A. Introduction:

Since mainstream additive manufacturing solutions do not currently fill the need for electrically conductive components, the objective of the research project which I have actively participated in is to understand the relationship between microstructure, processing, and final properties of copper-based alloys fabricated via selective laser melting. The primary role of my involvement in this research project was to investigate the fundamental relationships integral to the additive powder metallurgy process called selective laser melting, with particular focus on performing particle shape, size, and size distribution analyses. The following report outlines the knowledge I have gained including the role powder metallurgy plays in this additive manufacturing process and the impact of particular powder properties on the properties of manufactured parts intended for electrical applications.

The long-term benefit of this research is to provide industries involved in the additive manufacturing of copper-based alloys, produced from powders, for commercial electronic components with insight into the microstructural development which occur during fabrication via selective laser melting.

A.1 Additive Manufacturing Overview:

Additive manufacturing is a relatively recent manufacturing technique where a component is built up layer-by-layer, as dictated by a 3D computer aided drawing (CAD). The schematics Figure 1 and Figure 2, shown below, depict the flow of the process, and a rectangular model sliced into thin layers, respectively.

![Figure 1 - Diagram showing the formation of a 3D product from a computer model](image1)

![Figure 2 - Schematic of a rectangular model sliced into thin layers](image2)

In its inception, additive manufacturing was first considered a rapid prototyping process, used for creating models and prototypes; however, rapid prototyping processes have gained increasing attention and has progressively been implemented into the manufacturing of tools, forms, and components, typically for small scale productions [1]. Rapid prototyping processes have many names including layer manufacturing, additive fabrication, rapid manufacturing, freeform manufacturing, toolless manufacturing, and direct digital manufacturing [2]. These names specify some of the various ways the same general process is perceived: layer manufacturing suggests a product is made layer-by-layer; additive fabrication conveys the manufacturing of a product is fabricated by adding material rather than subtracting material as in the case of conventional machining; rapid manufacturing implies that products are manufactured faster in comparison with conventional processes, especially for products with complex geometries; freeform manufacturing indicates that the geometry of the product is not confined within the boundaries of tools and/or molds; toolless manufacturing implies that the intermediate state of making tools for the making a product has been eliminated, comparable to injection molding where the product type depends on the mold type/material and making different types of molds for different types of products is necessary; and direct digital manufacturing suggests that
the digital model is directly converted into a physical product without resorting to intermediate steps including making or selecting various types of tools [3, 4]. In order to minimize confusion, the American Society for Testing and Materials committee F42 recommends using additive manufacturing as the preferred general name [2].

A.1.1 Selective Laser Sintering/Melting Theory:
Selective laser sintering/melting is a type of additive manufacturing process where the term 'laser' implies that a laser is used for processing, 'sintering' implies that a powder is involved in the process, and the term 'selective' implies that not all of the powder is processed by the laser simultaneously, only when and where it is required, unlike during conventional sintering processes; 'melting' refers to the particular situation in which powders are completely melted [2]. It should be noted however, that this name actually encompasses two slightly different processes. The 'selective laser sintering' process joins powders by partial melting, while 'selective laser melting' joins powders by full melting, but the two have been combined for convenience [5, 6]. Similar to the variety of names for additive manufacturing discussed above, the same process has also been named laser cusing, direct metal laser sintering, laser generating, direct laser forming, and direct laser fabrication; but the name 'selective laser sintering/melting' is more widely used and recognized [2].

Selective laser sintering/melting is a layer manufacturing process with the aim to make layers of powders of predefined geometry and fusing them using a laser beam [2]. The process follows the proceeding general sequence: (a) a substrate is lowered to a depth equal to the powder layer thickness, (b) a powder layer is spread onto the substrate, and (c) the deposited powder is scanned by a laser beam to fuse the powder in the predefined, selected, area. This sequence is repeated until the product is fabricated [2]. Figure 3 shows this schematically, and a more detailed explanation of this process follows.

![Figure 3 - Schematic depiction of the selective laser sintering/melting process](image)

Initially, the powder is held in a container, or hopper, and adjacent to it is a roller which carries the powder toward the build platform, or substrate, which is placed on a piston such that the vertical position can be controlled during the process. Any excess powder is carried away by the roller into a trash receptacle on the opposite side of the substrate. A scanning mirror enables the laser beam to be scanned across the deposited powder layer, termed the powder bed, fusing it into a solid. This description depicts the basic necessities of the selective laser sintering/melting process: (1) the formation of a powder bed, (2) the consolidation of the powder via a laser beam, and (3) a mechanism that enables (1) and (2) to be repeated. The entire process, including the powder feeding, deposition system, scanning, temperature, atmosphere, and build, are computer controlled [2].
A.1.2 Key Parameters:

Parameters requiring consideration in selective laser melting include the powder (shape, size, and size distribution), the laser (power, type, spot size, and mode), and the overall process (scan spacing and speed, layer thickness, scanning and building strategy, and atmosphere) [2]. However, in the present work, the powder properties (shape, size, and size distribution) were of main concern, and independent works discussing and investigating the other key parameters should be sought for more information.

