Laser-Matter Interactions in Directed Energy Deposition

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Abstract

Additive manufacturing is a promising process that has the capability to build components with complex geometries for structural and biomedical applications. Due to the rapid and localized directional solidification of molten metallic alloys, unique phase transformations occur at the melt pool that can provide for components with greater strength and other improved mechanical properties. However, knowledge gaps remain at how additive manufacturing processing influences the resulting properties. Knowledge gaps include the mechanisms behind powder flow behavior entering the melt pool, how powder flow influences characteristics of the melt pool, how porosity forms during the process, and the anisotropy of microstructures, porosity and mechanical properties in an additive manufactured part.

This work utilized both in-situ and ex-situ methods to characterize the powder-blown additive manufacturing process, or directed energy deposition (DED), of 316L stainless steel (SS), Ti-6Al-4V, and Inconel 718. In-situ high-speed X-ray imaging allowed real-time observation of the liquid-solid interface of a fluctuating melt pool and porosity formation during the process. In-situ infrared imaging also enabled the capture of temperature variation during the process. Ex-situ methods included characterization of the resulting parts with optical microscopy, X-ray diffraction, Vickers microhardness indentation, and tensile testing, etc. Collaboration with computational efforts allowed for calibration, validation of thermal models that enable prediction of properties of an additive manufactured part.
The process-structure-property relationships of additive manufactured parts were found for three materials: 316L SS, Inconel 718, and Ti-6Al-4V. Observations of the laser-matter interactions during the directed energy deposition process showed how powder flow influenced the melt pool at various powder flow rates and laser power values. The thermal history of additive manufactured parts was shown to be influenced by laser power, scan speed, and tool path. Stainless steel components were shown to exhibit varying microstructures and porosity structures with varying laser powers in addition to anisotropy of these structures within the same component. Thin walls of DED-processed Inconel 718 with differing laser scan strategies were shown to varying cooling rates, microstructures, and residual strains. The process-structure-property relationships of cubic Ti-6Al-4V components were compared to the relationships in micro-scaled Ti-6Al-4V single clads.

Overall, additive manufacturing is dependent on material properties and how they interact with a laser. This work proposed a few frameworks and insights into how process parameters influence thermal histories, microstructure, porosity, and properties of a part. Emerging directions in additive manufacturing of crystalline material based on these frameworks include process control and new materials development.
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## Nomenclature

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>(a)</td>
<td>Absorptance, absorptivity</td>
</tr>
<tr>
<td>(a)</td>
<td>Ellipsoid axis in the hatch direction (mm)</td>
</tr>
<tr>
<td>(A)</td>
<td>Cross-sectional area (mm(^2))</td>
</tr>
<tr>
<td>(A)</td>
<td>Material constant that describes frequency with which liquid atoms attach to solid nucleus</td>
</tr>
<tr>
<td>(A(\tau))</td>
<td>Autocorrelation function</td>
</tr>
<tr>
<td>(b)</td>
<td>Ellipsoid axis in the scan direction (mm)</td>
</tr>
<tr>
<td>(c)</td>
<td>Ellipsoid axis in the build direction (mm)</td>
</tr>
<tr>
<td>(C_P)</td>
<td>Specific heat capacity (J·kg(^{-1})·K(^{-1}))</td>
</tr>
<tr>
<td>(C_{P_L})</td>
<td>Liquid specific heat capacity (J·kg(^{-1})·K(^{-1}))</td>
</tr>
<tr>
<td>(C_{P_S})</td>
<td>Solid specific heat capacity (J·kg(^{-1})·K(^{-1}))</td>
</tr>
<tr>
<td>(d)</td>
<td>Depth of clad into the substrate (mm)</td>
</tr>
<tr>
<td>(D)</td>
<td>Dilution ratio</td>
</tr>
<tr>
<td>(e)</td>
<td>Principal load axis</td>
</tr>
<tr>
<td>(E_D)</td>
<td>Energy density (J·mm(^{-2}))</td>
</tr>
<tr>
<td>(f)</td>
<td>Focal length of incident light (mm)</td>
</tr>
<tr>
<td>(f)</td>
<td>Volume fraction of a new phase</td>
</tr>
<tr>
<td>(f_{\alpha'})</td>
<td>Volume fraction of the (\alpha') phase in Ti-6Al-4V</td>
</tr>
<tr>
<td>(G)</td>
<td>Thermal gradient (K·mm(^{-1}))</td>
</tr>
<tr>
<td>(h)</td>
<td>Hatch spacing (mm)</td>
</tr>
<tr>
<td>(h)</td>
<td>Height of the clad (mm)</td>
</tr>
<tr>
<td>(h_c)</td>
<td>Convection heat transfer coefficient (J·mm(^2)·K(^{-1}))</td>
</tr>
<tr>
<td>(H)</td>
<td>Heat input per length (J·mm(^{-1}))</td>
</tr>
<tr>
<td>(\Delta H_L)</td>
<td>Change in enthalpy (J·g(^{-1}))</td>
</tr>
</tbody>
</table>
\( i \) \((-1)^{1/2}\)

\( I \) Light intensity in medium (W·mm\(^{-2}\))

\( J \) J-factor, a fractal parameter for composites

\( k \) Extinction coefficient

\( k_{\text{part}} \) Partition coefficient in the CtFD model

\( k_{\text{wave}} \) Wave number

\( l \) Length of the melt pool (mm)

\( L \) Latent heat of fusion (J·g\(^{-1}\)·K\(^{-1}\))

\( \dot{m} \) Powder mass flow rate (g·s\(^{-1}\))

\( M'_c \) Mechanical property of the overall composite

\( M'_b \) Mechanical property of the bulk, single phase material

\( M'_w \) Mechanical property of the second phase (air in porosity analysis)

\( n \) Index of refraction

\( \tilde{n} \) Complex index of refraction

\( Q \) Laser power (W)

\( Q \) Activation energy for transformation (J)

\( r \) Reflectance, reflectivity

\( r \) Transformation rate (s\(^{-1}\))

\( r_b \) Laser beam radius (mm)

\( r_p \) Powder flow radius (mm)

\( R \) Universal gas constant (J·K\(^{-1}\)·mol\(^{-1}\))

\( R \) Growth rate, interfacial velocity (mm·s\(^{-1}\))

\( R_{\text{RMS}} \) RMS roughness (mm)

\( s \) Width, length, or height of a component (mm)

\( t \) Transmittance

\( t \) Layer thickness (mm)
\( t \) Time (s)
\( t_p \) Laser interaction time (s)
\( T \) Temperature (K)
\( T_i, T_{liquidus} \) Liquidus temperature (K)
\( T_{M_s} \) Starting martensite temperature (K)
\( T_s, T_{solidus} \) Solidus temperature (K)
\( T_\infty \) Ambient temperature (K)
\( \dot{T} \) Cooling rate (K·s\(^{-1}\))
\( \dot{T} \) Normalized cooling rate (K·s\(^{-1}\))
\( \Delta T_0 \) Equilibrium freezing range; difference between liquidus and solidus temperatures
\( v \) Laser scan speed (mm·s\(^{-1}\))
\( \dot{v} \) Volumetric powder flow (mm\(^3\)·s\(^{-1}\))
\( V \) Volume (mm\(^3\))
\( V_s \) Volume of the single, bulk phase (mm\(^3\))
\( V_w \) Volume of the second, porous phase in a composite (mm\(^3\))
\( x \) Hatch direction
\( y \) Scan direction
\( z \) Build direction
\( Z \) Vertical distance, depth (mm)
\( \alpha \) Absorption coefficient
\( \alpha \) Thermal diffusivity (mm\(^2\)·K\(^{-1}\))
\( \alpha \) Ti-6Al-4V matrix phase
\( \alpha' \) Ti-6Al-4V martensitic phase
\( \alpha'' \) Ti-6Al-4V phase
\( \beta \) Volumetric thermal expansion coefficient (K\(^{-1}\))
\( \beta \) Ti-6Al-4V phase
\( \gamma \) Surface tension (N·m\(^{-1}\))

\( \gamma \) Inconel 718, 316L SS phase

\( \gamma' \) Inconel 718

\( \gamma'' \) Inconel 718

\( \frac{d\gamma}{dT} \) Marangoni coefficient (N·m\(^{-1}\)·K\(^{-1}\))

\( \delta \) Inconel 718 phase

\( \Gamma \) Gibbs Thomson coefficient

\( \varepsilon_0 \) Permittivity (F·m\(^{-1}\))

\( \varepsilon \) Emissivity

\( \eta \) Absorption coefficient

\( \eta_m \) Powder catchment efficiency

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

\( \kappa_l \) Liquid thermal conductivity (W·m\(^{-1}\)·K\(^{-1}\))

\( \kappa_s \) Solid thermal conductivity (W·m\(^{-1}\)·K\(^{-1}\))

\( \lambda_0 \) Laser wavelength (mm)

\( \lambda_{P\text{DAS}} \) Primary dendrite arm spacing (mm)

\( \lambda_{S\text{DAS}} \) Secondary dendrite arm spacing (mm)

\( \mu \) Magnetic susceptibility (H·m\(^{-1}\))

\( \nu \) Poisson’s ratio

\( \rho \) Density (kg·m\(^{-3}\))

\( \rho_l \) Liquid density (kg·m\(^{-3}\))

\( \rho_{\text{powder}} \) Powder density (kg·m\(^{-3}\))

\( \rho_s \) Solid density (kg·m\(^{-3}\))

\( \sigma \) Conductivity (S·m\(^{-1}\))

\( \sigma_s \) Stefan-Boltzmann constant (W·mm\(^{-2}\)·K\(^{-4}\))
\( \tau \)  Distance between two points (mm)

\( \tau \)  Characteristic time scale for solidification cooling (s)

\( \omega \)  Angular frequency (rad·s\(^{-1}\))
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Chapter 1: Introduction

1.1 Motivation

The motivation for this work is the need to understand and characterize additive manufacturing (AM) processing, materials, and properties. Drivers to advance AM research include better structural materials, biomedical applications, and energy efficiency.

Research in AM aims to build a growing variety of materials for emerging functionalities. With growing demand for novel crystalline materials, advanced manufacturing processes are required to process these functional materials with flexibility and scalability. Examples of motivators for advanced materials research in AM include:

- Initiatives for rapid qualification of new alloys for flight-critical components (Pollock 2016) present opportunities and challenges for additive manufacturing (AM) (Seifi, Salem et al. 2016).

- Materials such as permanent magnets, where AM technologies can alleviate manufacturing obstacles (Li, Tirado et al. 2016).

- AM processing can overcome the strength-ductility trade-off by building structural components with increased strength and ductility compared to those built with conventional processing (Wang, Voisin et al. 2018).
AM of crystalline materials enhances human welfare by providing customized biomedical products. The majority of people above 40 years old struggle with various extents of joint disease (Long and Rack 1998). Examples of motivators for biomedical applications in AM include:

- High-strength titanium alloys built with AM processing can provide light-weight, load-bearing joint implants with porous structures and cell ingrowth (Hao, Li et al. 2016).
- Complex architectures and topological optimization of biodegradable metals, including titanium alloys that can accelerate the osseointegration process in the human body (Wang, Xu et al. 2016).
- Custom-fit skulls (Winder, Cooke et al. 1999) and implants (Liu, Leu et al. 2006) based on a computer-aided design (CAD) file converted from 3D imaging of the patient’s body.
- A supply chain study of AM feasibility for implants showed that installing a facility with several AM machines near hospitals could substantially decrease the time (from many months to days) and cost required to build and transport the implants to patients (Emelogu, Marufuzzaman et al. 2016).

AM technology is posed to contribute to the reduction of energy consumption and decrease the environmental impact of manufacturing. The U.S. manufacturing sector accounts for about 25% of the nation’s energy consumption (Office 2018). Examples of motivators advancing AM for greater energy efficiency and savings in greenhouse gas emissions include:

- AM can reduce waste as less material is required to manufacture a component and does not require cutting fluids (Luo, Ji et al. 1999).
• A study estimated the energy and greenhouse gas savings to be up to 2.8 GJ and 217 million tons of CO₂e for AM-processed aircraft parts of a fleet (Huang, Riddle et al. 2016). This shows how AM processing can improve energy efficiency and decrease negative impacts on the environment.

• The impact of AM on the manufacturing supply chain is that it reduces warehousing, transportation, and packaging requirements by enabling on-demand, customizable manufacturing (Huang, Liu et al. 2013).

The growing needs for new materials, biomedical applications, and energy efficiency are currently behind the goals of the Department of Energy’s Advanced Manufacturing Office (AMO) (Office 2018) and the Materials Genome Initiative (Hernandez 2017), with the overall goals of enhancing human welfare and economic growth. In its pursuit to reduce energy consumption of the manufacturing sector by 50% by 2025, taxpayer investment of $12B in its first year (2016-2017) has resulted in a net benefit of $230B to the U.S. economy (Energy 2018). The AMO reflects the needs in the United States’ manufacturing sectors, including improving energy efficiency and promoting economic growth by advancing manufacturing research (Office 2018). The Materials Genome Initiative aims to discover and implement new materials (Hernandez 2017) for improved AM processing, structures, and overall economic growth.

Advances in manufacturing research rely on understanding and harnessing the underlying fundamental physical phenomena of manufacturing processes. In turn, fully capturing the influence of processing on a material leads to a cascading effect of more advanced processes and
novel materials that are suitable for those processes. Advanced AM is required to process these materials with flexibility and scalability (Frazier 2014).

As a light source that emits localized heat, lasers are common devices in modifying, building or machining materials. However, there are knowledge gaps with how lasers interact with materials in AM. The knowledge gaps in laser-matter interactions that pertain to this work include:

- Powder flow behavior during a powder-blown AM process and how powder particles interact with a moving laser beam.
- Convection and fluid flow within a melt pool induced by laser melting of a crystalline material.
- Cooling behavior at the melt pool and how it depends on varying processing conditions during laser-matter interactions.
- Porosity formation, evolution, characteristics, and distribution in AM materials.
- Process-cooling-structure-properties relationships in AM materials that can provide means for process control.

Based on the listed knowledge gaps, laser-based AM faces the following detailed challenges, which the state-of-the-art work in Section 1.2 and the thesis work will address:

1. With small variations in process parameters, large deviations in the final manufactured part arise. Process control for a desired part requires an understanding of how changing process parameters influence the final part.
2. *In-situ*, high-resolution, process monitoring of the high-speed and small-scale physical phenomena (temperature, phase changes, defect formation, chemistry changes, etc.) is difficult to achieve.

3. Many conventional materials are not compatible with laser processing due to transmission, reflectivity, or inefficient absorption of the laser into the material. This leads to defects in laser-processed materials such as porosity, warping, a decrease in structural integrity, and residual stresses. Advanced *ex-situ* characterization, in addition *in-situ* process monitoring, is required to capture properties at multiple scales to determine material compatibility.

The overarching objective of this work is to investigate a range of laser-matter interactions in the context of advanced manufacturing processing.

1.2 Overview of additive manufacturing

1.2.1 Opportunities and challenges in additive manufacturing

Most additive manufacturing (AM) of crystalline materials requires a laser to melt and fuse material together. AM has been growing in ubiquity for customized and complex parts in a wide variety of industries, including biomedical and aerospace (Frazier 2014). The nature of layer-by-layer processing allows for components with geometries that are not achievable with conventional manufacturing. For example, processing a part with an interior that is hollow or with a network of cooling channels would not be possible with conventional casting and milling of a single component, but would require the joining of multiple components.
With AM, researchers and industries can build a part with complex geometries, multiple features with a single process, and achieve superior mechanical properties. GE Aviation modified its manufacturing of a jet engine fuel nozzle from the welding and brazing of more than twenty separate parts to building the entire nozzle in a single AM process with 25% less mass (Kellner 2018). A survey of several types of additive manufacturing processing of Ti-6Al-4V, including laser-based powder-blown processing, laser-based powder-bed, and electron beam melting, showed that tensile properties were greater than those processed with conventional manufacturing, including cast, forged and wrought parts (Lewandowski and Seifi 2016).

A major challenge of AM is the anisotropy of the properties in as-built components due directional, rapid cooling during laser scanning (Diegel, Singamneni et al. 2011). Solidification rates during laser-based additive processes are as high as about $10^6$ K/s (Frazier 2014), an unprecedented rate, leading to unique phase transformations that result in unconventional microstructures such as lamellar dendrites or irregular porosity. Before a prediction of the global properties of an AM part can be accomplished, an understanding of the localized properties is required to predict and control areas that could potentially lead to defects. Predicting the “weakest links” of an AM component is essential to ensure that quality standards are met (Peter and Yamamoto 2013). The scope of material compatibility with AM is also limited. Key knowledge gaps that remain in AM research include the understanding of laser-matter interaction across spatial and temporal scales and how material compatibility with AM can be improved. Filling these knowledge gaps can help pave the way to design and build new, functional materials and parts.
1.2.2 Types of AM processes

According to the ASTM F42 standard that defines terminology for AM technologies (Technologies and Terminology 2012), additive manufacturing is defined as any process that joins materials “to make objects from 3D model data, usually layer upon layer, as opposed to subtractive manufacturing methodologies.” There are seven categories of processes that fall into two groups. Five categories pertain to mostly polymer processing and include material extrusion, vat photopolymerization, sheet lamination, binder jetting, and material jetting while two processes, powder bed fusion and directed energy deposition (DED), build metallic and ceramic components.

**Polymer processing:** Vat photopolymerization, or stereolithography, consists of a light source that cures a vat of liquid photopolymer (Gibson, Rosen et al. 2015). A build platform moves downward to allow the light, which scans using a galvo scanning mirror, to cure layer by layer. Material jetting requires inkjet print heads that emit materials to be cured by an attached UV light source (He, Wildman et al. 2016). Materials from jetting can include a gradient of “digital” materials that combine photopolymers with specific concentrations and microstructures from flexible rubber, high strength ABS, transparent materials, etc. Binder jetting uses a powder bed of plaster-like particles that sit on a build platform and are leveled with a roller (Chen and Zhao 2016). An inkjet print head ejects droplets from a liquid adhesive supply that binds the powders together. The build platform moves down to allow for a layer by layer build. Post-processing requires an immersion into an acrylamide glue. Sheet lamination uses an ultrasonic welding head that laminates sheets of material tape together in a layer by layer fashion, while an end mill runs at the same time to create features (Mueller 2012). Material extrusion by means of fused deposition modeling extrudes a
melted wire filament of material with an extrusion nozzle (Rocha, Perez et al. 2014). Materials range from thermoplastic, nylon, and ABS.

**Metal and ceramics processing:** Powder bed processing of metallic parts is also known as selective laser sintering (SLS), selecting laser melting (SLM), direct metal laser sintering (DMLS), direct metal laser melting (DMLM), and electron beam melting (EBM), where either a laser or electron beam serves as the heating element. A bed of metallic powder particles is levelled with a spreader onto a build platform, which lies above a piston that moves down with each layer. The heat source scans across the powder bed with a galvo mirror scanning system and either sinters, or fully melts, the powder particles together (Khairallah, Anderson et al. 2016). Selective laser sintering (SLS) was first licensed for a patent in 1987 by Dr. Carl Deckard and Professor Joe Beaman at the University of Texas Austin (Beaman and Deckard 1990). The first metal AM machine became commercially used in 1989 by Nova Automation, or DTM, while the first metal AM machine became commercially available in 1995 (Lipson and Kurman 2013).

1.2.3 Overview of the DED process

The DED process is also known by many alternative names, including Directed Light Fabrication (DLF), Laser Engineered Net Shaping (LENS®), Direct Laser Deposition (DLD), Direct Metal Deposition (DMD), and powder-blown additive manufacturing. In 1994, researchers at Los Alamos National Laboratory developed DLF by introducing a nozzle-based powder flow into laser welding (Lewis, Nemec et al. 1994). By incorporating CAD and CNC capabilities, DLF could perform rapid fabrication of steel components with complex geometries. In the mid-to-late 1990s,
researchers at Sandia National Laboratories developed a DED process known as LENS®, which incorporated multiple nozzles, or a coaxial nozzle with multiple streams, for more effective powder delivery (Atwood, Ensz et al. 1998). Since then, the LENS process has been commercialized by OPTOMEC (Hofmeister, Wert et al. 1999). In the past decade or so, companies have incorporated hybrid metal AM-subtractive machines along with the dozens of metal AM companies that have arisen (Karunakaran, Suryakumar et al. 2010).

The development of solid-state continuous wave lasers with powers of more than 100 W and shorter wavelengths down to 450 nm have coincided with AM development in the last 15 years. AM technologies are moving away from CO₂ lasers at the 10.6 µm wavelength to solid-state lasers at infrared wavelengths between 1050 and 1080 nm or shorter (Majumdar and Manna 2003). These high-powered solid-state lasers provide better laser beam quality, efficiency and melt materials more effectively (Penzkofer 1988).

The powder-blown AM process, or DED, is characterized by the interaction of a heat source with powder particles that flow from a nozzle into a melt pool (Gibson, Rosen et al.). DED has been growing in pervasiveness, mostly in building large-size components, performing component repair, and coating. In comparison to powder bed systems, DED undergoes different heat transfer mechanisms with more rapid directional solidification as building an additional layer does not depend on the time it would take to spread a new layer of un-melted particles as in powder bed processes (Frazier 2014). In addition, DED has greater flexibility as the deposited particles can be a mixture of materials that flow from multiple nozzles with the capability to build functional
gradient material (FGM) structures (Griffith, Keicher et al. 1996), (Parthasarathy, Starly et al. 2011), (Popovich, Borisov et al. 2017).

In DED, powder is pneumatically conveyed via an inert gas into a molten pool, which is generated by a laser beam on an existing substrate, as seen in Fig. 1.1. As the laser moves throughout the part at some CNC feed rate, the molten pool solidifies to form a solid material track. A fully dense 3D geometry can be created by overlapping these tracks side-by-side and depositing consecutive layers on top of the previous layers. The dimensional consistency of the completed structure relies on the uniformity and repeatability of the width and height of each individual clad track. Dimensions depend on process parameters such as laser power, laser scan speed, powder flow rate, and substrate temperature (Frazier 2014). Laser beam power determines the heat input into the melt pool. Various tool paths, such as rastering from 0 to 180 degrees or from 0 to 90 degrees, will generate parts with differing thermal histories.

![Figure 1.1. Schematic of the DED process (Wolff et al. 2017).](image)

Parameters such as layer thickness and hatch spacing also determine the amount of fusion among powder particles, amount of re-melting of previous layers, and the overall thermal history of the part. As studies like (Gong, Rafi et al. 2014) show, when the layer thickness is too low, the
underlying layer does not fuse adequately with the melt pool boundary, leading to un-melted powder particles or lack of fusion porosity. In the same vein, too low of a hatch spacing can result in lack of wetting and thermal conduction of the melt pool boundary. This insulates the melt pool boundary by the surrounding powder bed or flowing powder, where the slower cooling results in surface tension effects that initiate porosity formation.

Powder delivery in laser-based processes requires controlled mass flow into a melt pool area that is dependent on the laser beam diameter. Most powder delivery systems provide a stream of inert gas to carry powders onto the substrate with a Gaussian mass density profile, with the most effective builds occurring at the intersection of the foci of both the laser and powder. Industrial DED systems use a focused laser beam diameter of 500 µm to 5 mm (Frazier 2014) and design their delivered powder flow so that mass flows into a focused area about the size of the laser beam diameter to ensure powders fuse in the melt pool.

Table 1.1 lists several DED studies that processed the materials of interest in this work, including 316L SS, Inconel 718, and Ti-6Al-4V. Processing conditions varied with material and laser specifications. In most studies, the reasons behind process parameter selection are not specified, as commercial systems provide pre-determined process parameter sets. The laser-matter interactions in DED are sensitive to each parameter in a unique set of processing conditions.
<table>
<thead>
<tr>
<th>Processed material; part geometry</th>
<th>Laser power (W)</th>
<th>Laser beam diameter (mm)</th>
<th>Wavelength (µm)</th>
<th>Scan speed (mm/s)</th>
<th>Layer thickness (mm)</th>
<th>Hatch spacing (mm)</th>
<th>Powder size (µm)</th>
<th>Powder flow rate</th>
</tr>
</thead>
<tbody>
<tr>
<td>316L SS; cuboids (Ziętala, Durejko et al. 2016)</td>
<td>400</td>
<td>Not reported</td>
<td>1.070</td>
<td>15</td>
<td>0.30</td>
<td>0.45</td>
<td>44-50</td>
<td>5 rpm</td>
</tr>
<tr>
<td>316L SS; cylinders (Gray Iii, Livescu et al. 2017)</td>
<td>380</td>
<td>Not reported</td>
<td>1.070</td>
<td>11.2</td>
<td>0.30</td>
<td>0.46</td>
<td>53-180</td>
<td>105 mg/s</td>
</tr>
<tr>
<td>316L SS; cylinder, disk, thin hexagonal wall (Yang, Yee et al. 2017)</td>
<td>360</td>
<td>1.8</td>
<td>1.064</td>
<td>16.3</td>
<td>0.25</td>
<td>0.39</td>
<td>44-149</td>
<td>167 mg/s</td>
</tr>
<tr>
<td>Inconel 718; thin walls (Zhao, Chen et al. 2008)</td>
<td>2350</td>
<td>3</td>
<td>10.60</td>
<td>8</td>
<td>Not reported</td>
<td>N/A</td>
<td>44-150</td>
<td>100 mg/s</td>
</tr>
<tr>
<td>Inconel 718; single clads and thin walls (Li, Xiao et al. 2017)</td>
<td>600</td>
<td>1</td>
<td>Not reported</td>
<td>6</td>
<td>0.5</td>
<td>0.5</td>
<td>Not reported</td>
<td>100 mg/s</td>
</tr>
<tr>
<td>Inconel 718; cuboids (Stevens, Toman et al. 2017)</td>
<td>250</td>
<td>0.24</td>
<td>1.064</td>
<td>2-5</td>
<td>0.254</td>
<td>0.508</td>
<td>44-150</td>
<td>5 rpm</td>
</tr>
<tr>
<td>Ti-6Al-4V; thin walls (Wu, Liang et al. 2004)</td>
<td>222-516</td>
<td>1-2</td>
<td>10.60</td>
<td>3.3-16.7</td>
<td>0.3</td>
<td>N/A</td>
<td>100-250</td>
<td>67-333 mg/s</td>
</tr>
<tr>
<td>Ti-6Al-4V; cruciform (Carroll, Palmer et al. 2015)</td>
<td>2,000</td>
<td>4</td>
<td>1.070-1.080</td>
<td>10.6</td>
<td>0.89</td>
<td>2.29</td>
<td>89 (avg)</td>
<td>133 mg/s</td>
</tr>
<tr>
<td>Ti-6Al-4V; cuboids (Wolff, Lin et al. 2017)</td>
<td>710-900</td>
<td>1.83</td>
<td>1.070</td>
<td>10</td>
<td>1.25</td>
<td>0.95</td>
<td>45-150</td>
<td>120 mg/s</td>
</tr>
<tr>
<td>Ti-6Al-4V; thin wall (Lia, Park et al. 2018)</td>
<td>2,000</td>
<td>3</td>
<td>Not reported</td>
<td>10.6</td>
<td>Not reported</td>
<td>N/A</td>
<td>50-150</td>
<td>225 mg/s</td>
</tr>
</tbody>
</table>

### 1.3 Research objectives and thesis overview

Considering the laser-matter interactions in additive manufacturing, technical challenges persist, including the understanding of the multi-scale and multi-material interactions. *In-situ* monitoring can help uncover these interactions. In addition, additive manufacturing requires a degree of predictability in the structure and properties of the final part. Access to *in-situ* monitoring with high temporal and spatial resolution to capture the physics in laser-matter interactions is vital but also a challenge.
The research objectives and outline of this work rely on a modified chain-link model (Olson 1997), which provides an overarching view of the capability of advanced manufacturing processing. The following objectives investigate the influences of laser-matter interactions on thermal history, microstructure, defects and mechanical behavior of a material, as seen in the overarching thesis outline schematic in Fig. 1.2:

- How does material compatibility influence the thermal history, structure, defects and properties in the DED process?
- What physical phenomena occur at the melt pool during the DED process that will allow us to predict the resulting build by improving assumptions in thermo-mechanical models and optimizing process parameters?
- How does thermal history, especially rapid solidification in DED, influence the resulting microstructure, porosity and mechanical behavior in a component?

The subsequent chapters will address these research questions. Chapter 2 provides a background on material compatibility in laser processing and the material selection used throughout this work. *In-situ* monitoring of a micro-scale DED process to capture the laser-matter interaction during powder flow is shown in Chapter 3. The influence of industrial-scale powder flow with *ex-situ* characterizations of laser-matter interactions in DED is discussed in Chapter 4. The influence of processing on thermal histories of various DED-processed materials is highlighted in Chapter 5. The microstructures in as-built DED components because of the phase transformations during
rapid solidification are explained in Chapter 6. Porosity formation during the DED process and the relationships between cooling and porosity characteristics are reviewed in Chapter 7. Chapter 8 considers how thermal history, microstructure and porosity influence the final properties of DED components while Chapter 9 provides an overall summary and opportunities for future work.

Figure 1.2. Outline of the thesis.

The relationships between each chapter highlight the key knowledge gaps addressed in this work as well as some of the enduring challenges, which are motivations for future work. Appendix A discusses laser-matter interaction in the form of surface texturing using a pulsed laser, and the relationships between processing conditions and final part structure. The goal of understanding the relationships between each chapter is to design better materials and processes.
Chapter 2: Material Compatibility in Laser-Matter Interactions

In laser-based processing, laser-matter interaction varies with the properties of both the laser and the workpiece material. The efficacy of laser melting or ablation of a workpiece depends on both. Considering material compatibility in laser-matter interactions is crucial to determine which process parameters, such as laser power, to use. Overall, investigating the influence of processing conditions in any laser-based process is not universal among materials. Therefore, a review on laser specifications and material properties is given in this chapter.

The types of metallurgical materials investigated in this work for its laser-matter interactions in melting for additive manufacturing are outlined in Fig. 2.1. Stainless steels, nickel super alloys, and titanium alloys are certified materials for aerospace, biomedical, and automotive applications (Pollock 2016). They are widely used in conventional melting processes, including casting and laser welding (Kalpakjian, Vijai Sekar et al. 2014). However, the interaction of a localized high-powered laser beam with flowing metallic powder particles in DED processes is not well understood (Gao, Zhang et al. 2015). Other materials, such as refractory materials like silicon, or transparent glasses, require greater laser power intensities for melting or ablation. These materials are discussed in Appendix A and are less compatible with most off-the-shelf lasers, with the exception of femto-second pulsed lasers.
The various laser types with their wavelengths, power intensities, beam profiles, gain mediums, interaction times, and applications in manufacturing are discussed in Section 2.1. Processing and thermal histories have been observed to have a wide variety of effects, including residual stresses, and mechanical behavior, depending on the material. For example, introducing dwell time between each layer during a build has been shown to decrease the residual stress in titanium alloys (Denlinger, Heigel et al. 2015) due to possible stress relaxation at high temperatures and its transformation from a body centered cubic (BCC) to HCP (hexagonal close packed) structure that could anneal the stress. On the other hand, dwell time has been shown to increase residual stress in Inconel 625, a nickel super alloy with accumulated distortion (Denlinger, Heigel et al. 2015).

The change in processing conditions, particularly laser power, scan speed, beam diameter, and powder mass flow rate, influences laser-matter interactions. The change in laser power intensity determines whether the workpiece material will form a melt pool in the conduction or keyhole
modes. Laser power intensities in conventional laser welding and the induced melt pool during the process are discussed in Sections 2.2.

The workpiece material’s chemical, surface, optical, thermal, and mechanical properties also influence the laser-matter interaction. Subsequent sections (2.3 to 2.5) discuss the importance of the workpiece material’s optical, thermal, and mechanical properties during processing. In addition, these sections will compare the properties of the materials used in both the DED and laser-induced plasma micro-machining studies in this work.

2.1 Laser power intensity and properties in processing

This section provides a literature review of the various laser specifications in manufacturing, with an emphasis of lasers in conventional processing. Knowledge of conventional processes is necessary to capture the context of the innovation in “advanced manufacturing” processes. Conventional manufacturing can be defined by a process that requires contact with a tool of greater hardness than that of the workpiece and are known to consume a substantial amount of power (Benedict 2017). This definition guarantees any laser-based process as unconventional. However, many laser-based processes such as welding are widely used, standardized, and understood in industry. With rapid technological advancement in the manufacturing sector, the boundaries as to what makes a process conventional or unconventional constantly shift. The development of new lasers with a wide variety of wavelengths, gain media and power intensities coincides with technology transfer that arises from new manufacturing processes.
Aside from lasers used for metrology, medical applications, and isotope separation, lasers used in material processing involve either forming, joining, machining or surface engineering (Majumdar and Manna 2003). These processes can require a change of phase, from solid to vapor or from solid to liquid. For surface hardening, shock-peening, bending, and annealing, laser processing does not require a phase change. Laser properties, particularly interaction time and peak power intensity, determine whether a phase change will occur.

In addition to interaction time and peak power intensity, laser gain media, operating wavelength and laser beam profile play a role in material processing. A pumping source provides energy to the gain medium, exciting electrons from low to high energy states (Silfvast 2004). Spontaneous emission occurs with decay, or during electronic transitions from a higher energy state to a lower one. The gain medium amplifies the laser beam by means of spontaneous and stimulated emission of photons. Gain media are either liquid, gas, semiconductor, or a solid-state crystal or glass. The type of gain medium determines the wavelength and the peak power in continuous wave lasers (Silfvast 2004). This is because of the various methods of energy supply to excite the electrons change the wavelength output, whether it is an electric current for gas and diode lasers or flash lamps for solid state lasers. The various means of energy supply for the gain medium determine the energy level of the amplified photon and the bandwidth of the gain medium. The energy level of the amplified photon in the gain medium is inversely proportional to the wavelength produced by the laser, as determined by Planck’s equation. The energy efficiency of the pumping system also determines the peak power output for continuous wave lasers, with high-efficiency electric current CO₂ lasers with power intensities up to 50 kW, or up to 500 kW/mm² (Majumdar and
Manna 2003). However, for mode-locked pulsed lasers, the resonant cavity surrounding the gain medium determines the periodicity of various oscillating modes, resulting in a burst, or pulse of light with super-positioned power (Grelu and Akhmediev 2012). Optical amplifiers within the laser system also determine the laser profile, whether the laser power has a Gaussian profile or a top-hat, or uniform density profile.

Laser properties and their manufacturing applications are outlined in Table 2.1 with key recent studies within the last ten years and the "first" notable study noted with a (*) for each manufacturing application. The combination of wavelength, repetition rate, interaction time, peak power intensity and beam profile determines how the laser interacts with the workpiece’s free electrons (Majumdar and Manna 2003). These interactions include lattice heating when the laser’s photon energies are not high enough to eject electrons from the workpiece material. Instead, the workpiece lattice’s dislocations and grain boundaries scatter the excited electrons in the workpiece material, resulting in overall heating. Heating due to laser irradiation in the workpiece material depends on the laser’s power and thermal diffusivity of the workpiece material. The irradiated material can reach temperatures that lead to melting and vaporization (Majumdar and Manna 2003). As indicated in Table 2.1, laser cutting and drilling result in a phase change from solid to vaporization. Laser welding, brazing, and sintering change the workpiece material from solid to liquid. Laser forming, bending, and shock peening do not result in a phase change, but result in phase transformations from solid to solid.
Table 2.1. Laser properties and their manufacturing applications.

<table>
<thead>
<tr>
<th>Manufacturing application</th>
<th>Laser gain media</th>
<th>Wavelength</th>
<th>Repetition rate range</th>
<th>Interaction time</th>
<th>Power intensity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cutting of tissue** (Hall 1971)</td>
<td>CO₂</td>
<td>10.6 µm</td>
<td>-</td>
<td>Continuous wave</td>
<td>12-15 W</td>
</tr>
<tr>
<td>Cutting of carbon fiber reinforced plastic (Herzog, Jaeschke et al. 2008)</td>
<td>Nd:YAG</td>
<td>1064 nm</td>
<td>0.01-0.1 Hz</td>
<td>0.1-20 ms</td>
<td>80-160 W</td>
</tr>
<tr>
<td>Cutting of nickel-based superalloy (Dubey and Yadava 2008)</td>
<td>Nd:YAG</td>
<td>1060 nm</td>
<td>18-28 Hz</td>
<td>0.6-1.4 ms</td>
<td>200 W</td>
</tr>
<tr>
<td>Drilling of copper* (Von Alimen 1976)</td>
<td>Nd:YAG</td>
<td>Not reported</td>
<td>Not reported</td>
<td>0.1-100 µs</td>
<td>100 MW/cm² (peak)</td>
</tr>
<tr>
<td>Drilling of Cu (Weck, Crawford et al. 2008)</td>
<td>Ti:sapphire</td>
<td>800 nm</td>
<td>1 kHz</td>
<td>150 fs – 35 ps</td>
<td>100 MW (peak)</td>
</tr>
<tr>
<td>Drilling of alumina ceramic (Hanon, Akman et al. 2012)</td>
<td>Nd:YAG</td>
<td>1064 nm</td>
<td>5-20 Hz</td>
<td>1-6 ms</td>
<td>5-9 kW (peak)</td>
</tr>
<tr>
<td>Welding of 304 SS* (Locke and Hella 1974)</td>
<td>CO₂</td>
<td>10.6 µm</td>
<td>-</td>
<td>Continuous wave</td>
<td>16 kW</td>
</tr>
<tr>
<td>Welding of Ti-6Al-4V (Akman, Demir et al. 2009)</td>
<td>Nd:YAG</td>
<td>1064 nm</td>
<td>0.2-500 Hz</td>
<td>0.3 - 50 ms</td>
<td>4.5 kW (peak)</td>
</tr>
<tr>
<td>Welding of 304 SS (Yan, Gao et al. 2010)</td>
<td>CO₂</td>
<td>10.6 µm</td>
<td>-</td>
<td>Continuous wave</td>
<td>4 kW</td>
</tr>
<tr>
<td>Brazing of 304 SS and Al 5052* (Jeon, Kim et al. 1998)</td>
<td>CO₂</td>
<td>10.6 µm</td>
<td>-</td>
<td>Continuous wave</td>
<td>600 W</td>
</tr>
<tr>
<td>Brazing of alumina and steel (Rohde, Südmeyer et al. 2009)</td>
<td>CO₂</td>
<td>10.6 µm</td>
<td>-</td>
<td>Continuous wave</td>
<td>360 W</td>
</tr>
<tr>
<td>Brazing of Ti-6Al-4V and A6061 (Song, Nakata et al. 2013)</td>
<td>Ytterbium</td>
<td>1070 nm</td>
<td>-</td>
<td>Continuous wave</td>
<td>4 kW</td>
</tr>
<tr>
<td>Sintering* (Beaman and Deckard 1990)</td>
<td>Nd:YAG</td>
<td>1060 nm</td>
<td>-</td>
<td>Continuous wave</td>
<td>100 W (peak)</td>
</tr>
<tr>
<td>Sintering of poly(L-lactide) (Zhou, Lee et al. 2008)</td>
<td>CO₂</td>
<td>10.6 µm</td>
<td>-</td>
<td>Continuous wave</td>
<td>50 W (peak)</td>
</tr>
<tr>
<td>Sintering of porous Ti (Traini, Mangano et al. 2008)</td>
<td>Ytterbium</td>
<td>1054 nm</td>
<td>-</td>
<td>Continuous wave</td>
<td>200 W</td>
</tr>
<tr>
<td>Forming* (Geiger and Vollertsen 1993)</td>
<td>CO₂</td>
<td>10.6 µm</td>
<td>-</td>
<td>Continuous wave</td>
<td>850 W</td>
</tr>
<tr>
<td>Forming of 17-4PH SS (Lin, Cao et al. 2012)</td>
<td>CO₂</td>
<td>10.6 µm</td>
<td>-</td>
<td>Continuous wave</td>
<td>5 kW (peak)</td>
</tr>
<tr>
<td>Forming of 304 SS (Maji, Pratihar et al. 2014)</td>
<td>Ytterbium</td>
<td>1070 nm</td>
<td>-</td>
<td>Continuous wave</td>
<td>2 kW (peak)</td>
</tr>
<tr>
<td>Bending of steel foils* (Vollertsen, Komel et al. 1995)</td>
<td>Nd:YAG</td>
<td>1060 nm</td>
<td>-</td>
<td>Continuous wave</td>
<td>13.4-45.5 W</td>
</tr>
<tr>
<td>Bending of borosilicate glass (Wu, Zhang et al. 2010)</td>
<td>CO₂</td>
<td>10.6 µm</td>
<td>-</td>
<td>Continuous wave</td>
<td>70 W (peak)</td>
</tr>
<tr>
<td>Bending of 304 SS (Gollo, Mahdavian et al. 2011)</td>
<td>Nd:YAG</td>
<td>1060 nm</td>
<td>1-1,000 Hz</td>
<td>0.2-20 ms</td>
<td>400 W (average)</td>
</tr>
<tr>
<td>Shock peening of manganese* (Chu, Riggsbee et al. 1995)</td>
<td>Nd:glass phosphate</td>
<td>1054 nm</td>
<td>Not reported</td>
<td>600 ps</td>
<td>1 TW/cm² (peak)</td>
</tr>
<tr>
<td>Shock peening of thin films (Zhang, Yao et al. 2004)</td>
<td>Nd:YAG</td>
<td>355 nm</td>
<td>Not reported</td>
<td>50 ns</td>
<td>4 GW (peak)</td>
</tr>
<tr>
<td>Shock peening of Ti-6Al-4V (Zhang, Zhang et al. 2010)</td>
<td>Nd:glass</td>
<td>1064 nm</td>
<td>0.5 Hz</td>
<td>10 ns</td>
<td>1 GW (peak)</td>
</tr>
<tr>
<td>Shock peening of nickel-based superalloy (Bhamare, Ramakrishnan et al. 2013)</td>
<td>Nd:glass</td>
<td>1054 nm</td>
<td>Not reported</td>
<td>25.3-28.6 ns</td>
<td>9-16 GW (peak)</td>
</tr>
</tbody>
</table>
2.2 Overview of laser-based welding

Laser-matter interactions in directed energy deposition (DED) additive manufacturing are like those in laser welding in terms of workpiece material, laser power intensity, and interaction time. As a conventional manufacturing process that precedes the multi-layer builds in additive manufacturing, laser-based welding concepts set the foundation in laser-matter interactions for melting. The concepts of the melt pool, conduction mode laser processing, and keyhole mode laser processing are introduced in this section.

Laser-based welding is a type of a welding process as an alternative to electron beam, plasma or arc welding. The most common type of laser used in welding is the continuous wave, CO$_2$ laser with a 10.6 µm, far-infrared wavelength (Duley, Duley et al. 1999). Lasers reach maximum power values of about 50 kW, with powers of 1 to 15 kW used to weld high-strength metals, like steels (Duley, Duley et al. 1999). Laser absorption in the workpiece determines the heat transfer from the surface to surrounding material. Laser absorption in a metallic workpiece is defined by the laser interaction with the free electrons in a metal workpiece, with electron movement against the crystal lattice and defects in the workpiece. The electron transfer converts the laser energy into thermal energy in the workpiece, resulting in a temperature rise, and finally melting wavelength (Duley, Duley et al. 1999). Figure 2.2 shows the surface power density of CO$_2$ lasers in comparison to other welding mediums, such as electron beam and plasma welding. As seen in the figure, laser welding has a range of joining mechanisms, from radial conduction melting to the keyhole mode of melting (Lancaster 1984). In contrast to the CO$_2$ lasers in welding that operate at
10.6 µm, most DED processing uses solid-state lasers at near infrared wavelengths, or at 1.07 µm, modifying the optical properties of the workpiece material during processing.

When the workpiece material surpasses its melting point, the welding process reaches the conduction mode, where a radial “melt pool”, or a molten puddle of liquid workpiece material forms at the workpiece surface. Because the material’s absorptivity tends to increase with its temperature and the laser’s wavelength, the absorption of the laser into the workpiece increases during the DED process (Paquin 1995). At even higher laser power intensities, the temperature rises to vaporize the workpiece material, causing a recoil pressure that forms a cavity, or keyhole (Ki, Mazumder et al. 2002). The laser absorption increases substantially in the keyhole mode, as the cavity allows for inter-reflection of the laser beam within the workpiece. The keyhole mode of welding is useful for the joining of materials with thicknesses in the tens of millimeters (Kaplan 1994). However, when powder flow is introduced, modifying the laser welding process into a
powder-blown AM process, laser absorption fluctuates and modifies the nature of the keyhole mode laser-matter interaction.

Figure 2.3 shows the mechanisms of a general weld cross-section moving from the left to the right, with the slag area representing the melt pool, where Marangoni convection stirs the melt flow (Lancaster 1984). Marangoni convection is defined as the mass transfer away from areas in the melt pool of low surface tension, causing stirring within the melt pool. The Marangoni effect is induced by the gradient in surface tension due to the temperature distribution and species concentration at the melt pool surface (Tsai and Kou 1989). A pulsating cavity induced by the laser irradiation, or flux in this case, causes the welding process in Fig. 2.3 to be between the conduction and keyhole modes (Lancaster 1984). The Marangoni effect in the melt pool is also prevalent in the DED process, with process parameters determining whether the mode is conduction or keyhole mode.

![General mechanisms in a cross-section of a welding process](image)

Figure 2.3. General mechanisms in a cross-section of a welding process (Lancaster, 1984).

When laser welding approaches the keyhole mode, an induced vapor-plasma plume appears above the melt pool. The plume is a mixture of ionized gas in the environment, ionized shielding gas,
and the vaporized workpiece material. There are some additional key parameters other than laser power density that influence the resulting melt pool flow, plume formation, and resulting defects (Lancaster 1984). These additional parameters include the composition and the flow rate of the inert shielding gas, and the scan speed of the laser. The scan speed contributes to the interaction time of the laser with the workpiece material, influencing the overall laser power intensity.

Overall, understanding the influence of laser power density and interaction time in welding is key to understanding the laser-matter interaction when introducing metallic powder in additive manufacturing. Additive manufacturing be another way to describe multi-layer welding, with the same mechanisms of Marangoni flow in the melt pool, plume formation and porosity formation.

2.3 Optical properties of materials

The electromagnetic interaction with the free electrons of the workpiece material is the initial step during the overall laser-matter interaction. If a workpiece material is highly reflective or transparent, insufficient interactions and absorption of the laser’s photons with the free electrons of the workpiece material do not occur. Therefore, the optical properties of the workpiece determine the degree to which laser-matter interactions occur.

