EFFECTS OF LASER INTERFACE SINTERING ON MONOLAYER COPPER MICRO-PARTICLES USING A CARBON DIOXIDE PULSED LASER SYSTEM

A Thesis by

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Bachelor of Engineering, University of Hertfordshire, 2013

Submitted to the Department of Mechanical Engineering
and the faculty of the Graduate School of
Wichita State University
in partial fulfillment of
the requirements for the degree of
Master of Science

December 2018
EFFECTS OF LASER INTERFACE SINTERING ON MONOLAYER COPPER MICRO-PARTICLES USING A CARBON DIOXIDE PULSED LASER

The following faculty members have examined the final copy of the thesis for form and content, and recommend that it be accepted in partial fulfillment of the requirement for the degree of Master of Science with a major in Mechanical Engineering.

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Rajeev Nair, Committee Chair

______________________________________
Gisuk Hwang, Committee Member

______________________________________
Krishna Krishnan, Committee Member
DEDICATION

To my family
ACKNOWLEDGEMENTS

I would first like to take the opportunity to mention, that my advisor, Dr. Rajeev Nair has influenced me to achieve the best of my ability as an engineer. He has introduced and mentored me through an area of research that I have grown fond of and thereby academically motivating me to complete my studies. With his guidance and assistantship support, through my education, I am forever indebted to him as it has evolved me in becoming a better person and engineer.

I would like to give my sincere gratitude to Dr. Gisuk Hwang for his involvement with my work. Along with Dr. Nair, they have both given me the opportunity of working in a project that has allowed me to work and experience the different fields of engineering. I am grateful to the NASA Cooperative Agreement Notice, Grant Number 80NSSC18M0030, for giving me the resources and time to work on such a unique project.

I am deeply thankful to my friends Akhil, Ashwini, Ramya and Kamlesh for their constant support and encouragement through my times of hardship as well as success over the course of completing my research. I would like to express my sincere gratitude to Mahmood Al Bashir for working with me throughout the course of this project. His experience and mentorship has greatly helped me in the completion of this research, and I hope to be of help to him through his endeavors.

I would like to thank my parents Asok and Reepa Sit for providing me with their unconditional love and support throughout my life and giving me the courage to pursue my goals. Finally I would like to thank Aditi and Savitri Sit, my two sisters and best friends, from whom I have always gained the positive support and inspiration to move forward in life.
ABSTRACT

Manufacturing of complex and intricate structures using the laser sintering process are in great demand for various applications such as thermal management, light weight structures, filtration, biomedical systems, and electronic components. Different methods of laser sintering can offer a variety of functions for the structures. However, due to the numerous amounts of factors affecting the quality of sintering, and the bonding between the particles, studies are carried to analyze these factors and their effects on the fabricated structures. The single-component laser surface-sintering method has the unique ability of generating porosity throughout the structure. This study shows the design and methodology of installing a CO$_2$ pulsed microsecond laser capable of these functions, as well as the procedure and analysis of factors affecting successful sintering of copper micro-particles. Results show the successful fabrication of laser sintering of a monolayer of copper micro-particles using a pulse period of 1000 μs, pulse width of 150 μs and a defocused beam with a spot size of 4.73 mm. Experimental tests such as, surface roughness, porosity and thermal conductivity tests and simulations were also carried out to validate the quality of sintering, as well as comparing the results with that of conventionally (furnace) sintered samples to show similar manufacturing quality. The obtained results provide a better understanding and a foundation for the steps towards the fabrication of a 3D porous structure.
# TABLE OF CONTENTS

<table>
<thead>
<tr>
<th>Chapter</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. INTRODUCTION</td>
<td>1</td>
</tr>
<tr>
<td>2. LITERATURE REVIEW</td>
<td>5</td>
</tr>
<tr>
<td>2.1 Direct Method</td>
<td>5</td>
</tr>
<tr>
<td>2.2 Indirect Method</td>
<td>6</td>
</tr>
<tr>
<td>2.3 Two Component Method</td>
<td>6</td>
</tr>
<tr>
<td>3. INSTALLATION OF CO₂ LASER SYSTEM AND SUPPORTING SYSTEMS</td>
<td>11</td>
</tr>
<tr>
<td>3.1 Laser System</td>
<td>11</td>
</tr>
<tr>
<td>3.2 Laser System Installation</td>
<td>15</td>
</tr>
<tr>
<td>3.3 Laser Alignment</td>
<td>18</td>
</tr>
<tr>
<td>3.4 Supporting Systems</td>
<td>20</td>
</tr>
<tr>
<td>4. EXPERIMENTAL PROCEDURE</td>
<td>24</td>
</tr>
<tr>
<td>4.1 Test Sample Preparation</td>
<td>24</td>
</tr>
<tr>
<td>4.1.1 Powder Delivery System</td>
<td>26</td>
</tr>
<tr>
<td>4.2 Laser Sintering Trials</td>
<td>27</td>
</tr>
<tr>
<td>4.3 Numerical Modeling</td>
<td>29</td>
</tr>
<tr>
<td>4.4 Sintering Approach</td>
<td>31</td>
</tr>
<tr>
<td>4.4.1 Single point approach</td>
<td>31</td>
</tr>
<tr>
<td>Chapter</td>
<td>Section Description</td>
</tr>
<tr>
<td>---------</td>
<td>---------------------</td>
</tr>
<tr>
<td>4.4.2</td>
<td>Single pass approach</td>
</tr>
<tr>
<td>4.4.3</td>
<td>Multiple pass approach</td>
</tr>
<tr>
<td>4.5</td>
<td>Design of Experiments</td>
</tr>
<tr>
<td>4.3</td>
<td>Conventional Furnace Sintering Procedure</td>
</tr>
<tr>
<td>5.</td>
<td>RESULTS AND DISCUSSION</td>
</tr>
<tr>
<td>5.1</td>
<td>Physical Properties of the Particle</td>
</tr>
<tr>
<td>5.2</td>
<td>Roughness Test</td>
</tr>
<tr>
<td>5.3</td>
<td>Wettability Test</td>
</tr>
<tr>
<td>5.4</td>
<td>Thermal Conductivity</td>
</tr>
<tr>
<td>6.</td>
<td>CONCLUSION</td>
</tr>
<tr>
<td>7.</td>
<td>FUTURE WORK</td>
</tr>
<tr>
<td>8.</td>
<td>REFERENCES</td>
</tr>
<tr>
<td>9.</td>
<td>APPENDICES</td>
</tr>
<tr>
<td>I.</td>
<td>APPENDIX A</td>
</tr>
<tr>
<td>II.</td>
<td>APPENDIX B</td>
</tr>
<tr>
<td>III.</td>
<td>APPENDIX C</td>
</tr>
<tr>
<td>IV.</td>
<td>APPENDIX D</td>
</tr>
</tbody>
</table>
## LIST OF TABLES

<table>
<thead>
<tr>
<th>Table</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 - Absorptance (A) of a single-component powders measured with a CO₂ laser[26]</td>
<td>8</td>
</tr>
<tr>
<td>2 - Coherent Diamond J-3 Series Laser Specifications [38]</td>
<td>12</td>
</tr>
<tr>
<td>3 - Material Properties of Copper Particles</td>
<td>24</td>
</tr>
<tr>
<td>4 - Power levels at different parameters</td>
<td>28</td>
</tr>
<tr>
<td>5 - Significant factors affecting sintering</td>
<td>34</td>
</tr>
<tr>
<td>6 - Combinations of factors producing successful sintering</td>
<td>35</td>
</tr>
<tr>
<td>7 - Final fabrication parameters used for sintering</td>
<td>35</td>
</tr>
<tr>
<td>8 - Mass of water trapped in laser sintered sample</td>
<td>41</td>
</tr>
<tr>
<td>9 - Mass of water trapped in furnace sintered sample</td>
<td>42</td>
</tr>
</tbody>
</table>
**LIST OF FIGURES**

<table>
<thead>
<tr>
<th>Figure</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>Coherent Diamond J-3 Series Laser [38]</td>
<td>11</td>
</tr>
<tr>
<td>3</td>
<td>Laser Beam Quality Enhancement System: BQE-25 [40]</td>
<td>13</td>
</tr>
<tr>
<td>4</td>
<td>Safety Shutter with Diode Pointer SSDP-25 [42]</td>
<td>14</td>
</tr>
<tr>
<td>5</td>
<td>Kinematic Beam Bender: BBK-25 [43]</td>
<td>14</td>
</tr>
<tr>
<td>7</td>
<td>Complete laser system setup</td>
<td>17</td>
</tr>
<tr>
<td>8</td>
<td>Schematic of the laser setup</td>
<td>17</td>
</tr>
<tr>
<td>9</td>
<td>Spot size of the laser at various lengths from the focal point of the lens (conducted in the following parameters: pulse period-1000μs, pulse width 130μs, and number of pulses-100)</td>
<td>20</td>
</tr>
<tr>
<td>10</td>
<td>6000 Series DuraChill Water-Cooled 1.5HP Chiller[49]</td>
<td>21</td>
</tr>
<tr>
<td>11</td>
<td>XYZ-BSMA-LY-140H-300X300X300[50]</td>
<td>22</td>
</tr>
<tr>
<td>12</td>
<td>Argon Gas Enclosure with powder delivery setup</td>
<td>23</td>
</tr>
<tr>
<td>13</td>
<td>CuLox™ 6000 Series 200μm pure copper powder</td>
<td>24</td>
</tr>
<tr>
<td>14</td>
<td>Copper plate substrate</td>
<td>25</td>
</tr>
<tr>
<td>15</td>
<td>3D Printed and 3D Design of mold</td>
<td>26</td>
</tr>
<tr>
<td>16</td>
<td>Powder delivery system inner block with coupler and motor (left) and out block (right)</td>
<td>27</td>
</tr>
<tr>
<td>17</td>
<td>'Balling' effect of particles conjoining together</td>
<td>32</td>
</tr>
<tr>
<td>18</td>
<td>Laser sintered copper particles</td>
<td>38</td>
</tr>
</tbody>
</table>
LIST OF FIGURES (continued)

