

Rationalization of Crystallographic Texture Evolution in High  
Gamma Prime Ni-based Superalloys during Laser Powder Feed  
Directed Energy Deposition

A Thesis Presented for the  
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## **Dedication**

To my family, girlfriend, friends, mentors and my dog, all of whom pushed me to achieve more and supported me throughout this work.

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## Abstract

Single-crystal nickel superalloy components are widely used in high temperature aerospace applications due to their excellent high temperature strength and creep resistance. Mechanical properties of single-crystal nickel superalloys are affected by chemical composition and the method of manufacture. Production of these components is costly and repair offers opportunity to sustain system performance while reducing costs. Additive Manufacturing by Laser Powder Feed Directed Energy Deposition (DED) is a promising method for repair due to selective application of material and the ability to tailor heat input. However, laser processing of these materials presents several challenges due to the high potential for cracking and recrystallization. Limiting stray grain formation and cracking within repairs is critical to product performance. The evolution of single-crystal nickel superalloy DED microstructures is investigated to assess the influence of DED on microstructural features including stray grain formation and dendrite growth directions.

A systematic assessment on the influence of mass flux, travel speed, and laser power on deposit microstructure was completed using design of experiments and thermo-fluid process modeling. Two test cases were considered including single-track and multi-track deposits. The influence of varying substrate thickness was considered. Modeling was used to understand the influence of varying mass flux, laser power, travel speed, and deposit geometry on crystallographic texture. Model calibration was performed using experimental measurements. The influence of varying processing parameters on deposit texture evolution for multiple deposit geometries are presented.

Epitaxial growth was observed for multiple deposit geometries. The experimental results suggest that process parameter optimization offers a means to produce highly textured microstructures. Managing equiaxed grain formation is critical to obtaining epitaxial growth which agrees with prior work. The ranges of laser power, travel speed, and mass flux considered in this study can significantly influence deposit texture evolution and appear to be key process parameters. Reducing the thickness of the cap of equiaxed material tends to promote highly textured microstructures with alignment of

the solidification structure to [100]. The thickness of the equiaxed cap decreases with reducing mass flux and heat input. The results suggest a variation in the number of nucleation sites for solidification within the deposit.

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## Chapter One: Introduction

Single-crystal nickel superalloys are used in gas turbine engine designs due to their high temperature strength and creep resistance. Manufacturing of single-crystal nickel superalloys can be costly and encounter long lead times. The metallurgical and manufacturing complexities associated with producing these alloys often presents substantial risk to attaining acceptable repairs.

Operating conditions for these materials can promote various degradation mechanisms including wear, oxidation, sulfidation, and thermomechanical fatigue. These mechanisms can negatively impact engine performance and result in the removal of parts from service [1]. With a typical single-crystal blade costing as much as \$30,000 to produce [2], there is strong motivation to identify a method for repair which can offer substantial opportunity for cost savings.

The prior literature has mainly focused on autogenous, single-pass welding of single-crystal nickel superalloys. Research on electron beam and gas tungsten arc welding of CMSX-4 and PWA-1480 single-crystal nickel superalloys has shown that cracking and stray grain formation can be mitigated at relatively low travel speeds [3-5]. Recrystallization and cracking are not acceptable in these repairs. Factors such as material pedigree, surface condition, substrate geometry and repair volume/geometry significantly contribute to establishing a qualified repair technology.

While autogenous welding may present many benefits for production of OEM equipment, there are limitations to these processes when considering single-crystal nickel superalloy repair. Single-crystal nickel superalloy feedstock is not commonly available in wire form. Powder feeding systems are not commercially available for electron beam welding systems and are not commonly incorporated with arc welding processes. Arc welding processes tend to exhibit relatively higher heat inputs compared to laser or electron beam processes due to the relatively diffuse heat source power density distribution.

A single-crystal nickel superalloy repair process should restore the part to its original geometry, remove non-conforming material and add new material in its place.

Since repair performance is significantly influenced by the repair microstructure and the ability to limit stray grain growth and misoriented grain formation (not [100] directions), the ability to readily produce acceptable repairs presents substantial technical challenges.

Laser powder feed directed energy deposition (DED) offers opportunity to achieve a relatively low heat input while readily implementing a feedstock form compatible with single-crystal nickel superalloy chemical composition. In addition, the process offers process parameter adjustments and a high degree of user-defined deposition path features. This readily enables processing of a prescribed, localized region. Given these features and the evidence presented on welding of single-crystal nickel superalloys in the literature, there is evidence which suggests the DED process exhibits favorable characteristics as a single-crystal nickel superalloy repair technology.

The ability to predict or directly correlate process parameters with deposit microstructural evolution is not well understood for additive manufacturing (AM) processes. As the heat source passes over the substrate, there can be many cycles of heating and cooling. There exists a high probability to produce cracks and recrystallization when repairing single-crystal nickel superalloys. Expanding the manufacturing community's knowledge beyond single-pass welding to multi-pass processing is critical to enable a viable repair technology for single-crystal nickel superalloys.

Metal AM exhibits many physical similarities to multi-pass fusion welding. Both processes include energy absorption, heat transfer, melting, solidification, and solid-state phase transformations which influence microstructural evolution, mechanical properties, and performance. AM of single-crystal nickel superalloys is not well understood. The ability readily produce a highly textured microstructure (directional solidification) while limiting stray grain formation and undesirable growth directions (non [100] directions) requires further understanding of DED process solidification. The three-dimensional temperature profile evolution is important to deposit microstructure. Given that there are many spatial and time dependent physical processes occurring during

DED, process modeling offers a viable means to investigate the influence of key process parameters on temperature gradients, solidification velocities and solidification process physics which influence microstructural evolution. Such phenomena are not readily examined by any other means. Repeated heating and cooling during the deposition process does not readily allow for the direct correlation of process parameters and factors significantly contributing to microstructural evolution. Therefore, the approach of this work utilizes both design of experiments and process modeling to better understand DED microstructural evolution.

In this work, the texture evolution of various DED deposit geometries are assessed relevant to single-crystal nickel superalloy repair. A systematic evaluation of several key process parameters is completed to better understand variation of laser power, travel speed, and mass flux on texture evolution for laser powder feed DED. A systematic approach was applied which evaluates the DED process relevant to repair applications. Two fundamental test cases were assessed including single-track and multi-track deposits. The experimental evidence was used to calibrate the thermo-fluid process model. Laser power, travel speed, and mass flux were varied for multiple substrate geometries to capture the influence of these process parameters on deposit microstructure. A steady state, thermo-fluid model based upon Openfoam is used to calculate metrics critical to predicting columnar to equiaxed transition during cooling. The influence of varying process parameters on deposit microstructures for various deposit geometries is presented and notable trends are identified.

## Chapter Two: Background and Literature Review

Prior work has investigated the laser powder feed directed energy deposition (DED) process as well as the solidification process of single-crystal and directionally solidified metals [6-8]. While these prior works provide a theoretical framework for this study, prior work does not investigate using single-crystal substrates with material deposition using DED of the same chemistry. Models for the single-crystal solidification process and columnar-to-equiaxed transition have been developed [7-9]. Modeling efforts have also begun in trying to understand the repair of single-crystal material through application of DED [10-13]. These models use finite element models to understand the thermal history of the material to better understand the influence of processing parameters on the formation of the resulting microstructure.

### 2.1 Nickel Superalloy Metallurgy

Nickel superalloys are commonly used in aerospace applications where high temperature strength, creep resistance, and corrosion resistance are critical. Ni-base superalloys can constitute 40-50% of the weight of a typical jet engine [14]. Nickel superalloys generally have numerous alloying elements (5-10) which may amount to 40wt% of the material [14]. Nickel is a face-centered cubic material that undergoes no changes in crystal structure with heating or cooling. Among the common alloying elements in nickel superalloys are chromium and aluminum for oxidation resistance, lanthanum and yttrium for oxide scale stability, and titanium, tantalum, niobium tungsten and rhenium for strength [15]. In general, refractory elements with large differences in atomic structure and radii in comparison to nickel are added for solid solution strengthening of the  $\gamma$  nickel matrix. These elements include tungsten, rhenium, niobium and molybdenum [15].

Nickel superalloys are precipitation hardened, generally, through precipitation of  $\gamma'$  precipitates. Schematics of the crystal structure of the  $\gamma$  and  $\gamma'$  phases are shown in Figure 1. The  $\gamma$  matrix is a normal FCC. The  $\gamma'$  precipitates also have an FCC structure but are of the  $A_3B$  type, where A is nickel and B is an alloying element. In this structure,



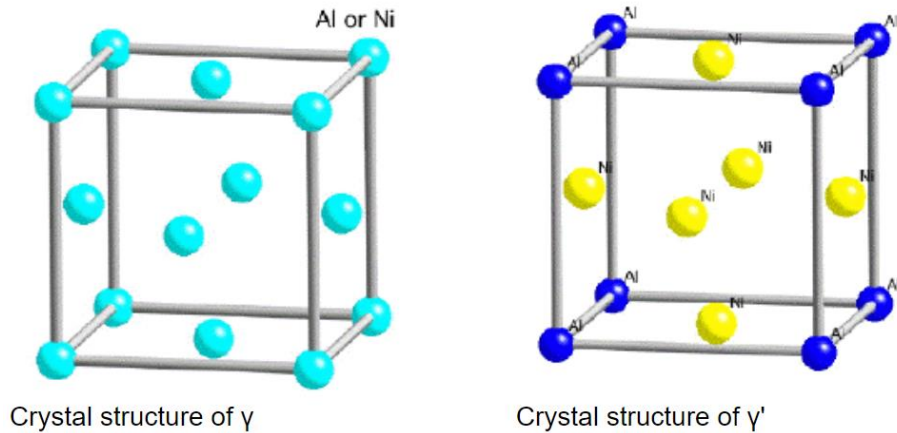


Figure 1: Gamma and gamma prime face-centered cubic crystal structures [16]. Aluminum and titanium alloying additions occupy the corner lattice positions in the  $\gamma'$  structure.

the B element occupies the corner lattice locations of the FCC [16]. Generally, the B element is titanium or aluminum.

The lattice mismatch between the  $\gamma$  and  $\gamma'$  phases is small (0-1%), allowing for coherent and semi-coherent interfaces to form [15]. Small  $\gamma'$  particles generally form as spheres as the spherical shape has the lowest energy. However, with coherent  $\gamma'$  particles, cuboidal precipitates have a lower interfacial energy [15].

A low interfacial energy between the precipitates and matrix results in high stability of the precipitates preventing significant coarsening at high temperatures. Gamma prime precipitates can have a maximum solubility limit as high as 90% allowing for significant strengthening by these particles and lending nickel superalloys its high temperature strength [16-18].

With  $\gamma'$  precipitates in nickel superalloys, an anomalous yield effect occurs. Between ambient temperature and 1023K, the yield stress increases with increasing temperature. After this inflection point in yield stress, increasing temperature decreases yield stress as shown in Figure 2. This is unlike most other materials where the yield stress decreases with increasing temperature. This anomaly, shown by the peak in Figure 2, has been attributed to an increasing fraction of strength imparted to the

material from the  $\gamma'$  phase up to 1023K where the properties are controlled by the  $\gamma'$  phase [19].

Design of single-crystal alloys looks to strengthen the  $\gamma'$  phase, increase the volume fraction of  $\gamma'$ , and optimize the lattice mismatch between the  $\gamma$  and  $\gamma'$  phases. Alloying with titanium and tantalum increase  $\gamma'$  strength as both increase the anti-phase boundary energy improving the resistance to  $\gamma'$  shearing. Aluminum, titanium and tantalum additions increase the phase fraction such that the pathways for dislocation motion in the  $\gamma$  matrix are minimized. Optimization of the lattice mismatch such that the  $\gamma$ - $\gamma'$  mismatch is similar lowers the driving force for precipitate coarsening. Finally, alloying with refractories, such as tungsten and rhenium, improves time-dependent properties if kept to an amount that does not promote the formation of detrimental phases such as the laves phase [19]. The microstructure of these alloys thus is void of grain boundaries with a large fraction of fine cuboidal  $\gamma'$  precipitates as shown in Figure 3. This microstructure allows for a high amount of strengthening from the  $\gamma'$  precipitates while producing a tortuous path for dislocation movement in the  $\gamma$  matrix. These further enhance mechanical properties of the single-crystal material.

#### 2.1.1 Manufacturing of Nickel Superalloy Products for Gas Turbine Applications

Nickel superalloys components can be produced through casting, powder metallurgy, forging or machining processes. For the components of interest in this work, gas turbine hot section components, the substrate material is generally produced by casting. Both single-crystal and directionally solidified nickel superalloys are generally produced by investment casting. A ceramic mold is fabricated by progressive layers of ceramic around a wax pattern which is later removed to produce the mold. Directionally solidified and single-crystal castings are solidified by withdrawing the component from the hot zone of the furnace to a cold zone at a controlled rate [14]. By maintaining a unidirectional thermal gradient, solidification occurs along a single-crystallographic direction. An example of the resulting mold and component is shown in Figure 4. The casting mold contains a single-crystal seed that resides at the base of the mold, residing

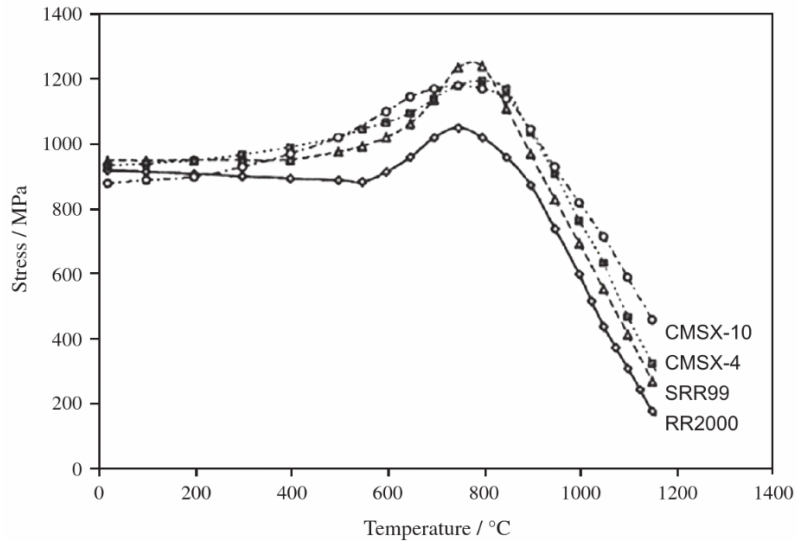


Figure 2: Variation of the yield stress of a number of single-crystal superalloys with temperature [19]. A peak occurs due to a yield point phenomenon occurs at 1023K (800 °C) due to  $\gamma'$  strengthening

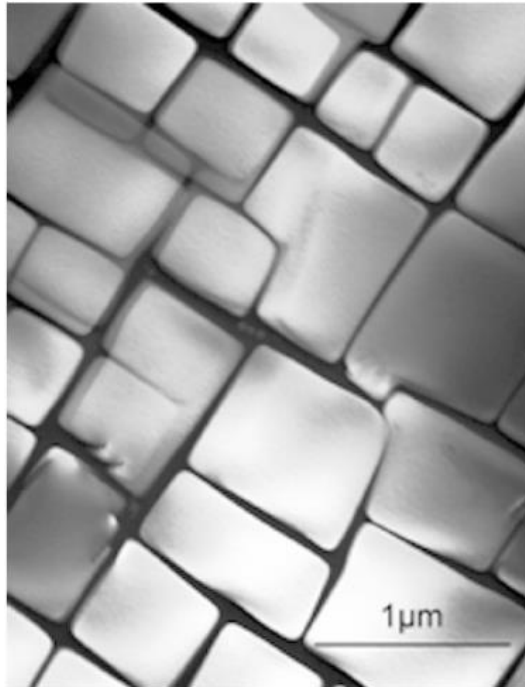


Figure 3: Microstructure of a single-crystal nickel-superalloy showing cuboidal  $\gamma'$  precipitates. This  $\gamma'$  precipitate shape is favored with a coherent interface between the  $\gamma$  matrix and  $\gamma'$  precipitate [19]

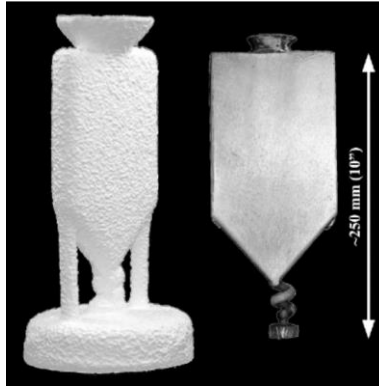


Figure 4: Ceramic mold with single-crystal starter at the bottom of the plate (left) and resulting single-crystal component following solidification and mold removal (right) [14]

at the end of the swirl in Figure 4. Following casting, heat treatments are performed to reduce chemical segregation and modify the precipitate distribution [14]. Single-crystal castings are still subject to formation of a number of defects due to processing and chemical segregation such as misoriented grains, porosity, hot tearing, and grain boundaries. These defects may limit mechanical properties. Processing is carefully controlled to prevent these defects and nondestructive test methods employed after casting to evaluate the completed casting [14].

Another manufacturing method of interest is powder metallurgy. Powder metallurgy is a popular manufacturing method for nickel superalloys which have a high level of refractory or  $\gamma'$  forming alloying additions. Large percentages of these additions make traditional manufacturing methods insufficient. Powder processing by gas or vacuum atomization allows for production of fine powders. With a small particle size, chemical segregation cannot occur. These high alloyed powders can then be consolidated to near net-shape with minimal chemical segregation and cracking that can occur with casting of ingots [14]. However, this method cannot produce single crystal material.

In creep sensitive applications, such as the hot section of turbine engines, the grain structure of the material becomes an important design factor. Directional

solidification improves creep resistance by formation of coarse grains which are highly aligned with the load direction. This improves the creep life as compared to equiaxed material. With even better creep life are single-crystal nickel superalloys. These alloys are void of all high angle grain boundaries and do not require grain boundary strengtheners. Removal of grain boundaries eliminates creep by grain boundary diffusion and an increase in incipient melting temperature [16]. The increase in incipient melting temperature allows for heat treatments at higher temperatures allowing for a higher volume fraction and finer dispersion of  $\gamma'$  particles.

Cooling rates of the nickel superalloy also effect the formation of  $\gamma'$  precipitates and dendritic substructure. With high solid-state cooling rates ( $>40$  K/min), a unimodal distribution of fine  $\gamma'$  precipitates forms. With lower solid-state cooling rates, a bimodal distribution of relatively large ( $>500$  nm) and small ( $>50$  nm) precipitates. With decreasing cooling rates from liquid to solid, secondary dendrite arms grow increasing dendrite spacing. This coarsening of the dendrite structure with decreasing solid to liquid cooling rate is shown in Figure 5 where the dendritic structure into the solidification direction is evaluated at four different cooling rates [14]. The size of the dendrite arm spacing is correlated to the amount of chemical segregation where a larger dendrite arm spacing is related to higher segregation [14].

### 2.1.2 Welding of Nickel Superalloys

Welding of nickel superalloys has been an area of interest in literature due to high demand for use of the alloy, the necessity for weldability in product design and the high costs associated with replacement over repair of components [20]. Dilute nickel superalloy chemistries can generally be welded and repair methods for these chemistries are well established in industry. However, the higher strength precipitation hardenable chemistries have proven difficult to weld. These chemistries are prone to solidification, heat affected zone and strain age cracking defects. Special considerations, such as the aging state, processing parameters, and post-weld heat treatments, must be made for acceptable welds in high  $\gamma'$  alloys [20]. Welding of these

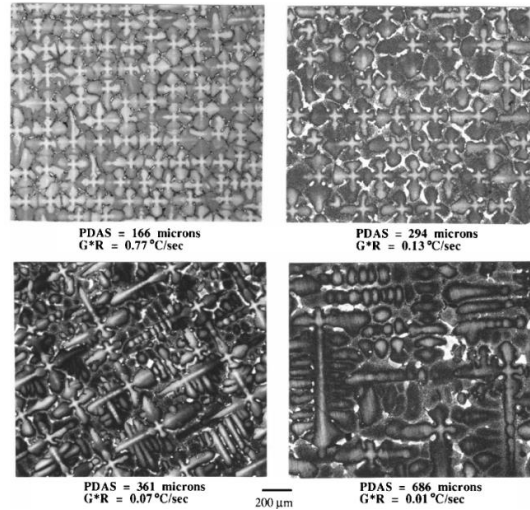


Figure 5: Primary dendrite arm spacing (PDAS) increases with decreasing cooling rate [14]

high  $\gamma'$  nickel superalloys can be conducted through gas tungsten arc welding (GTAW), electron beam, laser welding, friction welding and laser powder deposition.

Weldability in nickel superalloys is generally described by the amount of aluminum and titanium [20]. These elements are strong  $\gamma'$  formers and increasing the amount of these alloys increases the amount of  $\gamma'$  phase that is deleterious to weld quality. When the total amount of these two elements exceeds a critical amount, the alloy is considered “hard to weld” with increasing difficulty with increasing weight percent of these elements in the alloy [20]. Figure 6 shows weldability plot with a commercially available single-crystal alloy, CMSX-4, circled. From this plot, it is seen why single-crystal alloys are some of the harder to weld nickel superalloys due to the high volume of  $\gamma'$  necessary for the high strength and creep resistance necessary for their application.

In single-crystal alloys, researchers have investigated GTAW, electron beam, laser deposition methods especially for repair [3-5, 7, 8, 20-27]. Solidification cracking, strain age cracking and stray grain formation are the defect types that engineers look to avoid in these alloys [20]. In welding by electron beam and GTAW of these alloys,

### Weldability Assessment: strain age cracking susceptibility

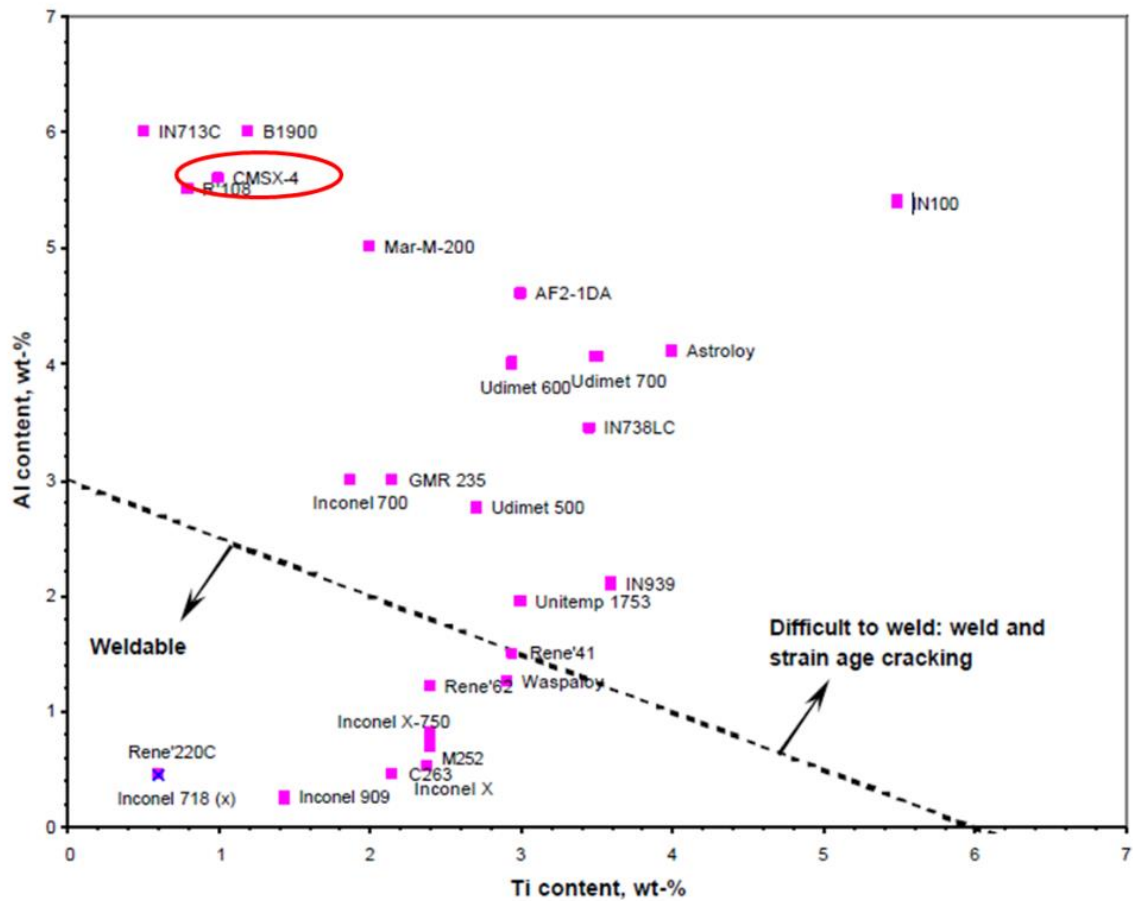


Figure 6: Weldability plot for nickel superalloys. High aluminum and titanium content ( $\gamma'$  formers) decreases weldability due to increased fraction of  $\gamma'$ . [20]

careful selection of processing parameters and preheat allowed for crack free material with minimization of stray grain formation [4]. Extensive work has been conducted on the mechanisms and mitigation of cracking in these alloys and can be found elsewhere [4, 5, 20, 27-29].

## 2.2 Laser Powder Feed Directed Energy Deposition (DED)

Laser powder feed directed energy deposition (DED), shows promise for repair of single-crystal nickel superalloys due to the relatively localized input of heat and high degree of user control of processing parameters and tool path. The material deposition allows buildup of new material. Localized input of heat has minimal heat impact on the component or substrate reducing the tendency for recrystallization and cracking [20]. The high solidification velocities associated with the localization of the heat source is also beneficial for reduction of chemical segregation, as the solute is trapped in the solid, and epitaxial growth from the substrate [20].

In Directed Energy Deposition, powder is fed to the molten pool using inert transport gas. These transport gases include the cover gas, which helps the powder flow out of the hopped, carrier gas, which carries the powder to the nozzle, and the shaping gas, which helps to shape the powder stream leaving the nozzle. Powder nozzle jets are aimed at the molten pool to feed powder metal which creates a raised track of material. A focused laser beam is aimed at the substrate which forms a molten pool. A schematic of the process is shown in Figure 7. The nozzle or workpiece are moved in the x-Y-direction to control the cross-sectional shape while the nozzle is moving in the z direction to control the layer height. Adding sequential layers in the z direction creates a three-dimensional part.

The microstructure of DED parts is typically columnar [30]. The orientation of the prior layer is generally maintained with columnar grains of that orientation forming and extending with additional layers. An example of this effect is shown in Figure 8, where an Inconel 718 multi-layered deposit was formed [30]. EBSD imaging shows the elongation and continuation of grains through multiple layers. Deposit microstructure can be affected by the substrate microstructure as shown in Figure 8. In this figure, the



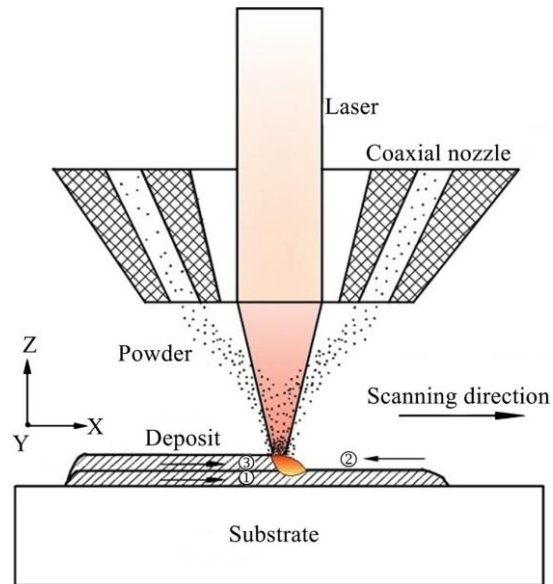


Figure 7: Schematic of the Directed Energy Deposition process [12]

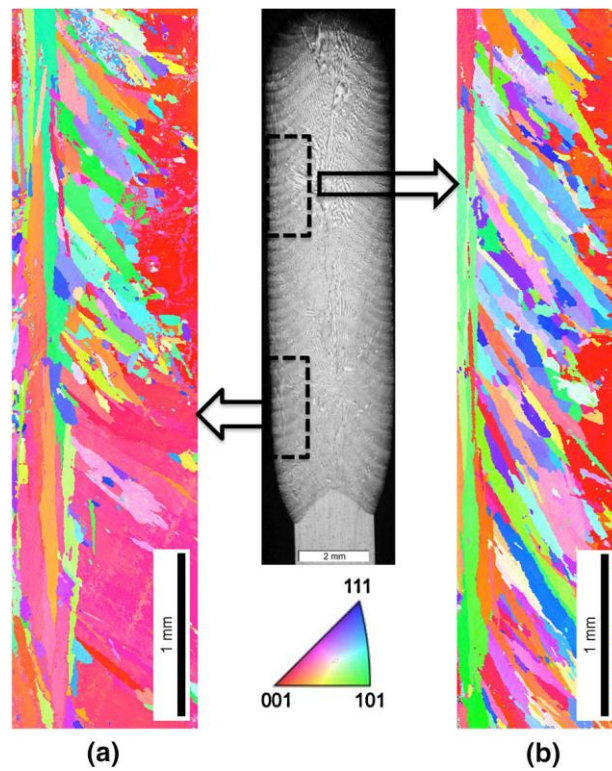


Figure 8: EBSD of transverse cross section of Inconel 718 Directed Energy Deposition multi-layer deposit. Grain orientation of the prior layer influences the orientation of the current layer [30]

microstructure of the prior layers can influence the orientation of the grains in the following layers. This results in directionally solidified material in these locations.

### 2.2.1 Key Process Physics and Parameters for Directed Energy Deposition

Important process parameters for the DED process include: nozzle-substrate offset distance, mass feed rate, laser power, laser spot size, travel speed and pause time. Adjusting these parameters allows for control of the amount of mass addition, mass capture, melt pool size and shape, defect formation, solidification structure, extent of solid-state phase transformations, and mechanical properties.

The mass feed rate is controlled by the powder feeder and process gases. It is typically measured in grams per minute. The powder feeder consists of a metering orifice which introduces volumes of powder into powder delivery tubes connected to powder nozzles. Mass feed rate is controlled by the rotational velocity of a drive motor. Process gases are broken into four classes: cover, shaping, carrier and nozzle. The primary processing gases controlling the mass flow rate are the cover and carrier gases with the cover gas having a larger influence [31].

The heat input is controlled by the laser power and the travel speed of the nozzle. Heat input is defined by:

$$Heat\ Input_{applied} = \frac{Laser\ Power}{Travel\ Speed} \quad (1)$$

Where P is the laser power in Watts and v is the travel speed in mm/s. Given equation 1, increasing laser power or a decreasing travel speed can increase the energy added to the melt pool for a given length of track. A decrease in travel speed can also increase mass flux if the process gas flow and powder mass feed rates are constant.

The shape of the nozzle and processing gases produce a converging-diverging powder stream as shown in Figure 9 [31]. The laser is also converging as it passes through the focusing lens. A change in the distance between the nozzle and substrate will affect the diameter of the laser, powder stream and the area in which they interact [32].

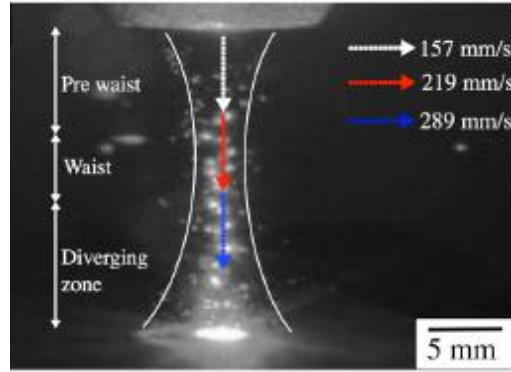


Figure 9: Converging-diverging powder stream shape during coaxial powder feeding in Directed Energy Deposition with particle velocities [31]

While a desired amount of mass may be targeted, the melt pool size, powder stream diameter, and amount of powder rejected from the molten pool will all affect the amount of mass captured by the melt pool during processing [33-35]. The melt pool size is determined by the laser spot size, laser power and travel speed.

Understanding the thermal history of the part is necessary for understanding the microstructural evolution and mechanical stresses within the part. Prior work in modeling of heat transfer and fluid flow in welding provides a starting point for the phenomena relevant in DED. This section aims to describe additional physics that must be accounted for in DED modeling as opposed to the modeling of welds.

During DED, the cover, carrier, and shaping process gases may act as a source of additional pressure on the melt and forced convection. Additionally, these gases act to shield the melt from surface active species reducing the complexity in modeling of the surface tension gradient that results in forced convection Marangoni flow.

The introduction of mass to the melt pool is the most impactful physical change in modeling of DED as opposed to prior work in autogenous welding. Powder particles, in addition to the mass they add to the system, can result in attenuation of the laser beam, absorption of energy from the laser, and additional impinging force on the melt in addition to the processing gases. Regarding the melting of particles, it is assumed that upon impact, the laser-particle interaction has heated the particles to the liquidus

temperature [36]. The combination of assuming that powder particles reach the liquidus temperature in flight and bring additional energy to the melt results in the conclusion that the addition of powder acts as a source of energy in addition to the laser. Models for attenuation and absorption of the laser are commonly included in DED modeling [11-13, 32, 34, 35, 37, 38]. This effect has also been seen in process calorimetry experiments as performed by Lia et al where calculated laser absorptivity decreases with powder addition [39].

It is known that while a powder feed rate may be prescribed by the process, not all this mass is captured to form the deposit. This is due to the area of the powder vs the area of the melt, the heat input to the system, and scattering of particles by the solid substrate surrounding the melt pool [33, 36]. The result is an additional parameter, capture efficiency, which must be defined or modeled to predict the deposit volume. Additional disruption of forced convection from particle impacts occurs, but its effects are not generally modeled [36].

A flow chart of much of the relevant physical phenomena during DED is presented in Figure 10. Heat input is affected by the type, dimension and travel speed of the laser, absorption by both the powder and substrate, attenuation due to the powder. The melt pool formed by this heat input is subject to the effects on conductive and convective heat transfer, losses to the surroundings due to convection and radiation, evaporation of metal due to superheating of the melt and wetting of the resulting melt with the substrate and gas. This melt pool is then subject to solidification theories and microstructural evolution which results in the properties resulting from the deposit. The DED process is very complex without a way to measure many of the relevant phenomena using in-situ measurements. Use of modeling can provide important insight into the process with selection of the phenomena relevant to the properties of interest.