2.3.1 Role of optical properties in processing

When the reflectivity of a workpiece reaches high thresholds, high laser power intensity with shorter interaction times is required to make any impact on the workpiece. Aside from surface effects, the intrinsic optical properties of the workpiece material are determined by electronic
transitions, lattice vibrations, and free-carrier effects in the material (Majumdar and Manna 2003). The free-carrier effects consist of the electrons in the material absorbing photons, exciting them to a higher state, influencing the overall absorptivity of the material (Brown and Arnold 2010). All types of materials have electronic transitions that contribute to their index of refraction. Lattice vibrations contribute to optical properties in insulators and semiconductors. Free-carrier effects influence both the absorptivity and reflectivity properties in metals in visible and infrared wavelengths. In general, optical properties are based on the complex index of refraction (Paquin 1995):

\[ \tilde{n} = n + ik \] 

(2.1)

where \( n \) is the refractive index and \( k \) is the extinction coefficient. The refractive index indicates the velocity of a laser’s phase propagation through a material and its angle of refraction. The extinction coefficient indicates the amount of laser attenuation during propagation. These values change with the laser’s wavelength and the material’s temperature. A greater extinction coefficient indicates how much of the material will absorb the laser beam, which could lead to melting or vaporization:

\[ \alpha = 4k/\lambda_0 \] 

(2.2)

where \( \alpha \) is the absorption coefficient and \( \lambda_0 \) is the laser wavelength in vacuum. Refractive indices typically indicate reflectivity and transmittance of the laser beam through the material:

\[ r = \frac{(n-1)^2+k^2}{(n+1)^2+k^2} \] 

(2.3)

\[ r + t + a = 1 \] 

(2.4)

where \( r \) is the reflectance of the material, \( t \) is the transmittance, and \( a \) is the absorptance, or the ratio of radiant flux lost by absorption to the total incident radiant flux. Kirchhoff’s law of thermal
radiation states that absorptance is equivalent to emittance, where emittance is the ratio of radiated emitted power of a surface to the emissive power of a blackbody at the same temperature. For opaque materials with transmittance close to zero, emittance is determined by:

\[ r + \varepsilon = 1 \]

(2.5)

where \( r \) is the reflectance and \( \varepsilon \) is the emittance of a material (Paquin 1995).

Figure 2.4 (Ready 1997, Kennedy, Byrne et al. 2004) showcases how the optical property of reflectivity changes with laser wavelength for various materials at room temperature. High-powered diode lasers (HPDL) emit wavelengths of between 800 and 940 nm due to the electrons recombination in media like InGaAs/GaAs or GaAlAs/GaAs (Haag and Rudlaff 1997), which determines the wavelength emission. At wavelengths below 940 nm, most materials have lower reflectivity values, as seen in Fig. 2.4. HPDLs result in the greatest absorption into most metals, with absorptivity of more than 0.4 in carbon steel, compared with an absorptivity of less than 0.1 when processed with a 10.6 µm CO\(_2\) laser. However, HPDLs usually do not have enough laser power to melt and fuse metallic particles together with its typical power of less than 50 W, with the exception of those that are assembled into a laser stack (Kennedy, Byrne et al. 2004). Due to its high wavelength, 10.6 µm CO\(_2\) lasers interact with most metals at their highest reflectivity values (Haag and Rudlaff 1997).
Other than a reflectivity of 1, there is no maximum limit for laser processing, if the laser power is high enough to overcome the material’s optical properties at the surface. In addition to the laser power limit, another limitation to processing of highly reflective materials is the possibility for back reflection from the material’s surface, which can cause damage to the laser’s fiber optic (Naeem 2013). Solid-state laser processing (Nd:YAG lasers as seen in Fig. 2.4) have wavelengths in the IR or near-IR wavelengths, allowing for more efficient processing of most metallic materials due to decreased reflectivity as well as a high power input from these lasers. However, for refractory metals like polished silver (with polished referring to only specular reflection), copper, and aluminum, the reflectivity is at or above 0.8 at wavelengths when processed with Nd:YAG and CO₂ lasers, and are not suitable for most laser processing beyond the sub-surface of the workpiece material.

Figure 2.4 Change in reflectivity with wavelength for various materials. Figured modified from (Ready 1997) (Kennedy, Byrne et al. 2004) with permission from Elsevier.
With the ongoing development of high-powered (> 100 W) lasers with wavelengths less than 500 nm, including blue lasers from NUBURU that operate at 450 nm for up to 150 W continuous wave power (Zediker 2016) and diode-pumped solid-state ultraviolet lasers from Powerlase that operate at about 350 nm for up to 180 W nanosecond pulsed power (See, Chantzis et al. 2017), processing of materials like polished silver can become more effective. These wavelengths are shown in Fig. 2.4, with the reflectivity of silver decreased to below 0.5. Overall, optical properties of materials determine if and how the laser can interact with the material. The combination of laser properties and the workpiece's optical properties can provide a breakdown of the amount of laser energy absorbed, transmitted, reflected, and emitted from the workpiece surface.

2.3.2 Role of surface machining on optical properties

The initial interaction of a laser and the workpiece occurs at the workpiece surface. The surface properties of a workpiece material determine scattering of the laser either away or into the workpiece (Tay, Wang et al. 2003). Grinding, polishing, or electrochemical operations to modify the surface roughness can modify the refractive index of the workpiece surface. Surface machining operations can increase the reflectivity of the material and the angle of refraction of the beam into the material.

Gloss or the geometric attribute that causes a “shiny” appearance (Assender, Bliznyuk et al. 2002) is the optical finish of a surface and is directly linked to the topography of the material's surface. Gloss is quantitatively defined as proportional to the amount of incident light that is reflected at the specular reflectance angle, as opposed to diffused light, which is scattered light at many angles
The superposition of specular and diffuse reflection is the total reflectivity of a material’s surface. Figure 2.5 shows the difference between diffuse reflection and specular reflection (gloss) (Beckmann and Spizzichino 1987).

Figure 2.5. Diffuse and specular reflection relative to incident light (Beckmann and Spizzichino 1987).

Specular reflectance of unpolarized light is predicted from the Fresnel formula and is dependent on the material’s refractive index, \( n \), and the topography of the material’s surface, which influences the angle of incidence, \( \theta_i \) (Assender, Bliznyuk et al. 2002):

\[
 r_s = \frac{1}{2} \left[ \left( \frac{\cos \theta_i - \sqrt{n^2 - \sin^2 \theta_i}}{\cos \theta_i + \sqrt{n^2 - \sin^2 \theta_i}} \right)^2 + \left( \frac{n^2 \cos \theta_i - \sqrt{n^2 - \sin^2 \theta_i}}{n^2 \cos \theta_i + \sqrt{n^2 - \sin^2 \theta_i}} \right)^2 \right] \tag{2.6}
\]

where \( r_s \) is the specular reflectance. The above equation can be simplified by using the overall root mean square surface roughness, \( R_{RMS} \), of a material to determine the overall reflectance of a material’s surface (Assender, Bliznyuk et al. 2002), in addition to the definition in Eqn. 2.3:

\[
r = \exp \left[ - \left( \frac{4\pi R_{RMS} \cos \theta_i}{\lambda_0} \right)^2 \right] \tag{2.7}
\]

where \( r \) is the overall reflectance (including diffusive reflectance) and \( \lambda_0 \) is the wavelength of incident light, or of the laser beam. However, this relationship does not take in-plane distribution
of surface undulations into account, which determines localized light scattering. Various surface machining processes can provide various surface patterns with the same RMS roughness, resulting in varying ways incident light is reflected. A more localized relationship between reflectance with surface roughness can be calculated as the 1D scattered intensity:

\[ I(\omega) = r^2 \int \exp\left[-k_{\text{w}}^2 R_{\text{RMS}}(1 - A(\tau))\right] \exp\left(-\frac{j k_{\text{w}} \tau}{f}\right) d\tau \]  

(2.8)

where \( \omega \) is angular frequency, \( R \) is the material’s reflectance, \( A(\tau) \) is the autocorrelation function at the surface, \( \tau \) is the distance on the surface between two points, \( k_{\text{w}} \) is the wave number, and \( f \) is the focal length of incident light (Assender, Bliznyuk et al. 2002)

### 2.4.3 Continuous wave laser processing for melting

In materials where free-carrier effects dominate, such as metals, magnetic susceptibility and electrical conductivity also influence absorptivity. In a perfect metal, where the imaginary part of its dielectric constant is much greater than its real component, both optical properties and the conductivity are fully determined by the motion of free electrons, as denoted by the following relationship for the absorption coefficient at a material’s surface (Paquin 1995):

\[ \alpha = \left(\frac{\omega \mu \sigma}{2}\right)^{\frac{1}{2}} \]  

(2.9)

where \( \omega \) is is the angular frequency, \( \mu \) is the magnetic susceptibility of the material, and \( \sigma \) is the conductivity. For continuous wave laser processing of metals, the absorption and emissivity based on Eqns. 2.2, 2.3, 2.5, and 2.9 are used to determine the degree of melting of the material as the thermal properties described in Section 2.4 below are indicative of a radiant energy-matter interaction that can be described by optical properties. However, optical properties can be
expressed as spectral, or are a function of wavelength at constant temperature, or total, which is the integrated optical property over all wavelengths as a function of temperature (Paquin 1995).

As discussed in the previous sections on laser specifications, laser-matter interactions vary dramatically with laser wavelengths. Therefore, it is imperative to discuss which laser wavelengths are used during AM, as the absorptivity and emissivity of the workpiece material depends on this laser specification. Most of the experiments in this work used a 1070 nm wavelength solid-state laser for 316L SS and Ti-6Al-4V processing and a 1020 nm wavelength laser for Inconel 718 processing. At room temperature, optical properties for the base element for the three aforementioned alloys (316L SS, Ti-6Al-4V, and Inconel 718) can be calculated, assuming that each material has a mirror-finish surface with only specular reflectance. These properties are listed in Table 2.2 (Johnson and Christy 1974). There is limited data on the total optical properties of metallic alloys, as determining these values experimentally is a challenge, requiring a photo-receiver, oscilloscope, and microscopy (Tolochko, Khlopkov et al. 2000). Many models that attempt to emulate the laser-matter interactions during additive manufacturing use constant reflectivity and emissivity values even though these properties can change dramatically with rapid melting and solidification. A more thorough understanding of the optical properties used in laser-based additive manufacturing is necessary to optimize processing efficiency, develop more accurate models, and understand how to process novel materials.
Table 2.2. Optical properties of alloys used in additive manufacturing (Johnson and Christy 1974).

<table>
<thead>
<tr>
<th>Optical property</th>
<th>Iron</th>
<th>Titanium</th>
<th>Nickel</th>
</tr>
</thead>
<tbody>
<tr>
<td>Index of refraction, ( n )</td>
<td>2.9613</td>
<td>3.4740</td>
<td>2.5388</td>
</tr>
<tr>
<td>Extinction coefficient, ( k )</td>
<td>4.0133</td>
<td>4.0113</td>
<td>5.6815</td>
</tr>
<tr>
<td>Reflectivity, ( r )</td>
<td>0.627</td>
<td>0.615</td>
<td>0.773</td>
</tr>
<tr>
<td>Emissivity (also absorptance), ( \varepsilon )</td>
<td>0.373</td>
<td>0.385</td>
<td>0.227</td>
</tr>
</tbody>
</table>

2.4 Thermal properties of materials

In addition to optical properties, other intrinsic properties of a material that influences its melting and vaporization during laser processing includes density, electrical conductivity, electrical resistivity, crystal structure, and chemical composition. As discussed in the previous section, the intrinsic electrical conductivity of a material influences the laser absorptivity as the laser directly interacts with the free electrons (Paquin 1995). The crystal structure determines the anisotropy of elastic, electric, magnetic, and thermal properties (Callister and Rethwisch 2011). The various crystal structures of stainless steel, titanium alloys and nickel-based super alloys are face centered cubic (FCC) and hexagonal closed pack (HCP) (Callister and Rethwisch 2011). However, these crystal structures change with temperature and especially with phase transformations. Table 2.3 outlines the physical properties at room temperature of cast and annealed alloys that influence the properties detailed in this work. Table 2.4, in turn, lists the chemical compositions of the three alloys used in additive manufacturing in this thesis.

Table 2.3. Physical properties of alloys used in additive manufacturing.

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Density ((\text{kg/m}^3))</td>
<td>8000</td>
<td>4420</td>
<td>8220</td>
</tr>
<tr>
<td>Electrical resistivity ((\Omega \cdot \text{m}))</td>
<td>(7.4 \times 10^{-7})</td>
<td>(1.6 \times 10^{-6})</td>
<td>(1.3 \times 10^{-6})</td>
</tr>
<tr>
<td>Crystal structure</td>
<td>FCC</td>
<td>HCP/BCC</td>
<td>FCC</td>
</tr>
</tbody>
</table>
Table 2.4. Chemical composition of alloys used in additive manufacturing.

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Al</td>
<td>-</td>
<td>5.50-6.75</td>
<td>0.20-0.80</td>
</tr>
<tr>
<td>B</td>
<td>-</td>
<td>-</td>
<td>0.006 max</td>
</tr>
<tr>
<td>C</td>
<td>0.030</td>
<td>&lt;0.080</td>
<td>0.80 max</td>
</tr>
<tr>
<td>Cr</td>
<td>16.00-18.00</td>
<td>-</td>
<td>17.00-21.00</td>
</tr>
<tr>
<td>Co</td>
<td>-</td>
<td>-</td>
<td>1.00 max</td>
</tr>
<tr>
<td>Cu</td>
<td>-</td>
<td>-</td>
<td>0.30 max</td>
</tr>
<tr>
<td>Fe</td>
<td>Bal.</td>
<td>&lt;0.040</td>
<td>Bal.</td>
</tr>
<tr>
<td>H</td>
<td>-</td>
<td>&lt;0.015</td>
<td>-</td>
</tr>
<tr>
<td>Mn</td>
<td>2.00</td>
<td>-</td>
<td>0.35 max</td>
</tr>
<tr>
<td>Mo</td>
<td>2.00-3.00</td>
<td>-</td>
<td>2.80-3.30</td>
</tr>
<tr>
<td>N</td>
<td>0.10</td>
<td>&lt;0.050</td>
<td>-</td>
</tr>
<tr>
<td>Ni</td>
<td>10.00-14.00</td>
<td>-</td>
<td>50.00-55.00</td>
</tr>
<tr>
<td>Nb</td>
<td>-</td>
<td>-</td>
<td>4.75-5.50</td>
</tr>
<tr>
<td>P</td>
<td>0.045</td>
<td>-</td>
<td>0.015 max</td>
</tr>
<tr>
<td>S</td>
<td>0.030</td>
<td>-</td>
<td>0.015 max</td>
</tr>
<tr>
<td>Si</td>
<td>0.750</td>
<td>-</td>
<td>0.35 max</td>
</tr>
<tr>
<td>Ta</td>
<td>-</td>
<td>-</td>
<td>0.05 max</td>
</tr>
<tr>
<td>Ti</td>
<td>-</td>
<td>Bal.</td>
<td>0.65-1.15</td>
</tr>
<tr>
<td>V</td>
<td>-</td>
<td>3.50-4.50</td>
<td>-</td>
</tr>
</tbody>
</table>

2.5.1 Role of thermal properties in processing

Heating of a material during laser processing occurs when the deposited energy of laser irradiation converts to an elevated temperature profile that depends on the deposited laser’s energy profile and the thermal diffusion rate of the material. Thermal diffusivity, \( D \), depends on the material’s density, \( \rho \), thermal conductivity, \( \kappa \), and the specific heat, \( C_P \) (Paquin 1995):

\[
D = \frac{\kappa}{\rho C_P} \tag{2.10}
\]

Higher thermal conductivity is desirable in laser processing as this can minimize temperature gradients when there is a localized heat source irradiated into the material. A material with high specific heat requires more heat to cause a temperature change that might cause a distortion in the
lattice. This also means that high specific heat requires more energy, or laser power, to force a temperature change, either in cooling or heating.

In most metallic alloys, the vertical distance over which heat diffuses into a metallic workpiece is much greater than the thickness over which the laser is absorbed from the surface. This vertical distance, $z$, can be determined by the thermal diffusivity of the material and the interaction time of the laser, $t_p$ (Paquin 1995):

$$z = \sqrt{2Dt_p}$$  \hspace{1cm} (2.11)

In the one-dimensional heat flow condition, the heat balance equation can be expressed as (Mazumder and Steen 1980):

$$\rho C_p \frac{\partial T(z,t)}{\partial t} = Q(z,t) + \frac{\partial}{\partial z} \kappa \frac{\partial T(z,t)}{\partial z}$$  \hspace{1cm} (2.12)

where $T$ is the temperature density at a given depth, $z$, $Q$ is the laser power density at a given depth, and $t$ is time. Because material properties are temperature and phase dependent, this equation is usually solved using finite difference or finite element methods, as detailed later in Section 5, which discusses thermal history of additive manufactured parts.

The phase change of a material that undergoes laser processing is dictated by the resulting temperature profile and history. Depending on this history, the irradiated material may reach temperatures high enough for melting or vaporization (Majumdar and Manna 2003). During melting, a melt pool is created and defined by a liquid-solid interface, as will be detailed in Section 3 on DED processing. The liquid-solid interface moves based on the laser scanning method and speed (Gu, Meiners et al. 2012).
Additional thermal properties that play a role in laser processing include the coefficient of thermal expansion (CTE) of a material (Ion 2005). In general, lower thermal expansion leads to “better” laser-matter interaction, as this minimizes the effect of thermal gradients on dimensional changes of the final component. The material’s CTE can change with temperature and varying phase transformations in the crystal structure. For example, a single crystal of a cubic metal will have an isotropic CTE, while a hexagonal structure like Ti-6Al-4V has an anisotropic CTE (Paquin 1995).

The thermal properties of latent heat of fusion and latent heat of vaporization also play a role in laser processing as the material undergoes phase changes. These phase changes can dramatically increase the optical properties of the refractive index, and emissivity (Boone, Zhu et al. 2018). Changes in state will also modify the density and thermal properties like specific heat. For example, once a material reaches its vaporization temperature during laser processing, the metallic vapor can ionize and form a plasma. This plasma plume can attenuate the laser and decrease the absorptivity to almost zero, allowing for rapid cooling of the material (Miller and DebRoy 1990).

Table 2.5 lists and compares the thermal properties of the alloys used in this work. During laser processing, the energy distribution of the irradiated laser is often enough to heat the material to its vaporization point. Though thermal properties such as thermal conductivity and specific heat change with temperature, they are usually limited to a discrete value for each phase in thermal models. The thermal properties in the solid phase are at room temperature, whereas the properties at the liquid phase are just above the liquidus temperature of the material. The coefficient of thermal expansion, CTE, is the value close to the solidus temperature of the material while it is
still solid. The variations in thermal properties among alloys show that varying laser power and interaction times are required depending on the intrinsic material properties. For example, using a laser power of 1,000 W for both 316L SS and Ti-6Al-4V would result in different temperature profiles, thermal gradients, residual stresses, geometry compliance, and microstructures, which is explained more thoroughly in Section 2.4.2 and Chapter 6.

Table 2.5. Thermal properties of alloys used in additive manufacturing.

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Solidus temperature, K</td>
<td>1663</td>
<td>1878</td>
<td>1533</td>
</tr>
<tr>
<td>Liquidus temperature, K</td>
<td>1713</td>
<td>1933</td>
<td>1609</td>
</tr>
<tr>
<td>Boiling point, K</td>
<td>3090</td>
<td>3560 (Ti)</td>
<td>3186 (Ni)</td>
</tr>
<tr>
<td>Solid specific heat capacity, J·kg⁻¹·K⁻¹</td>
<td>500</td>
<td>553</td>
<td>435</td>
</tr>
<tr>
<td>Liquid specific heat capacity, J·kg⁻¹·K⁻¹</td>
<td>770</td>
<td>1500</td>
<td>755</td>
</tr>
<tr>
<td>Solid thermal conductivity, W·m⁻¹·K⁻¹</td>
<td>13.9</td>
<td>7.2</td>
<td>11.4</td>
</tr>
<tr>
<td>Liquid thermal conductivity, W·m⁻¹·K⁻¹</td>
<td>18.0</td>
<td>27.0</td>
<td>31.3</td>
</tr>
<tr>
<td>Latent heat of fusion, kJ·kg⁻¹·K⁻¹</td>
<td>270</td>
<td>360</td>
<td>290</td>
</tr>
<tr>
<td>Coefficient of thermal expansion, 10⁻⁶·K⁻¹</td>
<td>22.7</td>
<td>10.0</td>
<td>16.0</td>
</tr>
</tbody>
</table>

2.5.2 Thermally-induced phase transformations during laser processing

Phase transformations that occur due to either temperature changes or during plastic deformation lead to a different crystallographic structure of the final component. Thermally-induced phase transformations occur during heating and cooling at various rates, leading to a change in the unit cell, intermetallic phases, precipitates like carbides, etc. Phase transformations can also cause changes in the grain structure, size, and shape (Porter, Easterling et al. 2009).

Stainless steel 316L is an austenitic steel with a face centered cubic unit cell structure and is usually represented as the γ steel phase (Gill, Vijayalakshmi et al. 1986). Austenitic steels usually have a
greater composition of nickel and less chromium compared to the other iron allotropes of ferrite and duplex (Plaut, Herrera et al. 2007). However, austenitic stainless steels frequently result in delta ferrite in the microstructure after solidification, resulting in a decrease in ductility (Plaut, Herrera et al. 2007). Often, the presence of delta ferrite introduces interface fracture between the ferrite and matrix austenitic phases as austenite recrystallizes at a different temperature from ferrite (Gill, Vijayalakshmi et al. 1986).

Figure 2.6 (Plaut, Herrera et al. 2007) shows the TTT diagram (isothermal cooling that shows the phase transformation rate of austenitic steel) that shows the various phase transformations that occur between room temperature and the liquid state. For additive manufacturing, time to cool between the solidus and liquidus and solidus temperatures are on the order of milliseconds, typically resulting in recrystallization and martensitic phases. During rapid solidification, dendritic solidification structures can arise by delineating the liquid, “mushy”, and solid zones (Flemings 1974).
The room-temperature structure of Inconel 718 is like 316L SS in that it has a FCC crystal lattice structure, with the possibility of precipitates and metastable stable phases during heating and cooling (Knorovsky, Cieslak et al. 1989). Precipitates include $\gamma''$, $\gamma$, $\delta$, and carbides. The metastable strengthening $\gamma''$ phase transforms to the stable $\delta$ phase after heating, which can cause a degradation of mechanical properties. Rapid cooling can also result in the general dissolving of $\gamma''$ precipitates, which also weaken the final component. Figure 2.7 shows the TTT diagram for Inconel 718, and how the deleterious $\delta$ phase nucleates at the grain boundaries (Özgün, Gulsoy et al. 2013).
During rapid cooling, columnar grain structures that span several layers in selective laser melting, niobium carbides, and Laves phase on grain boundaries in a mostly \( \gamma \) solution have been observed (Chlebus, Gruber et al. 2015), (Idowu, Ojo et al. 2007). Depending on the thermal cycling and gas atomization process, a complex evolution of \( \gamma' \), \( \gamma'' \), and \( \delta \) phases can arise (Sames, Unocic et al. 2014).

Titanium alloys such as Ti-6Al-4V incorporate both the aluminum to stabilize its \( \alpha \) phase and vanadium to stabilize its \( \beta \) phase. When pure titanium heats up to its \( \beta \) transus temperature of 1,155 K, it undergoes a phase transformation from its \( \alpha \) phase, which is a hexagonal close packed structure, to its \( \beta \) phase, which is a body centered cubic structure (Ahmed and Rack 1998). However, alloying elements can adjust the transition temperature and drastically increase the strength of the alloy. At room temperature, Ti-6Al-4V is a combination of both alpha (\( \alpha \)) and beta
(β) phases, as aluminum is an α stabilizer and vanadium is a β stabilizer, though it is mostly an α phase matrix (Ahmed and Rack 1998).

Depending on cooling rate and prior heat treatment the micro constituents and microstructures are divided into several types, namely grain boundary allotriomorph α, globular or primary α (called bi-modal microstructure when the globular α is surrounded by Widmanstätten platelets), Widmanstätten, basketweave, and martensitic. A recently described microstructure is the bi-lamellar, in which the retained β phase, lying between the α platelets in a Widmanstätten structure, itself contains thinner secondary α platelets (Ahmed and Rack 1998).

For very slow cooling rates from high up in the α+β region or above the β-transus temperature the β phase mainly transforms into a globular type of α. Increasing the cooling rate enhances the α nucleation rate in the β grain boundaries thereby enhancing the formation and growth of α platelets into the prior β grains. The length and width of these α platelets are determined by the cooling rate, an increased cooling rate enhancing the nucleation rate and slowing the diffusion process (growth rate). At a certain point the cooling rate is fast enough for nucleation of α to occur inside the prior β grains as well, leading to the formation of the basketweave structure. Finally, if quenched, the β phase will fully or partly transform into a martensitic type of α’. This martensite exists in two different forms, α’ having hexagonal structure and α’’ having an orthorhombic crystal structure (Zeng and Bieler 2005). The grain boundary allotriomorph α phase starts to form as soon as the temperature drops below the β transus temperature. To what extent it then continues to grow along β grain boundaries depends on the cooling rate (Broderick, Jackson et al. 1985).
Figure 2.8 is a schematic of the microstructures resulting from various cooling rates (Kelly 2004). With high cooling rates (>410 K/s), martensite α’ forms, with matrix α forming at intermediate cooling rates (20<CR<410), and primary alliotriomorphic α at the grain boundaries or Widmanstatten α forming at slow cooling rates (CR< 20) (Kelly 2004).

![Figure 2.8](image)

Figure 2.8. An illustration of microstructure that occur in Ti-6Al-4V after quenching from different temperatures (Kelly 2004).

### 2.6 Mechanical properties of materials

Mechanical properties of a material rely on its crystal structure and vary with the crystallographic direction. In polycrystalline materials, grain boundaries are transition boundaries where atoms are at equilibrium and have a higher free energy than those within the grain boundaries (Paquin 1995). The atoms at the boundaries are the main agents of flow stress, fracture, and dislocations. Dislocations at grain boundaries are areas of high stress concentrations that can activate slip on a skew plane in a neighboring crystal, leading to deformation (Paquin 1995).
Mechanical properties can be divided into the elastic/plastic and fracture properties. The elastic properties of a metal can be described by a matrix of constants called the elastic stiffness constants. Because of symmetry considerations, there are a maximum of 21 independent constants that are further reduced for more symmetrical crystal types. For example, cubic materials have three constants (Bower 2009). These constants lead to the calculation of elastic moduli such as the Young’s modulus (stiffness, or the ratio of stress in a completely elastic region to the corresponding strain) and Poisson’s ratio (ratio of the absolute value of the lateral strain rate to the corresponding axial strain rate). These properties are functions of temperature and can also vary with crystallographic orientation (Bower 2009). Overall, they are sensitive to any modifications from manufacturing processes.

Strength is a measure of a material’s resistance to fracture (or onset of plastic deformation). Strength is highly dependent on material flaws, including dislocations, and therefore on the manufacturing process that may introduce flaws or strengthening mechanisms that limit dislocation propagation in a material (Hill 1998). These strengthening mechanisms include precipitation hardening, grain boundary strengthening, transformation hardening, work hardening, and solid solution strengthening. More details on these strengthening mechanisms are given in Chapter 8, which discusses the mechanical properties of additively manufactured 316L SS, Ti-6Al-4V, and Inconel 718 in this work.

Other mechanical properties include fracture toughness and hardness. Fracture toughness measures a metal’s resistance to brittle fracture, where low toughness corresponds to brittleness. This is
usually a measurement of the energy required for crack nucleation, with the amount of energy released corresponding to whether the material will undergo plastic deformation (ductile fracture) (Hill 1998). Hardness is a relative measurement of a metal’s resistance to abrasion, or wear, and is determined empirically, with varying values depending on the indentation method. Hardness has a qualitative relationship with strength and is dependent on temperature and the crystallographic orientation with respect to the tested metal’s surface. The mechanical properties of the alloys discussed in this work at room temperature are listed in Table 2.6. The materials listed in Table 2.6 are either cast or wrought with some strengthening mechanisms introduced for wide use in industry. However, there are large ranges of values for each material, depending on the manufacturing process.

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
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</tr>
</thead>
<tbody>
<tr>
<td>Young's modulus, GPa</td>
<td>193</td>
<td>110-119</td>
<td>200</td>
</tr>
<tr>
<td>Poisson's ratio</td>
<td>0.25</td>
<td>0.31-0.37</td>
<td>0.29</td>
</tr>
<tr>
<td>Yield strength, MPa</td>
<td>290</td>
<td>862-880</td>
<td>1100</td>
</tr>
<tr>
<td>Ultimate tensile strength, MPa</td>
<td>558</td>
<td>950-1,200</td>
<td>1375</td>
</tr>
<tr>
<td>Elongation at break, %</td>
<td>50</td>
<td>14</td>
<td>25</td>
</tr>
<tr>
<td>Fracture toughness, MPa·m$^{1/2}$</td>
<td>112-278</td>
<td>84-107</td>
<td>-</td>
</tr>
<tr>
<td>Hardness, Brinell (HB)</td>
<td>217</td>
<td>334</td>
<td>&lt;363</td>
</tr>
</tbody>
</table>

The role of laser processing on the mechanical behavior of materials lies primarily in how rapid heating and cooling modifies the crystallographic structure, introduces phase transformations, can result in residual thermal stresses within the material, and propagate dislocations within the material. For example, localized, high-energy laser processing prevents any necessary annealing that can remove stresses and minimize stress-induced birefringence in the material. Laser
processing usually maximizes the influence of localized thermal history, introducing greater degrees of anisotropy of mechanical properties in the material.

2.7 Summary

Careful consideration of laser specifications and properties of the workpiece material is required for effective processing. In this work, 316L SS, Inconel 718, and Ti-6Al-4V powder are used to build components using the DED process. Optical properties of each material used in this work determine laser-matter interactions, particularly in laser attenuation and absorptivity. Laser absorptivity results in melting, and sometimes vaporization of the material. Thermal properties determine the cooling behavior and geometry of the liquid-solid interface during the DED process. Each material undergoes phase transformations with varying rates of heating and cooling, which determine its mechanical properties.

The next chapter (Chapter 3) provides an overview of the DED process, where process parameters are uniquely tuned for different materials. Chapter 3 also takes a deeper look at the laser-matter interactions during a low-dosage DED process of Ti-6Al-4V powder particles. Ti-6Al-4V is an ideal material to observe using in-situ X-ray imaging due to its low density and X-ray mass attenuation. The combination of the titanium alloy’s optical, physical, thermal, and mechanical properties results in varying degrees of laser attenuation. Laser attenuation influences the melt pool boundary and any induced vapor or plasma at the melt pool surface, which in turn influences particle flow. Understanding the local interactions in the DED process can also lead to an understanding of how a material’s unique properties influence processing.
Chapter 3: Laser-Matter Interactions in Low-Dosage Directed Energy Deposition (DED)

This chapter focuses on the directed energy deposition (DED) process and its laser-matter interactions with low dosages of Ti-6Al-4V powder flow (Fig. 3.1). Low dosage of powder flow enables observation of how individual powder particles influence the melt pool during the DED process on a micro-scale. A high-resolution and time-resolved X-ray imaging technique, at the Advanced Photon Source, evaluates the interactions between the laser beam, flying particles, and the melt pool behavior during rapid solidification in DED. By expanding this advanced monitoring technique to AM processes, researchers can take advantage of the fundamental physics of the particle flow, fluid flow and heat transfer during laser-matter interactions to better model and predict for AM components.

Figure 3.1. Ti-6Al-4V will be discussed in this section on laser-matter interaction during low-dosage powder flow.
The motivation and an overview of the state-of-the-art in in-situ X-ray monitoring in AM processing are provided in Section 3.1. Section 3.2 discusses the setup and experimental conditions used in the in-situ high-speed X-ray imaging of the laser-particle interactions. The resulting Ti-6Al-4V particle behavior and its interaction with a moving laser beam from in-situ high-speed X-ray imaging are elaborated on in Section 3.3. A summary of the localized laser-matter interactions during the low-dosage DED process observed in this work and opportunities for future work are given in Section 3.4.

3.1 High-speed X-ray imaging in AM

Powder-blown laser additive manufacturing adds flexibility to the ability of building components with complex geometry. The understanding of the interaction of a laser beam and powder-blown deposition is limited. Therefore, in-situ monitoring is required to capture the influences of process parameters on powder flow and absorptivity of laser energy into the substrate. This work presents a piezo-driven powder deposition system that allows for imaging of individual powder particles that flow into a scanning melt pool. Here, in-situ high-speed X-ray imaging of the powder-blown additive manufacturing process of Ti-6Al-4V powder particles is the first of its kind and reveals how laser-matter interaction influences powder flow and porosity formation.

3.1.1 Motivation

This work aims to reveal the laser-particle interaction without additional environmental factors. The laser-induced vapor-plasma plume at the melt pool surface scatters and vaporizes powder particles. Control of powder delivery in AM processes is crucial as any change in mass flow
influences the thermal gradient and geometry of the resulting melt pool, and therefore, the mechanical behavior of the built component (Qi, Mazumder et al. 2006). A piezo-driven low-dosage powder delivery system allows for controlled powder flow to observe the interaction of individual particles and a moving laser beam, in contrast to observing a large mass, as in the actual process, of fast-moving particles and their interactions with each other and a moving laser beam.

In addition to observing particle flow during the process, a comparison between the keyhole and conduction modes during the process (as mentioned in the welding principles in Section 2.2) are made. Varying process conditions can change cavity depth evolution in relation to mass addition, mass subtraction, vapor plume formation, and porosity formation. The different modes of processing conditions include the keyhole mode and the conduction mode. At high laser power and slow scan speed, the laser interacts with the substrate to create a keyhole. In the keyhole mode, the laser beam induces a cavity, or depression zone, of ionized metal vapor into the substrate surface. As explained in the previous sections, particles entering the melt pool can attenuate the laser beam path. When the scan speed increases from about 100 mm/s to 500 mm/s, the energy density of the laser onto the substrate over time decreases. The resulting mode of laser-matter interaction changes from keyhole to conduction, where the substrate material is not vaporized within a deep cavity but creates a melt pool via laser conduction. Though there is still a laser-induced cavity, the penetration depth of the cavity in conduction mode does not result in keyhole porosity upon shrinkage.
3.1.2 The State of the art in in-situ X-ray process monitoring in AM

Researchers at Lawrence Livermore National Laboratory used ultra-high-speed imaging of laser-matter interactions in the powder bed fusion additive manufacturing process and found that vapor driven entrainment as opposed to widely believed recoil pressure led to particle spattering (Ly, Rubenchik et al. 2017). Surface tension arising from the Marangoni convection in the melt pool results in particle stirring and melting. The first of its kind, in-situ high-speed X-ray imaging of laser-matter interaction in a powder bed system using the Advanced Photon Source at Argonne National Laboratory revealed the capability to monitor the real-time growth of the melt pool geometry and its resulting cooling rate, particle ejection speeds out of the melt pool of tens of meters per second, and key hole porosity formation within 50 µs in a laser powder bed fusion system (Zhao, Fezzaa et al. 2017). A key study that contributes to the time-resolved mechanisms of laser-matter interactions in a powder-bed system includes ultrafast X-ray diffraction (Kenel, Grolimund et al. 2017) with a high temporal resolution of 10 ms, revealing the rapid phase evolution of titanium alloys. In-situ X-ray imaging of Marangoni convection driven porosity formation (Leung, Marussi et al. 2018) also reveals the mechanisms behind porosity migration in the melt pool. Though several studies have used in-situ X-ray monitoring for the powder-bed AM process, there have been no studies on in-situ of the powder-blown, or DED process, where different mechanisms may take place. Thus, the work presented in this thesis provides some of the first work that uses X-ray monitoring of the DED process in real time.
3.2 High-speed X-ray imaging experimental setup

To monitor the interactions of individual particles with a laser beam, including particle flow and the formation and evolution of defects, a piezo-driven powder delivery setup is brought into the 32-ID-B high-speed X-ray imaging beamline at the Advanced Photon Source (Argonne National Laboratory). The experimental setup includes the alignment of the piezo-driven powder delivery system with the galvo scanner, laser beam, X-ray beam, scintillator, right-angle mirror, and the high-speed camera.

3.2.1 Overall setup and triggering sequence

The overall schematic of the experimental system is shown in Fig. 3.2. The piezo-driven powder delivery system and sample are housed in a sealed chamber with an argon gas environment to be described in detail below. The 500 W continuous wave Ytterbium fiber optic laser is collimated and delivered through a galvo scanner that illuminates the beam onto the substrate in the chamber at scan speeds of up to 500 mm/s. The focus of the laser beam is about 80 µm and is aligned with the deposition area of the piezo-driven powder delivery system at the top surface of 500 µm thick Ti-6Al-4V Grade 5 sheet substrates.
The triggering sequence begins with the actuation of the piezo element in the powder delivery system. The signal from the piezo element switches the laser on. The “on” signal from the laser begins the laser scan by moving the galvo scanner mirrors. When the galvo mirrors are at a certain position that aligns the laser beam with the piezo-driven powder delivery system, the X-ray shutters to allow the X-ray beam traverse into the chamber.

An undulator, a component that consists of a periodic structure of dipole magnets, is inserted into an X-ray source to oscillate electrons and therefore, radiate energy. The gap width of the undulator components determines the energy flux of the X-ray beam. The undulator gap sets the harmonic energy of the beam to 24.4 keV and a corresponding wavelength of 0.508 Angstroms. A pair of slits defines the size of the X-ray beam. The slits provide a 2 by 2 mm field of view, and fast and slow shutters dictate the time intervals for the beam to pass through, as seen in Figure 3.2.

Figure 3.2. Schematic of the X-ray beamline with the DED process within the chamber.
The X-ray beam traverses through the observation windows of the chamber covered with Kapton film, which is larger than the 2 x 2 mm field of view. The X-ray beam penetrates through the top of the substrate to monitor the scan-build plane of the process where the laser-matter interactions occur. After the X-ray traverses through the region of interest, the scintillator outside of the chamber converts the beam into visible light, which triggers detection by the high-speed camera system.

After the X-ray beam traverses the material of interest, a scintillator converts the X-ray beam to visible light. A 45° mirror reflects the visible light into the high-speed camera, which serves as an imaging detector. The resolution of the resulting images is 1,024 by 1,024 pixels with a pixel size of 1.9 μm. The exposure time for each image is 500 ns with a frame rate of 50 kHz.

3.2.2 Piezo-driven powder delivery system

As discussed in the role of process parameters in the DED process (Section 1.2.3), industrial DED processing uses powder flow distributions with a diameter of up to 5 mm carried by an inert gas. However, capturing the interaction of a powder particle with a laser beam is difficult under industrial-scale flows. To better study the complex interactions between the laser beam and individual particles, a piezo-driven vibration-assisted powder delivery system is used (Fig. 3.3.) (Wu, Pritchet et al. 2018). A high-voltage amplifier controls the piezo element and provides a vertical vibration to induce a gravitational flow of powders out of the syringe-needle setup. The syringe serves as a powder hopper while the needle functions as a nozzle. The selected needle size is dependent on the surface properties, flowability, and size distribution of powder particles.
In the experiments in this work, the syringe was bent at 45 degrees relative to the laser beam path so that particles flow onto the substrate where the laser and X-ray beams are aligned, as seen in the close-up schematic in Fig. 3.4, which show a view of about 25 by 25 mm within the chamber. The field of view of the images is represented by the size of the X-ray beam, which is 2 by 2 mm.
The powder particle size, syringe diameter, needle length, and the humidity influence the powder mass flow rate. Additionally, the powder mass flow rate can be controlled by modifying the vibration frequency and amplitude. Of all the parameters, the most dominant factors that influence the powder mass flow rate are the syringe size and the vibration frequency and amplitude. Control of powder flow into a localized area presents a compact, low-cost, and efficient mechanism for powder delivery into a melt pool for imaging AM processes. The parameters used in this work include a voltage input of 400 V, a duty cycle of 10% and a range of 40 to 100 pulses for each experiment. The powders used are reused Ti-6Al-4V particles of 45 to 106 µm in diameter.

The DED process in this work uses a mass flow rate from 5 mg/s to 50 mg/s without inert carrier or shield gas, restricting the particle flow velocity. Low mass flow rates and a focused laser beam diameter of 80 µm allow for the isolation and imaging of individual particle flow relative to the melt pool. Industrial DED applications use powder mass flow rates of about 50 to 500 mg/s with carrier and shield gas (Frazier 2014).
3.3 Particle flow behavior

The following results show the behavior of powder particle flow from the piezo-driven device and how individual particles flow above the substrate surface. Because the particles are gravitationally fed into the melt pool, there are no external factors to drive particle deposition, such as inert carrier gas. The DED process introduces complexity to the laser-matter interaction in that the amount of laser energy absorbed by either particles or the substrate fluctuates with particle flow behavior. The type of particle flow behaviors observed show the uniqueness of DED phenomena, including how the laser-induced vapor-plasma plume scatters mid-air individual particles above the substrate surface, interactions among particles, vapor-induced pressure gradients, and a combination of both interactions with other particles and pressure gradients that can propel particles into the melt pool.

The interaction of the laser beam and substrate creates a melt pool with Marangoni convection that entrains particles into and out of the melt pool, behaving similarly to laser-matter interactions in powder-bed systems. For example, the introduction of cold particles into a stirring melt pool can lead to instability and growth of the melt pool. The surface tension of the melt pool introduces a Marangoni flow that entrains nearby flowing or stationary particles with low vertical momentum relative to the substrate surface. Figure 3.5 shows a keyhole mode experiment, or an experiment where a metallic vapor-filled cavity is present. Keyhole mode occurs when the laser's power density onto the substrate is great enough to create a cavity into the substrate that is filled with ionized vapor and ambient gas (Lee 2002). The laser power of the keyhole experiment is 150 W and uses a laser scan speed of 100 mm/s, with Fig. 3.5 showing the influence of mass addition on the porosity and cavity geometry.
Figure 3.5. Cavity, melt pool, and porosity evolution as mass (by means of powder particles) was added in an experiment with a laser power of 150 W and a scan speed of 100 mm/s. All frames are processed, and not raw, images.

The frames in Fig. 3.5 are processed images from the open-source software, ImageJ, where the pixel intensities in the raw images are divided by the pixel intensities of the initial frame before the laser turns on. Then, the brightness and contrast are adjusted so that the intensity range of all pixels range from 0 (black) to 2.5 out of 255 (white). High intensity, or light, pixels that represent particles above the substrate surface are artifacts from image processing, where a particle was in place in the initial frame. Low intensity particles above the substrate surface show a moving particle that is present in the frame and not an artifact. The intensity contrasts in the images around the melt pool reveal the phase state of the Ti-6Al-4V in the experiment. These contrasts depend on the density of the material at different phases. The melt pool above the substrate surface is represented by dark intensity pixels, representing the contrast of the liquid melt pool against the low density, lighter intensity ambient air pixels. The liquid melt pool below the substrate surface
has slightly lighter intensity pixels than the surrounding, slightly denser solid material, with the red outlines in Fig. 3.5 revealing the liquid-solid interface of the melt pool. The blue lines outline the gas-liquid interface, or the cavity, where the light intensity pixels represent the vapor phase of Ti-6Al-4V. These light intensity pixels that appear within the melt pool but outside of the cavity represent porosity formation, as indicated by the white outlines in Fig. 3.5.

3.3.1 Particle scattering

This section highlights the uniqueness of DED phenomena, including how the laser-induced vapor-plasma plume scatters mid-air individual particles more than 1 mm above the substrate surface, interactions among particles, vapor-induced pressure gradients, and a combination of both interactions with other particles and pressure gradients that can propel particles into the melt pool.

When a laser illuminates a substrate surface with high power density, material vaporizes, forming a cavity that penetrates below the substrate surface. Process parameters influence the pressure and velocity of an expanding vapor-plasma plume, which pushes particles away from the melt pool and scatters them. The vapor-plasma plume reaches and pushes particles more than 1 mm above the substrate surface, resulting in particle scattering speeds between 1 and 10 m/s, depending on the size of the particle and its proximity to the melt pool. Scattering speeds were determined by tracking the trajectory of particles from 2D images. By considering the pixel size of 1.9 µm and a frame rate of 20 kHz, particle speeds and trajectories could be estimated. Smaller particles closer to the melt pool scatter with the greatest velocities, with accelerations up to 40 m/s². Particle scattering also leads to laser attenuation, or the obstruction of the laser beam. This results in
increased melt pool volatility and changes in the aspect ratio of the cavity. Change in mass addition and laser scattering in DED does not allow the melt pool to arrive at an equilibrium that a powder bed system would allow. In an inert environment, laser energy also ionizes the surrounding argon gas, causing a plasma plume that coincides with the metal vapor that jets from the substrate surface.

The scattering of the particles exactly reflects the laser-induced vapor-plasma plume at the melt pool. A velocity of about 1 m/s is required for a particle from more than 200 µm above the substrate surface to enter the melt pool (Qi, Mazumder et al. 2006), prohibiting most the piezo-driven and gravity-fed particles to enter the melt pool. This velocity threshold has been demonstrated in a numerical simulation of the DED process that captured the heat transfer of the laser-matter interactions during powder flow (Qi, Mazumder et al. 2006). The simulated forces and velocities of the vapor from the cavity during the laser scan were plotted and could only be penetrated with a high enough particle velocity.

3.3.2 Particle interactions above the melt pool

Though most particles that flow vertically toward the melt pool scatter due to the vapor-plasma plume at the substrate surface, high-speed X-ray imaging also captures individual particles that enter the melt pool. When in-flight particles interact with each other, they provide an additional force from their impact that allows the particles to penetrate the vapor plume and enter the melt pool. As seen in Fig. 3.6 at 50 µs, the laser beam scans toward the right, pushing in-flight powder particles away from the melt pool. The frames in Fig. 3.6 are raw images with increased contrast.
Figure 3.6. Interaction of two particles that rotate into the melt pool during a keyhole mode experiment where laser power is 250 W and the scan speed is 300 mm/s.

From 50 to 200 $\mu$s, the particle farther away from the underlying melt pool (circled in green and denoted by “1”) collides with another particle (also circled in green and denoted by “2”). At 200 $\mu$s, the surrounding vapor-plasma plume heats both circled particles to fuse them at their boundaries, as seen in the close-up of the particle interactions between 150 to 250 $\mu$s in Fig. 3.7.