<table>
<thead>
<tr>
<th>Figure</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>19 - Furnace sintered copper particles</td>
<td>38</td>
</tr>
<tr>
<td>20 - 3D profile of furnace sintered (left) and laser sintered (right) copper micro-particles</td>
<td>39</td>
</tr>
<tr>
<td>21 - Surface roughness of laser sintered particles</td>
<td>40</td>
</tr>
<tr>
<td>22 - Surface roughness of furnace sintered particles</td>
<td>40</td>
</tr>
<tr>
<td>23 - Basic visualization of thermal analysis experiment</td>
<td>44</td>
</tr>
<tr>
<td>24 - Experimental setup simulation with glass plates</td>
<td>45</td>
</tr>
<tr>
<td>25 – Temperature distribution through the monolayer copper sample</td>
<td>45</td>
</tr>
<tr>
<td>26 - Average temperature (°C) of sample over time (s)</td>
<td>46</td>
</tr>
<tr>
<td>27 – Coherent™ J-3 CO2 Laser technical drawing</td>
<td>57</td>
</tr>
<tr>
<td>28 - Laser system layout designed by HAAS™</td>
<td>58</td>
</tr>
<tr>
<td>29 - First iteration of setup installation</td>
<td>59</td>
</tr>
<tr>
<td>30 - Base plate drawing</td>
<td>60</td>
</tr>
<tr>
<td>31 - Outer container drawing</td>
<td>61</td>
</tr>
<tr>
<td>32 - Inner platform container drawing</td>
<td>62</td>
</tr>
<tr>
<td>33 - Leg support drawing</td>
<td>62</td>
</tr>
<tr>
<td>Abbreviation</td>
<td>Description</td>
</tr>
<tr>
<td>--------------</td>
<td>--------------------------------------</td>
</tr>
<tr>
<td>3-D</td>
<td>3 Dimensional</td>
</tr>
<tr>
<td>SLS</td>
<td>Selective Laser Sintering</td>
</tr>
<tr>
<td>LASER</td>
<td>Light Amplification of Stimulated Emission of Radiation</td>
</tr>
<tr>
<td>YAG</td>
<td>Yttrium Aluminum Garnet</td>
</tr>
<tr>
<td>Nd</td>
<td>Neodymium</td>
</tr>
<tr>
<td>CW</td>
<td>Continuous Wave</td>
</tr>
<tr>
<td>HAZ</td>
<td>Heat Affected Zone</td>
</tr>
<tr>
<td>BQE</td>
<td>Beam Quality Enhancer</td>
</tr>
<tr>
<td>IR</td>
<td>Infrared</td>
</tr>
<tr>
<td>CO₂</td>
<td>Carbon Dioxide</td>
</tr>
<tr>
<td>Cu</td>
<td>Copper</td>
</tr>
<tr>
<td>CuO</td>
<td>Copper Oxide</td>
</tr>
<tr>
<td>GUI</td>
<td>Graphical User Interface</td>
</tr>
<tr>
<td>DOE</td>
<td>Design of Experiment</td>
</tr>
</tbody>
</table>
LIST OF SYMBOLS

T  - Temperature

T₀  - Initial temperature of the material

I  - Power intensity

k  - Thermal conductivity

D  - Thermal diffusivity

t  - Laser irradiation time

A  - Absorptivity

R  - Reflectivity

P  - Power output of laser

r  - Radius of spot size

λ  - Wavelength
CHAPTER 1
INTRODUCTION

Additive manufacturing is a manufacturing process of creating an object by building it one layer at a time. It is similar to 3-D printing, where in the product is created by building something up. There are various types of additive manufacturing, with each offering specifically desired functionalities.

One form of additive manufacturing is the Selective Laser Sintering (SLS), which utilizes a high powered laser to bond particles of a material such as metal, plastic, ceramic, or glass[1]. SLS has recently become a popular and preferred manufacturing process choice for producing prototypes and parts requiring low-volume but high quality parts. The aerospace industry has lately taken advantage of the SLS process [2].

Laser, also known in its full form as Light Amplification by Stimulated Emission of Radiation, is the primary constituent to the SLS manufacturing process. As opposed to conventional sources of light, a laser produces a very narrow beam of light propagating over long lengths with minimal divergence. Light from a laser is produced by the release of photons from excited atoms [3, 4].

Initially an energy source, known as the pump source, is needed to supply the external energy to the gain medium. The pump source is usually in the form of an electrical discharge but could also be arc or flash lamps, chemical reactions, or even light from another laser. The gain medium, a material, is excited by the energy from the pump source and thereby creating a population inversion, more specifically inducing a spontaneous emission. This is when an atom at
an excited stage drops to its ground stage, producing a photon. The gain medium come in various materials ranging from liquids such as dye lasers, gases such as carbon dioxide, argon, or krypton, and solids such as glass or crystals like YAG (yttrium aluminum garnet), doped with impurities like chromium, neodymium [5].

The generated photons collide with other atoms in the medium to produce a stimulated emission. This is when the atoms drop from their excited stage to the ground stage, thereby producing two photons of the same frequency. This whole process is amplified by putting two parallel mirrors on either side of the medium, called an optical resonator. More accurately, one of the mirrors is fully reflective, whereas the other is partially reflective allowing some of the light to escape through the exit cavity as laser [5, 6].

There are essentially two major types of operations of lasers. The continuous wave (CW) operation is where the laser is continuously emitted and its power output is constant over time [7]. And the pulsed operation is any laser where the emission of light is non-continuous, i.e., the optical power emerges in pulses of specific duration. These pulse durations can range from microseconds to femtoseconds, depending on the type of pulse laser [8]. Short pulses can be generated by modulating or allowing a certain amount of light to be passed through at regular intervals. This proves inefficient as power is lost at the modulator. A q-switched laser allows the production of pulses with the duration in the nanosecond scale. A mode locking laser can emit extremely short pulses in the duration of picoseconds, or even femtoseconds allowing for very specific and delicate operations [9]. Short or ultrashort pulses have very high intensities because the laser intensity is concentrated in a very short-time duration, contributing to smaller heat affected zone (HAZ) and higher cooling periods. This is very useful in operations such as surface ablation, where the pulse duration allows the material to cool down and not melt and change phase [10].
The direction and behavior of the laser can be manipulated by the use of optical instruments. This is done to accommodate the functional objective of the laser system. Highly reflective mirrors at desired angles alter the direction of the laser and lenses are used to influence the size of the spot size by focusing and defocusing the laser beam.

Laser is the primary source of energy required to sinter particles in the SLS process of manufacturing. Sintering can happen naturally in mineral deposits or in manufacturing processes with materials such as metals, ceramics, glass, or plastics. The material is heated to a temperature of just below its melting point, which allows the boundaries of the particles to fuse together to form a solid structure. One example of conventional sintering processes is the ceramic/furnace sintering, which uses a furnace to apply the heat and pressure to a tightly packed arrangement of particles. After a desired cure time, and cooling period, the particles will have fused together to create a solid structure. Other types of sintering involve, liquid phase sintering, electric current assisted sintering, pressure-less sintering, microwave sintering and laser sintering [11].

Laser sintering has been in use since the mid-1980’s as rapid prototyping to create parts with high complexity [12]. Thermal energy from the laser sinters the particles in successive layers. As each layer is bounded, an additional layer of the particles are laid and the process repeats until a desired solid structure is achieved. The distinctive advantage of the process is that there is no need for support structures as the powder itself provides the necessary support [13]. There are two essential methods of the deposition of the material powder onto the substrate. One is the pre-blown powder deposition method, where the metal powder is deposited through an external nozzle onto the laser-substrate interface. This method is advantageous for rapid manufacturing of large products, but can produce higher tolerances for smaller products. The other method is the pre-
placed powder bed method. This method is more accurate for the building of very small products, up to the millimeter scale. However, this method is more time constrained [14].

With control over the initial parameters of the particle arrangement as well laser parameters, one can finely regulate the amount of porosity, surface roughness, thermal and material properties. The overall reduced manufacturing time is also a major benefit of the laser sintering process over its conventional processes.