### 2.2.2 Spatial and Temporal DED Process Considerations

With additional layers, the increase in z-height and pause between layers will affect processing of the next layer. Layer height is determined by the amount of material

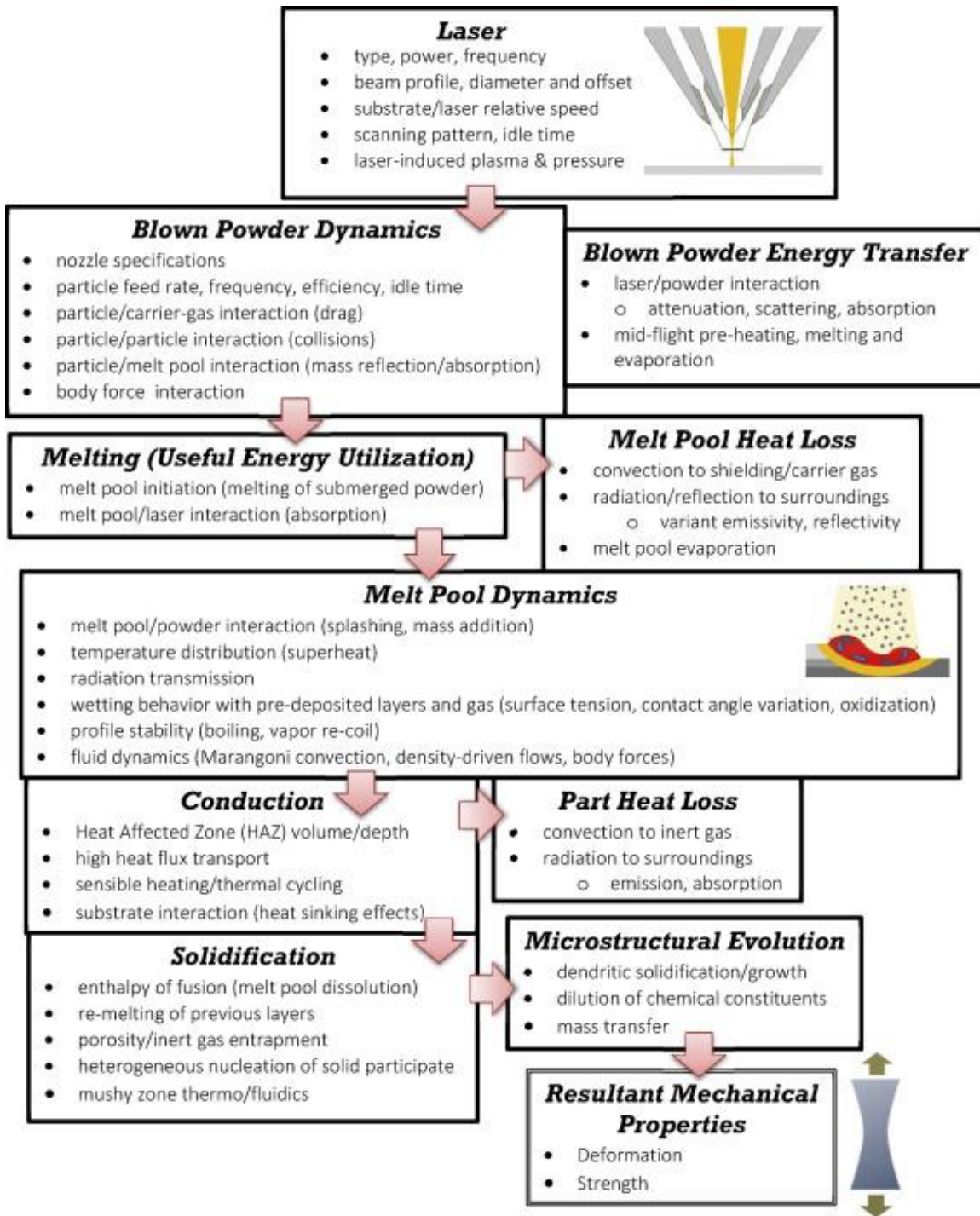


Figure 10: Flow chart showing relevant phenomena that occur during DED processing [36]

deposited on the previous layer. While many DED systems do not have active feedback loops to determine the layer height, it has been shown that the converging-diverging shape of the powder stream acts as a passive feedback system when the nozzle-substrate offset is maintained above the powder stream waist [32]. This offset will affect the amount of mass captured and laser spot size which will result in a change in the thermal conditions of the layer. Additionally, the pause time between layers will allow time for the substrate to cool. With an increase in the break between layers, the pause time, the substrate and prior layer will have additional time to cool. Over time, heat accumulation may increase the substrate temperature acting as an effective preheat. An increase in substrate temperature would result in a change in melt pool shape, size and mass capture.

### 2.3 Single-Crystal Solidification

During rapid cooling conditions seen in welding or additive manufacturing processes, solidification occurs epitaxially from partially melted grains. With reference to the crystallography of the substrate, solidification occurs along the direction most closely aligned with the highest thermal gradient. In most cubic materials including the face centered cubic (FCC) structure seen in nickel materials, this direction is  $\langle 100 \rangle$ .

Rappaz et al investigated the influence of crystallography on solidification using single-crystal welds [7]. With a known melt pool shape, a geometrical model was developed to describe the dendritic growth variations within the weld. Applying the minimum velocity (or minimum undercooling) criterion to a known melt pool shape of a single-crystal weld, the model relates favored growth direction, beam travel direction and direction of highest thermal gradient (the solidification front normal) to the selection of a dendritic growth variant [7]. Equation 2 is the governing equation of the dendrite growth model developed by Rappaz:

$$|\vec{v}_{hkl}| = |\vec{v}_b| \frac{\cos \theta}{\cos \varphi} \quad (2)$$

where  $|\vec{v}_{hkl}|$  is the dendrite growth speed with respect to the beam travel speed  $|\vec{v}_b|$ ,  $\varphi$  is the angle between solidification front normal and  $[hkl]$  direction, and  $\theta$  is the angle

between the solidification front normal and the beam travel direction. Schematically described in Figure 11, the model shows that only one growth direction will be active at any time which corresponds to the dendritic growth direction that most closely aligns with the solidification front normal [8].

This competition is true even in cases where a stray grain or misoriented growth occurs. As seen in Figure 12, solidification with a multitude of grains will initially begin epitaxially from partially melted grains. As solidification continues, competition between the most favorable growth variant results in certain growth variants of  $\langle 100 \rangle$  continuing to form while others are overtaken [29].

When welding along the (100) plane (in which the  $\langle 100 \rangle$  directions are aligned with the plane), dendritic growth is the same across the centerline (XZ plane) of the weld with a maximum solidification velocity equal to the beam travel speed. With rotations in the welding plane and direction, it is possible to achieve conditions where asymmetry of the weld across the centerline is not seen and solidification velocities exceed the beam travel speed. This effect was seen by Park et al in laser welds of Ni-base single-crystal welds of PWA 1480 as seen in Figure 13 [5].

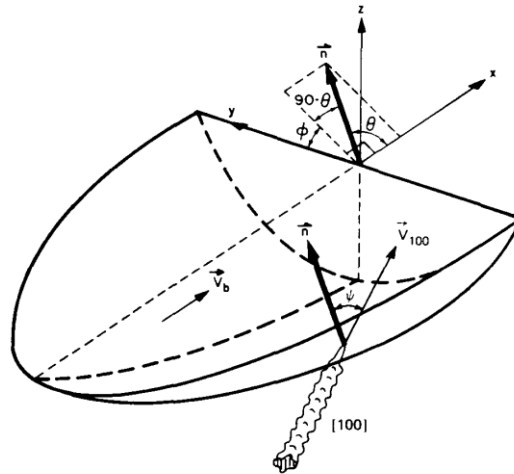


Figure 11: Dendritic selection during solidification of single-crystal material. Dendrite growth direction is determined by relationship between solidification front normal and favored dendrite growth directions [8]



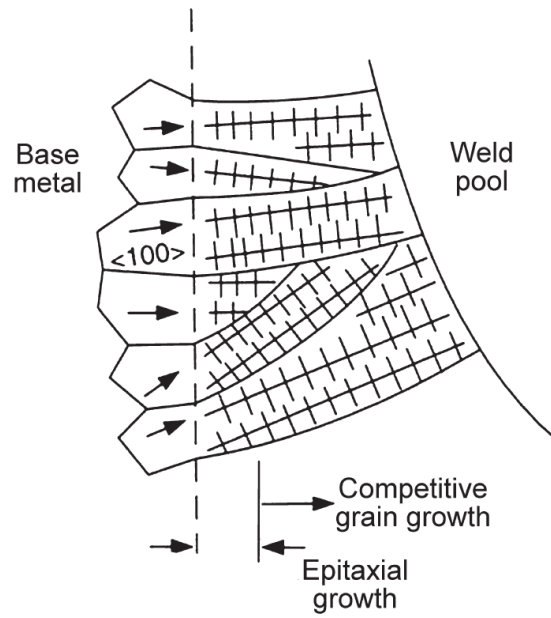


Figure 12: Competitive grain growth during weld solidification of a polycrystalline material. [28]

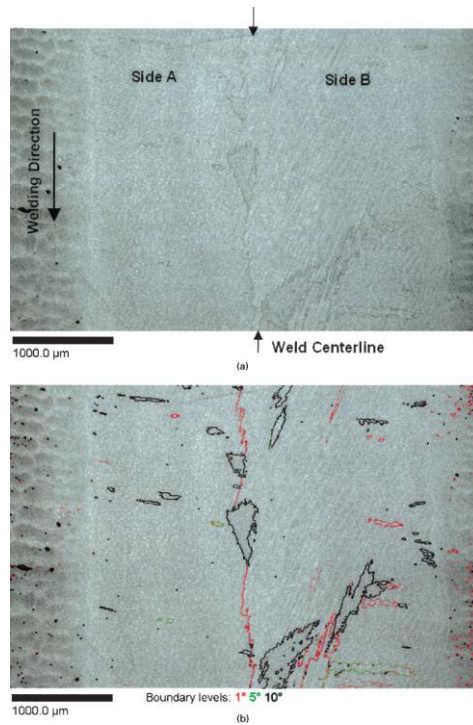


Figure 13: SEM imaging of top section of PWA 1480 asymmetric laser welds. Overlaid grain boundaries show unfavorable high angle grain boundaries at weld centerline. [5]



Understanding the active dendrite growth variant across the pool provides insight to the propensity for stray grain formation and cracking within the pool [4, 5]. For a given thermal gradient, an increase in the solidification velocity is directly related to an increase in the propensity for stray grain formation [4]. Additionally, it has been shown that at stray grains and the interface between dendrite growth variants increases the propensity for solidification cracking in conjunction with thermal stresses and the phases present [4, 5, 27]. While solidification cracking and its mechanisms are not within the scope of this work, it is important to understand the importance of eliminating stray grains and possible sources of cracking to produce acceptable deposit microstructures.

### 2.3.1 Thermo-Fluid Modeling of Single-Crystal Welds

Anderson et al looked to expand much of the earlier modeling work by Gaumann, Vitek, and others to include the effects of fluid flow [3]. These earlier works employed models neglecting the effect of fluid flow (usually using a Rosenthal solution) to determine the melt pool shape, thermal gradient and solidification velocity. The inclusion of fluid flow in modeling will result in changes to all three of these parameters [3].

Coupling a FEM thermo-fluid modeling with the dendrite growth variant model in three dimensions for laser welding produced similar trends as shown in prior work [3, 24, 26]. Stray grain content was shown to increase initially and then decrease with increase beam travel speeds. Increases in power were shown to result in an increase in stray grains. Incorporation of the dendrite growth variant model, which acts to limit the directions at which growth can occur, resulted in increases in solidification velocity but a decrease in thermal gradient. Temperature gradients were also lowest at the intersection between growth variants resulting in increased propensity for stray grain formation [3].

Using the assumption that these thermo-fluid simulation results are more accurate than the use of the conduction only Rosenthal solution models used by Gaumann, recalibration of the CET model for CMSX-4 was also performed [3]. Gaumann calibrated his model by comparing experimental results to simulations using the Rosenthal solution. Using data from new thermo-fluid modeling conducted,

Anderson's recalibrated CET model showed a small decrease in the nucleation site volume, increase in the fitting parameter  $a$ , and an increase in the material constant  $n$  [3]. In the literature review performed in this work, these CET model calibration values were not used outside of Anderson's work.

## 2.4 Columnar-to-Equiaxed Transition

To understand the formation of columnar and equiaxed grains, one must understand the role of thermal gradient ( $G$ ) and solidification velocity ( $R$ ). The formation of columnar and equiaxed grains influences the mechanical strength, especially creep properties, of the resulting material. In welding, laser, and electron beam additive manufacturing, the solidification velocity is defined by the melt pool shape and beam travel speed. The temperature gradient is influenced by heat flux which is a function of the heat source power density distribution, mass flux, travel speed, substrate geometry, and material thermophysical properties.

During welding or laser additive manufacturing, a range of grain morphologies can be achieved in which columnar and equiaxed grains may form upon solidification [29]. The substructure of these grains can also manifest itself in a wide range of structures from planar to columnar dendritic to equiaxed dendritic [29]. Solidification substructure selection can be understood through the concept of constitutional supercooling.

### 2.4.1 Constitutional Supercooling and Solidification Substructure

Constitutional supercooling occurs as solute is rejected from the solid upon solidification. This effect can be schematically seen through evaluation of the phase diagram, as shown in Figure 14 (a). The increase of solute in the liquid depresses the liquidus temperature in the melt, as shown in Figure 14 (b) and (c). With a high thermal gradient, solidification occurs along a planar front as any protrusions that form along the solidification front encounter liquid above the melting temperature which is unstable. With decreasing thermal gradient, protrusions on the solidification front encounter liquid below the melting temperature and continue to grow. This constitutional supercooling

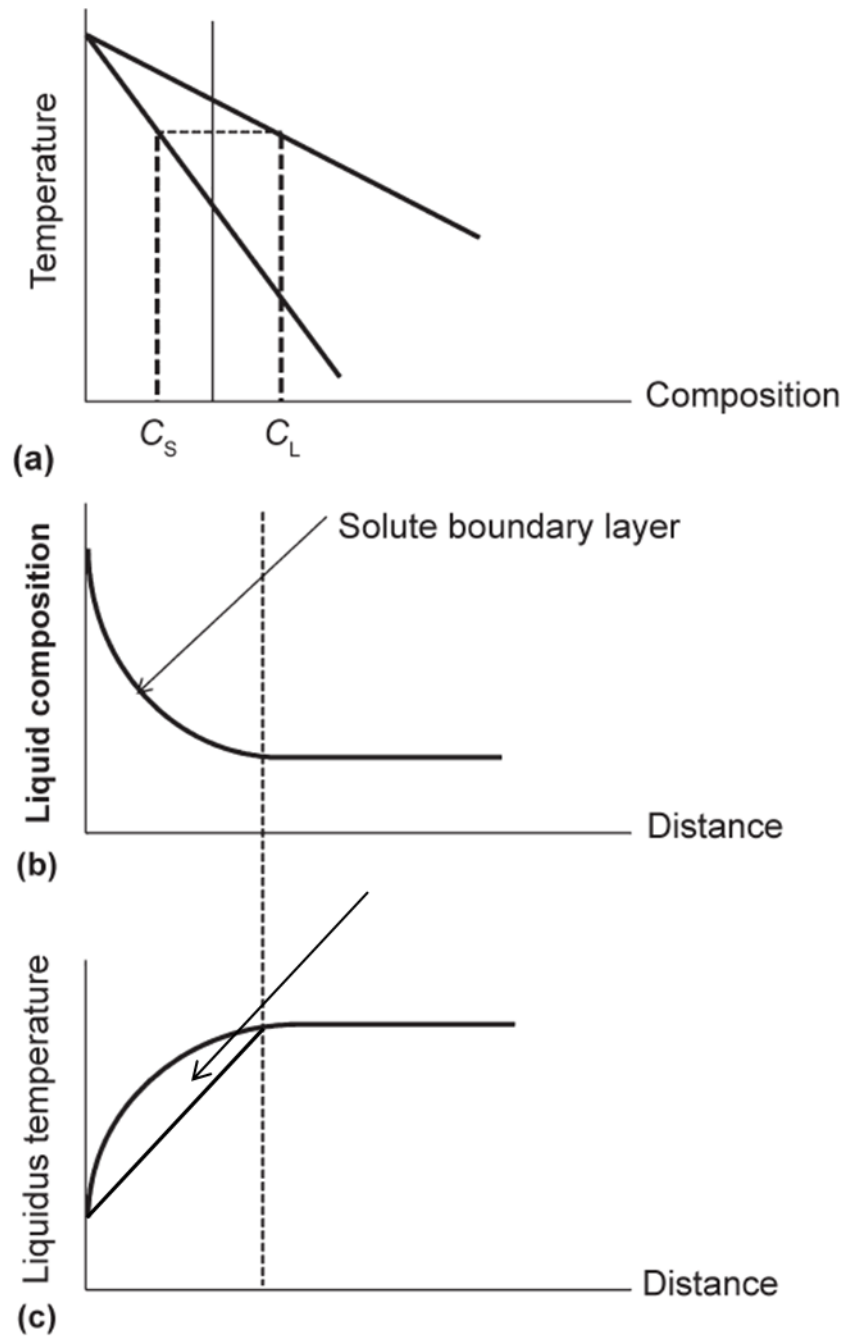


Figure 14: Schematic of constitutional supercooling showing solution rejection during solidification (a), solute boundary layer (b), and the resulting depression of the liquidus temperature (c) [29]

results in breakdown of the planar interface and dendritic growth is now favored. With an even lower thermal gradient, solidification may occur in front of the solidification front resulting in equiaxed dendritic growth [29]. The type of substructure that occurs can be directly related to the G-R combination within the molten pool, as shown in Figure 15.

#### 2.4.2 Columnar-to-Equiaxed Transition Model

The concept of constitutional supercooling can be further adopted to understand the formation of the solidification microstructure and the transition from columnar growth to equiaxed growth. The first columnar-to-equiaxed transition (CET) model was developed by Hunt [40]. Hunt showed that once a critical value of constitutional supercooling was reached, nucleation of equiaxed grains in front of the solidification front resulted in equiaxed growth. Depending on the amount of equiaxed grains in front of the solidification front, three steady state growth conditions can occur: fully columnar, fully equiaxed, or a mixed state of the two [40]. Gaumann further developed this model to include high solidification rates seen in laser welding through application of the Kurz-Giovanola-Trivedi (KGT) model for dendrite growth during rapid solidification [41].

The present form of the CET equation is shown in equation 3:

$$G = \frac{1}{n+1} \sqrt[3]{\frac{-4\pi N_0}{3 \ln(1-\Phi)}} \Delta T \left(1 - \frac{\Delta T_n^{n+1}}{\Delta T^{n+1}}\right) \quad (3)$$

where  $N_0$  is the nucleation volume density,  $\phi$  is the volume fraction of equiaxed grains,  $\Delta T$  is the dendrite tip undercooling for columnar growth,  $n$  is a material dependent parameter, and  $\Delta T_n$  is the undercooling for equiaxed grain nucleation. This form of the equation relates the thermal gradient to the volume fraction of equiaxed grains that may be formed under rapid solidification conditions. To calculate the equiaxed nucleation undercooling value, Gaumann used the relationship  $\Delta T_c = (aV_s)^{\frac{1}{n}}$  [41]. This realization resulted in parameters  $a$  and  $n$  serving as fitting parameters in Gaumann's CET model [9]. This relationship was developed by making simplifying assumptions to the dendrite tip undercooling based on the KGT theory. From this equation, it is possible to plot the

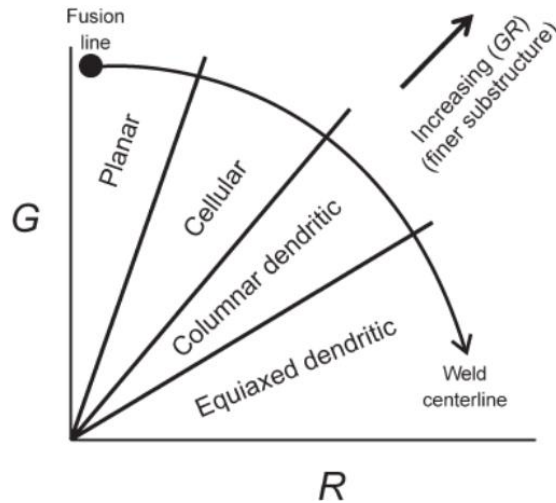


Figure 15: Influence of thermal gradient ( $G$ ) and solidification velocity ( $R$ ) on solidification substructure. Low  $G$  and high  $R$  results in formation of equiaxed grains in place of directional dendrite growth [29].

CET curves (columnar, equiaxed and mixed regions of growth) as a function of  $G$ - $R$ . A representative plot of the CET curves is shown in Figure 16.

#### 2.4.3 Sensitivity of CET Model and Effect of Processing Conditions

Haines et al performed a sensitivity analysis of the CET model evaluating both compositional and model parameters [42]. This work eliminated the fitting parameter  $a$  by using the KGT model to predict the undercooling values for columnar and equiaxed growth directly rather than the simplifying relationship used by Gaumann [41]. The sensitivity analysis concluded that the nucleation volume density ( $N_0$ ) has a significantly larger effect on the CET curves than the other model or compositional parameters [42].  $N_0$  cannot be directly measured and is generally a correction factor calibrated against experimental results. Additionally,  $N_0$  varies greatly and is a function of the process, environment and material among other factors.

Recent work has shown that by adjusting processing parameters, fully columnar or fully equiaxed material can be formed. Dehoff et al showed in the electron beam melting (EBM) additive process that changes in the processing parameters (and thus changes in  $G$  and  $R$ ) and scan strategies locally allowed for selection of columnar and

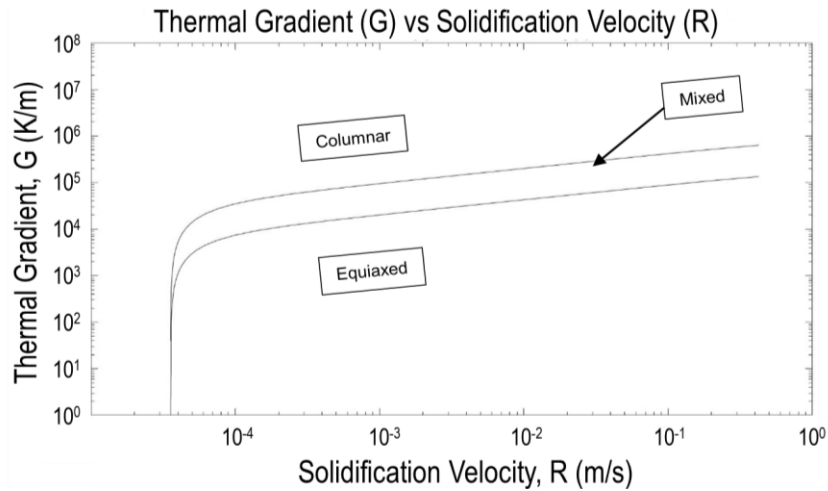


Figure 16: Representative columnar-to-equiaxed transition curves. Lower curve corresponds to equiaxed-to-mixed transition line while upper line corresponds to mixed-to-columnar transition line [42].

equiaxed material spatially within the part [43]. This effect can be seen through electron backscatter diffraction (EBSD) image in Figure 17. While this effect was achieved in the Ni superalloy Inconel 718 evaluated by Dehoff, there is no guarantee for a given material that parameter sets that result in columnar or equiaxed growth will also produce fully dense parts.

## 2.5 Combining Single-Crystal Solidification and CET Models

Combining the single-crystal solidification and CET models yields a deeper understanding of the evolution of texture and stray grains during solidification. For both decreases in thermal gradient or increases in solidification velocity, G-R plots for the CET show a shift towards mixed or equiaxed growth. Additionally, it has been seen in evaluation of single-crystal welds using the dendrite growth variant model, that the weld pool shape directly influences the solidification velocity. To fully understand the propensity for a stray grain to form at a given location within the pool, understanding and combining these two theories is necessary.

Work by Yoshihiro et al on surface remelted CMSX-4 showed that processing conditions that would result in high thermal gradient and solidification values allowed for

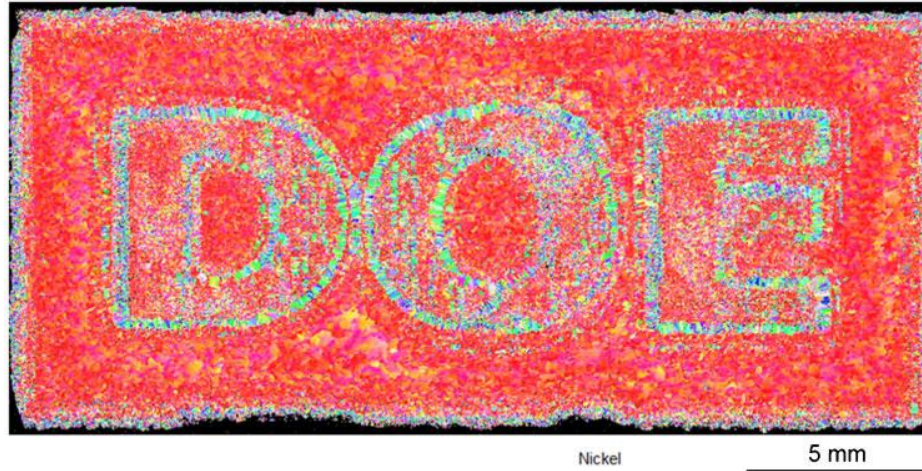


Figure 17: Electron backscatter diffraction image of part produced EBM showing spatial changes in processing parameters, G and R resulting in areas of columnar and equiaxed growth [43]

maintenance of the single-crystal substrate upon solidification. With decreasing heat input, the melt pool depth decreases and the shape of the pool changes. It can be seen that the depth and resulting shape of the pool plays an effect on the formation of stray grains as seen in Figure 18 [23].

Gaumann applied the CET model to the dendrite growth variant in experiments with CMSX-4 single-crystal deposits using epitaxial laser metal forming (E-LMF) [24]. Deposits were made with the  $\langle 100 \rangle$  normal to the surface. A modified Rosenthal approach was used for calculation of the melt pool shape, thermal gradients and dendrite solidification velocity. The change in G, R and propensity for stray grain formation were analyzed as a function of depth along the centerline of the melt. Propensity for stray grain formation was evaluated as  $K = G^n/R$  which is derived from the equation for the CET. In this form, a critical value of K ( $K_{crit}$ ) exists where the CET occurs [24].

Figure 19 shows the change in solidification velocity (a), thermal gradient (b) and K (c) with melt depth. At the base of the melt, the solidification velocity is low and the thermal gradient high with respect to the top of the melt. At the top of this melt, there is a loss of in epitaxial growth and an equiaxed region forms to the surface, as shown in



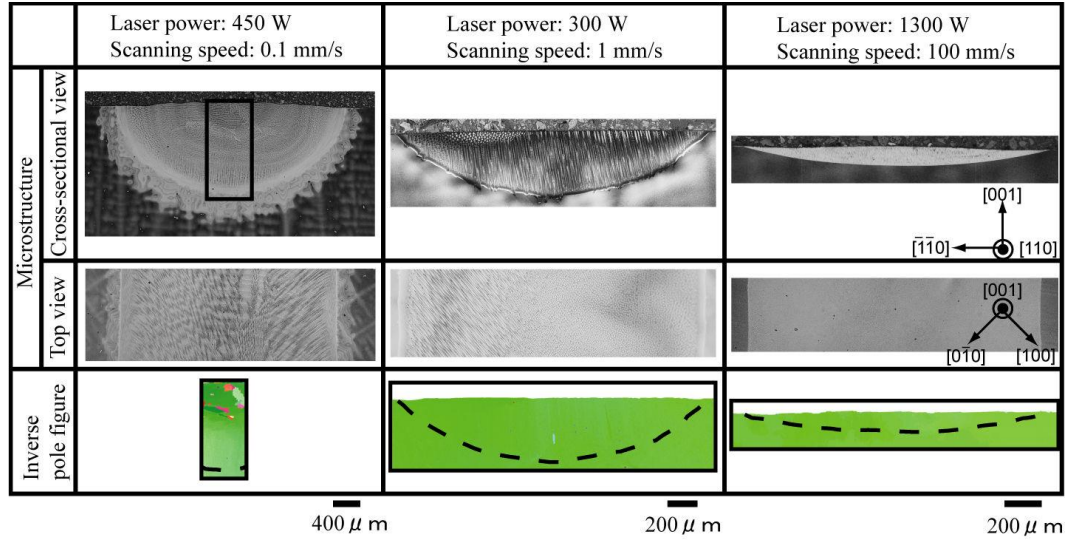


Figure 18: Microstructure and crystallographic orientation in laser surface-melted CMSX-4. Lower melt depths show less stray grain growth [23]

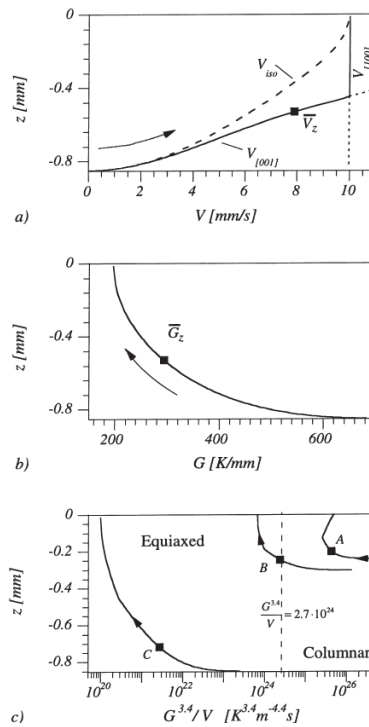


Figure 19: Change in solidification velocity (a), thermal gradient (b) and propensity for stray grain formation (c) as a function of melt depth accounting for both columnar-to-equiaxed transition and dendrite growth variant models [24]



Figure 20. Additionally, it is shown that with an increase in preheat temperature, the propensity for equiaxed growth increases as shown in Figure 21 [24]. The result is especially impactful for multi-layered deposits where residual heat in the previous layer will act as an effective preheat.

In a similar theoretical analysis with a wider range of processing parameters and welding orientations, the effect on crystallography was evaluated [26]. This study used the same analysis for calculation of melt pool shape, solidification velocity and thermal gradient. As shown in Figure 22, neglecting the dendrite growth variant model results in an underestimation of the stray grain area fraction.

## 2.6 Thermo-Fluid Modeling by Finite Volume Method

The concepts of dendrite growth variants and columnar-to-equiaxed transition show the importance of accurate determination of the melt pool shape, thermal gradient and solidification velocity. Thermal gradient and solidification velocity are time dependent effects that are difficult to measure in-situ or impossible for portions of the subsurface molten pool. Thus, the need for modeling of these phenomena is vital. In

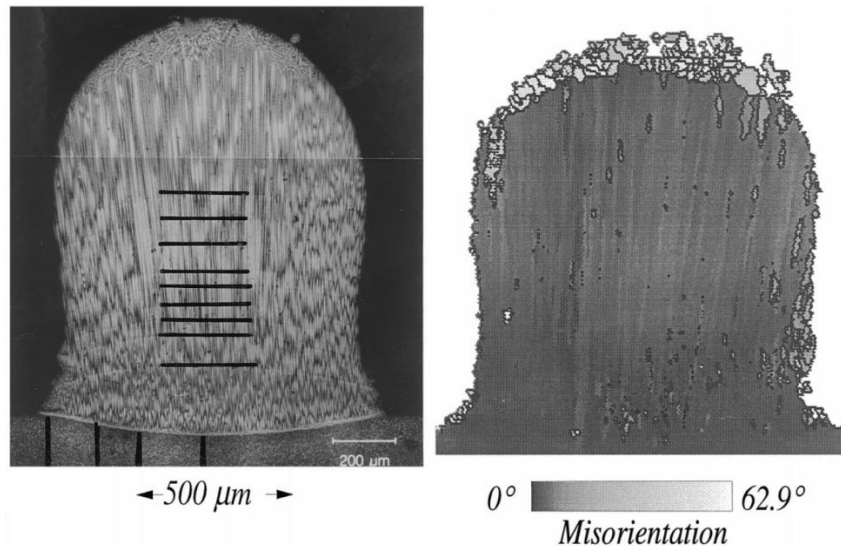


Figure 20: Optical micrograph (left) and EBSD (right) of the formation of multi-layered deposit on single-crystal substrate showing a misoriented equiaxed cap which forms at the surface. [25]

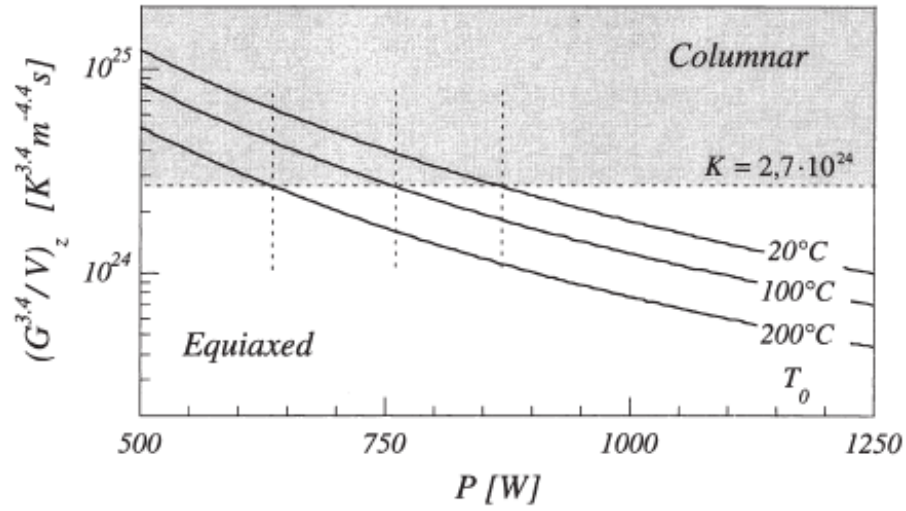


Figure 21: Increased propensity for equiaxed grain formation with increasing substrate preheat [24]

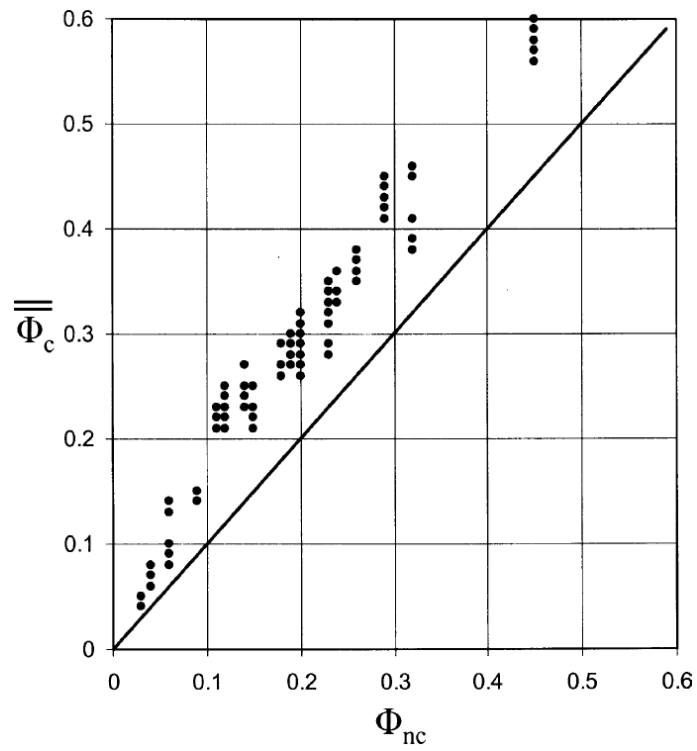


Figure 22: Effect of the inclusion of the Rappaz dendrite growth variant model on the area fraction of stray grains. The y axis is the area fraction with the dendritic growth variation model active while the x axis shows this value without the Rappaz model active [26]

this work, an OpenFoam based continuum computational fluid dynamics (CFD) finite volume method (FVM) thermo-fluid model developed by Coleman et al was used [44]. This model allows for calculation of the melt pool shape, thermal gradient and solidification velocity while accounting for the substrate shape, laser spot size and fluid flow which are important in modeling the phenomena in this work. Validation and calibration of the model was performed using experimental data in this work and alterations in model inputs for laser absorption, cell size and computation time step.