Figure 3.7. Close-up of the interactions in Fig. 3.6 of two in-flight powder particles that fuse at their boundaries.
These particles are sintered together and begin to propel with rotation into the melt pool from 200 µs to 600 µs, when the coalesced particles enter the melt pool. Increased mass leads to increased momentum, allowing the particles to penetrate the vapor-plasma plume barrier (Khairallah, Anderson et al. 2016) which is assumed to be emitted from the laser-induced cavity during the process. Although the barrier is not detected in the X-ray images, most powder particles with low speeds in their trajectories scatter away from the cavity, as mentioned in the previous section. The sintered particles move at a velocity of about 2 m/s, beyond the threshold velocity of about 1 m/s for particles to enter the melt pool. Increased mass and the momentum that initiate with the coalescence of two particles lead to a rotational motion that allows the particles to contribute mass to the DED process.

3.3.3 Particle flow due to pressure gradient

The pressure gradient induced by the vapor-plasma plume at the surface of the substrate also entrains individual particles into the melt pool. Vapor driven entrainment controls nearby particles when the particle is in the path of the laser beam and, therefore, the cavity-induced pressure gradient as seen in Fig. 3.8. The frames in Fig. 3.8 are raw images with increased contrast. In Fig. 3.8a, the laser scans from right to left. From 5,750 to 6,000 µs, vapor-plasma plume scatters the circled particle farther away from the substrate surface with no indication of rotation. At 6,050 µs, the particle contacts the laser beam and changes shape, indicating either rotation, melting, or both, as shown in Fig. 3.8b. As known from literature in laser processing (Khairallah, Anderson et al. 2016), it is assumed that the laser beam also vaporizes a section of the particle’s surface, creating
a localized metal vapor plume. The localized vapor plume causes a jet-like pressure gradient at the particle surface, pushing the particle toward the melt pool with a velocity of about 4 m/s. At such an elevated velocity, the particle penetrates the vapor-plasma plume without the aid of a carrier gas. This shows that localized vapor driven entrainment can capture a particle at least 200 μm above the surface of the melt pool.

Figure 3.8. Example of vapor entrainment during a keyhole mode experiment where the laser power is 150 W and the scan speed is 100 mm/s.

The molten or rotated particle in Fig. 3.8b at 6,050 μs also attenuates the laser beam path. Figure 3.9 illustrates additional evidence of laser attenuation at 6,050 μs frame from Fig. 3.8a by looking at the melt pool. In contrast to Figs. 3.7 and 3.8, Fig. 3.9 shows a processed image where the frame is divided by the initial frame to show the contrast of the cavity and melt pool boundaries. Figure 3.9 shows a segmented representation of the trajectory of the molten particle relative to the
underlying melt pool. The light-intensity areas within the melt pool include the laser-induced cavity and porosity. The cavity occurs where the substrate material is vaporized by the laser, generating a vapor-plasma plume. Compared to above the substrate surface, the cavity generates the greatest density and velocities of the vapor-plasma plume. In the keyhole mode of laser-matter processing, the laser-induced cavity extends to the bottom of the melt pool. However, when the laser is suddenly obstructed or turned off, the penetration of the cavity into the substrate simultaneously collapses and decreases in depth. This collapse causes rapid shrinkage and a recoil pressure within the melt pool, inducing two large keyhole pores below the cavity that are around 100 µm in size.

![Diagram showing laser scanning, cavity, and molten particle](image)

Figure 3.9. Example of vapor-driven entrainment of a single particle.

The decrease in the cavity penetration depth into the melt pool indicates loss of absorptivity of the laser into the substrate surface, causing sudden changes in the thermal history of the melt pool and of the additive manufactured build. Figure 3.8a shows that at 6,100 µs, the trajectory of the molten
particle diverts downwards towards the melt pool. Figure 3.8b shows that the particle regains its shape and size at 6,100 µs, indicating a particle that solidifies, rotates, or both. As the particle regains its shape and size during its downward trajectory toward the melt pool, the particle no longer blocks the laser beam path. The laser-induced cavity within the melt pool regains its full depth and coalesces with the large keyhole pores. This fluctuation in cavity depth and melt pool shrinkage reveal the fluctuations with the absorptivity of the laser into the substrate surface as a particle interacts with the laser beam.

3.3.4 Particle flow due to both pressure gradient and interaction with other particles

Figure 3.10 shows a conduction mode experiment where both a localized pressure gradient and particle lead to mass addition into the melt pool. As the laser scans toward the right, the vapor-plasma plume from the substrate surface entrains the two circled particles upward from 6,550 to 6,600 µs. At about 6,600 µs, the surface of the smaller particle contacts the laser beam, causing a localized vapor-induced pressure gradient at the particle surface. The smaller particle also melts into a liquid particle during the laser scan.
As seen at 6,650 µs, the molten smaller particle rapidly moves downward, contacting the larger circled particle. In this case, the temperature of these two particles was sufficiently high to cause fusing at the boundary and subsequent melting into a single larger particle, as seen from 6,650 and 6,700 µs. The coalescence into a single particle can be attributed to a few possible factors, including the fact that the smaller particle was in a molten state when it contacted the larger particle, conducting the larger particle to its melting point. Other possible factors for a full melt of both particles include the proximity of both particles to the laser beam at 6,650 µs and the possibility that the larger particle also contacted the laser beam before 6,550 µs. The momentum induced as the two particles coalesce does not create a rotational propelling motion into the melt.
pool as seen in Fig. 3.7, but mostly a linear trajectory downwards toward the melt pool. Even as the laser is turned off at 6,800 µs in Fig. 3.10, the coalesced particle is still on its trajectory toward the solidifying substrate surface. Because the cooling behavior of the melt pool is more rapid when the process is in conduction mode versus in keyhole mode, the coalesced particle only partially melts into the substrate.

3.4 Summary

Overall, the various mechanisms of particle flow behavior depend on the laser beam path and can attenuate the laser. The uniqueness of the work presented above, in contrast to powder-bed studies, is in particle deposition behavior in DED.

During gravity-fed powder flow, the laser-induced vapor-plasma plume that is emitted from the cavity typically scatters particles away from the melt pool. There were situations where gravity-fed particles interacted with each other by fusing at the boundaries and entered the melt pool. Entrainment away from the melt pool can lead particles to interact with each other or contact the laser beam path. These interactions induced a momentum or pressure gradient that allowed for particles to enter the melt pool. In turn, as particles flowed in the path of the laser beam, the resulting melt pool fluctuated due to fluctuating attenuation. Two instances of vapor entrainment of the particle occurred, with one instance of vapor entrainment scattered the particle upwards in the direction of the growing vapor-plasma plume. The second instance of vapor entrainment occurred when the particle contacted the laser beam, vaporizing a fraction of the particle surface. The localized vapor jet pushed the particle into the melt pool.
DED introduces complexity to the laser-matter interaction in that the amount of laser energy absorbed by either particles or the substrate fluctuates with particle flow behavior. This work highlights the uniqueness of DED phenomena, including how laser-induced vapor-plasma plume scatters mid-air individual particles more than 1 mm above the substrate surface, interactions among particles, vapor-induced pressure gradients, and a combination of both interactions with other particles and pressure gradients that can propel particles into the melt pool.

The purpose of *in-situ* high-speed X-ray imaging experiments in additive manufacturing is to capture and quantify physical phenomena during DED processes for controlled processing and materials development, which is still relatively unclear in the scientific community. High-speed X-ray images reveal the laser-matter interactions in various modes of DED processing can aid in the validation of thermal, thermo-fluid dynamic and thermo-mechanical models. Further studies are necessary to understand interesting mechanisms of particle entrainment, particularly of particles that attenuate the laser beam path. Controlling individual particle trajectories relative to a laser beam can lead to more conclusive observations about how particles can enter the melt pool.

This work reveals the necessity of an inert carrier gas to aid particle flow. Without carrier gas, most particles scatter away from the melt pool, whereas carrier gas allows particles to penetrate the laser-induced vapor-plasma plume. Future work that investigates the influence of carrier gas pressure and velocity is required to capture the phenomena in more representative DED processing. In addition to the use of carrier gas, industrial powder flow rates are about ten times greater than the rates used in this study, or about 500 mg/s in industry compared to up to 50 mg/s
used in this study. The laser beam diameter used in high-speed X-ray imaging was 80 µm, which is about 10 to 50 times smaller than that used in industrial DED processing. The laser scan speed used was between 50 and 500 mm/s, compared to the scan speeds of about 5 to 50 mm/s in industrial processing. Representative DED processing, especially that in industrial applications, requires different ranges of process parameter values. Monitoring of industrial scale laser-matter interactions requires larger scale metrology methods. As discussed in Chapter 4, measuring the resulting clad geometry provides insight into industrial scale laser-matter interactions during the DED process.
Chapter 4: Laser-Matter Interactions During Nozzle-Driven Powder Flow in Directed Energy Deposition (DED)

This chapter will discuss both experimental and theoretical laser attenuation during the DED process of Inconel 718 with a commercial powder feeding system. In laser-based DED, powder mass flow into the melt pool influences the cooling behavior and properties of a built part. Varying mass powder flow rates influence laser attenuation, melt pool convection, thermal history, and phase transformations of the solidifying melt pool. Section 4.1 discusses the dilution ratio and the importance of fusion between layers. A better understanding of the influence of powder flow is provided by investigating the efficiency of powder flow of Inconel 718 particles from a coaxial nozzle. Experimental methods to build and characterize single clads are given in Section 4.2. Sections 4.3 and 4.4 discuss the clad height and dilution structural characteristics, respectively while Section 4.5 provides a summary of laser attenuation during DED and how different processing conditions influence the thermal history of AM-processed material.

4.1 Capturing laser attenuation with dilution ratio

Varying powder flow rates at constant laser power and laser scan speed result in differing single-track cross sections with three categories of cross-section shapes (Weerasinghe and Steen 1987) that are characterized by the angle between the substrate and the tangent of the single-track cross-section edge, as seen in Fig. 4.1. Low powder flow rates result in high dilution, low clad height and large clad angle, whereas the opposite trends occur at high powder flow rates, indicating loss of adhesion.
Dilution is defined by (Toyserkani, Khajepour et al. 2004):

\[ D = \frac{d}{h + d} \]  (4.1)

where \( h \) is the height of the clad above the substrate and \( d \) is the clad depth below the surface of the substrate.

Dilution can be used to quantify the bond between the melted, deposited material and the substrate (Toyserkani et al., 2004). Dilution also elucidates the amount of laser attenuation by powder flow during the process. Substantial dilution ratios vary with deposited and substrate material. Dilution ratios, \( D \), greater than 0.6 for Inconel 718 clads onto steel substrates indicate excessive melting of the substrate material and cannot pass qualification standards (Lin 2013). During cladding of powder onto a substrate of a dissimilar material, minimal dilution is preferred to stabilize gradients in chemical composition (Shamsaei, Yadollahi et al. 2015). In a study on the dilution of laser-cladded gamma-TiAl intermetallic powders onto a titanium alloy substrate (Maliutina et al., 2016), low dilution values of 0.06 were optimal for high microhardness properties. In multi-layer builds, low dilution between layers contributes to lack of fusion. A dilution ratio of at least 0.15 is required.
for substantial bonding in a study of laser deposited Ti-6Al-4V powders onto a Ti-6Al-4V substrate (Tuhin Mukherjee, Zuback, De, & DebRoy, 2016). However, these threshold values are flexible and can be compensated for by adjusting the layer thickness when building multilayer structures. The dilution of a melt pool depends on the material’s thermal properties like conductivity, expansion, absorption, surface tension and chemical composition, etc., relative to the properties of the underlying layer or substrate.

Several studies have investigated process maps to determine optimal combinations of laser power and powder flow rate by using theoretical laser deposition models (Weerasinghe and Steen 1987). A model that included the mixing of powder in the melt pool showed that, in the ideal case where dilution is minimal, that clad height varied linearly with laser power and the laser scanning speed (Hoadley and Rappaz 1992). Olivera et al. (De Oliveira, Ocelik et al. 2005) found relationships between the parameters of powder flow rate, laser scan speed, and laser power with the clad height, clad width, clad area, molten area, and clad angle. Zhang et al. (Zhang, Yao et al. 2011) discussed the influence of process parameters in terms of specific energy, calculated by dividing actual power by beam diameter and scanning velocity. They found that the height of a cladding layer first increases, then decreases with increasing specific energy, concluding that specific energy affects size shape and formation rate of the clad. Liu and Li modelled a single clad track (Liu and Li 2005) and a thin wall (Liu, Li et al. 2007) created using a low power laser, concluding that the clad shape is dominated by the powder concentration at any point for the duration that the point is molten.
In powder-blown additive manufacturing, powder mass flow rates influence the geometry and dilution of a clad. Several studies have found that the thermal history and the cooling rate of the melt pool have a direct influence on the amount of dilution in additive processing (Farshidianfar, Khajepour et al. 2016), (Bennett, Wolff et al. 2017). Melt pool depth and dilution have a nonlinear relationship with process parameters (Fathi, Toyserkani et al. 2006). With relatively high dilution ratio values of greater than 0.5, the cooling rates from IR imaging at the surface were greater than 4,000 K/s, suggesting that most of the relatively low mass powder flow entered the cavity within the substrate and mixed with the substrate at a high level of energy density at the solidification front, or liquid-solid interface, at any given time.

The clad height and dilution ratio are metrics that indicate fusion of the DED melt pool onto the underlying substrate or underlying layers. Understanding of how processing conditions and thermal history influence dilution is limited. The experiments conducted in this chapter link process parameters to Inconel 718 clad structures, including height above the substrate and the dilution ratio. The use of a thermal fluid dynamic model in Chapter 5 allowed the calibration of the melt pool boundary conditions and the prediction of the overall clad structure.

4.2 Experimental Methods

A DMG MORI LaserTec 65 3D, a hybrid additive and subtractive five-axis machine tool, was used (Bennett, Wolff et al. 2017) to build single clads by melting deposited Inconel 718 (IN718) powders with a high-powered continuous wave laser, as seen in Fig. 4.3a. The machine included a fiber-coupled direct diode laser with a maximum power of 2,500 W at a wavelength of 1,020
The beam diameter at its focus was 3 mm. Gas atomized super alloy IN718 powders of particle size 50-150 µm were deposited onto the laser's focal spot. Argon at a flow rate of 7 L/min was used as both shielding and carrier gas.

Ten Inconel 718 clads of 50 mm in length were deposited on 1045 medium carbon steel discs, with 7 mm spacing between clads, as shown in Fig. 4.2b. The laser power was held constant on each substrate disc. The powder flow rate was controlled by the powder hopper, where the maximum powder flow rate reached up to about 450 mg/s, depending on the powder specifications. On each substrate disc, the powder flow rate increased from 56 to 453 mg/s to build the ten clads. The ten powder flow rates for the clads were 56, 109, 160, 209, 256, 300, 342, 381, 418, and 453 mg/s.

![Figure 4.2. a) Experimental setup of the DED of the IN718 single clads courtesy of Jennifer Bennett; b) the resulting ten clads with increasing powder mass flow on the AISI 1045 carbon steel substrate disc.](image)

The laser power values were 1,000 W, 1,200 W, 1,400 W, 1,600 W, 1,800 W and 2,000 W, resulting in six substrates with ten clads each for a total of 60 single clads. The influence of laser scan speed was not investigated in this study, as the speed was held constant during the build of
all the clads. The laser scan speed for all depositions was 1,000 mm/min, or 16.7 mm/s. To allow for cooling of the previous clads before subsequent clads were applied, the machine dwelled 120 seconds between the deposition of each clad. The clads were analyzed at three points within each clad for their thermal histories and changes in geometry. The points were 10, 25, and 40 mm from the start of the 50 mm long clad, indicating evaluation points that were close to the start, middle, and end of the builds for each clad. With six laser power values, ten powder flow rates, and three positions for each clad, there were a total of 180 evaluation points, as described in Table 4.1.

An Alicona InfiniteFocus white light optical measurement instrument that relies on 3D focus variation was used to measure the geometric profiles of each clad relative to the substrate surface. Five measurements were taken at the 10 mm, 25 mm and 40 mm positions of each clad totaling 900 measurements of all the 180 evaluation points. The vertical resolution of the measurements was 1 µm and the horizontal resolution was 5 µm. Some of the laser clads exhibited un-melted powder particles on the surface of the clad and the adjacent substrate surface. Wire electrical discharge machining separated the substrate discs at the cross sections to view clad dilution into

<table>
<thead>
<tr>
<th>Laser Power (W)</th>
<th>Laser scan speed (mm/s)</th>
<th>Powder Flow Rate (mg/s)</th>
<th>Position (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1,000</td>
<td>16.7</td>
<td>56, 109, 160, 209, 256, 300, 342, 381, 418, 453</td>
<td>10, 25, 40</td>
</tr>
<tr>
<td>1,200</td>
<td>16.7</td>
<td>56, 109, 160, 209, 256, 300, 342, 381, 418, 453</td>
<td>10, 25, 40</td>
</tr>
<tr>
<td>1,400</td>
<td>16.7</td>
<td>56, 109, 160, 209, 256, 300, 342, 381, 418, 453</td>
<td>10, 25, 40</td>
</tr>
<tr>
<td>1,600</td>
<td>16.7</td>
<td>56, 109, 160, 209, 256, 300, 342, 381, 418, 453</td>
<td>10, 25, 40</td>
</tr>
<tr>
<td>1,800</td>
<td>16.7</td>
<td>56, 109, 160, 209, 256, 300, 342, 381, 418, 453</td>
<td>10, 25, 40</td>
</tr>
<tr>
<td>2,000</td>
<td>16.7</td>
<td>56, 109, 160, 209, 256, 300, 342, 381, 418, 453</td>
<td>10, 25, 40</td>
</tr>
</tbody>
</table>
the substrate. After polishing the cross sections, dilution of the solidified melt pool was viewed and measured using optical microscopy with a Nikon Eclipse MA200 inverted materials microscope.

4.3 Height of Inconel 718 single clads

The interactions of laser processing power, powder flow rate and laser scan speed contribute to the clad’s geometry, surface roughness and the likelihood of any un-melted powders adhering to the clad surface. As indicated by two experiments in Fig. 4.3, there were un-melted powders at the surfaces in several experiments, indicating cold powders adhering to an already solidified melt pool and not melting into the clad.

Figure 4.3. Examples of the clad surface: left) end of the clad processed with 1,000 W laser power and 56 mg/s powder flow rate; right) end of the clad processed with 2,000 W and 56 mg/s.
As seen in Fig. 4.3, when the powder mass flow rate remained constant but the laser power increased from 1,000 to 2,000 W, the height increased from 0.15 to 0.25 mm. The difference in height suggests that the greater laser power melted a greater percentage of power. Laser attenuation due to powder flow when processed with the 1,000 W beam might have attributed to a clad height of only 0.15 mm. The width of the clad processed with 1,000 W was only about 2 mm, compared to the 2.5 mm width when processed with 2,000 W. Though the laser beam diameter was 3 mm, the laser power intensity of the beam was not enough to melt the Inconel 718 particles due to the Gaussian distribution of power throughout the beam. With a 2,000 W beam, the power distribution was high enough to induce melt pools and build clads with a width of about 2.5 mm. On the other hand, a mass flow rate of 453 mg/s that interacted with a 1,000 W laser power yielded a clad height of about 1.5 mm, compared to a height of 1.0 mm when processed with 2,000 W laser power. The decrease in height with an increase in power can be attributed to the clad dilution ratio, which is discussed in the next section.

When the laser power remained constant at 1,000W, the vertical height of the clads processed with 453 mg/s powder flow were about nine to ten times greater than the heights of the clads processed with 56 mg/s. This shows a linear relationship between powder mass flow rate and clad height. However, when the laser power remained constant at 2,000 W, the vertical height of the clads processed with 453 mg/s powder flow were only about four to five times greater than the heights of the clads processed with 56 mg/s, indicating some volume loss. This points to the dilution into the substrate because of the laser beam vaporizing, melting, and mixing the Inconel powder particles with the underlying carbon steel substrate material. On average, clad heights decreased
with increasing laser power, most possibly because of increased dilution of the IN718 powders. At greater laser power and powder flow rates, clad heights at the middle were greater than those at the start and end of clads, indicating greater dilution, whereas there was loss of dilution and adhesion for low laser power and powder flow rates. As shown in Fig. 4.4, variability of clad height increased with increasing powder flow. In the case of high laser power and low powder flow rates, the start of the clad was highly diluted into the substrate.

![Figure 4.4](image)

Figure 4.4. Z height of clad at various parameters and clad positions. At each powder flow and laser power the bars represent the cooling rate of the 10 mm (left), 25 mm (center), and 40 mm (right) points (Bennett, Wolff et al. 2017).

The metric of energy density per unit of mass can be used to capture the laser-matter interaction during the DED process. Because the laser scan speed of each clad was kept constant at 16.7 mm/s, this metric compares the influence of the laser power and powder flow, similarly to the powder density used in previous dilution studies (Maliutina, Si-Mohand et al. 2016). The energy density metric also compares the dilution results from both experiments and thermal model, as it will be
discussed in Chapter 5. Energy density is used to scale the relation between dilution and process parameters, with the definition of energy density as follows:

\[ E_D = \frac{Q}{\dot{m}} \]  

(4.2)

where \( Q \) is the laser power and \( \dot{m} \) is the powder mass flow rate.

Figure 4.5 exhibits exponential decay in the relationship between clad height and energy density, indicating that at lower ratios of laser power to powder flow rate the height of the clad was more sensitive to changes in laser power or powder flow rate. Exponential decay can be explained by the dependence of shrinkage on the change of Gibbs free energy and enthalpy during phase transformations of a metallic alloy. During a 3D build, start and stop points often occur at the same point on each layer. This can cause the start and stop point inconsistencies of deposited energy density to compound after many layers, creating a part that is out of tolerance and requires an increased amount of post machining. The results in Fig. 4.5 indicate that using a higher energy density during deposition could lead to greater dimensional consistency of a deposited component, as the standard deviation of clad geometry decreased with an increase in energy per unit mass (J/kg). Further discussion and possible tradeoffs are explored in Section 4.5, which summarizes this chapter.
4.4 Dilution of Inconel 718 single clads

The experimentally-determined geometrical dilution of the clad cross-sections increased with increasing cooling rates determined from IR images when they were processed with the same laser power. The cooling rate was determined by calculating the change in temperature between the solidus and liquidus temperatures from IR thermography that monitored the process. Further details on the thermal history of the Inconel 718 single clads are discussed in the next chapter (Section 5.4). Experimentally-determined cooling rates between solidus and liquidus temperatures, \( \dot{T} \), were normalized to energy density from Eqn. 4.2, \( E_D \), or the process parameters of laser power per unit of mass powder flow per second, as seen in:

\[
\hat{T} = \frac{\dot{T}}{(Q/\dot{m})}
\]

where \( \hat{T} \) is the normalized cooling rate between solidus and liquidus temperatures, \( Q \) is the laser power, and \( \dot{m} \) is the powder flow rate.
With increasing normalized cooling rate, $\hat{T}$, clad dilution decreased and reached zero after a threshold of about $0.4 \text{ K} \cdot \text{g} \cdot \text{J}^{-1} \cdot \text{s}^{-1}$, as seen in Fig. 4.6. The clads with zero dilution occurred when the powder flow rate was above the threshold relative to the laser's input energy. For example, at 1,200 W processing power, dilution occurred only when the powder flow rate was less than 209 mg/s, whereas there was dilution for every powder flow rate value with 2,000 W laser power. Figure 4.6 also shows that at relatively high cooling rates and high powder flow, or high normalized cooling $\hat{T}$, there was less mixing of liquid IN718 into the substrate depth. Under these conditions, most of the powder solidified above the substrate surface and exhibited shrinkage at the melt pool’s liquid-solid interface. Both the Marangoni convection within the melt pool and increased laser attenuation due to high powder flow influenced the rapid cooling rates at the surface of the melt pool.

Figure 4.6. Experimentally determined dilution values of single line clads and their corresponding cooling rates determined by surface IR thermography normalized by laser power divided by mass powder flow rate, or $\hat{T}$ as denoted in Eqn. 4.3.
Within the same clad processed with 1,000 W laser power and 56 mg/s powder flow, geometrical dilution varied at the cross sections 10, 25 and 40 mm from the start of the clad, as seen in Fig. 4.7. At each cross section, there was a high dilution ratio of greater than 0.5 and a cooling rate at the surface that was greater than 4,000 K/s. This suggests that most of the relatively low mass powder flow entered the cavity within the substrate and mixed with the substrate at a high amount of energy density, $E_D$, within the liquid-solid interface.

![Micrographs of three cross sectional areas (10, 25, and 40 mm) of a clad deposited with 1,000 W laser power and 56 mg/s mass powder flow deposition.](image)

When the mass powder flow reached 223 mg/s with a laser power of 1,000 W, there was not enough energy density, $E_D$, in the melt pool. An increased powder flow creates a blocking effect that scatters the laser, causing that the flowing powders, not the underlying substrate, absorb the laser energy. This resulted in clads with no depth into the substrate and dilution values of 0, meaning that all resulting IN718 clad material was above the substrate surface, as seen in Fig. 4.8. Reducing the blocking powder flow, increasing laser power, or decreasing scan speed could
increase dilution. The solidification cooling rates calculated from the change in temperature between the solidus and liquidus temperatures from IR thermography of clads processed with 1,000 W laser processing power and 223 mg/s and 453 mg/s dropped dramatically from about 2100 K/s to about 1,200 K/s across the clad. This is because of the increased mass and lower surface to volume ratio of the resulting build, taking longer time to cool. In addition, the surface tension resulting from Marangoni forces at the substrate were not substantial enough for such low energy densities, especially with a powder flow of 453 mg/s and laser power of 1,000 W, as seen on the right side of Fig. 4.8 where there was only partial adhesion of the clad onto the substrate and a potential crack forming at the clad-substrate interface.

![Image](image_url)

Figure 4.8. Clads at the 10 mm cross section, with left clad resulting from 1,000 W laser power and 223 mg/s powder flow and the right clad resulting from 1,000 W laser power and 453 mg/s powder flow. The resulting dilution values are 0 as there is no depth into the substrate.

The above-stated lack of dilution suggests that the combined effect of the blocking powder flow and laser power did not deliver enough energy to extend the bottom boundary of the melt pool into the substrate. Figure 4.6 uses only laser power and powder mass flow to calculate energy density, $E_D$, as the laser scan speed remains constant in all experiments. Figure 4.6 normalizes the cooling rate to the laser power and powder mass flow without taking the scan speed into account. Though
the influence of laser scan speed was not investigated in this work, this process parameter influences vaporization or melting of the substrate, powder mass accumulation at any given area, growth rate at the liquid-solid interface, and the overall cooling behavior of the clad. An energy density metric can incorporate the laser scan speed parameter by dividing the laser power by speed and resulting in Joules per millimeter. However, the emphasis of this work was on the influence of powder mass flow, which differentiates the DED process from powder bed AM processes.

4.5 Summary

This chapter aims to capture the global laser-matter interactions during the DED process by evaluating the resulting height and dilution of single clads. These structural characteristics indicate how much of the in-flight powder particles melt and fuse together relative to the underlying substrate material or layer. Clad height and dilution were compared to process parameters and cooling rates determined by in-situ IR thermography. Further details on the thermal histories, microstructures and mechanical properties of the Inconel 718 single clads are provided in subsequent chapters. In addition, these experimental results were compared to results from a thermal fluid dynamic CtFD model, which is explained further in Chapter 5 on thermal history. A comparison of two thermal models with experimental results is also given in Chapter 5.

The results in this chapter indicate that using a higher energy per unit mass, or energy density denoted by $E_D$, during deposition could lead to greater dimensional consistency of a deposited component, as the standard deviation of clad geometry decreases with an increase in energy per unit mass (J/kg). Higher energy densities also correspond to greater cooling rates, as there is less
mass to cool. In all experiments, the laser scan speed remained consistent, with an industrial standard (16.7 mm/s) speed used. Changes in scan speed, and therefore changes in cooling rate, were not captured in its influence on the energy per unit mass during deposition metric.
Chapter 5: Thermal History of DED-Processed Materials

This chapter focuses on the thermal cycling and history of additively-built materials. In DED, the thermal history is unique for rapid heating and cooling within a localized area. Process parameters such as laser power, scan speed, and powder mass flow determine the rate of moving liquid-solid interface of the melt pool (Section 5.1). The state-of-the-art of both experimental (Section 5.2.1) and computational (Section 5.2.2) methods in capturing the thermal history of a localized area of a build, are discussed. The state of the art in experimental methods for in-situ thermal monitoring has been discussed in Section 1.3.1. The materials discussed in this section are Inconel 718 and Ti-6Al-4V, as seen in Fig. 5.1. A discussion on the computational methods used in this work is provided in Section 5.3 A comparison of the methods used to determine thermal histories for Inconel 718 single clads and thin walls are detailed in Sections 5.4 to 5.5. A discussion and comparison of the methods used to determine thermal histories for Ti-6Al-4V are detailed in Section 5.6. Section 5.7 provides a summary of thermal histories during the DED process and opportunities for future work. This section will also provide an overview on how thermal processing influence microstructure formation.
5.1 Influence of process parameters on thermal history

Several physics-based models have investigated the influence of processing conditions, including the flow of powder particles, on the thermal history of the melt pool. In a study with a three-dimensional heat transfer numerical model of the DED process (Manvatkar, De et al. 2015), laser power, scan speed, laser beam diameter, and mass flow rate were used as inputs to calculate the amount of energy of the laser beam transferred to in-flight particles and their temperature rise. An increase in laser scanning speed increased the growth rate at the liquid-solid interface of the melt pool, therefore increasing the cooling rate between the solidus and liquidus temperatures. There was an observed melt pool growth with each layer of a 316 SS thin wall added. However, cooling rates decreased with increasing layers. For example, the cooling rates during solidification ranged from 4,500 to 6,000 K/s in the second layer of a component, whereas the cooling rates ranged from 1,000 to 1,500 K/s in the ninth layer (Manvatkar, De et al. 2015). The temperature history from the model was also compared to experiments, showing a steady increase in peak temperature in the melt pool with an increase in laser power.
A model captured the melt pool geometry at quasi-steady state during laser directed metal deposition, which is another term for the DED process, by coupling heat and mass flow (Pinkerton and Li 2004). Their model balanced the energy conducted through the liquid-solid interface and the absorbed laser energy, using empirical constants for varying materials. Their study modeled the build with 316L SS processed with a CO₂ laser beam. The geometry of the melt pool was assumed to be an ellipse with major and minor axes that corresponded to its length and width. With increasing laser scan speed, the melt pool geometry increased in its aspect ratio (length/width), with greater rates of increase with an increase in laser power. They investigated the melt pool geometry from the model for a thin wall geometry and compared to the solidified width and height of the walls from experiments. They found substantial agreement between the model and experimental results, observing that increasing laser power led to an increase in layer width and increasing powder mass flux led to increasing layer height (Pinkerton and Li 2004). However, this model cannot be generalized to varying powder flow rates, as powder assimilation efficiency into the melt pool and laser attenuation are kept constant. In addition, the model does not consider the influence of underlying heated layers on the melt pool geometry.

A 2D Rosenthal model determined process maps that evaluated the influence of laser power and scan speed on the cooling rate of the melt pool’s liquid-solid interface (Bontha, Klingbeil et al. 2009). The model investigated DED-processed thin wall builds of Ti-6Al-4V and was compared to a 2D nonlinear thermal FEM, which yielded similar results. They found that with increasing laser power, the thermal gradient at the liquid-solid interface, \( G \), increased and approached the
mixed columnar and equiaxed microstructure region. With an increase in laser scan speed, both the thermal gradient and growth rate, $R$, increased, leading to more columnar structures, as seen in Fig. 5.2 (Bontha, Klingbeil et al. 2009).

Finite element three-dimensional thermal kinetic modeling of the DED process provided the temperature histories into a microstructural model that determined the various $\alpha$ phase fractions in DED-processed Ti-6Al-4V (Baykasoglu, Akyildiz et al. 2018). Trends in the $\alpha$ colony phases were observed to increase with layers until the last layer, where there was an increase in cooling rate and decrease in the $\alpha$ colony phase. Simulation results of $\alpha$ lathe widths were compared with substantial agreement to experimental results, though there is a limitation in determining the phase fractions from experimental result. In addition, the model did not provide any indication into the localized distributions of the various phase transformations that occur in the DED-processed part.
A study implemented computational fluid dynamics to predict for the grain structures and sizes of Inconel 718 (Chandra and Rao 2017). Simulation results were calibrated and validated with experimental results from literature. Observed trends include an increase of equiaxed dendritic grain structures with increasing laser power and preheating of the substrate. Similar to the previous study, the temperature analyses that predict for grain structures are macro-scale to an entire melt pool and does not include phase transformations of small-scale precipitation. In addition, the computational fluid dynamics model is material dependent and its compatibility with other materials was not presented.

5.2 State-of-the-art methods to capture thermal history

5.2.1 In-situ experimental thermal monitoring

Thermal monitoring of laser-matter interactions includes infrared (IR) thermography (Hu and Kovacevic 2003), (Price, Cooper et al. 2012), (Moylan, Whitenton et al. 2014), (Rodriguez, Mireles et al. 2015), two-wave pyrometers (Islam, Purtonen et al. 2013), (Kriczky, Irwin et al. 2015), (Ma, Franco et al. 2017), (Khazadeh, Chowdhury et al. 2018), calorimetry (Trapp, Rubenchik et al. 2017), (Matthews, Trapp et al. 2018), (Lia, Park et al. 2017) and thermocouples (Hu, Chen et al. 2000), (Kelly 2002), (Heigel, Michaleris et al. 2015).

IR cameras capture the cooling rates, solidification rates, and thermal gradients at localized points of an additive manufactured part. However, limitations of IR camera monitoring include the low spatial and temporal resolutions. Without a close-up lens, IR cameras usually provide pixel sizes of about 100 µm, which is not high enough of a resolution to capture the thermal history of sub-
grain features, which are typically in the order of a hundreds of microns to sub-microns, as grain sizes of additive manufactured parts can reach hundreds of microns in size (DebRoy, Wei et al. 2017). Close-up lenses can provide pixel sizes of about 10 to 20 µm, but with a compromise to the field of view of about 50 by 25 mm, as opposed to the original field of view of about 500 by 250 mm. However, the use of close-up lens would be able to capture the thermal history of some of the sub-grain features. The temporal resolution is about 50 frame rate per second (fps), i.e., a resolution of 0.02 seconds. Depending on the material, this frame rate may not fully capture the change of temperature between the solidus and liquidus temperatures of a material, which typically takes anytime in the order of 0.5 sec to $10^{-4}$ sec with a corresponding rate of $10^2$ to $10^6$ K/sec. Furthermore, IR cameras do not provide absolute temperature and rely on the calibration of the emissivity value. However, the emissivity changes as a material undergoes vaporization, melting, and rapid cooling, and the emissivity value changes with the surface finish. Ongoing work aims to provide accurate temperature readings by calibrating the changing emissivity values with other in-situ processing methods that capture phase change (Boone, Zhu et al. 2018), (Cheng, Lydon et al. 2018).

Two-wave pyrometers are used as melt pool sensors that capture the aerial view and absolute temperature of the melt pool region during the process. Work with two-wave pyrometers in additive manufacturing includes control of the thermal history to control the functional response of additively manufactured shape memory alloys (Ma, Franco et al. 2017). In this work, the field of view was 26 by 20 mm, or 1,300 by 1,000 pixels, with a pixel size of 20 µm. The frame rate was 100 Hz and captured the melt pool with a temperature calibration of more than 3,400 ºC. Other
ongoing work with two-wave pyrometer monitoring, includes tracking the change in melt pool geometry to predict for powder spattering and porosity formation (Khanzadeh, Chowdhury et al. 2018). The resolution of the field of view was 752 by 480 pixels, with a pixel size of 6.45 µm. The frame rate was 6.4 Hz, as the average melt pool geometry at a specific localized location of the build, and not the cooling rate, was vital for prediction. The temperature range of the pyrometer was from 1,000 to 2,500 °C, allowing for substantial boundary detection of the liquid-solid interface of the melt pool, considering the size of the melt pool reached about 2 mm in the major axis.

Thermocouples in the additive manufacturing process are usually embedded in the substrate plate prior to build and capture the thermal history of the substrate (Heigel, Michaleris et al. 2015). This thermal history is extrapolated to the thermal history of the actual build by assuming boundary conditions of conduction, radiation, and convection of the melt pool. In a study of directed energy deposited Ti-6Al-4V thin walls, a thermo-mechanical model was validated by the thermocouple readings at various 1 by 1 mm areas of the substrate (Heigel, Michaleris et al. 2015). The thermocouples had a temperature uncertainty of 2.2 °C and had a maximum temperature reading of 482 °C, with an unknown temporal resolution. In a recent study (Trapp, Rubenchik et al. 2017) to capture real-time absorptivity in powder-bed processed 316L SS, thermocouples with 76 µm diameter wires were used within the substrate. The data collection rate was 14 Hz. Though thermocouples provide absolute temperature, they are limited by their maximum temperature capability, data collection rate, and spatial resolution. Persistent challenges in thermal monitoring
include temporal resolution, spatial resolution, capturing absolute temperature, and resolving for multiple scales of the process.

5.2.2 Thermal modeling

To assess the highly transient manufacturing process, a few models have been developed. There are two major types of models: the thermal model and the thermal-fluid flow model. Gao et al. (Gao, Zhao et al. 2016) developed a 3D thermal model using the finite element method (FEM) to calculate the thermal gradient and the solidification rate in the liquid-solid interface of an iron-based alloy mixed with titanium powders in laser cladding. The model incorporated the tool path and component geometry as inputs. The resulting cooling rates at different areas of a clad were compared to experimentally-determined solidification microstructures. Using a Fourier heat conduction model, the effects of preheating a 1030 carbon steel substrate on the cooling rates of deposited Fe–TiC composite particles in laser cladding were investigated in a thermal model (Emamian, Corbin et al. 2012). Microstructure analysis of Ti-C particles and microhardness testing were compared to the temperature distribution of the clad. However, fluid flow in the melt pool was not considered in these numerical models, which does not take melt pool convection into account and could drastically underestimate the cooling at the liquid-solid interface.

Yan et al. integrated the thermal-fluid flow process models, grain growth models and micromechanics models to investigate several process-structure-property relationships for powder bed additive manufacturing (Yan, Lin et al. 2018). Several thermo-fluid flow models for the DED process have also been developed. Qi et al. (Qi, Mazumder et al. 2006) built a three-dimensional
thermal fluid model to calculate the temperature distribution and liquid metal flow in the melt pool during the direct metal deposition (DMD) process of low-carbon steel powders. The trends in melt pool length, melt pool width, and clad height with increasing laser power were the same as those in experiments. He et al. (He and Mazumder 2007) captured powder deposition of ANSI H13 tool steel powders onto a low-carbon steel substrate in DMD by using a computationally intensive approach, the Level-Set method, to capture the melt pool interface. Trends in clad dilution with increasing laser power and scanning speed were like those in experiments. Kumar et al. (Kumar and Roy 2009) derived and solved sets of dimensionless conservation equations to predict the melt pool dimensions, temperature distribution and cooling rates during the laser cladding of iron powders. They found that the influence of Marangoni–Benard convection dominates the simulation results. Shin’s group (Wen and Shin 2010) developed a series of models to investigate the transport phenomena in off-axis high power diode laser cladding of stellite powder. The experimental and predicted values of powder velocity during deposition, clad height, and clad width were compared. Gan et al. (Gan, Yu et al. 2017) proposed a physical-based model with Marangoni convection to predict for the thermal distribution and solute transport in the melt pool during the direct laser deposition of cobalt-based powders onto a steel substrate. The temperature field results from their model provide solidification parameters such as thermal gradient, solidification rate, cooling rate, and morphology factor. These values were compared to the experimental clad profile and microstructures within the clad.
5.3 Computational methods used in this work

5.3.1 GAMMA thermal model

Process parameters are inputs into an in-house simulation code developed by a former student (Smith, Xiong et al. 2016). The code is named GAMMA and models the moving melt pool during a DED build. One can output the temperature history from any probed point within a built component. The GAMMA code demonstrates the evolution of the melt pool during a build. Overall, GAMMA is a thermodynamically consistent prediction of melt pool solidification that serves as a qualitative output of an in-house finite element software that takes process parameters, tool path, component geometry and the thermodynamic properties as inputs.

Deposited groups of powders onto the melt pool are approximated as solid clusters, where each element is approximated as a solid cluster and birthed during deposition with the laser input. The laser input is approximated by a moving boundary flux, represented by the element’s degrees of freedom in the solution. The major benefit of the in-house code as opposed to a commercial software like ABAQUS is the drastic reduction (about 1000x) in computation time for a full part simulation. This is achieved through optimization of time-dependent boundary conditions and element birth routines based on the experimental tool path. A single-processor simulation of a build could take up to about six days for a commercial software, whereas the same simulation would take less than ten minutes for the in-house code (Smith, Xiong et al. 2016).

The thermal model does not consider convective effects that occur due to Marangoni forces, yielding predicted cooling rates that are larger than what is seen in experiments or in computational
flow dynamics simulations. Previous studies show that these Marangoni convective cooling forces play a significant role in the cooling process for applications incorporating extreme thermal gradients such as welding (Ribic, Rai et al. 2008) and additive manufacturing (Francois, Sun et al. 2017). Convective influence in the melt pool can be captured by implementing a reduced order model or by enlarging the conductivity to capture additional cooling. However, the goal of the work presented here is to present a preliminary but general framework that combines experimental and computational approaches to link process, structure and mechanical properties of additively manufacture parts. The resolution of the additional physics and a high-fidelity localized analysis is out of scope of the presented work, though refining the resolution of the methods will be the primary focus of future work.

5.3.2 Computational thermo-fluidic (CtFD) model

To consider the complex fluid mechanism of liquid metal and mass addition phenomena, a self-consistent three-dimensional computational thermo-fluid dynamics (CtFD) model was developed for the DED processing of IN718 clads by Gan (Gan, Yu et al. 2017), (Gan, Yu et al. 2017). This section provides an overview of model formulation. The non-isothermal Navier-Stokes (N-S) equations, including mass, momentum and enthalpy conservation equations, were solved. A surface profile calculation based on minimizing free surface energy is used to deal with the transient change of top surface due to mass addition. The temperature distribution and liquid metal flow in the melt pool as well as clad geometry and composition dilution were predicted. The resulting temperature profile provided the solidification parameters, such as the cooling rate, that were evaluated to predict the dendrite arm spacing and microhardness distribution of a clad. The
model's assumptions follow those in the previous study (Gan, Liu et al. 2017), with the governing equations:

\[
\frac{\partial \rho}{\partial t} + \frac{\partial (\rho u_i)}{\partial x_i} = 0 \tag{5.1}
\]

\[
\frac{\partial (\rho u_i)}{\partial t} + \frac{\partial (\rho u_i u_j)}{\partial x_j} = \frac{\partial}{\partial x_j} \left( \mu \left( \frac{\partial u_i}{\partial x_j} + \frac{\partial u_j}{\partial x_i} \right) \right) - \frac{\partial p}{\partial x_i} - K_0 \frac{(1-f_l)^2}{f_l^2 + B} u_i \tag{5.2}
\]

\[
\frac{\partial (\rho h)}{\partial t} + \frac{\partial (\rho u_i h)}{\partial x_i} = \frac{\partial}{\partial x_i} \left( \frac{\kappa}{C_p} \frac{\partial h}{\partial x_i} \right) - \frac{\partial (\rho L f_l)}{\partial t} - \frac{\partial (\rho u_i L f_l)}{\partial x_i} \tag{5.3}
\]

where \( t \) is the time, \( u_i \) is the \( i \)th component of velocity, \( \mu \) is the viscosity, \( p \) is pressure, \( h \) is the enthalpy, which is related to the temperature \( T \), \( C_p \) is the specific heat capacity, \( \rho \) is the density, and \( \kappa \) is the thermal conductivity. The morphology parameter of the porous media is \( K_0 \), which depends on primary dendrite arm spacing (Mukherjee, Wei et al. 2018). The parameter \( B \) is used to avoid dividing by zero, and is \( 10^{-8} \) in this study (Gan, Liu et al. 2017). The latent enthalpy of fusion is \( L \), and \( f_l \) is volume fraction of the liquid phase. The thermal boundary condition at the liquid-gas interface is shown as:

\[
q_{\text{energy}} = \frac{2Q(1-\eta_p)\eta_l}{\pi r_b^2} \exp \left( -\frac{2\left((x-Vt)^2+y^2\right)}{r_b^2} \right) - h_c(T - T_{\infty}) - \sigma_s \varepsilon (T^4 - T_{\text{ref}}^4) \tag{5.4}
\]

where \( Q \) is the laser power, \( \eta_p \) and \( \eta_l \) are the absorptivity of powder flow and base metal, respectively. The laser beam radius is \( r_b \), \( V \) is the scanning speed, \( h_c \) is the convective heat transfer coefficient, \( T_{\infty} \) is the ambient temperature, \( \sigma_s \) is Stefan-Boltzmann constant, and \( \varepsilon \) is emissivity (Gan, Yu et al. 2016). The reference temperature is \( T_{\text{ref}} \). The metal absorptivity \( \eta_l(T) \) is estimated based on the Hagen-Rubens relationship as follows (Lee, Nordin et al. 2014):

\[
\eta_l(T) = \sqrt{8\varepsilon_0 \omega R_e(T)} \tag{5.5}
\]

where \( \varepsilon_0 \) is the permittivity of free space and \( R_e \) is the resistivity for Ni-Fe alloys. The energy absorbed by the laser-powder interaction is formulated as a source term in the enthalpy equation:
\[ S_e = \frac{2\eta_p}{\pi r_b^3} \exp \left( -\frac{2(x-Vt)^2+y^2}{r_b^2} \right) \]  

(5.6)

where \( d \) is the depth of the melt pool and was determined by iteration. Boundary condition at the liquid-gas interface for momentum conservation equations are (Gan, Liu et al. 2017):

\[ F_{L/G} = \gamma n \kappa + \nabla_s T \frac{dy}{dT} \]  

(5.7)

Where \( F_{L/G} \) is the momentum equation at the liquid-gas interface, \( \gamma \) is the surface tension of melt pool, \( n \) is the outward normal of the surface, and \( \kappa \) is the curvature of the surface. Previous work (Gan, Liu et al. 2017) details the simplified equations to capture the dynamic surface profile of the melt pool.

### 5.4 Inconel 718 thermal history of single clads

Both experimental and computational studies have investigated the influence of process parameters in the DED process on the resulting thermal histories of a DED-built component. Comparisons between experiments and model-based studies in this work evaluated the resulting melt pool geometry and cooling rates. This work looks at the influence of fluid dynamics in powder deposition thermal models as well as the capability to predict the thermal histories. Spatial profiles in such models were compared with thermal imaging of laser deposition processing of Ni-based superalloy Inconel 718. Experimentally determined clad dilution and microhardness results of Inconel 718 clads were compared to the cooling rates and dilution values in two thermal models. The GAMMA model provided thermal distribution and history during the process. The computational thermal fluid dynamic (CtFD) model considered the fluidic mechanisms of molten metal during powder deposition and the resulting transient changes of geometry. Based on the computed temperature and velocity distributions in the melt pool, cooling rate at the liquid-solid
interface were evaluated in this Chapter. This information will be further used in Chapter 7 to predict the microhardness distribution and dilution of a single line clad.