The research goal for this project is to fabricate interface-solid-sintered copper micro powder using a pulsed CO$_2$ microsecond laser as a first step towards the fabrication of 3D wick structures. The fabricated samples are experimentally validated by measuring the surface roughness, porosity, permeability and thermal conductivity of the surface as well as numerically validating the thermal conductivity in ANSYS®. Consequently, the successful laser sintering of the copper particles also demonstrate the advantage of reduced manufacturing time over the conventional furnace sintering operation.
CHAPTER 2
LITERATURE REVIEW

Laser sintering is the additive manufacturing process of applying heat onto particles of a material from a laser beam. The principle in laser surface-sintering is to apply just enough heat onto the material that the particles sinter along the interface and not melt completely. This temperature-gradient has to be precisely calibrated such that the melting occurs only along the particle interface of the bulk material and not proceed further into its core, and at the same time ensure that it is not low enough that no interface phase change is possible. Moreover, due to the voids present between the particles, desired porosity can also be achieved. By controlling the various parameters of the laser, many complex structures at the macro and micro level can be manufactured. The following describes the three general methods of laser sintering of metal powders.

2.1 Direct Method

This method involves the application of energy from a laser beam onto the particles of a material without the aid of any other different material. The temperature attained should be close to the melting point of the material but not exceed it. This softens the material and the particles adhere to each other by the interfacial grain contact area. The development of this process is based on material particle fusion [15, 16]. As the temperature approaches the melting temperature of the material, softening of the material occurs. This produces necking between the two particles, thereby adhering themselves onto one another. The accumulation of necking between the particles allows the powdered material to agglomerate and thus create the solid structure desired. The free surface energy reduction can credit the joining of the particles. However, uneven distribution of
heat, such as the localized increase of temperature due to the Gaussian nature of the laser beam can melt some particles even below the melting temperature. When this occurs, some particles may end up joining together due to surface tension and bigger spherical particles may arise. This phenomenon is known as ‘balling’ and is one of the trivial problems to overcome in solid-state laser sintering [17, 18]. Therefore, in the solid-state laser sintering process, the laser spot size, scan speed pattern and speed can all act as a major factor in the even distribution of heat onto the particles. Moreover, as the particles are spherical in nature and only a limited surface area is conjoined, porosity in a structure is generated and can thus be manipulated [19].

2.2 Indirect Method

As opposed to the direct method, the indirect method involves the addition of organic materials with a lower viscosity value than that of the primary metal material. This additional material is used as a coating which aids in the absorption of the infrared energy from the laser and hence, acting as a binder for better adhesion of the metal particles [20]. However, this method does require post processing.

2.3 Two Component Method

This method involves the use of two different metal powder. With different melting temperatures, $T_1$ and $T_2$, the laser parameters are adjusted so that the applied temperature ($T$) is between the two temperatures, such that, $T_1<T<T_2$. At this temperature, it is ensured that the voids between the sintered metal particles will be filled by the melting material of the other metal particles. This creates a structure without any voids and with better structural integrity [17]. However, such a method is not ideal when manufacturing structures with desired porosity.

The figure below shows visual description of the particle interactions during bonding for each method.
Laser sintering of metal powders can be conducted primarily using gaseous pulsed lasers (CO₂ laser, for example) or solid state lasers (such as Nd:YAG) [21] both operating in the infrared region. However, the amount of energy from the laser that is delivered onto the surface depends on the parameters defined by the user as well as the geometry of scanning or fabrication parameters [22, 23]. These are the laser power, spot size, hatch spacing, beam speed/frequency, and vector length. The laser power and spot size, determine the amount of power intensity delivered onto the material. The hatch spacing and vector length dictate the speed and amount of material being covered by the laser beam at a given moment. Each of these parameter themselves are then dependent on each other as well as secondary parameters such as the intensity of the pulse, duration of the pulse, number of pulses, and time duration between each pulse [24].

Figure 1 - Visual interpretation of the particle bonding interaction during laser sintering of single and two-component methods [11]
After the energy is delivered, it must be then analyzed to see how much of that energy is absorbed by the material for sintering to take place. This is dependent on parameters defined by the properties of the material to be sintered as well as some user defined parameters. As the energy comes from light, the optical properties of the material define the amount of light that is absorbed by the material. The absorptivity (A) of a material will dictate this. A higher value of A, requires a higher amount of power intensity input onto the material, and thus is more difficult to control the parameters [25]. Most common pure metals exhibit a lower absorptivity, such as pure copper at 0.26. However, if the material is a compound such CuO (Copper Oxide), the value of absorptivity greatly increases to 0.76 [26]. Absorptivity is calculated as, $A = 1 - R$, where, R is the reflectivity of the material [27, 28]. However, it is important to note that the absorptivity depends on the wavelength and the frequency of the laser that it is exposed with [29, 30]. For instance, it has been calculated that the absorptivity of a material at wavelength of $\lambda=1.06 \, \mu m$, is almost 1.5 - 2.5 times greater than at a wavelength of $\lambda = 10.6 \, \mu m$. Also, it is difficult to determine the exact value of the absorptivity of a material as it changes during fabrication, by removal of oxide films or at some cases the generation of oxides over the particles [31].

Table 1 - Absorptance (A) of a single-component powders measured with a CO$_2$ laser[26]

<table>
<thead>
<tr>
<th>Material</th>
<th>Absorptivity (A) at $\lambda = 10.6 , \mu m$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu</td>
<td>0.26</td>
</tr>
<tr>
<td>Fe</td>
<td>0.45</td>
</tr>
<tr>
<td>Sn</td>
<td>0.23</td>
</tr>
<tr>
<td>Ti</td>
<td>0.59</td>
</tr>
<tr>
<td>Cu-alloy (10% Al)</td>
<td>0.32</td>
</tr>
<tr>
<td>ZnO</td>
<td>0.94</td>
</tr>
<tr>
<td>Al$_2$O$_3$</td>
<td>0.96</td>
</tr>
<tr>
<td>CuO</td>
<td>0.76</td>
</tr>
<tr>
<td>SnO</td>
<td>0.95</td>
</tr>
</tbody>
</table>
Thermal properties of the material, such as the melting temperature and the thermal conductivity determine the amount of energy that is transferred within the material until sintering occurs. For this, the choice of powder will ultimately change all other parameters required for sintering [32]. A higher melting point of the material requires a higher amount of power intensity. Copper has a melting temperature of about 1081°C, and thus a temperature as close to this but not exceeding this must be achieved. This can prove to be difficult to control as opposed to the two-component method, where the melting of the secondary material can act as adherent aiding in the bonding process [33].

In terms of user parameters for the energy absorption, the temperature of the bed is also a factor. The part bed is the substrate onto which the material is to be sintered on. A higher bed temperature can greatly reduce the amount of power required to sinter. This reduces the temperature difference between the in-sintering and after-sintering, also simplifying the process to control all other parameters [32].

The discussed parameters are the major factors that determine the amount of power required to sinter a material together. However, there are many other finer parameters that may have an influence, are yet subject to further study. Such factors could vary from the chemical and physical process of the laser material interaction, control theories, desired geometry of the part, and the orientation of the laser setup, machine characteristics and post processing methods [32, 34].
Regardless, the above parameters are to be considered when developing a relation between the laser power and the initial temperature required to sinter. For this, thermal numerical models have to be employed that is discussed in further detail in Chapter 4.

Most of the laser sintering done in the industry today usually comprise of the two-component method for the manufacturing of parts for achieving better structural integrity and limited porosity [35, 36]. However, this study aims to conduct tests and examine the results of laser sintering performed using the single-component method on copper micro-particles for creating desired porosity as well as maintaining the properties of the material. This can be conventionally done by the use of furnace sintering. However, this is a method that requires a great amount of curing time as well as preparation time, from 6 to 10 hours [37]. This can be greatly reduced to within an hour by employing the single-component method laser sintering. This study aims to create a better understanding of the single-component laser sintering method of monolayer copper micro-particles and thereby creating a foundation for the fabrication of a 3 dimensional porous structure.
CHAPTER 3
INSTALLATION OF CO$_2$ LASER SYSTEM AND SUPPORTING SYSTEMS

To accomplish laser sintering of the copper particles, a laser system accompanied with various supporting systems were installed and tested.

3.1 Laser System

The primary component of the laser system comprises of the Coherent® DIAMOND J-3 Series OEM laser. It is a sealed, microsecond pulsed CO$_2$ laser. This means that it utilizes carbon dioxide as its active laser medium and its laser discharge mechanism is of pulsing characteristic. The latter characteristic is unique to this laser as a CO$_2$ laser is usually a continuous wave (CW) laser, i.e. a laser beam whose output power is constant over time. However, by employing a slab discharge design, the pulsing characteristic of the J-3 laser is able to reach peak powers greater than 750 W[38]. This allows for a wide operating power range as well as good power stability. With pulsed repetitions rates of up to 200 kHz, combination of wavelength selection and high peak power, the J-3 laser is capable of producing an excellent and clean beam quality essential for laser sintering of metal micro particles.