### 2.6.1 Conservation Equations

In a thermo-fluid model, the effects of heat transfer by fluid convection within the molten region and conduction through the solid and liquid are accounted for and solved using the conservation equations of mass, momentum and energy. These equations cannot be solved directly, and an equation of state is needed. For incompressible flow, an equation of state describing the pressure is developed from combining the continuity and momentum equations [45]. The conservation of mass, momentum and energy in base SI units using a continuum modeling approach are shown in equations 4, 5 and 6 respectively [44].

$$\frac{\partial \rho}{\partial t} + \nabla \cdot (\rho U) = 0 \quad (4)$$

$$\frac{\partial(\rho U)}{\partial t} + \nabla \cdot (\rho U U) = \nabla \cdot (\mu \nabla U) - \nabla p + S_U \quad (5)$$

$$\frac{\partial(\rho c_p T)}{\partial t} + \nabla \cdot (\rho c_p U T) = \nabla \cdot (K \nabla T) + S_T + \dot{Q} \quad (6)$$

where  $\rho$  is the density,  $U$  is the velocity vector,  $t$  is time,  $p$  is pressure,  $\mu$  is dynamic viscosity,  $c_p$  is the specific heat,  $T$  is temperature,  $k$  is the thermal conductivity,  $S_U$  is the momentum source term which accounts for thermal buoyancy and drag in the mushy zone, and  $S_T$  is the temperature source term which accounts for latent heat during

phase change.  $Q$  is the heat source which can be depicted as a volumetric, point or areal source [44].

### 2.6.2 Heat Source Model and Boundary Conditions

Goldak et al argues that the heat source in laser welding effectively applies energy to a volume of material [46]. The double ellipsoidal volumetric heat source proposed by Goldak, and adopted here, assumes a Gaussian heat flux where energy is distributed over a finite penetration depth. This model provides a large amount of flexibility as length, width, depth and the relative amount of heat flux in a given quadrant can all be varied. In this work, a spherical cross section is used comparable to the heat source used during experimentation [46].

Forced convection due to Marangoni effects, heat extraction by radiation and by forced convection are accounted for through applied boundary conditions. Forced convection is present during Directed Energy Deposition due to processing gases impinging on the molten pool. Marangoni flow is the result of the variation in surface tension due to temperature and surface-active species [47]. Flow always moves from an area of low tension to high tension [47]. The variation in surface tension,  $\frac{d\sigma}{dT}$ , is known as the surface tension or Marangoni coefficient.

Three conditions are possible for the surface tension coefficient; positive, negative and positive-to-negative. These conditions are schematically described in Figure 23 [47]. With a positive coefficient, fluid moves towards the symmetry plane of the molten pool. With a negative coefficient, fluid moves from the symmetry plane towards the periphery of the molten pool. A positive-to-negative coefficient is possible with surface active species or chemical differences across the melt surface. In this condition, separate vortexes are formed due to fluid flow switching directions as indicated by the change in sign of the surface tension coefficient [47].

### 2.6.3 Finite Volume Discretization

To linearize the equations for ease of computational solution, the conservation equations in the integral form are discretized [45, 48]. One such discretization method is

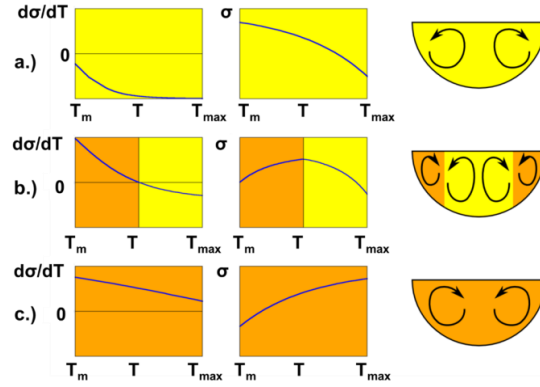


Figure 23: Fluid flow direction due to Marangoni flow as a function of the surface tension coefficient. Image a shows a negative Marangoni coefficient, b shows a positive-to-negative Marangoni coefficient, and c shows a positive Marangoni coefficient. [37]

the finite volume method. This method makes use of a grid consisting of numerous voxels of finite size which make up the entirety of the part. This allows for the conservation equation to be reduced to a series of linearized conservation equations which can be readily solved computationally. For a small increment in time, this approach approximates the exact solution [48].

With increasing the number of voxels in the modeling domain and decreasing the time step, the accuracy of the solution increases [48]. Each approach comes with a cost as increasing the size of the domain increases the number of equations necessary to describe the domain while decreasing the time step increases the number of times the series of equations must be solved. Thus, the time step, domain size and computation time must be considered. Generally, the time step and domain size are determined by the point of diminishing returns, where increasing the accuracy of the solution no longer outweighs the computational cost.

To determine the mesh size necessary for accuracy of measurements (i.e. melt pool dimensions) and calculated values (i.e. temperature, fluid flow velocity, etc.) for a three-dimensional simulation, a mesh sensitivity study is conducted. In this study, the time step and, generally, the cell aspect ratio are maintained constant while the size of

the voxel is reduced [44]. This process is continued until the change in accuracy with a reduction in voxel size is insignificant with respect to the computational errors and the expected accuracy necessary to resolve key features.

To improve computation time, the area in which melting of the domain occurs is refined to a smaller voxel size, as defined by the mesh sensitivity study, while the number of voxels and their size is increased away from the area of interest. Figure 24 shows an example mesh where the voxels within the area of interest are refined to a smaller dimension while away from the area of interest the voxels are enlarged.

Further improvement of the computational time can be achieved from adjustments to the time step. Increasing the time step, while still achieving convergence, allows for a reducing in computational time. For an FVM problem, the convergence limit is governed by the Courant number as shown in equations 7 and 8.

$$Co_U = \frac{U\Delta t}{\Delta x} \leq 1.0 \quad (7)$$

$$Co_T = \frac{u_b\Delta t}{\Delta x} \leq 1.0 \quad (8)$$

In equations 7 and 8,  $Co_U$  is the momentum Courant number,  $Co_T$  the thermal Courant number,  $U$  the fluid velocity,  $\Delta t$  the simulation time step,  $u_b$  the beam travel speed, and  $\Delta x$  the size of the voxel parallel to velocity term. The momentum Courant number, defined in equation 7, relates the voxel size to the distance in which fluid moves within a single time step.

For conservation of mass to be maintained, the fluid movement in a given time step cannot exceed the size of the voxel which equates to a Courant number greater than 1. For convergence of the momentum and mass conservation equations, the Courant number must be less than 1. The thermal Courant number, defined in equation 8, describes the ability of the thermal field to resolve the motion of the laser. Like the momentum Courant number, a thermal Courant number greater than 1 indicates that the time step is too long to resolve the motion of the beam. Since the beam travel speed is generally lower than the fluid velocity in the melt, the momentum Courant number

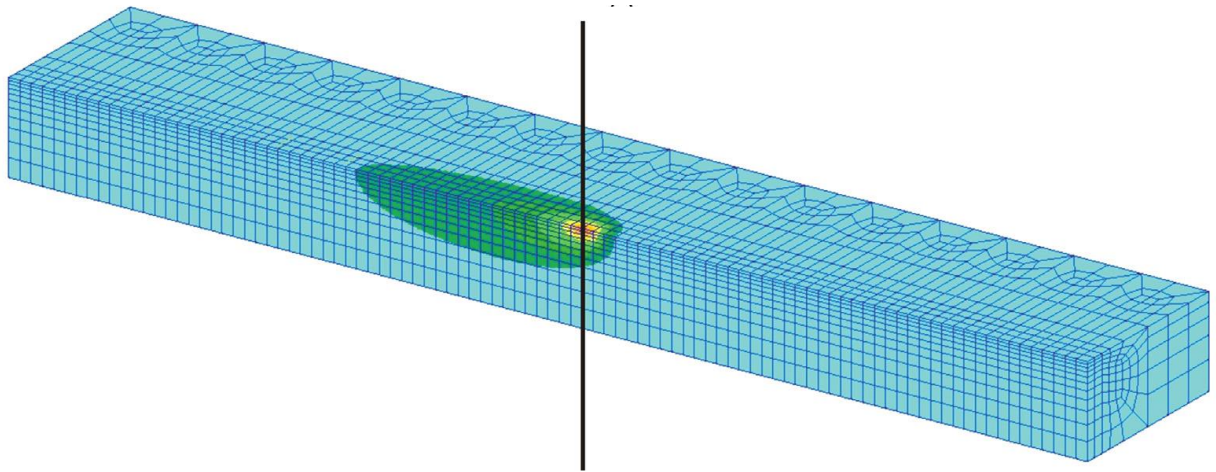


Figure 24: Example of a refined FEM mesh near centerline for a welding simulation [49]

determines the maximum time step allowed. In practice, a value less than unity (0.5) is generally used to ensure convergence and accuracy of the solution.

#### 2.6.4 Modeling of Directed Energy Deposition

Solving the temperature field during DED can be performed using many of the same modeling methods used for welding including the use of analytical models. While superior in computational time, use of these analytical models becomes more difficult during simulations of DED as the boundary conditions become more complicated with the addition of powder [36]. These types of simulations usually employ some form of the Rosenthal solution which includes the assumption of a semi-infinite plate heat transfer condition. This will cause problems when solving for thin walls as this assumption breaks down. Still, some authors have also used this approach to calculate the temperature field of the substrate to feed analytical models for the deposit height for build height predictions with success [37]. In spite of their limitations, these models can provide a starting point for modeling the DED process and important insight to the thermal history of the process [36].

Finite element models (FEM) allow for near-edge heating cases to be modeled such as thin wall builds seen during DED. These finite element models allow for the

thermal history within the deposit to be evaluated as well. Modeling of the deposit can be broken into three types: cell activation schemes, analytical models and free-surface models. Cell activation schemes prescribe cells to be used for modeling the solidification of the deposit. Upon solidification of the melt, the cells which serve as the deposited material are activated [49]. This type of model has the advantage over substrate only heating models in that they allow for thermal history of the deposit to be modeled as well. However, the deposit height must be preset by the user and approximates the true deposit shape as a flat top.

Increasing in complexity are analytical models which calculate the deposit shape and apply this to a FEM substrate. Variations of this model exist including models where the deposit is calculated using a geometric model coupled with processing parameters, substrate heating only and where the deposit is solved iteratively by initially calculating the deposit from substrate heating and converging to a solution with the deposit included [32, 50]. Knapp et al use a geometry model coupled with physical phenomena, calculated as a function of the processing parameters, to calculate the shape of the deposit and melt pool with good agreement. The deposit shape is approximated as a cylinder with an ellipsoidal front which is valid if the deposit width is less than the substrate width [35].

Analytical approaches to deposit shape provide a less computationally intensive scheme to calculate the more complex phenomena occurring during DED, improving upon cell activation or substrate heating only schemes.

Calculation and application of the deposit shape to the original substrate allows for more a more accurate calculation of the fluid flow and the calculation of the deposit shape. With calibration, this allows for prediction of deposit size, shape and volume without production of samples. Fluid flow can be better predicted with modeling of the deposit shape as the direction of forced convection by Marangoni forces will be properly applied along the curved surface of the deposit as opposed to the flat surface of the original substrate.



The most intensive models computationally and complex physically are free-surface models. Free-surface models track the liquid-gas interface using mathematical functions, such as the level set and volume of fluid method, and use of physical phenomena, such as the addition of mass into the mass conservation equation, surface tension and gas pressure forces, to solve for the buildup and motion of the liquid interface. These models require the use of a CFD scheme and usually are performed with a substrate larger than the resulting deposit width. With calibration, these models theoretically allow for an even more accurate prediction of the deposit shape, fluid motion and heat transfer.

DED free-surface models, such as the ones developed by Wen, Liu and Song, allow for predictions of track geometries [11-13, 38, 51]. Additionally, the models developed by Liu and Song can predict the thermal gradients and solidification velocities that are necessary for understanding the microstructure of the part [11-13, 38]. These models come at a heavy computational cost and limit their use to shorter time scales than that of the analytical, substrate only heating, and cell activation schemes described in this section.

As reviewed by Debroy, Table 1 shows a summary of the different types of models used in AM and the important features [52].

### 2.6.5 Modeling of Single-Crystal Deposits during Directed Energy Deposition

Modeling of the DED process for single-crystal repair has been conducted by Liu et al [11-13] and Liang et al [10]. Each take a different approach to understanding the effects of DED processing parameters on single-crystal solidification.

Liang et al take the approach of using an analytical model for processing maps to understand the evolution of the single-crystal microstructure with changes in processing parameters. In much of the literature on DED for single-crystal nickel superalloy repairs, the influence of the addition of powder is neglected. Additionally, use of an analytical model allows for a broader parameter sweep to be conducted in a shorter period of time. The result is a more populated processing map that Liang argues is more informative. The analytical model uses the CET model developed by Gaumann coupled

Table 1: Comparison of current modeling approaches which include heat and fluid flow calculations in additive manufacturing [52]

| Approaches                                                               | Features                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             |
|--------------------------------------------------------------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Analytical approach                                                      | <ul style="list-style-type: none"> <li>Analytically solves Rosenthal's heat conduction equation.</li> <li>Outputs are temperature fields, build dimensions and cooling rates.</li> <li>Computationally less expensive, simplified and easy to use.</li> <li>Ignores the dominant mechanism of heat transfer and known to produce large errors.</li> <li></li> </ul>                                                                                                                                                                                                  |
| Heat conduction models using finite element method (FEM)                 | <ul style="list-style-type: none"> <li>Solves steady state or transient energy conservation equation with convective and radiative boundary conditions.</li> <li>Outputs are 3D steady state or transient temperature distribution and build shape and size.</li> <li>Many existing software packages, easy to implement, can handle intricate geometries.</li> <li>Does not consider the effects of convective flow of liquid metal inside molten pool on the temperature field, therefore severely overestimates the peak temperature and cooling rate.</li> </ul> |
| Heat transfer and fluid flow models using finite difference method (FDM) | <ul style="list-style-type: none"> <li>Solves 3D transient conservation equations of mass, momentum and energy.</li> <li>Outputs are 3D transient temperature and velocity distributions, build shape and size, solidification parameters.</li> <li>Considers the effects of molten pool flow inside pool and therefore provides accurate temperature distribution.</li> <li>Often assumes flat top geometry of the deposit to make the calculations tractable.</li> </ul>                                                                                           |
| Level set method                                                         | <ul style="list-style-type: none"> <li>Tracks the free surface of the molten pool.</li> <li>Outputs are 3D temperature and velocity distribution of the deposit with free curved surface.</li> <li>The calculated deposit shape and size agree well with experiments.</li> <li>Computationally intensive and tends to suffer from non-conservation of mass.</li> </ul>                                                                                                                                                                                               |
| Volume of fluid (VOF) using finite difference method (FDM)               | <ul style="list-style-type: none"> <li>Tracks the free surface of the molten pool.</li> <li>Outputs are 3D temperature and velocity distribution of the deposit with free curved surface.</li> <li>Computationally intensive.</li> <li>Mass conservation maintained but at less sharp interface than LSM.</li> </ul>                                                                                                                                                                                                                                                 |

with an analytical model to calculate the heat transfer and consequently G and R. The addition of powder will alter the melt pool shape and, in the case of unmelted powder particles, increase the number of nucleation sites in the melt [10].

To compensate for these powder effects, a process parameter dependent model for  $N_0$  is developed relating  $N_0$  to heat input,  $\rho_{lp}$ , and linear powder feed rate,  $\rho_{lm}$ . The resulting equation is shown in equation 8 [10].

$$N_0 = N_1 \rho_{lp}^q (1 + N_2 \rho_{lm} + N_3 \rho_{lm}^2) \quad (8)$$

Calibration of the model was performed by measuring the point in the deposit where the CET occurred and using the analytical model to determine the G and R at that location [10].

Liu et al use a free-surface finite element model to predict the deposit shape, heat transfer and fluid flow in the melt. Directionally solidified samples were produced for evaluation of the free-surface model. The free-surface model accounts for attenuation of the laser by a Gaussian powder cloud, energy brought to the melt by powder heating, and uses a level set method to track the free surface and determine the deposit shape. The numerical model shows that with increasing layers, the residual heat in the previous layers results in an increase in the fluid flow velocity and melt depth of the new deposit [13]. The result is a higher propensity for remelting of an equiaxed cap. In later versions of this model, the addition of a combined CET and dendrite growth variant model is implemented in calculation of G, R and where stray grain (or equiaxed) growth will occur [11].

This model is able to predict the growth variants and stray grain areas for single-track and multi-track and hatched samples [11-13]. However, it does not account for the addition of powder and uses the CET parameters calculated by Gaumann for CMSX-4 for Rene N5 deposits on a directionally solidified DZ125 substrate.

## 2.7 Background Summary

Prior work has looked at single crystal solidification in autogenous welding, epitaxial laser metal forming and directed energy deposition on thick plate substrates [3,

5, 10-13, 21-26, 53, 54]. In processes with material addition, has been shown with a thick plate substrate that single crystal material can be produced with proper processing parameter selection [10-13, 21, 22, 24, 25]. In multi-layered deposit, remelting of the equiaxed cap was found to be the primary factor determining the maintenance of epitaxial growth [24, 25].

Prior work on single crystal solidification has incorporated process modeling to provide additional insight into the solidification conditions, namely the thermal gradients ( $G$ ) and solidification velocities ( $R$ ) [3, 10-13, 21, 22, 24, 25, 53]. Variations in  $G$  and  $R$  can result in a transition from single crystal/directionally solidified to equiaxed material. Incorporation of the Rappaz dendrite growth variant model and fluid flow (CFD modeling) have been shown to increase the accuracy of these processing models [3, 26]. A variation in  $N_0$  with processing conditions has also been discussed and a preliminary model produced [10]. Models for directed energy deposition have been created incorporating the addition of material through level set, volume of fluid, and analytical techniques [10-13, 32, 34-38, 49, 50, 55]. The effects of powder particles are incorporated through laser attenuation and absorption of energy by particles in flight.

This work looks to build upon prior work and evaluate the effects of a change in geometry on epitaxial growth through CDF modeling and design of experiments. The effects of powder particles on the formation of the deposit and resulting crystallographic texture is evaluated. The validity of neglecting powder melting upon impact with the melt, a common modeling technique, is called into question. The variation in  $N_0$  during processing is evaluated and the model build by Liang is expanded [10].

## Chapter Three: Technical Approach and Evaluation Methods

This Chapter will discuss the substrate preparation and powder used (Section 3.1), experimental setup and characterization techniques. Figure 25 shows a hierarchical structure detailing the experimental approach used to evaluate the influence of varying key process parameters on texture evolution. Of the four experimental setups evaluated, two test cases were considered including single-track and multi-track deposits. These cases served as a means to calibrate the thermo-fluid model and experimentally assess deposit geometry microstructures for a range of laser powers, travel speeds, and mass fluxes. Heat source proximity relative to the edge of the substrate was considered by varying substrate geometry. Single-track deposits were completed on thick plate and thin wall geometries. Thick plate condition included a single line deposits trial, in which laser power and travel speed were varied between passes, and a variable travel speed (spiral) trial, in which the travel speed is constantly varying at a constant laser power. The thin wall condition included through section heating. Single-pass and multi-pass (ten layers) samples were produced in the thin wall geometry to assess the effect of processing parameters and the time dependent changes in deposit microstructure and shape due to additional layers.

### 3.1 Substrate Preparation and Powder

All substrates and powder used were single-crystal, nickel superalloy (> 5 wt% Al, < 0.1 wt% C). Substrates were inspected prior to deposition to ensure crystal orientation was parallel and perpendicular to [100]. All substrates were subsequently machined from single-crystal castings using electro-discharge machining (EDM) and any recast layer was removed prior to deposition.

### 3.2 Variable Travel Speed Trial with and without Mass Addition

Table 2 summarizes the samples and respective process parameters used to complete the variable travel speed experimental trials. Deposition was completed along a spiral path achieved by rotating the substrate while positively translating the laser along a single axis of motion. The experimental design is similar to the spiral trace used

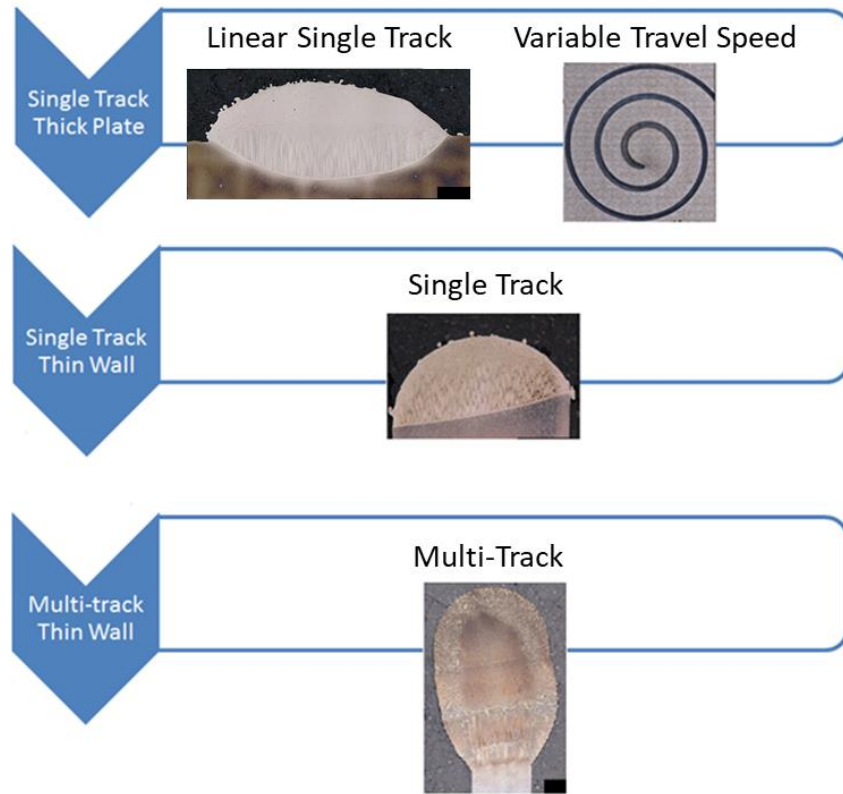


Figure 25: Schematic of hierarchical structure to experimental methods which systematically address varying DED process parameters, substrate geometry, and deposit geometry.

Table 2: Variable Travel Speed (Spiral) Sample Processing Parameters

| Sample ID | Normalized Power | Mass Addition |
|-----------|------------------|---------------|
| A         | 1.5              | No            |
| B         | 3.2              | No            |
| C         | 5.3              | No            |
| D         | 7.4              | No            |
| E         | 1.5              | Yes           |
| F         | 3.2              | Yes           |
| G         | 5.3              | Yes           |
| H         | 7.4              | Yes           |

by Fukumoto et al in evaluation of the microstructure of an Fe-Cr-Ni alloy [56]. Spirals consisted of three counterclockwise rotations at constant angular velocity. All samples were produced in an argon rich atmosphere ( $< 100$  ppm oxygen). Spiral deposits without mass addition were produced using four different laser powers. Two replicates were produced for each laser power considered with mass addition. Powder mass feed rate was constant for all experimental trials utilizing feed stock. Deposits were completed using a convergent laser beam except for samples C and D. A divergent laser beam was used in these cases due to the risk of back reflection and potential for damage to the laser optics. Transverse cross sections were removed by EDM at 135 and 180 degrees for each sample as shown in Figure 26. The linear travel speed at each location is shown in Table 3.

Spiral deposits without mass addition were imaged on the top (XY) and transverse (YZ) cross sections. Stitched optical micrographs of the XY (parallel to the laser direction) of the spiral with and without mass addition are shown in Figure 27.

### 3.3 Linear Single-Track Deposits on Thick Plate Substrate

Table 4 outlines the process parameter levels considered in the design of experiments completed for linear single-track deposits produced using a thick plate substrate. Three laser powers and four travel speeds were considered. Powder mass feed rate was constant throughout these trials. Figure 28 is a drawing of the deposit geometries produced and sectioning plan for metallographic evaluation of the transverse track microstructures. Four-line deposits were completed on each square coupon using four different travel speeds. Each deposit was spaced 0.25 inch apart with a sufficiently long pause time to limit heat accumulation. All three samples were sectioned perpendicular to the laser direction by EDM at 1/3 and 2/3 of deposit length (~0.58" and 1.16" respectively).

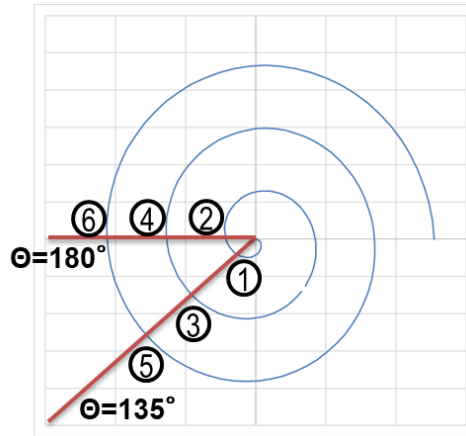


Figure 26: Spiral weld and deposit sectioning and location naming diagram

Table 3: Normalized Travel Speed at Selected Cut Locations in Variable Travel Speed Samples

| Location | Normalized Travel Speed |
|----------|-------------------------|
| 1        | 0.379                   |
| 2        | 1.137                   |
| 3        | 1.867                   |
| 4        | 0.091                   |
| 5        | 0.855                   |
| 6        | 1.622                   |



Figure 27: XY stitched optical images of deposits with without (top) and with (bottom) mass addition. Increasing laser power from left to right.



Table 4: Single-Track Thick Plate Deposit Parameter Sets

| Sample ID | Normalized Laser Power | Normalized Travel Speed |
|-----------|------------------------|-------------------------|
| I-1       | 1.5                    | 0.667                   |
| I-2       | 1.5                    | 1                       |
| I-3       | 1.5                    | 2                       |
| I-4       | 1.5                    | 3                       |
| J-1       | 2.0                    | 0.667                   |
| J-2       | 2.0                    | 1                       |
| J-3       | 2.0                    | 2                       |
| J-4       | 2.0                    | 3                       |
| K-1       | 3.2                    | 0.667                   |
| K-2       | 3.2                    | 1                       |
| K-3       | 3.2                    | 2                       |
| K-4       | 3.2                    | 3                       |

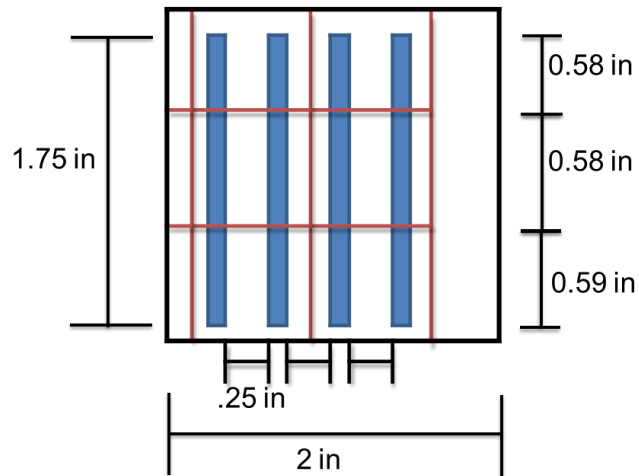


Figure 28: Single-track thick plate deposit specimen layout and sectioning locations

### 3.4 Single-Pass Thin Wall Deposits

Thin wall samples were cut from single-crystal castings using EDM and the EDM recast layer was removed before deposition. Single-pass samples were first produced to the parameter levels shown in Table 5. For the normalized mass feed rate of 2, full steps (i.e. 1, 2, 3, ...) were taken in the design of experiments. For normalized mass feed rates of 1 and 2.5, half steps (1, 1.5, 2, 2.5, ...) were taken in the design of experiments. A full factorial design of experiments was used.

Sample IDs for single layer deposits included in this work can be found in Table 6. Deposits were produced along the  $\langle 100 \rangle$  direction. Samples were sectioned at  $\sim 1/3$  of the length of the deposit using a precision saw. Longitudinal and transverse cross sections were mounted.

### 3.5 Multi-Pass Thin Wall Deposits

For single-pass samples in which the deposits width did not greatly exceed that of the thin wall substrate and insufficient melting of powder did not occur, multi-pass samples were produced. Multi-pass samples were produced through deposition of ten layers using a constant parameter set for each trial. The increase in Z-height between layers, or layer height, was determined by the measured deposit height from the corresponding single-pass parameter combination using a dial caliper. The parameter sweep for these multi-pass samples includes a variation in pause time. In most cases, this pause was varied at three levels. Deposits were produced along the  $\langle 100 \rangle$  direction. Samples were sectioned at  $\sim 1/3$  of the length of the deposit using a precision saw. Longitudinal and transverse cross sections were mounted. Sample IDs for multi-pass samples included in this work can be found in Table 7.

### 3.6 Polishing and Etching Techniques

All samples were mounted in conductive phenolic resin. Polishing of all samples was performed where removal of material during polishing was kept to a minimum by limiting grinding steps. Samples for optical imaging were polished to a 1  $\mu\text{m}$  finish before etching. Swab etching to expose substructure was performed using a glyceregia

Table 5: Single-pass thin wall condition parameter levels

| Normalized Power | Normalized Velocity | Normalized Mass Feed Rate |
|------------------|---------------------|---------------------------|
| 1                | 1                   | 1                         |
| 1.5              | 1.5                 | 2                         |
| 2                | 2                   | 2.5                       |
| 2.5              | 2.5                 |                           |
| 3                | 3                   |                           |
| 3.5              |                     |                           |
| 4                |                     |                           |

Table 6: Sample IDs for select thin wall condition samples included in this work

|      | Normalized Power | Normalized Velocity |
|------|------------------|---------------------|
| L-1  | 1                | 1                   |
| L-2  | 1.5              | 1                   |
| L-3  | 1.5              | 1                   |
| L-4  | 1.5              | 1.5                 |
| L-5  | 1.5              | 2                   |
| L-6  | 2                | 1                   |
| L-7  | 2                | 1.5                 |
| L-8  | 2                | 2                   |
| L-9  | 2.5              | 1                   |
| L-10 | 2.5              | 2                   |
| L-11 | 3.5              | 1.5                 |
| L-12 | 4                | 1.5                 |
| L-13 | 4                | 2                   |
| L-14 | 4                | 3                   |

Table 7: Sample IDs for select thin wall condition samples included in this work

| ID   | Normalized Power | Normalized Velocity |
|------|------------------|---------------------|
| M-1  | 1                | 1.5                 |
| M-2  | 1.5              | 1                   |
| M-3  | 1.5              | 1.5                 |
| M-4  | 2                | 1                   |
| M-5  | 2                | 1                   |
| M-6  | 2                | 1                   |
| M-7  | 2                | 1.5                 |
| M-8  | 2                | 1.5                 |
| M-9  | 2                | 1.5                 |
| M-10 | 2                | 2                   |
| M-11 | 2                | 2                   |
| M-12 | 2                | 2                   |

etchant solution of three parts hydrochloric acid (HCl), two parts glycerol and one-part nitric acid (HNO<sub>3</sub>). Samples for EBSD imaging were polished to a 1 µm before final vibratory polishing with colloidal silica.

### 3.7 Optical Microscopy and Measurements

Imaging of the dendritic structure was performed by optical microscopy using a LEICA DM4000 MLED and Keyence VHX-5000 with VH-Z100R 100x-1000x objective. LEICA images were taken at 50x and 100x. Keyence images of spiral samples were taken at 500x, thin wall longitudinal cross sections at 200x and thin wall transverse cross sections at 400x. In all cases where the entire cross section could not be imaged within a single frame, image stitching was performed using the imaging software for both the Keyence and LEICA microscopes. All measurements were taken using the measuring features in the Keyence software.

### 3.8 Electron Backscatter Diffraction (EBSD) and Measurements

All EBSD data was performed using a JEOL 6500 field emission scanning electron microscope (SEM) with an EDAX EBSD camera. Imaging was taken at a magnification of 50-65x using an accelerating voltage of 20kV and a current of 4 nA. Imaging data was taken treating the material as a normal FCC nickel crystal structure with a step size of 3 µm. Measurements were taken using Fiji (a distribution package of ImageJ) image analysis software [57].

### 3.9 Thermo-Fluid Modeling in OpenFoam

Modeling in this work was performed using the continuum CFD model developed by Coleman et. al [44] on a computer using two 6 core 3<sup>rd</sup> generation Intel Xeon processors. This model was previously developed by Coleman et al using an OpenFoam framework for powder bed additive manufacturing of nickel superalloys [44]. This model shows promise for further development to apply to DED processing. The model accounts for heat transfer due to conduction and convection while computationally efficient to allow for longer run times to be analyzed. The inclusion of

fluid flow allows for a more accurate prediction of the melt pool shape and peak temperature of the melt. Thus G, R and the propensity for stray grain formation can be more accurately determined. The model is unable to predict the deposit cap shape and does not account for the effects of mass addition or favored dendrite growth direction. Inclusion of the dendrite growth direction model produced by Rappaz [7] is valid for use in DED and could be included at a later time. While handled by this model, temperature dependent properties were not used. Additionally, the evaporation model was disabled, and a forced convection coefficient was added to the radiation heat transfer coefficient calculated by the radiation boundary condition.

For modeling of the single-track thick plate samples, the full substrate was meshed with the symmetry plane boundary condition applied at the centerline of the melt pool. In the case of the spiral welds and deposits, parameters of interest were selected, and the assumption was made that velocity was constant and linear. Modeling was conducted using the sample geometry of the single-track thick plate specimens. In both cases, measurements from experimental results were used to define a volume of interest where the mesh was refined for improved accuracy and resolution. The number of cells and cell size within this refined region was dictated by the melt width and depth with consideration for computational time. The element size in the refined mesh area of the high heat input case measured  $19.6 \times 14.2 \times 8.06 \mu\text{m}$ . In the low heat input case, the element size was  $19.6 \times 10.0 \times 2.5 \mu\text{m}$ .

Other modeling cases used intermediate cell sizes depending on the heat input of the parameter set of interest. All simulations for the two semi-infinite cases were run for at least 3 mm in length. This point was considered the point at which a steady state condition was reached. Computation time varied from 3-5 days.

Calibration of the OpenFoam model was performed to experimental substrate melt width, depth and curvature by changes in time step, element size and laser absorption. A high (K-1) and low (J-3) heat input were selected for calibration of the thermal model. The adjustable time step option within OpenFoam was employed with a maximum allowed Courant number of 0.75. Relevant materials properties were taken

from literature and are listed in Table 8 [3, 58]. For additional information on the model used in this work, the reader is referred to the paper describing this models formulation [44].

