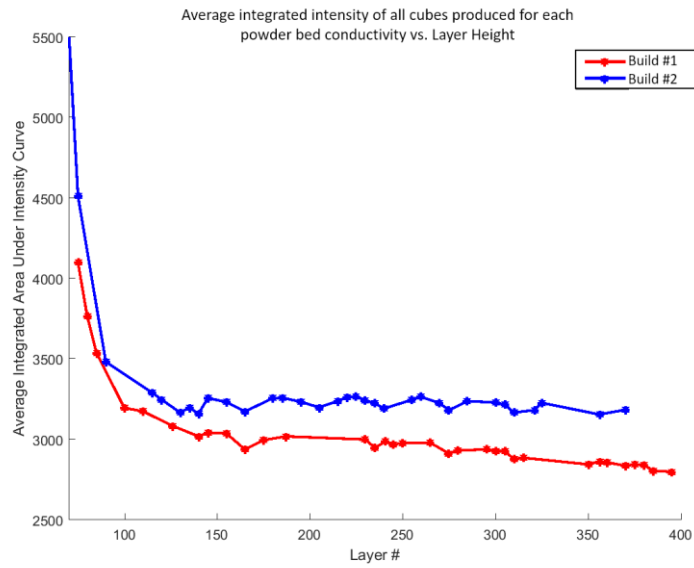


Figure 6-9 Comparison of the average integrated intensity for all cubes produced under both powder bed conductivity conditions. The second higher conductivity build shows a lower average integrated intensity for the material that has a slight decline as the build continues compared to the first build which remains relatively stable throughout the build.

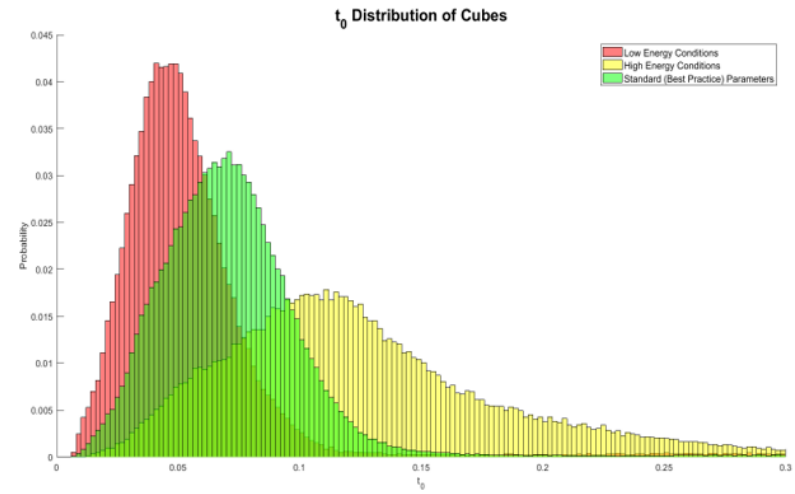
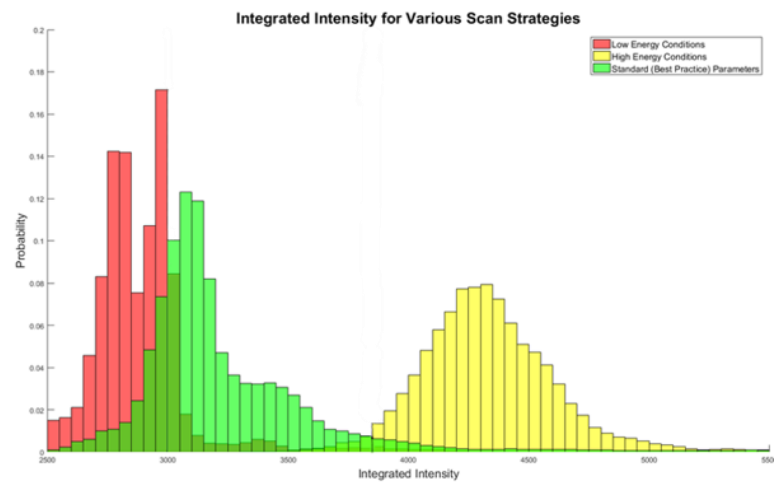


Figure 6-10 Comparisons of the integrated intensity (left) and decay rate t_0 (right) for all cubes within the build.

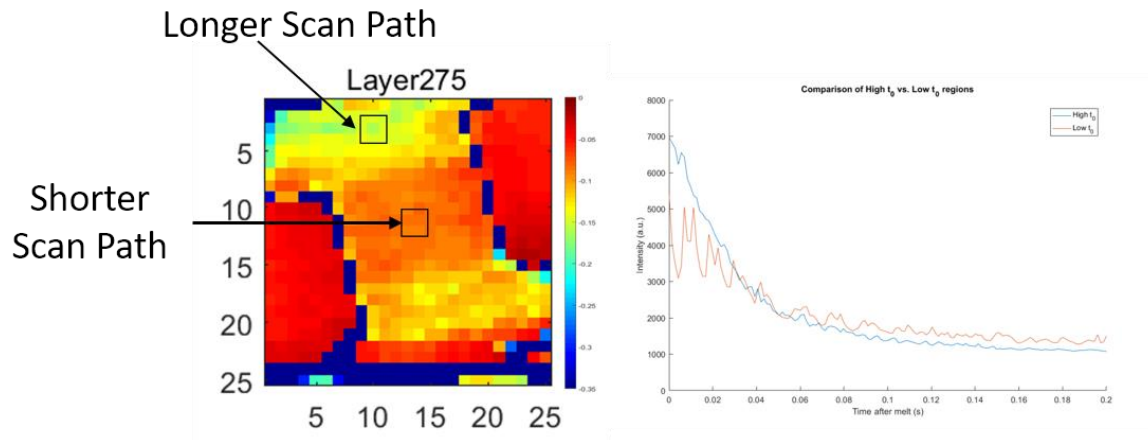


Figure 6-11 Image showing the relative intensity signals for the material in a cube. Note that the signal seen for the longer scan region rapidly decays after two initial passes the material rapidly decays and for the other material the rapidly passing beam maintains intensity for much longer.

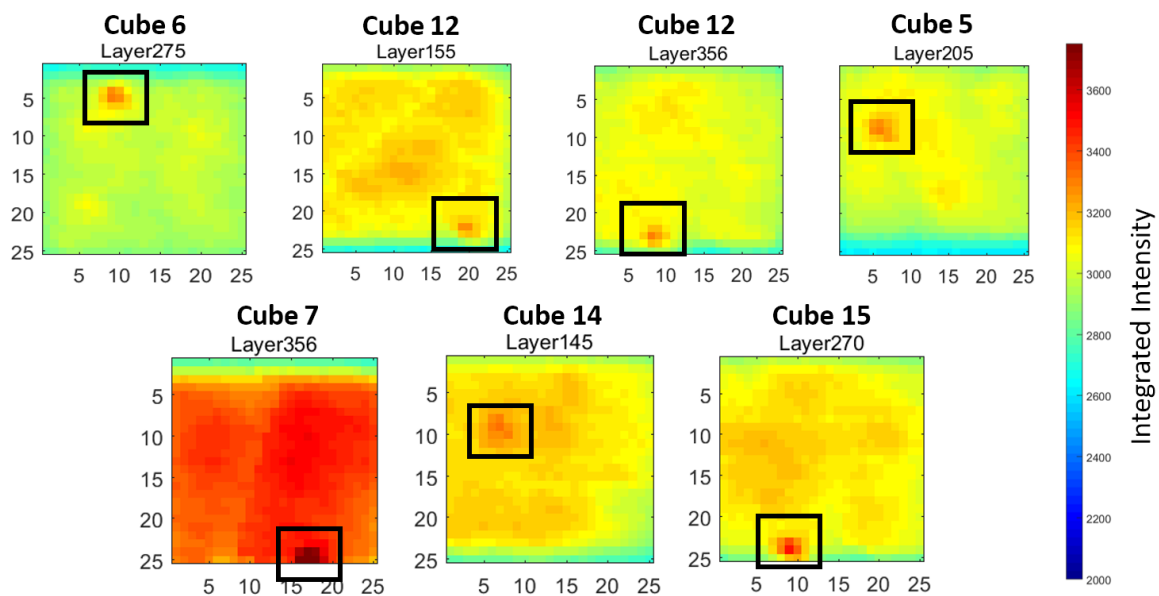


Figure 6-12 Images of layers within the cubes where regions of increase integrated intensity of the IR signal are shown. These regions could be locations of voids within the material or a location where spatter landed on the surface, however, further investigation of the cause is needed.

the material which would lead to an increased intensity for much longer or possibly voids within the material where the conductivity is reduced [74].

For both builds, microscopic imaging was collected for twenty cubes in the build to show the relationship between the process parameters, collected IR data, and the resultant material porosity. Digital image analysis was performed on all of the collected images, by converting the micrographs to a binary image that showed only the porosity present within the cube, and the total area fraction of porosity in the image was collected to determine the density of the cubes[71]. An image of the cubes can be seen in **Figure 6-13**, showing the cubes in order of increasing density for both of the builds. **Table A-6** shows the density values and associated energy density for both builds. As expected, the data shows that at the extremes of the parameter sets chosen the density begins to fall, with the lowest energy density values having more porosity than the higher energy density values. The cause of this difference is that low energy density values tend to lack-of-fusion porosity, which is characterized by a morphology that tends to include large, irregularly-shaped pores. These pores occur where the melted material does not adequately adhere to the substrate or where the individual beam paths do not have enough of an overlap with each other[58]. In contrast, the high energy density tends to lead to gas and keyhole porosity, which is characterized by small spherical and elliptical pores within the material produced [58]. These pores tend to occur when the increased melt penetration allows the material in the melt pool to collapse in on itself trapping a small amount of gas within the material [59], [61], [62]. Metallographic analysis reveals that Both of these pore types are produced during the build and correspond with energy density changes. With this data, the trends in porosity as an effect of the parameters can easily be seen, especially when mapping when the overall average value of the integrated intensity compared to the density of the material (**Figure 6-14**).