Figure 2 - Coherent Diamond J-3 Series Laser [38]
Table 2 - Coherent Diamond J-3 Series Laser Specifications [38]

<table>
<thead>
<tr>
<th>Specifications</th>
<th>Diamond J-3-10.6</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wavelength (µm)</td>
<td>10.6 ± 0.4</td>
</tr>
<tr>
<td>Output Power² (W)</td>
<td>≥ 250</td>
</tr>
<tr>
<td>Power Range (W)</td>
<td>10 - 250</td>
</tr>
<tr>
<td>Nominal Peak Power³ (W)</td>
<td>≥ 750</td>
</tr>
<tr>
<td>Mode Quality</td>
<td>&lt;1.2</td>
</tr>
<tr>
<td>Beam Waist Diameter at 1/e² (mm)</td>
<td>8.5 ± 1.0</td>
</tr>
<tr>
<td>Full –Angle Beam Divergence (mrad)</td>
<td>≤ 2.0</td>
</tr>
<tr>
<td>Pulse Frequency (kHz)</td>
<td>Single-shot to 200</td>
</tr>
</tbody>
</table>

To ensure that the light from the cavity of the laser is smoothened and is of a non-interruptive nature, additional components are installed to maintain and regulate the properties of the beam.

**Beam Enhancer:**

A beam enhancer is responsible to prevent any back reflections of the laser from re-entering the laser cavity. In addition the laser beam quality enhancement system is also used to preserve and maintain the intra-cavity conditions, mode quality, and power stability. Lastly, another very significant function of this system is to convert the laser beam from a linearly polarized beam to a circularly polarized beam [39]. This is done because there is better absorption of the photons by the material when the light beam is circularly polarized. [40]
In this case, the Laser Beam Quality Enhancement System BQE-25-10.6 by Haas is selected to ensure all the above mentioned functions.

**Safety Shutter:**

The safety shutter in a laser system ensures the safe and clean operation of the laser beam. It is designed to temporarily shut off the laser beam emission for user safety. As the shutter is closed the laser beam is reflected off the shutter blade into a beam dump to terminate the beam. The blade in the shutter is used as fail-safe operation and will automatically close during the absence of power. The safety shutter must also be installed co-linearly with the laser beam in order for successful termination [41]. A visible red light also emits from the shutter cavity once it is closed. This red light is linearly aligned with the path of the laser, and one can determine the location of the spot size as the light from the laser itself is not from the visible spectrum. For this laser system, the Safety Shutter with Diode Pointer SSDP-25-AU-AC-DDD was chosen. [42] The safety shutter also has the option to be liquid cooled and therefore requiring a separate de-ionized water cooling system.
Beam Bender:

A beam bender is used to change the direction of the beam. The orientation of the bender dictates the direction of the beam and is determined as per the user’s requirement. For this laser system and its installation, the BBK-25 Kinematic Beam Bender with a 25 mm clear aperture by Haas® is used. To redirect the laser 90°, the mirror of the beam bender was placed at 45°.

The optics of the bender consists of an orthogonal mirror correction that can provide precise and repeatable alignment. This results in the ease of optic removal and replacement without disturbing alignment. [43]
Process Head:

The process head is the final component before the laser beam is exposed and interacts with the work surface. The G1-25 Laser Process Head by Haas®, is designed for applications such as welding and cutting, however, it is not limited to just these applications. With its modular design is can be custom configured for all wavelengths thereby configuring it for different applications. For the purpose of this study, the optics consist of a 5-inch focal length concave lens. This converges the collimated laser to smaller spot size, thereby increasing the power intensity to its highest value at the focal point. To increase the spot size and thus decreasing the power intensity, the work surface is lowered further away from the focal point so that the light diverges from the focal plane [44].

![Figure 6 - Laser Process Heads: G1-25](image)

3.2 Laser System Installation

The general orientation of the laser and its components is determined by the user and its application. In this instance, it was decided that the laser beam will be projected horizontally and then directed downwards 90° towards the work surface (See Appendix A).
For this, the laser was mounted on top of a frame. The frame is constructed from hollow 1\text{in}^2 carbon steel tubing welded together using shielded metal arc welding (SMAW). A secondary frame of different dimensions was built for the support of the remaining components using SMAW process. The beam enhancer is fixed onto the exit cavity of the laser, whereas the safety shutter and beam bender are clamped using the C-clamps on top of the secondary frame. The laser process head is supported onto the secondary frame using a magnetic base/indicator holder and held vertically into place using a three-finger clamp.

To accommodate all safety regulations, the laser beam must be enclosed at all points up to the work surface. This is accomplished by connecting all components’ entry and exit cavities using a 1\text{inch} diameter aluminum tube. All tubes are anodized in black so that any misdirected or rogue light is absorbed by the coating and not destroy the tubing.

The entire laser system is installed on top of a Laboratory Grade 781-Series CleanTop® Optical Table. The surface of the breadboard table consists of M3 sized holes separated by 1\text{in}. The table is mounted on top of a TMC® 14-416-35 Pneumatic Optical Table Base [45]. Such a table setup is crucial to any laser setup as it provides a performance level for the least severe floor vibration environments [46]. A constant pressure of 75\text{psi} was set within all four legs of the table. Any pressure applied on top of the table also results in the uniform movement across the entirety of the table, negating any risk of the laser light deviating from its projected path.
Figure 7 - Complete laser system setup

Figure 8 - Schematic of the laser setup
3.3 Laser Alignment

As all components exist and function independently from the laser, it is crucial that they are aligned to the direction of the laser. Failure to do so results in the non-containment of beam, where the laser could interact with any material in its way and endangering the user. Due to the high power (since this is a class IV laser), this could destroy and produce flames and gases, thereby violating multiple safety regulations as enforced by OSHA (Occupational Safety and Health Administration). The section, 29 CFR 1910 in OSHA standards, directives, letters of interpretation, and national consensus standards relate to laser hazards, and has to be strictly adhered to. This laser emits light at a wavelength of 10.6 μm, which lies in the infrared region of the electromagnetic spectrum. As IR is invisible to the human eye, additional care was taken during the alignment phase by operating the laser at its minimum capacity.

At first, the laser is kept at a horizontal position and this is verified by the use of spirit levels at multiple points on the laser. The exact vertical and horizontal height of the exit cavity of the laser is measured by using the edges of the table as reference points. This position is critical, as this is the path of the laser and all other components will be placed along this path.

The safety shutter is placed horizontally on top of two anodized aluminum plates, where one of the plates is glued onto the secondary frame. The plates are sandwiched with four mechanical jack screws that are used to adjust the height and angle of the shutter. The entire component is fixed into place using a double-force, sliding arm clamp. Similar procedure was taken for the beam bender. However, this is fixed onto place using plumbers putty to minimize any sliding and rigged onto the frame using C-clamps. Both components were repeatedly checked for their horizontal position using the spirit levels.
Before activating the laser, all safety precautions were taken. Primarily eye safety and parametric conditions were set to minimum for testing purposes. To check for the quality of the spot size, a thermal alignment card provided by Haas® is used. All tests from the laser were carried out at 1000 microseconds of pulse period, 10 microseconds of pulse width and a total of 10 pulses. This ensures minimal power output from the laser and minimizes risks of damage to the components and the user.

Initially, a thermal card was placed at the exit of the beam enhancement system and laser light was shot, to check for a clean beam. After this, the laser was shot at a card placed at the entry point of the safety shutter. This ensured that the entry point of the safety shutter is aligned with the laser. After this point, the laser must also be aligned with the red diode emitting from the exit cavity of the safety shutter. This is done so that the red diode light can act as a substitute to the laser for ease of location of the spot size during operation.

Another thermal card is placed at the exit of the shutter, and the spot size is located. The mechanical jack screws are then used to adjust the position and angle of the safety shutter so that the spot size coincides with the red diode light at the exit. After doing so, a similar approach is conducted on the beam bender, where the coincidence of the laser spot size and red diode light are matched at the entry and exit of the beam bender. Any required adjustments were moderated by the use of the mechanical jack screws.

It must be noted that all the alignment test mentioned above are validated through the visual observations. This is eventually validated by the use of a power meter and the quality of the spot size at the focal point from the laser process head.
The laser process head is kept perpendicular to the horizontal orientation of the previous components. It is held vertically using a magnetic base/indicator holder. This is clamped onto the secondary frame using a C-clamp. The orientation of the process head is adjusted by moving the three-finger clamp to a desired position and locking it. This step is crucial to quality of the spot size, because all light that is focused from the lens within the process head has to exit the nozzle. Failure to do so results in an abnormal spot size on the thermal card. This step is a trial and error method, and adjustments have to be made until a perfectly circular spot size can be observed on the thermal card. Any protrusion on the spot size is due to the reflection of the laser onto the sides of the inner wall of the process head nozzle. Multiple tests were taken at different heights ranging from the focal point to around 3 inches from the focal point.

Figure 9 - Spot size of the laser at various lengths from the focal point of the lens (conducted in the following parameters: pulse period-1000μs, pulse width 130μs, and number of pulses-100)

3.4 Supporting Systems

To ensure that the laser system continues to function at optimum performance and that specific functions of the laser are fulfilled, additional supporting systems were designed, developed and installed.

Cooling System:

The cooling system for the laser ensures that the laser does not overheat during operation. For this, the laser is continuously supplied with a mixture of DOW Frost™ (96% solution of USP/EP grade polypropylene glycol and food grade di-potassium phosphate) and de-ionized water.
from a chiller system [47]. The system cooling and circulating this mixture is a 6000 Series DuraChill® Water-Cooled 1.5 HP Chiller [48]. The chiller unit is always turned on prior to operating the laser. A stagnant coolant temperature of 25 °C is continuously circulated through the laser unit.