5.4.1. Inconel 718 single clads experimental setup

A DMG MORI LaserTec 65 3D, a hybrid additive and subtractive five-axis machine, was used by Jennifer Bennett from DMG Mori and Northwestern University to build Inconel 718 single clads. A 2,500 W, 1020 nm, high-powered continuous wave laser melted deposited IN718 powders onto an AISI 1045 medium carbon steel substrate, as mentioned in Section 4.2.1.

During the deposition process, a digital infrared camera calibrated to 2,100 °C, the FLIR A655sc, captured thermal images of the melt pool from above at a rate of 24 fps, as seen in Section 4.2.1. The camera’s resolution was 640x480 with spectral range from 7.5 to 14.0 µm and a ±2°C accuracy. The camera recorded the infrared radiation emitted by an object and recorded the emission of temperatures ranging from 300°C to 2,000°C. Absolute temperature measurements using an IR camera required knowledge of the emissivity at the measurement point. The emissivity of a surface was its effectiveness in emitting energy as thermal radiation. The emissivity of the evaluated workpiece material was a function of surface condition, temperature, and wavelength of measurement. Since areas of interest in this study were undergoing a phase change from powder to molten to solid track, the emissivity values cannot be accurately determined. An emissivity value of 0.1 was chosen for all measurements. During in-situ thermal monitoring (Bennett, Wolff et al. 2017), the emissivity of the melt pool was not accurately determined as the material underwent phase transitions, resulting in a temperature reading that was not absolute. However,
an emissivity value of 0.3 was used with the IR camera, based on the approximate emissivity of Inconel 718 in its solid phase (Gan, Liu et al. 2017). Using the thermal images, the trends in cooling rate across the clad and over varying process parameters could be determined at each analyzed point on the clad, as described in Chapter 4. Thermal information was extracted from the IR images by thresholding the melt pool pixels and calculating the rate between the liquidus and solidus temperatures (Bennett, Wolff et al. 2017).

Wire electrical discharge machining separated the substrate discs at the cross sections to view clad dilution into the substrate. After polishing the cross sections, dilution of the solidified melt pool was viewed and measured using optical microscopy. The overarching assumption of measuring the dilution of the resulting clad was that the solidified geometry represents the geometry of the melt pool, or the area within the liquid-solid interface during the process.

5.4.2. Inconel 718 single clads thermal model setup

The in-house thermal models developed by collaborators in Section 5.3 were used to evaluate the thermal history of the Inconel 718 single clads. The material properties used for both the GAMMA and CtFD thermo-fluidic dynamic models are shown in Table 5.1, with process parameter inputs listed in Table 5.2. For the GAMMA model, a XY plane view of the computational domain is shown on the right side of Fig. 4.1b in the previous chapter. Radiative and convective cooling due to the environment were also imposed over the build surface. An unstructured hexahedral mesh with 101436 elements was used to discretize the GAMMA model’s domain.
Twenty-five simulation cases of single track laser deposition were numerically simulated using the CtFD model. The laser power values were 1,000 W, 1,250 W, 1,500 W, 1,750 W and 2,000 W. At each level of fixed laser power, the powder flow rate was incremented from 56 mg/s to 453 mg/s in 4 increments for a total of 25 simulation cases as shown in Table 5.2. The computational domain used with the CtFD model was 20 mm by 10 mm by 10 mm, which had grid discretization of 210 by 105 by 105 (total 2,315,250 cells). The simulation time in this study was 1,000 ms with a time step of 0.1 ms. The melt pool achieved steady state at 1,000 ms based on trial calculation. In each time step, the Navier-Stokes (N-S) and enthalpy conservation equations with latent enthalpy of phase change were solved iteratively. Based on the temperature field, a physics-based model provided the melt pool surface profile above the substrate (Gan, Yu et al. 2017).

Table 5.1. Thermo-physical properties of IN718 and AISI 1045 carbon steel substrate.

<table>
<thead>
<tr>
<th>Variables</th>
<th>Symbols</th>
<th>IN718 Value (Hosaeus, Seifter et al. 2001)</th>
<th>AISI 1045 Value (Elmer, Palmer et al. 2004)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Solid density</td>
<td>$\rho_s$ (kg m$^{-3}$)</td>
<td>7,734</td>
<td>7,872</td>
</tr>
<tr>
<td>Liquid density</td>
<td>$\rho_l$ (kg m$^{-3}$)</td>
<td>7,578</td>
<td>7,700</td>
</tr>
<tr>
<td>Solidus temperature</td>
<td>$T_s$ (K)</td>
<td>1,533</td>
<td>1,713</td>
</tr>
<tr>
<td>Liquidus temperature</td>
<td>$T_l$ (K)</td>
<td>1,609</td>
<td>1,768</td>
</tr>
<tr>
<td>Solid specific heat capacity</td>
<td>$C_{ps}$ (J kg$^{-1}$K$^{-1}$)</td>
<td>435</td>
<td>586</td>
</tr>
<tr>
<td>Liquid specific heat capacity</td>
<td>$C_{pl}$ (J kg$^{-1}$K$^{-1}$)</td>
<td>755</td>
<td>746</td>
</tr>
<tr>
<td>Solid thermal conductivity</td>
<td>$\kappa_s$ (W m$^{-1}$K$^{-1}$)</td>
<td>11.4</td>
<td>50.9</td>
</tr>
<tr>
<td>Liquid thermal conductivity</td>
<td>$\kappa_l$ (W m$^{-1}$K$^{-1}$)</td>
<td>31.3</td>
<td>50.9</td>
</tr>
<tr>
<td>Latent heat of fusion</td>
<td>$L$ (kJ kg$^{-1}$)</td>
<td>290</td>
<td>290</td>
</tr>
<tr>
<td>Dynamic viscosity</td>
<td>$\mu$ (Pa s)</td>
<td>5.3$\times$10$^{-3}$</td>
<td>5.3$\times$10$^{-3}$</td>
</tr>
<tr>
<td>Surface tension</td>
<td>$\gamma$ (N m$^{-1}$)</td>
<td>1.8</td>
<td>1.8</td>
</tr>
<tr>
<td>Marangoni coefficient</td>
<td>$\frac{d\gamma}{dT}$ (N m$^{-1}$K$^{-1}$)</td>
<td>-3.7$\times$10$^{-4}$</td>
<td>-4.3$\times$10$^{-4}$</td>
</tr>
</tbody>
</table>
### Table 5.2. Process parameters used in both thermal models.

<table>
<thead>
<tr>
<th>Variables</th>
<th>Symbols</th>
<th>Values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Laser power</td>
<td>$Q$ (W)</td>
<td>1,000, 1,250, 1,500, 1,750, 2,000</td>
</tr>
<tr>
<td>Beam radius</td>
<td>$r_b$ (mm)</td>
<td>1.5</td>
</tr>
<tr>
<td>Powder flow radius</td>
<td>$r_p$ (mm)</td>
<td>3</td>
</tr>
<tr>
<td>Scan speed</td>
<td>$ν$ (mm·s$^{-1}$)</td>
<td>16.7</td>
</tr>
<tr>
<td>Ambient temperature</td>
<td>$T_{\infty}$ (K)</td>
<td>295</td>
</tr>
<tr>
<td>Convection coefficient</td>
<td>$h_c$ (W·m$^{-2}$·K$^{-1}$)</td>
<td>100</td>
</tr>
<tr>
<td>Stefan-Boltzmann constant</td>
<td>$σ_s$ (W·mm$^{-2}$·K$^{-4}$)</td>
<td>$5.67 \times 10^{-14}$</td>
</tr>
<tr>
<td>Emissivity</td>
<td>$ε$</td>
<td>0.3</td>
</tr>
<tr>
<td>Permittivity</td>
<td>$ε_0$ (F·m$^{-1}$)</td>
<td>$8.85 \times 10^{12}$</td>
</tr>
<tr>
<td>Angular frequency</td>
<td>$ω$ (rad·s$^{-1}$)</td>
<td>$1.75 \times 10^{15}$</td>
</tr>
<tr>
<td>Mass flow rate</td>
<td>$\dot{m}$ (mg·s$^{-1}$)</td>
<td>56, 140, 223, 338, 445</td>
</tr>
<tr>
<td>Powder catchment efficiency</td>
<td>$\eta_m$</td>
<td>0.9</td>
</tr>
</tbody>
</table>

### 5.4.3. Inconel 718 single clad experimental cooling rates

The experimental cooling rate between the solidus and liquidus temperatures was determined from IR thermal images at the surface of each clad. Though the IR images did not provide absolute temperature, the cooling rate trend was assumed to reflect the cooling within the melt pool. Currently, experimental determination of cooling rate is restricted to only surface measurements and at coarse frame rates.

Thermal history was investigated via IR data at specific points of a clad: at 10, 25 and 40 mm from the start of the 50 mm long clad. Figure 5.3 demonstrates the influence of the powder flow and laser power parameters on the thermal history of each clad. Each subplot shows three curves, corresponding to the thermal history of the start, mid, and ends points from left to right. The dotted lines illustrate the solidus and liquidus temperatures of IN718 at 1,260°C and 1,336°C, respectively. These temperatures determined the solidification region of each clad and its resulting...
structure and properties. The cooling rate was calculated during this solidification region for the purposes of this study.

Figure 5.3 reveals that an increase of powder flow rate corresponded with an increase of thermal conductivity throughout the clad resulting in reheating of the clad reaching greater temperatures and slower cooling than those with less powder flow rates. Due to the thermal threshold of the IR sensor, the maximum temperature of several clads processed with 2,000 W could not be captured because they exceeded the threshold of about 2,100 °C. Figure 5.4 illustrates the thermal histories extracted from the IR images of two clads with extreme processing conditions.
As seen in Figs. 5.5 and 5.6, greater cooling rates occurred at lower powder flow rates and at lower laser powers. A larger difference between maximum temperature and solidification cooling rate at the sampled points on a clad was also observed at the lower end of powder flow rates, indicating that as power flow rate increased, there was slower cooling. Because microstructure is dependent on cooling rate, these results suggest that a more uniform clad microstructure would be created with a higher powder flow rate. The trends of cooling throughout the clad of the 1,000 W and 56 mg/s differ from the other clads processed with 1,000 W in that the peak cooling rate was found closer to the start of the clad instead of the end of the clad. This is possibly due the non-uniform and fluctuating nature of powder flow from a coaxial nozzle or because the clad processed with 56 mg/s was the first clad built on the substrate without any preheating of the underlying substrate.
Other than the aforementioned case, the end of the clads typically featured the most rapid cooling rate. A decrease in surface area that radiates to the chamber atmosphere explains the decreased cooling rate at the clad midpoint. Although the points closer to the start and the end points of the clads had similar surface areas, the 40 mm point close to the end of a clad exhibited a higher cooling rate because the laser turned off immediately after deposition and no longer conducted heat to that position. Latent heat and thermal conductivity explain the decreasing cooling rate, with increasing radiation contributing to high cooling rates. However, volume change and energy input need to be considered to explain the cooling differences between the positions close to the start and end of clads.

Figure 5.6 illustrates the change in cooling rate for each localized area of a clad corresponding with the area’s energy density per unit mass, $E_D$, which is dependent on laser processing power and powder flow rate. Not all powders from the coaxial nozzle melted into the laser-induced melt.
pool, which was apparent by un-melted powder particles on the surface of the clad and substrate. The powder usage percentage was unknown, though the powder flow distribution diameter was set to be the same as the laser beam diameter at 3 mm. The energy density per unit mass, $E_D$, does not consider the powder usage percentage but assumes that the overall powder flow rate influences energy density of the melt pool. Logarithmic behavior dominated the dependence of cooling on energy per unit mass, $E_D$, of deposited material. With lower powder flow rates, greater surface to volume ratio of the heated powders led to a greater energy per unit mass and a higher cooling rate. Decreasing powder flow rates also led to an increase in variation of energy densities and cooling rates due to a greater proportion of the powder absorbing laser energy and cooling within a diluted clad. As powder flow rates increased, cooling rates decreased, which may be a result of optimal, or minimal, dilution relative to the steel substrate. The trends in Fig. 5.6 show that at an individual powder flow rate, there was a linear relationship between IR thermography-determined cooling and energy density from process parameters, though the logarithmic relationship was observed for the entire range of powder flow rates.
As the solidification cooling rate decreased, the variation of the Z-height within a clad increased, as shown in Fig. 5.7. Figure 5.7 suggests that for localized areas of clads processed with large powder flow rates, dimensional consistency was highly dependent on slight changes of the cooling rate throughout the clad due to shrinkage. This finding aligns with observations of IN718 and other metallic alloys that rapid solidification resulted in unique phase transformations that led to eutectic phases, minimal changes in the Gibbs free energy (Griffith, Schlienger et al. 1999), minimal changes in volume and therefore a monotonic relationship of a consistent build with cooling rate.
Capturing the powder loss during the process would lead to more accurate relationships between powder flow rate, energy density, cooling rate, and clad height. To capture the convection forces within the clad and the moving melt pool interface, *in-situ* monitoring techniques that penetrate through the thickness of the material would be required. However, such techniques are time consuming and costly whereas parallelized computational models can provide online monitoring information on thermal histories and predict properties, which will be shown next.

5.4.4. *Simulated cooling rate of Inconel 718 single clad from GAMMA*

The GAMMA thermal model is an inexpensive tool to immediately capture the thermal history of a toolpath without considering the fluid convection in the melt pool. Mass powder flow rate was not an input into the simulation, with the birthing element size set at 500 µm above the substrate surface for clads of every powder flow rate. The cross section of the melt pool could be captured
at any time. Stephen Lin, a PhD candidate in the Liu/Wagner group, ran the simulations with the appropriate mesh size and time step. There was no solidification cooling within the melt pool except for the solidification front, or the liquid-solid interface at the tail of the moving melt pool. Figure 5.8 shows the thermal history of the single clad built with 56 mg/s powder mass flow at 1000 W and 2000 W. The dotted lines indicate the solidus and liquidus temperatures of Inconel 718. Each temperature profile indicates a height or depth from the surface, with Z=0 indicating the disc substrate surface. Positive Z values probe the clad temperatures that are above the substrate surface, with negative Z values below the surface.

Figure 5.8. Thermal histories from the GAMMA thermal model in the 1000 W and 2000 W processed discs. The depth into the substrate starts at -0.5 mm to denote the distance relative to the surface of the substrate (Z=0.0), with 0.5 mm above the surface of the substrate denoting the surface of the clad.

Unlike the IR-determined temperature profiles that were only of the clad surfaces, the resulting cooling rates from the GAMMA model exhibited variation with depth within the same clad. The resulting GAMMA-determined clads exhibited cooling rates that decreased with increasing laser power and gradient of cooling that decreased with time, as seen in Fig. 5.9. The cooling rate values
and trends at the surface of the clads were comparable to the cooling rate values and trends determined by IR thermography and what is known in literature. Due to the lack of fluidic convection in the melt pool and the lack of the capability to include powder flow rate, the GAMMA model did not capture the change in cooling rates with increasing powder flow rates.

The cross section of the clads processed by GAMMA had the same melt pool geometry at every time step for a given disc processed with the same laser power. As seen in Fig. 5.10, the depth of the melt pool at every time step of the 1,000 W processed disc was about 700 µm with a dilution ratio of 0.58. The depth of the melt pool at every time step of the 2,000 W processed disc was about 1,400 µm with a dilution ratio of 0.74. As compared to single clads processed with 56 mg/s and 1,000 W and 2,000 W, respectively, the predicted dilution ratios were comparable, with the solid-liquid interfaces of the melt pools also emulating the shape of the experimental solidified
clads. The experimental clad depths and dilution values can be used to further improve the GAMMA model by calibrating and validating the boundaries of the melt pool during simulation.

![Comparison of experimental single line clads processed with 56 mg/s and a) 1,000 W with a resulting dilution depth of 340 µm and a dilution ratio of 0.69 with b) GAMMA-determined single clad cross section with a 1,000 W laser input with a resulting depth of 700 µm and a dilution ratio of 0.58. c) Experimental clad processed with 56 mg/s and 2,000 W with a resulting dilution depth of 775 µm and a dilution ratio of 0.86 compared to d) GAMMA-determined single clad cross section with a 2,000 W laser input with a resulting depth of 1,400 µm and a dilution ratio of 0.74.]

Though the simulated results from GAMMA from Fig. 5.10 captured the influence of increasing laser power on the dilution ratio of the clad, the clad depths under the substrate did not match the experimental results. A possible reason for this is the lack of convection in the melt pool in the GAMMA model. In addition, the absorptivity of the laser into the substrate was possibly overestimated in the GAMMA model, whereas the powder flow during the experiments caused the laser absorptivity into the substrate to fluctuate and decrease, resulting in less depth penetration of the laser into the underlying substrate.
5.4.5. *Simulated cooling rate of Inconel 718 single clad from the CtFD model*

By considering the dominant fluid mechanisms of liquid metal in the melt pool, more realistic transport phenomena and solidification parameters could be obtained and evaluated. The results of the thermo-fluid dynamics model demonstrated the solidification cooling behavior within the melt pool and its liquid-solid interface. The proposed thermo-fluidic dynamic model, the CtFD model, determined the temperature gradient ($G$) and the growth rate ($R$) at the pool, which drive solidification. These thermal parameters influence the microstructure at the liquid-solid interface. The temperature gradient normal to the liquid-solid front is $G$ and $R$ is the liquid-solid front velocity, as detailed in (Gan, Liu et al. 2017). The cooling rate, $\dot{T}$, which can also be defined as $G \times R$, affects the grain and dendrite size and therefore the properties. For example, lower cooling rates lead to coarser grain sizes.

The comparison of cooling rates determined from IR thermography at the clad surface during experiments with simulation-determined cooling rates at the liquid-solid interface is shown in Fig. 5.11. As shown in Fig. 5.11, the increase of laser energy input led to a decrease of cooling rate. Higher laser power led to a larger melt pool and lower temperature gradient, which resulted in lower cooling rate at the liquid-solid interface of the melt pool. As the liquid metal convection was considered in the numerical model, the strong Marangoni convection in the melt pool significantly decreased the cooling rate at the liquid-solid interface.
The laser absorptivity of in-flight powder and of the underlying substrate were crucial to determine the temperature and velocity distributions in the model-determined melt pool. However, the absorptivity of in-flight powder flow was difficult to obtain from experimental measurements and can be affected by many factors. These factors include shielding gas flow, carrier gas flow, powder flow distribution, laser energy distribution within the beam, and the structure of powder feeding device. In this work, the absorptivity was calibrated by evaluating the experimental cross section geometries of the clad at varying process parameters. The absorptivity of in-flight powder substantially increased with the increase in powder mass flow rate from 0.10 to 0.39. This implies that a greater amount of laser energy was absorbed by the in-flight powder when the powder mass flow increased due to the block of powder flow above the melt pool. The increase of laser power also caused a slight increase in the absorptivity of the in-flight powder.
The comparison of cross section geometries between simulation and experimental resulted with calibrated absorptivity parameters are shown in Fig. 5.12. The calculated melt pool geometry with the temperature-dependent absorptivity at both laser powers of 1,000 W and 2,000 W agreed well with the experimental cross section results, implying that the proposed numerical model could be beneficial in capturing the overall interaction between the laser and the in-flight powder. This aids in the understanding and designing the multi-physics processes that occur in DED. The deviation between numerical and experimental results can possibly be ascribed to the assumptions of the temperature-independent thermal properties and Gaussian laser flux distribution.

Figure 5.12. Comparison of cross section geometry between simulation and experimental results at various laser power and powder mass flow rates.
Figure 5.13 shows a comparison of dilution ratios with energy density from both experiments and the CtFD simulation. There was no dilution of clads into the steel substrate when the mass powder flow rate was 256 mg/s or greater with a 1,000 W laser power. This reveals that no dilution occurred at energy densities, $E_D$, of less than $4.5 \times 10^6$ J/kg. An increased powder flow rate attenuated the laser, leading to in-flight particles absorbing the laser energy. Reducing the blocking powder flow or increasing laser power can increase dilution. With a high powder flow rate of 453 mg/s and low laser power of 1,000 W, there was only partial adhesion of the clad onto the substrate and a potential crack forming at the clad-substrate interface. With an increase in energy density, $E_D$, at the melt pool, dilution into the substrate increased and with greater efficiency of powder melting as seen in Fig. 5.13. The temperature distributions determined by the thermal-fluid dynamic CtFD model provided the dilution ratios from simulations. When the energy density input was less than the critical energy density, $4.5 \times 10^6$ J/kg, there was no dilution. When the energy density was greater than the critical value, dilution increased logarithmically.

![Figure 5.13. Comparison of experimentally and model-determined clad dilution values with energy density, $E_D$.](image)
5.5 Influence of dwell time on the thermal history of Inconel 718 thin walls

To study the influence of dwell time on thermal history, two Inconel 718 thin walls were built by Jennifer Bennett with the DMG Mori LaserTec 65 3D used for the Inconel 718 single clads study in Section 5.4. The dwell time was the amount of time during which the laser beam was turned off between each layer to allow for cooling. The dwell times for the two thin walls were zero and 60 seconds. The thin walls were built with 1,800 W of laser power, a 3 mm beam spot size, and a 6.7 mm/s laser scan speed. The powders were between 50 and 150 µm in diameter and were deposited with a 300 mg/s flow rate. The shield gas from the coaxial nozzle was argon and had a gas flow rate of 0.12 L/s. The walls were 120 layers tall with 500 µm of layer thickness. Figure 5.14 shows the schematic of the resulting thin wall build.

![Figure 5.14. IR camera image of a thin wall after the DED process.](image)

The FLIR IR camera from Section 5.3 was used to monitor the thermal history at a pixel level for each thin wall. The pixel size was 100 µm, with a 640 by 480 pixel resolution. The camera was calibrated up to 2,100 °C with a 50 fps frame rate. As mentioned earlier, the limitation of the IR
camera was that emissivity was assumed to be constant, therefore not providing absolute temperature, especially during phase changes. The thermal history at any pixel could be probed from the IR camera images, providing a means to evaluate the trend in cooling at a localized area or line of pixels. For the thin walls, there were about three to four pixels per layer height.

By evaluating a cross section of both thin walls near the top edge of the build, a comparison of thermal histories, solidification cooling rate, and thermal gradient could be made. For example, for the no dwell thin wall build, the top pixel of the cross section only exhibited one melting cycle (through the liquidus and solidus temperatures) while the 36th pixel below the top of the wall (10th layer from the top) exhibited eight re-melting cycles, as subsequent layers above led to heating of the 10th layer past the melting point, as seen in Fig. 5.15.

![Figure 5.15. Thermal histories taken from IR thermography at the top pixel and 36th pixel from the top for the no dwell IN718 thin wall.](image-url)
The solidification cooling rates, or the rate of cooling between the solidus and liquidus temperatures, were calculated based on the thermal histories of each pixel. These rates were measured at the last re-melting cycle for both the no dwell and 60 second dwell thin walls. Figure 5.16 shows the change in solidification cooling for the no dwell thin wall. The peaks in solidification cooling indicated the pixel at the interface of the layer, as there were three to four pixels per layer. As the probed point moved farther away from the top of the build, the localized area underwent a greater number of re-melting cycles, as seen in the thermal history inset figure in Fig. 5.16. Because the solidification cooling rate was measured at the last cycle, the slope of the rate decreased with more cycles, as the thermal gradient also decreased.

![Figure 5.16. Trend in solidification cooling rate from IR thermography in the no dwell thin wall.](image)

In contrast, the trend in solidification cooling rate for the 60 second dwell wall showed an increase while probing away from the top of the thin wall, as seen in Fig. 5.17. In addition, the solidification cooling rate values were orders of magnitude greater than those in the no dwell thin wall. Like the calculated solidification cooling rates from the no dwell wall, there were peaks in the cooling rates...
at the pixels where the layer interface was located for the 60 second dwell wall. The values of the peaks of the top four layers ranged from 500 to 5,000 K/s for the 60 second dwell thin wall, compared to the cooling rates of 40 to 140 K/s at the peaks of the no dwell thin wall. The change in trend and increase in values were because the dwell time allowed each layer to cool thoroughly before the build of the next layer. Thus, only the immediate underlying layer, and not any of the other underlying layers, underwent an additional re-melting cycle. With the deposition of each layer, the initial temperature of the underlying layer increased with more layers. At the top layer, the wall was slightly hotter than it was at the beginning of the 2nd layer from the top. Because of this, the resulting heating and cooling times decreased from those in underlying layers.

The change in thermal gradient in the top three layers was also evaluated for both walls. As seen in Fig. 5.18, the thermal gradient in the no dwell thin wall decreased while going down the pixels, corresponding to its trends in solidification cooling rates. The greatest thermal gradient was at the top pixel because of the gradient between the ambient temperature and the underlying hot thin
In the 60 second dwell thin wall, the thermal gradient vectors mostly occurred at alternating layers, with the increasing thermal gradients farther away from the top surface of the thin wall.

![Diagram of thermal gradients](image)

**Figure 5.18.** Trends in thermal gradient for both the no dwell and 60 second thin walls.

For both thin walls, the direction of the thermal gradient followed the laser scanning direction. The peaks in thermal gradient occurred at the bottom of each layer for the no dwell thin wall and at the top of each layer for the 60 second dwell thin wall. Introducing cooling of the underlying layers with dwell time also introduced a large difference in gradients within a layer, opposed to the gradual change in thermal gradients within a layer for the no dwell thin wall.

Later sections will discuss the microstructural and mechanical behavior changes with dwell time in the two thin walls. The ability to isolate and quantify the influence of thermal histories on the resulting component’s properties will allow for improved process control for desired material properties and the possibility to create new materials with unique phase transformations and bonding mechanisms.
5.6 Ti-6Al-4V thermal history

Thermal history of AM-processed Ti-6Al-4V include probing thermal histories from a GAMMA simulation at localized areas for an industrial-scale cubic component. Simulated cooling rates were calculated from varying orientations and locations in the components. These cooling rates were compared with structures observed experimentally, with further discussion on the porosity and mechanical behavior in Chapters 7 and 8, respectively. During micro-scale DED processing, cooling rates were calculated from high-speed X-ray images and showed the influence of mass addition into the melt pool on cooling behavior. The resulting porosity structures from the micro-scale DED process will be discussed in Chapter 7.

5.6.1. Ti-6Al-4V cubic component GAMMA cooling rates

This section aims to propose a framework to demonstrate the influence of the solidification cooling rate on porosity anisotropy (Chapter 6) and strength (Chapter 8). The proposed framework (Wolff, Lin et al. 2017) coupled thermal histories from computational simulations with experimental results to predict localized porosity and mechanical properties, aiming to establish the relationship between cooling rate and porosity characteristics. This section discusses how the cooling rates at localized points were determined.

A Laser Engineered Net Shaping ® system in the Quad City Manufacturing Laboratory in Rock Island, IL built three Ti-6Al-4V 40 mm by 40 mm by 40 mm cubic components. The DED process utilized a 1 kW Ytterbium continuous wave (CW), Gaussian profile, fiber optic laser coupled with
inert shield gas in an enclosed environment and four-axis capability. Four coaxial nozzles surrounded the laser beam and deposited Ti-6Al-4V Grade 5 powders with a size distribution from 45 to 150 µm in diameter.

Each Ti-6Al-4V component was built separately on an unheated Ti-6Al-4V substrate plate within the DED enclosure with the process parameters outlined in Table 5.3. The tool path alternated between a laser on the ON mode during a scan at 0 and 180 degrees between clads within each layer and on the OFF mode while moving the laser optic in the build direction, or the positive Z direction by the layer thickness.

<table>
<thead>
<tr>
<th>Output laser power (W)</th>
<th>Scan speed (mm/s)</th>
<th>Laser beam diameter (mm)</th>
<th>Powder mass flow rate (mg/s)</th>
<th>Hatch spacing (mm)</th>
<th>Layer thickness (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>710</td>
<td>10</td>
<td>1.83</td>
<td>120</td>
<td>1.25</td>
<td>0.95</td>
</tr>
<tr>
<td>800</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>940</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Tensile specimens were machined by wire-EDM from each cubic component in three different orientations as defined in Fig. 5.19 and Table 5.4. The dimensions of tensile specimens followed ASTM E8 standards (Officials and Materials 2004) with a gauge length of 10.0 mm, gauge width of 2.5 mm, and thickness of 1.2 mm. Tensile specimens with orientation “A” were machined from the XZ plane at various depths in the Y direction. These “A” specimens were subject to a uniaxial tensile direction, or the principal load axis, in the build direction, the Z axis. Tensile specimens with orientation “B” were machined from the XY plane with a principal load axis in the X axis.
Tensile specimens with orientation “C” were machined through the X direction on the XZ plane with a principal load axis in the scan direction, or Y axis.

![Diagram of orientations](image)

Figure 5.19. The various orientations of the machined tensile specimens are defined as “A”, “B”, and “C”.

<table>
<thead>
<tr>
<th>Orientation</th>
<th>Sample plane</th>
<th>Unit vector normal to plane</th>
<th>Principal load axis</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>XZ</td>
<td>Y</td>
<td>Z</td>
</tr>
<tr>
<td>B</td>
<td>XY</td>
<td>Z</td>
<td>X</td>
</tr>
<tr>
<td>C</td>
<td>YZ</td>
<td>X</td>
<td>Y</td>
</tr>
</tbody>
</table>

Table 5.4. Description of each orientation tested.

A thermodynamically consistent prediction of melt pool solidification served as a qualitative output of an in-house finite element software that takes process parameters, tool path, component geometry and the thermodynamic properties of Ti-6Al-4V as inputs (Smith, Xiong et al. 2016). The in-house code, the GAMMA code, demonstrated the evolution of the melt pool’s solidification front during a build. In this case, the model was a 40 mm by 40 mm by 40 mm Ti-6Al-4V component with 212,112 linear heat transfer hexahedral elements and 224,315 nodes. Further details on the GAMMA code are provided earlier in this chapter in Section 5.2.2. Values used for various material properties for Ti-6Al-4V and processing parameters are given in Table 5.5:
Table 5.5. Ti-6Al-4V material and process parameter values used for the GAMMA thermal model.

<table>
<thead>
<tr>
<th>Variables</th>
<th>Symbols</th>
<th>Values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Convection heat transfer coefficient</td>
<td>$h_c$</td>
<td>$5 \times 10^{-5}$ J·mm$^{-2}$·K$^{-1}$</td>
</tr>
<tr>
<td>Emissivity</td>
<td>$\varepsilon$</td>
<td>0.2</td>
</tr>
<tr>
<td>Stefan-Boltzmann constant</td>
<td>$\sigma_s$</td>
<td>$5.6704 \times 10^{-14}$ W·mm$^{-2}$·K$^{-4}$</td>
</tr>
<tr>
<td>Ambient temperature</td>
<td>$T_{\infty}$</td>
<td>300 K</td>
</tr>
<tr>
<td>Laser processing power</td>
<td>$Q$</td>
<td>710 W, 800 W, 940 W</td>
</tr>
<tr>
<td>Laser beam radius</td>
<td>$r_b$</td>
<td>0.915 mm</td>
</tr>
<tr>
<td>Heat capacity</td>
<td>$C_p$</td>
<td>0.6 J·g$^{-1}$·K$^{-1}$</td>
</tr>
<tr>
<td>Density</td>
<td>$\rho$</td>
<td>$7.99 \times 10^{-3}$ g·mm$^{-3}$</td>
</tr>
<tr>
<td>Thermal conductivity</td>
<td>$\kappa$</td>
<td>0.02 W·mm$^{-1}$·K$^{-1}$</td>
</tr>
<tr>
<td>Change in enthalpy</td>
<td>$\Delta H_l$</td>
<td>286 J·g$^{-1}$</td>
</tr>
<tr>
<td>Liquidus temperature</td>
<td>$T_l$</td>
<td>1726.1 K</td>
</tr>
<tr>
<td>Solidus temperature</td>
<td>$T_s$</td>
<td>1607.4 K</td>
</tr>
</tbody>
</table>

The solidification cooling rate for a location of the component was determined at each element of the computational model. Each element was 0.625 mm in width, 0.625 mm in length and 0.95 mm in height, with a volume of 0.371 mm$^3$. The cooling rate of each location, or point of the component, was representative of this volume. The FEM process thermal model resulted in a temporal resolution of 0.001 seconds, which was high enough to capture the characteristic material properties to be experimentally examined and to capture the effect of latent heat in solidification. This study assumed that the dominant influence during solidification occurred during the last re-melting cycle, as many areas of a component were prone to re-heating and re-melting during fusion of additional layers. As seen in the temperature profile in Fig. 5.20, there were two cycles of melting over time at the highlighted green element, with the last re-melting cycle as the circled region between the solidus and liquidus temperatures of Ti-6Al-4V. To calculate the solidification cooling rate at an individual probed point, the last re-melting cycle was evaluated between the liquidus and solidus boundaries of Ti-6Al-4V.
Figure 5.20. Melt pool evolution during the model's element birth; thermal history of a probed point (highlighted green element) with the solidification cooling rate calculated at the circled re-melting cycle.

The output of the thermal model was the thermal history at every node during the DED build. A snapshot of the build at about 5,000 seconds showed that the melt pool was near the front of the component as a new layer fused with the subsequent layer as shown in Fig. 5.20. The melt pool was represented by red elements that had temperatures greater than the melting temperature, i.e., greater than 1,877 K. The group of red elements that represented the melt pool totaled to about 15 mm$^3$ at any given time throughout the build. Compared to the melt pool volume throughout the build, the element size of less than 0.4 mm$^3$ was refined enough to resolve the thermal gradient within the melt pool. The model captured the rapid cooling of the component while subsequent layers underwent re-melting cycles when the laser traversed over. Figure 5.20 shows the 800 W processed component at a point and time window while the model's outputs for the 710 W and 940 W processed components showed similar re-melting cycles and melt pool evolution at the same point and time window but with slightly different numerical values.
The thermal history at any point of the component can be plotted to show the solidification cooling rate between the liquidus and solidus temperatures at multiple re-melting cycles, as denoted by the dotted lines in Fig. 5.20. Porosity micrographs (2D images) were taken at the probed points of the thermal model. The solidification cooling rate values at these points completed the link between process and thermal history in the proposed framework. The cooling rates were also shown to have relationships with porous microstructure at the same points, which will be discussed in Chapter 7 on the porosity in DED-processed Ti-6Al-4V.

The GAMMA code provided the localized thermal history at any point of a component’s build, including at the melt pool boundary, or liquid-solid interface. As mentioned in previous sections, the GAMMA code did not include the fluid dynamics and Marangoni flow within the melt pool. In addition, the influence of powder flow rate, or mass addition into the melt pool, was not considered in GAMMA analyses. *In-situ* monitoring of a changing melt pool because of localized interactions of the laser beam with individual powder particles can provide additional insight on localized thermal histories. As discussed in the next section, high-speed X-ray imaging during the process investigated the temperature changes at the melt pool and how cooling behavior changed with mass addition into the melt pool.

5.6.2. Ti-6Al-4V *piezo-driven flow with X-ray imaging*

In the study discussed in Chapter 3 on the interaction of a laser beam with piezo-driven powder flow of Ti-6Al-4V particles, cooling and convection velocity of the melt pool was also determined.
Cooling behavior can be attributed to process parameters, powder flow, and mass change into or out of the melt pool.

Mass changes in the melt pool influenced the dynamics of the melt pool, its cooling behavior and therefore, the structure and properties of the final part. Mass change in the melt pool was monitored and calculated by tracking the size of any particle that entered or ejected out of the melt pool, counting addition as positive mass change and spatter as negative mass change. Mass subtraction did not consider evaporation. The cooling rate was determined by using the difference between the extreme temperatures of the liquid phase for Ti-6Al-4V. More specifically, the temperature change was the difference between the vapor/liquid and liquid/solid temperatures (3315 K and 1928 K, respectively) (Gale and Totemeier 2003). Time change was determined by counting the number of frames it took for the melt pool to solidify and remain stationary after the laser turned off. The resulting cooling rate was compared with the total mass change of each pass in each keyhole experiment, as seen in Fig. 5.21.
Figure 5.21. Cooling rate with the change of mass in the melt pool for each pass during each keyhole mode experiment. Each data point represents a pass in one experiment.

The trends in cooling for both sets of keyhole experiments matched what previous modeling studies predicted (Thompson, Bian et al. 2015), (Yang, Gu et al. 2018), (Stender, Beghini et al. 2018). The cooling rates in the first pass were greater than those in the second pass. At the beginning of the first pass, the laser heated up a substrate with an initial state at ambient temperature, resulting in a large thermal gradient. When the second pass began, the substrate was still hot from the first pass and the thermal gradient after the pass was not as significant. The difference in cooling rates between the first and second passes were greater for the 250 W, 300 mm/s set of experiments than that of the 150 W, 100 mm/s set of experiments because the more powerful laser caused a greater thermal gradient during the first pass, but also caused a hotter substrate when the second pass began. As the amount of mass in the melt pool increased, the cooling rate generally decreased as more time was required to melt and cool a greater amount of material in a larger melt pool. The dependence on mass change was greatest during the first pass.
because more time was required to melt additional material, or particles, in ambient temperature and resulted in rapid cooling whereas the initial elevated temperature at the beginning of the second pass overcame the influence of mass change at ambient temperature. The greater scan speed in the 250 W, 300 mm/s experiments as compared to the 150 W, 100 mm/s experiments resulted in a greater spread in cooling rates and increased dependence on mass during the first pass.

The cooling rates of the industrial-scale Ti-6Al-4V cubic component calculated the solidification rate between the solidus and liquidus temperatures, or the “mushy” zone. In contrast, the cooling rates of the micro-scale DED study were calculated from the moment the laser turned off (gas phase), throughout the liquid phase, and until the melt pool solidified. This resulted in cooling rates in the industrial-scale with values of about $10^3$ K/s, while the cooling rates in the micro-scale DED study calculated the rate from the peak temperature for values of about $10^6$ K/s. Limitations in the analyses of the X-ray images included the inability to differentiate the liquidus temperature of the melt pool, which could be used to provide the solidification cooling rate in the “mushy” zone. Future work would include advanced image processing to provide the ability to identify the liquidus temperature in the melt pool during high-speed X-ray imaging. An overall cooling rate from peak temperature to the solid phase can provide an indication of what types of phase transformations could occur during cooling, though spatially and temporally-resolved temperature changes in the melt pool would provide a more accurate calculation of cooling rates.

With a laser beam size of 80 μm, the sensitivity of laser absorptivity and cooling to mass change was greater than that of commercial additive manufacturing systems that use laser beam diameters
of up to 5 mm. This study revealed a small-scale representative investigation into the influence of mass change in the melt pool on the cooling behavior of a build.

5.7 Summary
Thermal histories of DED-processed component determined the structure, microstructures, and properties. Monitoring of changes in thermal and cooling behavior provided insight into heat transfers during the DED process. This allowed one to observe how changes in process parameters directly influence heat transfer. Most in-situ thermal monitoring methods only provided surface measurements. In this work, IR thermography was used to monitor the changes in temperature at the surface of Inconel 718 clads and thin walls. Cooling behavior and thermal gradients were calculated from IR images. Coupling experimental and computational work was vital to capture, calibrate and validate thermal phenomena during the build. Comparing cooling behavior from IR thermography and the CtFD model provided energy density and cooling rate thresholds for structural characteristics of Inconel 718 clads. Including Marangoni convection and fluid dynamics in the CtFD model allowed for a more thorough view of how process parameters influenced localized thermal histories.

An in-house code, GAMMA, used material properties and processing parameters as inputs to monitor the temperature changes at any spatial or temporal point during DED processing of Ti-6Al-4V cubes. To capture the influence of powder mass flow rate and localized cooling of the melt pool, images from in-situ high-speed X-ray imaging during two passes tracked how individual particles modified cooling behavior.
Future work includes bridging the multiple scales in cooling behavior with both experimental and computational methods. Experimentally, work is being done on how to calibrate emissivity values to provide absolute temperatures during IR thermography. The use of thermocouples within substrates and two-wave pyrometers for melt pool monitoring can aid in calibrating for absolute temperature. Coupling two or more experimental thermal monitoring methods, including high-speed X-ray imaging, can advance and bridge the multiple spatial and temporal scales of the thermal histories observed in the DED process. With advances of experimental techniques, thermal modeling will have more accurate datasets for calibration and validation. Future work in thermal modeling can incorporate the influence of powder flow rate, including powder flow behavior and changes in melt pool size. This can also lead to improving thermal models that capture the volume change and warping induced by thermal stresses in laser deposition.

The next chapters explore the influence of thermal history on microstructure, porosity, and mechanical behavior. Chapter 6 evaluates the resulting phase transformations of DED-processed materials as a type of ex-situ thermal monitoring. By measuring secondary dendrite arm spacing of solidified materials, localized cooling rates can be calculated. Observations on the various phase transformations, dendritic arm spacing, cellular structures, grain structures, and solidified melt pool boundaries reveal the rapid cooling of DED-processed materials.
Chapter 6: Microstructure of DED-Processed Materials

This chapter focuses on the resulting as-built microstructures of additively-built materials. Because of rapid, directional cooling in DED-processed components, unique phase transformations and microstructures occur. Process parameters, and therefore the thermal histories at localized areas, determine the crystalline structure (Section 6.1). This section will discuss how characteristics of thermal processing including cooling rate, solidification rate, and thermal cycling influence microstructure formation. Section 6.2 provides an overview of the experimental methods used in this work to determine the microstructure, including phase, chemical composition, dendritic structures, and grain structures. The materials discussed in this section are 316L SS (Section 6.3), Inconel 718 (Section 6.4), and Ti-6Al-4V (Section 6.5), as seen in Fig. 6.1. Section 6.6 provides a summary of the microstructures observed in this work.

Figure 6.1. Materials discussed in DED-processed microstructures.
6.1 The influence of thermal history in rapid solidification

This section links process to thermal history to microstructure. Section 6.1.1. discusses how thermal history determines phase transformations, grain nucleation, and solidification structures like dendrites, Section 6.1.2. provides a brief literature review on the microstructures observed from the DED process. One of the overarching goals in additive manufacturing research is to obtain specific microstructures that will improve the mechanical properties of a metal, such as grain-size refinement and solid-solution strengthening.

6.1.1 Introduction to mechanisms during solidification

Changes in microstructure often require a phase transformation to occur, which are mostly diffusive transformations in additive manufacturing. Phase transformations also include “transformations that maintain the type and number of phases, transformations that alter the phase composition, and transformation into metastable phases” (Porter, Easterling et al. 2009). Other changes in microstructure that maintain the type and number of phases include allotropic transformations, recrystallization, and grain growth. Metastable phases include martensitic transformations, which have been observed in the 316L SS and Ti-6Al-4V microstructures in this work. Diffusive phase transformations require both nucleation and growth. Nucleation occurs when new particles, or nuclei, form new phases. These nuclei usually form at grain boundaries and defects. The nuclei grow, pushing against the surrounding matrix material, or the original phase. Usually the transformation rate is thermally activated (Mittemeijer 1992) and can be quantified by the Arrhenius equation, \( r = A \exp \left( -\frac{Q}{RT} \right) \), where \( A \) is a constant that typically describes the
frequency with which liquid atoms attach to the solid nucleus, \( Q \) is activation energy for the transformation, \( R \) is the universal gas constant, and \( T \) is the absolute temperature.

During the rapid cooling in the DED process, the transformation rate is usually slower than the cooling rate between the solidus and liquidus temperatures, resulting in metastable microstructures. Rapid solidification occurs when most of the latent heat of fusion is absorbed by the molten metal, making the external rate of heat loss negligible (Mehrabian 1982). Microstructure formation during solidification depends on the interfacial morphology, which is a function of the thermal gradient at the liquid-solid interface of the melt pool and the interfacial velocity, also known as the growth rate (Cohen and Mehrabian 1980). Therefore, rapid solidification usually results in dendritic structures, depending on the interfacial velocity (m/s) and the temperature gradient (K/m) of the liquid-solid interface (Cohen and Mehrabian 1980). The average cooling rate can be defined as a function of both the thermal gradient and interfacial velocity (Cohen and Mehrabian 1980):

\[
\dot{T} = G \times R
\]  

(6.1)

where \( G \) is the temperature gradient at the liquid-solid interface and \( R \) is the interfacial velocity, or growth rate. Figure 6.2 shows the resulting microstructures during solidification (Cohen and Mehrabian 1980).
While the thermal gradient and growth rate drive the type of microstructure, the cooling rate, $\dot{T}$, drives the refinement of microstructure during solidification. With an overall increase in average cooling rate, microstructures become more refined during the phase transformation. These solidification structures nucleate in the “mushy” zone within the interface, which comprises both the liquid and solid phases. Figure 6.3 shows the schematic of the common microstructures that result from rapid solidification (Flemings 1974), with Fig. 6.3a providing a more detailed graphic of Fig. 6.2 with its resulting microstructures, and Fig. 6.3b illustrating the solidification structures. Dendritic formation occurs from either heterogeneous nucleation or from a dendrite multiplication mechanism. When enough agitation is introduced to the solidification process, the solid and liquid zones are separated, dissipating the mushy zone. This leads to spheroidal growth sites, as seen in the equiaxed, non-dendritic structure in Fig. 6.3 (Flemings 1974).
Individual dendrites are much smaller than grain structures with a grain usually composed of a full dendritic structure. Dendritic structures are purely due to solidification, whereas grain boundaries occur due to the crystallographic misorientation between two neighboring grains (Brandon 1966). Figures 6.4a and 6.4b shows how grain boundaries might look with many columnar or equiaxed dendrites within them (Flemings 1974). Though each grain in the microstructure is composed of multiple dendritic arms, the arms grow from the same nucleation point, resulting in an identical crystallographic orientation for all dendritic arms within a grain. The multiple dendritic arms from the same nucleation point with identical crystallographic orientation compose a dendritic structure. Thus, each grain is composed of a dendritic system.
The secondary dendrite arm spacing (SDAS) is the spacing between secondary arms, as seen in Fig. 6.4c which differentiates primary and secondary dendrite arms and their spacings. The SDAS in rapidly cooled components can indicate the cooling rate during solidification in a localized area.

The overall relationship of SDAS to cooling rate is:

$$\lambda_{SDAS} = A(\dot{T})^{-n}$$  \hspace{1cm} (6.2)

where $A$ and $n$ are constants that depend on the particular alloy (Glicksman and Voorhees 1984).