Also of note, is that in some cubes produced during the second build, there seems to exist a gradient in porosity where the material will either start relatively dense and become more porous as the build continues, or will eventually become dense after starting out with some porosity (**Figure 6-15**). In these few cases, a slow drift in the integrated intensity values characterizes the cause of this change in porosity. **Figure 6-16** shows a case where this occurs and the relative location of the change from porous to fully dense within the build. Note, that within these builds the processing parameters are held at a constant value, meaning that what

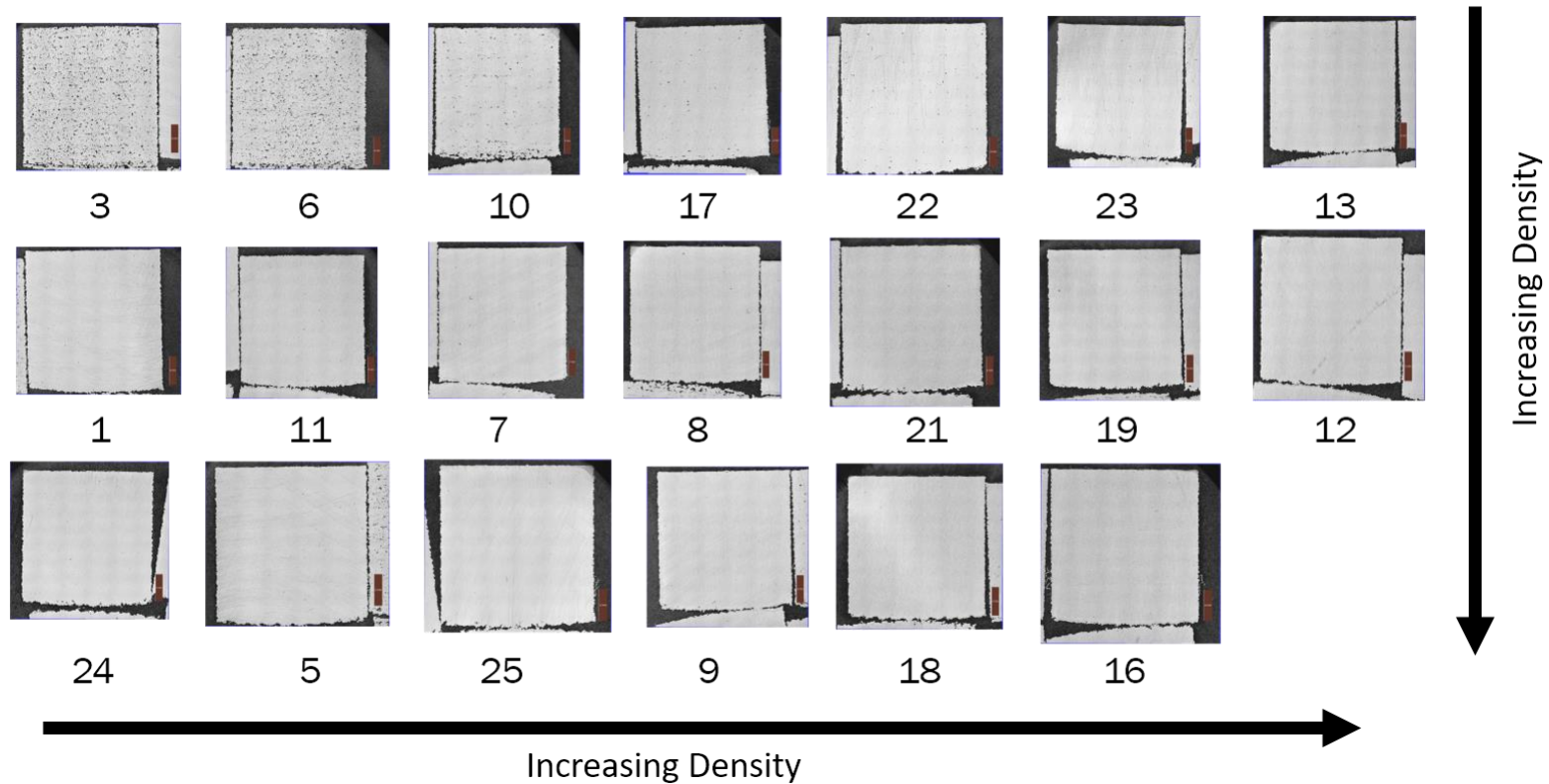


Figure 6-13 Series of images depicting the cubes ranked from least dense (top left) to densest (bottom right). As expected the best parameter sets were among the densest with the extremes of the variations ranking among the lowest.

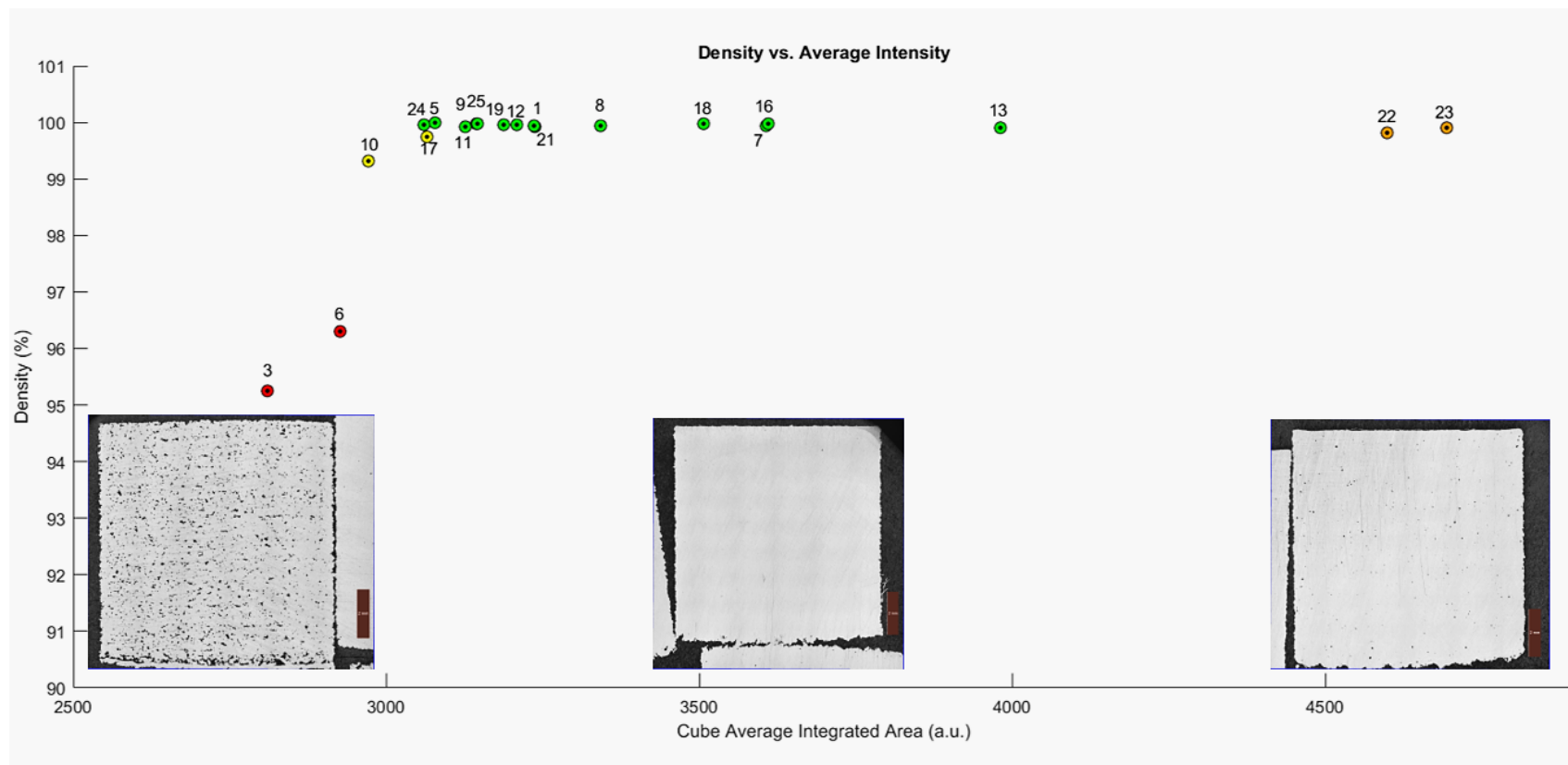


Figure 6-14 Comparison between the average integrated area for all of the cubes and their relative densities.

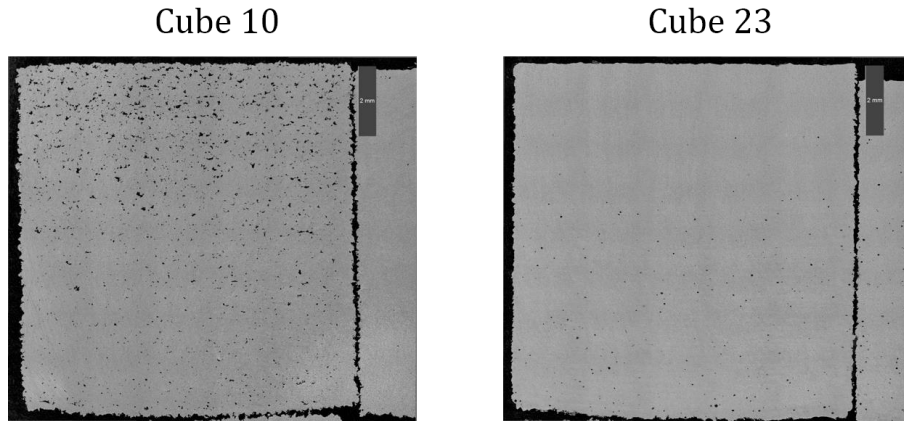


Figure 6-15 Images of cubes from the second build where the porosity shows a gradient in the material from some porosity to more porous (left) and porous to mostly solid (right). It is believed that this is due to the changes in the powder bed conductivity.