![6000 Series DuraChill Water-Cooled 1.5HP Chiller](image)

**Figure 10 - 6000 Series DuraChill Water-Cooled 1.5HP Chiller[49]**

**AC-DC Converter System:**

This system is responsible for changing the AC current to a high voltage DC current for priming the laser. This is rated at 48 VDC.

**XYZ Stage System:**

The laser system itself is of a stationary nature, i.e. the beam itself will not change in any direction. The XYZ-BSMA-LY-140H from IntelLiDrives™, comprises of 3 NEMA 17 motors and low profile actuators with precision ball screws. This provides movement in the three degree of freedom and is ideal for laser applications due to its high accuracy, repeatability and positioning capabilities of up to 1 micron [50].
The operation of the system is controlled through a software provided by the manufacturer. All movements are controlled via commands given through code within the software. Speed, acceleration and repeatability can also be controlled (See Appendix C).

![Figure 11 - XYZ-BSMA-LY-140H-300X300X300][50]

**Inert Atmospheric Chamber:**

An inert atmospheric chamber was constructed to ensure that any gases present in the work environment does not interfere with the laser-material interaction. Due to the high heat involved in laser processes, oxidation of the material is of concern as it can change material properties. To minimize the oxidation, the work piece is always placed inside of a partially closed box which is purged of the oxygen by continuously supplied argon gas into it. The flow rate of the argon gas passing into the enclosure is 14.83 ft³/hr.

The chamber was constructed out of laser cut acrylic sheets bonded together using solvent based acrylic glue. Argon gas is supplied into the chamber and the oxygen content is continuously
monitored using an oxygen meter. As the oxygen content reaches to an acceptable level of less than 5%, lasing operations can begin.

Safety Systems:

The Coherent™ DIAMOND J-3 Series OEM laser is rated as a Class IV laser, which is the highest and most dangerous class of laser. Such a laser has the ability to burn the skin and cause permanent eye damage. Moreover, due to the high heat generated from the beam, ignition of combustible material can also cause fire risks. Precautions are taken to minimize these risks. The laser is equipped with a key switch and safety interlock mechanism for any unintentional starting of the laser. The laboratory is locked at all times and only authorized personnel are equipped to the use of the laser. All members present in the room are also to wear laser safety goggles at all times. There is a laser indication light outside the room and the door is lined with an industrial grade black curtain for the absorption of any rogue laser light escaping the room.
CHAPTER 4
EXPERIMENTAL PROCEDURE

The following chapter aims to explain the experimental procedure of the study, from the test sample preparation to the laser sintering process. A brief experimental procedure of the conventional approach of furnace sintering is also explained.

4.1 Test Sample Preparation

The material used for sintering is a pure copper micro-powder with a mesh size of 60/80 and an average particle diameter of 200 μm. The copper powder was commercially available and known as CuLox™ 6000 series [51]. The material thermal and physical properties are shown below:

Table 3 - Material Properties of Copper Particles

<table>
<thead>
<tr>
<th>Melting Point</th>
<th>Density</th>
<th>Thermal Conductivity</th>
<th>Specific Heat at 25°C (298K)</th>
<th>Reflectivity</th>
</tr>
</thead>
<tbody>
<tr>
<td>1083°C (1356K)</td>
<td>8960 kg/m³</td>
<td>401 W/m-K</td>
<td>385 J/kg-K</td>
<td>0.74</td>
</tr>
</tbody>
</table>

Figure 13 – CuLox™ 6000 Series 200μm pure copper powder
All sintering tests were conducted on the copper particles placed on top of a copper plate disc substrate. This was a circular copper disc with a thickness of 0.5 mm and a diameter of 1.5 in.

Figure 14 - Copper plate substrate

To ensure that a single layer of copper particles are laid onto the copper plate, various iterations of different methods were used. The discs are cleaned with methanol prior to powder placement to ensure a smoother surface so that the particles don’t stick due to moisture. During the first instances of sample preparation, the copper particles were spread across the plate and spread about the disc by tapping it. Visual inspection of the sample validated the quality of the monolayer. However, this method proved to be both tedious and inaccurate as there were some overlap, and there were areas where particles were not packed together.

The subsequent iterated method was to create a 3D printed mold of the copper plate. It was designed such that it incorporates the depth of the copper plate and the copper monolayer of 200μm. The copper plate was then placed onto the mold and powder was placed on top of it and then spread around.
However, this also proved to be ineffective due to tolerance limits provided by the 3D printer. Due to the small scale of the particles, the printed design was not consistent enough at the micro-meter level.

4.1.1 Powder Delivery System

Through these iterations, it was identified that the problems lie with inconsistent particle arrangement, and good repeatability of layering. To solve this, a customized functioning powder delivery system was designed and constructed. Aluminum blocks were cut using Computer Numerical Control (CNC) machining with a very high precision. The design is such that the center piece of the block moves up or down linearly with set increments. By controlling the increments it is possible to achieve a successful monolayer of copper particles (see Appendix B).

The movement of the center block is done using a NEMA 17 motor. This is controlled via an A4988 driver by micro-stepping with the help of an Arduino (Blononano™) and a 12V DC power supply. A GUI (graphical user interface) is used, where both the direction of the movement and length of the movement can be controlled as small as 0.025 mm or 25 microns using a lead screw of 2 mm pitch held together to the motor with a flexible coupler (see Appendix D).
The basic principle of the powder delivery system is to lower the platform by 200 μm, pouring the powder on top of it and then removing any excess powder using ‘card’ like shaped material. This ensures that there is a monolayer of copper on the copper plate substrate.

4.2 Laser Sintering Trials

All sintering was performed using the Coherent™ Diamond J-3 series pulsed CO₂ laser. It operates at a wavelength of 10.6 μm in the infrared region, has a peak power of 750 W and an average power of 250 W.

As all laser sintering setups vary in terms of initial conditions and as this is study is unique in its own nature, numerous sintering trials were performed and the optimum parameters were obtained using the trial and error method. The parameters involved to achieve successful sintering are the pulse period, pulse width, number of bursts, scan speed, spot size, percentage overlap, and repetition.

The pulse period, measured in micro-seconds, is the duration of time of one cycle of the repeating pulse. The pulse width, also measured in micro-seconds, is the total lasing time within
pulse period. The number of bursts corresponds to the total number of pulses supplied before the lasing is complete. These three parameters can be controlled by the laser control module. The scan speed is the speed at which the workbench moves. This is set by the software interface controlling the movement of the motors in the XYZ stage. The percentage overlap of the raster scan is also controlled via the XYZ stage. The spot size is the total area of laser exposure on the material. This can be increased or decreased by moving the laser-material interaction area closer or further away from the focal point of the lens in the laser process head.

At first the power levels at different parameter settings of the laser was determined. This is done by using a power meter to absorb the laser, and then indicating the amount of power. The pulse period was kept at a constant of 1000μs and the pulse width was kept as a variable. The power was measured and tabulated at different intervals of pulse width.

<table>
<thead>
<tr>
<th>Pulse Period (μs)</th>
<th>Pulse Width (μs)</th>
<th>Pw/Pp</th>
<th>Bursts</th>
<th>Power (1st attempt) (W)</th>
<th>Power (2nd attempt) (W)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1000</td>
<td>10</td>
<td>0.01</td>
<td>1000</td>
<td>2.1</td>
<td>2.1</td>
</tr>
<tr>
<td>1000</td>
<td>20</td>
<td>0.02</td>
<td>1000</td>
<td>6.1</td>
<td>6.6</td>
</tr>
<tr>
<td>1000</td>
<td>30</td>
<td>0.03</td>
<td>1000</td>
<td>10.7</td>
<td>11.2</td>
</tr>
<tr>
<td>1000</td>
<td>40</td>
<td>0.04</td>
<td>1000</td>
<td>14.8</td>
<td>16.2</td>
</tr>
<tr>
<td>1000</td>
<td>50</td>
<td>0.05</td>
<td>1000</td>
<td>19.7</td>
<td>21.2</td>
</tr>
<tr>
<td>1000</td>
<td>60</td>
<td>0.06</td>
<td>1000</td>
<td>23.4</td>
<td>24.8</td>
</tr>
<tr>
<td>1000</td>
<td>70</td>
<td>0.07</td>
<td>1000</td>
<td>27.1</td>
<td>30.9</td>
</tr>
<tr>
<td>1000</td>
<td>80</td>
<td>0.08</td>
<td>1000</td>
<td>30.3</td>
<td>33.5</td>
</tr>
<tr>
<td>1000</td>
<td>90</td>
<td>0.09</td>
<td>1000</td>
<td>33.3</td>
<td>39.4</td>
</tr>
<tr>
<td>1000</td>
<td>100</td>
<td>0.1</td>
<td>1000</td>
<td>38.7</td>
<td>43.4</td>
</tr>
<tr>
<td>1000</td>
<td>200</td>
<td>0.2</td>
<td>1000</td>
<td>78.8</td>
<td>83.3</td>
</tr>
<tr>
<td>1000</td>
<td>300</td>
<td>0.3</td>
<td>1000</td>
<td>110.6</td>
<td>117.3</td>
</tr>
<tr>
<td>1000</td>
<td>400</td>
<td>0.4</td>
<td>1000</td>
<td>140.6</td>
<td>145.1</td>
</tr>
<tr>
<td>1000</td>
<td>500</td>
<td>0.5</td>
<td>1000</td>
<td>170.5</td>
<td>169.6</td>
</tr>
</tbody>
</table>
This gives an understanding of the average amount of power output from the laser at different pulse width/pulse period combinations. All the tests were performed at a distance of 3 inches from the focal point of lens. This results in a spot size of about 4.8 mm.