An analysis of the cooling rate and SDAS of various DED-processed materials are discussed in this chapter.

6.1.2 Microstructures of alloys observed in DED

Based on the solidification fundamentals in the previous section, DED process parameters have the potential to be determined to generally control the thermal gradient and interfacial velocity of the melt pool’s liquid-solid interface. For example, modifying laser power generally influences the thermal gradient and modifying laser scan speed generally influences the interfacial velocity at the melt pool boundary. Additional parameters such as carrier gas, powder mass flow, toolpath and
part geometry also influence the thermal gradient and interfacial velocity. This section provides a brief literature review of the microstructures observed and achieved with control methods for 316L SS, Inconel 718, and Ti-6Al-4V during the DED process.

In laser-based DED, the four main mechanisms of nucleation include dendrite fragmentation, grain detachment, heterogeneous nucleation, and surface nucleation (Kou 2003). During dendrite fragmentation, the convection in the melt pool can fragment the tips of the dendrites in the “mushy” zone. The fragments serve as nuclei for new grain formation. In grain detachment, the convection in the melt pool can cause partially melted grains to separate from the “mushy” zone at the boundary of the melt pool. If they do not melt fully and dissipate within the melt pool, these detached grains can also serve as nuclei for new grain formation. During nucleation, solutes that differ in chemical composition from the melt pool matrix material serve as nuclei for new grain formation in the melt pool. With the presence of an external cooling mechanism, surface nucleation can occur at the melt pool surface and grow downward. Overall, equiaxed refined grains driven by melt pool convection are desirable during DED for improved mechanical behavior (Kou 2003), (Farshidianfar, Khajepour et al. 2016).

As discussed in Chapter 2, 316L SS is an austenitic steel that undergoes phase transformations into ferrite or a mixture of austenite and ferrite during solidification. In a study by the Seely group (Yadollahi, Shamsaei et al. 2015), the microstructure of a single 316L SS cylindrical rod was compared to that of a group of nine rods built in the same test and platform. The aim was to evaluate how the thermal history of additional builds influences the final part properties. They
found that the grain size refined from an average of 60 to 45 µm in components built among the nine rods. With heat treatment of the single rod, the elongated grains recrystallized to an average size of 80 µm. Heat treatment also depleted the ferrite phase from about 10% of the phase composition in the as-built sample to 0%. In another study of DED-processed 316L SS (Guo, Zou et al. 2017), the microstructures resulting from differing building directions (0° and 90°) were compared. In the 0° build direction, the resulting solidification structure exhibited a refined equiaxed structure. In the 90° build direction, epitaxial nucleation of large, columnar dendritic grains occurred at the melt pool boundaries because of the increased thermal gradient in the vertical build direction. The overall cooling rate was lower than that of the 0° build direction, resulting in grain coarsening (Guo, Zou et al. 2017). Real-time control of the microstructure for DED-processed 316L SS could be achieved by measuring the cooling rate during the process with an IR camera (Farshidianfar, Khajepour et al. 2016). They found that cellular grains dominated the top of the clad structures due to high cooling rates, while columnar grains dominated the bottom of the clad structures due to lower cooling rates.

The phase transformations that occur during the solidification of Inconel 718 include precipitation of equilibrium $\gamma'$ (Ni$_3$Al), metastable $\gamma''$ (Ni$_3$Nb), equilibrium $\delta$ (Ni$_3$Nb), Laves (Fe$_2$M), $\eta$ (Ni$_3$Ti) and carbide phases (Sames, Unocic et al. 2014). The presence of $\gamma''$ precipitates usually strengthens the face centered cubic ($\gamma$) nickel matrix material while the $\delta$ phase typically reduces its strength and controls grain size. The Laves phase typically leads to embrittlement in segregated interdendritic regions where there is a greater concentration of Nb, the solute strengthening agent in Inconel 718. A study by Zhao et al. (Zhao, Chen et al. 2008) investigated the microstructure of
DED-processed Inconel 718 where mostly columnar dendrites grew epitaxially along the crystallographic orientation parallel to the build direction. The spacing between the primary or secondary dendritic arms revealed the rapid solidification of the melt pool, with an average primary arm spacing of about 5 µm (Zhao, Chen et al. 2008). In a study by the Attallah group (Parimi, Ravi et al. 2014), the microstructures of Inconel 718 as a result of two different tool paths and two laser power values were compared. The three build strategies are shown in Fig. 6.5, with B1 representing the unidirectional toolpath at 390 W laser power, B2 the bi-directional toolpath at 390 W laser power, and B3 the bi-directional toolpath with 910 W laser power. Figure 6.5 also shows the resulting dendrite and grain orientation due to the varying build strategies.

![Figure 6.5. Schematic of the dendritic/grain orientation of the entire component built with the (a) B1 uni-directional path with 390 W laser power; (b) B2 bi-directional path with 390 W; (c) B3 bi-directional path with 910 W (Parimi et al., 2014).](image)

The varying dendritic structures were oriented at the angle of resulting heat flux due to the laser source in the scanning direction and the heat flux due to the substrate in the build direction. With the B1 and B2 build strategies, the microstructure was a mixture of fine and coarse grains that alternated in orientation, depending on the toolpath, as seen in Fig. 6.6. At the 910 W laser power,
the grain structure was coarse and columnar, with large Laves phase particles, δ phase and carbides (Parimi, Ravi et al. 2014).

Phase transformations during rapid solidification of DED-processed Ti-6Al-4V usually result in coarse columnar β-grain structures. Solidification microstructures for titanium alloys do not form dendritic structures as the β phases transforms into β+α phase, dissolving the dendritic structure in the “mushy” zone if β dendrites nucleate (Bermingham 2008). The “mushy” zone is the region of “mixed phase between the fluid melt and the frozen solid” that moves from the solidified area into the fluid area (Hills, et al. 1982).

Thermal history can be used to determine the phase compositions of the α, α′ (martensite), and β phases in Ti-6Al-4V, where the β phase leads to greater ductility and the α, α′ phases lead to greater strength but more brittle behavior. During heating of Ti-6Al-4V, a phase transformation
occurs from $\alpha$ to $\beta$ phase above the transus temperature of 1273 K, with the $\beta$ phase growing by diffusion. During cooling, three transformations can occur, including diffusive $\beta$ to $\alpha$ and $\alpha'$ to $\alpha$ transformations and a diffusionless $\beta$ to $\alpha'$ transformation.

A study by the Voice group (Wu, Liang et al. 2004) found that during the DED process, basket weave structures with $\alpha$ and $\beta$ lathes form. The size of the lathes increased with increasing laser power and decreasing scan speed. In a study that investigated the anisotropy and location dependence of DED-processed microstructures (Carroll, Palmer et al. 2015), it found that grain coarsening occurred at the top of a thin wall (a build with no hatching), with finer microstructures at the bottom near the substrate. The Palmer group investigated the role of part geometry on microstructures during the DED process (Keist and Palmer 2016). determined, showing how grain structure coarsened with the cruciform structure and became more columnar with a thicker geometry, as seen in Fig. 6.7. The $\alpha$ lathe widths refined with thicker geometries, indicating greater cooling rates in bulk structures, as shown in Fig. 6.8. However, quantitative cooling rate differences were not determined with the process or from the resulting microstructures, providing an opportunity for future work (Keist and Palmer 2016).
Figure 6.7. Optical macrographs from the (a) L-shape thin wall; (b) L-shape thick wall; (c) cruciform thin wall; (d) cruciform thick wall. The schematic in the bottom row outlines the prior β grains (Keist & Palmer, 2016).

Figure 6.8. Representative optical micrographs from the (a) L-shape thin wall; (b) L-shape thick wall; (c) cruciform thin wall; (d) cruciform thick wall (Keist & Palmer, 2016).

6.2 Experimental methods to characterize microstructure

Because most of the DED-processed components investigated in this work exhibited substantial surface roughness due to the resolution limited to the powder size, destructive methods were
required to investigate the microstructure. Destructive methods include sectioning, grinding, polishing, and etching.

After polishing the cross sections of a material, the image from an optical microscope can be captured by normal, photosensitive cameras to generate a micrograph. Optical microscopy typically uses a CMOS or CCD camera to capture a digital image with resolutions of up to hundreds of nanometers. This method is typically the first and easiest when evaluating dendritic spacing, overall phase compositions, porosity, and possibly grain boundaries.

Scanning electron microscopy (SEM) uses an electron beam to interact with a surface topography of a material. The excited atoms of the material’s surface emit secondary electrons, which the SEM detects to produce an image. The resolution of SEM ranges from sub-nanometer scale to about 20 nm. Energy-dispersive X-ray spectroscopy (EDS) can be used with SEM to characterize the chemical composition of a localized area on the material’s surface (Humphreys 2004). Because each element has its unique atomic structure, EDS uses an X-ray detector to produce unique peaks. SEM is useful to investigate small-scale and 3D microstructures, such as lathes or fracture surfaces in additive manufactured components. Many additive manufactured components consist of defects like porosity and inclusions. EDS is crucial to differentiate pores and oxides, identify precipitates such as carbides, and identify microsegregation from phase transformations like Nb-enriched areas. Identifying oxygen content in an additive manufactured component using EDS can determine how much atmospheric gas leaks into the build chamber.
X-ray powder diffraction aided in identifying phases in additive manufactured components, with each phase having a unique lattice spacing, and therefore, a unique diffraction pattern. Micro-Laue X-ray diffraction was conducted at the Advanced Photon Source (APS) in Argonne National Laboratory (Liu and Ice 2014). The 34-ID APS beamline uses the polychromatic Laue micro-diffraction technique to examine the structure of materials with sub-micron spatial resolution in all three dimensions. Properties that can be measured include local crystallographic orientations, orientation gradients and strains. The source is Undulator A with energy range from 7-30 keV, a beam size of 300 x 300 nm, energy resolution of 1e-4. Figure 6.9 shows a schematic of micro-Laue diffraction and how detectors can provide crystal orientation with a white beam and localized lattice spacing differentials, or residual strain, with a monochromatic beam.

Figure 6.9. Schematic of Micro-Laue diffraction at APS 34-ID beamline (W. Liu & Ice, 2014).
6.3 Microstructures of DED-processed 316L SS

6.3.1 Cylindrical rods for tensile testing

A method to determine the cooling rate during processing is to evaluate the microstructure morphology within an additively built component. The SDAS value in a localized area in the solidification structure has been shown to be related to the cooling rate of metallic alloys, where the relationship for 316L SS alloys is (Zheng, Zhou et al. 2008):

$$\lambda_{SDAS} = 80\dot{T}^{-0.33}$$  \hspace{1cm} (6.3)

where $\lambda_{SDAS}$ is the secondary dendrite arm spacing and $\dot{T}$ is the cooling rate. Methods to determine solidification structure in 316L SS included optical microscopy to view the melt pool shape and morphology, phase distribution, PDAS, and SDAS.

Stainless steel 316L cylindrical rods (Fig. 6.11 left) for tensile testing were built at Northern Illinois University using an OPTOMEC laser engineered net shaping (LENS) machine for DED processing. The OPTOMEC LENS 750 machine used an IPG ytterbium laser at 1070 nm with a maximum of 3 kW of laser power and a beam diameter of 1.5 mm. The diameter at the two ends of the cylindrical rod, used for clamping in the tensile test, was 9.5 mm, while the diameter of the central part of the rod, the gage area, was 6.0 mm, and the length of the rod was 160 mm. The rods were built on the platform with the surface of one grip at the surface of the substrate plate. The overall path of the laser consisted of a 0 to 180 degree scanning strategy, where the build direction, Z, was along the tensile direction. The 160 mm length was the height of the overall build relative to the substrate plate. The laser power input was 450 W with a measured output of 290 W,
laser scan speed was 12.7 mm/s, the hatch spacing was 0.559 mm, the layer thickness was 0.381 mm, and the powder mass flow rate was 112.5 mg/s.

In the rod structures, solidification microstructure changed from the bottom of the build to the top surface, indicating the changes in cooling rate, interfacial velocity (growth rate), and the thermal gradient in each area. Refined equiaxed grains formed at the melt pool interface, or the solidification front between each layer, indicating high cooling rates. The solidification front is the formation of the “mushy” zone at the end of the moving melt pool, where solidification first occurs. Epitaxial dendritic or cellular growth occurred epitaxially within the melt pool toward the center of the melt pool cladding surface of the layer (scan-hatch plane), with growth from the melt pool boundaries, as seen in Fig. 6.10. The periodic melt pool boundary ripples in Fig. 6.10 indicated the solidification front. Because the build-hatch plane is shown in Fig. 6.10, the vertical spacing of the solidification fronts of the melt pool boundary indicated the process parameter of layer thickness and the horizontal spacing of the solidification fronts indicated the hatch spacing parameter.
Near the bottom of the rod at the 46th to 48th layers (out of a total of 420 layers), or 18 mm above the substrate in the 160 mm tall rod, there were large amounts of the ferrite phase (dark blue in Fig. 6.11) in the austenitic matrix (light intensity in Fig. 6.11). The micrographs show the melt pool boundaries, with the fusion, or “mushy” zones where small, equiaxed grains dominated. The boundaries, or the interface between each layer, exhibited the greatest cooling rates. Within the melt pool boundaries, equiaxed and columnar dendritic structures formed and coarsened, as shown in Fig. 6.12. The SDAS spacing ranged from 1 to 3 µm, with an average of about 1.5 µm, indicating a cooling rate of more than $1.5 \times 10^4$ K/s near the bottom of the rod. The rapid cooling rate is indicative of the large thermal gradient between the first several layers of the built component and the substrate plate, which was initially at room temperature.
Figure 6.11. Microstructure at the bottom section of a DED-processed 316L SS tensile rod.

Figure 6.12. Columnar and equiaxed dendritic structures near the bottom of the rod with both micrographs indicating the same plane and orientation.
Moving along to the top of the rod, microstructures gradually became more cellular, the amount of ferrite phase increased, and the SDAS for dendritic structures increased with overall coarsening, indicating decreasing cooling rates. From about the 100th to 200th layers (out of 420 total layers) of the rod, the SDAS values ranged from 1 to 4 µm with an average of about 2 to 2.5 µm, indicating decreasing cooling rates of about $2.0 \times 10^4$ to $3.6 \times 10^4$ K/s. The differences in cooling rates within each layer at the center of the rod were also greater than the differences in cooling rates closer to the bottom of the rod. This can be attributed to lower thermal gradients between the built layer and the heated underlying layers. The variability in structure and cooling rate could arise from overlapping hatching, where clads within a layer closer to the edge would radiate more heat and result in less heat conduction from surrounding built material. Refined structures were found at the layer interfaces, which were sites of nucleation. Between the 200th and 320th layers of the rod, the SDAS increased to an average of about 3 to 5 µm, indicating decreasing cooling rates of about $5 \times 10^3$ to $2 \times 10^4$ K/s.

Figure 6.13 shows the XY (vertical axis is the hatch direction and horizontal axis is the scan direction) plane near the top of the tensile rod. With this planar view, there is no directional growth, and the structure is mostly equiaxed. The size of the cellular structure ranged from 5.9 to 7.3 µm in diameter. There were also carbides near the cellular boundaries, indicating
segregation during phase transformation. At localized areas around the melt pool boundaries near the top of the tensile rod, small amounts of columnar structures appeared with refined SDAS of 0.5 to 1.5 µm, indicating cooling rates as high as 5e6 K/s. This was possibly attributed to nucleation that occurs at the “mushy” zone with high convection velocities, leading to dendrites that did not coarsen during the process because there was not as much of a heat input above the layer. The refined cellular and planar structures at the top of the rod indicated high cooling rates and greater thermal gradients. This can be attributed to less number of re-melting cycles that occur near the top of the rod, where subsequent melting cycles decreased the cooling rate between the solidus and liquidus temperatures in addition to decreasing the thermal gradient with an underlying heated layer.

6.3.2 Charpy components for impact testing

In a study to investigate the competing effects of grain structure and porosity in DED-processed 316L SS components, Charpy specimens that underwent impact testing were evaluated. Charpy specimens were built with dimensions of 55 mm long, 10 mm in height, and 10 mm in width in Northern Illinois University’s OPTOMEC LENS 750 machine. Two sets of process parameters were used. One parameter set used a laser beam diameter of 3 mm, 475 W laser power input, laser scan speed of 2.1 mm/s, powder mass flow rate of 17.5 mg/s, hatch spacing of 958 µm, and a layer height of 201 µm. The second parameter set used a laser beam diameter of 3 mm, 850 W laser power input, laser scan speed of 2.1 mm/s, powder mass flow rate of 48 mg/s, hatch spacing of 1486 µm and a layer thickness of 464 µm. Each build composed of 84 layers with a 0 to 90 degree scan strategy, where the scanning direction was the length (55 mm) of the Charpy sample.
After the impact testing, the Charpy parts were studied for mechanisms behind ductile deformation and failure. Grain structure and porosity are two dominant factors affecting strength, elongation, and fracture behavior. Ductile materials are known to be weakened because of microstructural defects, i.e., through void nucleation by inclusion debonding, void growth, and void coalescence (Hill 1998). At the same time, the Hall-Petch effect correlates grain size to yield strength (Hansen 2004). The mechanisms behind failure are typically due to the porosity in low-density parts, whereas failure in fully dense parts are dominated by grain structures. The DED process allows for a large variety of combinations of different levels of porosity and grain structure within a single part. It is therefore extremely important to study the competing effects of grains and porosity, which is detailed in subsequent chapters and the work of collaborators.

Using micro-Laue diffraction at the 34-ID beamline in APS, crystallographic structure and orientation were determined at cross-sections that were near the top surface of both 475 W and 850 W processed Charpy parts. Using a white beam and rastering resolution of 2 µm, a volume of 300 µm in the scan direction, 600 µm in the build direction, and 100 µm in the hatch direction (penetration depth of the X-ray in 316L SS) was probed to determine the crystallographic structure for the 475 W processed Charpy part.

The resulting structure of the 475 W-processed part is shown in Fig. 6.14. The left side shows the crystallographic orientations in the YZ plane, or the scan-build plane of the Charpy part. The right side of Fig. 6.14 shows the crystallographic orientation of the XZ plane, or the through thickness
of the part at the center of the YZ plane, or at “A” cross section. The layer interfaces were apparent in the YZ plane, with part of the top layer extending toward the 200 \( \mu m \) mark, the second layer from the 200 to 0 \( \mu m \) mark, the third layer from the top in the range of 0 to -200 \( \mu m \) mark, and part of the fourth layer from the top in the range of -200 to -300 \( \mu m \) mark. These interfaces reflected the process parameter of layer thickness, which was set to 201 \( \mu m \). The spatial orientations of the grain boundaries reflected the laser scanning strategy. When the laser scanned from right to left, the grains nucleated and grew at the melt pool interface from right to left. With an increasing thermal gradient from the bottom to the top of the build, the angle of the elongated grains relative to the layer interface decreased. The average grain size in the YZ plane was about 25 \( \mu m \) in the minor axis and 80 \( \mu m \) in the major axis, or elongated direction. Grain boundaries in the XZ, or hatch-build plane showed a more equiaxed grain structure, though the penetration depth, or the hatch dimension of the cross-section, was about the same size as the hatch spacing. To observe a clearer grain structure pattern in the XZ plane, a greater area that shows more of the hatch spacing interfaces is necessary.

Figure 6.6. Crystallographic structure of a 475 W-processed Charpy part determined with micro-Laue diffraction.
The resulting structure of the 850 W-processed part is shown in Fig. 6.15. Using a white beam and rastering resolution of 5 µm, an area of 600 µm in the scan direction and 1,750 µm in the build direction was probed to determine the crystallographic structure at localized 5 by 5 µm areas. With a layer thickness of 464 µm, four layers can be observed in the probed area. However, the layer interfaces were not clear as coarsening of grains inclined toward the (111) direction occurs through the layers. In addition, the laser scanning strategy is not apparent as the grains coarsen from left to right throughout the four layers. Like those in the 475 W-processed part, the thermal gradients of the melt pool boundaries also increased going up the build direction, as indicated by the decreasing angle of grain nucleation relative to the layer interface. The structure is similar to those presented in literature (Parimi, Ravi et al. 2014) in Figs. 6.5 and 6.6.

Figure 6.7. Crystallographic structure of a 850 W-processed Charpy part determined with micro-Laue diffraction.
which show that using high laser power led to a high enough heat input for grains to grow epitaxially from the bottom of the part, or by the substrate heat sink, instead of re-nucleating at the melt pool boundaries. The grains were spatially oriented in the same direction throughout the layers (left to right), as the nucleation occurred when the laser scanned from left to right. It is not clear what the average grain sizes are due to the boundaries extending beyond the probed area. However, Fig. 6.15 shows that the grain coarsening through the layers resulted in sizes of at least 1 mm in the major axis, coarsening grains through at least three layers. Chapter 7 will investigate the porosity observed in these Charpy specimens and how they related to process parameters and thermal histories.

Overall, microstructural analyses of 316L SS set the precedent in this work that microstructure, and therefore mechanical properties, are heavily influenced by local thermal history (cooling rates, thermal gradients, temperature of the melt pool), location in the component, especially relative to the substrate heat sink (as observed in the cylindrical rod study), and with process parameters like laser power, as observed in the Charpy impact study. Chapter 8 will discuss the resulting mechanical behavior of the components described in this section.

6.4 Microstructures of DED-processed Inconel 718

6.4.1 Inconel 718 single clads

In the Inconel 718 single clads study discussed in previous chapters, the localized primary (PDAS) and secondary dendrite arm spacing (SDAS) throughout the clad were determined to compare microstructure to experimentally-determined cooling rates, which were calculated between the
liquidus and solidus temperatures from IR thermography. The dendrite spacing varied with depth in the clad and proximity to the clad surface, with an example of the dendritic structure for a 2,000 W-processed clad with 456 mg/s powder mass flow in Fig. 6.16.

![Dendritic Structure Example](image)

Figure 6.16. Example of secondary dendrite spacing within the clad cross section.

Spacing values at the center of the clad were evaluated for trends and for the CtFD model prediction. The cooling rate-microstructure relationships used for cooling rate determination and model prediction were (Hunt 1984):

\[
\lambda_{PDAS} = 2 \left( k_{part} \Gamma \Delta T_0 D_l \right)^{0.25} G^{-0.5} R^{-0.25}
\]

\[
\lambda_{SDAS} = 34 \dot{T}^{-0.25}
\]

where \( \lambda_{PDAS} \) is the primary dendrite arm spacing and \( \lambda_{SDAS} \) is the secondary dendrite arm spacing. The thermal gradient, \( G \), growth rate, \( R \), and the cooling rate, \( \dot{T} \), were calculated at the liquid-solid interface of the melt pool from the CtFD simulation. The partition coefficient is \( k_{part} \), \( \Gamma \) is the Gibbs Thomson coefficient, \( \Delta T_0 \) is the equilibrium freezing range, or difference between liquidus and solidus temperatures, and \( D_l \) is the liquid diffusivity.
SDAS values at the center of the clad were evaluated for trends and CtFD model prediction. Overall trends indicated that dendrite arm spacing decreased with decreasing powder mass flow rates. This corresponds with the increasing cooling rates observed in IR camera monitoring of clads with decreasing powder mass flow rate (Bennett, Wolff et al. 2017). As shown in Fig. 6.17, PDAS and SDAS from both experiments and the CtFD model decreased as function of power with an increase of the energy density, $E_D$. Results from both experiments and simulation matched well.

![Figure 6.8. Comparison of experimentally-determined and model-determined average dendrite arm spacing with energy density: (a) Primary dendrite arm spacing, (b) Secondary dendrite arm spacing.](image)

Although the center microstructure from experiments was used for the CtFD model calibration and validation, there was a range in solidification structure within a single clad. The range in microstructure gave reason for the morphology of micro-hardness throughout the clad, as discussed in Chapter 8. As shown in Fig. 6.18, large grain boundaries can be delineated in a single clad, with refined equiaxed grain structure at the clad-substrate interface. Because the substrate was 1045 carbon steel, an intermetallic region at the melt pool boundary formed with carbides, and Nb-rich microregated zones. Throughout the rest of the clads, epitaxial dendritic growth
occurred with a mixture of columnar and equiaxed dendrites. The spacings of the dendritic structures indicated the cooling of the clad. However, for clads with low cooling rates or with high gradient in cooling from the clad-substrate interface to the surface, the Laves phase appeared. The Laves phase was more apparent near the top surface of the clad, where the cooling rate of the melt pool decreased but the thermal gradient was at its peak. The single clad study revealed that the interaction of the laser beam with powder mass flow determined the cooling rate and microstructure morphology, with greater mass flows resulting in decreasing cooling rates and detrimental phases like the Laves phase.

Figure 6.18. Grain boundaries and the various dendritic structures present in an Inconel 718 single clad built onto a 1045 carbon steel substrate.

6.4.2 Inconel 718 thin walls

IN718 single clads expanded to the building of thin walls, or a build with 120 layers in the Z (build) direction but no re-melting in the lateral directions, as introduced in Chapter 5. IR camera temperature readings of the surface were coupled with thermal models to capture temperature histories within the build as well. Dwell time, or the amount of time that allows for cooling of the
component between each layer, was the key factor in the thin wall components. IN718 walls with no dwell time between each layer experienced much slower solidification cooling rates that components with a 60 second dwell time. The large difference in thermal history led to differences in solidification structure and crystallographic structure and residual strain determined with micro-Laue X-ray diffraction.

In both no dwell and 60 second dwell structures, the heat input with 1,800 W laser power was high enough to cause grain coarsening throughout both builds toward the top layer. The introduction of a dwell time did not prevent grain coarsening, as seen in the solidification structures in Fig. 6.19. This suggests that dwell time did not allow for substantial cooling for nucleation at the melt pool boundaries. The left side of Fig. 6.19 shows the no dwell processed thin wall at the top 15 layers (out of 120 total layers) at the XZ cross section, or the thickness of the thin wall, where there was no hatch spacing. The right side shows the same cross section of the thin wall built with 60 seconds of dwell time, which also exhibited grain coarsening through layers.
Zooming into the microstructures at the top layer shows that Laves phase formed due to low cooling rates. The Laves phase can be observed for both the no dwell (left) and 60 second dwell (right) cross sections in Fig. 6.20. In the no dwell part, the Laves phase dominated the top layer, or about 400 μm from the top. Even though IR thermography showed that the cooling rate at the top layer of the no dwell thin wall was at its maximum for the build, the thermal gradient at the interface between the top layer and the underlying layer was a site for grain nucleation. This nucleation led to the Laves phase at the top layer. Though underlying layers showed lower cooling rates from IR thermography, they were composed of coarsened grains that grew through layers
due to high heat input and lower thermal gradients between layers. In the 60 second dwell wall, the Laves phase formed in only sections of the top layer with a thickness of about 100 to 200 µm, in contrast to the thickness of 400 µm in the no dwell wall, indicating that the overall increase in cooling rates throughout the part with dwell time decreased the amount of Laves phase.

![Image of Laves phase](image)

Figure 6.20. Laves phase at the top of both the Inconel 718 cross sections with the left showing the no dwell XZ plane and the right showing the 60 second dwell XZ plane. Figure obtained with help from Haiguang Liao.

By evaluating the solidification structures throughout the rest of the layers, changes in cooling rate can be observed between the no dwell and 60 second dwell components. Figure 6.21 shows the solidification structures at the 12th layer from the top. In the no dwell component, the Laves phase still appeared, along with columnar dendrites. The columnar dendrites had a spacing of about 6.8 µm, indicating a cooling rate of about 625 K/s by using the constants used in the CtFD model in Section 6.4.1. In the 60 second dwell component, the secondary arm dendrite spacing was about 3 to 4 µm, indicating an increased cooling rate of up to $1.65 \times 10^4$ K/s.
6.5 Microstructures of DED-processed Ti-6Al-4V

For the tensile specimens extracted from the DED-processed Ti-6Al-4V cubic components, powder X-ray diffraction (XRD) was carried out to evaluate the phase composition differences for each orientation. Optical microscopy allowed for the imaging of solidification microstructure and the porosity distribution after each specimen was ground, polished and ultrasonically cleaned. The cross-sections evaluated under optical microscopy were sectioned from the tensile specimen grips using an aluminum silica blade.

Based on the mechanical behavior of the tensile specimens detailed in Chapter 8, the differences between each orientation within the 800 W processed cubic component were apparent. To determine whether porosity or phase composition dictated the difference in mechanical behavior for the three orientations, XRD analysis for each orientation, denoted “A” for the XZ (hatch-build) plane, “B” for the XY (hatch-scan) plane, and “C” for the YZ (scan-build) plane, was used to examine whether phase composition was a deciding factor, with the results in Fig. 6.22.
The XRD pattern showed negligible differences in phase composition for each orientation, with similarities to conventionally-processed, annealed Ti–6Al–4V. The resulting XRD pattern showed that phase composition had little influence on the variations in mechanical behavior for each orientation. This was the motivation to analyze porous structure in-depth and to evaluate if porosity had the largest influence on mechanical behavior. However, the powder XRD analysis provided averaged peaks over a 10 by 10 mm area, too large to evaluate the changes in phase compositions at localized areas within a DED-processed part.

The solidification structure of the Ti-6Al-4V cubes at the grips of the machined tensile specimens were observed, with microstructures that were equiaxed, columnar or bimodal. Figure 6.23 shows examples of micrographs that show the differences of the orientations within the 800 W laser power cubic part. Binary versions of these 2D micrographs were taken to measure the size, shape and distribute of these pores to help demonstrate the relationship between process parameters,
mechanical behavior and microstructure in J-factor determination, as discussed in Chapters 7 and 8. The numbers next to each orientation denotes the location of the micrograph relative to the surface of the cubic component, with each subsequent number denoting an additional 15 mm from the surface.

![Figure 6.9. Change in structure with orientation and distance from the surface.](image)

As explained in Chapter 5, a key aspect of the anisotropy analysis in the DED-processed Ti-6Al-4V specimens was the cooling rate at localized areas, with the hypothesis that the changes in rapid cooling dictated the changes in stiffness and strength of the material throughout a DED-processed component. Figure 6.24 shows the change in microstructure with the change in orientation and cooling rate, which was determined by probing the same point in the GAMMA thermal model. In the “A” orientation microstructure at about 7.5 mm away from the surface, the \( \beta \) phase transformation led to diffusional \( \alpha \) phase at prior \( \beta \) grain boundaries, with a “basketweave” Widmanstätten morphology indicative of low cooling rates between 0 and 20 K/s. However, the
GAMMA-determined cooling rate of the 0.65 mm x 0.65 mm x 0.95 mm voxel at the last re-melting cycle was about 600 K/s.

In the “B” orientation at about 7.5 mm away from the surface, an increase in cooling rate resulted in finer $\alpha$ phase lathe widths and “massive”, or matrix HCP $\alpha$ structures, indicated by the light intensity areas in Fig. 6.24b. The cooling rate determined by GAMMA at this area was about 900 K/s, though the observed structure typically appears after cooling rates between 20 and 175 K/s (T. Ahmed and H.J. Rack, 1998).

![Figure 6.24](image)

Figure 6.24. SEM images that show the change in $\alpha$ lathe width and cooling rate with orientation with a) microstructure in the “A” orientation and a GAMMA-determined cooling rate of about 600 K/s; b) microstructure in the “B” orientation and a GAMMA-determined cooling rate of about 900 K/s.

Figures 6.25 and 6.26 show examples of the martensitic phase present in the “B” and “C” orientations at 4.5 mm away from the surface of the 40 mm by 40 mm by 40 mm cubes, where high cooling rates resulted in $\alpha'$ martensitic transformation. The HCP martensitic $\alpha'$ phase is composed of long orthogonally oriented martensitic plates with acicular morphology with substructures that are composed of dislocations and stacking faults (T. Ahmed and H.J. Rack,
Cooling rates that result in the martensitic $\alpha'$ phase in Ti-6Al-4V alloys are typically greater than 500 K/s.

The GAMMA-determined cooling rates at the last re-melting cycles between the solidus and liquidus temperatures of these areas were about $1.0 \times 10^3$ and $1.2 \times 10^3$ K/s for the “B” and “C” orientations, respectively. The martensitic $\alpha'$ phase lamellar widths in the “B” orientation at 4.5 mm away from the surface was 3 to 10 µm, as seen in Fig. 6.25. With the GAMMA-determined cooling rate of $1.0 \times 10^3$ K/s, the martensitic structure formed preferentially to the grain boundaries. In the “C” orientation in Fig. 6.26, martensitic formation occurred with less preferential orientation to the surrounding grain boundaries and with a lamellar width of about 1 to 6 µm. The refinement of the martensitic lamellar widths qualitatively corresponds to the increasing trend in the GAMMA-determined cooling rates, though the predicted GAMMA cooling rates are quantitatively overestimated.

Figure 6.26. Martensitic formation in the “B” orientation, 4.5 mm from the surface.
Overall, the trends in the resulting Ti-6Al-4V microstructures observed in the 800 W process cubic component correspond to the trends in the GAMMA-determined cooling rate. However, the GAMMA-determined cooling rates do not predict the presence of α “basketweave” structures typical of cooling rates less than 20 K/s. In addition to cooling rates in the “A” orientation, the GAMMA cooling rates quantitatively overestimate the cooling behavior of the “B” and “C” orientations, which may be due to the lack of convection in the melt pool, the large voxel size, and the introduction of constant re-melting, which would also introduce constantly evolving phase transformations.

![Martensitic transformation in the “C” orientation, 4.5 mm from the surface.](image)

**6.6 Summary**

Evaluating microstructures in DED-process materials show that components are consistently heterogeneous with localized changes in phase composition. The heterogeneity in microstructure
is related to the changes in thermal behavior, even within the same layer or clad. Rapid directional solidification give rise to unique phase transformations, which include grain coarsening and dendritic growth. In 316L SS, microstructure morphology was observed with changing distance from the build plate for cylindrical rods and laser processing power for Charpy impact components. Changes in microstructure were observed with varying processing conditions for Inconel 718 clads and thin walls. The microstructure morphology in the Inconel 718 components can be compared to the trends observed in the cooling rates from IR thermography and a thermal fluid dynamic model, the CtFD model. Microstructure morphology in a DED-processed Ti-6Al-4V cubic component was observed with varying orientations, locations from the surface of the cube, and with corresponding cooling rates from the in-house GAMMA thermal model. Future work could include running analyses of the Ti-6Al-4V cubic components with the CtFD model and comparing the cooling rates with experimental microstructures. Cooling rates in 316L SS and Inconel 718 have been tied to their localized secondary dendrite arm spacings while cooling rate in Ti-6Al-4V can be tied to its α phase volume fraction.

Though the GAMMA and CtFD models effectively predict trends in cooling rates of a DED-processed component, the next step is to improve on their temperature-dependent microstructure prediction capabilities. However, this is limited to the spatial and temporal resolutions in the models. Constant thermal cycling leads to microstructures that are like those in annealed materials, with uncertainty into which cycles determine the resulting microstructure. Though the rapid solidification cooling rates during the DED process have been shown to be about $10^2$ to $10^6$ K/s, microstructures that indicate slow cooling, such as the Laves phase in Inconel 718 or the
Widmanstätten α phase in Ti-6Al-4V, have been observed. Future work would include more in-depth studies in micro-scale thermal monitoring and exactly matching the monitored areas to the localized microstructure of the built material. Control of temperature would also lead to microstructure control, including grain refinement and controlled crystallographic orientations. The scales presented in this work were only on the micro and meso scales. However, many of the characteristics that arise from phase transformations include precipitates, particularly in 316L SS and Inconel 718, that can be in the order of 1-10 nm scale. Additional hierarchical, multi-scale studies on the nano-scale precipitates structures, their formation because of the unique thermal histories, and their influence on mechanical behavior are required.

The next chapter discusses another structural characteristic that is common in DED-processed components – porosity. Both phase transformations and porosity are results of the thermal histories that occur at localized areas, with porosity occurring more from shrinkage or processing inconsistencies. Depending on the material, its density, and function, the porosity structure often competes with the material’s crystallographic structure during mechanical testing. The next chapter continues the characterization of the materials discussed in previous sections.
This chapter discusses how porosity occurs because of the thermal histories in DED processing. The Marangoni flow in the melt pool can lead to formation of entrapped gas pores, rapid directional solidification can lead to shrinkage, and lack of fusion among metallic particles can leave cavities in the processed component. Process parameters can provide insight on the types of porosity in the component and how they form (Section 7.1). Section 7.2 provides an overview of the experimental methods used in this work to determine porosity content and characteristics. The materials discussed in this section are 316L SS (Section 7.3) and Ti-6Al-4V (Sections 7.4 to 7.6), as seen in Fig. 7.1. Section 7.7 provides a summary of the microstructures observed in this work and opportunities for future work.

Figure 7.1. Materials discussed in porosity in DED processing.
7.1 Porosity in laser-processed materials

7.1.1 Influence of process parameters on porosity

Two general categories of porosity types appear during DED processes due to the non-uniform heat transfer during the process: interlayer and intralayer porosity (Wang, Pratt et al. 2009). Literature illustrates that there is a relationship between porosity and global energy density (GED) where interlayer porosity is found at the low extreme of GED values and intralayer porosity is found at the high extreme of GED values for a given process and material (Yan 2015), as seen in Fig. 7.1. GED can be defined as a combination of several process parameters (Kruth, Vandenbroucke et al. 2005):

\[ GED = \frac{Q}{2\nu r_b} \]  \hspace{1cm} (7.1)

where \( Q \) is the laser power in J/s, \( \nu \) is the laser scan speed in mm/s, and \( r_b \) is the laser beam radius in mm. However, most of the work that use the GED parameter are for powder bed, or selective laser sintering /melting studies where the influence of powder mass flow is not captured.

Figure 7.2. Relationship between porosity and global energy density in laser additive manufacturing processes.
This work shows that varying process parameters, including powder mass flow, can lead to various varying porosity distributions, shapes, and sizes. Varying process parameters result in varying laser attenuation during the process, which result in differing mechanisms of porosity formation. In the discussion on porosity in Ti-6Al-4V components (Sections 7.4 and 7.5), the hypothesis tested is that different solidification mechanisms and cooling rates during the DED process lead to various porosity distributions.

Previous studies on DED-processed 316L showed that increase in scan speed, an intermediate and stable power feed rate minimized the area fraction of porosity (Majumdar, Pinkerton et al. 2005). However, previous work showed conflicting conclusions concerning the influence of laser power density on porosity. Majumdar et al. suggested that increasing power density had no effect on porosity but resulted in coarsened grain size when coupled with a decrease in scan speed (Majumdar, Pinkerton et al. 2005). Syed et al. suggested that increased laser power density can decrease porosity (Syed, Pinkerton et al. 2005) and a study of DED-processed H13 tool steel revealed that high laser power density also resulted in low porosity levels regardless of powder mass flow rate (Choi and Chang 2005). A quantitative characterization study of porosity in another austenitic steel, 304L SS, demonstrated that components with larger void to particle size ratios tended to result in lower porosity. In addition, allowing gas to escape from the melt pool surface and laser re-melting of the surface layers could also further reduce porosity content (Susan, Puskar et al. 2006).
An example of how solidification cooling influenced porosity characteristics such as shape and size looked at the ordered porosity in the build direction versus the porosity in the scan direction and their influence on the anisotropic mechanical behavior of Ti-6Al-4V components (Sterling, Torries et al. 2016). The study found that there was insufficient fusion and increased rapid cooling between layers (Sterling, Torries et al. 2016). Porosity formation due to solidification during casting of Al-4.5 pct. Cu plates was mathematically modeled using shape descriptors of radius of pore and area fraction (Kubo and Pehlke 1985). A comprehensive study looked at the influence of cooling rate on pore diameter, area fraction of pores, microstructure, and mechanical properties for cast LM13 alloy (Hosseini, Shabestari et al. 2013). The study demonstrated that increasing cooling rate decreased porosity area fraction and size in addition to improving mechanical properties. However, many DED-processed materials have cooling rates between the solidus and liquidus temperatures that range from $10^2$ to $10^6$ K/s whereas cast materials have cooling rates that range from 1 to 100 K/s (Frazier 2014). The anisotropy in tensile behavior of DED-processed, wrought and cast Ti-6Al-4V were compared and were found to be influenced by the interlayer porosity due to crack initiation at pore sites as opposed to intralayer porosity (Alcisto, Enriquez et al. 2011).

### 7.1.2 Lack of fusion porosity

Porosity due to the lack of fusion is interlayer or inter-hatch porosity at the interfaces between each layer or hatch spacing of melted powder (Cunningham, Narra et al. 2016). This is a result of the discrepancy in temperature distribution within the melt pool, including the unwanted presence of un-melted powder particles that flow into the underlying, colder layers (Cunningham, Narra et al.
2016). Lack of fusion porosity occurs when there is an insufficient overlap of the rapidly cooling liquid-solid interface (Tang, Pistorius et al. 2017), which typically results from laser power values that are too low, laser scan speeds that are too high, powder mass flow rates that are too high, or a combination of the factors. Other characteristics of lack of fusion porosity includes irregular and elongated shape that appears periodically, depending on the layer thickness or hatch spacing (Kobryn and Semiatin 2001).

7.1.3 Entrapped gas porosity
Intralayer porosity is a result of vaporization within the melt pool, which can entrap gas, such as oxides, into the component (Ng, Jarfors et al. 2009). X-ray computed tomography has demonstrated the presence of interlayer and intralayer porosity in DED-processed stainless steel and titanium alloys and that altering process parameters influence the size, shape and distribution of the pores, especially entrapped gas pores (Ng, Jarfors et al. 2009). The size and shape of these pores are anisotropic due to the subsequent layers of material re-melting and the surfaces of a component undergoing more convection and radiation of heat (Ng, Jarfors et al. 2009). Gaseous porosity and voids in DED-processed metal components can result in decreased strength and overall degradation of mechanical properties. Instability in powder flow, which results in fluctuating laser attenuation, contributes to entrapped porosity. A study (Ng, Jarfors et al. 2009) showed that the powder stream can trap the delivered shielding gas into the melt pool and increased with greater convective flow in the melt pool due to higher laser power. Additional studies (Gong, Rafi et al. 2014), (Zhao, Wang et al. 2009) also showed that disturbed thermocapillary powder flow contributed to high porosity with pore sizes approximately one to three µm and less refined
dendrite structure. The average porosity values among the studies were about 5.0% with pore sizes ranging from 1 to 60 µm in diameter (Gong, Rafi et al. 2014). The entrapped gas porosity can be composed of oxygen contamination from insufficient sealing of the environment but is more commonly composed of the inert gas, such as argon, used for the carrier, shield, or environment gas.

The Marangoni-driven flow in the melt pool overcomes the flotation effect, usually retaining any of the formed entrapped gas porosity within the melt pool (Ng, Jarfors et al. 2009). The convection and geometry of the melt pool determine the flow direction of the pores within the melt pool. In some instances, when the solidification rate is low enough, entrapped gas porosity rises to the surface of the melt pool and escapes before solidification occurs (Ng, Jarfors et al. 2009). Entrapped gaseous porosity has been linked to moisture of the powder particle surface, porosity within the powder particles, and an increasing solidification rate that does not allow for the entrapped porosity to escape. Entrapped gas porosity resides within the melt pool boundaries after solidification. Other characteristics include a spherical shape, with sizes of usually less than 10 µm in most applications, though entrapped porosity of up to 50 or 100 µm in diameter have been observed (Gong, Rafi et al. 2014).

7.1.4 Keyhole porosity

Chapter 3 introduces the concept of keyhole laser processing, where the laser power is high enough and the scan speed is low enough to vaporize the underlying the substrate. Vaporization of the metallic substrate, or underlying deposited powder layer, induces a cavity that penetrates deep into
the substrate and is surrounded by the melt pool (Ly, Rubenchik et al. 2017). When the melt pool cools near the bottom, shrinkage occurs and keyhole porosity well below the melt pool surface forms. The keyhole pore can collapse during solidification but can also be filled with shielding inert gas or metallic vapor. Though keyhole porosity is like entrapped gas porosity, it is formed during vaporization of the underlying material (Lancaster 1984).

7.2 Experimental methods to characterize porosity

7.2.1 Archimedes method

The Archimedes method (Spierings, Schneider et al. 2011) can measure the bulk porosity volume fraction and the overall density of a DED-processed component. The specific type of Archimedes measurement used in this work was the suspension technique. For the 316L SS Charpy parts, the Archimedes method used for each specimen five times. For the Ti-6Al-4V cubic component study, the Archimedes method was used for each of the three cubic components prior to machining and three times for each tensile specimen prior to tensile test for a total of 123 complete measurements. The resolution of the Archimedes method for each specimen was 0.001 g/mm$^3$, with the volume of each specimen less than 50 mm$^3$. The accuracy of this method has been demonstrated to be 0.03% for volumes less than 50 mm$^3$, which is comparable to the accuracy of pycnometry, at 0.01% (Hughes 2005). After failure, porosity and density were measured three times using the Archimedes method for both halves of each Charpy or tensile specimen. The mean values and standard deviations of porosity and density values for each Charpy or tensile specimen were evaluated. Negligible differences of about 0.1% between the measurements before and after the impact or tensile tests were observed.
Compared to other density and porosity measurement methods for additive manufactured parts, the Archimedes method has demonstrated superior repeat accuracy of components with high density (i.e., above 95% density) and is the most reliable and cost-effective method to measure the density of entire parts (Spierings, Schneider et al. 2011). Though the 316L SS Charpy parts exhibited high levels of porosity, the density of each Ti-6Al-4V part examined in this work was above 95%. The drawbacks of the Archimedes method include possible variations in measurements due to air bubbles that adhere to the surface of the part and is exacerbated by increases in surface roughness (Hughes 2005). In addition, the presence of surface porosity can introduce water to flow into the part if they are not appropriately sealed, which would skew the density measurement (Hughes 2005). The parts measured did not exhibit surface porosity. Surface roughness was not an issue because the parts were machined from the AM component using wire-EDM with stress relief.

7.2.2 Microscopy

Evaluated cross-sections of the observed specimens were cut from the tensile specimen grip area of the Ti-6Al-4V specimens using an aluminum silica blade. The resolution of the 2D optical microscopy (OM) method was determined by the pixel size of the micrographs, which was 0.65 µm by 0.65 µm. The accuracy of the 2D OM method by thresholding each individual micrograph is greater than that of an automatic global thresholding method where the same threshold cutoff value to identify pores is applied to all micrographs. In addition, the coefficient of variation of the 2D OM method is less than 0.1 mm/s with increasing accuracy with greater sample density.
Hughes 2005). The drawbacks of the 2D OM method includes potential inconsistency of imaging thresholding among various micrographs, micrographs that are not representative of a part and only capture two dimensions of porous structure, and the time required to section and polish at many locations.