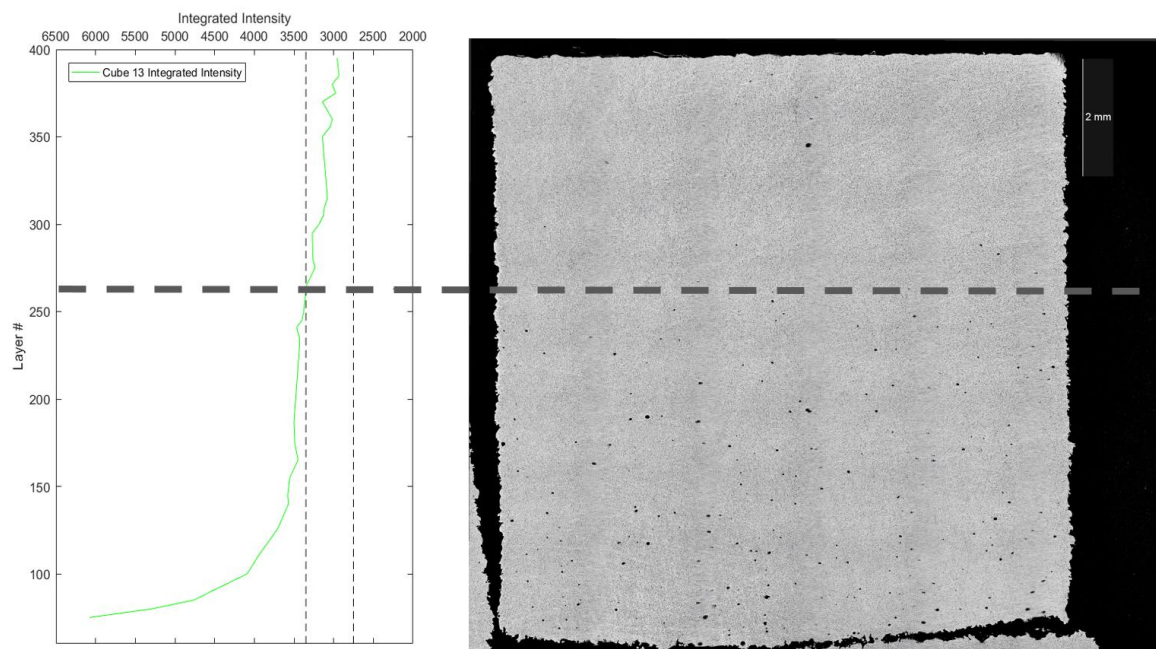


Figure 6-16 Cube porosity (right) compared to the average integrated intensity per layer(left). The graph of the average integrated intensity has been rotated so and scaled so that the data seen in the image coincides with the image. Note that the layer in which the material's average integrated intensity drops below a particular value, the material begins to exhibit higher density.

is being monitored here are the various environmental effects and effects from the machine that cannot be predicted or controlled within the build process by traditional parameters such as the beam power, speed, and point to point spacing.

6.2 Monitoring site-specific porosity

This section will discuss the data analysis performed and results gathered from an experiment that sought to determine the effectiveness of IR thermography as a method for detecting the location and size of porosity similar to methods that can be found in literature but with increased surface visibility [33]. The goal was to develop a method that could give the user an idea of the shape and location of porosity below the current surface throughout the build to quickly determine whether or not a part will meet their given specifications.

6.2.1 Data Analysis

A build containing created defects (Section 3.2.2) was created utilizing the SLS process to attempt to understand the ability of thermographic imaging to detect porosity, both on and below the current surface during the build. In this experiment, cylindrical voids with varying diameters and depths were placed in a grid in the center cylindrical build to test the sensitivity of thermographic measurements for detecting porosity locations. Upon close analysis of the thermal signatures of these regions, a clear difference is seen in how the material behaves when viewing the melt over a void or solid material during the melting process. As the beam rasters along the surface, a given point within the path will undergo several heating cycles in which the intensity of the pixel that the camera detects will peak and then subsequently decay over a small amount of time. However, the peak values and rate of decay of the intensity within a pixel differs when the pixel is located directly over a void as opposed to being located over solid material. When melting over a pore in multiple passes, the intensity of the IR signal will have slightly higher peaks that are maintained for more passes of the beam (**Figure 6-17**). Also, the decay rates of these signal peaks over time appear slightly longer. **Figure 6-18** shows both of these particular phenomena for the two regions directly after the first pass of the laser. This increase in intensity occurs when there is a subsurface pore because the trapped powder and gas below the surface has significantly reduced conduction compared to the solid material which causes the heat from the melting process to take longer to diffuse into the substrate. Also, if there is a region within the melt that goes unmelted, the surface layer of powder will have a slightly higher emittance when compared to the surrounding, melted material. This

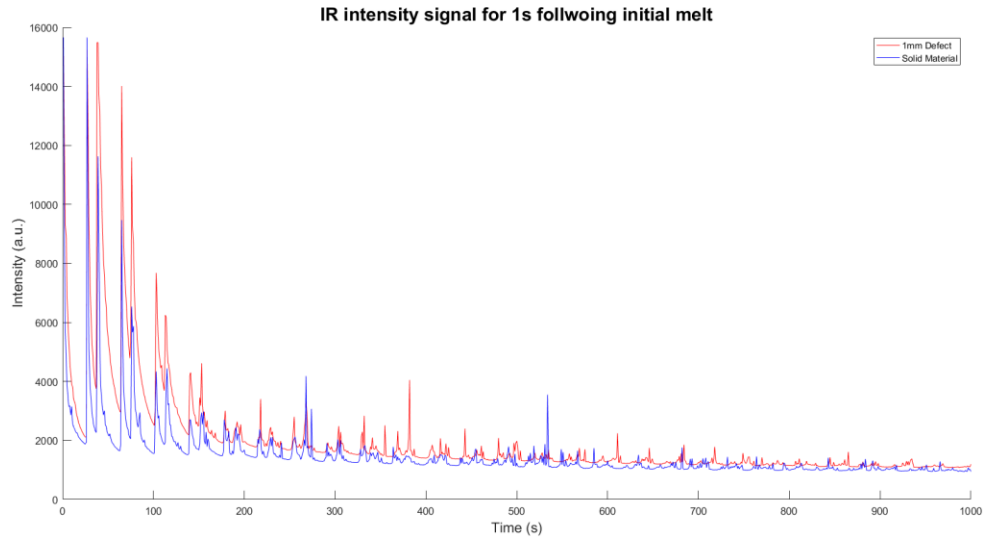


Figure 6-17 IR intensity signal for both the region over a 1mm void and over the solid material.

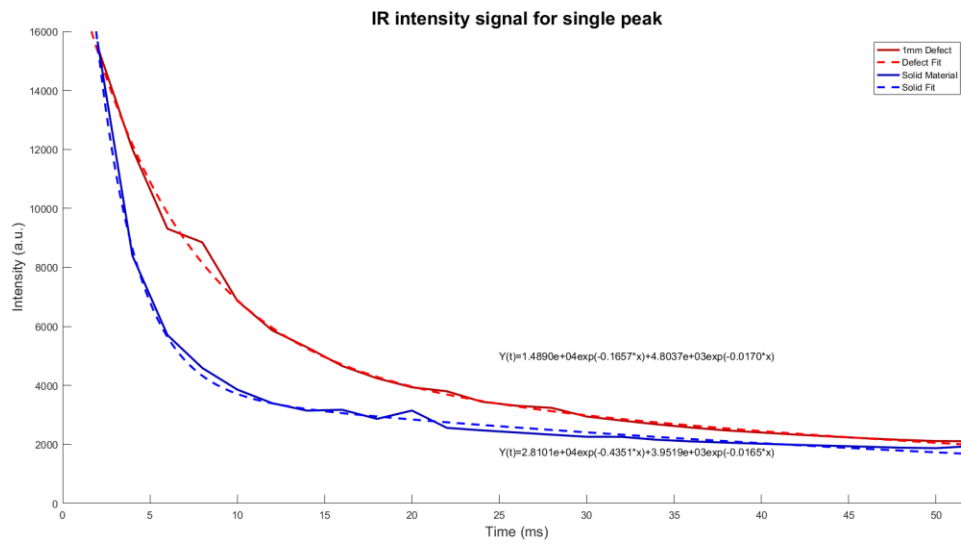


Figure 6-18 Comparison of the first pass of the beam for the material over a 1mm void and the solid material. Note the drastically different decay rate of the two materials.

higher emittance will also contribute to increased intensity signals over time (**Figure 6-19**). This decrease in decay rate and amplified intensity for sub-surface voids and surface discontinuities is consistent with what has been seen before in literature in works such as Krauss et al. and Dinwiddie et al. and shows that detection is possible with the correct processing and quantification [33], [40].

The first approach utilized to detect subsurface porosity was to attempt to extract a decay rate value through the use of curve fitting algorithms. However, because of the processing power required to analyze each curve created by a beam passing over the material in a pixel, and the difficulty of producing a robust curve fit (as seen in section 6.1), using this analysis may not prove to be an accurate or efficient method for detecting porosity. A novel way to detect quantifiable differences with relatively little computing power is to calculate the area under the curve for each set of data. The area under the signal curve, or integrated intensity, was calculated by using numerical integration using the trapezoidal method for 2 seconds of the melt curve following the maximum intensity value of each pixel, assumed to be coincident to when the beam melts the material at that pixel. As seen in **Figure 6-20**, the differences in the intensity values represented as an area are much more apparent and allow for the quantification of a significant amount of information about the IR signal as a single characteristic number for every pixel within the image. These values can then be analyzed to determine whether porosity or a surface discontinuity exists.

6.2.2 Analysis results and discussion

By mapping the integrated intensity over the surface of the material, an image such as the ones seen in **Figure 6-21** can be created for any given layer. In these images, the regions that are built directly over the intentionally created voids will have much higher integrated intensity values than the surrounding material. By selecting a threshold value of the integrated area associated with voids within the material, in this case, any pixels that were more than 15% above the average surface value, and filtering out the pixels which fell below this threshold, another set of images can be generated showing the location of these voids (**Figure 6-22**). Taking the values for the integrated intensity across all of the monitored layers and processing them with data visualization software allows for the creation of a three-dimensional of the part showing the location of the manufactured defects within the material (**Figure 6-23** and **Attachment 3**). **Figure 6-24** shows a flowchart of the data analysis process for better visibility.