A.1.2.1 Powder Shape, Size, and Size Distribution:

The powder is the basic building-block of this process; its shape, size, chemistry, and properties influence every stage of the process. Powder bed density is a measure of how loose powder will fit with each other; high powder bed density is preferred in selective laser sintering/melting, and spherically-shaped powders give rise to maximum powder bed density and flowability [2]. The size of the powder is an important parameter which warrants consideration because as the particle size decreases, the surface area as well as the gaps between particles increases, resulting in a less dense powder bed and potentially porous final product [2]. In the case of two and three different size spherical powders, for example, when they are tightly fitted adjacent to each other, the unfilled space between the particles is triangular in shape. In order to fill these gaps, an additional powder of smaller shape is needed, thus increasing the powder bed density. Figure 4 below depicts how bimodal and trimodal size distributions of spherical powders fit together. However, utilizing powder mixtures of various sizes does not always produce a fully dense material, for which additional processing steps may be necessary [2].

![Figure 4 - Packing of bimodal (left) and trimodal (right) size distributions of spherical powders](image)

The schematic representations shown in Figure 4 assume that all of the particles are perfectly spherical and the size distributions are ideal. In reality however, this is never the case; filling the gaps between particles also depends on the probability that each particle settles in the gap according to its size. Increasing this probability would require additional processing steps, such as compaction or consolidation, but these intermediate actions increase the number of steps required in the selective laser sintering/melting process, which consequently increases the cost and time of production, and can prove to be detrimental to any small features within the build layer [2].

A.2 Conventional Material & Alloy Properties:

Copper (Cu, Z=29) has many common features with other precious metals, including its atomic and electronic structure [7]. As copper is a face-centered cubic, monovalent atom, its electronic structure is responsible for many significant physical properties including its characteristic color and high electrical conductivity, chemical stability, and compressibility [7]. Electrical conductivity strongly depends on the purity of the material as well as the processing techniques utilized during refining and manufacturing [8]. Both residual porosity and impurities
lower conductivity, however the high temperature process associated with sintering removes these particular impurities [8].

Additions of tin (Sn, Z=50) in copper-based alloys can lead to improvements in strength, as well as corrosion and wear resistance: low-alloy Cu-Sn alloys, (< 5wt.% Sn) are intended for plastic forming, whereas concentration of tin between 5-10wt.%, mainly strengthens the alloy despite making it harder to deform in cold processes; tin concentrations greater than 8wt.% make performing cold processing impossible due to the generation of fragile phases; and copper alloys containing more than 10wt.% tin are used as-cast with particular applications including wear-resistant or optically reflective elements [7].

Metallic products for electrical applications are characterized by their resistivity, or inverse conductivity. The average reported electrical resistivities of pure copper and tin are $1.72 \times 10^{-8} \ \Omega \cdot m$ and $11.5 \times 10^{-8} \ \Omega \cdot m$, respectively, while their average reported thermal conductivities are 400 W/m·K and 66.6 W/m·K, respectively [9, 10].

A.3 Conventional Manufacturing Processes:
Conventionally manufactured components for electronic applications are fabricated in various ways depending on the intended purpose. Providers of these components tend to keep their manufacturing methods proprietary, however general information on fabrication of such parts can be found. For example: connectors are manufactured via rolling into plates, sheets and/or strips and subsequently machined or stamped [11]; seamless waveguide tubes and bus pipes are hot and/or cold worked (extruded) and soft annealed [12, 13]; and rods and wires are hot and/or cold worked (drawn) and annealed [14].

B. Results & Discussions:
Industrially provided pre-alloyed Cu-4.3Sn powder fabricated via air atomization was observed and analyzed for particle shape, size, and size distribution analyses. Static image analysis was performed on more than 1,500 finely dispersed particles on carbon tape using a Hitachi SU3500 scanning electron microscope, and particle measurements were performed using Image-J computer software. Metallography was performed using standard preparation techniques and samples (mounted powder and fabricated cube) were etched in Klemm's I for light optical microscopy under polarized light.