Table 8: Material Thermophysical Properties for use in Thermo-Fluid Modeling

| Properties                      | Magnitude                  |
|---------------------------------|----------------------------|
| Liquidus Temperature            | 1653.00 K                  |
| Solidus Temperature             | 1593.00 K                  |
| Density (solid)                 | 8700.00 kg/m <sup>3</sup>  |
| Density (liquid)                | 7740.00 kg/m <sup>3</sup>  |
| Reference Density               | 7740.00 kg/m <sup>3</sup>  |
| Specific heat capacity (solid)  | 397.00 J/(kg K)            |
| Specific heat capacity (liquid) | 636.00 J/(kg K)            |
| Thermal conductivity (solid)    | 27.2 J/(m s K)             |
| Thermal conductivity (liquid)   | 35.68 J/(m s K)            |
| Dynamic viscosity               | 0.0053 kg/(m s)            |
| Thermal expansion coefficient   | $1.25 \times 10^{-4}$ 1/K  |
| Dendrite arm spacing            | $5.0 \times 10^{-6}$ m     |
| Latent heat of fusion           | $2.40 \times 10^5$ J/kg    |
| Emissivity                      | 0.13                       |
| Surface tension gradient        | $-1.37 \times 10^{-3}$ N/K |
| Laser absorptivity              | 0.375                      |

## Chapter Four: Results and Discussion

### 4.1 Variable Travel Speed Trial with and without Mass Addition

Increasing power and decreasing spiral radius (and thus linear travel speed), resulted in an increase in melt pool width and depth for spiral deposits without mass addition. These trends are shown in Figure 29 for increasing laser power and travel speed. The same trends are seen in the samples with mass addition shown in Figure 30. Additionally, the height of the deposit increases with increasing power and decreasing travel speeds. Increased laser power was used in sample F-1 as compared to E-1 resulting in an increase in melt pool dimensions. An increase in travel speed was used in sample F-2 as compared to F-1 resulting in a decrease in melt pool dimensions. These results are as expected for an increase in heat input. From the height maps in Figure 31, it is evident that there is thermal stress accumulation, seen by the curling of the substrate corners, which increases with laser power.

The only cracking in the thick plate geometry samples is seen in sample D where a centerline crack is present from the spiral center through 810-degrees of rotation (two full 360-degree rotations plus an additional 90-degrees of rotation). Nickel superalloys are susceptible to solidification cracking. The centerline crack, in addition to the thermal stresses apparent in the samples, is indicative of this type of cracking. However, evaluation of the thermal stresses and cracking are outside the scope of this work.

Certain locations on the outermost ring of the spiral experience edge heating effects as opposed to the semi-infinite heat transfer condition expected. This can be seen at the normalized travel speed 1.867 location for Sample E and F spirals, shown in Figure 32. The edge heating effect results in dendrite growth occurring only in the Z(build)-direction, especially near the substrate edge, in these samples.

#### 4.1.1 Thermo-Fluid OpenFoam Model Comparison to Experimental Results

Since the model does not handle the effects of mass addition, the model was validated to the results from the spirals without mass addition. Samples A-1, A-6 and C-5 were selected for analysis. A comparison of modeling results to experimental trials is depicted



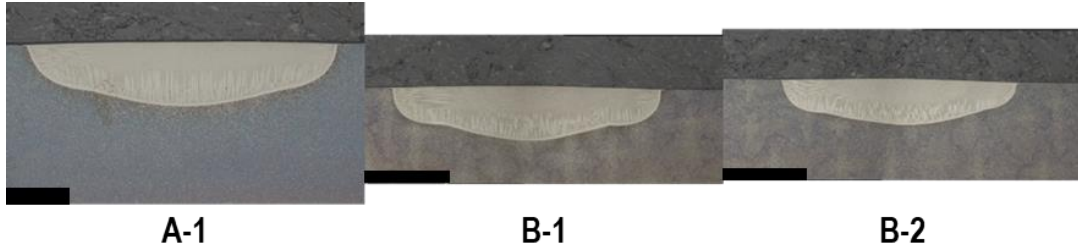


Figure 29: Effect of increasing laser power (A-1 to B-1) and increasing travel speed (B-1 to B-2) on melt pool size in spiral deposits without mass addition

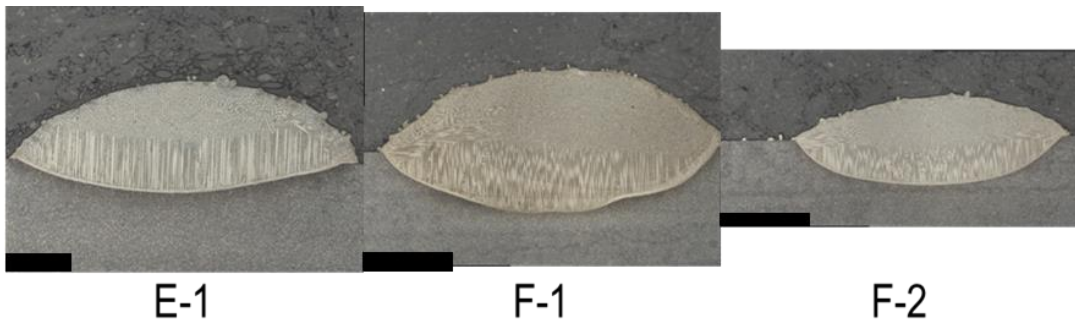


Figure 30: Effect of increasing laser power (E-1 to F-1) and increasing travel speed (F-1 to F-2) on melt pool size in spiral deposits with mass addition

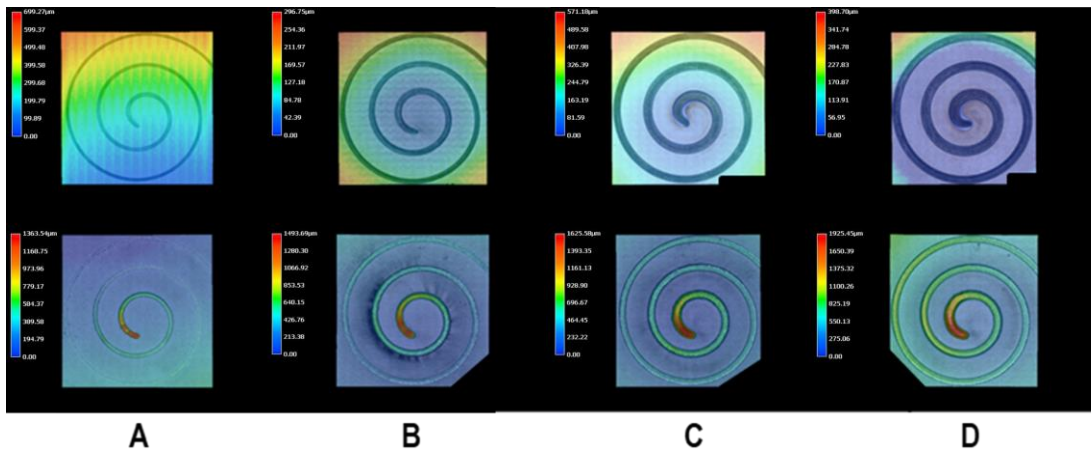


Figure 31: XY stitched height maps of deposits without (top) and with (bottom) mass addition. Increasing laser power from left to right. Curling of substrate is seen which increases with increasing laser power.

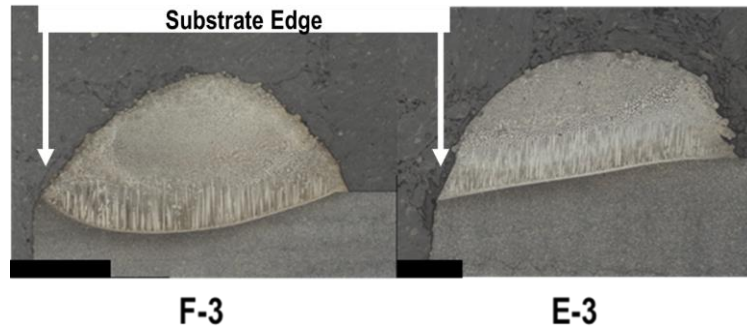


Figure 32: Spiral deposit samples (F-3 (left) and E-3 (right)) with mass addition where edge-heating effects were seen

in Table 9 and shown schematically for a low heat input in Figure 33 and a high heat input in Figure 34. As expected by the lack of a mass addition model, the OpenFoam model predicts these dimensions much better than their deposit counterparts. Deposit transverse cross sectional shapes are predicted with good agreement with the highest error in these cases at 21%. The overprediction in the deposit samples and better agreement in the welding cases can be attributed to mass addition as discussed in Chapter 4.1.2. Further validation and calibration of the model is necessary to better predict the melt pool shapes especially in the case with mass addition.

#### 4.1.2 Effect of Powder Addition on Transverse Melt Pool and Modeling Results

Height, width and volume of the deposit increase with decreasing travel speed (smaller radial distance) and increasing power. Asymmetry of the deposit is seen in some cases such as F-2, H-3 and H-4 in Figure 35. This is likely due to off-centered alignment of the powder cloud and melt pool and is seen in both the spiral and single-track deposits discussed in Chapter 4.2. The addition of powder also reduces the propensity, or eliminates, the growth of dendrites in the x and Y-directions where the z direction is parallel to the heat flux direction. This can be seen in Figure 36 and is likely due to the reduction in melt pool depth with the addition of powder.

Increasing travel speed will reduce mass flux and deposit volume. As shown in Figure 37 and Figure 38, an increase in laser power increases mass capture efficiency

Table 9: OpenFoam model calibration showing melt pool dimensions, percent error, max temperature and max flow velocity for spiral (without mass addition) cases

|                     | Experiment Value          | Modeling Result | Experiment Value            | Modeling Result | Experiment Value          | Modeling Result |
|---------------------|---------------------------|-----------------|-----------------------------|-----------------|---------------------------|-----------------|
| Sample ID           | A-1                       |                 | A-6                         |                 | C-5                       |                 |
| Percent Error Width | 19.69%<br>(over predicts) |                 | -2.04%<br>(under predicts)  |                 | 10.25%<br>(over predicts) |                 |
| Percent Error Depth | 4.99%<br>(over predicts)  |                 | -20.97%<br>(under predicts) |                 | 1.73%<br>(over predicts)  |                 |
| Max Temperature     | N/A                       | 2224.6 K        | N/A                         | 2036.45 K       | N/A                       | 2404.09 K       |
| Max Velocity        | N/A                       | 2.17389 m/s     | N/A                         | 2.53072 m/s     | N/A                       | 2.33365 m/s     |

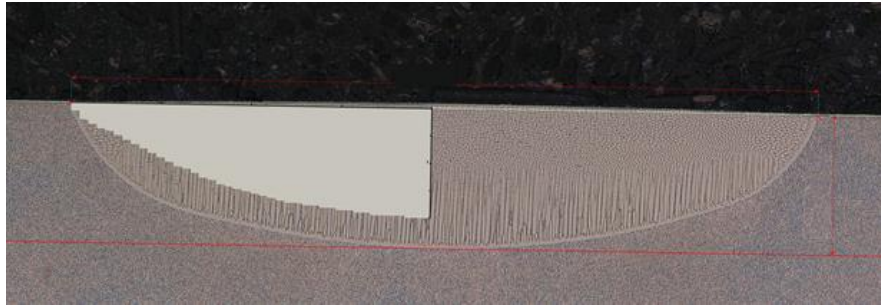


Figure 33: Calibration of OpenFoam model to low heat input sample A-6 (without mass addition). Curvature shows good agreement with a maximum error of 20.97% occurring in depth.

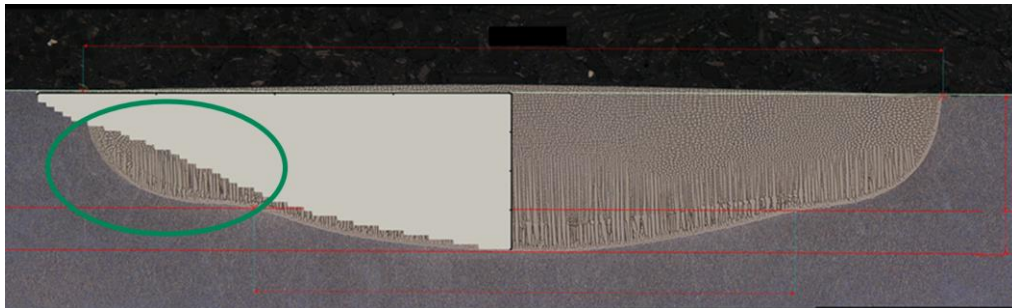


Figure 34: Calibration of OpenFoam model to high heat input sample C-5 (without mass addition). Agreement in curvature is not as good as the low heat input sample due to bulge shown by green circle. However, a superior maximum error of 10.25% occurring in width is achieved.

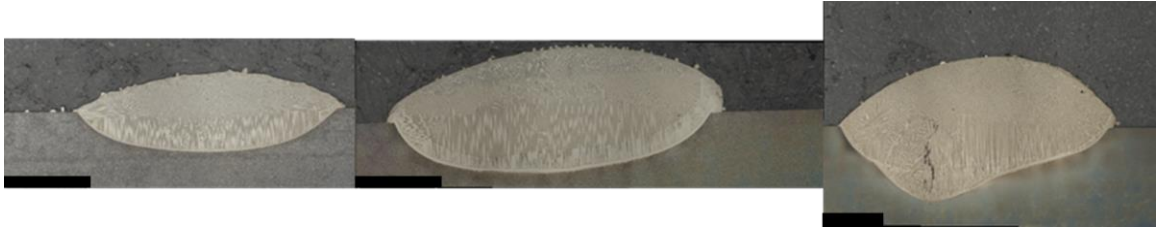


Figure 35: Select variable travel speed (spiral) deposit samples F-2 (left), H-3 (center) and H-4 (right) where asymmetry of the deposit and substrate melting was noted

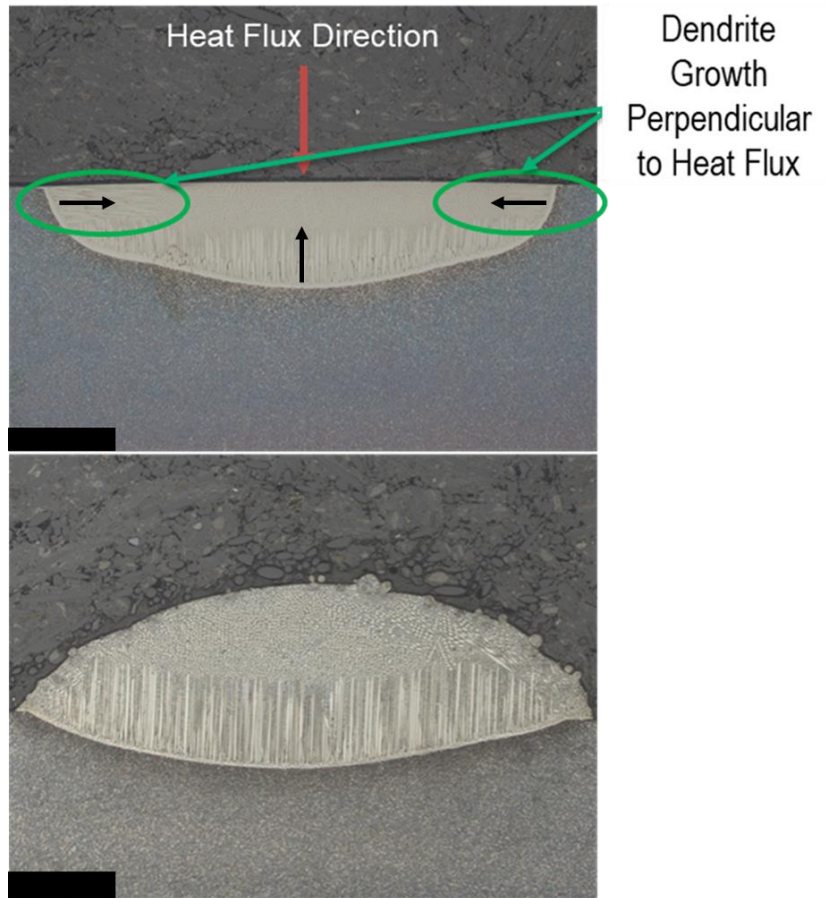


Figure 36: Sample A-1 (top, no mass addition) and E-4 (bottom, with mass addition). Reduction or elimination of dendritic growth perpendicular to the heat flux direction occurs with addition of powder. Dendrite growth directions shown for sample A-1 by black arrows.

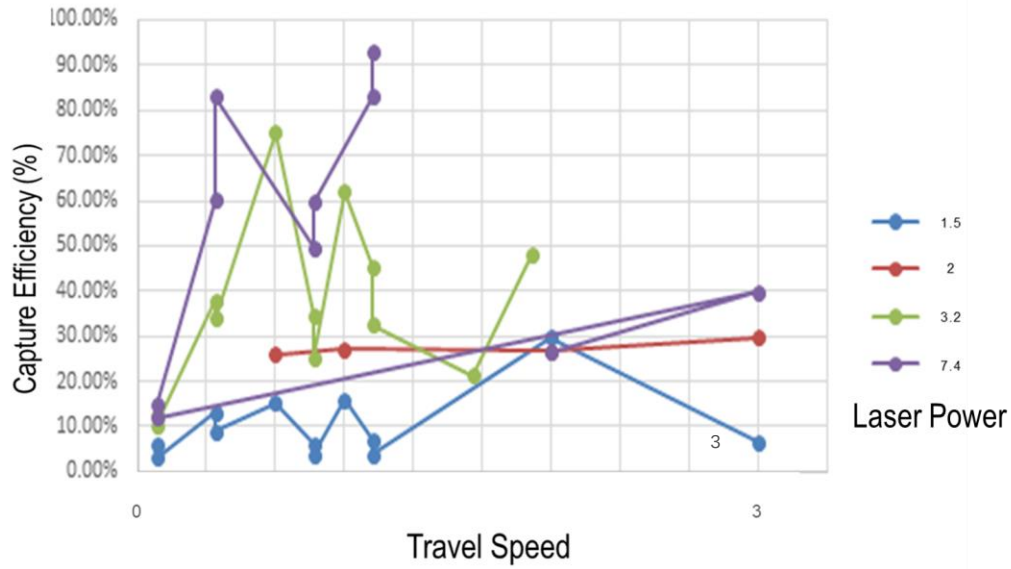


Figure 37: Capture Efficiency in thick plate samples vs travel speed as a function of laser power. Capture efficiency increases with increasing laser power and shows increasing trend with travel speed.

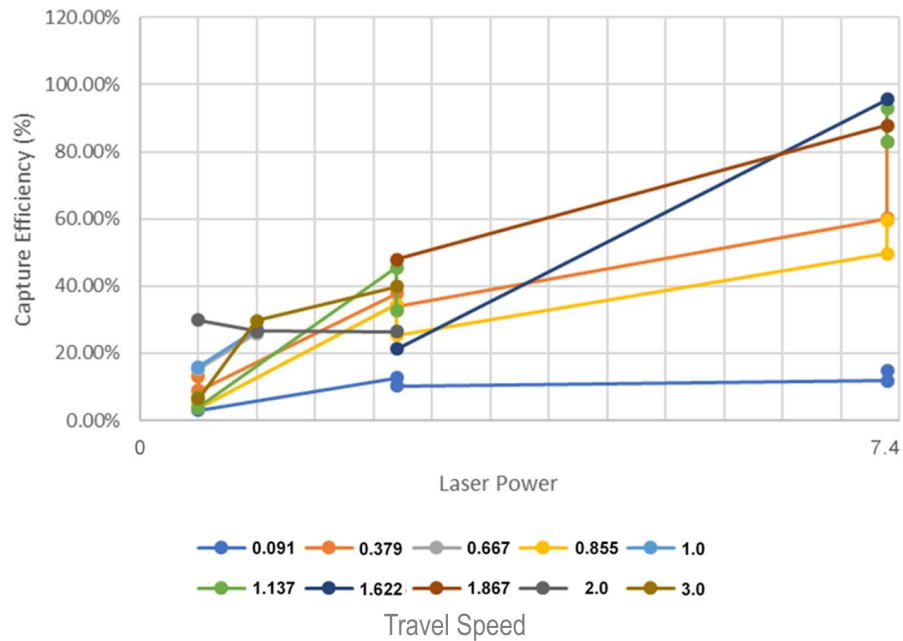


Figure 38: Capture Efficiency in thick plate samples vs laser power as a function of travel speed. Capture efficiency increases with increasing laser power and generally increases with travel speed.

while changes in travel speed do not show a discernible trend. The raw data for these plots is included in Table 13 in Appendix 3 where mass capture is calculated by equations 10, 11 and 12 in Appendix 2. As shown by sample E, in Figure 27, critical limit may exist where mass capture is negligible due to excessively low heat input.

For a powder particle to become a part of the melt, the particle must receive enough energy to fully melt. It is common in modeling of the DED process to assume that powder particles act as a source of energy. **Inspection of the dimensions of the spiral welds and deposits questions the validity of this assumption.** Figure 39, Figure 40, and Figure 41 show the change in width, depth and cross-sectional area, respectively, with the addition of mass as a function of laser power and travel speed. The raw data for these plots can be found in Table 14 in Appendix 3 for reference. A positive change is indicative of an increase in melt pool size with the addition of powder.

Comparison of the cases with and without mass addition in Figure 39, Figure 40 and Figure 41 show that a decrease in substrate melt depth and total substrate melt cross sectional area occurs with the addition of mass. As shown in samples D-1 and D-2 in Figure 42, in samples without mass addition, the melt pool shape approaches one that would be expected with keyhole formation. With material addition for the range of mass feed rates considered, the melt pool shape at these same conditions resembles the shape expected during conduction mode welding. These samples with mass addition are shown in Figure 42 samples H-1 and H-2. This decrease in melt area can be attributed to powder particles acting as a heat sink, removing heat from the melt to melt the particles. This result is supported by the results of the thin wall samples, in Chapter 4.5. It is also contrary to other work and the assumptions made in DED modeling as described in Chapter 2.6.4.

Powder particles acting as a heat sink will result in a depression in the peak temperature, change in the solidification temperature gradient especially near the surface and cause an uneven gradient of temperature across the surface of the melt. These changes will also cause a reduction and disruption in the Marangoni forces changing the flow field and the shape of the melt pool. As described in Chapter 2.3, the



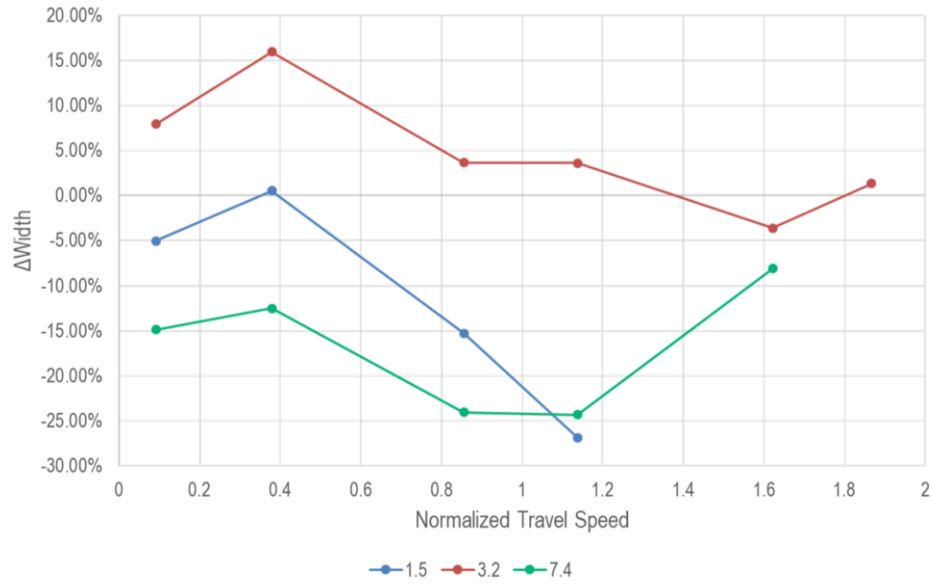


Figure 39: Measured percent change in substrate melt width as a function of laser power and travel speed relative DED without mass addition. Width decreases with powder addition (negative percent change) for 1.5 and 7.4 laser powers but generally increases at 3.2.

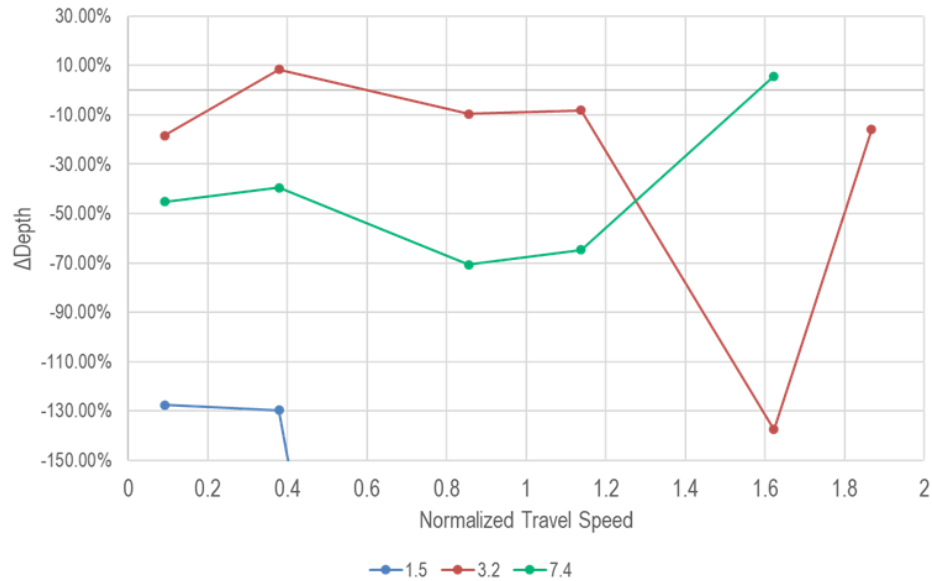


Figure 40: Measured percent change in track depth of deposits with mass addition relative to samples without mass addition as a function of laser power and travel speed. Depth decreases with powder addition (negative percent change) for all laser powers.

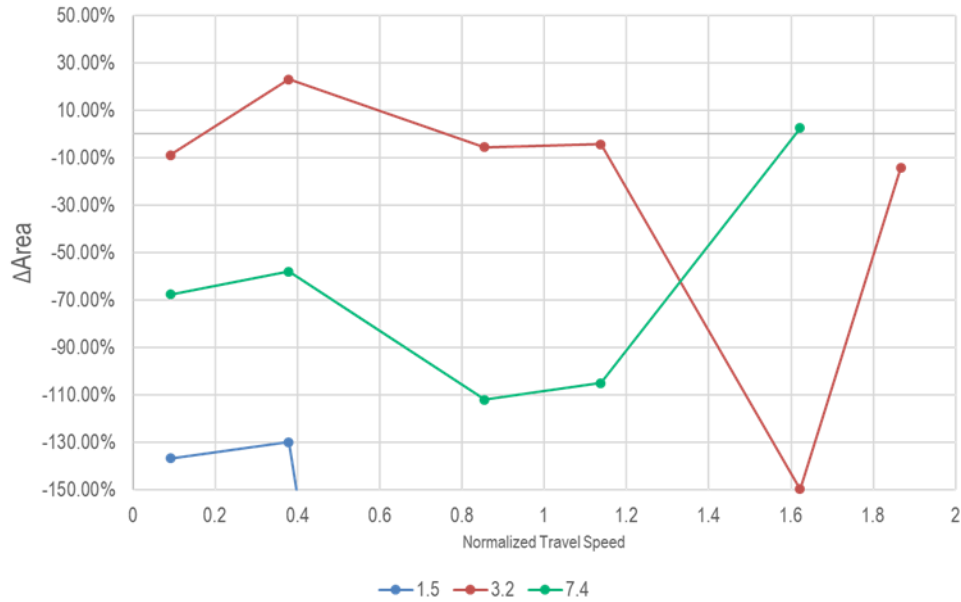


Figure 41: Change in substrate melt cross sectional area of deposits with mass addition relative to samples without mass addition as a function of laser power and travel speed . Cross sectional area decreases with powder addition (negative percent change) for all laser powers.

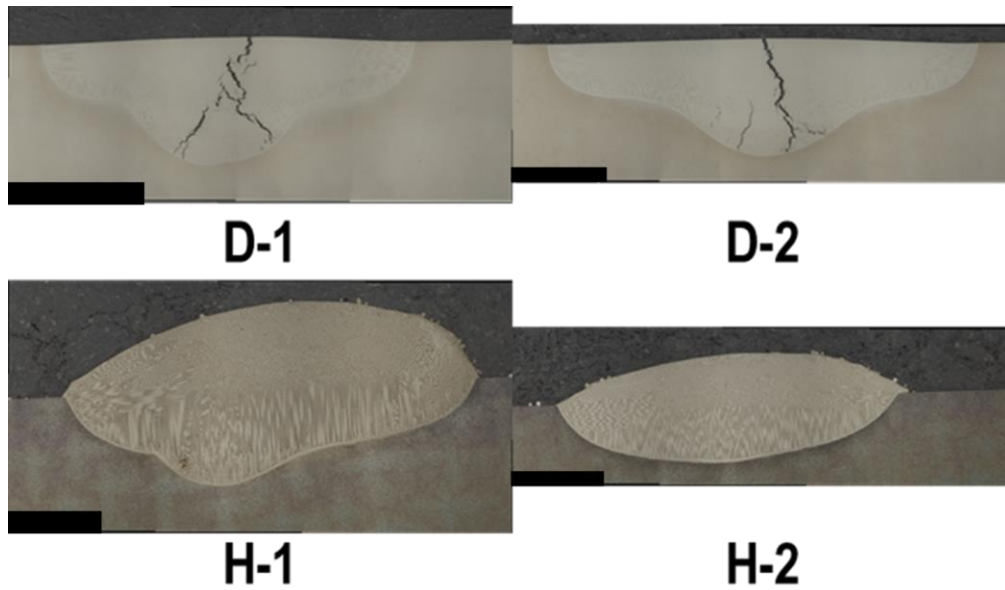


Figure 42: Change in substrate melt shape from one expected with the beginning stages of keyhole formation without powder (top, D-1 and D-2) toward one expected in conduction mode with the addition of powder (H-1 and H-2)



change in melt pool shape will result in additional changes to the thermal gradient and solidification velocity changing the propensity for stray grain formation.

Seen in modeling of the thick plate samples without mass addition in Chapter 4.1 without accounting for material deposition, the model shows good agreement (within 21%) in curvature, depth and width. The reduction in depth due to the addition of powder describes the over prediction of depth in the single-track deposit cases during model validation as discussed in Chapter 4.2.1. As seen in Figure 40, there tends to be a reduction in substrate melt depth with the introduction of powder feedstock.

Comparison of modeling results for the A-1 and C-5 spiral conditions are shown in Table 10 and Table 11. Modeling predictions overlaid onto experimental transverse cross section optical images are shown in Figure 43 and Figure 44. At both higher and lower powers, the weld depth is better predicted by the model for the no mass addition sample as compared to the deposit sample with mass addition. Since the model does not consider mass addition, there may be a higher propensity for overprediction of weld depth when comparing to the experimental cases with mass addition. Since the no mass addition cases considered agree well with the modeling results, the overprediction of melt depth qualitatively agrees with this trend. The model exhibits a fair accuracy when compared with experimental results shown in Table 10 and Table 11. With the addition of a subroutine in the model to account for mass addition, it can be hypothesized that the error in calculated dimensions for the deposit may be reduced. An overview of modeling results with and without mass addition is presented in Figure 45 and Figure 46.

#### 4.1.3 Effect of Processing Parameters on Epitaxial Growth

Using image analysis techniques<sup>1</sup> calibrated to sample C, shown in Figure 47, and applied to samples A, B and D, shown in Figure 87 in Appendix 2, color maps of the dendritic growth directions that correspond to the optical microscope images of the top (XY) sections of the spiral welds were produced. In these color maps, the red color

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<sup>1</sup> Supporting code developed by Peter Zeglen is included in Appendix 5.

Table 10: Modeling vs experimental results for melt pool dimensions for sample A-1

|                     | Experimental Value (weld)  | Experimental Value (Deposit) | Modeling Result |
|---------------------|----------------------------|------------------------------|-----------------|
| Sample ID           | A-1                        |                              |                 |
| Percent Error Width | -50.93%<br>(over predicts) | -49.54%<br>(over predicts)   |                 |
| Percent Error Depth | -7.51%<br>(over predicts)  | -141%<br>(over predicts)     |                 |
| Max Temperature     | N/A                        | N/A                          | 2224.6 K        |
| Max Velocity        | N/A                        | N/A                          | 2.17389 m/s     |

Table 11: Modeling vs experimental results for melt pool dimensions for sample C-5

|                     | Experimental Value (weld) | Experimental Value (Deposit) | Modeling Result |
|---------------------|---------------------------|------------------------------|-----------------|
| Sample ID           | C-5                       |                              |                 |
| Percent Error Width | -10.3%<br>(over predicts) | -6.32%<br>(over predicts)    |                 |
| Percent Error Depth | 1.73%<br>(under predicts) | -7.69%<br>(over predicts)    |                 |
| Max Temperature     | N/A                       | N/A                          | 2404.09 K       |
| Max Velocity        | N/A                       | N/A                          | 2.33365 m/s     |

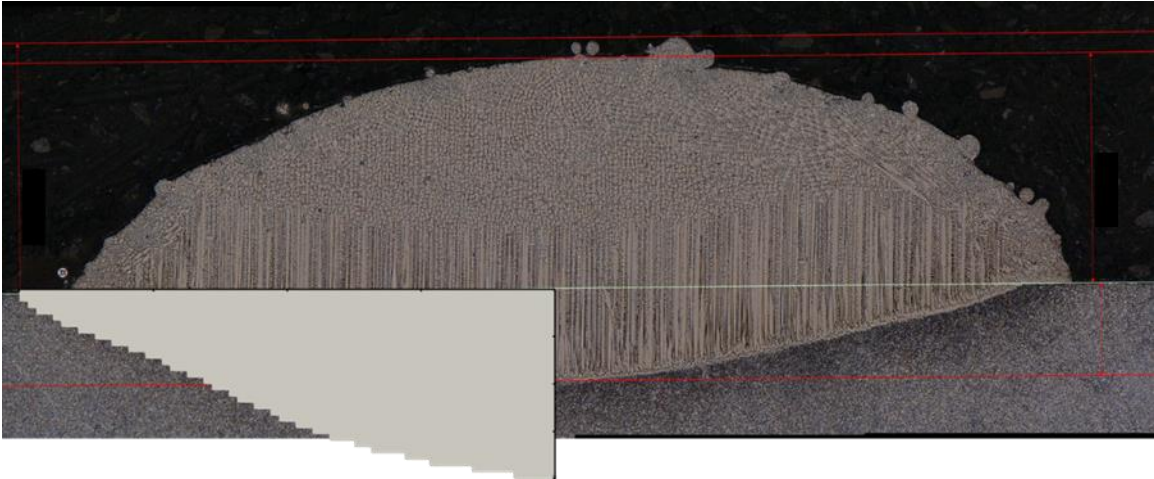


Figure 43: Comparison of OpenFoam model to sample A-1 spiral deposit with mass addition. Width and depth are overpredicted with good agreement in curvature.