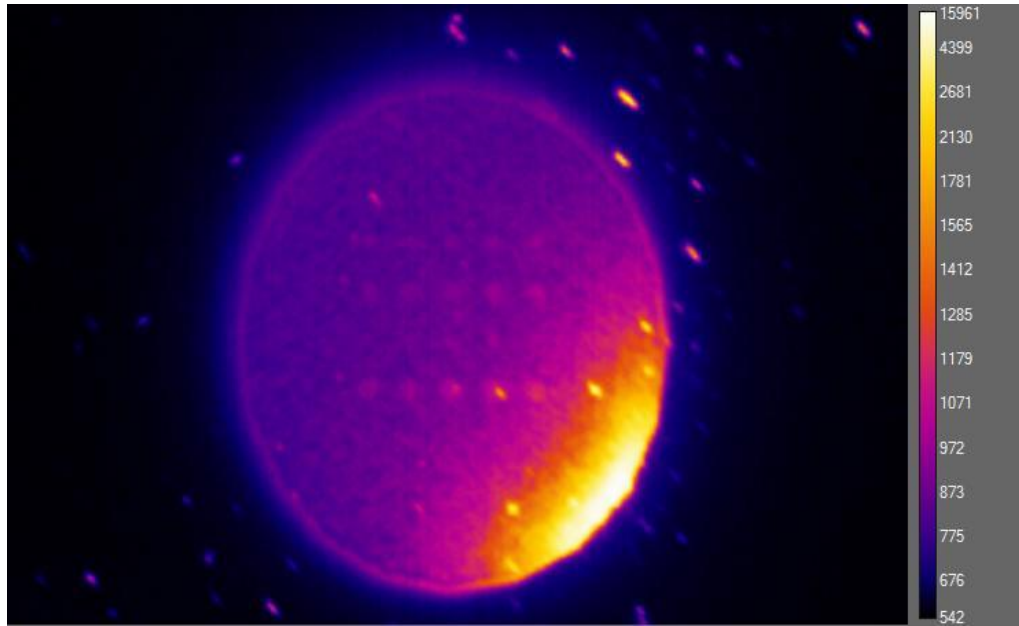


Figure 6-19 IR image taken from the build process during the melt, note each of the voids within the layer are brighter than the surrounding material due to the difference in emissivity.

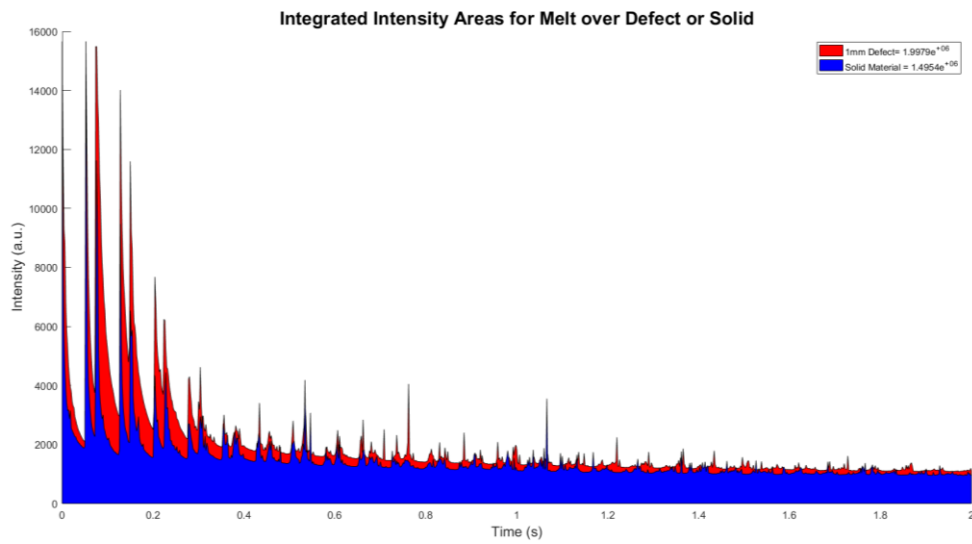
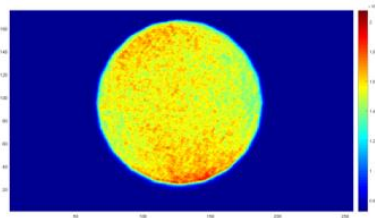
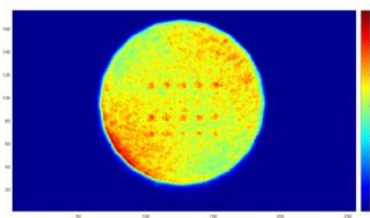


Figure 6-20 Comparison of the area under the curve for material located over a 1mm diameter defect and the solid part. Note the drastically different decay rates of each beam pass as well as the longer overall decay time of the material that is deposited over a void.

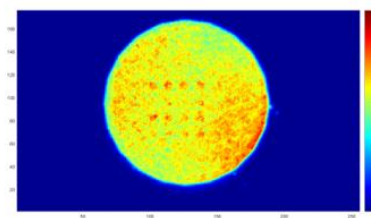
Layers 349-360 Integrated Area Surface Maps



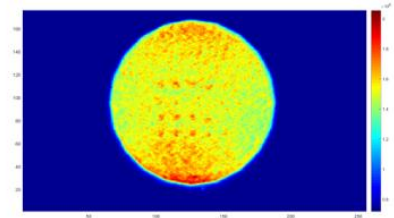
No voids present.



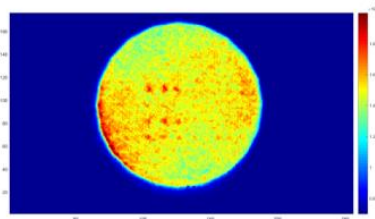
First layer where all voids are present.



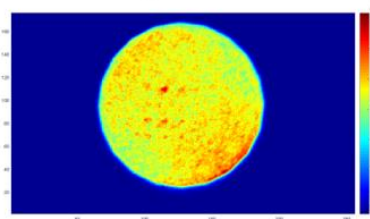
First set of voids melted over (far right).



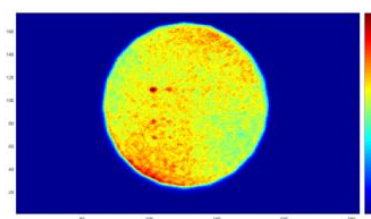
Second set of voids melted but still visible.



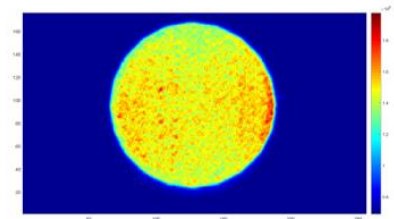
Next set of voids covered only two far left sets remain



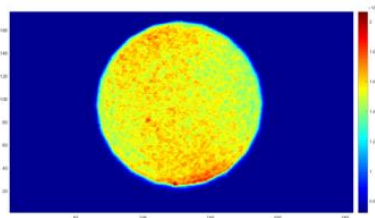
Second to last voids melted over only one set remains



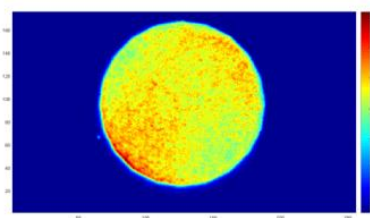
First melt over the last set of voids.



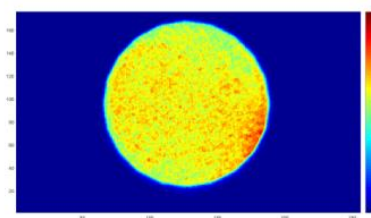
First layer over all sealed voids.



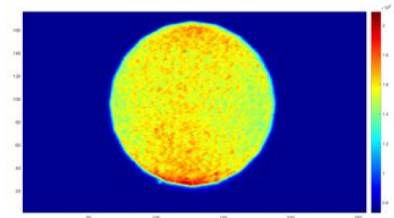
Two layers over the sealed voids



Three layers over sealed voids almost no voids are detectable.



No voids detectable.



Final layer in the set of imaged layers no voids readily visible.

Figure 6-21 Images of the integrated area values of the material's surface for a ten-layer defect set. Note how in the case show the defects can be seen both in-layer and underneath the surface of the material when the laser passes over the material.

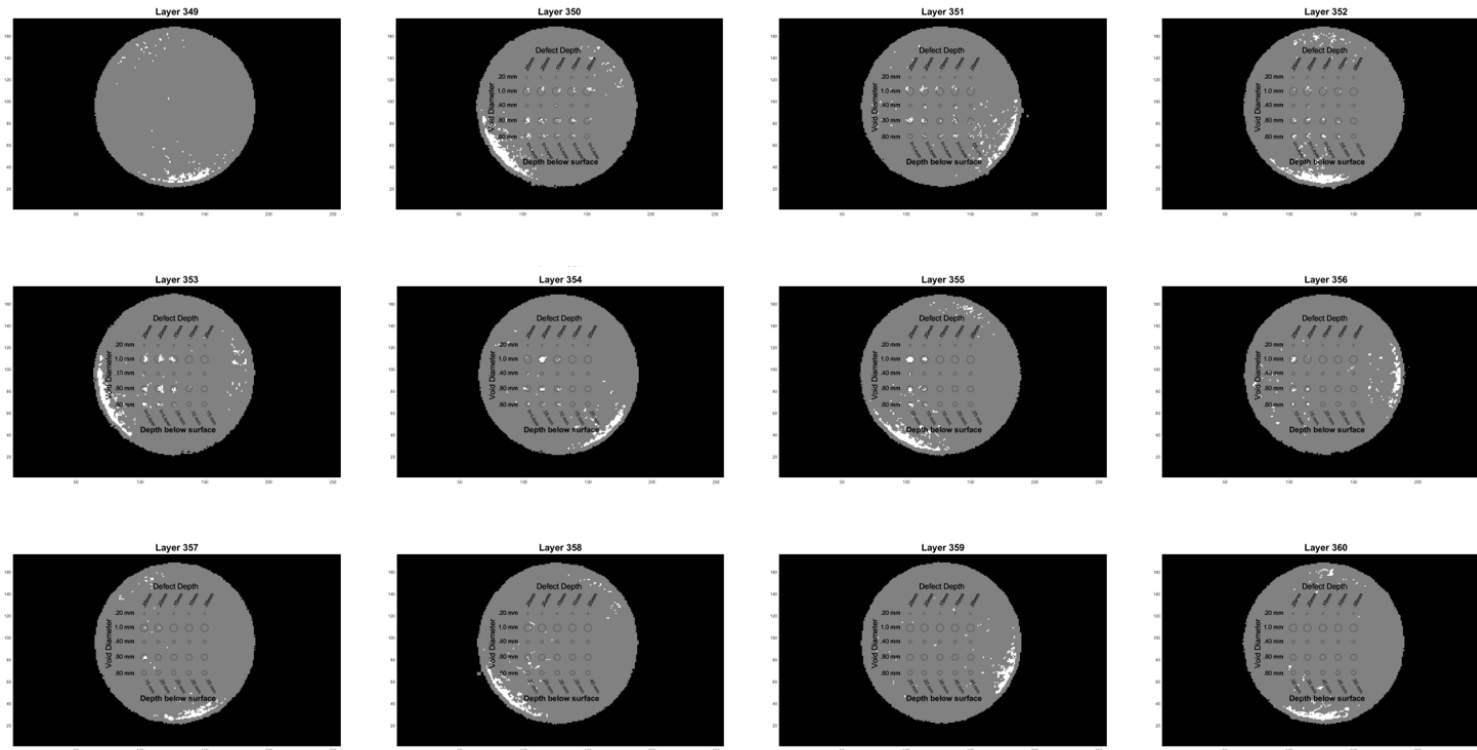


Figure 6-22 Images of the threshold images that are used to identify potential defect locations within the material.