Image analysis of 2D micrographs provided the area, roundness and nearest neighbor distance of each pore and the area fraction of pores in each micrograph, based on descriptor-based methodology for designing and predicting microstructure material systems for polymer nanocomposites (Xu, Li et al. 2014). Past work on polymer nanocomposites identified key descriptors of nanoparticles such as dispersion, geometry and composition and used the simulated annealing algorithm to reconstruct microstructure maps for finite element (FEA) analysis of mechanical behavior (Xu, Li et al. 2014). This work identifies descriptors for pores in metallic alloys to reconstruct microstructure maps for FEA and takes this a step further to link the descriptors to processing conditions and solidification behavior.

7.2.3 Micro-computed X-ray tomography
Micro-computed X-ray tomography uses a bending magnet X-ray source with an energy range of 11 to 35 keV. The part is mounted on a rotary platform with X, Y, and Z alignment motors. As the part rotates, the X-ray penetrates the material, providing cross-sectional images along the height of the part. The micro-computed X-ray tomography method used was conducted at sector 2-BM at the Advanced Photon Source (APS) in Argonne National Laboratory. With a field of view of 2
by 2 mm in each cross-section, with the observed length up to 20 mm, the resolution is about 1 µm.

7.2.4 High-speed X-ray imaging

High-speed X-ray imaging at the 32-ID beamline at APS also observed entrapped porosity due to Marangoni convection and keyhole porosity due to vaporization. This work used the high-resolution, time-resolved X-ray imaging technique at the Advanced Photon Source to evaluate the laser-matter interactions in DED, as detailed in Chapter 3.

7.3 Porosity versus GED in 316L SS Charpy parts

Several 316L SS Charpy parts were investigated for their porosity values compared to the GED value based on process parameters. Table 7.1 displays the process parameters with calculated and measured energy densities for 12 specimens. Calorimetry measurements add calibration done by Northern Illinois University determined the measured energy densities, which include the influence of the powder mass flow rate. Table 7.2 lists the porosity and density measurements for the corresponding specimens using the Archimedes and image analysis methods. Overall, image statistical analysis results were consistent with Archimedes results.
Table 7.1. Process parameters for DED-processed 316L SS Charpy specimens.

<table>
<thead>
<tr>
<th>Part #</th>
<th>Laser power output (W)</th>
<th>Laser beam diameter (mm)</th>
<th>Laser scan speed (mm/s)</th>
<th>Powder flow rate (mg/s)</th>
<th>Theoretical GED (J/mm$^2$)</th>
<th>Net heat input (J/mm$^2$)</th>
</tr>
</thead>
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<tr>
<td>1</td>
<td>290</td>
<td>1.5</td>
<td>12.7</td>
<td>112.5</td>
<td>15.2</td>
<td>-</td>
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<td>2</td>
<td>234</td>
<td>1.5</td>
<td>11.9</td>
<td>112.5</td>
<td>13.1</td>
<td>-</td>
</tr>
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<td>112.5</td>
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<td>8.5</td>
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<td>49.8</td>
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<td>8.5</td>
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<td>167.7</td>
<td>48.4</td>
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</tbody>
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Table 7.2. Comparison of porosity and density techniques and measurements.

<table>
<thead>
<tr>
<th>Part #</th>
<th>Archimedes density (kg/m$^3$)</th>
<th>Archimedes porosity (%)</th>
<th>Archimedes porosity standard deviation (%)</th>
<th>Binary image analysis porosity (%)</th>
</tr>
</thead>
<tbody>
<tr>
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<td>7953</td>
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<tr>
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</table>

Figure 7.3 plots the porosity measurements carried out by the Archimedes method as a function of the net heat input energy density, which considers the theoretical GED and measured calorimetric
results. Fig. 7.3 reveals a curve like that of the hypothesized GED versus porosity curve based on lack of fusion or gas entrapment as causes for higher porosity in Fig. 7.2. This shows that the optimal range of net heat inputs for minimized porosity content in 316L SS rectangular components was about 25 to 35 J/mm$^2$ for porosity values under 0.20%. Lack of fusion left to the optimal range was most probably due to increased scan speed and powder mass flow, which did not allow for effective particle bonding, as seen in Figure 7.4. Slower laser scan speeds could have led to gas entrapment, or higher porosity levels to the right of the optimal range. Figure 7.4 shows the fracture surface of Charpy part #10, which had the highest porosity content due to the lack of fusion between layers. Un-melted powders at the fracture surface and between layers led to damage and variation of mechanical properties throughout the part.

![Figure 7.3. Energy density versus porosity content of Charpy parts 4-12.](image-url)
Figure 7.4. Fracture surface of part 10 with the vertical axis representing the build direction and the horizontal axis representing the hatch direction.

7.4 Global porosity characteristics in Ti-6Al-4V cubic components

To establish relationships between porosity and resulting mechanical behavior in each tensile specimen, Generalized Mixture Rule (GMR) analysis was used for the tensile specimens machined from the Ti-6Al-4V cubic components detailed in Chapter 5. The GMR is a fundamental composite theory that aims to mathematically define the relationship between a poly-phase structure and the resulting mechanical properties. For porous materials, the GMR can reflect the random nature of the solidified microstructure with porosity distribution with properties like elastic constants, yield strength, and ultimate tensile strength (Ji, Gu et al. 2006). The mechanical properties analysis of this global study continues in Chapter 8.

7.4.1 Using generalized mixed rule and J-Factor

The fractal parameter $J$ is a numeric value that applies to isotropic materials and is controlled by the distribution of the phases. This fractal parameter is a key factor in these relationships in that $J$ captures the continuity and connectivity of the pores in the material (Wolff, Lee et al. 2016). This
fractal parameter is controlled by the shape, size distribution and spatial distribution of the pores. The general GMR equation is:

$$M_c = (1 - V_w)M_s + V_w M_w$$  \(7.2\)

where \(M_c\) is the mechanical property of the overall composite, \(V_w\) is the volume of the porous phase, \(M_s\) is the mechanical property of the bulk, single phase material, and \(M_w\) is the mechanical property of the second phase, which in this case, is air and where the properties are equivalent to zero. For more specific porous materials, the following equations apply where Eqn. 7.3 is an approximation for materials for very low porosity values.

$$\frac{M_c}{M_s} = (1 - \rho)^{\frac{1}{J}} = V_s = \left(\frac{\rho_c}{\rho_s}\right)^{\frac{1}{J}}$$  \(7.3\)

$$\frac{M_c}{M_s} = 1 - \frac{1}{J} \rho$$  \(7.4\)

The use of curve-fitting \(J\) factor in GMR relationships was to predict either the mechanical behavior or microstructure by having information of the other. This work aimed to determine the \(J\)-factor based on the mechanical test results and the porosity measurements from 2D optical microscopy images. The pores that were evaluated were the interlayer pores due to the lack of fusion, as they contributed to an increase of porosity and led to mechanical failure. These pores were assumed to be ellipsoids, or ellipses in 2D image analysis. Figure 7.5 shows the flow chart to determine the \(J\)-factor and its link to mechanical properties. This included determining a thermal strain parameter that incorporated the process parameters, especially laser power, and using this thermal strain parameter to determine the ellipsoid pore dimensions.
7.4.2 Determination of thermal strain parameter

To account for the influence of process parameters, dimensional inaccuracy due to thermal distortion during the process was calculated to determine the amount of lack of fusion that would cause porosity (Mukherjee, Zuback et al. 2016). A non-dimensional thermal strain parameter, $\varepsilon^*$, was calculated based on the Buckingham $\pi$-theorem and by using the process parameters as variables (Bird 2002). Table 7.3 lists the variables and their dimensions in the MLT system.
Table 7.3. Variables used in the dimensional analysis for thermal strain parameter.

<table>
<thead>
<tr>
<th>Variable – Material Properties</th>
<th>Dimension</th>
</tr>
</thead>
<tbody>
<tr>
<td>Volumetric thermal expansion coefficient, ( \beta )</td>
<td>( \theta^{-1} )</td>
</tr>
<tr>
<td>Temperature gradient, ( \Delta T = T_{\text{liquidus}} - T_{\text{solidus}} ), which refer to the temperature difference between the liquidus and solidus temperatures of Ti-6Al-4V</td>
<td>( \theta )</td>
</tr>
<tr>
<td>Characteristic time scale for solidification cooling, ( \tau )</td>
<td>( T )</td>
</tr>
<tr>
<td>Thermal diffusivity, ( \alpha = \kappa/\rho_{\text{powder}}C_P ), where ( \kappa, \rho_{\text{powder}}, C_P ) are thermal conductivity, density of powder and specific heat for Ti-6Al-4V, respectively</td>
<td>( L^2 T^{-1} )</td>
</tr>
<tr>
<td>Absorption coefficient, ( \eta )</td>
<td>Dimensionless</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Variable – Process Parameters</th>
<th>Dimension</th>
</tr>
</thead>
<tbody>
<tr>
<td>Heat input per length, ( H = \eta Q/\nu ) where ( \eta, Q, \nu ) refer to the absorption coefficient, laser beam power (W) and the scan speed of the laser beam (mm/s)</td>
<td>( \text{MLT}^{-2} )</td>
</tr>
<tr>
<td>Volumetric powder flow, ( \nu = q/\rho_{\text{powder}} ), where ( q ) is the mass powder flow and ( \rho_{\text{powder}} ) is the density of Ti-6Al-4V powder, which is about 60% of bulk Ti-6Al-4V</td>
<td>( L^3 T^{-1} )</td>
</tr>
<tr>
<td>Cross-sectional area, ( A ) of the ellipsoid melt pool, with volume ( V = \frac{4}{3} \pi * (l) * (2l - h) * \left( \frac{Q}{\nu(2l-h)} - t \right) ), where ( l ) is the laser beam diameter and the length of the melt pool in the scan speed direction, ( 2l - h ) is the width of the melt pool in the hatch direction with ( h ) hatch spacing, and ( \frac{Q}{\nu(2l-h)} - t ) the depth of the melt pool with ( t ) layer thickness</td>
<td>( L^2 )</td>
</tr>
<tr>
<td>Width, length and height of the cubic component, ( s )</td>
<td>( L )</td>
</tr>
</tbody>
</table>

The resulting thermal strain parameter was expressed as a function of the DED process parameters and material thermal properties (Mukherjee, Zuback et al. 2016):

\[
\varepsilon^* = \frac{\beta \Delta T A}{\nu a^2 s^2 \sqrt{\rho_{\text{powder}}}} H^{3/2}
\] (7.5)

Table 7.4 provides the resulting thermal strain parameters for each load axis, \( e \), in a cubic component, for the three laser processing powers. Each load axis had a different cross-sectional area for the melt pool, which influenced the weight of the parameter. The thermal strain parameter was a weighting factor to \( J \)-factor calculations.

Table 7.4. Thermal strain parameters for the various laser power and directions in %.

<table>
<thead>
<tr>
<th>Principal load axis</th>
<th>710 W</th>
<th>800 W</th>
<th>940 W</th>
<th>( C_1 \varepsilon^2 + C_2 \varepsilon^* + C_3 = )</th>
</tr>
</thead>
<tbody>
<tr>
<td>( e_x )</td>
<td>0.217</td>
<td>0.260</td>
<td>0.331</td>
<td>( b^2 + c^2 )</td>
</tr>
<tr>
<td>( e_y )</td>
<td>0.286</td>
<td>0.343</td>
<td>0.436</td>
<td>( a^2 + c^2 )</td>
</tr>
<tr>
<td>( e_z )</td>
<td>0.543</td>
<td>0.650</td>
<td>0.827</td>
<td>( a^2 + b^2 )</td>
</tr>
</tbody>
</table>
7.4.3 J-factor analysis

To determine how process parameters influenced the $J$-factor and to determine the $J$-factor itself, 2D optical micrographs were captured for each tensile specimen. Porosity in the 2D micrographs was investigated for their statistical distributions in roundness, circularity and their distances from one another. Though pores were aligned in binary images for the “A” and “B” orientations, the characteristics of circularity, roundness and distribution varied in the localized area. By using the approximation of the Taylor series in the GMR relationship in Eqn. 7.3., the $J$-factor was calculated for each data set by knowing the bulk porosity values and the ultimate tensile strength (UTS).

By calculating these three independent variables for hundreds of pores for various tensile specimens in each power and orientation, probability distribution curves were determined for each variable to determine the a, b, and c dimensions of the ellipsoid pores in the various orientations. By using values and the thermal strain parameters, unknown constants were determined to solve for the $J$-factor of the various laser powers and orientations.

This study continues in Chapter 8 to discuss the influence of the porosity structures on mechanical behavior. In this chapter, an investigation on characteristics of individual pores in the tensile specimens was conducted to analyze pore’s contribution to the GMR and J-factor analysis for failure predictability.

Overall, an in-depth investigation of 2D optical micrographs to make calculations on each pore can be insightful as to how the size and shape of the pores vary with laser processing power,
orientation and location within the cubic component. These relationships would link process parameters and porosity, and are discussed in the next section, which provides a localized analysis of the Ti-6Al-4V cubic components.

7.5 Localized porosity characteristics in Ti-6Al-4V cubic components

7.5.1 Overview of need for localized characterization

Expanding on the global study of porosity in the Ti-6Al-4V specimens, the range in porosity characteristics and cooling rates within each component motivated the need for a localized study. Based on the hypothesis that different solidification cooling rates during a DED build of a Ti-6Al-4V component lead to various porosity distributions, a localized study that bridged thermal histories from GAMMA and 2D binary micrographs was conducted.

The anisotropy in tensile behavior of DED-processed, wrought and cast Ti-6Al-4V is influenced by the interlayer porosity due to crack initiation at pore sites as opposed to intralayer porosity (Alcisto, Enriquez et al. 2011). The tensile behavior in DED-processed Ti-6Al-4V has been shown to depend on the orientation of the build and the location of a specimen relative to the substrate (Carroll, Palmer et al. 2015). In summary, previous studies have shown that DED-processed Ti-6Al-4V components exhibit porosity and strength anisotropy. Modeling efforts in electron beam additive manufacturing evaluated the thermal evolution of the melt pool of a powder bed fused part with varying initial porosity levels (Shen and Chou 2012). Several studies have predicted microstructural characteristics, such as dendritic spacing of stainless steel alloys, using cooling rate (Thijs, Verhaeghe et al. 2010). Evaluating porosity in a DED-processed component is a simple
method to evaluate a microstructural characteristic and its link with cooling. This forms the crux of a proposed framework that demonstrates the relationships between cooling and porosity.

This localized study proposes a framework to demonstrate the influence of the solidification cooling rate on strength and porosity anisotropy. Recent studies in AM lack more descriptive pore characteristics while investigating the relationships between process parameters and mechanical properties. Linking process parameters and mechanical properties emphasizes the need for an understanding of localized properties because of the high variability and anisotropy of a DED-processed component. The proposed framework introduced in Chapter 5 (Wolff, Lin et al. 2017) couples computational simulations with experimental results to predict localized porosity, aiming to establish the relationship between cooling rate and pore shape descriptors.

7.5.2 Dependence on solidification cooling

Binary micrographs were taken at localized areas aligned with the probed points with calculated solidification cooling rates from the thermal model, as shown in Fig. 7.6. The size, shape and distribution of the individual, aligned interlayer pores in the cubic component led to proper characterization of the relationship between the DED process parameters and mechanical behavior. Each pore was assumed to be an ellipsoid and had semi-principal axes lengths of a, b, and c per the XYZ coordinate system.

The dimensions of the pores changed with direction and location within the cubic component. Due to more frequent re-melting, porosity at bottom layers tend to be more irregularly shaped and less
common than those in top layers and locations closer to the surface of the component. As seen in Fig. 7.6, many areas of the component exhibited aligned porosity with layer thickness spacing, hatch spacing, or both. Pores in Fig. 7.6 also ranged from irregularly shaped on the left side toward more spherical pores. Though the cubic component had a relatively simple geometry, the heat transfer and resulting solidification cooling behavior at every point in the build were highly variable and complex, particularly at the start and end points of the tool path within a layer.

![2D porosity map](image)

Figure 7.6. 2D porosity map at the same point the solidification cooling rate was calculated in Fig. 5.20 in Chapter 5.

DED processing of more complex geometries require an understanding of the microstructure and solidification cooling behavior at every area to guarantee structural integrity. Localized study on cubic components is a step into gaining insight into how porosity microstructure and solidification cooling are linked. By linking the predicted cooling rate, pore shape and dispersion descriptors, a gradient of interlayer and intralayer porosity appeared as solidification cooling rate increased. Larger, irregularly shaped interlayer pores appeared when the solidification cooling rate increased due to lack of fusion and thermally-driven volume change at the melt pool. On the other hand, there were fewer interlayer pores, leading to a decreased area fraction of pores and increased
nearest neighbor distance as solidification cooling rate increased. These relationships are illustrated in Fig. 7.7. Though the GAMMA thermal histories over-predicted the cooling rates at the localized areas of the DED-processed Ti-6Al-4V component, the trends in cooling are assumed to be consistent. However, the inaccuracy in cooling rate values could influence the results in Fig. 7.7 in that the range of “real” cooling rates would be more narrowed as “real” processing undergoes less re-melting of the underlying layers due to powder flow attenuating the laser.

![Figure 7.7](image)

Figure 7.7. Relationship between simulation-determined solidification cooling rate and experimentally-determined (a) pore size, b) pore roundness, c) area fraction of porosity and d) dispersion of pores measured using average nearest neighbor distance (NND).

The fit types for Fig. 7.7 were tested by calculating residuals and goodness of fit tests like RSME. The fit types were reasonable with the experimental data because of the relationship between
increasing solidification rate and the exponential decay of solute, or pore concentration relative to the distance from the solid-liquid interface during solidification (Davis 2001), (Galenko and Jou 2005). The shape descriptors of the pores, including the number of pores and pore shape, are related to the spatial and temporal concentration of pore concentration and behavior that changes with solidification cooling.

Figure 7.7a plots the average pore size at each point where the solidification cooling rate of the last re-melting cycle was probed. As the solidification cooling rate increased, the average pore size also increased most probably due to the presence of more irregularly shaped, lack of fusion pores. In addition, the standard deviation of the pore size also increased with increasing solidification cooing rate, as the heterogeneity of the porosity microstructure increased. The relationship between the experimentally-determined average pore size and computationally-determined solidification cooling rate was found to have a logistic growth function, defined by a shape descriptor, $S_{\text{size}}$ in the following equation:

$$S_{\text{size}} = \frac{A_{\text{size}}}{1 + B_{\text{size}} \exp \left( -C_{\text{size}} \frac{d \tau_{l-s}}{d t_{l-s}} \right)}$$  \hspace{1cm} (7.6)

where $\frac{d \tau_{l-s}}{d t_{l-s}}$ is the calculated solidification cooling rate between the liquidus and solidus temperatures. Parameters for the logistic growth function are given in Table 7.5.

<table>
<thead>
<tr>
<th>Pore size parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>$A_{\text{size}}$</td>
<td>$7.00 \times 10^3 \mu m^2$</td>
</tr>
<tr>
<td>$B_{\text{size}}$</td>
<td>$1.40 \times 10^2$</td>
</tr>
<tr>
<td>$C_{\text{size}}$</td>
<td>$3.65 \times 10^{-3}$</td>
</tr>
</tbody>
</table>
The logistic growth function is a statistical model that predicts a period of growth as a descriptor value transitions from exponential growth to slow growth once a saturation limit is reached. Physical limits in the DED process, the solidification velocity due to diffusion of solute in the liquid Ti-6Al-4V, slows the growth of the pore size descriptor and dominates the saturation limit (Altgilbers, Hofmeister et al. 2003). The logistic growth function has been used in solidification modeling, including those that incorporate neural networks (Santos, Fortaleza et al. 2005) and smooth modeling based on the latent heat evolution approach to evaluate the volume liquid fraction function during solidification (Tavakoli 2017).

Figure 7.7b plots the average pore roundness at each evaluated solidification cooling rate. As the solidification cooling rate increased, the average pore roundness decreased because of the increased irregularly shaped pores. The relationship between the experimentally-determined average pore roundness and computationally-determined solidification cooling rate was an exponential decay function. Exponential decay relationships are observed in heat transfer applications and in the concentration of liquid media in binary and ternary alloys during solidification (Tiller, Jackson et al. 1953). The exponential decay function for pore roundness during solidification is defined by a shape descriptor, $S_{\text{round}}$, in the following equation with parameters in Table 7.6:

$$S_{\text{round}} = A_{\text{round}} \exp \left( -B_{\text{round}} \frac{dT_{l-s}}{dt_{l-s}} \right)$$  \hspace{1cm} (7.7)
Table 7.6. Pore roundness parameters for exponential decay curve fit.

<table>
<thead>
<tr>
<th>Pore roundness parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>( A_{\text{round}} )</td>
<td>1.6</td>
</tr>
<tr>
<td>( B_{\text{round}} )</td>
<td>( 1.2 \times 10^{-3} )</td>
</tr>
</tbody>
</table>

Figure 7.7c plots the porosity area fraction at each evaluated solidification cooling rate. As the solidification cooling rate increased, the number of pores decreased due to sufficient melting and fusion of the titanium alloy powders. The relationship between the experimentally-determined porosity area fraction and computationally-determined solidification cooling rate was an exponential decay function. The curve fit suggests that the porosity in the Ti-6Al-4V component built with the given process parameters did not achieve less than a 0.2% area fraction. Hot isostatic pressing (HIP'ing) is required for a denser part, though an optimized combination of process parameters may achieve full density. A previous study on porosity in cast aluminum alloy (Gaviphatt, Puncreobutr et al. 2015) suggested that gas diffused porosity follows an exponential decay function because with increasing cooling rate, the less time entrapped gas can diffuse into pores. The exponential decay function for porosity area fraction during solidification is defined by a shape descriptor, \( S_{\text{frac}} \) in the following equation with parameters in Table 7.7:

\[
S_{\text{frac}} = A_{\text{frac}} \exp \left( -B_{\text{frac}} \frac{dT_{l-s}}{dt_{l-s}} \right) + C_{\text{frac}} \quad (7.8)
\]

Table 7.7. Porosity area fraction parameters for exponential decay curve fit.

<table>
<thead>
<tr>
<th>Pore area fraction parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>( A_{\text{frac}} )</td>
<td>( 7.00 \times 10^{-2} )</td>
</tr>
<tr>
<td>( B_{\text{frac}} )</td>
<td>( 1.261 \times 10^{-3} )</td>
</tr>
<tr>
<td>( C_{\text{frac}} )</td>
<td>( 2.000 \times 10^{3} )</td>
</tr>
</tbody>
</table>
Figure 7.7d plots porosity dispersion in terms of nearest neighbor distance for every pore at each evaluated solidification cooling rate. As the solidification cooling rate increased, the distance between each pore increased due to the number of pores decreasing. This suggests that less spherical pores due to entrapped gas and more irregularly shaped pores due to lack of fusion occurred at greater cooling rates. Gas entrapped pores were not restricted to aligned spacing of layer thickness or hatch spacing, and therefore, were found to be closer to other pores. The relationship between the experimentally-determined porosity dispersion and computationally-determined solidification cooling rate was a logistic growth function. This function is related to the pore size, roundness and area fraction, as the distance between each pore depends on these shape descriptors. The physical limits that determines the saturation value are the process parameters, most notably the layer thickness and hatch spacing where more pores due to lack of fusion occur. The logistic growth function for porosity dispersion during solidification is defined by a shape descriptor, $S_{disp}$ in the following equation with parameters in Table 7.8:

$$
S_{disp} = \frac{A_{disp}}{1+B_{disp} \exp\left(-C_{disp} \frac{dt}{\text{th}}\right)} + D_{disp}
$$

(7.9)

<table>
<thead>
<tr>
<th>Pore dispersion parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>$A_{disp}$</td>
<td>$6.566 \times 10^2 \mu m$</td>
</tr>
<tr>
<td>$B_{disp}$</td>
<td>$1.835 \times 10^4$</td>
</tr>
<tr>
<td>$C_{disp}$</td>
<td>$1.100 \times 10^2$</td>
</tr>
<tr>
<td>$D_{disp}$</td>
<td>$1.484 \times 10^2 \mu m$</td>
</tr>
</tbody>
</table>

Localized analysis of the porosity descriptor dependence on solidification cooling completed the second link in the framework to connect cooling rate and porosity. Relationships between porosity
descriptors and process parameters controlled characteristics, such as solidification cooling rates, are applicable for prediction purposes. The ability to probe the cooling rate of DED-processed components with complex geometries would aid the prediction of types of porosity and its descriptors in specific locations of the component. Furthermore, knowledge of porosity and its descriptors can provide statistically representative elements to predict mechanical behavior at localized areas.

Limitations to the analysis that evaluated the relationships between porosity descriptors and solidification cooling are that there are nuances in both experimentally and computationally determined data. Porosity presented in 2D micrographs may not be fully representative of the localized microstructure of the part due to differences in the machine environment during a build. The thermal model presented in this study was not calibrated or validated yet and only served as a qualitative means of comparison. The presence of $\alpha$, $\beta$, and $\alpha'$ (martensitic) phases in the microstructure can provide more information on changes in porosity and the resulting mechanical behavior. However, identifying relationships between cooling and microstructure provides a valuable step in the overall proposed framework. This is particularly vital with DED-processed components with complex geometries and varying thermal histories and structure.

Though the presented framework reveals porosity characteristics at a local level (within a layer and hatch spacing), the mechanisms as to how porosity form was not fully captured. The GAMMA thermal model that provided the cooling rate information assumed that powder flow rate and laser attenuation remained constant during DED processing. Overall, the framework provides an
efficient *ex-situ* characterization study. However, porosity formation and characteristics within the melt pool area were captured in real-time, as detailed in the next section. High-speed X-ray imaging allowed for the observation on how mass addition, mass subtraction, and overall laser attenuation influence porosity formation.

### 7.6 In-situ porosity characterization during Ti-6Al-4V piezo deposition

X-ray imaging captures the formation of defects in real-time and shows how processing conditions influence defect evolution. Varying process conditions can change cavity depth evolution in relation to mass addition, mass subtraction, vapor plume formation, and porosity formation. The different modes of processing conditions included the keyhole mode and the conduction mode. The keyhole mode in DED resulted in keyhole porosity and the conduction mode resulted in some entrapped gas and balling of unfused particles.

#### 7.6.1 Marangoni convection behavior of the melt pool

The interaction of the laser beam and substrate created a melt pool with Marangoni convection that entrained particles into and out of the melt pool, behaving similarly to laser-matter interactions in powder-bed systems. For example, the introduction of cold particles into a stirring melt pool can lead to instability and growth of the melt pool. The surface tension of the melt pool introduced a Marangoni flow that entrained nearby flowing or stationary particles with low vertical momentum relative to the substrate surface.
The frames in Fig. 7.8 are processed images where the raw data was divided by the initial frame. High intensity, or light, pixels that represent particles above the substrate surface were artifacts from image processing, where a particle was in place in the initial frame. Low intensity particles above the substrate surface show a moving particle that was present in the frame and not an artifact. The intensity contrasts in the images around the melt pool revealed the phase state of the Ti-6Al-4V in the experiment. These contrasts depended on the density of the material at different phases. The melt pool above the substrate surface was represented by dark intensity pixels, indicating the contrast of the liquid melt pool against the low density, lighter intensity ambient air pixels. The liquid melt pool below the substrate surface had slightly lighter intensity pixels than the surrounding, slightly denser solid material, with the red outlines in Fig. 7.8 revealing the liquid-solid interface of the melt pool. The blue outlined the gas-liquid interface, or the cavity, where the light intensity pixels represented the vapor phase of Ti-6Al-4V. When porosity formation occurred, light intensity pixels that represent a vapor phase of pore appeared below the substrate surface and within the liquid-solid interface.
Figure 7.8. Cavity, melt pool and porosity evolution as mass was added due to surface tension in an experiment with a laser power of 150 W and a scan speed of 100 mm/s.

The frames from 450 to 600 µs in Fig. 7.8 are outlined in green to indicate when the particle entered and stirred within the melt pool. The geometry of the cavity fluctuated when a particle entered the melt pool. The vapor-plasma plume entrained a particle into a trajectory that followed the laser scan direction until the particle contacted the surface of the melt pool. At that point, as seen at 450 µs, surface tension entrained the particle into the melt pool due to the particle's low vertical momentum.

In Fig. 7.8, porosity formation was tracked by an identifier, where the first number was a unique ID of a pore in chronological order and the second number was the lifetime of the pore in terms of number of frames. For example, the label “2-1” at 400 µs was the second pore to appear during the process and 400 µs was the first frame for that pore to appear. At 500 µs, “2-2” dissolved into the cavity, resulting in the unique second pore present for two frames during the process. Because
a span of 50 µs passed between each frame, the lifetime of “2-2” could have been between 50 and 150 µs. Pore “2” decreased in size from 325 to 312 µm² until it dissolved in the melt pool. Figure 7.8 shows that as particle mass entered the melt pool, there was laser attenuation into the melt pool. Laser attenuation led to a decrease in laser-induced cavity depth, the formation of porosity below the cavity, and overall fluctuations in melt pool geometry. As a particle melted into the melt pool, the cavity and melt pool regained their stable geometries. The melt pool either engulfed the porosity below the cavity or porosity stirred within the melt pool and dissolved into the surrounding molten Ti-6Al-4V material. Pore “3” decreased in size from 490 to 47 µm² until it dissolved into the melt pool at 650 µs and pore “4” maintained its size of about 30 µm² until it also dissolved at 650 µs.

Spattering arose from large pressure gradients induced by the vapor-plasma plume and aided in the stability of the melt pool during the process. Spattering led to the ejection of hot or molten particles from the melt pool that could deposit on other area of the build, leading to surface defects or increased roughness. An example of the mass subtraction via melt pool ejection is presented in Fig. 7.9 which was during the same experiment as shown in Fig. 7.8 but 600 µs after the particle entered the melt pool. Figure 7.9 also shows the processed images to reveal the change in phase contrast during the process. As seen in Fig. 7.9 at 1450 µs, three hot particles about 10 µm in diameter or smaller were ejected from a volatile melt pool to the right of the cavity. The direction of the particle ejection was in the same direction of the laser scan with speeds of up to tens of m/s. During ejection, the recoil pressure from the vapor-plasma plume arising from the cavity caused the instability of the cavity geometry, increased the cavity depth, and caused spattering of hot
particles. Afterwards at 1500 µs, the cavity and melt pool geometry regained stability, although some porosity from 100 µs prior remained in the melt pool.

**Laser Scanning**

![Figure 7.9. Cavity, melt pool and porosity evolution as mass was subtracted due to spattering in an experiment with a laser power of 150 W and a scan speed of 100 mm/s. All frames are processed images.](image)

During spattering at 1,450 µs, the cavity engulfed porosities “12-1” and “13-1, enlarging the width and depth of the cavity. The sizes of pores “11” and “14” increased during spattering at 1,450 µs, where pore “11” increased from 217 to 271 µm² and pore “14” increased from 57 to 72 µm². After spattering at 1,500 µs, the pore sizes decreased to 169 µm² for pore “11” and to 42 µm² for pore “14”. Though pores “11” and “14” shrunk in size, they did not dissolve into the melt pool or become engulfed by the cavity until about 200 to 300 µs after spattering.

**7.6.2 Formation and evolution in keyhole mode melting**

At high laser power and slow scan speed, the laser interacted with the substrate to create a keyhole mode. In keyhole mode experiments, the laser beam induced a cavity, or depression zone, of ionized metal vapor into the substrate surface. As explained in the previous sections of this study,
particles entering the melt pool could attenuate the laser beam path. The motivation of this section is to observe if and how laser attenuation during mass addition or subtraction led to instabilities in the melt pool and possible keyhole porosity formation.

Figure 7.10 shows the evolution of the laser-induced cavity during two passes in a keyhole mode experiment with 150 W laser power and 100 mm/s scan speed. The powder delivery driven system used a voltage input of 400 V, a 10% duty cycle for each pulse of vibration, and a frequency of 100 Hz for ten pulses. The second pass began 2.5 ms after the first pass and initiated where the first pass ended. As shown in Fig. 7.10, three phenomena were tracked in addition to the depth of the cavity. White circles denoted instances of mass addition, or when particles entered the melt pool via surface tension, particle interaction or pressure gradients. White diamonds show when mass spattered out of the melt pool. Data points surrounded by dashed squares denote when porosity formed during the process. The time between the two passes where the cavity depth is zero denotes the delay time of 2.5 ms when the laser was turned off. There were eight trials of keyhole experiments with the same conditions described. Though the parameters of the piezo-driven powder delivery device were held constant for all eight trials, the flow of particles was not consistent. Among the eight trials of experiments, there were variations in the number of individual particles flowing into the melt pool, particle sizes, and their interactions with each other. This resulted in varying fluctuations in cavity depth and porosity formation among passes or experimental trials.
Cavity depth in Fig. 7.10 was directly related to laser absorptivity into the substrate, or how the laser attenuated. The dip in laser absorptivity was attributed to both the formation of a laser-induced vapor-plasma plume and flowing particles that flowed into the laser beam path. When particles close to the substrate surface entered the melt pool by surface tension, there was no substantial laser attenuation. However, particles that flowed directly into the melt pool from the piezo-driven powder delivery system were more likely to influence laser attenuation. During the first pass in Fig. 7.10, the cavity depth reached zero at around 600 µs due to a particle of 92 µm in diameter blocking the entire path of the laser beam, which was about 80 µm in diameter. During the second pass, the substrate was at an elevated temperature, compared to the substrate at room temperature during the first pass. This caused the cavity depth to reach its constant maximum value more quickly than it did during the first pass.
In Fig. 7.10, porosity formation, or the initial frame of at least one keyhole pore, typically occurred when the cavity depth decreased dramatically, or collapsed. As discussed in Chapter 3, large decreases of cavity depth coincided with porosity formation as the melt pool shrunk with decreased laser absorptivity. For both passes in all eight trials of experiments, keyhole porosity formed at the end of each pass when the laser turned off. Considering the eight trials of two-pass experiments with the same conditions, keyhole porosity formed 49.6% of the instances the cavity geometry decreased to a local minimum depth and 6.1% of the instances the cavity geometry increased to a local maximum depth. Tracking particle flow into the melt pool can provide an indicator of possible keyhole porosity formation within the melt pool. In addition, tracking cavity depth can indicate whether any nucleated keyhole porosity remains or later stirs into the cavity or melt pool.

Mass addition of particles can destabilize melt pool geometry and cooling behavior. In almost all keyhole mode experiments, keyhole porosity formed when the laser turned off and rapid shrinkage occurred in the melt pool. The formation of keyhole porosity every time the laser turned off reveals the potential debilitating start/stop effect of laser processing on the final structure and mechanical behavior of a DED-processed build. Tracking the geometry of the cavity can aid in observing the instabilities of the melt pool during a build and the likelihood of keyhole porosity formation during mass addition or subtraction.

7.6.2 Formation and evolution in conduction mode melting

When the scan speed increased from 100 mm/s to 500 mm/s, the energy density of the laser onto the substrate over time decreased. The resulting mode of laser-matter interaction changed from
keyhole to conduction, where the substrate material was not vaporized within a deep cavity but created a melt pool via laser conduction. The motivation in this section is to determine whether tracking the cavity depth evolution, mass addition, mass subtraction and porosity formation in conduction mode experiments can reveal how changing processing conditions influences the final DED-processed build. Figure 7.11 shows the evolution of cavity depth during two conduction mode experiments of two passes each with the same exact process parameters. The modified parameters from the keyhole mode experiments in the previous section (7.6.1), include the increased scan speed to 500 mm/s and a decrease in delay time between the two passes from 2.5 ms to 500 µs. The powder-delivery system parameters were the same as those in the previous section; 400 V input, a 10% duty cycle for each pulse of vibration and a frequency of 100 Hz for ten pulses.

![Figure 7.11. Evolution of the laser-induced cavity geometry during two conduction mode experiments with two passes each.](image)
During the conduction mode, the peak in cavity depth consistently reached a peak at about 350 µs and then decreased into a stable depth at about 1,250 µs. The peak consistently occurred at the same time with different trials of conduction mode experiments due to heat transfer delay from the laser beam to the surrounding melt pool relative to the laser beam's scan speed. Both experiments in Fig. 7.11 had in-flight particles that attenuated the laser beam path, with a dramatic decrease in laser absorptivity into the underlying substrate and where the cavity depth reached about 10 µm.

Though particles flowed into the laser beam path, no mass was added into the melt pool during the first pass. The laser-induced vapor-plasma plume at the surface of the substrate caused surrounding particles to scatter. Particles closest to the substrate surface scattered with a velocity between 1.5 and 2.5 m/s, accelerating to 3-4 m/s at 1 mm above the substrate surface. Because of the increased laser scan speed to 500 mm/s, the moving laser beam interacted with in-flight particles with greater frequency. Therefore, the laser-induced localized pressure gradient at particle surfaces scattered particles with greater frequency than a slow-scanning laser in the keyhole mode. Particles that contacted the high-speed laser beam path scattered with velocities from 4-6 m/s, with most particles traversing upward and outward away from the melt pool.

The expanding vapor-plasma plume from the substrate surface scattered any particle from flowing close to the laser beam. In the conduction mode, laser-matter interaction led to decreased laser absorptivity into the substrate and hindered particle flow without the aid of carrier gas. The combination of decreased laser absorptivity into the substrate and fewer particles flowing into the path of the laser beam resulted in no keyhole porosity formation. Thus, the changes in cavity depth
during the second pass in Fig. 7.11 were relatively repeatable without any obstruction to the laser beam. In one of the trials in Fig. 7.11 a pore of about 2 µm in diameter formed during the first pass and quickly stirred into and dissipated into the melt pool. When the cavity depth reached a peak during the second pass, two pores of about 2-3 µm in diameter formed and stirred into the melt pool. These were considered temporary gas entrapped pores, where the slow laser scan speed allowed for surrounding gas to enter the melt pool. One of the micro-pores traversed toward the substrate surface and dissipated. The solidification front, or the solid-liquid interface at the tail of the laser scan path, entrapped and solidified the other micro-pore, causing a defect close to the surface.

7.6.3 Effect of laser power on porosity formation and evolution

Cavity depth, mass addition, and defect evolution during one pass in both keyhole and conduction modes were compared, as seen in Fig 7.12. The comparison was to highlight the influence of increased laser power intensity on the resulting DED build. Both modes of experiments in Fig. 7.12 had a laser power of 250 W, as compared to the conduction and keyhole modes of experiments with laser power of 150 W in Figs. 7.10 to 7.11. The keyhole mode had a scan speed of 50 mm/s and the conduction mode had a scan speed of 500 mm/s.

Increased laser power intensity on the substrate generally increased overall cavity depth. However, there was a clear difference in the evolution of both cavity depth and aspect ratio (depth/width) with an increased laser power from 150 to 250 W. During the conduction mode at 250 W laser power and 500 mm/s scan speed used in Fig. 7.12, the cavity depth did not increase substantially
from the conduction mode experiments at 150 W and 500 mm/s as shown in Fig. 10. Because of the higher scan speed, pressure-gradient driven scattering of particles was frequent throughout the experiment. During the keyhole mode, the process parameters of 250 W and 50 mm/s caused a cavity depth about two times greater than those in the keyhole mode experiments with 150 W and 100 mm/s. The overall power intensity over time dramatically increased. With increased laser power and slower scan speed, the cavity depth took longer to become stable at about 750 µs from 400 µs with 150 W. This caused a peak in the aspect ratio of the keyhole cavity depth at 750 µs as well. In the keyhole mode with greater laser power, there were less fluctuations in cavity depth.

Figure 7.12. Evolution of the laser-induced cavity depth (top) and aspect ratio, or depth/width, (bottom) for both conduction and keyhole modes.
Mechanisms of mass addition during the process also changed with increased laser power. Contrary to the conduction mode experiments at 150 W, mass addition and subtraction occurred at 250 W. As seen in Fig. 7.12, there were instances of mass addition and subtraction during the conduction mode as denoted by the white circles and diamonds. At greater laser power, the resulting melt pool during conduction mode increased in size and surface area. Greater surface area allowed for Marangoni convection to entrain more particles near or on the substrate surface to enter the melt pool. These particles entered the melt pool during the beginning of the experiment before the cavity depth and aspect ratios stabilized.

During the keyhole mode at 250 W, there was mass addition at the beginning of the experiment, as denoted by the white circles in Fig. 7.12. Marangoni convection in the melt pool entrained these particles mostly at the beginning of the keyhole mode of experiments, before the cavity depth and aspect reached stable values. The resulting vapor-plasma plume from the cavity had greater density and velocity than the plume generated when the laser power was 150 W. The expanding vapor-plasma plume scattered flowing particles from 4 to 8 m/s outward and upward, away from the melt pool. At an increased laser power from 150 to 250 W and a decreased scan speed from 100 to 50 mm/s, the vapor-plasma plume did not allow any surrounding particle to travel into the path of the laser beam. The change in process parameters did not allow laser attenuation. The vapor-plasma plume traveled as a tail that increased in angle relative to the laser beam as the scan speed increases. With a decreased scan speed from 100 mm/s to 50 mm/s, the resulting plume was more aligned with the laser beam path. The vertically-aligned plume jetted any particle above the
melt pool away from the surface. This allowed for particles with low vertical momentum relative to the substrate surface and at the melt pool boundaries to enter the melt pool.

Increased laser power intensity generally decreased the frequency of porosity formation. During the conduction mode with 250 W, an increased laser power did not cause any entrapped micro-pore, unlike those formed during the conduction mode experiments with 150 W. Increased energy into the substrate led to greater Marangoni convection within the melt pool. This greater Marangoni force stirred any induced micro-pore during the process. During keyhole mode processing with an increased laser power, porosity formation only occurred once the laser turned off. This was because decreased scan speed from 100 to 50 mm/s resulted in a slower moving solidification front and less shrinkage that would otherwise have contributed to keyhole porosity.

7.6.4 Influence of two beam passes

With two-pass experiments, cooling behavior was observed after laser processing on both room temperature and heated substrates. During rapid cooling in DED, porosity traversed through the melt pool with melt pool convection velocity. Resulting porosity expanded, shrunk, dissipated at the surface, or was constrained by the solidified melt pool after cooling. Scanning the laser for a second pass did not necessarily remove or change the resulting keyhole porosity from the first pass. Mass change in the melt pool can be monitored and influenced the cooling rate in both passes. Trends in cooling behavior show that powder mass flow can have a significant influence in the thermal history and structure of the melt pool during the first pass.
Tracking the cavity geometry evolution during laser-matter interaction allows observation of the moving liquid-solid interface that surrounds the cavity zone where the laser vaporizes the substrate, as seen in Figs. 7.10 and 7.11. The moving solidification front introduced more keyhole porosity formation at the solid-liquid interface. Porosity usually stirred within the melt pool, shrinks, and then dissipates. However, the melt pool boundary also acts as a constraint on keyhole pores as they are pushed out of the solid-liquid interface into the surrounding substrate. Porosity location, shape and size from the two-pass experiment in Figs. 7.10 to 7.11 were tracked after the laser was turned off and the melt pool solidified.

A fully solidified melt pool was determined when the melt pool, and any feature within the melt pool (keyhole pores, micro-pores, and any pixel that change in intensity) became stationary. Figure 7.13a shows a small pore constrained by the liquid-solid interface at 1,850 µs, right before the laser was turned off. As soon as the laser was turned off at 1,900 µs, two additional micro-pores with entrapped gas formed at the liquid-solid interface (porosity group “17-6”) and increased in size from about 50 to 350 µm². In subsequent frames until the melt pool fully solidified at 3200 µs, pore group “17” oscillated in size from 300 to 340 µm² until it reached an equilibrium at about 320 µm².

As soon as the laser turned off in 1,900 µs, shrinkage caused a keyhole pore, porosity group “18”, within the melt pool to form. At 1,950 µs, the keyhole pore (“18-2”) expanded from 470 to 1213 µm² as the solidifying melt pool was still stirring and shrinking. In subsequent frames, porosity
group “18” decreased in size and maintained equilibrium when substrate was fully solidified at 3,200 µs, reaching a size of about 260 µm².

![Figure 7.13.](image)

Figure 7.13. Evolution of keyhole porosity after the laser was turned off and until the substrate was solidified for two passes. The experimental conditions were 150 W laser power, 100 mm/s, 400 V voltage input into the piezo-driven powder delivery system with a pulse.

There was a 2.5 ms delay from the time the laser was turned off in the first pass to the beginning of the second pass, with each pass lasting 2,000 µs of laser illumination. During the delay, the constrained porosity from the first pass, groups “17” and “18”, remained stationary. During the second pass, these constrained porosity groups from the first pass remained within the melt pool without significantly changing size or shape and appeared in the final solidified part after the second pass.

Eight groups of pores formed and dissipated into the melt pool during the second pass, resulting in two large keyhole pores, groups “25” and “27”, at 6,200 µs, immediately before the laser turned off. When the laser turned off at 6,250 µs, pores “25-10” and “27-1” coalesced into “25-11” and another keyhole pore, “28-1”, formed closer to the surface because of rapid shrinkage. After 250
μs, at 6,500 μs, the coalesced pore split into two pores (porosity groups “25-16” and “29-5”). The large keyhole pore from 6250 μs, “28-1”, shrunk and moved vertically toward the surface, as seen at 6,500 μs with porosity group “28-6”. Until the build solidified at 3000 μs, pore “28” dissipated at the substrate surface, “29” epitaxially traveled toward the surface and slightly decreased in size, and “25” was constrained by the liquid-solid interface. In addition to the porosity that remained from the first pass, “17” and “18”, two large keyhole pores of almost 100 μm in size, resulted after the part solidified after two passes. The capability to monitor the evolution of porosity location, shape and size during and after a build can reveal the mechanisms behind defects in additive manufactured parts, as small fluctuations to the laser power, scan speed and overall absorptivity influence the solidified structure.

7.7 Summary

Currently, the industrial standard for DED-processed parts is to build them with minimal porosity, which usually translates to using process parameters that avoid any balling or lack of fusion. However, porosity can arise from entrapped gas or keyholing and influence how the material behaves. Measuring the bulk porosity is a starting point to understanding a material’s density, but porosity characteristics such as distribution, shape, and size also contribute to its behavior. Alignment of porosity commonly seen in components with lack of fusion at the layer or hatching interfaces. Though aligned porosity is usually detrimental to structural components, this type of microstructure can be beneficial to biomedical components for osseointegration. This chapter provided an overview of types of porosity and how they form during the DED process. Bulk porosity in 316L SS components were evaluated with their processing conditions, or global energy
density. Porosity analysis for Ti-6Al-4V was more in-depth in that localized characteristics were compared to orientation, location in the component, and localized cooling rate determined by the GAMMA in-house code.