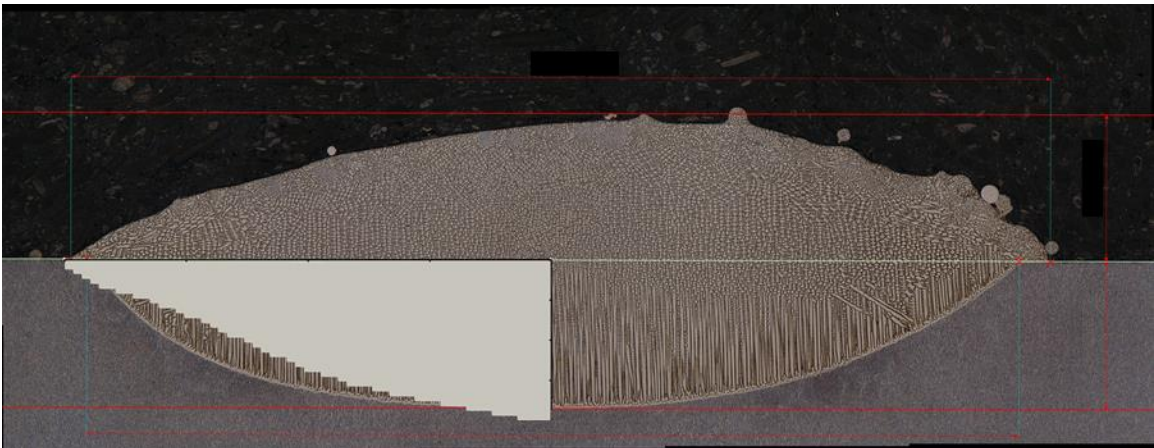


Figure 44: Comparison of OpenFoam model to sample C-5 spiral deposit sample with mass addition. Depth is overpredicted with good agreement in width.

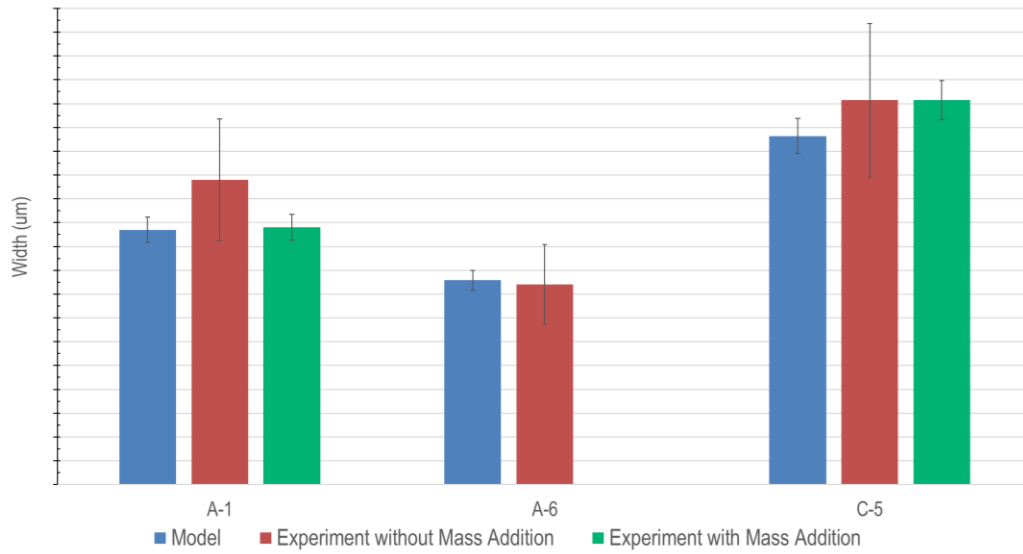


Figure 45: Comparison of modeling results for thick plate spiral samples to experimental results with error bars (20%) for melt depth with and without mass addition

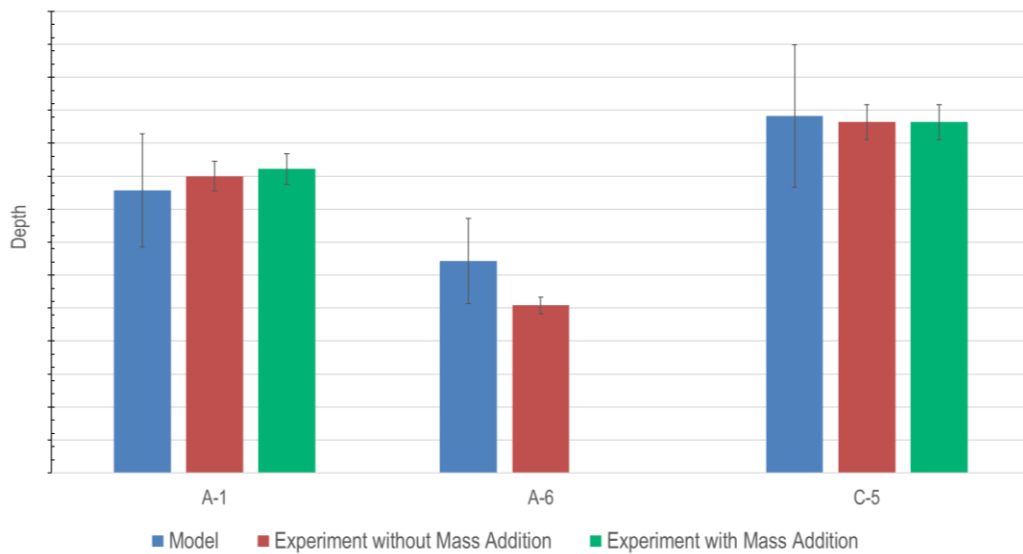


Figure 46: Comparison of all modeling results for thick plate spiral samples to experimental results with error bars (20%) for melt width with and without mass addition

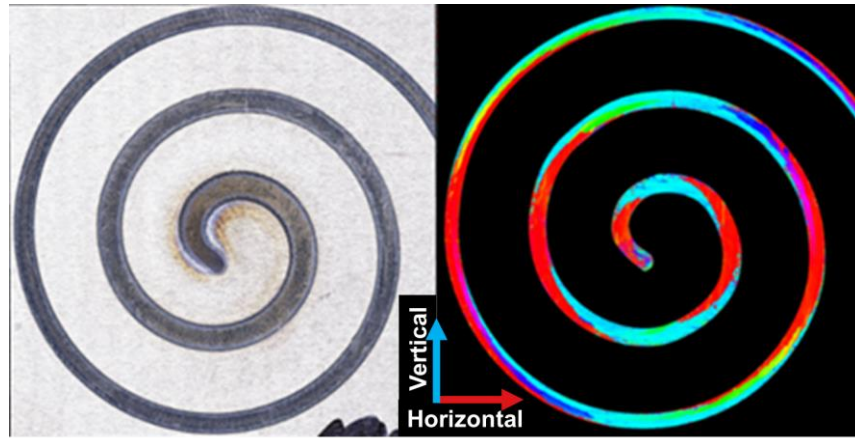


Figure 47: Spiral deposit without mass addition stitched optical (left) and image analysis dendrite orientation color map (right) for Sample C. Red corresponds to horizontal and light blue to vertical dendrites.

corresponds to horizontal (X) dendrite growth, light blue corresponds to vertical (Y) dendrite growth and other colors to directions off-axis from the horizontal or vertical with respect to the image. Through evaluation of the raw and image analysis images, competition between dendrite growth directions is highly evident. At each multiple of 90-degrees and at low speeds, dendrite growth along the top surface is perpendicular, either  $[100]$  or  $[010]$  assuming  $[001]$  is into the weld depth, to the laser motion. At higher speeds, dendrite growth along the laser travel direction is seen. This effect has been seen in prior work [3, 4, 53]. In between each 90-degree location, the transition from completely  $[100]$  growth to completely  $[010]$  is seen. At the middle of this transition, 45-degree variants, the weld pool is approximately split with half the dendrites solidifying in the  $[100]$  direction and the other half in the  $[010]$ . These effects are all expected and confirm previous work through application of the Rappaz dendrite variant model [7, 8]. Use of this image analysis tool may provide an indication of the amount of stray grain formation at a given location however work to investigate the validity of this approach was not completed.

Transverse cross sections of the spiral welds show [001] growth occurring from the weld pool bottom with competing growth in the [010] or [100] occurring from the melt pool sides inward. Amount of dendrite growth from the side walls increases with melt pool depth as shown in Figure 48. This observation can be expected as the dendrite growth variants perpendicular to the side wall will become more active with higher curvature, again agreeing with the Rappaz dendrite variant model [7, 8]. Due to the spiral path and the small velocity gradient created from the outer to inner radius of the pool, asymmetry in the melt pool shape was expected. However, upon inspection of the transverse section imaging, little melt pool shape asymmetry can be seen. Spiral weld samples agree well with prior work and single-crystal solidification theories with the spiral path.

Epitaxial growth was generally maintained in the bulk with an equiaxed cap forming at the surface of the deposit which agrees with the results and trends of Gaumann and others [10-13, 24, 25]. The size of the equiaxed cap increases with increasing mass flux, laser power and decreasing travel speed. This equiaxed cap forms likely due to a decrease in thermal gradient and solidification velocity at the later stages of solidification occurring at the surface in addition to an increase in nucleation sites as noted by Liang [10].

#### 4.1.4 Spiral Path for Process Parameter Development

The spiral path experiment allows for rapid and flexible analysis of the effect of travel speed at a given power within one experimental run. In DED processing, the spiral path also allows for a rapid analysis of capture efficiency needed for deposition models. The production of meaningful samples allows for the spiral path experiment to be a more effective way to conduct parameter sweeps during process parameter development. One can access a large range of velocities, as determined by the angular velocity and radius chosen, with the ability to easily access intermediate values. This reduces the possibility of needing additional processing runs to acquire wanted parameter combinations saving material, analysis and machine time.

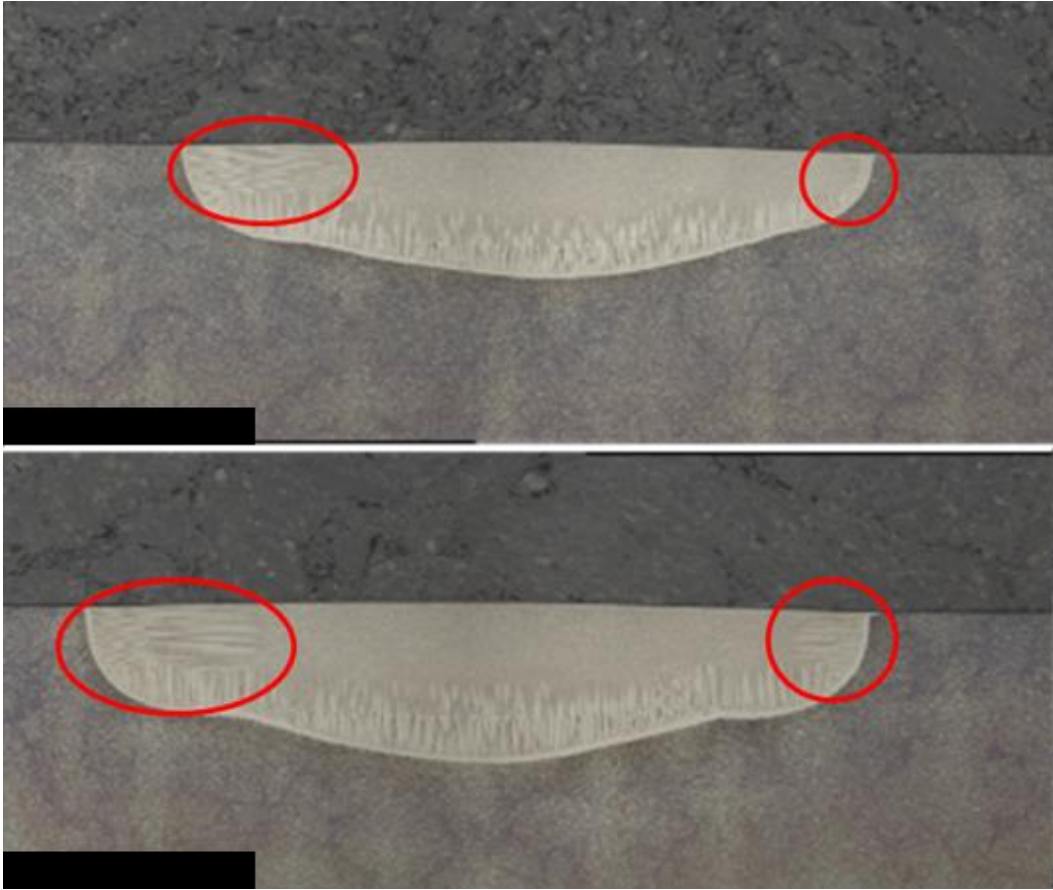


Figure 48: With increasing melt pool depth, the amount of dendrite growth occurring perpendicular to the heat flux direction, circled with red, increases.



Use of a spiral path can provide a large number of possible samples in a single experimental run but one needs to understand the effects of a spiral path as opposed to the linear path in single-track experiments. The spiral design imposes non-steady state conditions due to the constantly accelerating instantaneous travel speed, a travel speed gradient across the pool due to the distance between the inner and outer radius, and an increase in substrate temperature during production. However, these effects are minimal, as shown by Fukumoto in his work with a ferrous alloy [56], and analysis of the samples in this work. Samples produced in this method provide good material indicative of the processing parameters and phenomena occurring in the melt while allowing for the rapid and flexible analysis desired in the early stages of process parameter development.

#### 4.2 Linear Single-Track Deposits on Thick Plate Substrate

Single-track, thick plate substrate deposits with mass addition transverse cross sections are shown in Figure 81, Figure 82, and Figure 83 for samples I, J, and K, respectively. EBSD with corresponding dark field images of these same cross sections are shown in Figure 84, Figure 85 and Figure 86 for sample I, J, and K, respectively. These figures can all be found in Appendix 2 for reference. The same trends for increases in melt pool width and depth are seen in these samples as were observed for the spiral welds and deposits.

##### 4.2.1 Thermo-Fluid OpenFoam Model Comparison to Experimental Results

Comparison of experimental and modeling melt pool dimensions, percent error in modeling dimensions, peak temperature and max fluid flow velocity for modeling calibration are provided in Table 12. Figure 49 and Figure 50 show modeling results for high heat input case (K-1) and low heat input case (J-3) overlaid onto experimental results. Good agreement in width and curvature are achieved in both cases. Depth is greatly overpredicted in the low heat input case. Good agreement in melt depth is seen in the high heat input case. This discrepancy may be due to powder addition and powder particles acting as a heat sink.



Table 12: OpenFoam model calibration showing melt pool dimensions, percent error, max temperature and max flow velocity for single-track thick plate cases with mass addition

|                     | Experimental Value       | Modeling Result | Experimental Value       | Modeling Result |
|---------------------|--------------------------|-----------------|--------------------------|-----------------|
| Sample ID           | K-1                      |                 | J-3                      |                 |
| Percent Error Width | 1.52% (over predicts)    |                 | -13.79% (under predicts) |                 |
| Percent Error Depth | -12.15% (under predicts) |                 | 117.39% (over predicts)  |                 |
| Max Temperature     | N/A                      | 2396.24 K       | N/A                      | 2139.96 K       |
| Max Velocity        | N/A                      | 2.41251 m/s     | N/A                      | 2.89959 m/s     |

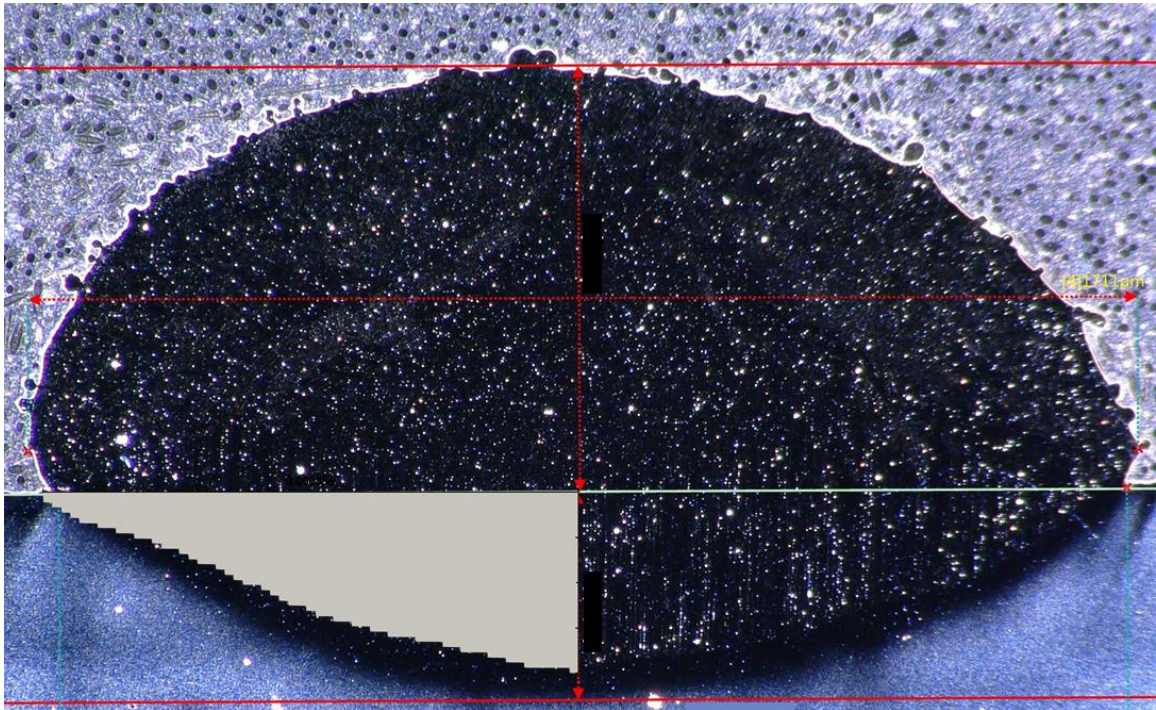


Figure 49: Validation of OpenFoam model to K-1 single-track thick plate substrate sample. High heat input case shows good agreement in melt pool dimensions and curvature.

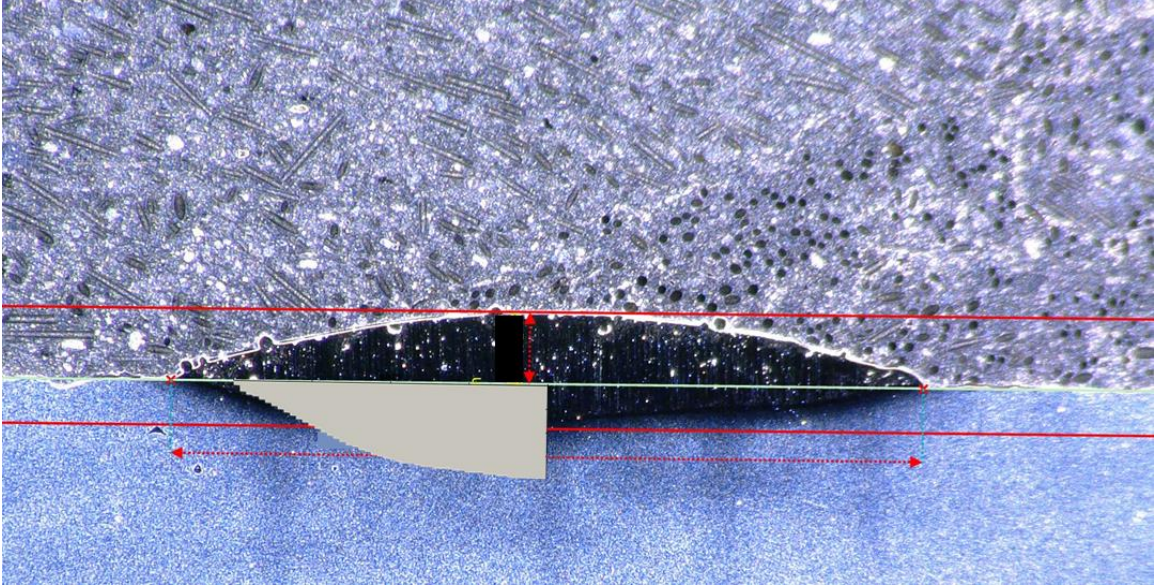


Figure 50: Validation of OpenFoam model to J-3 single-track thick plate substrate sample. Low heat input case shows poor agreement in melt pool dimensions but good agreement in curvature.

#### 4.2.2 Effect of Processing Parameters on Epitaxial Growth

When the extent of substrate melting is relatively low (melt pool volume), off-axis growth occurs at the solid-liquid interface. This effect can be seen in EBSD of samples I-3 and J-4, in Figure 51, where lines of off-axis growth occur within the deposit interior. With constant travel speed, an increase in power results in a relatively larger melt pool volume, increased mass capture and increased equiaxed cap depth (thickness of the equiaxed cap at the crown of the deposit. Schematically defined in Figure 88 in Appendix 2). This trend is depicted in Figure 52 where increasing laser power results in a larger melt cross sectional area and an increase in the equiaxed cap. Holding laser power constant, increasing travel speed results in a decrease in curvature (angle of repose), volume of deposited material, and equiaxed cap depth. This trend is depicted in Figure 53. EBSD of samples with increasing travel speed show decreased melt dimensions and decrease in the equiaxed cap. A change in travel speed had less of an effect on cap depth than changes laser power.

With a decrease in melt pool depth, to the limit as described previously, epitaxial growth is maintained, and cap depth is decreased. Additionally, as shown in optical microscopy of the spiral deposits with mass addition, decreased melt pool depth increases the fraction of [001] dendrites to others. Similar trends have been seen in literature. As seen in single-crystal welds by Yoshiro and depicted in Figure 18, a decrease in melt pool depth has shown a decreased propensity for stray grain formation. The current work agrees with this trend.

#### 4.3 Summary of Thermo-Fluid Modeling Results

Thermo-fluid modeling conducted in this study applies the OpenFoam model developed by Coleman et al [44]. In this work, the boundary conditions were adjusted to add a forced convection heat transfer coefficient, use materials properties that were constant with temperature and ignore the effects of vaporization.

Results for samples without mass addition were presented previously in Chapter 4.1.1. Comparison of modeling and experimental results for samples produced without



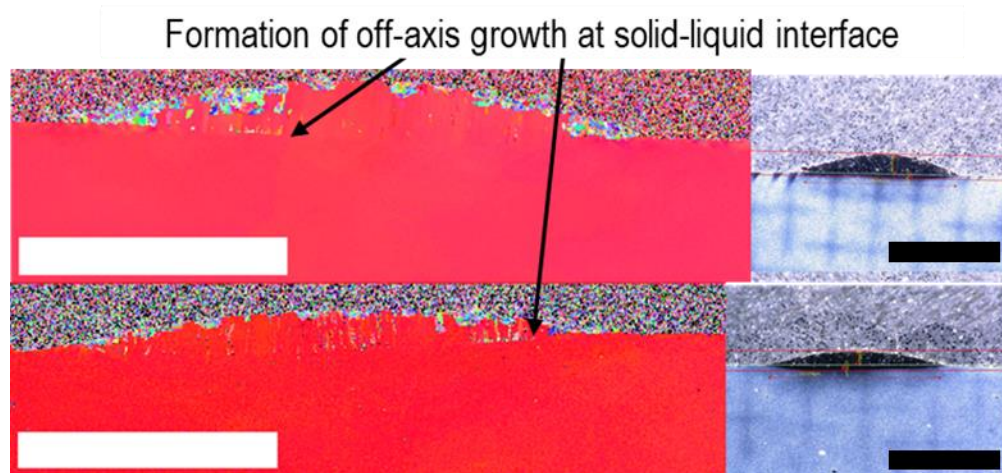


Figure 51: EBSD of sample I-3 and J-4. With shallow melt pools in the thick plate condition, off-axis growth sites, denoted by the black arrows, can occur at the solid-liquid interface with very low heat inputs.

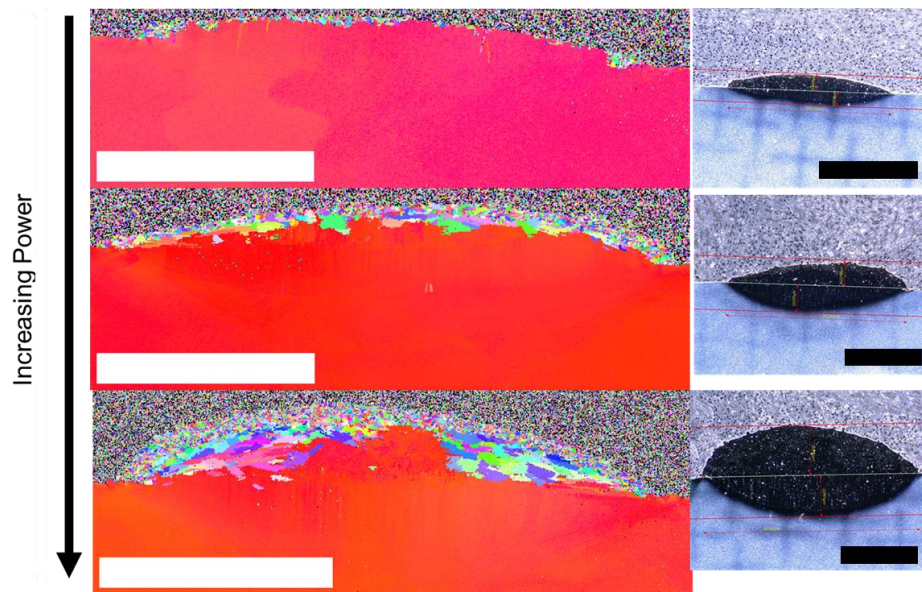


Figure 52: EBSD and dark field imaging for thick plate sample at I-2 (top), J-2 (center) and K-2 (bottom) showing increasing equiaxed cap depth with increasing laser power.

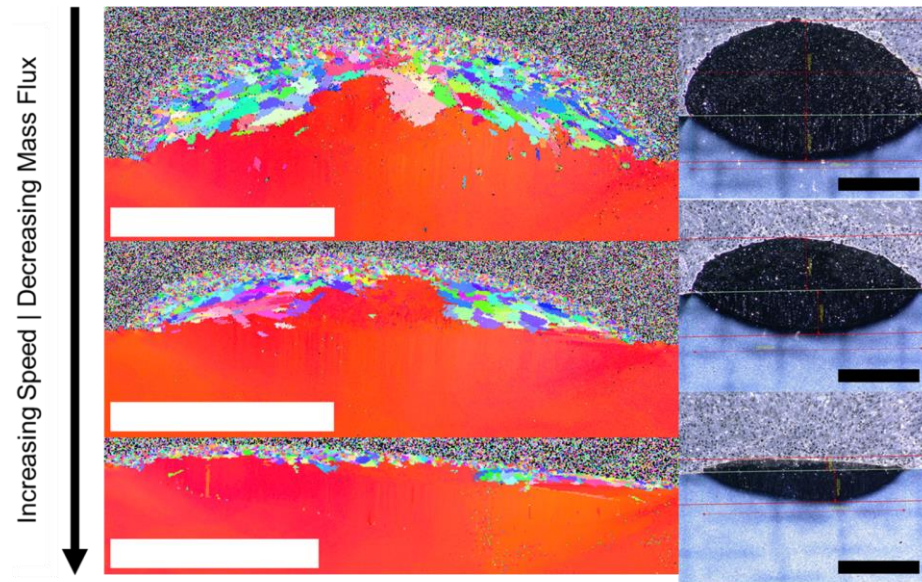


Figure 53: EBSD and dark field imaging for thick plate samples K-1 (top), K-2 (middle) and K-3 (bottom) showing decreasing equiaxed cap depth with increasing laser power.

mass addition show a maximum percent error of 20.97% and slight deviations in transverse cross-sectional curvature. Deviations in curvature may be due to deviations in Marangoni force from the constant negative value used in this study. While acceptable agreeance in width, depth and curvature was achieved in these samples, further calibration and validation of the thermal model is recommended.

This model, which was previously developed for powder bed AM processes, does not account for the addition of mass which occurs during DED processing. This allows for predictions of thermal gradients and solidification velocities for the substrate remelting region of samples with mass addition. As shown in Chapters 4.1.1 and 4.2.1, the addition of mass which acts as a heat sink can affect the dimensions of the melt pool. As shown in Table 10, Table 11, and Table 12, modeling predictions with mass addition over predicted the melt volume. This overprediction agrees with experimental observations, as shown by the change in cross sectional area shown in Figure 41.

With the addition of a subroutine to handle the effects of mass addition and further calibration, it is hypothesized that better agreement for the mass addition case can be achieved. Further increases in modeling accuracy may be achieved through addition of subroutines to describe the dendrite growth variant selection, spatial variation in  $N_0$ , deposit shape and convergence of the laser beam. Uncertainty in DED process modeling and methods to improve the model in this work are further discussed in Chapter 5.

#### 4.4 Thick Plate Substrate Processing Maps

Processing maps were produced evaluating cap depth, capture efficiency and change in transverse melt area vs laser power and travel speed. These metrics are important to describe the propensity to maintain epitaxial growth with additional layers, the amount of mass captured by the melt and consequentially the energy absorbed by powder. The amount of mass captured is directly correlated to the deposit volume.

The effects of laser power and travel speed on cap depth mimic those seen in Figure 54 and Figure 55 for capture efficiency. As seen in Figure 54, with increasing laser power, cap depth increases and then plateaus. In Figure 55, the increase in cap

depth due to power is again noticeable. With an increase in travel speed, cap depth decreases. Thus, as the mass flux, heat input, and deposit volume decrease, the equiaxed cap depth decreases. Evaluation of the slope of the lines in Figure 54 and Figure 55 shows that laser power generally has a stronger effect on cap depth than travel speed for the range of parameters considered. This agrees with the trends seen in Figure 52 and Figure 53.

#### 4.5 Single-Pass Thin Wall Deposits

The impact of variation in section geometry on track geometry and texture evolution is presented in this section starting with single-track samples. Single-track deposits were completed along a thin edge to explore the influence of substrate geometry and heat source proximity to the edge of the substrate on deposit texture evolution.

In Figure 56, the effect of the change in substrate geometry can be seen through EBSD imaging. In the thick plate case, the deposition height is limited, with epitaxial growth in the substrate and deposit until the formation of a thin equiaxed cap. With the reduction of conductive pathway in the thin wall substrate geometry, the deposit height increases, off-axis growth at the solidification interface is seen and the equiaxed cap depth increases. This is likely due to the change in solidification thermal gradient, solidification velocity, curvature (angle of repose) and powder melt interactions in the constrained geometry.

##### 4.5.1 Effect of Processing Parameters and Deposit Shape on Single-Track Specimens

Figure 57 shows increasing laser power and decreasing travel speed coarsen deposit microstructure. With increasing laser power, thermal gradients decrease allowing for coarsening of the microstructure. With decreasing travel speed, solidification velocities decrease allowing also allowing for coarsening of the

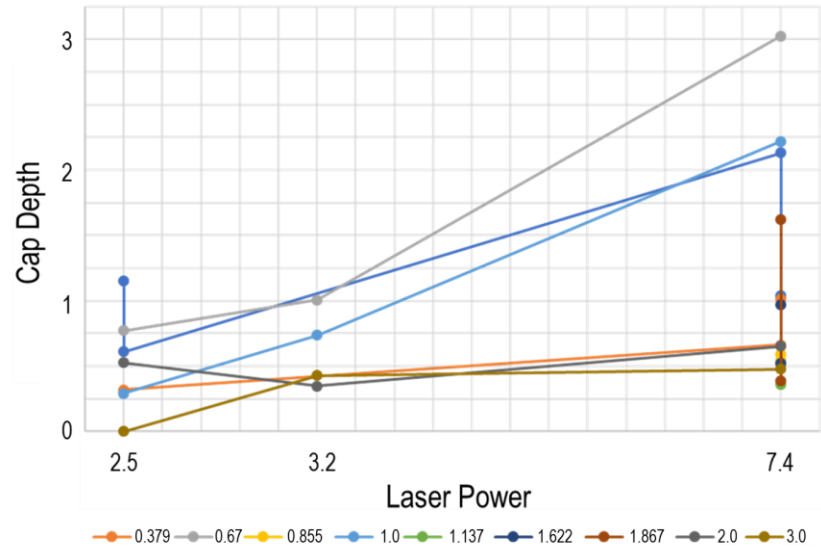


Figure 54: Thick plate sample cap depth vs laser power (x-axis) and normalized travel speed (different colored curves). Equiaxed cap depth increases with increasing laser power.

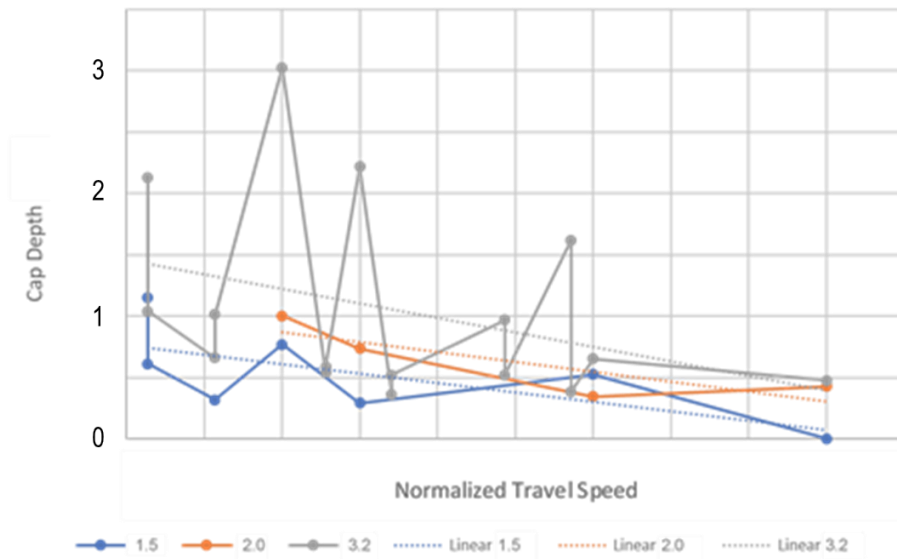


Figure 55: Thick plate sample cap depth vs travel speed (x-axis) and normalized laser power (different colored curves). Decreasing trend in cap depth is seen with increasing travel speed.



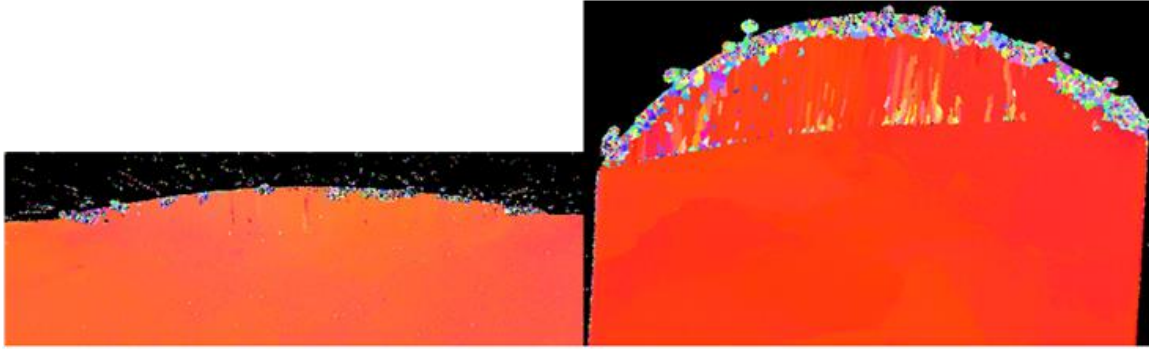


Figure 56: Effect of change in substrate width on single-track deposit texture and microstructural features in thick plate (J-3, left) and thin wall (L-8, right) geometries. Cap depth and amount of off-axis growth increase in thin wall geometry.