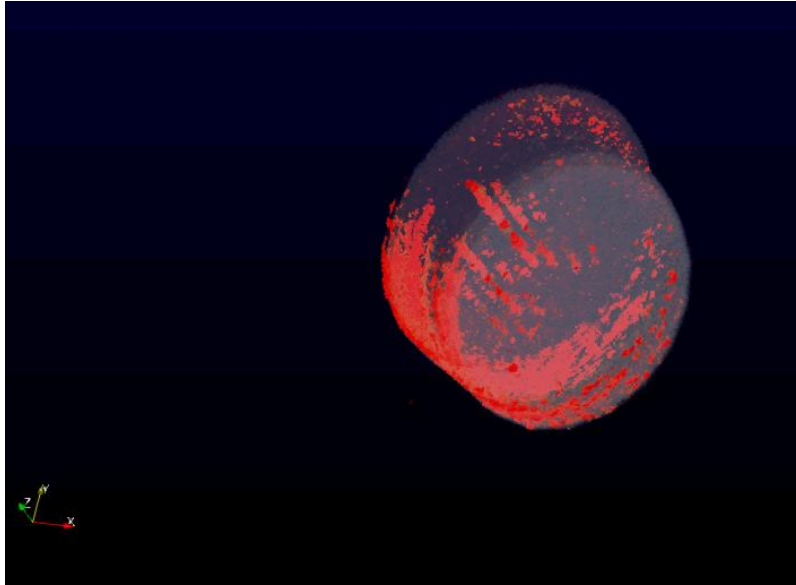


Figure 6-23 3D representation of the observed build with potential defect locations highlighted in red.
The edges show potential defects due to the increase of beam scanning frequency.

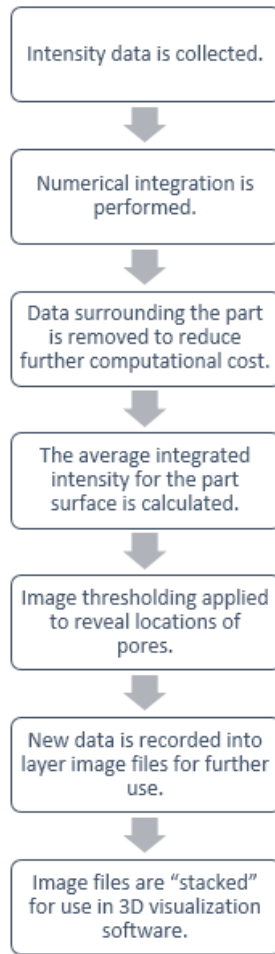


Figure 6-24 Flowchart describing the data analysis used for locating pores within the material.

Following these calculations, further data analysis can then be performed to show various trends within the data correlating to voids.

A trend between the engineered voids' size and the integrated intensity value that is collected within subsequent layers can be seen. Larger voids can easily be seen within the data with considerably larger integrated intensity values on average when compared to smaller voids. Conversely, the smallest created pores (which are on the order of the minimum detectable feature size, $200\mu\text{m}$) or are relatively shallow (less than three layers in height or $150\mu\text{m}$), are unable to be seen in any of the imaging. **Figure 6-25** shows this relationship between the size of these defects and their measured integrated intensity through a series of graphs each showing the effects of the diameter and height effects on the integrated intensity. From the figure, one can see that when the voids are sufficiently small in height, they become nearly indecipherable from the average surface temperature. However, as the defects' size increases, they start to become readily differentiable from the average surface integrated intensity, with the largest pores giving significantly higher integrated intensity compared to the surface.

The lack of the presence of the $200\mu\text{m}$ defects within the imaging follows the Nyquist-Shannon criteria, which states that the smallest detectable feature must measure twice that of the camera's instantaneous field of view [8]. Since a pixel in the imaging collected in this case is $200\mu\text{m} \times 200\mu\text{m}$, the imaging would only adequately detect these defects if they fell directly in the center of a pixel. Falling anywhere other than the center of a pixel that would not cause a change in the pixels intensity that would be significant enough to register the subsurface defects. Also, the defects that are less than three layers thick ($<150\mu\text{m}$ in height) are also often unable to be seen in the results when melted over as seen in **Figure 6-21**. These smaller voids are assumed to have been melted over but the data collected cannot determine if this is true. In order to confirm the presence, or lack thereof, of intentional defects in the material, X-ray tomography was performed on the part (**Figure 6-26**) and showed that smallest intentional defects that were not able to be detected by thermal imaging were rarely present within the material signifying that the detection technique is relatively capable of correctly detecting defects that formed.

As previously mentioned, the voids are also not only able to be seen when below the surface but while within the layer as well (negative depth below the surface in **Figure 6-25**).

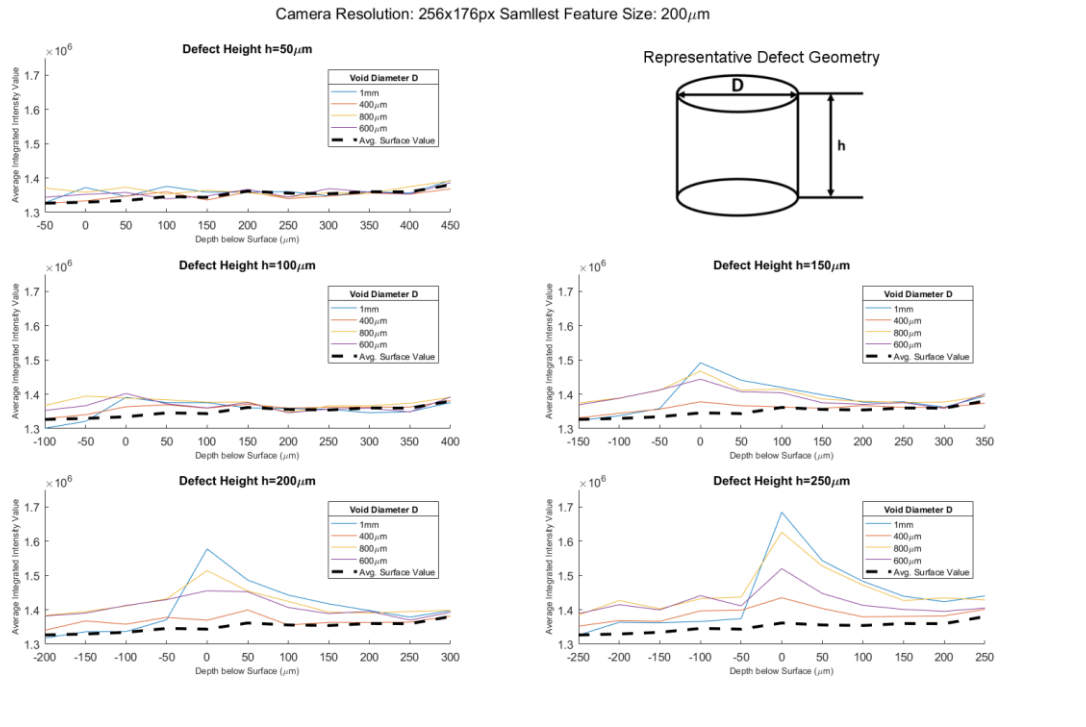


Figure 6-25 Series of graphs showing the relationship between the height and diameter of the defects in the material and the integrated intensity values collected while the material was within the layer (negative depth below the surface) or below the surface.

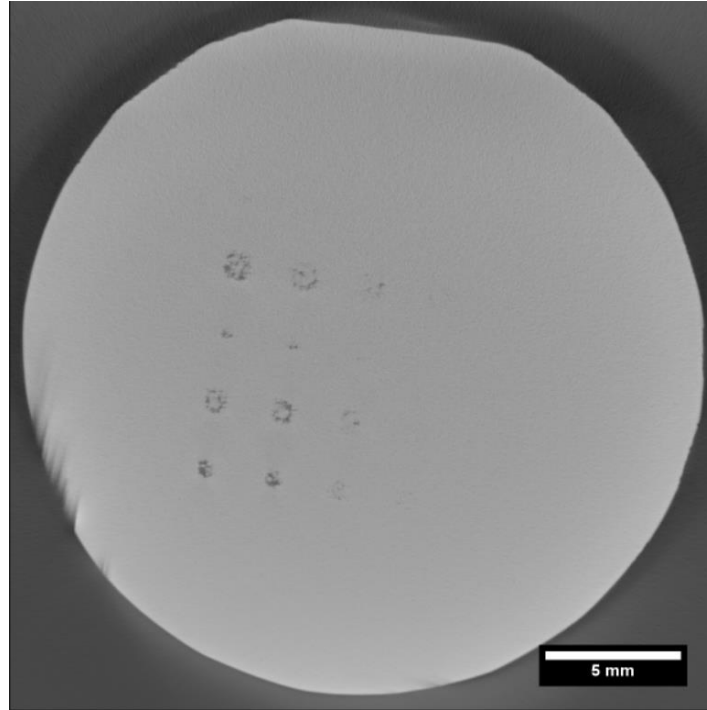


Figure 6-26 X-Ray CT image of the defects present in the part. Note the lack of the defects that are small ($<400\mu\text{m}$ in diameter and $<150\mu\text{m}$ in height) due to apparent remelting.