4.3 Numerical Modeling

To get better idea of the amount of power required to sinter the particles, a numerical model is used. This model develops a relation between the laser power and initial temperature in the material surface. Assuming that the surface temperature of the material exposed to the laser beam is of uniform energy distribution, and that no losses occur in the system, the power can be calculated by considering the final temperature to be 10 °C below the melting temperature of copper.

This simple 1D analytical model is used:

\[ T = T_o + \frac{2I}{K} \sqrt{\frac{Dt}{\pi}} \]  

where,

\( T \) = final temperature of the material

\( T_o \) = initial temperature of the material

\( I \) = power intensity

\( K \) = material thermal conductivity

\( D \) = material thermal diffusivity

\( t \) = laser exposure time

The final temperature of the copper is assumed to be 1075 °C, initial temperature to be the room temperature of 25 °C, \( K \) is 401 W/mK, \( D \) is \( 1.11 \times 10^{-4} \) m\(^2\)/s. The total time that each particle
is exposed to the laser beam can be determined as a factor of the scan speed, pulse period, pulse width and number of pulses. Here, it is assumed that all energy is supplied to each particle without any losses and that no conduction occurs between consecutive particles making it a closed system.

The various parameters used are given below:

Pulse period = 1000 = 0.001 s
Pulse width = 50 = 0.00005 s

Therefore,

Total lasing time per pulse:

\[ t = 0.001 \times 0.00005 = 50 \times 10^{-6} \, s \]

The total time of lasing operation over the length of a single particle is calculated by dividing the spot size by the scan speed:

Total lasing operation time:

\[ t = 4.73 \times 0.5 = 11 \, s \]

The total time of lasing operation is 11 s, and therefore by incorporating the scan speed of 0.5 mm/s and total lasing time of 11 s, the total irradiation time for each particle is given as:

Total about of irradiation time:

\[ t = \frac{11}{0.001} \times (50 \times 10^{-6}) = 0.55 \, s \]

The power intensity, I, can be calculated using the parameters as follows:

\[ I = \frac{2P(1-R)}{\pi r_o^2} \]  

(2)
where,

\[ P = \text{power output of laser} \]
\[ R = \text{reflectivity of material} \]
\[ r = \text{radius of spot size} \]

Reflectivity of copper at operating wavelength of 10.6 μm is 0.74 and the radius of the spot size is 2.15 mm. By solving equations 1 and 2, the power required to sinter was calculated to be about 42 W. However, it must be noted that this value is calculated assuming a closed system, where in, there are no losses from the laser energy, and that no heat is being conducted from other particles. These are big assumptions to make, but this gives an approximation of the minimum amount of power required to attain sintering.

Sintering was done in an inert atmospheric environment by placing the samples within an argon gas enclosure. Argon gas was continuously supplied to the enclosure and the testing was only performed when oxygen levels reached to levels below 5%.

All sintered samples were immediately observed under an Amscope™ ME300 microscope to validate the level of sintering. The structural integrity of the samples were checked by lightly tapping them and checking to see how easily it would disintegrate.

4.4 Sintering Approach

For successful sintering, multiple methods were employed. The following describes the three main approaches of sintering.

4.4.1 Single point approach

This test involved no movement of the copper substrate and direct lasing onto a single point. The parameters used for this was at 1000μs of pulse period and a pulse width of about 150μs.
The bursts were not kept limited and instead kept at a continuous mode. This resulted in a small circular area of partially sintered copper particles. It was observed the center portion of the area was affected more by the heat of the laser than the outer areas. This is due to the Gaussian nature of the laser beam. The microscopic image, as shown in figure 16, revealed partially melted and partially sintered particles.

![Microscopic image of partially sintered copper particles.](image)

**Figure 17 - 'Balling' effect of particles conjoining together**

Even though partial sintering was achieved these tests were successful in terms of providing a ‘ballpark’ for the parameters to be used in the successive tests.

### 4.4.2 Single pass approach

This approach involved the linear movement of the sample in one direction to observe the effects of continuous lasing over a period of time over different points of the sample. This approach did not result in any successful sintering as the heat distributed throughout the sample varied greatly. It was found that during the initial periods of the test, no visible effects were observed. However, during the end portion of the test, there was burning of the copper particles due to the excess heat built up through heat conduction and convection, internal inverse
bremsstrahlung effects, from the initial heat supplied on the copper particles and copper plate substrate. Inverse bremsstrahlung effects are collisional absorption of energy that occurs in inertial confinement fusion systems when hydrodynamic expansion of the plasma causes an electron passing through the field of an ion to absorb radiation, raising the energy and temperature of the electron particle. This increases the heat of the system.

Observation under the microscope revealed that there was no change in half of the particles and burnt particles at the latter half. This approach consequently showed that the varying distribution of heat across the sample was a factor that needed to be taken into account.

4.4.3 Multiple pass approach

Using the observations from the previous methods, it was decided that a multiple pass approach may lead to a better distribution of the heat across the sample. This approach involved multiple passes across the sample with varying degrees of overlap. This ensured that all particles had almost equal amount of laser exposure, as well as heat exposure from conduction. With a pulse period of 1000μs and pulse period of 140μs, successful sintering was achieved. Structural integrity of the sample was also of a better quality than those achieved with previous approaches.

4.5 Design of Experiments

By employing these approaches a rigorous design of experiments (DOE) was followed to find the significant parameters which had substantial effect on the outcome of the experiment.

The following factors have an effect on the sintering of the copper powder:
Based on the literature and studies done previously, the following factors are considered to be of importance. The factors along with their classification, level and range is described below:

<table>
<thead>
<tr>
<th>Factors Name</th>
<th>Classification</th>
<th>Level</th>
<th>Upper Range</th>
<th>Lower Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pulse period</td>
<td>Numerical</td>
<td>-1 to 1</td>
<td>500μs</td>
<td>1500μs</td>
</tr>
<tr>
<td>Pulse width</td>
<td>Numerical</td>
<td>-1 to 1</td>
<td>10μs</td>
<td>200μs</td>
</tr>
<tr>
<td>Scan Speed</td>
<td>Numerical</td>
<td>-1 to 1</td>
<td>0 mm/s</td>
<td>2 mm/s</td>
</tr>
<tr>
<td>Number of hatch passes</td>
<td>Numerical</td>
<td>-1 to 1</td>
<td>0</td>
<td>5</td>
</tr>
<tr>
<td>Spot Size</td>
<td>Numerical</td>
<td>-1 to 1</td>
<td>1.5 mm</td>
<td>5.3 mm</td>
</tr>
</tbody>
</table>

As five factors are chosen with response (quality of sintering), a 2⁵ or 32 runs of the experiment can have enough degrees of freedom to analyze the system. Only one material of copper with set properties and size, and only the CO₂ laser was used for all tests. The room temperature, humidity was considered to be constant. By running all the combinations through the methods mentioned previously, the following combinations of factors resulted in the best quality of sintering. The output value of sintering is not quantitative and therefore is qualitative, i.e., either...
sintering was achieved or not. If there was observed sintering present, then the quality of it was checked by handling the sample and checking the structural integrity.

The following combinations produced a satisfactory level of sintering:

Table 6 - Combinations of factors producing successful sintering

<table>
<thead>
<tr>
<th>Pulse period (μs)</th>
<th>Pulse width (μs)</th>
<th>Scan speed (mm/s)</th>
<th>Number of hatch passes</th>
<th>Spot Size (mm)</th>
<th>Sintering</th>
</tr>
</thead>
<tbody>
<tr>
<td>1000</td>
<td>160</td>
<td>0.5</td>
<td>1</td>
<td>4.73</td>
<td>Yes</td>
</tr>
<tr>
<td>1000</td>
<td>140</td>
<td>0.5</td>
<td>3</td>
<td>4.73</td>
<td>Yes</td>
</tr>
</tbody>
</table>

The final parameters used for achieving sintering are as follows:

Table 7 - Final fabrication parameters used for sintering

<table>
<thead>
<tr>
<th>Pulse Period (μs)</th>
<th>Pulse Width (μs)</th>
<th>Bursts</th>
<th>Power (W)</th>
<th>Scan Speed (mm/s)</th>
<th>Spot Size (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1000</td>
<td>140</td>
<td>Continuous</td>
<td>49</td>
<td>0.5</td>
<td>4.73</td>
</tr>
</tbody>
</table>

4.3 Conventional Furnace Sintering Procedure

The traditional sintering involves the heating of the sample in a furnace. A single layer of copper particle is laid on a 38 mm diameter copper disk. The particles are held in place using a machined stainless steel mold. To ensure an inert condition between the stainless steel and copper particles, a sonicator was used to uniformly coat the particles at a temperature of 38 °C for 90 mins. A ceramic crucible is placed under the mold, before placing it in the furnace. Argon gas is circulated through the furnace to reduce oxidation. The temperature of the furnace is gradually increased to 950 °C and maintained as such for 3 hours, before cooling it back down to the room
temperature. Experimental tests conducted on the laser sintered samples are compared to that of the furnace sintered samples to validate the level and quality of the laser sintering.
CHAPTER 5
RESULTS AND DISCUSSION

Laser sintering was achieved on 200 μm sized copper particles, for creating a monolayer structure. This process uses the light from the pulsed-laser to expose the copper particles with a high amount of heat flux in a short span of time (microseconds). Specific conditions have to be met for successful sintering to take place. The applied heat cannot increase the material’s temperature above 1085 °C, as this melts and conjoins the particles together completely to-the-core. This decreases the porosity and uniformity of the desired structure. Applying a considerably less temperature than 1085 °C, results in the failure of bonding of the particles, and no sintering occurs. Therefore, using a design of experiment and trial and error methods, optimum parameters were discovered to create successful sintering.