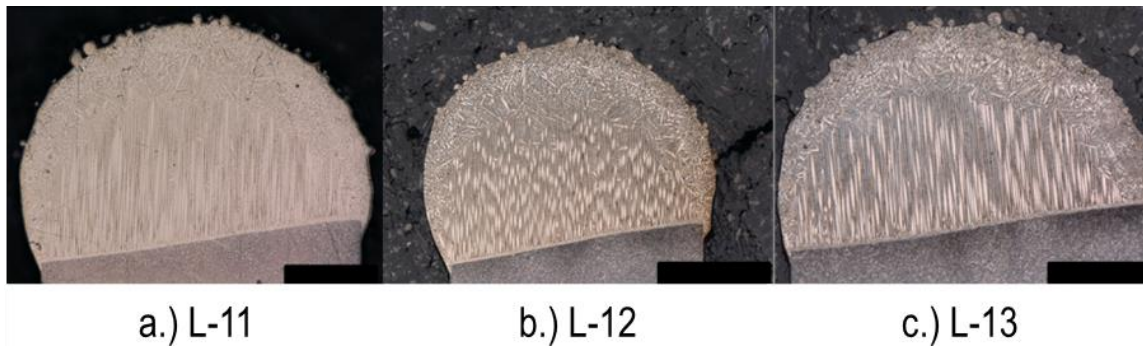


Figure 57: Coarsening of dendrite structure occurs with increasing power (L-11 to L-12) and decreasing travel speed (L-12 to L-13).

microstructure. Dendrite growth predominantly occurs along the Z-direction. No dendritic growth perpendicular to the heat flux direction was observed for the process parameter combinations considered in this study.

Figure 58 shows photomicrographs comparing single-pass deposits on a thin wall substrate (L-11) and a thick plate substrate (H-5) microstructure. Solidification occurred inward from all locations along the melt pool resulting in some growth occurring perpendicular to the heat flux direction in the case of thick plate sample H-5. Reducing the volume of substrate material in the Y-direction which can extract heat due to conduction may cause dendrite growth in the Y-direction to be less favorable. The removal of interfaces where the dendrite growth direction changes may reduce the chance of stray grains, cracking and reductions in solidification velocity due to dendrite growth variant selection, as reviewed in Chapter 2 [5, 7].

Asymmetry of the molten pool shape was observed for single-pass, thin wall substrate deposits. Figure 59 shows photomicrographs demonstrating track geometry asymmetry. In image (b) of Figure 59, a concave-convex interface is presented. Track geometry asymmetry may arise due to laser alignment or working with an excessively large laser spot size. Further work is needed to understand the significance of these factors and the corresponding mechanisms promoting track geometry asymmetry. Track geometry asymmetry is important as it correlates with asymmetry in deposit equiaxed cap depth. The side of the track where melt depth is higher tends to correspond to a thinner cap depth. Epitaxial growth is dependent upon the ability to limit the presence of remnant equiaxed grains as deposition proceeds. Therefore, to maintain epitaxial growth over multiple layers, a relatively deeper molten pool will be needed as cap depth (area fraction of equiaxed grains) increases.

Figure 60 is a photomicrograph of a deposit microstructure where half of the deposit shows columnar material while the other half shows equiaxed material. This is likely due to excessively low heat input resulting in sintering or partial melting of powder particles and subsequently recrystallization. The half of the track with a lower number density of partially melted powder particles exhibits columnar grain morphology.

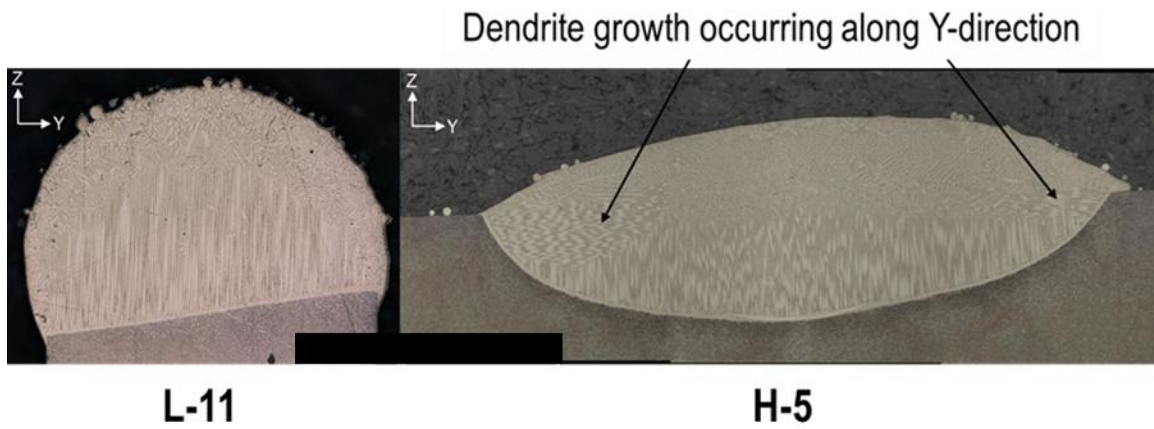


Figure 58: Changes in dendrite growth variants seen with change in substrate geometry with no change in processing parameters

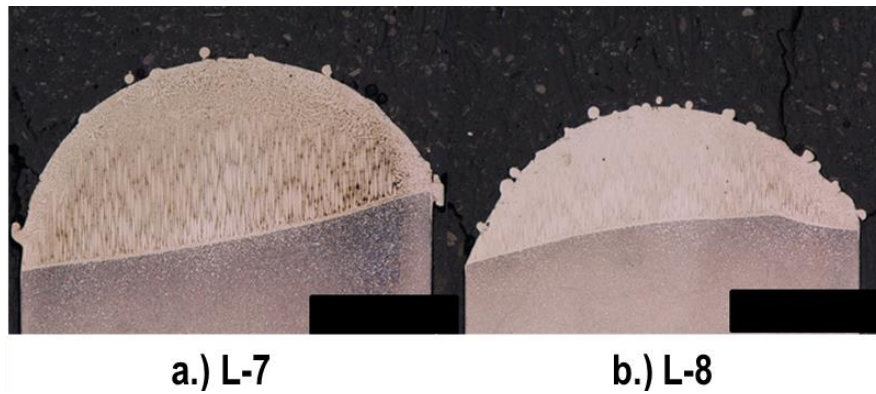


Figure 59: Change in melt pool shape likely due to an off-centered laser and changing processing parameters in samples L-7 and L-8. Decreasing heat input due to increased travel speed from L-7 to L-8.

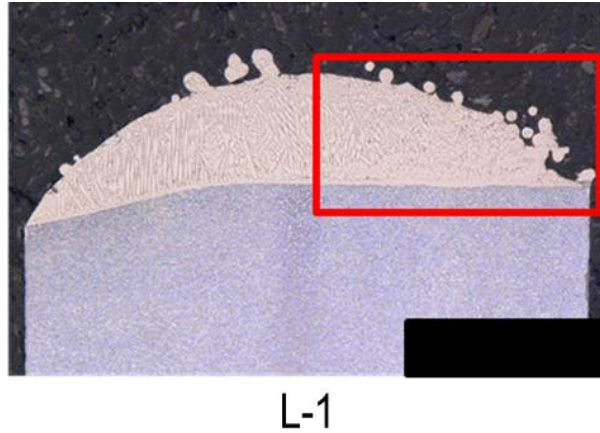


Figure 60: Low heat input and melt pool asymmetry results in sintering of powder, boxed in red, and lack of epitaxial growth resulting in equiaxed material. This area is shown by the red box.

In cases where the maximum track width exceeds the substrate width, the thickness of the equiaxed material on the deposit side increases, as seen in Figure 61. Deposit microstructures tended to show a strong propensity for epitaxy within the width of the original substrate (highlighted by red lines on Figure 60 a) despite the variation in equiaxed material on the deposit sides. These trends highlight the relative significance of convection and conduction for these deposit and substrate geometries. The data tend to suggest that even for thin wall substrate geometries, conduction through the bulk is critical to maintaining epitaxial growth.

With increasing melt volume, a bifurcated melt pool may form. Evidence of this formation is shown in Figure 62 where there is increased epitaxial growth at the center of the deposit. For a bifurcated melt pool to form, multiple eddy currents must be formed [59]. The formation of these currents in this processing condition may be due to the laser interacting with the sides of the substrate in addition to the substrate top due to the laser spot size exceeding the original substrate width. This forms three faces and directions of heat flux which can result in the formation of this phenomena as shown in Figure 63. Further work is needed to confirm if this is the cause of the bifurcated pool.

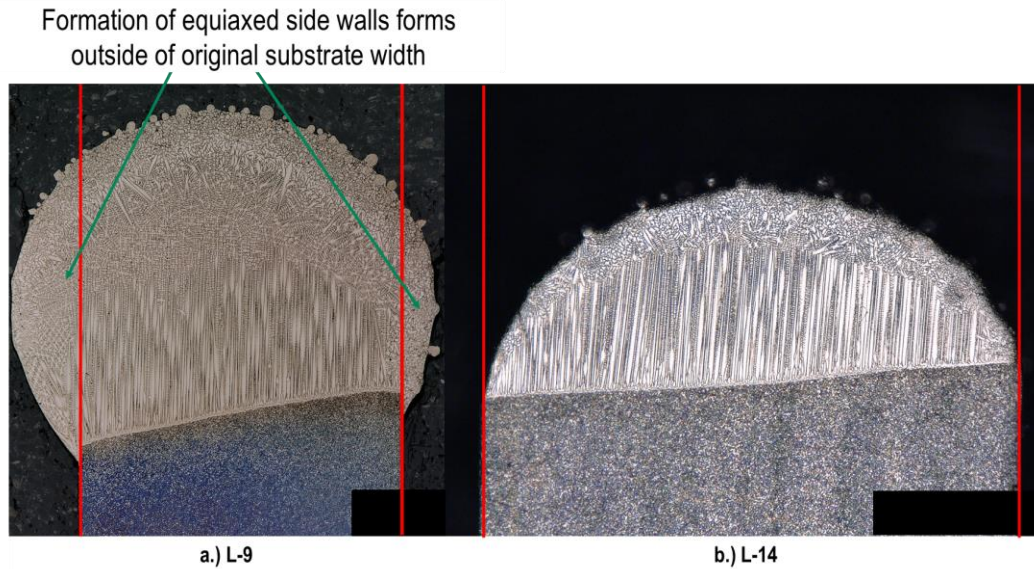


Figure 61: Photomicrographs of two deposit microstructures whose maximum track width is a.) greater than (L-9) and b.) less than (L-14) the width of the substrate. In L-9, off-axis columnar and equiaxed material forms in the deposit material which falls outside the red lines denoting the substrate width.

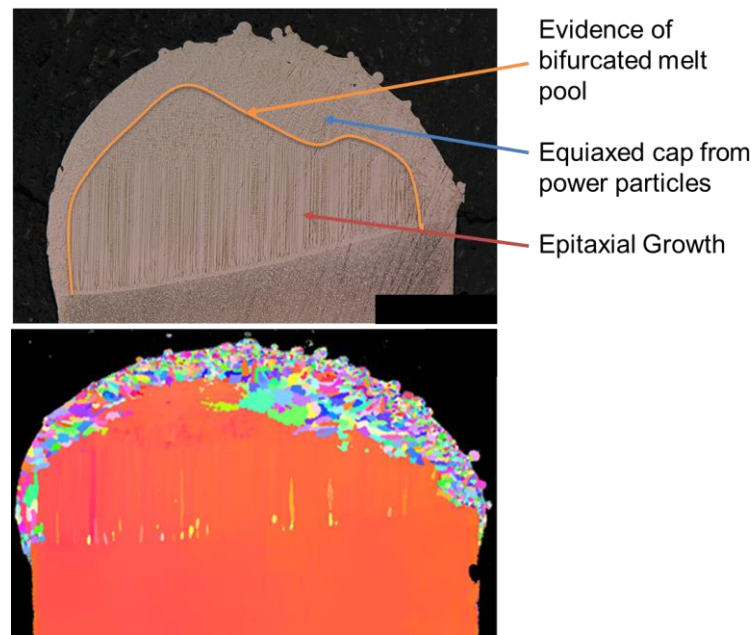


Figure 62: Evidence of bifurcated melt pool in deposit L-3 shown by optical microscopy (top) and EBSD (bottom) imaging. Peak in epitaxial growth is likely due to bifurcation resulting in solidification at this point before solidification of equiaxed cap around it.

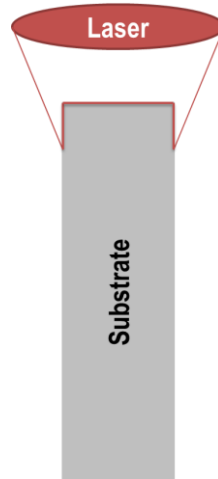


Figure 63: Laser substrate interaction in thin wall substrate geometry where substrate width is smaller than the laser diameter. Heating of the substrate occurs on the substrate surfaces highlighted in red.

#### 4.5.2 Role of Powder Particles in Formation of Equiaxed Cap

The addition of powder particles has two effects on the formation of the equiaxed cap for thin wall substrate. First, powder may form an equiaxed “cap” on the surface of the melt. This occurs because powder acts as a heat sink, as discussed in 4.1.2. If powder acts as a heat sink, the presence of solid powder particles can promote rapid top surface solidification and form a solid crust. Figure 64 shows a deposit microstructure where the track top surface crust has solidified, and dendritic growth opposes the bulk epitaxial solidification structure. This microstructure suggests that solidification of the material within the bulk can lag that of the outer surface of the track. Within the molten bulk, growth competition can occur between epitaxial growth and solidification from the outer crust inward [29]. Decreasing heat input reduces the cross-sectional area of the observed molten core. The effect of mass flux on the cross-sectional area of the molten core could not be determined. Thermal gradients and solidification velocities within the bulk and crust appear to be significant and deserved further consideration in modeling of the DED process. Second, an increase in mass feed rate increases equiaxed cap depth as shown in Figure 65. A reduction in mass



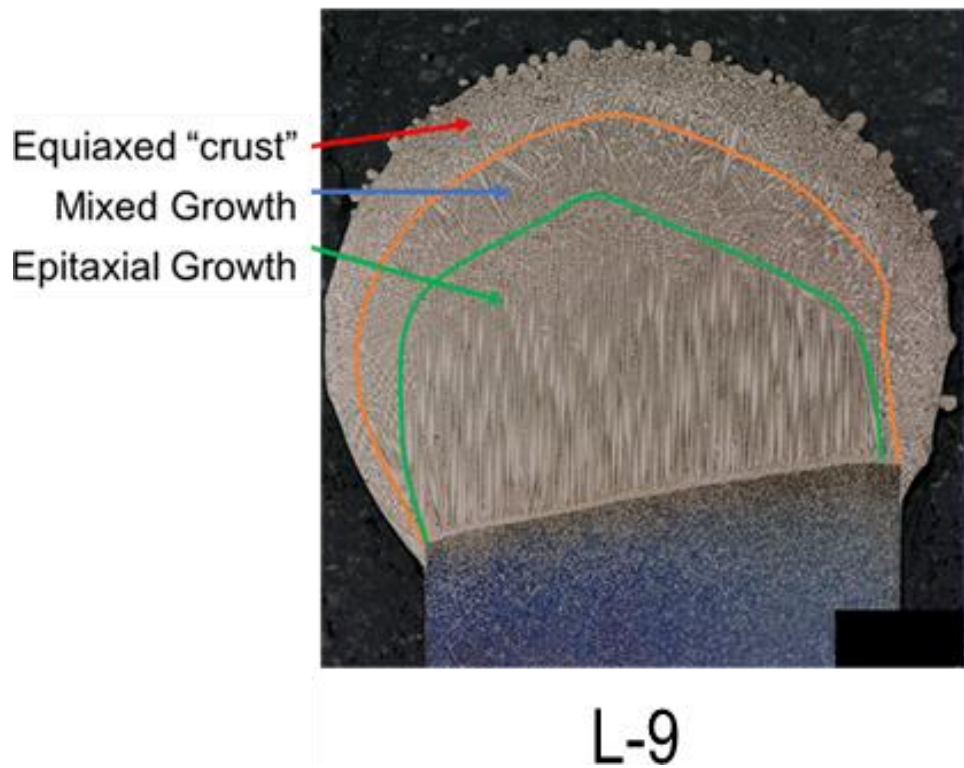


Figure 64: Formation of equiaxed crust during deposition in thin wall case. Formation of solid equiaxed crust results in growth from the substrate and crust inward resulting in mixed growth region.

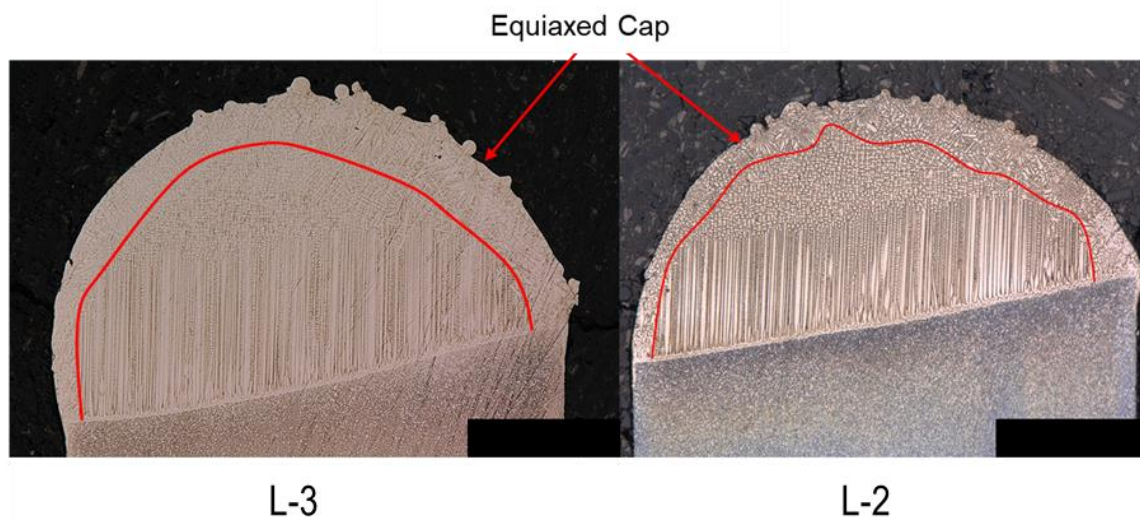


Figure 65: Width of equiaxed cap increases with increasing mass feed rate (mass feed rate increases from L-2 to L-3)

feed rate, and thus mass flux, will reduce the amount of partially melted powder particles in the deposit, reduce  $N_0$  and thus the propensity for equiaxed material to form.

#### 4.6 Multi-Pass Thin Wall Deposits

An experimental assessment was completed to better understand the impact of remelting on deposit texture evolution over ten layers. Laser travel is assumed to always occur along the X-direction. The build direction is the positive Z-direction. The same trends for increases in cap depth are seen in these samples as were observed for single-pass thin wall deposits.

##### 4.6.1 Effect of Processing Parameters on Deposit Geometry and Epitaxial Growth

In the multi-layered samples, an additional parameter, pause time, was added to the design of experiments. This parameter is directly related to the amount of residual heat in the deposit. Decreasing pause time generally resulted in an increase in deposit volume and deposit height. Figure 66 shows three deposit microstructures produced using three different pause times which decrease from left to right. At relatively long pause times, a change in texture, decrease in deposit volume and decrease in deposit height was observed. In a sample where epitaxial growth was limited, this large increase in pause time resulted in an increase in the amount of epitaxial growth.

Time dependent variation in deposit geometry was observed in multi-layered deposits. This effect can be seen in Figure 67, where the deposit width is maintained within the width of the substrate until a point where the amount of melting is increased to the point where the melt volume, and subsequently the deposit width, increases to a point where the deposit width is no longer less than the original substrate width. The time dependent variation in track geometry may be influenced by heat accumulation during the deposition process and tends to increase with increasing heat input.

Variation in epitaxy was observed in samples M-2 and M-5. Images showing variation in epitaxy for these samples is shown in Figure 68 (a) and (b) where low laser power and low travel speed conditions were used. It is theorized that transient



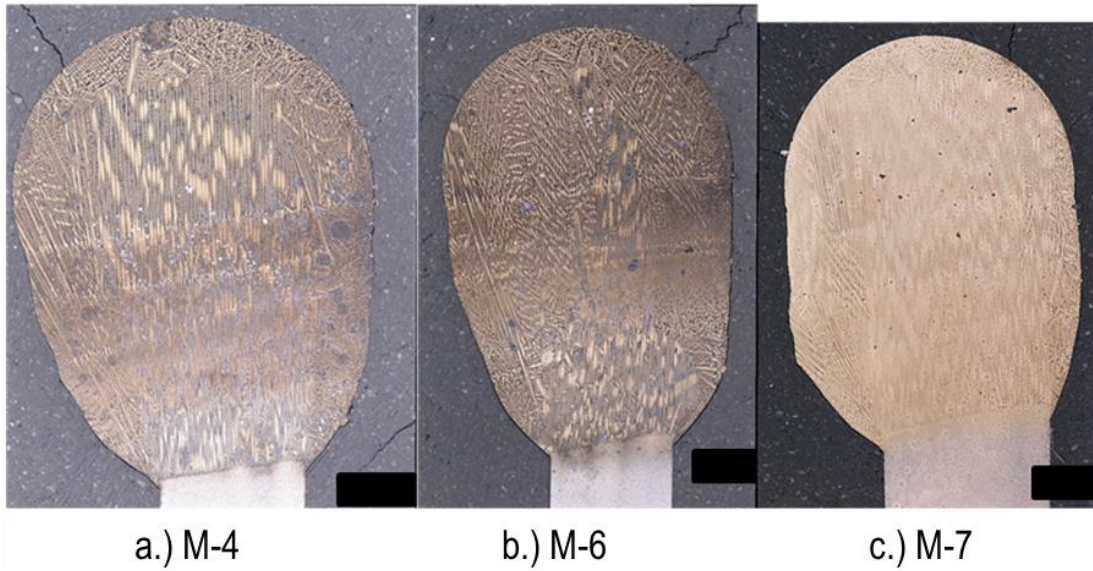
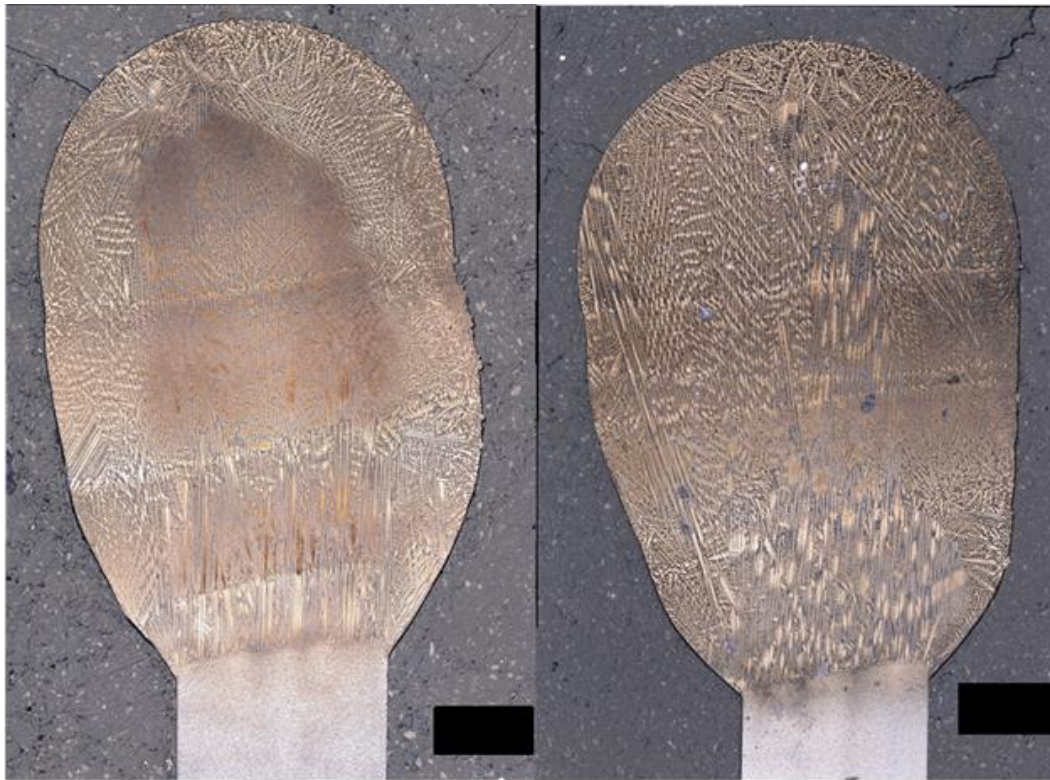


Figure 66: Effect of increasing pause time (left to right) on epitaxial growth and deposit volume, shape and height. Increasing pause time decreases deposit volume, height and increases epitaxial growth.



Figure 67: Change in deposit width from less than the width of the original substrate to greater than the original substrate width at higher layer numbers results in off-axis columnar growth. Off-axis growth occurs only at locations where the deposit width exceeds the substrate width.



a.) M-2

b.) M-5

Figure 68: Epitaxial growth after some layers is lost with epitaxial growth dominating the remaining layers at two high heat input cases

conditions may promote off-axis growth and equiaxed grains to form as the DED process progresses (with deposition of additional layers).

With decreasing powder mass feed rate, a reduction in deposit volume, deposit height, and equiaxed cap depth is seen. This is seen in Figure 69 shows deposit microstructures obtained while varying mass feed rate. Reducing mass feed rate resulted in thinner cap depth, lower track width, and smaller deposit volume.

With complete melting of powder, decreasing mass flux, heat input and substrate temperature have all been shown in this work to improve epitaxial growth with addition of layers. In samples where epitaxy was not maintained at higher mass feed rates, a decrease in feed rate can allow for epitaxy to be maintained. This is likely due to a reduction in cap depth due to powder introduction and increased melt depth since more energy is used to melt the substrate or prior layer as less energy is extracted by powder particles for melting. In samples where a loss of epitaxy occurs at higher layers, decreasing the substrate preheat or decreasing the heat input may mitigate or eliminate deposit width increases and off-axis or equiaxed growth.

#### 4.6.2 Effect of Single-Track Texture and Remelting on Multi-Layered Epitaxy

The primary influence on epitaxial growth with multiple layers is the amount of epitaxy in the first layer. Variation in track geometry also tends to correlate with variation in deposit texture evolution. Texture from the first layer of deposition is usually maintained and carried through additional layers if it is not completely remelted by the next layer. These effects can be seen in EBSD imaging in Figure 70. While the equiaxed cap is more readily remelted, off-axis growths and equiaxed material at the side walls are not as easily removed by remelting. This can result in columnar off-axis growth locations throughout the deposit resulting in directionally solidified material. This is shown in Figure 70 where EBSD of a single-track near-edge and a multi-layered near-edge at the same parameters is compared. The small equiaxed cap is remelted at the center leaving only the equiaxed cap formed at layer 10 and a thin side wall of equiaxed material. This side wall is a result of the equiaxed cap at the substrate edges which is not completely remelted. While no equiaxed material is seen in the deposit



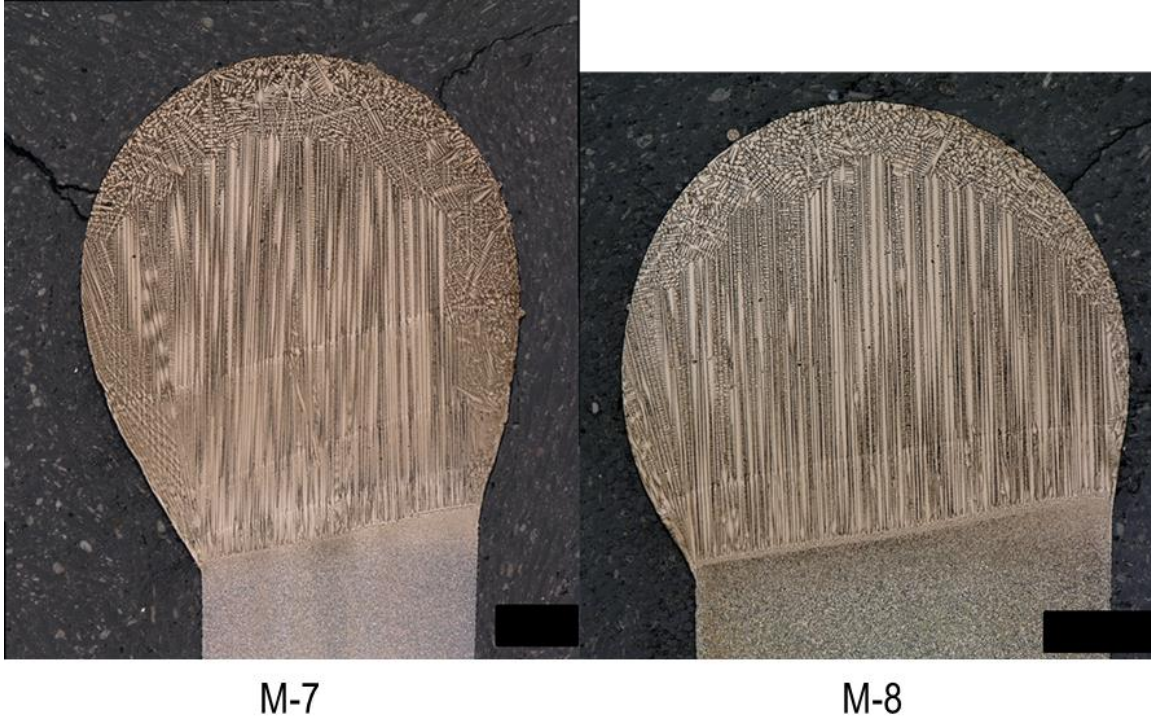


Figure 69: Effect of reduction in mass feed rate (M-7 to M-8) on epitaxial growth, equiaxed cap formation and deposit shape, volume and height. Equiaxed cap and deposit height, width and volume decrease.

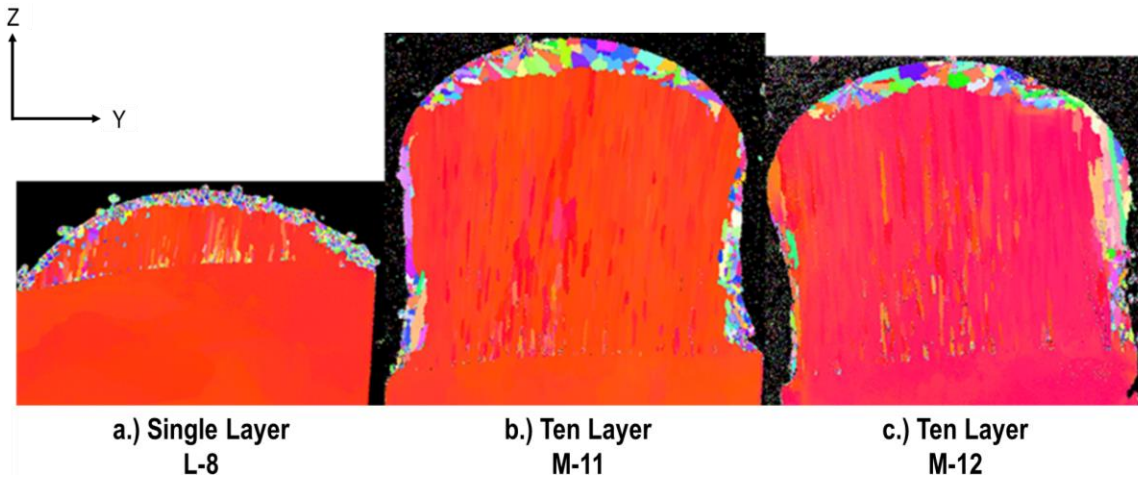


Figure 70: Effect of off-axis growth which occurs at the liquid-solid boundary on texture evolution in multi-layered deposits. Without complete remelting of off-axis growth sites, off-axis columnar growth occurs through multiple layers.

bulk, off-axis growth sites which formed at layer 1 are not completely remelted resulting in off-axis growth that progresses from layer 1 through layer 10.

As shown in this work, as well as literature as described in Chapter 2, epitaxy was maintained in samples where the equiaxed cap was completely remelted in the subsequent layer pass. In production of multi-layer samples in this study, single-pass samples that showed excessive substrate heating and deposit widths that exceeded that of the original substrate were avoided. This had the added effect of avoiding many of the samples with thick equiaxed caps.

Substrate and time dependent deposit geometry play a profound role in the ability to maintain single-crystal growth and must be accounted for during process parameter development. With a change in substrate geometry or narrowing of the heat transfer pathway that results from multiple layers on a wider substrate, the conditions that result in epitaxial growth may change. The microstructural features that form may change as well. Off-axis growth was seen in all thin wall substrate geometries. In comparison, off-axis growth was far less prominent in thick plate substrate deposits. Accounting for the various microstructural features that can form as a result of a change in substrate geometry, and thus the heat transfer conditions and pathway, is necessary in the development of a successful parameter set for single-crystal repairs.

#### 4.7 Thin Wall Substrate Processing Maps

Processing maps for thin wall deposits were produced showing the effects of varying laser power and travel speed on cap depth and cap percent. Cap percent is the depth of equiaxed cap to the total height of the melt. This calculation can be found in equation 23 in Appendix 2.

Cap depth tends to decrease with decreasing power and increasing travel speed. This trend can be seen in Figure 71. Increasing travel speed tends to exhibit a greater influence on cap depth at higher laser powers. The variation in slope may be indicative of a complex interaction between these two variables and other process parameters, such as mass flux. Further investigation of this theory is necessary.