However, increase in the integrated intensity of the voids that are within the layer does not apply for voids that are 1mm in diameter. For every height of the cylindrical defects, the 1mm diameter voids do not see as significant of an increase in integrated intensity when they are within the layer. In fact, the 1mm diameter defects consistently hold the lowest integrated value within the melt layer even until they are melted over for the first time in which they surpass all other void morphologies, becoming easily visible. It is most likely that this decreased intensity of 1mm diameter defects within the layer may also be due to decreased thermal conductivity making it difficult to heat the volume of powder and be detected adequately. Meaning, that since these voids have more relatively insulative powder the amount of heat provided by the surrounding solid material is not significant enough to increase the temperature of the powder to make a measurable difference in the material emittance.

Finally, even though the data collected shows strong correlations with the initially designed voids in the material, there are still some regions that seem to correlate to defects. However, there should be only solid material underneath. These areas follow two morphologies: large regions that occur in the beginning or end regions of the surface melt scan, and small, single- pixel regions that occur in random locations across the surface being observed. The most prevalent of these regions of interest is the noticeable difference in the integrated intensity value at the beginning and end of the melt path across the material (**Figure 6-27**). The cause of the increase in intensity is most likely an effect of the scan strategy and the frequency at which the material passes a single pixel within the material. As the melt begins, the beam must make several short passes in rapid succession to melt the material. When this occurs, the number of intensity peaks within a short amount of time is significantly increased (**Figure 6-28**). As a result, the data collected over a such a short amount of time would have a significantly higher integrated area under the intensity curve. Also of note is that the leading edge of the melt path (the lower and left side regions of the imaging) has a higher intensity than that of the end. This change in measured integrated intensity is due to ejecta produced during the process. Since the integrated intensity data is collected over a two second period, the data that is collected at the beginning of the build will undergo high-frequency melting, and then as the beam moves away from the region, it will decay to the background intensity signal. However, during this time there are several small peaks in the data that are representative of spatter passing through these pixels when this occurs there is noise generated within the signal that will increase the overall integrated intensity for the region (**Figure 6-28**). Conversely,

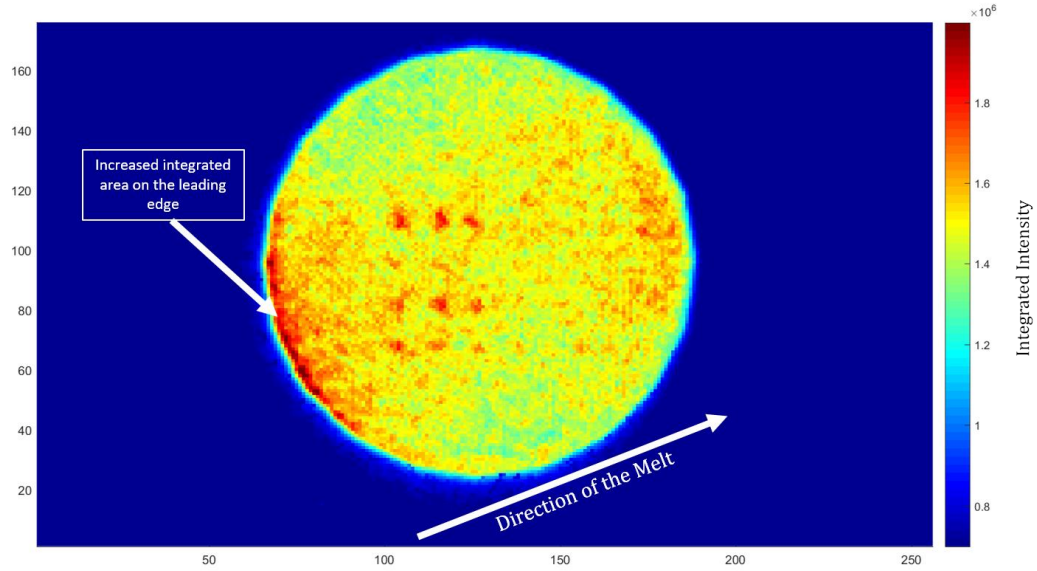


Figure 6-27 Image showing the increased integrated area for the leading and trailing regions of the surface's scan strategy.

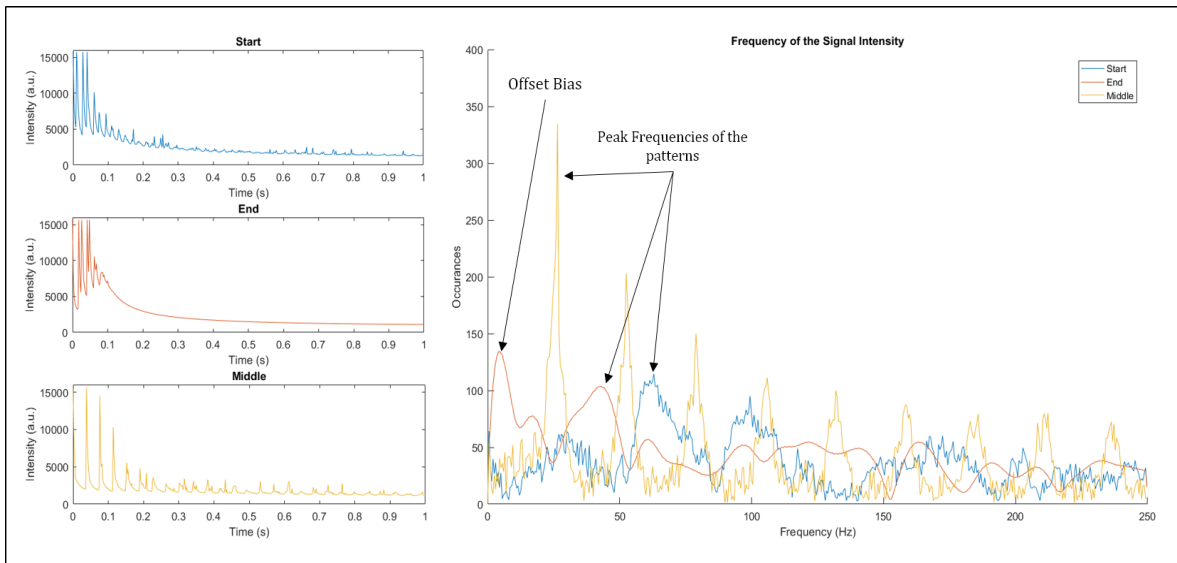


Figure 6-28 Representative intensity curves(left) for the material at the beginning, end, and middle of the parts melt, and the frequency of the same regions (right). Note the offset bias of the end melt.

material that is seen in the ending regions of the melt is not affected by the spatter because, following the beam passing through this area, the melting for the layer is complete, and the data follows a typical decay with no more noise created by ejecta. Without the peaks produced by the spatter the material sees less intensity and therefore the integrated area under the intensity curve is reduced. Developing a method to reduce the noise created by ejecta is not a trivial endeavor, and further research is needed to create a method to remove its influence on the analysis results, and the phenomenon has been discussed in the literature [75].

6.3 Sources of error, limitations, and future work

Although the data encompassed within this work shows that IR thermography could be used as a defect detection and monitoring tool, there are still concerns that need to be addressed to determine its full feasibility.

Material ejecta and scan strategy effects can contribute to false determinations of defect regions. In order to increase the reliability of this measurement tool, further experimentation and analysis need to be performed to determine a way to remove the error involved in regions that are characteristic of these effects or, in the case of the starting and ending regions of the scan path, if defect detection is unaffected. In this data the areas where the laser scan begins and ends do not have any intentional defects present and if another test were performed showing the same notable increase in the integrated intensity of the material or if some unintentional defects are actually present this may not be an issue. A method for detecting the presence of ejecta and then determining whether or not it lands on the part surface needs to be assessed so that it does not affect the results of the data adversely.

Another issue within the presented information was inconsistency within the data collection process. In the data provided in Section 6.1, there were several instances where the values of some of the average values for the integrated intensity, maximum intensity, or the decay times would be inconsistent with what is expected, creating peaks or valleys in the data. It was later determined that these inconsistencies were produced when the imaging of the build would end before melting had been completed for these cubes during the layer (**Figure 6-29**). When the imaging does not capture the melt of these locations, it causes the analysis methods used to find the maximum of the data that is captured which is relatively close to the value of the powder bed. This fault in the analysis leads the value of the maximum intensity and the integrated area of the data to register much lower in that region and for the decay rate

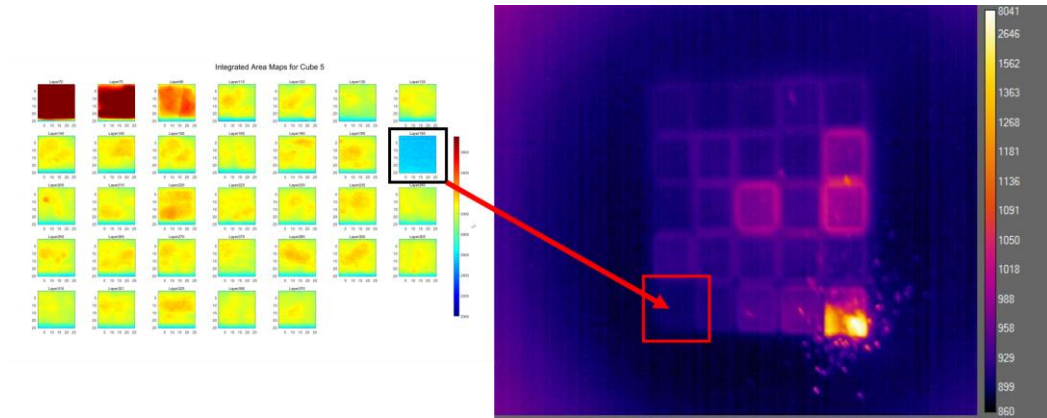


Figure 6-29 Surface maps for the integrated area of a cube for all layers (left) shows a dramatic decrease in the measurement for layer 195. Upon reviewing the IR video for the layer, it was discovered that the video ended early and the cube had not been melted in the video yet.

of the signal to appear much higher than expected. This error shows that even though the processing technique could be robust it is ultimately at the mercy of the data collection techniques and consistency in data collection is a significant factor.