5.1 Physical Properties of the Particle

To validate the quality of sintering the observational results for the laser sintering samples are compared to that of a traditionally (furnace) sintered sample. The quality of sintering is primarily checked by visual observation under a microscope. The bonding of the particles are checked by lightly moving the sample around. If these particles do not show any melted features and minimal protrusion are observed, it can be said that successful sintering has taken place.
In figure 17, minimal protrusion can be seen, i.e. little ‘bumps’ on the surface of the particles. This can occur due to the localized heating from the laser. To minimize this, providing a better heat distribution across sample can be done, by better focusing of the sample and finer overlapping. Nevertheless, the laser sintered sample contains similar physical characteristics as its furnace sintered counterpart, as shown below.
To get an improved understanding, the particles were placed under a MicroXam-100 optical profilometer by KLA-Tencor to develop a three-dimensional profile of the structure. Multiple images were taken at varying focal planes to generate a 3D image profile of the particles. The figures below shows the similarities of the structure with that of the conventionally sintered samples.

![Figure 20 - 3D profile of furnace sintered (left) and laser sintered (right) copper micro-particles](image)

### 5.2 Roughness Test

A roughness test was conducted using the profilometer to visualize the spatial arrangement of the particles and thus the porosity of the structure. The roughness chart below in figures 21 and 22, shows a graphical representation of the cross-sectional view of the sample. The region between the peaks indicate the gap between the particles, which contributes to the porosity and the depressions represent the void particles themselves. Again, comparing this to that of the conventionally sintered samples for validation, it can be observed that the gaps and depressions show similar behavior. It must be noted that the depression of the laser sintered chart are not smooth indicating the 'bumps’ on top of the particles arising from localized heating. This is much smoother in the furnace sintered results due to much more even heating of the particles.
Additionally, the surface roughness values are also similar, further proving that the same quality of primary sintering can be achieved as done by conventional sintering. This also indicates that the laser sintering process has an immense advantage over furnace sintering due the high reduction in manufacturing time.

![Figure 21 - Surface roughness of laser sintered particles](image)

![Figure 22 - Surface roughness of furnace sintered particles](image)

5.3 Wettability Test

A wettability test is carried out to determine the amount of porosity of the structure. This is done by first measuring the dry weight of the sample and then measuring it after dipping in
water. The overall difference in the weight signifies the volume of water that is trapped in the porous sample. Using this, the porosity index is calculated to be the ratio of the volume of water to that of the sample.

Table 8 - Mass of water trapped in laser sintered sample

<table>
<thead>
<tr>
<th>Mass before water entrapment (g)</th>
<th>Mass after water entrapment (g)</th>
<th>Mass of water (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.0868</td>
<td>2.1318</td>
<td>0.04505</td>
</tr>
<tr>
<td>2.0425</td>
<td>2.09108</td>
<td>0.04858</td>
</tr>
<tr>
<td>2.0636</td>
<td>2.11362</td>
<td>0.05002</td>
</tr>
<tr>
<td>2.0895</td>
<td>2.13302</td>
<td>0.04352</td>
</tr>
<tr>
<td>2.0668</td>
<td>2.11018</td>
<td>0.04338</td>
</tr>
</tbody>
</table>

Calculating the volume of water:

\[
\text{volume of water} = \frac{\text{mass of water}}{\text{density of water}}
\]

\[
= \frac{4.505 \times 10^{-5}}{997} = 4.518 \times 10^{-8} m^3
\]

Calculating the volume of the sample:

\[
\text{volume of sample} = \frac{\text{mass of sample}}{\text{density of copper}}
\]

\[
= \frac{0.0020868}{8960} = 2.33 \times 10^{-7} m^3
\]

Therefore, the porosity index can be calculated from the ratio of both volumes:
\[
\text{porosity index} = \frac{6.302 \times 10^{-8}}{2.33 \times 10^{-7}} = 0.194
\]

Similarly, the same was carried out for the furnace sintered samples:

<table>
<thead>
<tr>
<th>Mass before water entrapment (g)</th>
<th>Mass after water entrapment (g)</th>
<th>Mass of water (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5.05909</td>
<td>5.12037</td>
<td>0.06128</td>
</tr>
<tr>
<td>5.05846</td>
<td>5.11864</td>
<td>0.06018</td>
</tr>
<tr>
<td>5.05808</td>
<td>5.12092</td>
<td>0.06284</td>
</tr>
<tr>
<td>5.06370</td>
<td>5.12640</td>
<td>0.06270</td>
</tr>
<tr>
<td>5.06600</td>
<td>5.12565</td>
<td>0.05965</td>
</tr>
</tbody>
</table>

Calculating the volume of water:

\[
\text{volume of water} = \frac{\text{mass of water}}{\text{density of water}}
\]

\[
= \frac{6.284 \times 10^{-5}}{997} = 6.302 \times 10^{-8} \text{m}^3
\]

Calculating the volume of the sample:

\[
\text{volume of sample} = \frac{\pi d^2}{4} \times 200 \times 10^{-6}
\]

\[
= \frac{\pi \times (38 \times 10^{-3})^2}{4} \times 200 \times 10^{-6} = 2.268 \times 10^{-7} \text{m}^3
\]

Therefore, the porosity index can be calculated from the ratio of both volumes:
\[
\text{porosity index} = \frac{6.302 \times 10^{-8}}{2.268 \times 10^{-7}} = 0.277
\]

Therefore, by conducting the wettability tests, it is measured that the porosity index of the laser sintered samples is about 0.194, whereas it is 0.277 for the conventionally sintered samples. This discrepancy is within the allowed limits, but this study also shows that it is possible to achieve similar amounts of porosity through the laser sintering method. Moreover, by altering parameters such as the raster scan speed, overlap frequency and laser parameters, desired amounts of porosity can be also be achieved.

5.4 Thermal Conductivity

The thermal conductivity of the sintered particles was measured on the principle of a simple thermal analysis. The method incorporated the application of a heat source on one side of the sample. Temperature was kept at a constant 200°C. The copper sample was placed between two sheets of glass for insulation. And constant measurements were taken at both ends of the sample to observe how much time was taken for the entire sample to reach equilibrium. An infrared thermometer gun was used to measure the temperature. Tests showed that it took about 5 seconds for the cold end of the sample to reach about 200°C.
This was compared to a thermal analysis performed in ANSYS™. A geometry of the sample was modelled by designing one particle of 200μm and then replicating it on a 2D plane in a rectangular orientation. The length of the designed sample was 7.2 mm and width of 1.82 mm. The boundary conditions applied consisted of an insulation on top and bottom. The initial conditions involved the application of 200 °C on one side and other was set at room temperature of 22 °C. After applying the thermal and physical properties of copper, the analysis showed a total time of 4.37 seconds for the system to reach equilibrium, i.e. the other end also reaching 200°C.
Figure 24 - Experimental setup simulation with glass plates

Figure 25 – Temperature distribution through the monolayer copper sample
A transient thermal analysis was conducted to simulate the experiment. A triangular mesh type of 0.02 size was used in a solid element type configuration. The boundary condition set was that no transfer of heat other than conduction and convection is to occur within the simulation. Geometrically, all particles are in contact with the glass on the top and bottom, and are open at the end portions. Initial conditions were setting one end of particles at 200°C and the other at 25°C. All material properties used for this simulation are same as those of copper found in Table 3.
CHAPTER 6

CONCLUSION

This study involved the laser sintering of copper micro-particles of 200 μm diameter, using a CO₂ pulsed microsecond laser. Prior to sintering, the laser system was installed along with its supporting systems. During this process, the many functionalities and parameters of the laser were understood, which aided in the laser sintering tests. There was successful demonstration of sintering of a monolayer copper micro-particles at pulse period of 1000 μs, pulse width of 150 μs, spot size of 4.73 mm and scan speed of 0.5 mm/s. The quality of sintering is validated by comparison with conventionally furnace sintered samples. The surface roughness of both sintered samples show similar gaps and spatial arrangement of particles. The 3D profile images of both samples also show no significant differences in the physical appearance of the sintered particles.