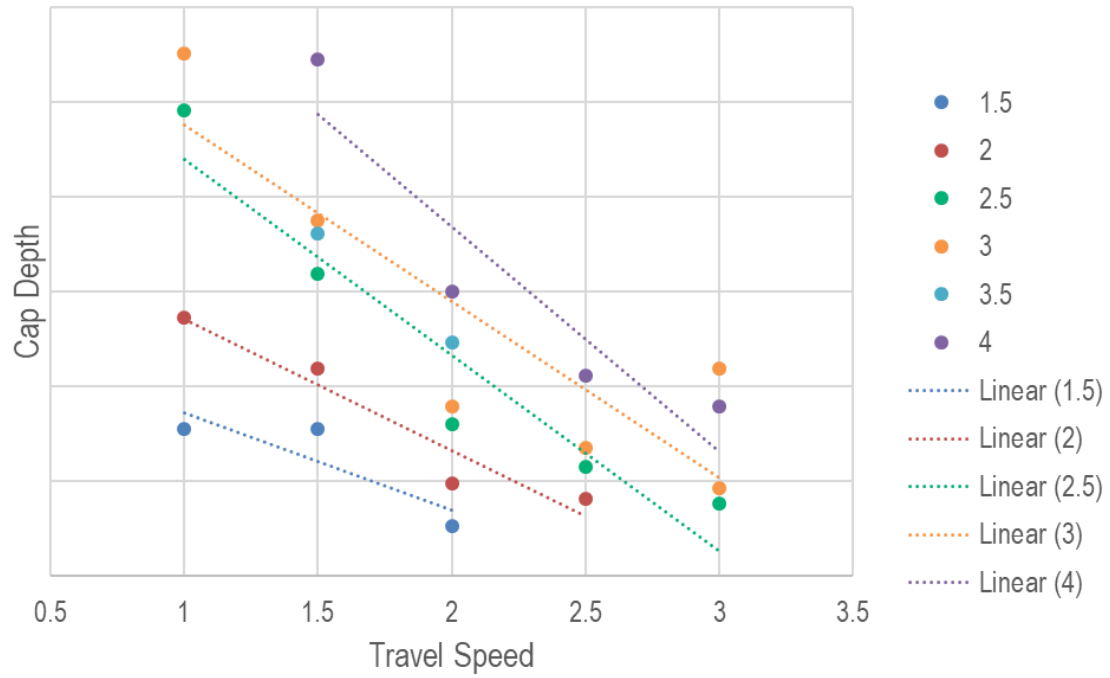


Figure 71: Cap depth vs normalized travel speed as a function of normalized laser power. Increasing travel speed and decreasing laser power decrease cap depth.

Figure 72 is a graph showing cap percent vs normalized travel speed vs normalized laser. An inflection point occurs at a velocity of 1.5 with a slightly increasing trend after the sharp increase at 1.5. The inflection point is likely the result of mass addition acting as a secondary effect in this plot. Comparison of the plot of cap percent and cap depth shows that while cap depth always decreases with decreasing laser power or increasing travel speed it is not always proportional to the increase in deposit height resulting from the increase in heat flux. This is important for maintenance of epitaxial growth as at the processing conditions which lie on the inflection point the amount of remelting as compared the depth of weld pool may be higher. Work is necessary to further understand the implications of the inflection point in cap percent.

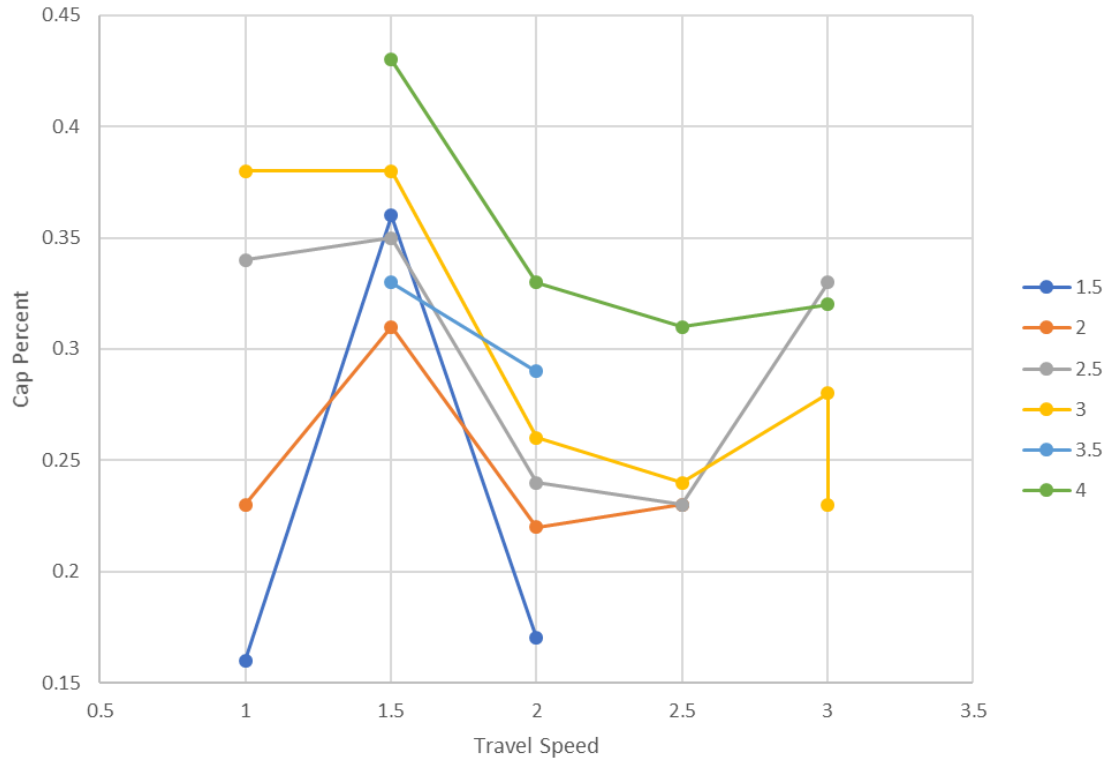


Figure 72: Cap percent vs normalized travel speed as a function of normalized laser power. Increasing travel speed initially increases cap percent with eventual plateau at higher travel speeds. Increasing power increases cap percent.



## Chapter Five: Future Work

There are several opportunities for research either beyond the scope, or as a follow-up study, of the present work that have been identified in the course of the work. These future studies would enhance understanding of the Directed Energy Deposition (DED) process, the effect of DED parameters on single-crystal solidification and improved modeling of the DED process with or without a single-crystal substrate.

### 5.1 Uncertainty in Processing and Modeling of Directed Energy Deposition and Single-Crystal Solidification

Capture efficiency is a primary source of error in DED process modeling. While models have been produced, such as the one used by Knapp et al in their predictions of DED deposits [35], accuracy of these models are not perfect. These imperfections introduce errors to modeling of the temperature field, flow field, solidification thermal gradient, and solidification velocity in addition to the accuracy of the deposit shape and size these models look to predict. Capture efficiency is a function of numerous parameters, such as melt pool top surface area, laser power, mass flux, powder size and material. This makes capture efficiency models with high accuracies difficult to develop. Models which predict deposit shape and size are thus only as good as the accuracy of the capture efficiency value or model which is used.

Understanding the formation of columnar and equiaxed material by these models relies on calibration of the CET model. The CET model is a function of several process and material related values as discussed in Chapter 2.3. As shown by Haines [42], this model is most sensitive to changes in the number of nucleation sites. This value is sensitive to the process as well as the environment and cannot be measured directly. As such, calibrations of this model are rarely valid outside of the process and environment in which the calibrations occur. Still, analysis in several works, including this study, rely on calibration values for the CET model from literature. This may introduce further error into models which use the CET model for microstructural predictions.

As discussed in Chapter 4, results of this study show a spatial variation in  $N_0$  likely due to the effects of powder-melt interactions. While Liang et al propose a model for variation in  $N_0$ , due to powder, as a function of mass flux and power, this model will not capture the effects of a Z-height variation in the  $N_0$  value. For accurate representation by CET model, better understanding of the nature of the  $N_0$  value is necessary, including a  $N_0$  model that predicts variations due to height, heat input and other factors. Until this is determined, recalibration of the model to a new process or processing environment is recommended. In the DED process, further work into the variation in  $N_0$  due to powder interactions and heat input is needed.

While models for powder interactions during DED exist for laser attenuation, powder heating during flight, and the amount of powder introduced to the melt for a given powder flux distribution, no models include the melting of powder at the surface or its distribution along the surface. The energy removed from the melt due to the melting of powders, as shown in the change in substrate remelt depth in the semi-infinite plane heat transfer condition or the formation of a crust along the surface of the deposit in the near-edge heat transfer condition, affects the melt pool shape and the formation of the equiaxed cap. It has been shown in literature that the introduction of powder reduces the peak temperature of the puddle. This results in a decrease in forced convection due to Marangoni forces. If the powder particles act as a heat sink, melting of powder particles would also form disruptions in the temperature field, and thus the Marangoni force field, further impacting the flow field, shape of the melt and propensity to form stray grains.

Models for powder flight direction and distribution exist but are rarely packaged in modeling of the heat transfer and single-crystal solidification process. Understanding the flight path, time and completeness of melting are important in understanding the effects of powder on  $N_0$  and stray grain formation. As seen by Babu et al, introduction of particles into the fast-moving solidification fronts seen during welding and additive manufacturing can result in incomplete melting of particles [60]. Figure 73 shows the incomplete dissolution of a dissimilar particle during laser surface alloying. This would

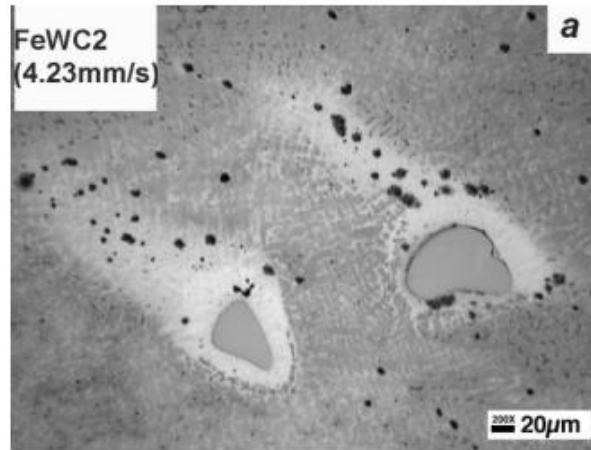


Figure 73: Incomplete dissolution of powder particles in laser surface alloying sample [60]

results in an increase in  $N_0$  and the possibility that the particle can act as an off-axis growth or stray grain initiation site. In understanding the formation of the equiaxed cap, off-axis growth and stray grain formation during DED repairs of single-crystal material, inclusion of these factors would be desirable. However, for computation time considerations, inclusion of these powder interaction models is difficult making this another source of possible modeling error.

While many models for the solidification of single-crystal welds and deposits have been produced, the uncertainties in modeling of the DED process are not fully understood. Inherent in the DED process itself are fluctuations in laser power, travel speed, and mass flux that occur during deposition. Additionally, deviation from the laser power, travel speed and powder flux distribution set in the controller occur with use. These fluctuations can have a large effect on the melt pool size, thermal gradient and solidification velocities. While changes in laser power and travel speed can be quantified using commercially available data logging systems, accurate methods of quantification of instantaneous mass flux and area do not currently exist. With changes in offset distance between the part and nozzle, due to process fluctuations, layer heights of the part not matching the value set in the controller or curvature of the deposit surface result in changes in the powder distribution area and laser spot size. This will

result in additional changes in heat flux and mass flux. These fluctuations due to the process itself are difficult to quantify and their inclusion in modeling will necessitate adding more calibration parameters and will increase the computation time.

## 5.2 Improvements to Modeling Method

Results of this work show that the effect of mass addition to the thermodynamics of the DED process is significant and that effective modeling of the DED process must account for the powder flow into the melt pool, especially in prediction of G-R in DED deposits, single-crystal solidification and deposit shape. Improved modeling of the DED process should include a model for powder capture into the melt pool and energy consumed in heating of powder particles and in powder particle melting, to accurately predict the formation of the deposit, shape and size of substrate melting, amount of mass capture, and solidification conditions.

A mass addition model should include extraction of energy from melt and the location at which this extraction occurs. Shown by the modeling results in the thick plate case, a single laser efficiency value may not account for the amount of energy extracted by the powder nor its distribution. These effects are important for understanding the shape and size of substrate melting and predictions of deposit shape and solidification conditions. An analytical formulation for the mass addition model is recommended in the case of understanding single-crystal solidification. Use of a FEM to solve for the heat transfer and fluid flow conditions alone is computationally heavy. With the addition of a VOF or analytical formulation of the deposit shape, this computational load will significantly increase. Using another analytical layer to calculate the heat extraction by the powder would allow for a larger area to be modeled in a reasonable amount of time.

In this work, the substrate size was assumed to be exactly what was specified and the Z-height increase between layers equivalent to the single-track layer height. This assumption brings some amount of error into the production of layered deposits. However, the extent of this error is not known. In addition to the change in powder flux and its distribution, evaluated by Haley [32], the converging laser will result in a different spot size at the surface for a change in offset height. Evaluated by Haley [32], it is

known that the DED process design provides a passive feedback system to level out variations in deposition height. This study agrees with these findings. However, this study did not evaluate the change in heat flux and solidification that may result due to the converging-diverging nature of the laser. Additionally, it is known that variations in absorbed laser power, mass flux and travel speed can be seen during production. Understanding how these variations do or do not affect laser spot size, capture efficiency, and solidification conditions is needed to maintain the correct processing conditions during multi-layered deposits.

While variations in applied laser power and travel speed can be evaluated using data logging systems, powder mass flux and layer height are not measured so easily. Use of real time layer height measurements, through a technique such as laser profilometry, may allow for the addition of a new feedback system to accurately set the Z-height increase during processing. This could leverage the passive system described by Haley to correct small fluctuations in height while keeping the deviation in laser spot size and heat flux within a smaller bound. Additional research and development is necessary to understand the variation in mass flux during processing.

Further work should be performed to determine if the direction in which the powder particle travels towards and into the melt pool affects the resulting texture of the deposit. The size of the powder stream may also affect the amount of powder that is completely melted. In powder fed single-crystal repairs, this work, and those reviewed in the literature, show the existence of an equiaxed cap that forms at the surface. This cap may be due to effects impacting solidification velocities and thermal gradients near the point of impact of powder particles and increases in  $N_0$  in this area with incomplete melting of the particles. Further experiments are needed to understand the effects of variation in spatial mass flux, and powder stream impingement location/angle on epitaxial growth and the formation of the equiaxed cap.

Further work should be performed in further understanding the effect of a change in substrate thickness on epitaxial growth. While there is a noticeable change in the

microstructural features in the thin wall case (such as the increased amount of off-axis growth at the solidification interface), the reason for this change is not clear.

Use of modeling in the thin wall geometry is also advised. While a FEM can be applied to this type of geometry, time constraints prevented the application of the model to this case. Using modeling of the thin wall geometry, with the addition of a deposit shape model, allows for one to understand the evolution of melt size, propensity to melt through the equiaxed cap, and evaluation of the increased preheat caused by the addition of layers. It is known that an increase in preheat temperature will increase melt size. Using modeling to understand this effect, one can determine the point at which the increase in deposit width to greater than the substrate width is favorable and the amount of (remelting of prior layer (s)). Both of these effects have been shown to be important in maintaining epitaxial and single-crystal growth. Understanding the preheat also allows for evaluation of the limit in which columnar growth is no longer favored by the CET. An increase in preheat may result in a decrease in thermal gradient, pushing the solidification conditions toward equiaxed or mixed growth. It is feasible that a sufficient increase in preheat will make columnar growth impossible.

For future application of the modeling efforts in this work, accuracy should be improved through the addition of an analytical model to predict deposit geometry, dendrite growth variant model, an update of the heat source model and a powder-laser interaction model. An analytical model to predict deposit geometry allows for an increase in accuracy and modeling capability while not increasing the computational load as substantially as a free-surface model. Application of the dendrite growth variant model is necessary for the accurate prediction of solidification thermal gradient and solidification velocity. Updating of the laser model such that the effect of the converging laser on heat input is considered allows for the overflow effect seen in the near-edge heating, and the more complex shape of the deposit to be better handled. Adding in a subroutine to account for the powder-laser interactions begins the process of producing a model to include the effect of energy extraction due to powder and its distribution along the surface.

### 5.3 Variation in $N_0$ during Directed Energy Deposition

Review of the literature showed efforts to describe a variation in  $N_0$  [10]. This work supports continued development of a  $N_0$  variation model. A  $N_0$  model should include the heat input and mass flux, as was proposed by Liang, with the addition of a model to handle the variation with Z-height. This work agrees with the existence of a variation in  $N_0$  with height. However, without accurate modeling of the change in G-R in the deposit or understanding of how to experimentally measure  $N_0$ , determination of the extent of this change cannot be easily determined.

Using the results from modeling, columnar-to-equiaxed transition curves were produced, shown together in Figure 74 and separately in Figure 90 and Figure 91 in Appendix 5. The red CET curves were produced using the code produced by Haines et al [42] using the  $N_0$  values determined by Gaumann in his work with CMSX-4 [24]. From these curves, the CET model predicts that all modeling cases would result in equiaxed material. Analysis of experimental results shows that this is not the case as single-crystal material is achieved in the remelted substrate portion of the deposits. The black CET curves were produced using the  $N_0$  value determined by Haines for the EBM process. These curves show all processing conditions falling within the columnar region. While variations in G-R in the deposit are expected (these were unable to be measured by the current modeling approach), the experimental and modeling results point towards a variation in  $N_0$  as a function of Z(build)-height.

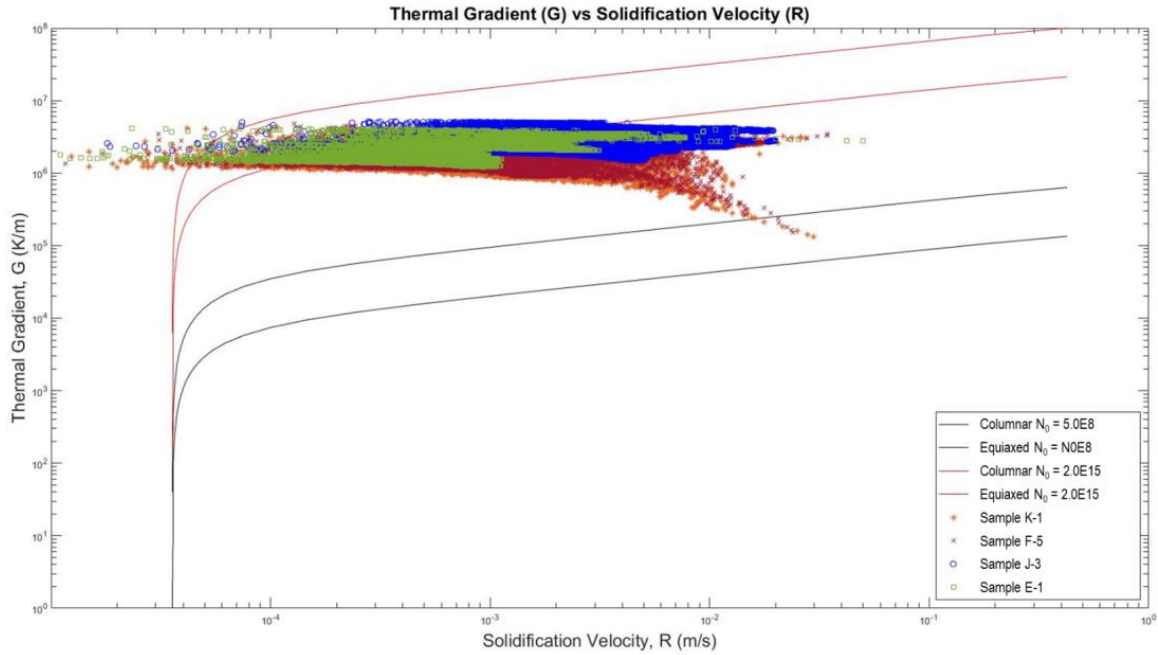


Figure 74: Columnar-to-equiaxed transition curves including data from OpenFoam model.  $N_0$  calibrated by Gaumann predicts equiaxed or mixed growth while a very conservative  $N_0$  from electron beam melting process predicts columnar growth as seen in region of these samples which was modeled (substrate region which was melted).



## Chapter Six: Summary and Conclusions

The results of this study show that with through process parameter optimization, epitaxial growth may be more favorable in Directed Energy Deposition (DED). This was demonstrated for multiple substrate geometries and heat transfer conditions when the laser was near the edge of the substrate (thin wall geometry) and relatively far from the edge of the substrate (thick plate geometry). The ability to maintain epitaxy is dependent upon laser heat flux, powder mass flux, and substrate geometry.

Furthermore, it is shown that:

1. Epitaxial growth was successfully demonstrated across various geometries and relationships between several key process parameters and deposit microstructural texture characteristics were identified.
2. Remelting of the equiaxed cap is a key factor influencing epitaxial growth in multi-layered deposits.
3. Depth of the equiaxed cap which forms in all samples can be reduced through decreasing heat and mass flux. When the substrate geometry is relatively thin, maintaining the deposit width equal to or less than the substrate width reduces or eliminates the formation of equiaxed side walls on the deposit.
4. Powder particles can act as a heat sink for the range of process parameters considered.
5. The addition of mass plays a significant role in the dynamics of the melt pool and solidification.
6. Dendrite growth competition between solidification occurring from the deposit bulk and outer equiaxed crust can substantially influence microstructure.
7. The number of nucleation sites ( $N_0$ ) within the melt may change as a function of deposit height.
8. Transient phenomena may occur which promote variation in deposit texture over multiple layers which do not directly correlate with laser power, travel speed, and mass flux alone.

9. Production of spiral path samples for process parameter development is useful and efficient to assess a wide range of process parameters.

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## Appendices

## Appendix 1: Single-Track, Thick Plate Substrate Geometry Deposits

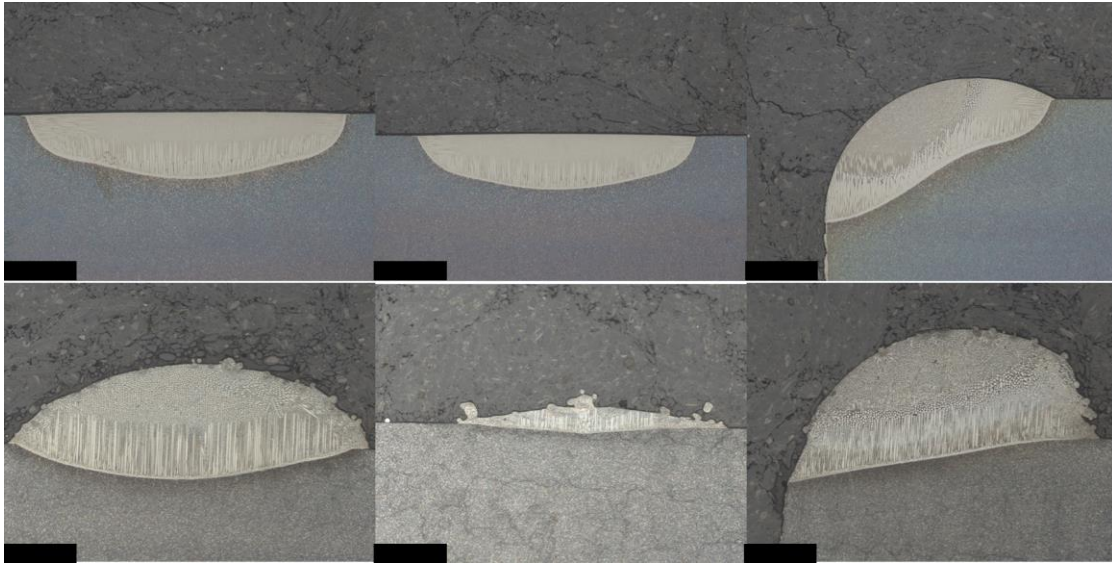


Figure 75: Spiral Sample A-1 through A-3 track geometry without mass addition (top) and Sample E-1 through E-3 track geometry with mass addition (bottom) transverse cross sections from 180-degree section location. Travel speed increasing from left to right.

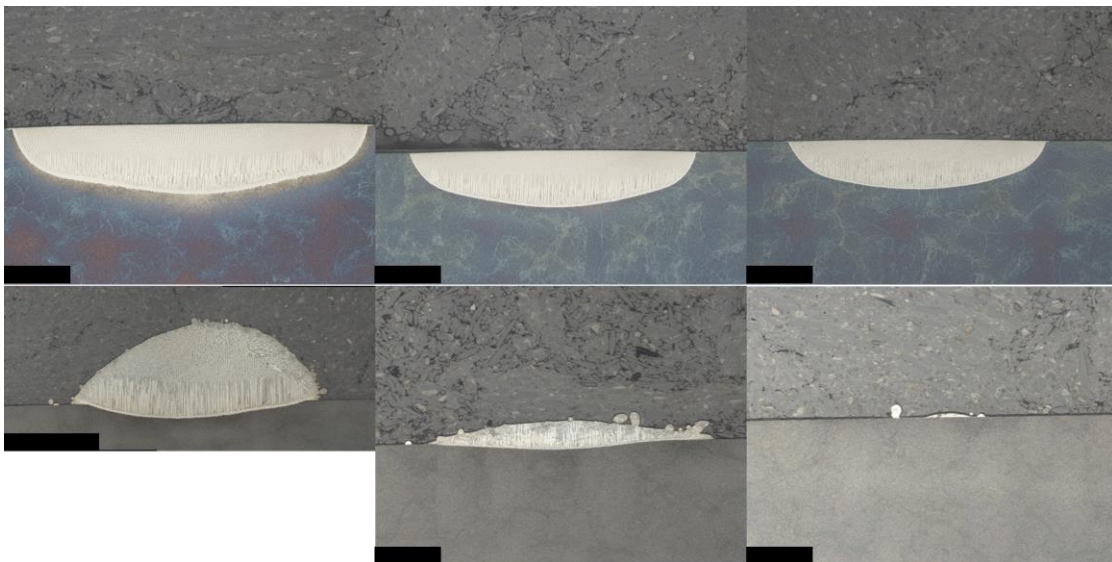


Figure 76: Spiral Sample A-4 through A-6 track geometry without mass addition (top) and Sample E-4 through E-6 track geometry with mass addition (bottom) transverse cross sections from 135-degree section location. Travel speed increasing from left to right.



Figure 77: Spiral Sample B-1 through B-3 track geometry without mass addition (top) and Sample F-1 through F-3 track geometry with mass addition (bottom) transverse cross sections from 180-degree section location. Travel speed increasing from left to right.

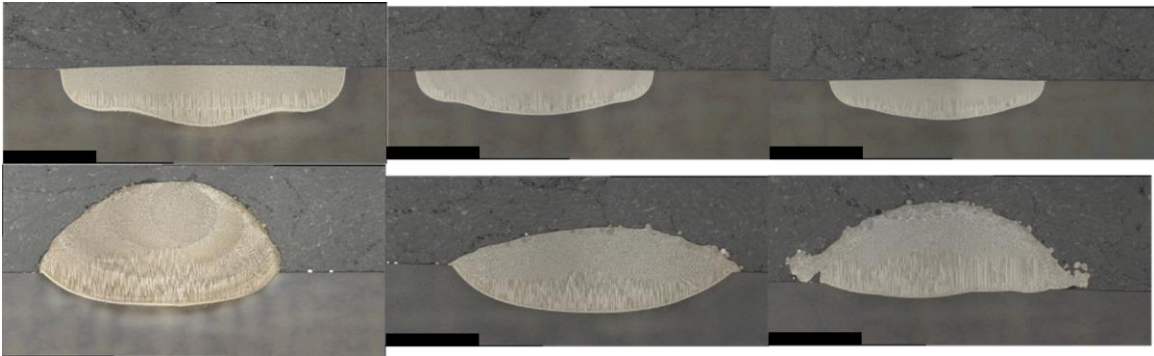


Figure 78: Spiral Sample B-4 through B-6 track geometry without mass addition track geometry (top) and Sample F-4 through F-6 track geometry without mass addition (bottom) transverse cross sections from 135-degree section location. Travel speed increasing from left to right.



Figure 79: Spiral Sample D-1 through D-3 track geometry without mass addition (top) and Sample H-1 through H-3 track geometry without mass addition (bottom) transverse cross sections from 180-degree section location. Travel speed increasing from left to right.

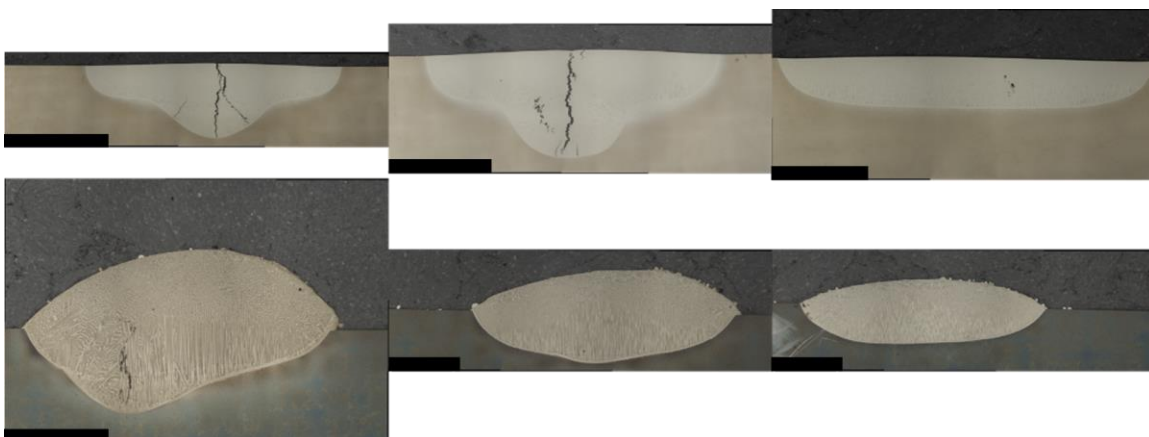


Figure 80: Spiral Sample D-4 through D-6 track geometry without mass addition track geometry (top) and Sample H-4 through H-6 track geometry without mass addition (bottom) transverse cross sections from 135-degree section location. Travel speed increasing from left to right.

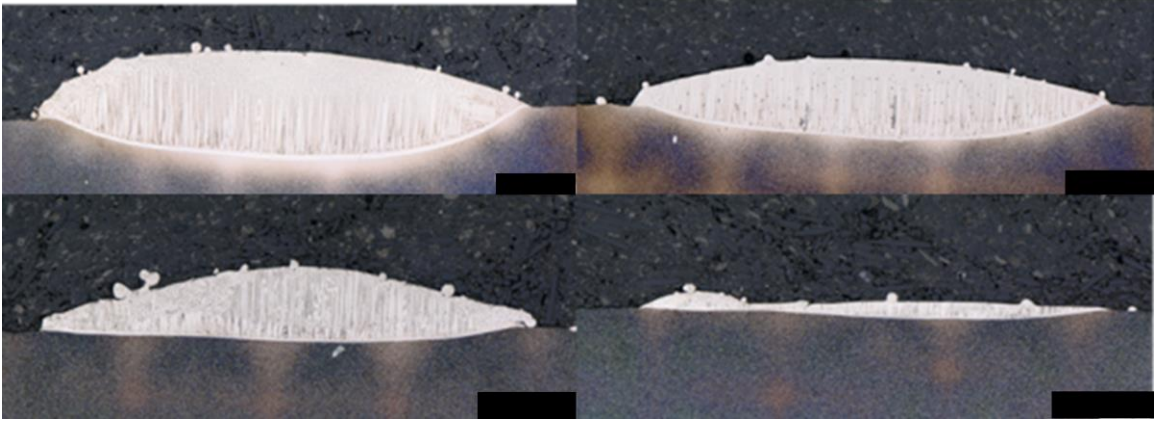


Figure 81: Sample I-1 (top left), I-2 (top right), I-3 (bottom left) and I-4 (bottom right) optical microscopy transverse cross sections for single-track thick plate deposits.



Figure 82: Sample J-1 (top left), J-2 (top right), J-3 (bottom left) and J-4 (bottom right) optical microscopy transverse cross sections for single-track thick plate deposits.



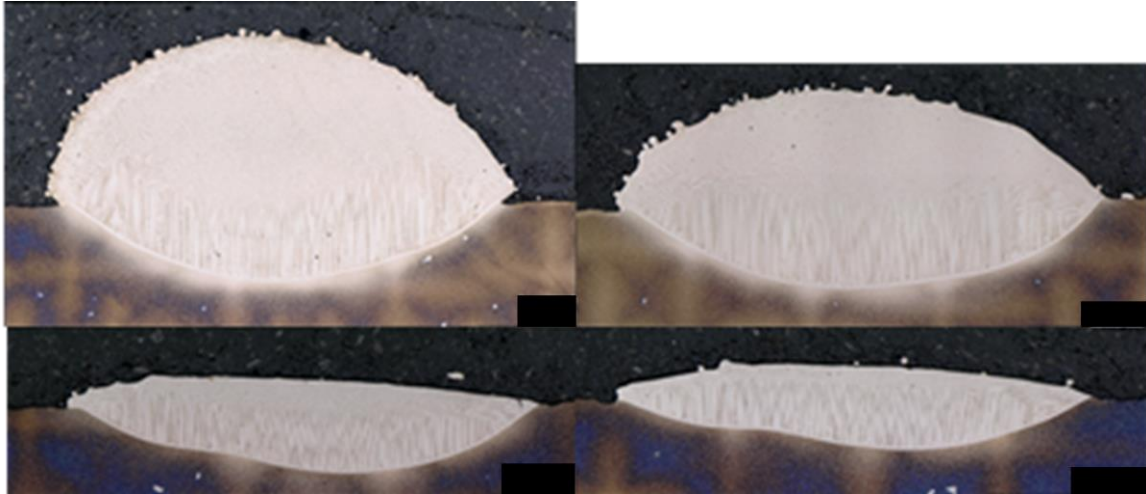


Figure 83: Sample K-1 (top left), K-2 (top right), K-3 (bottom left) and K-4 (bottom right) optical microscopy transverse cross sections for single-track thick plate deposits.

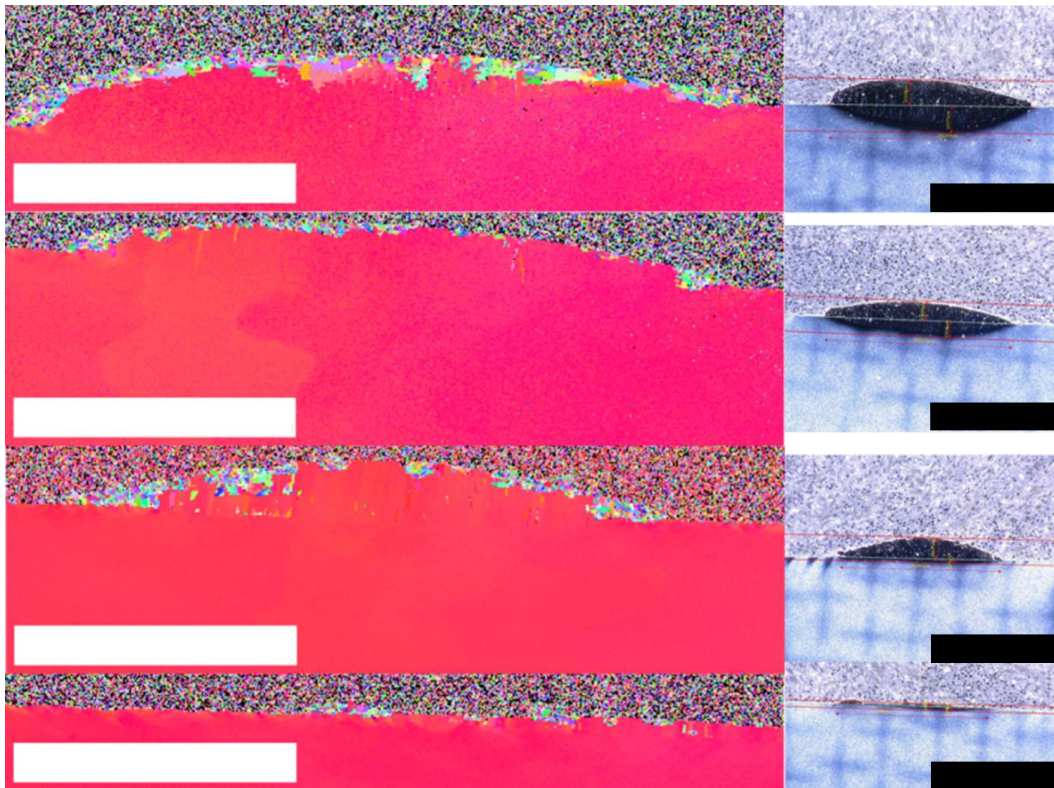


Figure 84: Sample I-1 (top) through I-4 (bottom) thick plate single-track transverse cross section EBSD and corresponding dark field images. Travel speed increases from top to bottom.