Compared to the GAMMA cooling rates and thermal history, *in-situ* high-speed X-ray showed that less laser penetration into the underlying layer would occur in “real” processing. Compared to powder bed additive manufacturing, *in-situ* X-ray high-speed imaging of the DED process reveals that powder flow absorbs energy from the laser, preventing the formation of a keyhole mode of the melt pool. Contrary to previous assumptions about porosity in additively manufactured parts, keyhole and entrapped gas porosity observed in the low-dosage DED process were not large or spherical. Entrapped gas porosity within the melt pool formed from the cavity, or the vapor depression zone where the plasma-vapor plume originates. This porosity typically ranged in size from a few to tens of microns in diameter and would stir within the melt pool due to convection. If the buoyancy force overcame convection in the melt pool, the pore would rise to the surface and dissipate. Keyhole porosity formed at the bottom boundary of the cavity due to rapid cooling, and therefore collapse, of the cavity. Both types of pores would undergo stirring, shrinking, expansion, and dispersion into multiple smaller pores. During this porosity evolution, the characteristics of shape, size and orientation would change with the cooling behavior of the melt pool. When metallic powder particles entered the melt pool or the melt pool underwent spattering, the cooling behavior would change, also modifying the trajectory of porosity evolution. Therefore, *in-situ* X-ray high-speed imaging of the DED process is critical to understand the formation, evolution, and later the control of porosity in the part.
Future work includes further *in-situ* imaging of porosity formation to understand the mechanisms in which defects arise. Collecting a large amount of porosity data for various DED-processed materials can be used to normalize and predict porosity using data science methods. These methods will provide means to calculate a normalized energy density value that is dependent on process parameters and result in a predicted porosity value. Coupling porosity characteristics with microstructure provides a more thorough understanding of how a DED-processed material can behave. Future work will either use porosity characteristics and microstructure as inputs into mechanical (such as phase field) models or use thermal histories from thermal models to predict for porosity and microstructure. In the same vein as using process parameters to control microstructure, modifying process parameters during the process can control porosity characteristics based on understanding its mechanisms of formation.

The next chapter will take the porosity analysis further by looking at the relationships between porosity and mechanical behavior. As porosity characteristics varied with orientation, location, and laser processing power, mechanical behavior also exhibited heterogeneity and anisotropy.
Mechanical behavior of a DED-processed component determines whether a material can be certified and verified for wider use. This chapter discusses the mechanical behavior observed in DED-processed materials because of processing, thermal history, phase transformations, and porosity discussed in previous chapters. A review on the mechanical behavior of DED-processed materials is provided in Section 8.1. Section 8.2 provides an overview of the experimental methods used in this work to determine mechanical behavior. The materials discussed in this section are 316L SS (Section 8.3), Inconel 718 (Section 8.4), and Ti-6Al-4V (Sections 8.5 and 8.6), as seen in Fig. 8.1. Section 8.7 provides a summary of the mechanical properties observed in this work and opportunities for future work.

Figure 8.1. Materials discussed in the mechanical behavior in DED processing.
8.1 Review of the mechanical behavior of DED-process materials

Rapid solidification during the DED process leads to phase transformations and possible porosity that influence tensile and shear behavior. In general, the resulting phase transformations during laser-based additive manufacturing strengthens the material when negligible porosity is present. The presence of porosity, depending on the alignment and orientation, can lead to premature failure due to crack propagation. However, resulting mechanical properties of a DED-processed component are process and material-dependent. The phase transformations and mechanisms of porosity formation influence the final component performance.

8.1.1 Tensile behavior

A study on 316L SS processed using laser powder-bed fusion showed that the multi-scale microstructural features that appear because of rapid solidification can increase both strength and ductility (Wang, Voisin et al. 2018). This revealed how laser-based additive manufacturing could overcome the strength-ductility trade-off that usually occurs during steel strengthening. Solidification cellular structures, low-angle grain boundaries, solute segregation, and dislocations from selective laser melting strengthened the material while heterogeneous, multi-scale microstructures led to work-hardening (Wang, Voisin et al. 2018). As shown in Fig. 8.2, specimens built with increased additive manufacturing exhibited increased strength and ductility compared to those built with traditional methods.

Like tensile behavior in powder-bed based additive manufacturing, components built with the DED process also exhibited increased strength compared to tensile behavior of components
processed with traditional manufacturing methods. Because varying heat input influenced the resulting phase transformations, DED process parameters were sensitive to changes in mechanical behavior.

![Figure 8.2](image_url)

Figure 8.2. The tensile engineering stress-strain curves for the two laser powder bed studies compared to the those from conventional manufacturing (Wang, Voisin et al. 2018).

In a wire-fed laser deposition study that tested the tensile behavior of Ti-6Al-4V specimens, changes in process parameters and heat treatments were used (Brandl, Palm et al. 2011). Two process parameter sets were used with different laser power and scan speed values, but with the same overall heat input per unit length (laser power divided by scan speed). In the as-built specimens, yield and ultimate tensile strengths were similar, though the specimens with greater laser power and scan speed exhibited greater ductility, with elongation values 30-40% greater than those built with less laser power and scan speed, as shown in Fig. 8.3. The microstructures were basket-weave $\beta+\alpha$ phase with some martensitic $\alpha'$ phases (Brandl, Palm et al. 2011). With heat treatment that strengthened the material, fine lathe and martensitic structures were observed. With
heat treatment that decreased the strength of the material, more of the $\alpha$ phase was observed. Within the plate, bimodal refined microstructures were observed.

Figure 8.3. Tensile engineering stress-strain curves of wire-fed Ti-6Al-4V for different processing conditions, with P38 representing the high laser power and scan speed condition and P58 representing the low laser power and scan speed condition (Brandl, Palm et al. 2011).

In addition to process parameters influencing the microstructure and therefore the mechanical behavior, orientation also plays a role. The rapid solidification that occurs in the component is directional, the tensile and shear behavior in a DED-processed component can exhibit anisotropy. In a study that used 304L SS powders (Wang, Palmer et al. 2016), two process parameter sets were used to build thin walls, with tensile specimens machined in the longitudinal direction, the transverse direction, and from the baseplate. The study found a decrease in yield and ultimate tensile strengths with the higher laser power process parameter set. However, the strength from the baseplate was greater than the specimens from the wall because of martensitic transformation, while nitrogen in the 304L powder stabilized the austenitic phase within the wall (Wang, Palmer et al. 2016).
Other mechanisms introduced by additive manufacturing can increase the strength, including carbide precipitation in steels and nickel superalloys, so that plastic flow behavior would cause the slip to occur by particle shearing (leading to low shearing mode). The presence of oxide inclusions can also lead to either hardening or embrittlement, depending on the oxide chemical composition. Carbon monoxide gaseous bubbles were found to rupture grain boundaries in addition to linking voids that led to ductile fracture of Inconel 718 (Qi, Azer et al. 2009). Aluminum oxides and ferrous oxides were used as solute strengthening atoms that provided an elastic interaction energy in various alloys (Benjamin 1970). In rare cases, sulfide inclusions in the material led to impurity atom embrittlement by reducing a steel’s resistance to pitting corrosion, though there were inherently some levels of sulfur in the steel powder to increase machinability (Campbell 1992).

With smaller grain sizes, dislocation movement throughout the material is more readily impeded, hindering the onset of plasticity and increasing the strength of the material. The Hall-Petch relationship for grain boundary strengthening can provide insight on strengthening (Hansen 2004):

$$\sigma_y = \sigma_0 + \frac{k_y}{\sqrt{d}}$$  \hspace{1cm} (8.1)

where $\sigma_y$ is the yield strength of the material, $\sigma_0$ is its initial stress for dislocation movement, $k_y$ is the strengthening coefficient and a material constant, and $d$ is the average grain diameter. However, the chemical composition, porosity, and inclusions of DED-processed parts can counteract grain boundary strengthening and strain gradient hardening to ease dislocation motion and grain boundary sliding.
8.1.2 Toughness and hardness

Toughness of additive manufactured parts can be measured with Charpy impact testing, and quantified by the amount of energy per unit volume that the material absorbs before fracture (Toshiro, Isamu et al. 1986). In a study that compared conventionally processed 316L SS with additive manufactured selective laser melted (SLM) parts (Yasa, Deckers et al. 2009), the toughness, or impact resistance, of cast 316L SS after annealing was about 160 J, compared to the resistance of about 60 J for SLM parts. They found that porosity played a role in reducing toughness in that the interlayer porosity in the AM 316L SS parts were sites of crack propagation. In addition, the average bulk porosity of the SLM-processed 316L SS was about 1.5 % (Yasa, Deckers et al. 2009). In previous studies of Charpy impact testing for the toughness of DED-processed Inconel 625 specimens, the resistance against crack propagation did not change with the orientation of the part relative to the build direction (Paul, Ganesh et al. 2007). However, hot finishing and annealing of the specimens increased the impact resistance from about 103 J to 110 J (Paul, Ganesh et al. 2007).

In a study of selective laser melted Ti-6Al-4V +10Mo, impact resistance was about 3.8 J for specimens that were built horizontally (orthogonal to the build direction) and 1.4 J for specimens built along the build direction, as compared to Ti-6Al-4V specimens with impact resistance of about 11.5 J (Vrancken, Thijs et al. 2014). They found that the direction of crack growth tended to follow the layer interface, with the horizontally built specimen having crack growth that started in the build direction in the layer interface, and then grow in the horizontal direction (Vrancken, Thijs et al. 2014).
In additive manufactured parts, hardness was more dependent on the phase composition and the presence of segregation, rather than on porosity. In a study of DED-processed 316L SS, microhardness in the build and scan directions were compared (Ziętala, Durejko et al. 2016). The Vickers microhardness in the build direction was about 272±35 HV, whereas the microhardness was 289±16 HV in the scan direction. This was due to the increase in dislocation concentration in the austenitic cellular structures, with larger gradients in microhardness (due to greater thermal gradients) in the build direction (Ziętala, Durejko et al. 2016).

In DED-processed Inconel 718, strengthening elements such as Nb and Mo became supersaturated in the matrix material due to high cooling rates (Moiz 2013). High cooling rates also refined the microstructure. Both the refined microstructure and supersaturation of the strengthening elements led to high Vickers hardness values of about 254 HV, which was greater than the hardness value in conventionally processed Inconel 718 of about 240 HV (Moiz 2013). In DED-processed Inconel 718 cuboids (Stevens, Toman et al. 2017), an increase of average hardness was attributed to a decrease in linear heat input into the component, with some localized areas of the cuboid reaching about 310 HV due to carbide precipitation.

In DED-processed Ti-6Al-4V cubic components, the Vickers microhardness values increased with an increase volumetric energy density (Nassar, Keist et al. 2015). The greater cooling rates in the components with greater energy density resulted in martensitic α’ phases, as well as the precipitation of the intermetallic Ti₃Al phase, contributing to hardness.
8.2 Experimental methods to determine mechanical behavior

8.2.1 Uniaxial tensile testing

All uniaxial tensile testing was conducted in the Central Laboratory for Materials Mechanical Properties (CLaMMP) at Northwestern University. The tension test load frame included a servo-hydraulic machine with 100 kN capacity, the Sintech 20 G. For the majority of the tensile tests in this work, a standard quasi-static test was used and consisted of attaching a 10 mm extensometer to each tensile specimen. The strain rate used in each test was 0.25 mm/min. The geometry of the tensile specimens were 10 mm in gage length, 2.5 mm wide in the gage area, and 1.2 mm in thickness, according to the sub-sized dimensions in the ASTM E8 standard (Officials and Materials 2004).

For some tensile specimens, digital image correlation (DIC) was used to capture and calculate strain in real time. In the DIC tests, quasi-static testing used a strain rate of 1 mm/min. Speckled patterns on the tensile specimens were used to create a random pattern. A CCD camera, coupled with the VIC-Snap software, captured the change in the speckled pattern with up to 9 fps. The resulting images had a resolution of 3,376 X 2,704 pixels, with pixel sizes of 5 by 4 µm. To perform DIC analysis, a seed point was set on the initial reference frame, and displacements were calculated from the images of the deformed part throughout the test. The displacements were smoothed to calculate the strain values using the VIC 2D software. An in-house MATLAB code with a modified correlation function analyzed two subsets at a time to create a virtual extensometer. The resulting strain values were matched with the load values from the Sintech 20G machine to provide a stress-strain curve.
8.2.2 Charpy impact testing

Charpy impact testing consisted of using a Tinius Olson 1177 apparatus with a capacity of 358 J of impact resistance. The apparatus consisted of a pendulum that impacted the V-notch of each Charpy specimen, per the dimensions and procedures in the ASTM E23 standard (Standard 2009).

8.2.3 Microhardness

Vickers microhardness measurements were taken within the clad with a Duramin 5 hardness tester using a press load of 2.94 N for five seconds. At least five points were evaluated for Vickers hardness for each region of interest in a DED-processed part, ensuring that the microhardness gradient, and therefore the heterogeneity of mechanical behavior from the extreme sides of the additive manufactured component were captured.

8.3 Mechanical properties of DED-processed 316L SS

8.3.1 Tensile properties

The influence of the net heat input on tensile behavior was observed for 316L SS. The net heat input was determined by calorimetry measurements performed at Northern Illinois University, which was retrofitted into the OPTOMEC machine. Table 8.1 lists the process parameters and the resulting net heat inputs used for the 316L SS tensile parts. The laser beam diameter during the build of all specimens was 1.87 mm. The “216387” parts were all built on the same substrate, during the same build. They were built with the thickness side on the substrate, resulting in a thin wall. Though the “216387_1” and “216387_2” tensile parts used the same processing conditions,
their net heat inputs varied because the “216387_1” tensile part was built first, resulting in a retained heat during subsequent builds. In addition, the “216387_1” and “216387_4” tensile parts were built closer to the edge of the substrate, resulting in less of a heat sink. The “217405” tensile parts were built individually on the center of the substrate to isolate the influence of heat from adjacent part builds.

Table 8.1. Processing conditions for 316L SS tensile parts.

<table>
<thead>
<tr>
<th>Part name</th>
<th>Input laser power (W)</th>
<th>Laser scan speed (mm/s)</th>
<th>Powder mass flow (mg/s)</th>
<th>Hatch spacing (mm)</th>
<th>Layer thickness (mm)</th>
<th>Net heat input (J/mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>216387_1</td>
<td>750</td>
<td>12.7</td>
<td>267</td>
<td>1.06</td>
<td>0.36</td>
<td>14.7</td>
</tr>
<tr>
<td>216387_2</td>
<td>750</td>
<td>12.7</td>
<td>267</td>
<td>1.06</td>
<td>0.36</td>
<td>22.2</td>
</tr>
<tr>
<td>216387_3</td>
<td>950</td>
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<td>167</td>
<td>1.12</td>
<td>0.53</td>
<td>38.9</td>
</tr>
<tr>
<td>216387_4</td>
<td>950</td>
<td>8.5</td>
<td>167</td>
<td>1.12</td>
<td>0.53</td>
<td>34.3</td>
</tr>
<tr>
<td>217405_1</td>
<td>950</td>
<td>8.5</td>
<td>167</td>
<td>1.06</td>
<td>0.43</td>
<td>28.9</td>
</tr>
<tr>
<td>217405_2</td>
<td>750</td>
<td>12.7</td>
<td>267</td>
<td>1.06</td>
<td>0.36</td>
<td>14.5</td>
</tr>
</tbody>
</table>

The resulting tensile behavior showed that the net heat input did not have much influence on the elongation and strength of the tensile parts, as seen in Fig. 8.4. Though the “216387_1” and “217405_2” tensile parts were processed with the same parameters and had similar net heat inputs, the ultimate tensile strength of the “217405_2” part, which was built at the center of the substrate without adjacent builds, was about 60 MPa greater than that of the “216387_1” part.
In addition, the strength of the part processed with 950 W laser power and on the center of the plate, “217405_1”, was up to about 70 MPa greater than those processed with 950 W (the “216387_3” and “216387_4” parts) but built with other parts on the substrate. Table 8.2 shows the resulting mechanical properties of the parts, compared to the results of conventionally-processed 316L SS. Five tensile specimens were machined from wrought and cold worked 316L SS and tested to evaluate their properties. Overall, the DED-processed parts surpassed the conventionally processed parts in strength, but with the strength-ductility tradeoff.
### Table 8.2. Resulting mechanical properties of 316L SS tensile specimens.

<table>
<thead>
<tr>
<th>Part name</th>
<th>Net heat input (J/mm)</th>
<th>Young's modulus (GPa)</th>
<th>0.2% Offset yield strength (MPa)</th>
<th>Ultimate tensile strength (MPa)</th>
<th>Uniform elongation</th>
<th>Strain at break</th>
</tr>
</thead>
<tbody>
<tr>
<td>216387_1</td>
<td>14.7</td>
<td>165.2</td>
<td>187.7</td>
<td>624.5</td>
<td>0.40</td>
<td>0.48</td>
</tr>
<tr>
<td>216387_2</td>
<td>22.2</td>
<td>182.0</td>
<td>430.3</td>
<td>602.3</td>
<td>0.30</td>
<td>0.33</td>
</tr>
<tr>
<td>216387_3</td>
<td>38.9</td>
<td>184.2</td>
<td>430.1</td>
<td>603.8</td>
<td>0.27</td>
<td>0.40</td>
</tr>
<tr>
<td>216387_4</td>
<td>34.3</td>
<td>189.6</td>
<td>430.1</td>
<td>621.0</td>
<td>0.29</td>
<td>0.44</td>
</tr>
<tr>
<td>217405_1</td>
<td>28.9</td>
<td>183.3</td>
<td>510.3</td>
<td>671.4</td>
<td>0.40</td>
<td>0.51</td>
</tr>
<tr>
<td>217405_2</td>
<td>14.5</td>
<td>233.1</td>
<td>517.5</td>
<td>686.0</td>
<td>0.30</td>
<td>0.49</td>
</tr>
</tbody>
</table>
| Conventionally processed (wrought and cold worked) | -                     | 193                   | 234-290                         | 560-586                         | 0.35-0.45          | 0.38-0.51       

### 8.3.2 Charpy impact testing

Charpy parts detailed in Chapters 7 and 8 for their microstructure and porosity were found to have impact resistance values related to their porosity content. This aligned with literature (Yasa, Deckers et al. 2009), that found crack propagation in the direction of the build, or layer interface, direction due to lack of fusion of powder particles.

Figure 8.5 shows the trend of the bulk porosity and impact resistance of the 316L SS Charpy parts. With an increase in porosity within the specimen because of lack of fusion, crack propagation was more likely and there was less resistance to fracture. Contrary to literature, impact resistance values were greater than the 160 J for conventionally processed 316L SS, indicating better fusion during the DED process than that of the SLM process.
8.4 Mechanical properties of DED-processed Inconel 718

In the Inconel 718 single clads study discussed in previous chapters, the microhardness of the deposited clad depended on its microstructure, which was in turn determined by the cooling rate during processing. The high temperature toughness and ductility of IN718 is provided by the face-centered cubic (FCC) nickel crystal structure ($\gamma$-phase) matrix of the alloy [(Reed and Rae 2015)].

The precipitation strengthening of $\gamma'$, $\gamma'''$ to the $\gamma$ matrix and the intercrystalline strengthening of platelet-like $\delta$ phase to the grain boundaries contribute to increased microhardness (Wang, Guan et al. 2012). Other possible precipitates during phase transformations of IN718 include carbides and the eutectic Laves phase, which is known to be detrimental to the mechanical properties of IN718 (Reed and Rae 2015).

Experimental surface measurements of cooling rate based on IR thermography changed dramatically from the solidification region and at lower cooling temperatures where $\gamma'$ and $\gamma'''$
precipitation strengthening may have occurred (Wang, Guan et al. 2012). As a result, microhardness values along the top of the clad, or those that were more indicative of the surface IR thermography readings, were compared to the cooling rates normalized to the amount of laser power per unit of mass powder flow, \( \hat{T} \), as seen in Fig. 8.6. Thermal properties such as cooling rate dictated the geometry of the melt pool and the solidified clad. The trends in cooling dictated the trends in changing microstructure, and therefore the mechanical behavior in the clad.

As seen in Fig. 8.6, the microhardness of the parts increased with normalized cooling rate, \( \hat{T} \), up to a value of approximately 0.4 K·g·J⁻¹·s⁻¹, the same threshold value at which the dilution ratios of the clads became zero, and after which microhardness values leveled off at about 250 HV. This trend agreed with the findings of Wang et al., showing that grain refinement caused by rapid cooling increased the hardness of deposited clads (Wang, Guan et al. 2012). Except for one outlying clad measurement of about 300 HV at low normalized cooling due to possible precipitation, all the measured hardness values were considerably below the hardness value of conventionally processed and heat treated IN718 hardness value which is 383 HV (Wang, Guan et al. 2012).
Figure 8.6. Experimentally determined average Vickers microhardness values and their respective cooling rates determined by IR thermography normalized by laser power divided by mass powder flow rate, or $\hat{T}$.

The outlying hardness value of 300 HV occurring at a cooling rate of approximately 3,000 K/s was comparable to the conventionally processed IN718 value. This high value could have been the result of inadvertent reheating of the clad during the processing of the adjacent clad, resulting in a post-process heat treatment. For the remaining clads that were only heated and cooled once, the low hardness values agreed with the findings of Stevens et al. (Stevens, Toman et al. 2017), who observed that if the deposited material cooled too quickly for adequate segregation to occur, there was less Nb available to strengthen the part. In multi-layer builds, this low hardness was observed at the top layer because it only underwent one cycle of melting (Stevens, Toman et al. 2017).

The results in Fig. 8.6 are averages of multiple microhardness measurements along the top of the clad, or close to the surface. Clads with relatively large cross-sectional areas (dilution ratios near zero or one) had a greater gradient of microhardness values, with increased hardness at the liquid-solid interface within the substrate. This agreed with the results of Stevens et al. (Stevens, Toman
et al. 2017) who found that there was an increased occurrence of the Laves phase near clad boundaries. The increased microhardness closer to the melt pool interface was a result of increased cooling rates at the bottom of the clad due to Marangoni convection during the process. Overall, the melt pool interface underwent rapid quenching that led to grain refinement and a distinguishable layer band (Wang, Guan et al. 2012). These findings were like those in a study that used a quasi-continuous wave laser to control the cooling rates in Inconel clads and therefore resulted in equiaxed refined grain structures, Nb microsegregation, and suppressed Laves phase formation (Xiao, Li et al. 2017).

Figure 8.7 shows the microhardness model calibration and validation with the experimentally-determined microhardness values. Although the effects of cooling rate and SDAS on hardness were neglected in the microhardness prediction model, an approximate relation between composition (equivalent nickel) and microhardness could be used with reasonable accuracy. This implies, that rather than the variation in dendrite arm spacing, the composition changed due to substrate melting (dilution) dominated the variation in microhardness. In traditional metallurgy, microhardness highly depended on dendrite arm spacing for a specific material system (Knapp, Mukherjee et al. 2017). The deviation between numerical and experimental results was possibly a result of the non-linear solidification mechanisms such as rapidly quenched segregates, which was not considered in the microhardness analysis module of the CtFD thermal fluidic dynamic model.
An experimental study of single clads processed with the DED additive manufacturing process with various powder mass flow rates and laser power inputs demonstrated how the laser-material interaction led to discrepancies in dilution into the substrate and a change in mechanical properties. Based on IR thermography measurements at the surface of the IN718 clads, an increase of solidification rates normalized to laser power and mass powder flow led to decreased dilution into the substrate as more conduction into the substrate material and laser scattering with the deposited material occurred. Microhardness values initially increased with solidification rates normalized to the amount of laser power for each unit of mass powder flow, then leveled off at a value of about 250 HV. Microhardness also consistently changed with depth into the clad. These results can help calibrate and validate thermal models that capture the change of the melt pool and the overall structure and properties of the resulting build. Future experimental studies will investigate the scattering and absorption of powder during deposition as well as the exact temperatures and
cooling rates that lead to complex phase transformations and segregation that occur in IN718 that can lead to significant changes in mechanical behavior.

A comparison of solidification rates, depths of the clad into the substrate, dilution ratios and microhardness values are summarized in Table 8.3. The proposed thermal fluid dynamic multiphysics model, the CtFD model, considered physics such as powder mass flow and fluid dynamics of the melt pool that captured the solidification cooling rate within the melt pool with high accuracy. The model also predicted for microhardness at localized points within a build. The proposed model was the first that evaluated cooling rate and predicted microhardness of DED-processed IN718 based on the calibration of experimental results. Future work includes incorporating a more detailed dendritic microstructure model to accurately predict material properties of the resulting material. The future experimental work of determining phase compositions and macro and micro segregation will also further improve the microhardness prediction module of the CtFD model.

<table>
<thead>
<tr>
<th>Property</th>
<th>Experiments</th>
<th>CtFD model</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cooling rate at the substrate surface (K/s)</td>
<td>800-6000</td>
<td>800-6000</td>
</tr>
<tr>
<td>Clad depth into the substrate (μm)</td>
<td>0-800</td>
<td>0-1000</td>
</tr>
<tr>
<td>Clad dilution ratios at 56 mg/s and 1000, 2000 W</td>
<td>0.69, 0.86</td>
<td>0.75, 0.90</td>
</tr>
<tr>
<td>Microhardness (HV)</td>
<td>190-300</td>
<td>180-260</td>
</tr>
</tbody>
</table>

The proposed inexpensive and well-tested numerical framework can generate a large amount of high-quality prediction data, including temperature field, velocity field, melt pool dimensions, heating and cooling rates, solidification parameters, and microhardness. Future work will use this
data to train a reduced-order model (ROM) to obtain the linkages between processing parameters of the DED-processed build and the resulting mechanical properties. A data-based online monitoring and feedback control modeling approach will be applied to the localized DED process and other relevant multi-scale and multi-physics processes. Data-driven relationships between process, structure and property can provide online monitoring and process control for desirable solidification rates at localized points to drive ideal phase transformations.

8.5 Global Ti-6Al-4V tensile properties

8.5.1 Experimental methods

Mini-tensile specimens were machined by wire-EDM from DED-processed Ti-6Al-4V cubic components in three different orientations as illustrated in Fig. 8.8. The processing conditions for the three Ti-6Al-4V cubic components are detailed in Chapter 5. The planar distance between each tensile specimen in each orientation was about 1.5 mm. The tensile dimensions followed ASTM E8 standards for sub-sized sheet tensile specimens (Officials and Materials 2004) with a gauge length of 10.0 mm, gauge width of 2.5 mm, and thickness of 1.2 mm. Tensile specimens in the “A” orientation were machined from the XZ (scan-build) plane and varied in the Y (hatch) direction and with Z, the build direction, as their uniaxial tensile axis. Tensile specimens in the “B” orientation varied in the Z direction and had X as their tensile axis. Tensile specimens with orientation “C” orientation varied in the X direction and had Y, the scan direction, as their tensile axis, as discussed in Chapter 6.
Eight tensile specimens were machined in three orientations from the cubic component processed with 800 W, resulting in 24 tensile specimens from the 800 W cubic component. For the 710 W cubic components, six specimens in each of the “B” and “C” orientations were tested from the 710 W cubic components, and six specimens from the “C” orientation was tested for the 940 W cubic component. The reduced number of machined specimens for the 710 W and 940 W components were due to machining costs and the fact that superior mechanical behavior had been observed in the “B” and “C” orientations. There were 42 total number of tensile specimens from all three cubes. Scanning electron microscopy (SEM) was used to evaluate the fracture surfaces of several fractured tensile specimens to carry out fractography image analysis.

8.5.2 Determination of the J-factor

Based on the global analysis study for Ti-6Al-4V detailed in Chapter 7, the J-factor could be determined based on the porosity content and the mechanical behavior of the specimens in the differing orientations and laser processing powers. To properly characterize the relationship between process parameters and mechanical behavior, the size, orientation and shape of the
individual pores were captured. To do this, each orientation was characterized by the normal vector to its plane, \( \vec{n} \), and the tensile test load axis, \( \vec{e} \), as shown in Table 7.3 in the previous chapter. Moment of the DED-processed component was calculated as a composite where the bulk material was a cube and the pores were \( N \) number of aligned ellipsoids, as seen in Figure 8.9. The axis about which the moment of inertia was calculated was through the center of the cube, with each axis the principal axes of \( X \), \( Y \) and \( Z \).

Figure 8.9. Visual representation of a bulk component with aligned ellipsoid pores with pore dimensions \( a \), \( b \), \( c \).

The moment of inertia of the composite was expressed as a tensor. If \( m \) was the mass of the \( Ti-6Al-4V \) bulk material, \( a, b, c \) were the dimensions of the ellipsoid pores. The number of pores in the \( \vec{e}_x, \vec{e}_y, \vec{e}_z \) load axes were denoted by \( N_1, N_2, N_3 \), respectively. The number of pores in the \( X \) direction, \( N_1 \), was the width of the cubic component divided by the hatch spacing in the \( \vec{e}_x \) load direction, \( N_2 \), or the number of pores in the \( Y \) direction, was the length of the cubic component divided by the hatch spacing in the \( \vec{e}_y \) load direction, and \( N_3 \), or the number of pores in the \( Z \) direction, was the height of the cubic component divided by the layer thickness in the \( \vec{e}_z \) load direction.

The total moment of inertia of the porous phases in the cubic component was described as:
\[ \mathbf{I}_{\text{tot}} = \begin{bmatrix} I_{xx} & I_{xy} & I_{xz} \\ I_{yx} & I_{yy} & I_{yz} \\ I_{zx} & I_{zy} & I_{zz} \end{bmatrix} = m \begin{bmatrix} \frac{s^2}{6} - \frac{N_4}{5} (b^2 + c^2) & -\frac{N_4}{5} (b^2 + c^2) & -\frac{N_4}{5} (b^2 + c^2) \\ -\frac{N_2}{5} (a^2 + c^2) & \frac{s^2}{6} - \frac{N_2}{5} (a^2 + c^2) & -\frac{N_2}{5} (a^2 + c^2) \\ -\frac{N_3}{5} (a^2 + b^2) & -\frac{N_3}{5} (a^2 + b^2) & \frac{s^2}{6} - \frac{N_3}{5} (a^2 + b^2) \end{bmatrix} \] (8.2)

Determination of the \( J \)-factor can be described in relationship in the following equation:

\[ J = \exp(C_4 (\mathbf{n} \cdot [I] \cdot \mathbf{e})) \] (8.3)

Table 8.4 lists the \( \mathbf{n} \) and \( \mathbf{e} \) vectors for each orientation. The \( a, b, c \) dimensions of the ellipsoid pores were experimentally determined by measuring distributions of the area and roundness of the pores in the tensile specimens of the varying orientations where \( a > c > b \), based on image analyses of the experimentally-determined micrographs.

<table>
<thead>
<tr>
<th>Orientation</th>
<th>( \mathbf{n} ), vector normal to plane</th>
<th>( \mathbf{e} ), principal load axis</th>
<th>Area of ellipsoid</th>
<th>Roundness of ellipsoid</th>
<th>( \mathbf{n} \cdot [I] \cdot \mathbf{e} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>([0 \ 1 \ 0])</td>
<td>([0 \ 0 \ 1])</td>
<td>( \pi ac )</td>
<td>( c/a )</td>
<td>(-\frac{N_3}{5} (a^2 + b^2))</td>
</tr>
<tr>
<td>B</td>
<td>([0 \ 0 \ 1])</td>
<td>([1 \ 0 \ 0])</td>
<td>( \pi ab )</td>
<td>( b/a )</td>
<td>(-\frac{N_1}{5} (b^2 + c^2))</td>
</tr>
<tr>
<td>C</td>
<td>([1 \ 0 \ 0])</td>
<td>([0 \ 1 \ 0])</td>
<td>( \pi bc )</td>
<td>( b/c )</td>
<td>(-\frac{N_2}{5} (a^2 + c^2))</td>
</tr>
</tbody>
</table>

A roundness value of 1.0 indicated a perfect circle. Roundness values ranged from 0 to 1. Area and roundness data for each specimen were interpreted as Weibull distributions, \( f_{\text{area}}(u) \) and \( f_{\text{roundness}}(v) \). Each Weibull distribution was defined by shape parameters, \( c_{\text{area}}, d_{\text{area}}, c_{\text{roundness}} \) and \( c_{\text{roundness}} \). The Weibull probability distribution functions (pdf) for area and roundness were defined by:
\[ f_{\text{area}}(u; c_{\text{area}}, d_{\text{area}}) = \begin{cases} \frac{d_{\text{area}}}{c_{\text{area}}} \left( \frac{u}{c_{\text{area}}} \right)^{d_{\text{area}}-1} e^{-\left( \frac{u}{c_{\text{area}}} \right)^{d_{\text{area}}}}, & x \geq 0 \\ 0, & x < 0 \end{cases} \quad (8.4) \]

\[ f_{R}(v; c_{\text{roundness}}, d_{\text{roundness}}) = \begin{cases} \frac{d_{\text{roundness}}}{c_{\text{roundness}}} \left( \frac{v}{c_{\text{roundness}}} \right)^{d_{\text{roundness}}-1} e^{-\left( \frac{v}{c_{\text{roundness}}} \right)^{d_{\text{roundness}}}}, & x \geq 0 \\ 0, & x < 0 \end{cases} \quad (8.5) \]

The dimensions of \( a, b, c \) for the ellipsoid pores provided inputs into the moment of inertia and the \( J \)-factor. GMR analysis led to the development of a relationship between physical characteristics of the pores, including the shape and size distribution, to the \( J \)-factor for each case of laser power and orientation in the cubic component.

8.5.3 Influence of location relative to the surface of the component

Experimental results helped established relationships among the many aspects of the DED-processed builds, including porosity, phase composition and mechanical behavior. Three tiers of analysis were conducted: the influence of the location of the tensile specimen relative to the surface of the cubic component on mechanical behavior; influence of orientation (“A”, “B”, and “C”) of the tensile specimen on mechanical behavior; and the influence of laser processing power of the tensile specimens on mechanical behavior.

For each orientation and cubic component, each tensile specimen was in a unique position within the cube relative to the surface. This was crucial because each unique position within a component underwent a distinct thermal history – tensile specimens located near the surface of a component underwent more convection and radiation of heat to the surrounding air and shield gas, whereas material closer to the core of the component underwent re-melting and conduction. Therefore,
mechanical behavior changed with the location of the tensile specimen relative to the surface of the cubic component, or with the cooling rate of any unique position.

Figure 8.10 shows how mechanical behavior changed within the “A” orientation in the 800 W processed cubic component where each curve represents the distance of the tensile axis of the specimen to the surface of the cube. Based on Figure 8.10, the tensile specimens increased in strength as the distance from the surface of the cubic component increased, or as the tensile specimens neared the core, re-melted area of the cubic in the Y scanning direction. Specimens in the “A” orientation had the greatest thermal residual stresses as each specimen had a large gradient of cooling rates, due to the re-melting at the bottom and the radiation near the top of the tensile specimens. However, specimens closer to the core failed at lower elongation values and had greater Young’s moduli, implying that embrittlement occurred closer to the re-melted areas in the “A” orientation.

Figure 8.10. Tensile behavior of 800 W “A” specimens at various distances from cubic component surface.
Figures 8.11 and 8.12 show the influence of location relative to the surface on mechanical behavior for the “B” and “C” orientations. Tensile specimens in the “B” orientation also increased in strength from the surface to the core of the block in the negative Z direction, or from the top of the build to the central layers of the cubic component. Like the tensile specimens in the “A” orientation of the 800 W cube, the tensile specimens in the “B” orientation exhibited greater embrittlement and strength with an increase in distance from the surface of the cube. The Young’s modulus decreased until 6 mm and increased from 7.5 mm to the center of the component. This may have been due to the alignment of pores that coalesced at the layer interface with the tensile axis as well as the difference in cooling rate. However, elongation of the tensile specimens was about twice the elongation values of the tensile specimens in the “A” orientation. The tensile specimens in the “C” orientation exhibited superior properties compared to the specimens in the “A” and “B” orientations.

Figure 8.11. Tensile behavior of 800 W “B” specimens at various distances from cubic component surface.
As the tensile specimens moved toward the center of the cube, where the greatest re-melting occurred, there was an overall increase in yield and ultimate tensile strengths. However, elongation values within the cube in the “A” orientation did not show a steady trend. This could possibly be due to the placement of hatch-layer porosity, where pore alignment in the center of the tensile axis exhibited less elongation than specimens with pore alignment closer to the surface of the tensile specimen. Pore alignment may have also influenced the Young’s modulus.

8.5.4 Influence of orientation

Based on the results in Section 8.5.1, the differences between each orientation within the 800 W processed cubic component were apparent, with XRD analysis detailed in Chapter 7. In addition to investigating the porosity of each tensile specimen, testing tensile specimens in DED-processed Ti-6Al-4V cubic components processed with different laser powers were evaluated. Figure 8.13 shows the stress-strain curves for the 710 W processed cube in the “B” and “C” orientations, and the 800 W processed cube in the “A”, “B”, and “C” orientations. The 710 W cube exhibited the same trends as those observed in the 800 W processed component, including an increase in
elongation in “A” orientation, an increase in strength in the “A” orientation, and an increase and slight embrittlement at greater distances from the surface of the cubic components. Though yield strength and ultimate tensile strength (UTS) values were comparable between the 710 W and 800 W processed tensile specimens, the elongation increased with the increase in laser power.

By comparing the bulk porosity volume fraction and the ultimate tensile strength of the tensile specimens in the 710 W processed component, relationships between mechanical behavior and porosity were clear in certain orientations of the component than in others. Figure 8.14 illustrates the dependence of mechanical behavior on bulk porosity in the 710 W processed component in the “A”, “B” and “C” orientations. The UTS values were normalized by the maximum UTS value observed in DED-processed Ti-6Al-4V, which was about 1200 MPa (Vilaro, Colin et al. 2011). Greatest anisotropy, or influence of porosity on mechanical behavior, was found in the “A” orientation, where lack of fusion porosity at the layer interface persisted. Lack of fusion porosity at the hatch interface in the “B” orientation also influenced mechanical behavior.
Figure 8.14. Relationship between bulk porosity and mechanical behavior in three orientations for 710 W.

By comparing the bulk porosity volume fraction and the ultimate tensile strength of the tensile specimens in the 800 W processed component, relationships between mechanical behavior and porosity were clear in certain orientations of the component than in others. Figure 8.15 illustrates the dependence of mechanical behavior on bulk porosity in the 800 W processed component in the “A”, “B” and “C” orientations.
Figure 8.15. Relationship between bulk porosity and mechanical behavior in three orientations for 800 W.

Pores due to lack of fusion were results of the large thermal gradients at layer interfaces that did not properly fuse together. Toward the side surface of the component, there was more spherical and micro-scale porosity due to vaporization, leading to more bulk volume porosity closer to the side surface. Close to the top surface, phase transformations occurred over a relatively short time, leading to fast cooling and porosity from shrinkage. Tensile specimens from layers closer to the core of the component had slower cooling and less shrinkage due to re-melting and re-heating from layers above. However, specimens closer to the core of the component also exhibited more vaporization porosity.
Tensile specimens in the “A” orientation, which had its principal load axis in the build direction, exhibited the greatest anisotropy of ultimate tensile strength, as seen in Fig. 8.15. Mechanical behavior in the “A” orientation was highly dependent on porosity in the tensile specimens because of the alignment, shape and size of the pores in the direction. The pores were at the layer interfaces with a spacing of 0.95 mm and aligned throughout the tensile specimens so that about ten rows in the build direction of large, irregularly shaped pores coalesced and led to failure. Not only did the “A” oriented tensile specimens had porosity aligned at the layer interface, but also exhibited large thermal gradients throughout the part as the specimens included material close to the bottom of the part as well as the top, or areas that spanned ten layers in the build direction.

Tensile specimens in the “B” orientation had a principal load direction in the hatch direction. Mechanical behavior in the “B” orientation was also dependent on porosity but not to the degree that the tensile specimens in the “A” direction were dependent. The rows of pores in the “B” specimens were aligned at the hatch spacing between each clad of the same layer, which were 1.25 mm apart, resulting in about eight rows in the X axis of large, irregularly shaped pores. Because each tensile specimen only included material from one or two layers in the Z axis with material from about eight hatched clads, the thermal gradients throughout the specimens were not as large as those in the specimens in the “A” orientation. Tensile specimens closer to the core of the component had slower cooling and less shrinkage due to re-heating from layers above. However, specimens closer to the core of the component exhibited more vaporization porosity.
Tensile specimens in the “C” orientation had a principal load direction in the scan direction. Mechanical behavior in the “C” orientation was not as dependent on porosity as the behavior in the “A” and “B” orientations. This was because the tensile specimens included material from two or three layers in the Z axis and a single clad, or hatch, in the X axis. Thus, not only was there less of a thermal gradient in the tensile specimens in the “C” orientation, but pores were not aligned in rows normal to the load axis of the tensile specimens in the “C” orientation. Though there was a trend of increasing ultimate tensile strength with proximity to the core of the component due to an increase in martensitic formation and more re-heating cycles at the core, there was no clear trend of increasing bulk volume porosity with proximity. This is due to the changing type of porosity in the “C” orientation from keyhole porosity at the surface to entrapped gas porosity closer to the core of the cube.

The dependence of mechanical behavior on the orientation of the tensile specimen within the bulk component for tensile specimens close the surfaces of the cube is further illustrated in Fig. 8.16. Specimens in the “C” orientation exhibited greater ductility and higher load bearing capacity than those in the other orientations. This phenomenon was because specimens in the “C” orientation did not experience changes in the hatch direction nor in the build direction, and hence, less porosity due to lack of fusion at the layer or hatch interfaces throughout the principal load axis as those specimens at the “A” and “B” orientations would. The results shown in Figs. 8.15 and 8.16 illustrated the need for a deeper understanding of orientation- and location-dependent behavior, such as shearing or crack initiation, which was dictated by the thermal history of localized areas.
8.5.5 Influence of laser processing power

For a comprehensive evaluation of laser processing power, tensile specimens in “C” orientation in the 940 W processed cubic component were also tested. Figure 8.17 shows the mechanical behavior of tensile specimens in all three cubic components (710, 800, 940 W laser processing power) in the “C” orientation. Tensile specimens in the 940 W cubic component reinforced the observation in Section 8.5.2, where elongation and strength increased with increased laser processing power. Tensile specimens in the “C” orientation from the 940 W cubic component exhibited consistency and showed relatively similar elongation and strength values within a margin of error.
Fractography analysis of the fracture surface of representative tensile specimens was used to determine the mechanism of failure, whether it was elastic incompatibility, plastic deformation, coalescence of plastic cavities, or boundary sliding. Figure 8.18 shows representative fracture surfaces for each processing laser power and orientation.

For the fracture surfaces in the “A” orientation, fracture occurred at the interface of each layer with noticeable un-melted powders. River lines were apparent in both fracture surfaces and were characteristic of brittle fracture. However, the 800 W “A” fracture surface exhibited more of a cup-cone fracture, indicating that ductile fracture occurred.

For the fracture surfaces in the “B” orientation, fracture occurred at the interface of each hatch spacing, or overlap between scanning beads within the same layer. There were also noticeable un-melted powders that were aligned, indicating the location of the hatch overlap. Compared to “A”
orientation, the fracture surfaces exhibited more dimples, which were characteristic of ductile fracture, with the 800 W surface exhibiting more complex topography.

<table>
<thead>
<tr>
<th></th>
<th>710 W</th>
<th>800 W</th>
<th>940 W</th>
</tr>
</thead>
<tbody>
<tr>
<td>“A”</td>
<td><img src="image1.png" alt="Image" /></td>
<td><img src="image2.png" alt="Image" /></td>
<td><img src="image3.png" alt="Image" /></td>
</tr>
<tr>
<td>“B”</td>
<td><img src="image4.png" alt="Image" /></td>
<td><img src="image5.png" alt="Image" /></td>
<td><img src="image6.png" alt="Image" /></td>
</tr>
<tr>
<td>“B”</td>
<td><img src="image7.png" alt="Image" /></td>
<td><img src="image8.png" alt="Image" /></td>
<td><img src="image9.png" alt="Image" /></td>
</tr>
</tbody>
</table>

Figure 8.18. Fracture surfaces of representative processing powers and orientation.

For the fracture surfaces in the “C” orientation, there was a negligible amount of un-melted powders compared to the other orientations, with a decrease in un-melted powders when the laser processing power increased. In addition, an increase in processing power showed an increase in topography and cup-cone fracture, indicating an increase in ductile fracture.
By examining the fracture surfaces, the changes in elongation and strength with processing power and orientation observed during mechanical testing were validated. Relationships between the bulk porosity with the resulting mechanical behavior were made. By plotting out this relationship in Fig. 8.19, the slope of each data set indicated the degree of anisotropy with the orientation direction and processing power. Based on Fig. 8.19, the 800 W processed “A” orientation exhibited the greatest anisotropy, or greatest variation in mechanical behavior with a change in porosity. This meant that changes in porosity in the 800 W “A” oriented tensile specimens greatly influenced the mechanical properties as compared to 940 W “C” oriented tensile specimens where there was little change in mechanical behavior with a change in porosity.

This study presented an experimental analysis of DED-processed Ti-6Al-4V components built with various laser processing powers in to understand the basis for achieving superior mechanical properties in AM components as compared to cast titanium alloys.

Figure 8.19. UTS vs. Bulk Porosity for each tensile specimen.
8.6 Localized Ti-6Al-4V tensile properties

The localized analysis of Ti-6Al-4V tensile properties combined numerical results from a thermal model with experimental 2D micrographs to illustrate porosity distribution throughout the component. The results established relationships between cooling rate, microstructure, and mechanical behavior in the proposed framework. To link process parameters, thermal history, and porosity to mechanical behavior, the statistical relationships from Chapter 7 were used.

Using the statistically determined relationships between the experimentally determined porosity shape descriptors and the simulated cooling rate calculation, 2D or 3D representative porosity maps were generated. Finite element analysis via ABAQUS meshed the porosity map with tetrahedral elements in 2D and an implemented Johnson-Cook damage model for Ti-6Al-4V (Milani 2009) to illustrate how pores in the AM component changed shape and led to shear bands in various orientations of tensile load. The Johnson-Cook material parameters are listed in Table 8.5:

<table>
<thead>
<tr>
<th>A (MPa)</th>
<th>B (MPa)</th>
<th>n</th>
<th>m</th>
<th>Melting Temp (K)</th>
<th>Transition Temp (K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>862</td>
<td>331</td>
<td>0.34</td>
<td>0.8</td>
<td>1064</td>
<td>980</td>
</tr>
</tbody>
</table>

In this study, a 2D porosity map illustrated a case study for a cooling rate between the solidus and liquidus temperatures of 750 K/s. Figure 8.20 is an example of a meshed 2D representative map at this solidification cooling rate. The 2D finite element geometry was composed of two materials –
air in the white void areas and Ti-6Al-4V as the matrix material, defined by the Johnson-Cook damage model.