The wettability test conducted on both samples show that the laser sintered tests result in a porosity index of 0.194 and 0.277 for conventionally sintered tests. Similar amounts of porosity, within the acceptable limits, denote that laser sintering is a viable alternative to the conventional sintering for additive manufacturing of porous structures. Moreover, with changes in laser parameters, one can also generate structures with different amounts of porosity as required, while maintaining a similar quality of sintering.

The thermal conductivity of the sintered particle was checked and validated with ANSYS simulation. Experimentally heat was applied at one end of the sample and temperature was measured at the other end. The total amount of time taken for the other end to reach the same temperature measured 5 seconds. Simulation was also validated over ANSYS by measuring the time it takes for the sample to reach an equilibrium temperature of 200 °C on the cold side. 3.84
seconds was taken for it to reach equilibrium. In conclusion, taking into account the loss of heat through radiation in the experiments, results were in close tolerance with the simulated one.

These experimental validations prove that a laser sintering can achieve similar results to that of furnace sintering. A substantial reduction in fabrication time by an order of magnitude of 6-8 hours, can prove advantageous when providing fast prototyping. Such a feature can be crucial for the prototyping phase of projects involving complex structures in the microscale.
CHAPTER 7
FUTURE WORK

This study aimed to showcase the validity of laser sintering of a monolayer of copper micro-particles, and thus paving a foundation for sintering of multilayered three-dimensional structures. However, further development on this study can be done to help in achieving this goal. The primary focus should be in the heating of the copper substrate, which can act as a catalytic for the bonding of the particles to the substrate. This allows for a stronger sintered bond. Heating can be applied externally using infrared lamps/heaters, or the substrate could be heated using resistive heating methods.

Better sample preparation methods should be looked into. For example, a more mechanized powder delivery system, where the entire device is programmed and controlled through a coded software rather than the user manually leveling the particles would be very beneficial. The particles themselves can be coated using a transmissive anti-reflective coating to better absorb the light from the laser. This can prove to be very advantageous as this will greatly compensate for the reflective index of copper.

Different combinations of scanning and overlapping can be performed, by changing the raster scan speed and orientation. This will also have an effect on the type of sintering and could also alter the initial conditions of the laser parameters.

More testing involving different metal (for example like, aluminum, nickel and their alloys) as well as non-metal (diamond, graphite) powders should also be considered. The high reflectivity and thermal conductivity of copper acts as a natural deterrent when applying heat via laser light. Other types of material with lesser values of these properties could allow for easier and more
refined sintering results. And consequently, the addition of successive layers on top of the monolayer can be subjected to various tests allowed for three-dimensional structures, such as a water contact angle measurement test for analyzing the hydrophobic/hydrophilic properties of the surface. Better porosity and permeability tests can also be validated.
REFERENCES
REFERENCES


Figure 27 – Coherent™ J-3 CO2 Laser technical drawing
Figure 28 - Laser system layout designed by HAAS™
Figure 29 - First iteration of setup installation

- Connecting to LASER
- NEED 4x M4 screws
- NEED another ADAPTER
- CUT Al plate 2.75 in
- For supporting BEAM 1
- CUT coupler 1.50 in
- Connecting BEAM 2

- CUT tube 40.00 in
- Connecting COUPLER 3
- CUT coupler 1.00 in
- Connecting COUPLER 4

- CUT square Al plate 4.00 in with 2 in x 2 in square pocket
- For supporting COUPLER 5
- CUT coupler 1.00 in
- Connecting BEAM BENDER 1

- CUT tube (TBD)
- Connecting COUPLER 5 to COUPLER 6
- CUT coupler 1.00 in
- Connecting PROCESS

- CUT tube 4.00 in
- Connecting COUPLER 1
- CUT coupler 1.00 in
- Connecting tube 1 to SAFETY

- CUT coupler 1.00 in
- Connecting SAFETY SHUTTER to TUBE 2
- CUT Al plate 5.00 in
- For supporting SAFETY 1

- CUT coupler 1.00 in
- Connecting SAFETY SHUTTER to TUBE 2
- CUT coupler 1.00 in
- Connecting SAFETY SHUTTER to TUBE 2

- CUT coupler 1.00 in
- Connecting SAFETY SHUTTER to TUBE 2
- CUT coupler 1.00 in
- Connecting SAFETY SHUTTER to TUBE 2

- CUT coupler 1.00 in
- Connecting SAFETY SHUTTER to TUBE 2
- CUT coupler 1.00 in
- Connecting SAFETY SHUTTER to TUBE 2

- CUT coupler 1.00 in
- Connecting SAFETY SHUTTER to TUBE 2
- CUT coupler 1.00 in
- Connecting SAFETY SHUTTER to TUBE 2
II. APPENDIX B

Figure 30 - Base plate drawing
Figure 31 - Outer container drawing
Figure 32 - Inner platform container drawing

Figure 33 - Leg support drawing
III. APPENDIX C

The following code allows for an automatic linear movement along the x-axis. When switched on, the x stage will move at speed of 0.5mm/s for a total of 5 cm before moving back in the opposite direction. This movement will oscillate back and forth until the switch is turned off.

XYZ Code:

"HSPD=30000
LSPD=100
ACC=500
EO=1
V1=0
WHILE 1 = 1
 IF DI1 = 1
 IF V1 != 1
 STOPX
 WAITX
 JOGX+
 V1=1
 ENDF
 ELSEIF DI2 = 1
 IF V1 != 2
 STOPX
 WAITX
 JOGX-
 V1=2
 ENDF
 ELSE
 STOPX
 WAITX
 V1=0
 ENDF
 ENDFWHILE
END

;******************************************************************************
SUB 31
ECLEARX
ENDSUB
"
IV. APPENDIX D

The following code allows for the linear movement of the lead screw thereby moving the platform onto which sintering is to take place. The code generates a GUI through which the user first sets the movement direction, where an input of ‘U’ denotes upwards, and ‘D’ denotes downwards. Then the user simply inputs the amount of movement in mm into the GUI. The output will result in the turning of the motor and movement of the leadscrew. It must be noted that the resolution of the micro stepping is 0.025mm and therefore only movements with intervals of 0.025mm can be made.

PDS Code:

"//09012018

// defines pins numbers
const int stepPin = 3;
const int dirPin = 4;

// Define step mode control pin (to control MS1, MS2, MS3 of A4988 stepper driver (ref: datasheet)
int step_factor=2;
const int MS1=5; // e.g for step_factor=2 (half step) MS1=HIGH, MS2=LOW, MS3=LOW
const int MS2=6;
const int MS3=7;
const int enable=8; // control the enable pin of the stepper driver A4988

//Stepper Parameters
float theta = 1.8; // 1.8 deg. per step from motor datasheet (NEMA 17)

// Actuation parameter

float stroke_length = 0; // user defined input (unit should follow the unit of screw pitch (mm))

int Tpulse = 0; // required number of pulses; N.B: Truncated or rounded integer value; cal. in the code section

// Serial Input Variables

char rx_byte = 0;
String rx_str = "";

void setup() {

    // Defining the Outputs from arduino nano

    pinMode(stepPin, OUTPUT); // provides required number of pulses for the desired actuation (mm)

    pinMode(dirPin, OUTPUT); // controls the rotation direction

    // Step control mode defining output

    pinMode(MS1, OUTPUT);

    pinMode(MS2, OUTPUT);

    pinMode(MS3, OUTPUT);

    pinMode(enable, OUTPUT);

    // To stop driver working before setting the control parameters

    digitalWrite(enable, HIGH); // enable pin is active low so making it high will disable the driver
//stepPin and dirPin initialisation
digitalWrite(stepPin, LOW);
digitalWrite(dirPin, LOW);

//Driver and Step Mode initialisation (coded for half stepping mode)
digitalWrite(MS1, HIGH);
digitalWrite(MS2, LOW);
digitalWrite(MS3, LOW);
digitalWrite(enable, LOW); //activating the driver

//Serial communication initialisation to input stroke length
Serial.begin(9600); //9600 baud rate

void loop() {

    if(Serial.available()>0) {
        rx_byte = Serial.read(); // get the character

        if (rx_byte != '\n') {
            // a character of the string was received
            rx_str += rx_byte;
            //Serial.println(rx_str);
        }
    }

}
else
{
    stroke_length=rx_str.toFloat();
    Serial.println(rx_str);
    rx_str="";
}

if(rx_byte=='D' || rx_byte=='d')
{
    digitalWrite(dirPin,LOW);
    Serial.println("Direction Update: CCW");
}

if (rx_byte=='U' || rx_byte=='u')
{
    digitalWrite(dirPin,HIGH);
    Serial.println("Direction Update: CW");
}

if(stroke_length>0)
{
    //digitalWrite(dirPin,HIGH);

    Tpulse=(360*step_factor*stroke_length)/(8*theta); //pitch of the lead screw is 8 mm
    Serial.print(stroke_length);
    Serial.print(", ");
    Serial.println(Tpulse);
// Makes total pulses for required rotation High - CW, Low - CCW
for(int x = 0; x < Tpulse; x++) {
    digitalWrite(stepPin, LOW);
    delayMicroseconds(500);
    digitalWrite(stepPin, HIGH);
    delayMicroseconds(500);
}
stroke_length=0;
Tpulse=0;
// rx_str="";
delay(2000);
}"
}