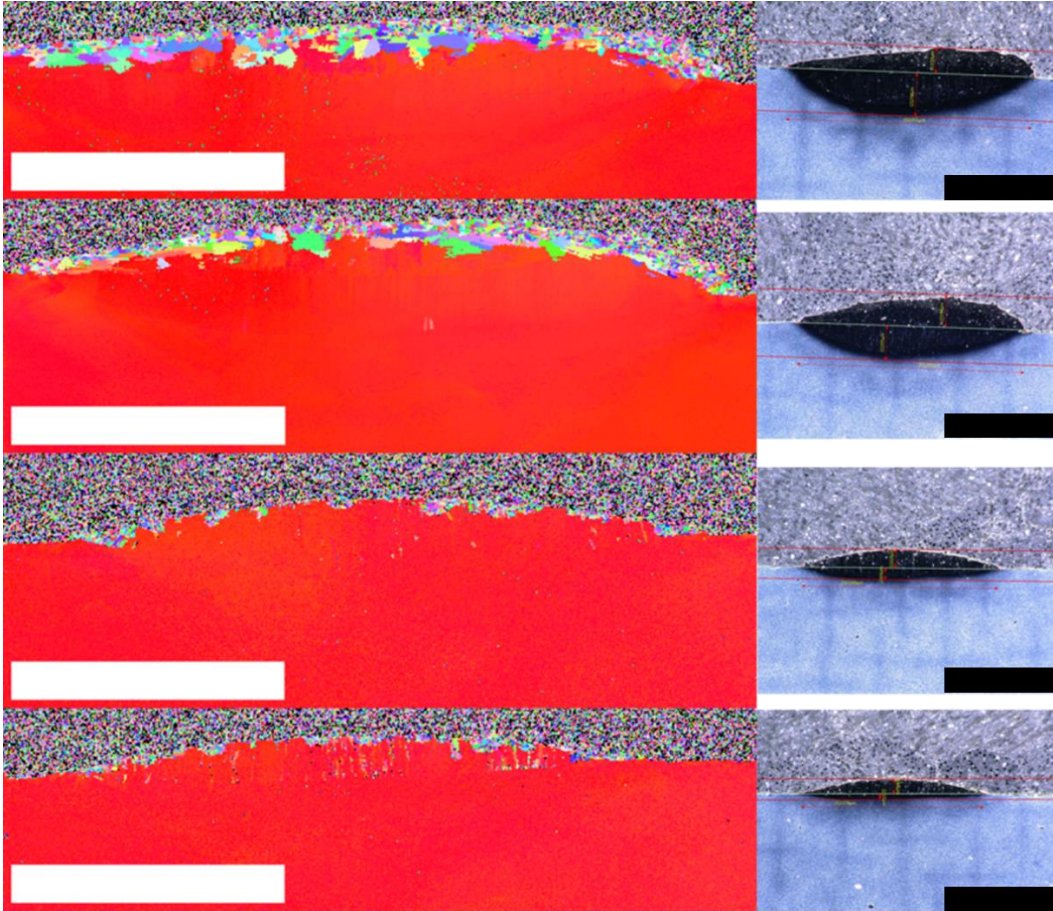


Figure 85: Sample J-1 (top) through J-4 (bottom) thick plate single-track transverse cross section EBSD and corresponding dark field images. Travel speed increases from top image to bottom.



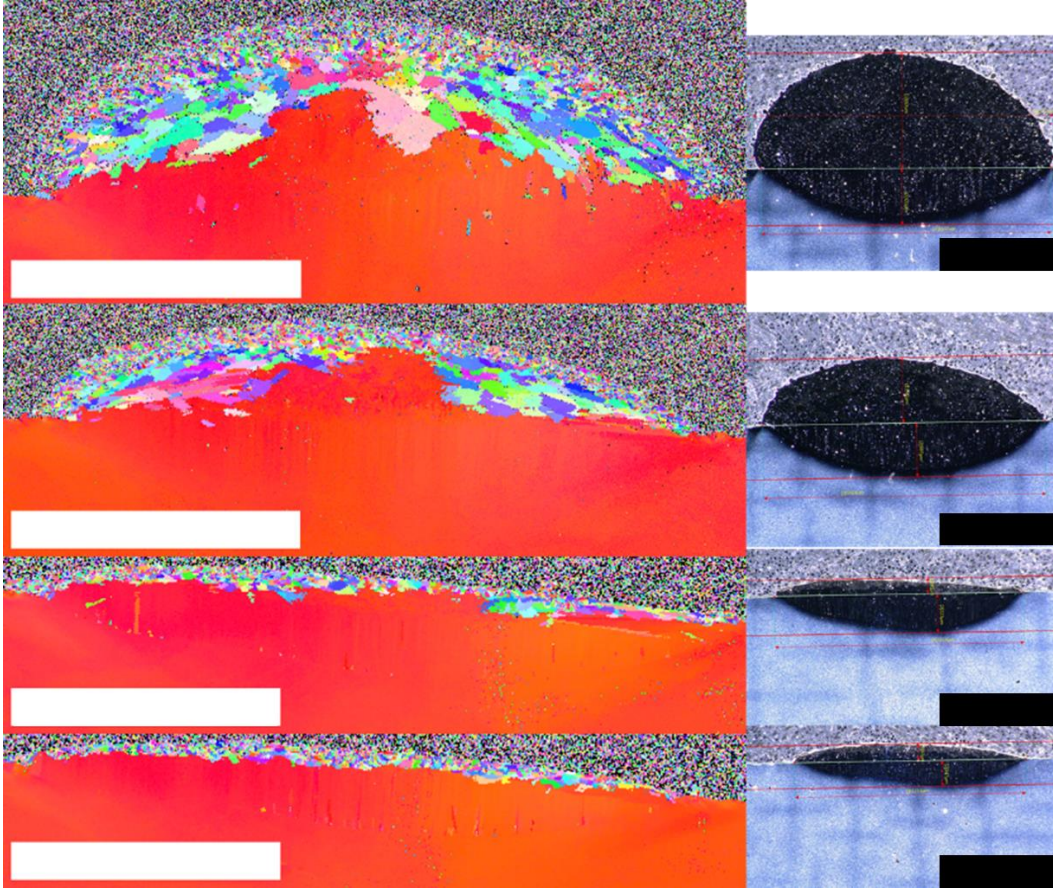


Figure 86: K-1 (top) through K-4 (bottom) thick plate single-track transverse cross section EBSD and corresponding dark field images. Travel speed increases from top image to bottom.



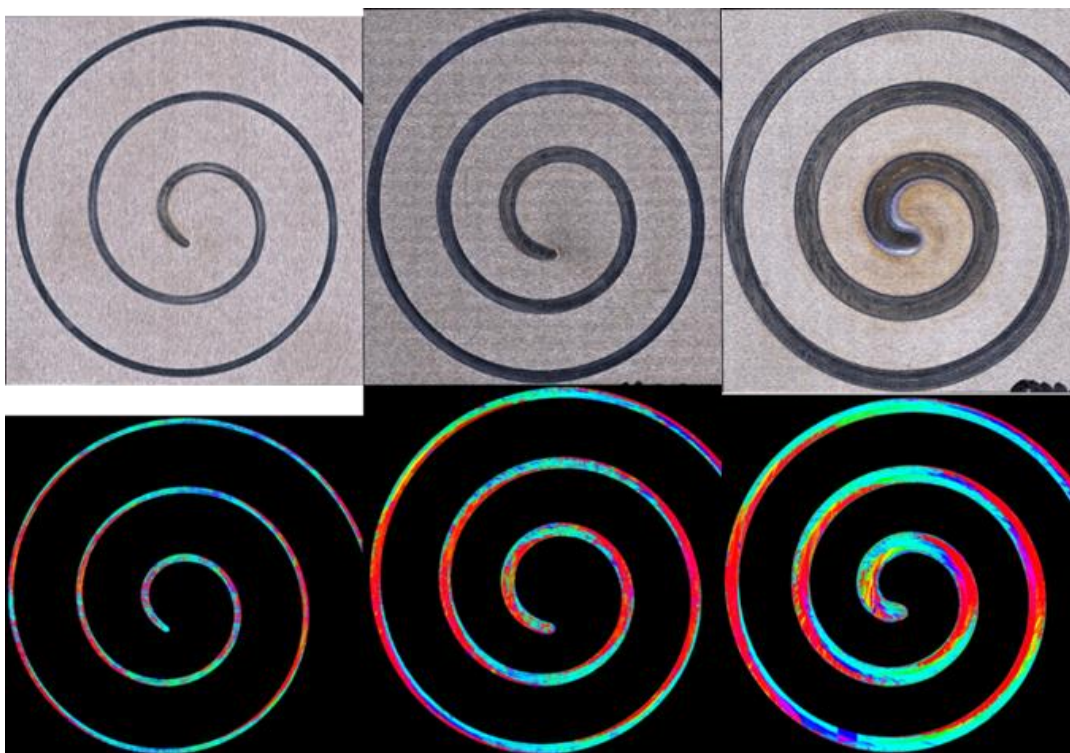


Figure 87: Spiral deposit without mass addition stitched optical (top) and image analysis color maps (bottom) for Sample A (left), Sample B (middle) and Sample D (right). Red corresponds to horizontal and light blue to vertical dendrites.

## Appendix 2: Measurement Equations and Schematics

Equation 9 was used to calculate the change in substrate melting (weld) width, depth or substrate melt area given:

$$\Delta x = \frac{x_{deposit} - x_{weld}}{x_{deposit}} \times 100 \quad (9)$$

where the variable x signifies width, depth or area. Area was approximated by best fit of an ellipsoid shape. A schematic of this measurement is provided in Figure 89.

The theoretical cross sectional area of the deposit in millimeters is defined by equation 20

$$A_{theoretical} = \frac{\frac{\dot{m}}{60} \cdot \frac{L}{v}}{\frac{\rho}{1e6}} \quad (10)$$

where  $\dot{m}$  is Powder feed rate in g/min,  $v$  is laser travel speed in mm/s,  $L$  is length in mm and  $\rho$  is density in kg/m<sup>3</sup>. The actual transverse cross sectional area is determined by equation 21 by assuming that the deposit is elliptical in shape.

$$A_{deposit} = \frac{\pi \cdot w_d \cdot h_d}{2} \quad (11)$$

Thus, the capture efficiency is a function of the theoretical and actual deposit areas as defined by equation 22.

$$Eff_{capture} = \frac{A_{actual}}{A_{theoretical}} \times 100 \quad (12)$$

Equation 23 shows the calculation for cap percent which related the cap depth to the total height of the deposit and substrate melting.

$$Cap\ Percent = \frac{Cap\ depth}{Deposit\ height + substrate\ melt\ depth} \times 100 \quad (13)$$

$$\text{Cap Depth} = \text{Total Height} - \text{SC/DS Height}$$

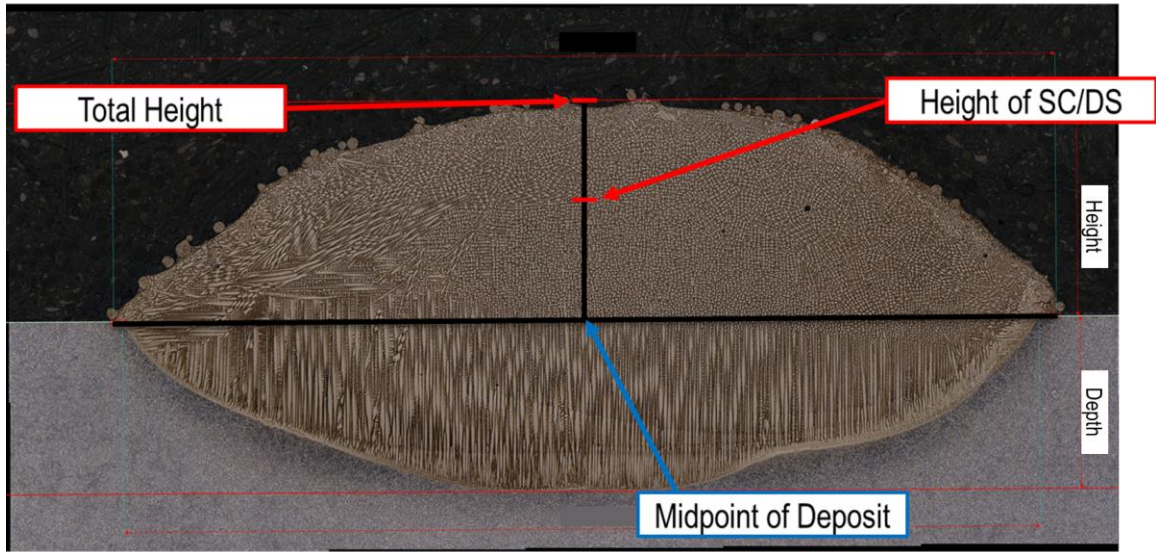


Figure 88: Cap depth measurement schematic. Cap depth is measured from the highest point of the deposit along the build direction

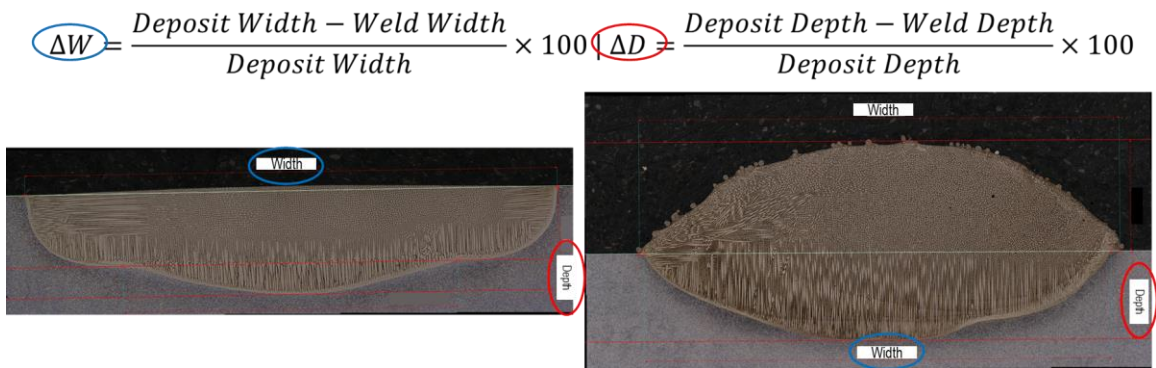


Figure 89: Calculation of change in substrate melt dimensions

### Appendix 3: Thick Plate Substrate Geometry Deposit Calculations and Measurements

Table 13: Thick plate deposit calculated capture efficiency

| Sample | Normalized Power | Normalized Travel Speed | Capture Efficiency |
|--------|------------------|-------------------------|--------------------|
| D-1    | 1.5              | 0.379                   | 10.95%             |
| D-2    | 1.5              | 1.137                   | 5.42%              |
| D-4    | 1.5              | 0.091                   | 4.56%              |
| D-5    | 1.5              | 0.855                   | 4.86%              |
| I-1    | 1.5              | 0.667                   | 15.25%             |
| I-2    | 1.5              | 1                       | 15.96%             |
| I-3    | 1.5              | 2                       | 29.82%             |
| I-4    | 1.5              | 3                       | 6.49%              |
| J-1    | 2                | 0.667                   | 25.97%             |
| J-2    | 2                | 1                       | 27.13%             |
| J-3    | 2                | 2                       | 26.58%             |
| J-4    | 2                | 3                       | 29.64%             |
| F-1    | 3.2              | 0.379                   | 35.92%             |
| F-2    | 3.2              | 1.137                   | 39.04%             |
| F-3    | 3.2              | 1.867                   | 47.94%             |
| F-4    | 3.2              | 0.091                   | 11.48%             |
| F-5    | 3.2              | 0.855                   | 29.96%             |
| F-6    | 3.2              | 1.622                   | 21.23%             |
| J-3    | 3.2              | 2                       | 26.50%             |
| J-4    | 3.2              | 3                       | 39.79%             |
| H-1    | 7.4              | 0.379                   | 71.64%             |
| H-2    | 7.4              | 1.137                   | 87.95%             |
| H-3    | 7.4              | 1.867                   | 87.82%             |
| H-4    | 7.4              | 0.091                   | 13.29%             |
| H-5    | 7.4              | 0.855                   | 54.65%             |
| H-6    | 7.4              | 1.622                   | 95.54%             |

Table 14: Change in substrate melt width, depth and area for the spiral deposit samples E, F and H with introduction of mass addition

| Sample ID | Normalized Laser Power (W) | Normalized Travel Speed (in/min) | $\Delta$ Width (substrate melt) | $\Delta$ Depth | $\Delta$ Area (substrate melt) |
|-----------|----------------------------|----------------------------------|---------------------------------|----------------|--------------------------------|
| A-1       | 1.5                        | 0.379                            | 0.53%                           | -129.76%       | -129.78%                       |
| A-2       | 1.5                        | 1.137                            | -26.87%                         | -535.43%       | -708.76%                       |
| A-3       | 1.5                        | 1.867                            |                                 |                |                                |
| A-4       | 1.5                        | 0.091                            | -5.03%                          | -127.59%       | -136.72%                       |
| A-5       | 1.5                        | 0.855                            | -15.27%                         | -546.89%       | -648.98%                       |
| A-6       | 1.5                        | 1.622                            |                                 |                |                                |
| B-1       | 3.2                        | 0.379                            | 15.94%                          | 8.41%          | 23.03%                         |
| B-2       | 3.2                        | 1.137                            | 3.63%                           | -8.21%         | -4.30%                         |
| B-3       | 3.2                        | 1.867                            | 1.35%                           | -16.00%        | -14.43%                        |
| B-4       | 3.2                        | 0.091                            | 7.95%                           | -18.43%        | -9.00%                         |
| B-5       | 3.2                        | 0.855                            | 3.65%                           | -9.58%         | -5.57%                         |
| B-6       | 3.2                        | 1.622                            | -3.60%                          | -137.46%       | -149.70%                       |
| D-1       | 7.4                        | 0.379                            | -12.49%                         | -39.53%        | -57.94%                        |
| D-2       | 7.4                        | 1.137                            | -24.33%                         | -64.61%        | -104.92%                       |
| D-3       | 7.4                        | 1.867                            |                                 |                |                                |
| D-4       | 7.4                        | 0.091                            | -14.89%                         | -45.12%        | -67.58%                        |
| D-5       | 7.4                        | 0.855                            | -24.04%                         | -70.76%        | -111.97%                       |
| D-6       | 7.4                        | 1.622                            | -8.08%                          | 5.60%          | 2.40%                          |

## Appendix 4: Code for Spiral Color Maps

Code for dendrite variant color maps for spiral weld images:

Author: Peter Zeglen

```
import cv2 as cv
import numpy as np
import math

#This is the only part you need to change when applying the code to a different image
#You just put in the correct image file, and the masked version
#Input spirals should have background and mushy area removed/masked
#The output will be written to "demo.bmp"
image = cv.imread("Spiral_NoPowder_newlighting.tif")
mask = cv.imread("Spiral_NoPowder_newlighting.tif",
cv.IMREAD_GRAYSCALE)[:image.shape[0], :image.shape[1]] == 0
print(image.shape)
print(mask.shape)
color = np.zeros((image.shape[0], image.shape[1], 3), np.float32)
for i in range(int(image.shape[0]/1000)+1):
    for j in range(int(image.shape[1]/1000)+1):
        im = image[(i*1000):(i*1000 + 1000), (j*1000):(j*1000+1000), :]

        masked = cv.dilate(
            cv.erode(cv.threshold(np.uint8(np.min(im, axis=2)), 200, 255,
cv.THRESH_BINARY)[1], np.ones((3, 3))),
            np.ones((7, 7))
        ) > 100
        im = np.mean(im, axis=2)
```

```

cv.imshow("im", im/255)

im = np.float32(im)

def deriv_blur(values, sigma):
    i_sigma_x = cv.GaussianBlur(src=values*np.mgrid[:values.shape[0],
:values.shape[1]][0, :, :],
                                ksize=(-1, -1),
                                sigmaY=sigma,
                                sigmaX=sigma)/np.square(sigma)
    x_sigma_x = cv.GaussianBlur(src=values,
                                ksize=(-1, -1),
                                sigmaY=sigma,
                                sigmaX=sigma)*np.mgrid[:values.shape[0], :values.shape[1]][0, :,
:]/np.square(sigma)

    i_sigma_y = cv.GaussianBlur(src=values*np.mgrid[:values.shape[0],
:values.shape[1]][1, :, :],
                                ksize=(-1, -1),
                                sigmaY=sigma,
                                sigmaX=sigma)/np.square(sigma)
    y_sigma_y = cv.GaussianBlur(src=values,
                                ksize=(-1, -1),
                                sigmaY=sigma,
                                sigmaX=sigma)*np.mgrid[:values.shape[0], :values.shape[1]][1, :,
:]/np.square(sigma)
    return x_sigma_x - i_sigma_x, y_sigma_y - i_sigma_y

```

```

def derivative(values, sigma):
    x2, y2 = deriv_blur(np.square(values), sigma)
    x, y = deriv_blur(values, sigma)
    dx = x2 - 2*x*cv.GaussianBlur(values,
                                    ksize=(-1, -1),
                                    sigmaY=sigma,
                                    sigmaX=sigma)
    dy = y2 - 2*y*cv.GaussianBlur(values,
                                    ksize=(-1, -1),
                                    sigmaY=sigma,
                                    sigmaX=sigma)
    return dx, dy

```

```

def derivative_covariance(f, g, sigma):
    x2, y2 = deriv_blur(f*g, sigma)
    xf, yf = deriv_blur(f, sigma)
    xg, yg = deriv_blur(g, sigma)
    dx = x2 - \
        xf*cv.GaussianBlur(g, ksize=(-1, -1), sigmaY=sigma, sigmaX=sigma) - \
        xg*cv.GaussianBlur(f, ksize=(-1, -1), sigmaY=sigma, sigmaX=sigma)

    dy = y2 - \
        yf*cv.GaussianBlur(g, ksize=(-1, -1), sigmaY=sigma, sigmaX=sigma) - \
        yg*cv.GaussianBlur(f, ksize=(-1, -1), sigmaY=sigma, sigmaX=sigma)

    return dx, dy

```



```

def norm_deriv(x, y, sigma = 5):
    x2_w_x, x2_w_y = derivative(x, sigma)
    y2_w_x, y2_w_y = derivative(y, sigma)
    xy_w_x, xy_w_y = derivative_covariance(x, y, sigma)

    x2 = cv.GaussianBlur(np.square(x), ksize=(-1, -1), sigmaY=sigma, sigmaX=sigma) - \
        np.square(cv.GaussianBlur(x, ksize=(-1, -1), sigmaY=sigma, sigmaX=sigma))
    y2 = cv.GaussianBlur(np.square(y), ksize=(-1, -1), sigmaY=sigma, sigmaX=sigma) - \
        np.square(cv.GaussianBlur(y, ksize=(-1, -1), sigmaY=sigma, sigmaX=sigma))

    xy = cv.GaussianBlur(x*y, ksize=(-1, -1), sigmaY=sigma, sigmaX=sigma) - \
        cv.GaussianBlur(x, ksize=(-1, -1), sigmaY=sigma, sigmaX=sigma) * \
        cv.GaussianBlur(y, ksize=(-1, -1), sigmaY=sigma, sigmaX=sigma)
    dif_x = .5*(y2_w_x - x2_w_x) + .25*(2*(x2 - y2)*(x2_w_x - y2_w_x) + 8*xy_w_x*xy)
    dif_y = .5*(y2_w_y - x2_w_y) + .25*(2*(x2 - y2)*(x2_w_y - y2_w_y) + 8*xy_w_y*xy)

    total = np.sqrt(np.square(xy_w_x) + np.square(xy_w_y) +
np.square(dif_x)+np.square(dif_y))
    return total

def eigen_value(im, size=37):
    x = cv.Sobel(im, -1, 1, 0)
    y = cv.Sobel(im, -1, 0, 1)
    x[mask] = 0
    y[mask] = 0

```

```

x2 = cv.GaussianBlur(np.square(x), ksize=(size, size), sigmaY=-1, sigmaX=-1) - \
    np.square(cv.GaussianBlur(x, ksize=(size, size), sigmaY=-1, sigmaX=-1))
y2 = cv.GaussianBlur(np.square(y), ksize=(size, size), sigmaY=-1, sigmaX=-1) - \
    np.square(cv.GaussianBlur(y, ksize=(size, size), sigmaY=-1, sigmaX=-1))
xy = cv.GaussianBlur(x*y, ksize=(size, size), sigmaY=-1, sigmaX=-1) - \
    cv.GaussianBlur(y, ksize=(size, size), sigmaY=-1, sigmaX=-1)*cv.GaussianBlur(x,
ksize=(size, size), sigmaY=-1, sigmaX=-1)

```

```

l1 = x2 + y2 + np.sqrt(np.square(x2 - y2) + 4*np.square(xy))

```

```

l1 /= 2

```

```

angles = np.arctan2(l1 - x2, xy)

```

```

return angles

```

```

e = eigen_value(im)

```

```

e[e < 0] += np.pi

```

```

color[(i*1000):(i*1000 + 1000), (j*1000):(j*1000+1000),0] = 360*(e -
np.min(e))/(np.max(e) - np.min(e))

```

```

color[(i*1000):(i*1000 + 1000), (j*1000):(j*1000+1000),1] = np.float32(1)

```

```

color[(i*1000):(i*1000 + 1000), (j*1000):(j*1000+1000),2] = np.float32(1)

```

```

print(i)

```

```

hsv = cv.cvtColor(color, cv.COLOR_HSV2BGR)

```

```

hsv[:, :, 0][mask] = 0

```

```

hsv[:, :, 1][mask] = 0

```

```
hsv[:, :, 2][mask] = 0  
cv.imwrite("demo.bmp", 255*hsv)
```

## Appendix 5: G-R Supporting Images

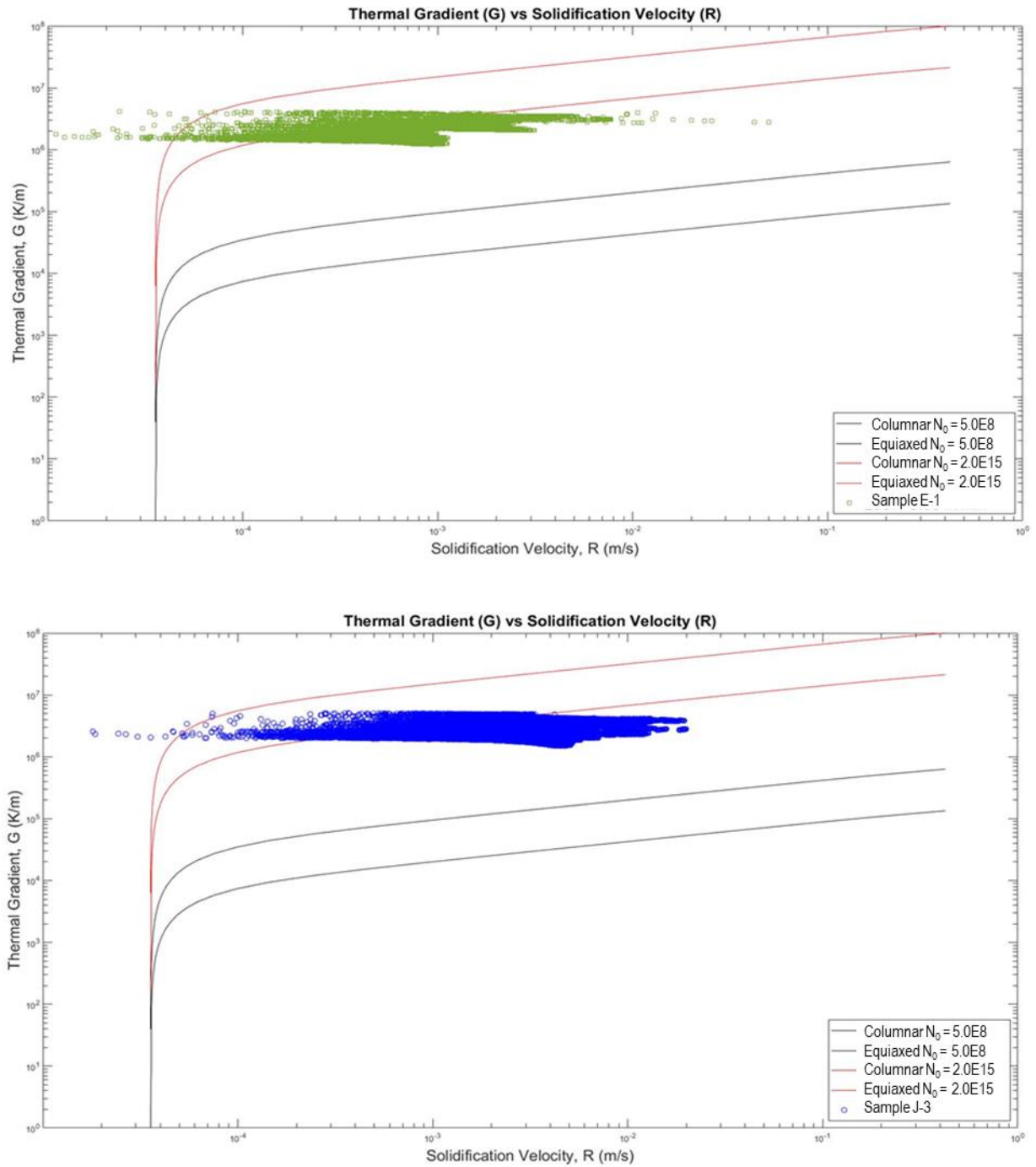


Figure 90: Columnar-to-equiaxed transition curve for sample E-1 (top) and J-5 (bottom) with Gaumann calibrated  $N_0$  (red) and electron beam melting  $N_0$  (black) curves

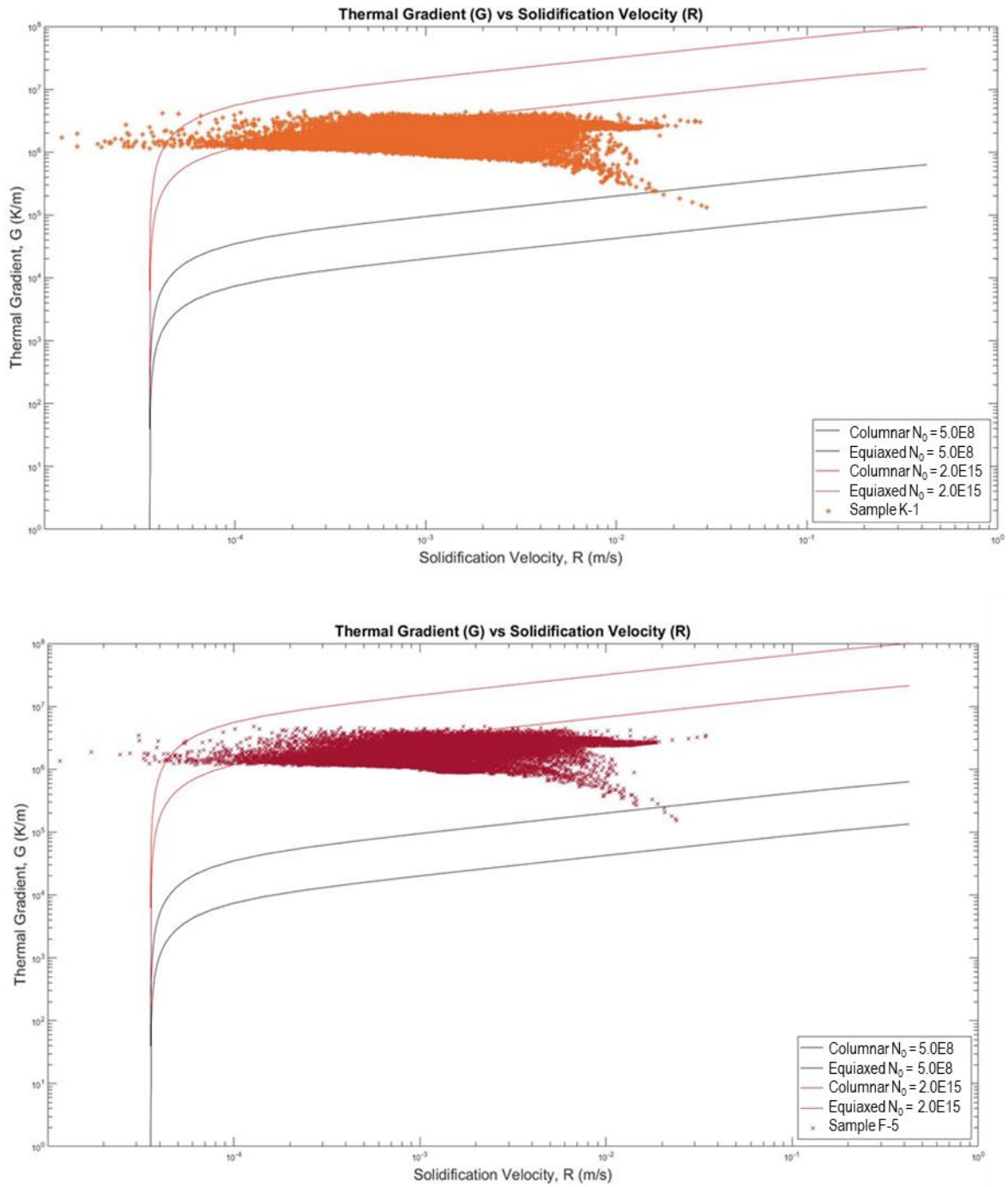


Figure 91: Columnar-to-equiaxed transition curve for sample K-1 (top) and sample F-5 (bottom) with Gaumann calibrated  $N_0$  (red) and electron beam melting  $N_0$  (black) curves

## Vita

Kevin M. Smith was born in Clearwater, FL to parents Michael and Maryann Smith in 1995. He has a sister Annika and a brother Ryan. Kevin graduated high school in 2013 from the East Lake High School engineering magnet and was a participant on the swim and dive and tennis teams. After graduation, he spent four years at the Georgia Institute of Technology as a member of the varsity men's swimming and diving team while completing his Bachelor of Science degree in Materials Science and Engineering. Upon completion of his Bachelors, Kevin began work on his Master of Science in Materials Science & Engineering at the University of Tennessee – Knoxville in 2017 under Dr. Suresh Babu with a focus in additive manufacturing. Kevin's research was conducted at the University of Tennessee – Knoxville as well as Oak Ridge National Laboratory's Manufacturing Demonstration Facility (MDF). Upon completion of his Masters, Kevin moved to West Palm Beach, FL to continue his career working for Aerojet Rocketdyne.