![Figure 8.20. A 2D porosity map illustrated a case study for a solidification cooling rate of 750 K/s.](image)

By modeling the 2D representative element as a uniaxial tensile test specimen, shear bands and void coalescence were assumed to lead to failure. Other than using the Johnson-Cook hardening law of the matrix material, Ti-6Al-4V, there were no damage or failure models embedded in the RVE, therefore, there was no softening behavior predicted from FEA. Nevertheless, the incorporation of the damage model in the matrix material could be incorporated in future work.

The mechanical behavior of a tensile specimen consisting of a porosity map corresponding to a solidification cooling rate of 750 K/s was comparable to global mechanical properties in Fig. 8.16 of the tensile specimens, as shown in Fig 8.21a-d.

Figures 8.21a-c show the RVE with applied load at three different orientations and show how the localized pores change shape depending on the alignment and orientation. Figure 8.20d shows all three localized ABAQUS results with the global experimental results. Compared to the global experimental results in Fig. 8.16, the local mechanical behavior also exists as variation in the
elastic modulus with orientation, and the modulus is highest for the “C” direction because of the lack of aligned pores in the tensile specimens in that direction. Hence, mechanical behavior can be observed on a point-by-point basis, with a proposed framework that can ultimately predict for porosity and mechanical properties at localized areas.

Figure 8.21. Shear bands and stress fields surrounded pores for a cooling rate of 750 K/s with the resulting stress-strain curves for the localized are: (a) the load was applied at the right of the RVE; (b) the load was applied at the top of the RVE; (c) the load was applied at a 45 angle; (d) All three local RVE curves with the global stress strain curves (see Fig. 8.16) at the three tested orientations.
DED-processed components exhibited highly inhomogeneous and anisotropic behavior due to the distinguished thermal histories in each localized area, which were highly dependent on process parameters, tool path, component geometry, and material compositions. Porosity was a microstructural feature found in many DED-processed components. Larger, irregular shaped pores were more prevalent in areas with greater solidification cooling rate due to lack of fusion, whereas areas with lower solidification cooling rate resulted in vaporization and gas entrapped pores.

This study implemented a framework approach to investigate the relationships between the process parameters that were used as inputs into a thermal model that tracked melt pool evolution, the cooling rate during solidification of the melt pool, porosity characteristics, and the resulting mechanical behavior. Localized information within AM materials was required to predict for potential points of failure and for process control. The size, roundness and dispersion of pores provided sufficient information for modeling for various types of damage. The statistical relationships between experimentally-determined porosity descriptors and simulation-determined solidification cooling rate in this study can lead to reduced order modeling efforts.

8.7 Summary

Charpy impact, microhardness, and tensile testing were used to quantify the performance of DED-processed materials. The tested mechanical properties were related to process parameters, thermal histories, microstructures and porosity. Ultimately, these efforts enable the prediction of structure and behavior as a function of process parameters and closed-loop “online” monitoring during the DED process. Monitoring of the process leads to desirable parts and increased confidence in AM, which can fuel large-scale production of high-functioning parts and new materials. Furthermore,
“online” monitoring for denser parts avoids additional cost and time for post-processing, such as hot isostatic pressing (HIP’ing). A better understanding of local relationships and properties furthers AM research and heightens the capabilities of AM materials.

Mechanical testing can provide data to advance thermal-mechanical models. Refined finite element analysis of the porous representative volume elements can implement 3D porosity maps and better meshing methods for hexahedral elements. Multi-scale porosity assessment can be implemented into a modified Gurson–Tvergaard–Needleman material model to predict ductile behavior of a variety of DED-processed materials. Future work can also include collaborating with those with models that couple microstructure with porosity data. Future work can also explore other testing techniques, including fatigue testing, creep, and corrosion for material performance.
Chapter 9: Overview

This chapter provides an overview of the conclusions from this work. Monitoring of the DED process and characterizing the final parts show how final material properties are sensitive to processing conditions. Section 9.1 summarizes the research with Section 9.1.1 discussing the conclusions gleaned from characterizing the DED process relative to porosity formation and thermal history. Section 9.2 provides ideas for future work in laser-matter interactions, particularly in characterizing the process (Section 9.2.1) and using an understanding of additive manufacturing processing to build new materials (Section 9.2.2).

9.1 Summary of research

The presented work considered DED processing of 316L SS, Inconel 718, and Ti-6Al-4V powder particles. The interactions of the laser and particles were investigated by characterizing the process and the resulting built materials. Processing conditions had a significant influence on resulting thermal histories, microstructures, and mechanical behavior. Porosity in as-built additively manufactured parts is a unique defect that is often the initiation site for crack propagation and other failure modes. This work investigated the relatively unknown mechanisms in which porosity forms and evolves during the DED process using in-situ methods (9.1.1) as well as characterized the process-structure-property relationships with porosity using ex-situ characterization methods (9.2).
9.1.1 Characterization of the DED process

At the time of this work, the physics that occur at the melt pool during DED additive manufacturing are relatively unknown with few assumptions made about surface convection for predictive thermo-mechanical models. This work characterized the process using IR thermography for large powder flow rates and high-speed X-ray imaging for low powder flow rates. Work with the high-speed 32-ID beamline at the Advanced Photon Source (APS) in Argonne National Laboratory monitored porosity formation and melt pool dynamics of DED in real time, demonstrating the influence of powder deposition. Physical phenomena observed include Marangoni flow, melt pool spattering, entrainment, porosity formation, porosity stirring, porosity shrinkage, epitaxial solidification, and vapor-driven powder scattering.

Characterization of the DED process requires an investigation into the laser power absorptivity of in-flight powder particles and the underlying substrate. *In-situ* X-ray monitoring captured how particles absorbed the laser beam by measuring the changes in the cavity and melt pool geometries. Porosity formed in keyhole mode due to a collapse of the cavity and evolved by either shrinking due to gas solubility within the melt pool, “popped” to the surface due to buoyancy overcoming melt pool convection, or was pinned at the solid-liquid interface as the melt pool solidified. Rapid cooling often led to porosity in real-time, with an increase in powder flow influencing whether porosity was keyhole porosity or resulted in micro-pores. Increased powder flow into the melt pool also led to decreased cooling rates, as observed by both X-ray imaging of a micro-scale DED process and IR thermography of an industrial-scale DED process. *Ex-situ* characterization of laser
absorptivity was made by measuring the dilution of clads and their dependence on process parameters, including laser power and powder flow rate.

9.1.2 Characterization of the DED-processed materials

Additive manufacturing processes allow for rapid, scalable and flexible processing of materials that have exhibited superior mechanical properties. Solidification rates observed in this work during DED were as high as about $10^6$ K/s, leading to materials that underwent unique phase transformations and formed porosity of various shapes, sizes, and orientations. This work revealed aimed to link process parameters with thermal history and porosity; and porosity with surrounding microstructure and mechanical behavior.

This work investigated the links between process parameters used in processing Inconel 718, such as the dwell time between layers or powder flow rate, and the resulting microstructures like dendritic arm spacing. Collaborative work on thermal models led to verification and further development of these models by incorporating geometric, microstructure, and microhardness studies. Relationships in DED-processed Ti-6Al-4V were made between processing conditions and thermal histories at varying orientations and locations. Cooling rates were linked to structural and porosity size, shape, orientation, and distribution. Porosity was found to be dependent on processing conditions, such as build direction, hatch spacing, layer thickness, and laser power. In turn, the various distributions, shapes, sizes, and orientations of porosity influenced tensile behavior. These relationships led to global and localized frameworks for predicting for resulting material properties of DED-processed materials.
9.2 Future research

Advances in manufacturing research rely on understanding and harnessing the underlying fundamental physical phenomena of manufacturing processes. Future research will require interdisciplinary collaborations in photonics, X-ray science, materials science and operations research. More specifically, work in the phase changes and deformation of materials during processing and how the driving physics can lead to novel materials, hybrid processes and systems where repeatability in additive manufacturing is not restricted to individual machines but is universal across the process.

9.2.1 In-situ process monitoring of additive manufacturing

Due to rapid directional solidification in additive manufacturing, particularly in the DED processes, materials undergo unique phase transformations and change in properties and mechanical behavior. To measure and predict for component performance, in-situ process monitoring is necessary for the various physical phenomena that occur during the interaction between a laser beam, in-flight metallic particles, and the melt pool. In addition to continuing synchrotron-based high-speed imaging of the melt pool at user facilities, changes in localized material properties, including precipitates and phase transformations at the melt pool and of the entire part can be monitored.

These changes in material properties also include thermal conductivity and heat flux, which could be used to keep track of phase changes during a build. In addition to measuring changes in the built material, future work can include the monitoring of variations in the conditions surrounding
the melt pool, including the dramatic increase of the pressure of inert gas, which increases the refractive index of the gas (Borzsonyi, 2010). The high-pressure gradients of the expanding vapor-plasma plume at the melt pool combined with increased pressure of the surrounding inert gas could cause laser attenuation or scattering of powder particles during deposition (Klassen, 2014). Ray tracing of the laser’s interaction with in-flight particles can be achieved with further monitoring, including high-speed infrared thermography. The ability to quantify the changes in laser absorptivity in the process will allow for predictive analyses of the thermal history in the DED-processed component. Experimentally observed changes in material properties and processing conditions can be used as inputs to mechanics-based models that incorporate thermal changes and ray-tracing of the laser beam. Process monitoring that investigates the physics underlying changes in material properties can inform ways to design for new materials and verify materials and system models that enable process control.

Future work includes monitoring of new methods of processing functional materials such as magnets. New methods of processing magnetic materials include introducing an electromagnetic solenoid ring that surrounds the local melt pool, controlling the convection, and therefore the stirring of the melt pool. The stirring of the melt pool would prohibit grain growth of large columnar grains and encourage nucleation of refined grains. During solidification within the external electromagnetic field, the refined grains would magnetize as they fall below the Curie temperature and align into magnetic domains. Melt pool stirring would be particularly beneficial for the processing of magnetic metal matrix composites (MMCs) by homogenously distributing permanent magnet particle as the reinforcing material. Changes in magnetic properties during the
build of permanent magnets or other ferromagnetic materials can be monitored using an array of Hall sensors within the build plate. In addition, observing the process using \textit{in-situ} high-speed localized X-ray diffraction can capture the micro- and nano-scale phase transformations during solidification that lead to magnetic domains.

\textbf{9.2.2 New materials development and process modifications}

As additive manufacturing emerges and is posed to be an integral part of the next industrial revolution, researchers find that this also opens the door for a materials design revolution, especially for complex components in biomedical and aerospace applications that require specific properties and rigorous certification (Pollock, 2016).

Coupling experimental observations from \textit{in-situ} process monitoring and thermal models can provide an overall understanding of DED processing. The ability to control for unique thermal histories can give rise to qualification of rapidly solidified materials development. Materials in development that would be ideal for DED processing can be classified as "4D printed materials" because of their functional responses and dynamic properties during and after processing. For example, the DED process can take advantage of rapid solidification for the design and thermal control of functionally responsive materials such as shape memory alloys.

As a continuation of the processing of magnetic materials, exploration of new magnetic alloys can arise from new processing monitoring techniques. Multiple feeds of materials with varying chemical compositions can enter the laser-induced melt pool, functionalizing the built part as the
melt pool solidifies. A variety of magnetic reinforcements with different shapes, sizes, and orientation can functionalize the part in controlled ways. In addition, low-cost and highly complex permanent magnets, magneto-caloric materials and other ferromagnetic materials can be designed by combining the magnetic responses and properties of various materials based on experimental observations and predictive models that show how magnetic properties change. New materials would also include those with optimal emissivity and absorptivity ranges relative to the laser beam.

Designing for new materials expands upon materials characterization and delves into advanced processing and systems that allow for repeatability and optimization. Possible process modifications include modifying the laser source and how the laser beam reaches the in-flight powder or substrate. Pulsed lasers, modulated lasers, and lasers with lower wavelengths than those in the near-infrared region can be used to melt metallic powders more effectively. Pulsed and modulated lasers would alter the interaction time of the material with the heat source, effectively controlling the cooling rate at localized areas. Pulsed laser micro-texturing of laser beam optics like microlens arrays and adaptive optics can also provide more efficient laser beam delivery and desired thermal histories of the melt pool. Modifying the process into a scalable and fast-paced ink-jet writing-like process can allow for a combination of multiple materials. Adding an external magnetic field to a combination stream of materials will also influence the magnetic and mechanical properties of conventional materials to create new alloys.

A well-known challenge in additive manufacturing is the large thermal gradients present in DED-processed components that usually lead to warping. Future work could include the ability to control
large thermal gradients to create "pop-up" materials that deform into its desired shape. On the systems side, a metric that combines all \textit{in-situ} measurements of the process and couples with predictive models can control varying additive manufacturing machines. Though the DED process is reaching maturity in industrial sectors, there are still many prospects in advancing the processing conditions and systems related to the DED process.
References


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Yan, F. a. X., Wei and Olson, Gregory (2015). Microstructural investigation of LENS processed 316L stainless steel. TMS Annual Meeting and Exhibition in Orlando, FL.


Appendix A: Laser-Induced Plasma Micro-Manufacturing (LIPMM)

This section of the thesis work focuses on laser-induced plasma micro-manufacturing (LIPMM) for surface texturing and material ablation. First, overviews of pulsed laser manufacturing and of the LIPMM process are provided (A.1). This work addresses the multi-material capability of the LIPMM process (A.2), including transparent and reflective materials. This work also investigates the influence of an applied magnetic field on material removal in laser-matter interactions and laser induced plasma-matter interactions during the LIPMM process. Crystalline and non-crystalline materials were processed, including polymers, glasses, and silicon, as seen in the highlighted materials in Fig. A.1.

Figure A.1. Materials investigated with LIPMM processing.
A.1 Introduction

A.1.1 Picosecond laser ablation

Picosecond laser ablation allows for high peak intensities at each pulse to vaporize the material. At the time of this work, this laser-based process is deemed the “traditional” processes and is the previous rung in the stepladder for advanced laser-based manufacturing processes. This work discusses new steps in this continuing progression of research, which is the laser-based processes of laser-induced plasma micro-manufacturing (LIPMM).

Picosecond laser ablation requires a pulsed laser, shifting the interaction time of the laser with the free electrons in the workpiece from melting modes to more vaporization modes. Pulsed lasers have pulse widths that range from milliseconds to femtoseconds. The decrease in melting modes is due to the interaction time of the laser with the workpiece material. Due to the short interaction time of each laser pulse, the vaporization does not occur because of a gradual temperature rise. The short interaction time in a pulsed laser allows the laser power intensity to reach peak values that are orders of magnitude greater than those in a continuous wave laser and vaporize a material in a localized area.

Most pulsed lasers that operate with pulse width, $\tau_p$, of tens of picoseconds or less are operated by mode-locked lasers with wavelengths from the ultraviolet (UV) range to the infrared. In a mode-locked laser, an element in the laser resonator formulates the ultrashort pulses that circulate between the two mirrors in the resonator. The circulation of pulses results in a periodic train of pulses with a specific repetition rate (J.A. Armstrong, 1967). In between each pulse and with
periodic train, laser energy is stored in the laser medium, resulting in peak power values up to 100 MW for lasers with pulse widths in the tens of femtoseconds and tens of MW for picosecond lasers. Repetition rates of these picosecond and femtosecond pulse emissions, or the frequency of the pulses, range from 10 kHz to 10 MHz, resulting in average power values of 0.1-10 W (S. Kobstev, et al., 2009).

Figure A.2 compares the maximum peak power values for pulsed lasers to that of continuous wave lasers. Pulse width is measured at the full width at half maximum (FWHM) of the pulse’s power profile, or the temporal width at half of the peak power. For shorter pulses at minimum repetition rates, the peak power is greater, as the laser resonator stores the energy for each pulse duration and periodicity. More specifically, the peak power for picosecond lasers is on the order of $10^7$ W, as denoted in red in Fig. A.2. The coupling of greater peak power and shorter interaction time in comparison to the melting mode of continuous lasers allows for picosecond laser ablation of areas as small as the sub-micron scale (Pronko, et al., 1995).
Material removal with picosecond laser ablation occurs when the laser beam directly interacts with the free electrons in the workpiece and does not allow for a temperature rise in the surrounding area. This results in less heat affected zone and more of an instantaneous shock that removes particles from the surface. Figure A.3 shows the range of laser-matter interaction based on the interaction time (L Lucas, et al., 2012). Picosecond lasers can machine a range of materials, including polymers, tool steels, and titanium (BN Chichkov, et al., 1996). However, the interaction time of picosecond pulsed lasers is not short enough to interact with transparent and highly reflective materials. Picosecond laser pulses are still limited by the photo-ablation limit of materials, including properties such as transparency, reflectivity and surface finish for scattering. Picosecond lasers also exhibit thermo-mechanical modes of ablation, including inducing shock waves and a small amount of heat affected zones due to thermal conduction.

A.1.2 Overview of laser-induced plasma micro-machining (LIPMM)

Laser induced plasma micro-machining (LIPMM) (Kumar Pallav, Saxena, & Ehmann, 2013) is an alternative laser-based process and a relatively new method of micro-machining. LIPMM utilizes plasma generated in a dielectric to physically interact with the workpiece. LIPMM can reduce machining time up to tenfold and can overcome the limitations of other conventional micromachining processes, including laser ablation. LIPMM eliminates transmission and
reflection losses by converting light energy into thermal and mechanical energy. Figure A.4 shows a schematic of how a plasma plume is formed during the LIPMM process.

During plasma generation, dielectric breakdown occurs inside the dielectric media, which may be distilled water, kerosene, mineral oil or another transparent dielectric liquid. Figure A.5 shows an example of plasma generated in a dielectric. When the peak irradiance of the ultra-short pulsed laser beam is greater than the dielectric ionization threshold potential, there is a generation of free electrons leading to the formation of a highly localized plasma zone through optical breakdown of the dielectric. The laser induced plasma is then brought in physical contact with the workpiece, which results in vaporization of the material near surface.
The unique mechanism that occurs in LIPMM is a thermo-mechanical mechanism of material removal instead of photo-ablation. The mechanical component occurs when the plasma’s energy produces shock waves radially from the expanding plasma plume and results in cavitation, replacing the dielectric and applying pressure to the workpiece. The thermal component of the mechanism leads to vaporization. At the end of the pulse, the dielectric fills back into the void and flushes the debris off the surface of the workpiece, thereby facilitating material removal.

A.2 Multi-material capability

A.2.1 Background on machining brittle materials

Brittle materials such as glass and functional ceramics play key roles in the semiconductor, energy, communication, biomedical and aerospace industries. Precision micro-texturing of these materials has been a critical focus in these applications. However, brittle materials pose difficulties in machining due to their susceptibility to crack propagation and fracture (TG Bifano, et al., 1991).
Ductile mode mechanical machining avoids brittle fracture by a below-critical load that induces plastic deformation to remove the material (S Shimada, et al., 1995). Tool-based processes such as diamond turning, grinding, micro-EDM and micro-milling have demonstrated their ability to texture micro features in brittle materials (TG Bifano, et al., 1991). However, these processes are limited by tool wear, high processing times, and the requirement of sub-micron cutting conditions for ductile mode machining (S Shimada, et al., 1995).

Tool-less methods such as short-pulsed and ultra-short-pulsed laser ablation are promising micro-texturing methods with characteristics of superior resolution, control and precision as compared to the afore-mentioned mechanical micro-machining processes (L Uriarte, 2006). However, laser ablation, particularly with nanosecond and picosecond pulse duration, is limited to materials with specific surface or optical properties. Specifically, material removal of brittle materials with high transparency and reflectivity is difficult with laser ablation due to the inefficient coupling of the laser beam with the material owing to optical losses (JF Ready and DF Farson, 2001).

LIPMM has demonstrated its ability to machine a variety of materials such as ceramics, metals, polymers and materials with surface characteristics including transparency and high reflectivity (Pallav, Han et al. 2013, Pallav, Saxena et al. 2013). LIPMM eliminates transmission and reflection losses by converting light energy into thermal and mechanical energy. The results presented in this paper demonstrate the viability of LIPMM on polymethyl methacrylate (PMMA) or acrylic glass, a common transparent material, and on a silicon wafer, a highly reflective material.
Two modes of material removal classify the mechanisms of machining of brittle materials into: plastic deformation on the characteristic slip plane and brittle fracture on the characteristic cleavage plane. These planes correspond to the plane with the maximum shear or tensile stress during machining. Plastic deformation occurs when the resolved shear stress exceeds the critical value in the easy slip direction while cleavage occurs when the resolved tensile stress normal to the cleavage plane exceeds a critical value before plastic deformation (TG Bifano, et al., 1991).

In the domain larger than the micron-scale, the brittle mode of material removal dominates due to micro-cracks and dislocations in the stress field during machining and the fact that the critical resolved shear stress is insensitive to the defects [1]. In the micron-scale domain and smaller, the number of defects in the stressed field during machining decreases and the critical resolved tensile stress normal to the cleavage plane increases, resulting in the ductile mode of material removal. However, both critical stress values are dependent on the environmental temperature (S Shimada, et al., 1995).

A.2.2 Experimental methods – material selection

Effective micro-texturing of optically transparent and reflective materials is vital in applications such as micro-optics, photovoltaic cells, waveguides and diffraction (J Brandrup, 1999). Conventional laser ablation using femtosecond, high energy picosecond or UV wavelength lasers may possibly machine transparent materials, but this process is usually inefficient and expensive (M Shirk, 1998). On the other hand, LIPMM is a low energy, picosecond pulsed laser based process. To show the viability of LIPMM to machine transparent and reflective materials that
would otherwise be difficult to machine with laser ablation or other conventional processes a set of representative materials was selected. In this study LIPMM was used to texture micro-channels on transparent brittle polymer, PMMA, and a reflective ceramic, silicon. Table A.1 summarizes some of the key properties for PMMA acrylic and silicon.

PMMA, or acrylic glass, is an optically transparent thermosoftening plastic and is considered a brittle polymer (J Brandrup, 1999). The acrylic used in this study had a sub-micron finish. Micro-textured PMMA can be used as a super-hydrophobic surface which is used for DNA biofilm deposition, microarray chip manufacturing, and biomechanical probes for the characterization of cells and tissues (A Accardo, 2001). Micro-texturing of PMMA has been a challenge due to its brittle behavior under load, its sensitivity to scratching, and crack propagation during machining (A Accardo, 2001), (S Megelski, 2002).

<table>
<thead>
<tr>
<th>Property</th>
<th>PMMA (Brandrup, 1999)</th>
<th>Si (Petersen, 1982)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tensile strength</td>
<td>~70 MPa</td>
<td>~7000 MPa</td>
</tr>
<tr>
<td>Young’s modulus</td>
<td>3.3 GPa</td>
<td>155 GPa</td>
</tr>
<tr>
<td>Fracture toughness</td>
<td>0.7–1.6 MPa.m$^{1/2}$</td>
<td>1.05–1.19 MPa.m$^{1/2}$</td>
</tr>
<tr>
<td>Hardness</td>
<td>63 -97 Kg/mm$^2$</td>
<td>850 Kg/mm$^2$</td>
</tr>
<tr>
<td>Melling temperature</td>
<td>160°C</td>
<td>1414 °C</td>
</tr>
<tr>
<td>Thermal conductivity</td>
<td>1.93 W/mK</td>
<td>141 W/mK</td>
</tr>
<tr>
<td>Dielectric constant</td>
<td>3.5 (Low Freq.)</td>
<td>11.7</td>
</tr>
<tr>
<td></td>
<td>2.57 (High Freq.)</td>
<td></td>
</tr>
<tr>
<td>Index of refraction (at 532 nm)</td>
<td>1.51</td>
<td>3.49</td>
</tr>
</tbody>
</table>

This study also investigates the machining of a silicon wafer with a mirror-finish surface quality with a 10 nm Ra value. Silicon is a highly brittle ceramic with a high melting temperature and,
therefore, very susceptible to crack propagation. Micro-textured silicon is in high demand for efficiency-enhanced solar cells, as well as other semiconductors and MEMS devices (P Papet, 2006).

A.2.3 Experimental methods – laser setup and process parameters

The LIPMM apparatus consists of a commercially available Nd-YVO₄ laser with 8 ps pulse duration operating at its second harmonic 532 nm wavelength. The workpiece of either PMMA or Silicon is secured flat on the bottom of a petri dish with a transparent dielectric media, in this case distilled water, poured into the dish to about 2 mm above the surface of the workpiece. The workpiece setup is, at room temperature, mounted on a 5-axis motion stage with a translation resolution of 10 nm and rotational resolution of 0.0001 degrees. During machining, the Gaussian beam (M² < 1.2) of the laser is brought to focus within the distilled water with a 25 mm triple-element focusing lens to a diffraction-limited focus of 10 μm spot size (1/e²). Here, a stable plasma plume forms and is translated in the Z-direction to make contact with and ablate the surface of the workpiece by a combined thermal and mechanical action as described above.

The experiments consisted of texturing multi-pass channels 100 μm in length by first translating the plasma plume to create one micro-channel and then translating over the original channel for three additional overlaps at the same feed rate to increase the channel depth. Each pass over the channel was constituted of consecutive plasma discharges that created overlapping micro-craters at a feed rate of 12 mm/min. Micro-channels were machined at ten varying heights of the laser beam focal spot relative to the surface of the workpiece due to poor visibility of the CCD camera
and to determine the machining depths best suited for each material. Figure A.6 demonstrates the ten varying machining depths. With each depth, the beam focal spot was offset vertically relative to the workpiece at 50 µm intervals between successive channels.

![Figure A.6. Representation of micro-channel fabrication at different positions of focal spot relative to the workpiece surface.](image)

The three frequency values used were 10, 20 and 50 kHz, yielding a maximum pulse energy of 6 μJ with 10 kHz after reflection and transmission losses within the beam delivery system.

A.2.4 Experimental methods – Microtexture characterization

All features were characterized using an Alicona Infinite Focus Measurement (IFM) 3D metrology system with sub-micron optical resolution. Polarization and white light were utilized to view the transparent and reflective material more effectively. Optical microscopy was used to characterize any surface cracking and heat affected zones (HAZ) by using a Nikon Ephilpot TME Inverted Microscope.

A.2.5 Results and discussion – PMMA (acrylic)

As an optically transparent and brittle polymer, acrylic is difficult to micro-texture by conventional laser ablation. Though ten channels were processed using LIPMM at varying Z depths, from 250
μm below the determined focal spot to 200 μm above, only four or five channels were machined into recognizable channels, depending on the frequency setting. Figure 3 shows the four visible channels machined using a 50 kHz pulse repetition rate at depths from 100 μm below the camera’s focal point on the left to 50 μm above on the right. Looking at Figure A.7, surface cracking surrounding the channels is apparent. The maximum depths were found to be up to about 30 mm for the 10 kHz frequency, and aspect ratios were up to about three.

![Figure A.7. Machined channels at 50 kHz repetition rate.](image)

Figure A.8 shows the profile of a channel machined at 50 μm below the focal point of the laser beam and plasma on the workpiece and the channel with the greatest depth. Here, the depth of the profile is 28 μm and the width is 10 μm, or a channel aspect ratio of 2.8. The average depth of the channel machined with process parameters of 10 kHz and at 50 μm below the focal point is 31.67 μm with an average aspect ratio of 2.29. As seen in Figure A.8, there is a build-up surrounding the channel though this specific area of the channel maintains flatness at the bottom and sidewalls.
Figure A.8. Example a profile of a channel in PMMA at 10 kHz.

The graph in Fig. A.9 shows the variation of feature depth with power and Z machining depths. As seen in Fig. A.7 and as demonstrated by the variance of the data in Fig. A.9, the channels are not evenly machined as there is build-up within the channels. This is most probably due to the feed rate and the low thermal conductivity of the material.

There is an increase in channel depth with Z depth relative to the focal point at lower frequency or higher power settings, whereas the channel depth is comparatively constant with higher frequency and lower power. In general, channels with more even and consistent machined areas with greater channel depths and aspect ratios were found at the 10kHz frequency setting.

In addition to examining the channel depths, surface cracking was examined. Larger and more frequent surface cracking was found in channels machined at the 20 kHz and 50 kHz frequency levels. However, only cracking present in channels machined at 10 kHz were closely examined because of their greater depths and aspect ratios.
Figure A.9. PMMA average feature depth with LIPMM.

Figure A.10 is an example of this surface cracking of a channel machined at 50 µm below the focal point and at 10 kHz. During machining, crack propagation is relatively fast due to the PMMA’s low fracture toughness and hardness properties, resulting in channels with nonparallel channel sidewalls. The red circles indicate notable surface cracks that propagated from the machined channel perpendicular to the machining direction. A more magnified examination using scanning electron microscopy (SEM) could possibly reveal more micro-cracks and sub-micron cracks. The micro-cracks that propagated into the bulk material during machining were as long as 11.08 µm and there were more “large” cracks in the channels machined at 50 µm below the focal point. However, channels machined at depths above the focal spot displayed less even channel walls and more non-vaporized PMMA debris surrounding and within the channel. These micro-cracks could lead to toughening of the bulk material, but also result in poorer surface finish quality of the textured surface.
A.2.6 Results and discussion – silicon wafer

Elemental silicon is widely used in semiconductor electronics, such as integrated circuits and is like common ceramics in mechanical properties. A polished silicon wafer with a 100 nm graphene oxide layer is highly reflective, making it difficult to machine with conventional laser ablation, especially at the nano or pico second time scales, due to reflection losses. However, the picosecond laser used in LIPMM could machine channels with depths of up to about 50-60 µm. Unlike LIPMM in acrylic glass, the micro-texturing of silicon resulted in channels with little to no debris at all machining depths. Figure A.11 shows the surfaces of ten channels machined at 10 kHz as compared to the channels machined at 50 kHz, from 250 µm below the focal point on the left to 200 µm above the focal point on the right. It can be also observed, that surface cracking is minimal around the channels machined at 10 kHz.
The maximum depths were found at the 10 kHz frequency and at a Z machining depth of 100 µm below the focal point. The maximum depth is 64 µm with an aspect ratio of 2.13. Figure A.12 shows the profile of a channel machined with these optimal parameters. As it can be seen, the average depth of the channels is 55.25 µm with an average aspect ratio of 2.07. In addition, there is a slight build up surrounding the channel.
Figure A.12. Example profile of a Si channel at 10 kHz.

The graph in Figure A.13 shows the pattern of the feature depth as a function of power and machining depths for the five channels nearest the determined focal point above the workpiece surface. Like the LIPMM-processed PMMA, there is more of an increase of channel depth with Z depth relative to the focal point with lower frequency. At an intermediate frequency setting, the maximum depth is achieved when machining at the focal point. As the frequency increases and the power decreases, the variance throughout a channel increases due to uneven machining and debris build-up.

Figure A.13. Average feature depth in silicon with LIPMM.
Surface cracking of silicon occurred within the heat affected zones (HAZ) surrounding the machined channels, which were greater than any HAZ around the PMMA channels due to silicon’s much greater hardness. HAZ is susceptible to cracking due to the residual stresses caused by the shrinking of the un-melted silicon during cooling (ZB Hou, 1994). Therefore, existing cracks or susceptibility to crack propagation was measured by examining the HAZ surrounding the channel, especially at channel ends where the stress of the bulk material onto the channel are the greatest. Figure A.14 is an example of large cracks that propagated out the end of a Si channel.

![Image](image.png)

Figure A.14. Cracks up to 50 µm at the end of a Si channel.

As seen in Figure A.11, the HAZ area increases with greater laser frequency. Observed cracks and HAZ areas were found to be up to 50 µm long or thick surrounding the channel. The cracks and HAZ areas also decreased as the Z depth of machining increased to below the focal point, unlike the cracking pattern of LIPMM-processed PMMA.
A.2.7 Conclusions

This study contributes to a proof of concept of LIPMM’s capability to micro-texture two brittle materials that are machining challenges for conventional laser ablation. Moreover, LIPMM is a relatively inexpensive and tool-less process with fast feed rates and low power. Channels with depths of more than 35 µm in PMMA and more than 55 µm in silicon with aspect ratios greater than 2 for both materials were machined using LIPMM.

Several of the challenges in the LIPMM process include micro bubbles that form and adhere to the surface around the machined features during vaporization of the workpiece. These bubbles refract the laser beam and can disturb the resolution and geometry of the features during machining.

Future work includes investigating many more brittle materials of varying hardness, transparency and reflectivity, including glass, silicon carbide and many others. In addition, the LIPMM process is not thoroughly understood, and plasma manipulation via experimental work and multi-physics simulations can provide insight to the plasma-matter interaction and its many possibilities.

A.3 Magnetically-assisted LIPMM (MA-LIPMM)

For centuries, magnetic materials have contributed to technological and societal advancements, including the discovery of novel and improved materials and manufacturing processes, and even more contributions to the understanding of fundamental physics. Magnetic materials and external magnetic fields that induce magnetic properties are ubiquitous – they have applications in energy, aerospace, semiconductors, medical devices, quality control, among many others. New materials
and manufacturing processes that manipulate magnetic fields and properties have the potential to faster processing time and improved mechanical properties of materials. The purpose of this work is to investigate the influence of an applied magnetic field on material removal in laser-workpiece interaction and laser induced plasma-workpiece interaction.

A.3.1 Background on magnetically-assisted manufacturing

Magnetic-field-assisted polishing covers a wide range of surface characteristics by careful selection of magnetic particles and abrasive particles with roughness values from 100 µm to 1 nm. Magnetic field-assisted polishing can enhance surface characteristics such as wettability or reducing friction and can assess hard to reach areas such as the inner diameter of tubular components, and can modify roughness without altering form. The setup of a magnetic field-assisted polisher is independent of workpiece material – the process can efficiently finish ceramics, stainless steels, carbides, coated carbides and silicon. The flexible application of force and pressure distributions of the magnetic field assisted particles reduce the cost of assembly (H Suzuki et al., 1989).

The major limitation is in scalability. The high energy density from using either electromagnets or permanent magnets allows for minimal overheating for constant flux density and the process is overall low cost and easy to integrate into existing CNC equipment. The combination of the tangential force and normal forces exerted by the brush (filled with abrasive particles) onto the workpiece removes material from the top peaks of surface asperities. Fig. A.15 shows an example of magnetic-assisted polishing (H Yamaguchi, et al., 2007). Magnetic abrasive finishing has a
resolution of about 1 µm to 2 nm by means of iron particles for freeform finishing. Magnetoherological finishing is the shearing of viscous mixture of micron sized iron particles, abrasives and oil to impart a machining force onto the workpiece surface and is common for nonmagnetic materials and sub-nano scale polishing. Magnetic fluid finishing has a resolution of <1 nm and is used mainly for RMS and X-ray reflection applications (S Singh, et al., 2002).

In these finishing processes, an alternating magnetic field drives the magnetic fluid which contains abrasive slurry to flush mirror chips in micro-scale fabrication processes. The magnetic field drives the fluid but does not directly work on the workpiece. The media containing magnetic abrasive particles flows under pressure. The workpiece is surrounded by a specially designed electromagnet fitted into non-magnetic fixture plates. While media flows through the workpiece cavity, the magnetic lines of force from the electromagnet poles attract the abrasive particles towards the internal surface of the workpiece. In magnetically assisted abrasive flow machining, (MAAFM), media is subjected to extrusion pressure and additional magnetic pressure (S Singh, et al., 2002).
Conclusions from a MAAFM study have shown that the application of a magnetic field around the workpiece while being processed by flow-machining enhances the material removal rate for non-ferromagnetic work materials. The magnetic field did not appreciably improve the surface of aluminum workpieces (only about 2% contribution), but significantly improved the surface for brass (88%). The magnetic field has a strong interaction with number of cycles in material removal (S Singh, et al., 2002).

Most magnetic field assisted micromachining has been implemented as a supplementary process during EDM and other hybrid methods. A rotational disk fastened with two magnets and driven by an electrical motor is set underneath the EDM machining zone so that the magnetic force assisted device would facilitate the expelling of machining debris more easily and quickly. The implementation of a magnetic field in EDM reduces the adverse effect of accumulated debris and is measured by the number of abnormal electrical discharge waveforms observed due to massive machining debris accumulated in the machining gap. The material removal rate (MRR) of magnetically-assisted EDM is three times compared to conventional EDM with a decrease in surface roughness, improved process stability, smoother machined surface topography and reduced thickness of any recast layers and surface cracks on the machined surface (Lin, Y. C., & Lee, H. S., 2008). Further research has investigated this process in conjunction with ultrasonic vibration machining and other hybrid processes, with similar desirable results. Studies of EDM machining of sintered permanent magnets and magnetic materials showed the feasibility of machining high-resolution features on the order of a few microns (Abdullah, A., Shabgard, M. R., Ivanov, A., & Shervanyi-Tabar, M. T., 2009).
Other studies have demonstrated the use of a magnetic field in laser micromachining. One study looked at the influence of a magnetic field in laser ablation of a highly reflective Al alloy for drilling a micro-hole (Chang, Y. J., Kuo, C. L., & Wang, N. Y., 2012). With a static magnetic field, the machining depth increased by multiple times and the inlet diameter decreased by 42%, improving the aspect ratios of the drilled holes as well as improving the roundness. To micro-machine NdFeB (a thick ceramic magnetic material), a nanosecond pulsed Nd:YAG laser in air and water showed that cutting in water (possible plasma formation) had best cut quality, with a narrower cut width and elimination of debris (A Kruusing, et al., 1999). Further investigation of the underlying physical phenomena during the magnetic material and plasma interaction had not been done, however.

A.3.2 Plasma-magnetic field interaction

In plasma, electrons have complete freedom in movement as they are separate from atoms, accumulating into a collective gas consisting of positively and negatively charged particles, or ions and electrons (D Naujoks, 2006). In magnetically-confined plasma, magnetic surfaces impede the flow of the currents of the electrons and ions. The most efficient magnetically-confined plasma systems are tokamaks and stellarators where the magnetic field lines are bent into a torus in both poloidal and toroidal directions to avoid significant parallel losses, as shown in Fig. A.16 (S Akaslompolo, 2015).
The Debye shielding layer in plasma has a greater density of positive ions with an overall excess of a positive charge, which arises when the temperature of electrons in the plasma are an order of magnitude or greater than that of the ions and are more lightweight (Naujoks, D., 2006). This layer is defined by length $\lambda_D$ which decreases with an increasing plasma density and increases with increasing electron temperature because shielding occurs mostly because of the electrons and their high mobility (Naujoks, D., 2006).

The Debye sheath layer thickness determines the minimum length for electromagnetic fields to have an influence on the motion of particles, as seen in Fig. A.17 (Naujoks, D., 2006). The Debye length helps determines the influence of the effect of a magnetic field geometry. The electric sheath is a thin layer between a plasma and a solid material, where electrons are repelled and the slower ions are attracted. Without the presence of a magnetic field, this sheath length is about a few Debye lengths (S Akaslompolo, 2015). The magnetization parameter, $\xi$, and the angle, $\alpha$, between the magnetic field lines and the surface plane determine the effect of a magnetic field. The magnetization parameter is a unitless ratio of the gyro-radius of electrons and ions with the Debye length (Naujoks, D., 2006). For ideal fusion experiments where magnetic fields have a large influence on high density plasma in tokamak or stellarator systems, $\xi_e$ is about one, the electron
gyro-radius is nearly equal to the Debye length, and $\xi_i \gg 1$, or the ion gyro-radius is much larger than the Debye length (Naujoks, D., 2006).

![Figure A.17. Description of the Debye length (Naujoks, D., 2006).](image)

A.3.3 *Plasma-solid interaction for ablation*

In plasma-solid interactions, the solid material is subjected to a large power load and bombardment of high energy particles from the plasma (Naujoks, D., 2006). Simultaneously, there is cooling in the boundary layer, or sheath, due to low-energy plasma particles and the solid’s wall atoms entering the plasma (Naujoks, D., 2006). The interaction is complex due to the simultaneous changes in properties in both the plasma and the solid material. Some of the processes in this interaction include reflection or absorption of plasma power, resulting in melting or sublimation of the solid material; bombardment of particles onto the solid would result in backscattered electrons back into the plasma, sputtering of solid wall atoms and a conversion of some of these sputtered atoms into impurity ions within the plasma and implanted electrons on the solid wall; and a build-up of an electric potential between the plasma and solid (Naujoks, D., 2006). Most studies that focus on plasma-solid interaction concern interstellar plasma and orbiting electronics or gas discharge in controlled fusion with the vessel walls (G. Federici, 2001). However, there is little known about the coupled influences of the magnetic field and plasma on a workpiece material and the material properties on the plasma plume during machining. Studies on air plasma cutting
arc discharging have mostly investigated the heat transfer, power density and the fluid mechanics of the plasma torch and not the influence of the workpiece material nor the plasma-material interactions (Murphy, et al. 2013).

A.3.4 Capabilities of magnetically-assisted LIPMM

An applied magnetic field, or superposition of magnetic fields can determine the boundary of a plasma plume, or magnetized ionic gas. The temporal and spatial scales of 100 nm and 100 ns respectively capture the binary collisions of electrons and ions of the plasma in a static magnetic field (Bussard 1991, Guo, Hu et al. 2011). These increments are of the same order as the pulsed laser wavelength and take into account the Lorentz forces within the plasma plume, or the velocity of the moving charges under the influence of an external magnetic field (Bussard 1991, Dolan 1994, Ethier, Tang et al. 2005).

This work has investigated plasma energy density within a static, unidirectional magnetic field induced by a single Neodymium magnet at various configurations relative to the laser-induced plasma plume. The laser system used was a commercial (RAPID PS, Lumera) Nd-YVO4 solid-state ultra-short pulsed laser available in the Advanced Manufacturing Processes Laboratory (AMPL) at Northwestern. The laser emitted linearly polarized laser pulses of 8 ps pulse duration, at 532 nm wavelength (second harmonic). The beam was redirected to a substrate through a beam delivery system, which included a 5X beam expander and a diffraction limited focusing lens of 25 mm focal length. The beam had a Gaussian (TEM00) profile at laser exit. The petri dish with the distilled water dielectric was mounted on a five-axis motion stage with a translation resolution of
The question of feasibility of MA-LIPMM (Zank, G. P., 1999) arises from analyses of the effects of magnetically-assisted micro-EDM, which shows that an external magnetic field led to relatively higher debris transportation and helped with flushing during micro-machining of micro-features such as deep channels and holes (G Federici, 2001). Magnetic fields can change the shape of plasma; plasma expands along the direction of magnetic force and compresses in the direction normal to the force. In addition, a surrounding magnetic field can increase oscillating frequency of the plasma and therefore, increase its temperature and energy density (Zank, G. P., 1999). The spatial position of plasma relative to the surrounding magnetic field is a crucial parameter. This study observes the plasma and machining characteristics because of various magnetic field configurations around the plasma plume. In addition to observing plasma behavior, more specific objectives include maximizing the aspect ratio of machined spots from the plasma; that is, to move
the plasma downward to machine spots with more depth and with consistent diameters, and to achieve maximum squeezing of plasma to create machined channels on the workpiece.

There are multiple interactions and velocity values of charged particles at the plasma and solid material boundary, or sheath layer. There is simultaneous heating and vaporization at the material wall as well as a cooling of the edges of the plasma plume due to the influx of material wall atoms. It is important to capture the electric potential that results at this boundary to calculate the amount of solid material that is vaporized (Zank, G. P., 1999).

MA-LIPMM was used to machine micro-channels in 304L steel, an un-magnetic material (S Akaslompolo, 2015). The trend observed in material removal agrees with the imaging results, indicating that longitudinal magnetic fields (in both upward and downward directions) create the most intense plasma, followed by the transverse field. Since material removal is facilitated by a combined effect of thermal vaporization and mechanical erosion, plasma with a higher energy density yields higher material removal rates and throughput. The maximal channel depths obtained for the longitudinal and transverse fields was respectively 50% and 30% greater than that obtained in the absence of a magnetic field. A similar but less pronounced variation was also observed in channel width.

To demonstrate the influence of magnetic fields on plasma shape, four arrangements of permanent magnets were made to create confined magnetic fields to stretch or squeeze the plasma within the confinement of a laser pulse, creating deeper or elongated features in aluminum alloys. Table A.2
lists the four configurations (a – d) that were tested, along with the schematics of the setup, and essential observations. These preliminary experiments demonstrate the capability of creating channel-like to spot-like machining with a variety of permanent magnet arrangements. Craters were up to about 100 to 200 µm long, 10 to 30 µm wide and 30 to 150 µm in depth. The craters exhibited aspect ratios of up to about 6. Features machined on the surface of the workpiece exhibited channel-like characteristics closer to the surface of the surrounding magnets and more spot-like characteristics where the field was almost non-existent. The initial investigations on material removal by magnetically enhanced plasmas have established proof of concept, i.e., feasibility of enhancing material removal, aspect ratios, depth and shape using uni-directional and confined external magnetic fields.

All features were characterized using an Alicona Infinite Focus Measurement (IFM) 3D metrology system with sub-micron optical resolution. Polarization and white light were utilized to view the transparent and reflective material more effectively.
Table A.2. Overview of permanent magnet configurations and corresponding machined features.

<table>
<thead>
<tr>
<th>Configuration</th>
<th>(a) 8 neodymium magnets</th>
<th>(b) 12 neodymium magnets</th>
<th>(c) 1 neodymium magnet</th>
<th>(d) 12 neodymium magnets</th>
</tr>
</thead>
<tbody>
<tr>
<td>Magnetic field</td>
<td>5000 Gauss</td>
<td>6000 Gauss (at surface of magnets); 0 Gauss at plasma plume</td>
<td>4400 Gauss</td>
<td>6000 Gauss</td>
</tr>
<tr>
<td>Objective</td>
<td>Squeeze plasma for LIPMM by machining in center of configuration.</td>
<td>Observe how plasma shape changes within an attractive field.</td>
<td>Observe how plasma shape changes at different distances from a single magnet.</td>
<td>Squeeze plasma for LIPMM within a repellent field.</td>
</tr>
<tr>
<td>Machined feature</td>
<td><img src="image1.png" alt="image" /></td>
<td><img src="image2.png" alt="image" /></td>
<td><img src="image3.png" alt="image" /></td>
<td><img src="image4.png" alt="image" /></td>
</tr>
<tr>
<td>Observation</td>
<td>A deep crater is formed with tapered walls, indicating that the plasma was formed in a V-shaped profile due to squeezing close to the focal spot.</td>
<td>An elliptical crater is created but with less tapering of walls indicating uniaxial squeezing of plasma.</td>
<td>The plasma shape changes from circular to oval to more linear (perpendicular to the direction of magnetic lines of force) as it is brought near the magnetic north pole.</td>
<td>Less tapering of walls by repellent fields, indicating a plasma that elongates along the direction of propagation of the pulse (orthogonal to magnetic lines of force) for higher aspect ratio features.</td>
</tr>
</tbody>
</table>