A final concern that should be addressed is to determine whether or not the conductivity of the solid material will affect the feasibility of the site-specific detection of porosity by changing the frame rate needed. As mentioned in Section 6.2 the material melted above a pore maintains higher signal intensity for longer than when it is above solid material due to the difference in thermal conductivity of the powder compared to the solid material. This retained intensity is seen in the stainless steel 316L and the Ti-6Al-4V that was observed at high frame rates. However, if the material is much more conductive or insulative, the change in intensity could become less detectable especially at lower frame rates or excessively fast scan strategies. When the frame rate is low, or the material has relatively low thermal conductivity the data may not decay fast enough for the material to exhibit a differentiable change when compared to that of the porosity.

This methodology shows that IR monitoring has great potential to help locate and qualify porosity within parts during the build process with a fair degree of accuracy. With further experimentation and refinement, this methodology could easily be used to qualify builds and help increase the consistency and quality of the material being produced especially for high-performance parts. Ultimately this technique along with the other methodologies described within this work will help to increase the reliability and consistency of parts and increase industry acceptance by improving both the throughput and quality of builds as they are produced, especially in high-performance applications.

Chapter Seven: Conclusions and Future Work

This final chapter will discuss conclusions drawn in the research about the viability of the in-situ monitoring techniques described and the current limitations and needs to progress the technology further. Also, avenues for future work that could help to advance the techniques discussed will be to achieve higher accuracy and processing speed which could increase the robustness of the technology as a qualification and process control tool.

7.1 Conclusions

The research contained in this work shows that IR thermography could provide an avenue to the in-situ qualification of AM and expand the understanding of the AM process. The work presented in this thesis was able to determine:

1. A methodology to calibrate and correct the temperature profile of a part accounting for the change in material emissivity that occurs during the EBM process.
2. Using the newly corrected temperature profile and simple equations for calculating the thermal gradient and solidification velocity within the material, the microstructural development of the part built throughout an EBM manufactured build can be monitored in-situ.
3. Various measurements can be collected using signal intensity analysis of the SLS process most importantly the bulk material porosity determined by input energy density changes that are not controlled by machine parameters.
4. The application of the integrated intensity metric can reliably show engineered cylindrical defects that are larger than 100 μm in height and 400 μm in diameter within a build.

Each of these accomplishments could provide methodologies that potentially allow for in-situ qualification of AM produced parts. Even more importantly, these methods could provide a route to robust feedback control that would prevent costly build failure or errors within the final part. Even though this work shows that the use of thermographic data analysis has a great potential for process monitoring in AM processes, there are still numerous issues and limitations that, until they are addressed, will hinder the technology's ability to be used effectively.

7.2 Current Limitations and future work of thermographic data analysis in AM

7.2.1 Current Limitations and Concerns

As mentioned throughout this research, there are various limitations to the use of thermography as a method for in-situ process monitoring. However, these restrictions are a byproduct of a lack of research into the effects of various conditions and technological barriers on the accuracy of the measurements taken. For instance, as mentioned in several chapters the current resolution and frame rate limitations of IR camera technology can significantly impact the accuracy of the data collected. The lack of both spatial and temporal resolution leaves the technology incapable of understanding any information about the liquidus region of the melt pool, limiting the ability to obtain accurate representations for the thermal gradients and the solidification velocities of the material (**Figure 5-12**). The figure shows that when the camera's frame rate and resolution are limited to specific values, the relative thermal gradient and solidification will be restricted as well; causing points that would potentially have higher solidification velocities and thermal gradients to fall on these measurement limits. Along with the limiting effects of the resolution and frame rate, the actual calculations performed using these resolution values can be affected as well. For instance, in the calculation of the cooling rate of the material used to determine the materials solidification velocity, the slope of the curve following the melt was used. If the frame rate is too slow, it can cause the collected data to miss the peak intensity of the material or calculate the slope based on a point that is too far along the curve. Both of these faults will cause the data to under-calculate the cooling rate (as approximated in **Figure 7-1**), significantly impacting the solidification velocity. Also, if the conductivity of the observed material is very low, the decay rate of IR signal could cause issues for determining the integrated intensity values. Since the effectiveness of the integrated intensity value is dependent on detecting the slight changes in material conductivity due to defects and porosity, if the material conductivity is very low, these differences may not be as noticeable in the amount of time captured within a frame of the video. Extensive experimentation is needed to address these issues further and increase the capabilities of the technique.

Ideally, to achieve the correct understanding of the material during a build, the frame rate and resolution need to be appropriately sized based on the smallest features both in time

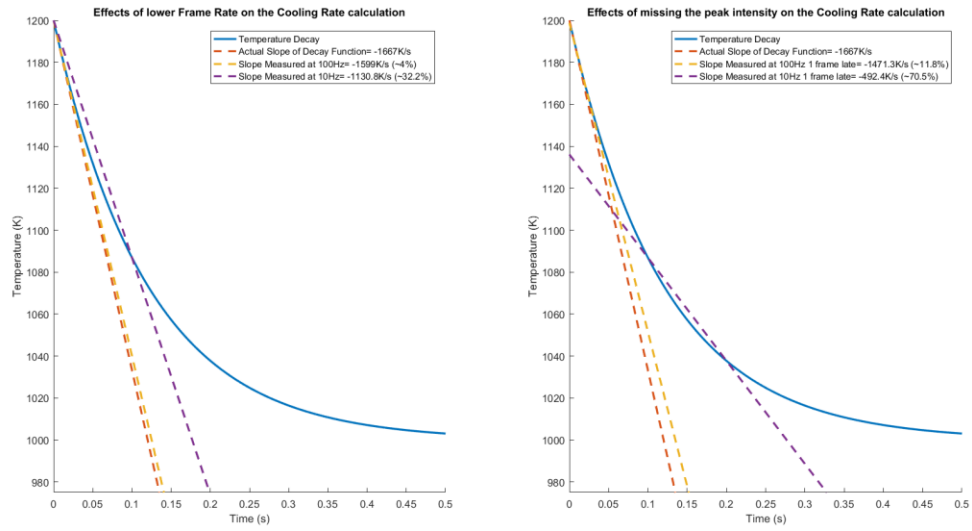


Figure 7-1 Conceptual examples of the error in cooling rate calculations brought on by missing the peak intensity of the curve by the equivalent of one frame (right) or by underestimating the slope of the curve due to time resolution (left).

and space that the user would want to understand. For instance, the principles of the Nyquist-Shannon sampling theory states that the sampling rates for time and space should be half of the smallest features of interest which would allow for accurate measurement and calculation of any phenomena of interest. As seen above, the frame rate and spatial resolution of the measurement system need to be carefully selected so that all of the relevant information that is required can be accurately collected and reduce the amount of error that occurs within the data. However, to achieve these resolutions both spatial and in time will significantly increase the amount of processing power and time needed as well as the amount of storage necessary for the data collected.

Another limiting factor of thermographic process monitoring is that the amount of data gathered for a build can become substantial. Even with low resolution, builds require 100 GB and higher resolution videos quickly approach terabytes (TB) of data storage for builds on the order of a few centimeters in height. The increase in data will also increase the amount of processing time required to give users the understanding they need to determine the quality of their builds. The processing times for the data included in this research ranged from several minutes in the case of the low-resolution data to hours for the high-resolution data collected. However, the processing times can be significantly reduced by removing unnecessary calculations and data. To do so, one can decrease size of the image to only the regions of interest within the build rather than the entire build, neglecting the powder bed data, or pick the slowest frame rate that is needed to understand the observed phenomena which can significantly reduce the size of the files as well as the processing time needed. Another methodology that was applied in the research here is to reduce the data into statistics or calculated values that can be rapidly read and processed rather than using the original data set. For instance, in Chapter 6 the data was collected and processed to values that reveal a great deal of information about the material within a pixel but require little storage. By reducing the amount of data, its subsequent analysis can be completed in seconds because the number of calculations needed is significantly reduced.

The reduction of data may need to be the standard for the current state of this technology because it can allow for calculations that are both computationally inexpensive as well as less time-consuming. However, as computational power keeps increasing and access to high-performance computing becomes more available the ability to perform real-time

calculation and analysis of data can be achieved. As for data storage, as high capacity storage becomes smaller and cheaper this concern could be addressed as well. Until then, the creation of quick calculations and data reducing techniques will need to be explored to make the most of the technology despite its limitations.

Apart from the technological limits of these techniques, there are also issues that arise from the physics of the process outside of the collection and processing of thermographic data. One such issue is the effect of surface topology on the resultant thermographic imaging. As previously mentioned, there were instances in the collected data in which swelling on the surface of the material created errors within the data. When the material starts to swell, the angle between the surface of the material and the camera sensor will change which leads to a drop in the amount of radiation detected by the material and increases the inaccuracy of the data. These changes in incident radiation need to be thoroughly researched to understand the effects on the resultant collected data. Swollen surfaces are not the only issue concerning surface topology; surface texture also could affect the resultant data collected. For instance, in the image seen in **Figure 7-2**, each of the spot melts has a bright, highly emissive center. Upon further analysis of similarly produced surfaces via confocal microscopy, the surfaces of these spots show a significant depression in the center of the spot. However, the effects have yet to be quantified, and a method to improve the resultant temperature calibration is needed as well.

It is imperative to address these concerns to improve not only the work presented in this material but the fields of process monitoring technology and additive manufacturing. Therefore, further work is needed to develop the technology and determine standardized methods need to be created to address the concerns and issues that arise due to these inaccuracies.

7.2.2 Future Work

The work discussed in this thesis was performed to help improve the capabilities and understanding of the AM process, specifically, powder bed fusion. The goals of this work were to help establish methodologies that could allow for in-situ qualification of AM produced parts and contribute to the advancement of the technology toward the ability to act as a process control system. Therefore, additional work needs to be completed to address the concerns raised by this research and further develop the technology and achieve these long-term goals.

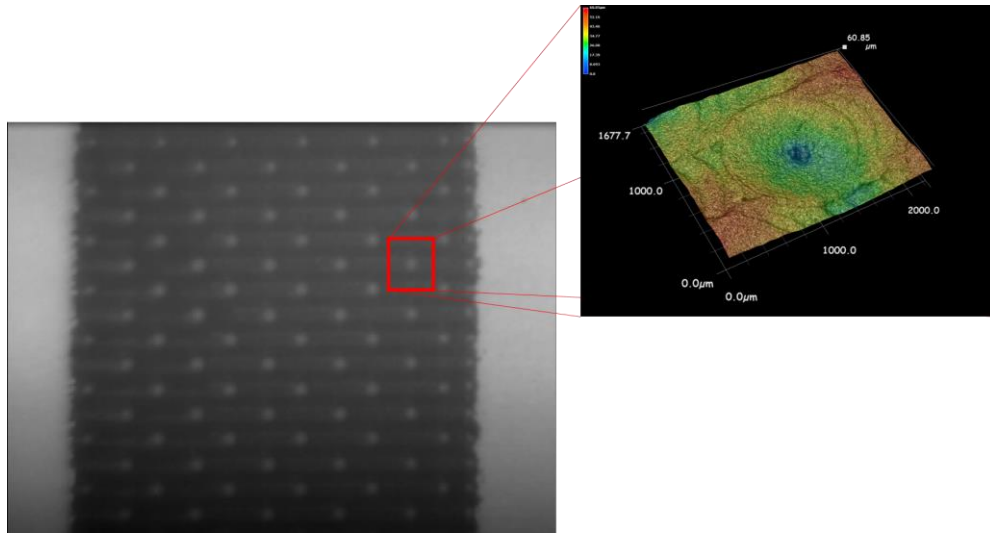


Figure 7-2 Image of a spot scan strategy taken in a build far after the end of melting for the current layer. The high-intensity spots were analyzed at 12x magnification using high-resolution confocal microscopy and show the surface topology for these regions.

The most pressing issue that needs to be addressed resulting from this work is to expand the methodologies presented to be used in both SLS and EBM processes. Currently, the analysis techniques presented in this work have only been developed for the EBM or the SLS processes independently, and therefore an appropriate temperature calibration process has not been created for the SLS process, and the porosity detection techniques provided have not been tested for EBM. These techniques need to be modified or expanded to encompass both processes as well as determining if they can be used on more materials without consequence.

Also, the concerns about the accuracy of the data and sources of error discussed in this research need addressing including error due to a lack of adequate resolution, the lack of a liquidus calibration and correction, and the error created by changing surface topology and powder bed inconsistencies. As for the understanding of the effects of surface topology and inconsistencies within the powder bed, more experimentation and subsequent analysis are needed to determine a method to detect specific surface geometries or regions where these inconsistencies occur and then correct them in-situ or with subsequent analysis. Hopefully, by understanding the way these surface features affect the data and then removing the associated error, the methods described in this work can become more reliable and allow for a proper avenue to process control and part qualification.

Along with correcting these current issues, the development of more algorithms and analysis procedures to address other issues that the AM process faces. These challenges include characterizing residual stress within the part, predicting other microstructural developments such as grain size and precipitation, and prevention of cracking within materials that are difficult to produce is imperative. By addressing these concerns within the process, users can be assured that parts manufactured using these processes will meet their performance needs, but currently, there are only a few methods that have been developed and tested [33], [34], [76], [77]. By expanding the capabilities of the technique, the usefulness of the technology can be improved and used to help further the understanding of the process and lead to developing a robust process control system. This knowledge and understanding will then be able to increase the consistency and reliability of additively manufactured material and increase industry confidence in the technology.

The end goal of this research and any subsequent research performed using IR thermography, as well as other process monitoring techniques, is the development of a robust

closed-loop feedback control system. Although there have been a few attempts at achieving this goal, there is still a lack of adequate results and development of such a system for the AM process[14], [36], [77]. By expanding the knowledge of process monitoring techniques and therefore increasing the accuracy of the technology a control system could become more feasible. However, to achieve this, a means of practically integrating the data collected by the sensors used in the machines to their control systems needs to be achieved, which is not a trivial task. Also, processing techniques such as those employed in this work need to be able to obtain the data, process the information gained, and then subsequently communicate the change to the machine at speeds on the order of the laser or electron beam motion which will require much more powerful computational expense and efficient processing methods. With expanded research into the field of AM process monitoring and the ever decreasing cost of high-performance computing and data storage, this goal could easily be obtained. As further research overcomes the current obstacles and limitations, the use of infrared thermography as a means to help improve the quality and reliability of additively manufactured metals will improve. This will allow for industry to provide better throughput and increase confidence in critical and complex materials.

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Appendices

Appendix A: Tables

Table A- 1 Table displaying relative advantages and disadvantages of the EBM and SLM processes.

<i>Process</i>	<i>Advantages</i>	<i>Disadvantages</i>
Electron Beam Melting (EBM)	Rapid build rate Lower Residual Stress Relatively low porosity	Poor surface finish Potential for in-situ aging Requires sandblasting to remove sintered powder.
Selective Laser Sintering (SLS)	Relatively fine part finish Relatively low porosity	Slow build rate Residual stress present

Table A- 2 Camera setups used.

<i>Experiment</i>	<i>Camera</i>	<i>Frame Rate (Hz)</i>	<i>Lens Size (mm)</i>	<i>Resolution (pixels)</i>	<i>IFOV (μm)</i>	<i>Integration time (μs)</i>	<i>ND filter</i>	<i>High-Speed Data Recorder (HSDR)</i>
Temperature correction/ Microstructure monitoring (Bracket) EBM	FLIR SC7600	10	25	480x380	350X 370	86.4	No	No
Microstructure monitoring (Cubes) EBM	FLIR SC7600	100	25	640x512	320 x 350	86.4	No	No
Bulk Porosity Detection	FLIR 8200	742.6	25	370x272	350 x 350	1245	Yes, ND2	Yes
Site Specific Defect Detection	FLIR 8200	500	50	256x176	200 x 170	222	Yes, ND2	Yes

Table A-3: Build parameters for 25 cube matrix experiment*.

<i>Cube #:</i>	<i>Hatch Spacing (μm)</i>	<i>Point Spacing (μm)</i>	<i>Exposure Time (μs)</i>	<i>V_{eff} (m/s)</i>	<i>Volumetric Heat Input (KJ/mm³)</i>	<i>Build Rate (cc/hr.)</i>
1	105	55	50	0.873	58	9.9
2	105	55	50	0.873	58	9.9
3	105	55	33	1.196	38	13.6
4	105	55	50	0.873	58	9.9
5	105	55	50	0.873	58	9.9
6	120	70	40	1.250	32	16.2
7	80	55	50	0.873	76	7.5
8	90	70	60	0.921	63	9.0
9	105	55	50	0.873	58	9.9
10	90	70	40	1.250	42	12.2
11	130	55	50	0.873	47	12.3
12	105	55	50	0.873	58	9.9
13	120	40	60	0.571	83	7.4
14	105	55	50	0.873	58	9.9
15	105	55	50	0.873	58	9.9
16	90	40	40	0.800	74	7.8
17	105	80	50	1.176	40	13.3
18	105	55	67	0.688	77	7.8
19	105	55	50	0.873	58	9.9
20	105	55	50	0.873	58	9.9
21	120	40	40	0.800	56	10.4
22	105	30	50	0.517	106	5.9
23	90	40	60	0.571	111	5.6
24	120	70	60	0.921	48	11.9
25	105	55	50	0.873	58	9.9

****Power was held at constant 200W***

Table A-4: List of Engineered Defects within the build and their grid coordinates.

<i>Row</i>	<i>Column</i>	<i>Height (μm)</i>	<i>Diameter (μm)</i>
1	1	250	200
1	2	200	200
1	3	150	200
1	4	100	200
1	5	50	200
2	1	250	1000
2	2	200	1000
2	3	150	1000
2	4	100	1000
2	5	50	1000
3	1	250	400
3	2	200	400
3	3	150	400
3	4	100	400
3	5	50	400
4	1	250	600
4	2	200	600
4	3	150	600
4	4	100	600
4	5	50	600
5	1	250	800
5	2	200	800
5	3	150	800
5	4	100	800
5	5	50	800

Table A-5: Weighting factors and frame numbers for use in the Savitzky-Golay filter calculations. The method of using this table is to sum the values obtained by multiplying the frames listed in column one with their respective weighting factor in column two and dividing that sum by the overall weighting factor given at the bottom of the table.

<i>Frame</i>	<i>Weighting Factor</i>
n-8	-21
n-7	-6
n-6	7
n-5	18
n-4	27
n-3	34
n-2	39
n-1	42
n	43
n+1	42
n+2	39
n+3	34
n+4	27
n+5	18
n+6	7
n+7	-6
n+8	-21
<i>Overall weighting factor</i>	323

Table A-6: Table comparing the calculated volumetric heat input and the cube densities from both of the observed cube builds on the Renishaw AM250.

<i>Cube #</i>	<i>Volumetric Heat Input (KJ/mm³)</i>	<i>Density (%)</i>	
		<i>Build #1</i>	<i>Build #2</i>
		<i>Normal Conditions</i>	<i>Increased Powder Bed Conductivity</i>
1	58	99.9157	99.9157
2	58	-	-
3	38	95.2460	90.8701
4	58	-	-
5	58	99.9967	99.9466
6	32	96.2980	94.0678
7	76	99.9332	99.9561
8	63	99.9334	99.9295
9	58	99.9735	99.9561
10	42	99.3130	99.9411
11	47	99.9159	99.7268
12	58	99.9550	99.9175
13	83	99.9010	99.8568
14	58	-	-
15	58	-	-
16	74	99.98196	99.9278
17	40	99.7400	98.5501
18	77	99.97535	99.9440
19	58	99.9504	99.9329
20	58	-	-
21	56	99.9459	99.9208
22	106	99.8124	99.62222
23	111	99.8995	99.8100
24	48	99.9625	99.53243
25	58	99.8704	99.86465

Vita

Jake Raplee was born in Terre Haute, IN, to parents Scott and Margee Raplee. He is the oldest of three children including himself, Joshua, and Sarah who both attended and attend the University of Tennessee respectively. He moved to Franklin, Tennessee and then to Brentwood, Tennessee, where graduated from Ravenwood High School with honors in 2010. After graduating, he then made his way to “The Loveliest Village on the Plains” to attend Auburn University with the Heritage Scholarship in pursuit of a degree in Mechanical Engineering eventually earning a minor in Business Administration. From there Jake traveled to “Rocky Top” to attend the University of Tennessee, Knoxville in the MS/MBA dual degree program to earn both a Master’s in Business Administration and a Master’s of Science in Mechanical Engineering with the latter granting him the opportunity to research under one of the university’s Governor’s Chair Professors, Dr. Suresh Babu. During his time at the university, he performed research in the field of additive manufacturing in conjunction with Oak Ridge National Lab’s Manufacturing Demonstration Facility working with the industry partners and researchers alike to help advance the field of additive manufacturing.