B.1 Particle Shape, Size & Size Distribution:
Atomization processes rely on a melt and the disintegration of that melt, as a result of rapid gas expansion near the melt stream, into droplets which solidify into particles [8]. Figure 5 shows the progression of the disintegration of the melt stream into droplets of various shapes and sizes.

Figure 5 - Schematic showing the formation of a metal powder by gas atomization
Gas atomization processes have a large number of operating variables including: gas type, residual atmosphere, melt temperature and viscosity, alloy type, melt feed rate, gas pressure, gas feed rate and velocity, nozzle temperature, and gas temperature; and these parameters can be tailored in order to control the powder characteristics depending on the intended use. The main advantages of gas atomization is in the product homogeneity and the good packing properties resulting from the produced spherical powder [8].

The air atomized Cu-4.3Sn powder observed in the present work were mostly spherical in shape as seen in Figure 6 below, however ellipsoid and ligament particle shapes were also present. Few particles exhibited having agglomerates or satellites, indicative of turbulence or particle reentry within the gas expansion zone, indicating good atomization process control.

Figure 6 - Scanning electron micrograph of air atomized Cu-4.3Sn powder particles showing mostly spherical and some ellipsoid and ligament particle shapes

Static image analysis of more than 1,500 air atomized Cu-4.3Sn powder particles was performed in compliance with ASTM E2651 standards for powder particle size analysis. Particles were finely dispersed on carbon tape, as shown in Figure 7, and measured using image analysis software. The distribution showed that 99% of the particles were less than or equal to 50µm in size (longest dimension), and Figure 8 shows the histogram summarizing the results.

Figure 7 - Scanning electron micrographs of finely dispersed powder used for particle size distribution determination
B.2 Microstructural Development:

During the atomization process, a number of possible microstructures can be produced as a result of various cooling rates experienced by individual particles during solidification. A range of possible structures exist from amorphous (rapid cooling) to dendritic (slow cooling), just as in conventional casting technologies. In Figure 9, it is possible that molten droplets impacted solidified particles during atomization, causing heterogeneous nucleated solidification, and dendritic grain growth radiating from the nucleation site [8]. Just as in conventional manufacturing processes, such as casting, these microstructural features can be observed in powder specimens. Micro- and nano-size equiaxed grain structures were also seen, showing large variability in the heat extraction from each particle.

Figure 9 - Polarized light optical micrograph of air atomized Cu-4.3Sn powder showing various grain structures, including dendritic, columnar, and equiaxed, resulting from inconsistent and non-uniform heat extraction

10mm$^3$ cubes of the Cu-4.3Sn alloy were fabricated by the industrial supplier using optimized laser parameters for a maximum density of approximately 96%. Polarized light optical micrographs were collected for both the build direction as well as the transverse direction, and some are shown in Figure 10 and Figure 11, respectively.
Figure 10 - Polarized light optical micrographs of fabricated cubes via selective laser melting with build direction indicated. First/bottom layer (left); Last/top layer (right)

Figure 11 - Polarized light optical micrographs of fabricated cubes via selective laser melting oriented transverse to build direction. Front cube-face (left); Rear cube-face (right)

Highly indicative of the selective laser melting process, melt pools were observed with noticeably elongated grains along the build direction in Figure 10. Furthermore, micro-voids are can be seen in areas separated by fully-dense material, also unique to this process. Figure 11 shows the microstructure produced in the transverse orientation to Figure 10. It is clear that the grain structure is more homogenous with fewer elongated grains, and less porosity than along the build direction. Additionally, at the bottom of the left-hand images and at the top of the right-hand images in both Figure 10 and Figure 11, the mounting material can be seen. The interface between the fabricated cube and the mount exhibit high surface roughness, which indicates poor process control during scanning, which is especially evident in Figure 10.

C. Conclusion:

My involvement in this project was focused primarily on: demonstrating my ability to conduct research on the properties of particular copper-based powders and their applications in electrical connectors manufactured by the additive manufacturing process called selective laser sintering/melting; metallographically preparing specimens of considered feedstock materials, as-printed parts, and powders for observation and analysis; providing supplementary measurements and analyses for my supervisors for statistical confirmation and validity; and implementing my expanding knowledge of powder metallurgy and particulate materials processing through hands-on experience and coursework in presentations and reports.

Through this opportunity, made possible by the Center for Powder Metallurgy Technology, I have been able to utilize and refine my skills in researching, characterizing materials via light optical and scanning electron microscopy, as well as investigating fundamental relationships integral to powder metallurgy through particle size (and size distribution) analyses, thus broadening my understanding of the diverse field of materials science.
and engineering. This experience and the skills developed throughout this project have proven invaluable as my academic, as well as professional, careers continue. I have leveraged such knowledge in employment as well as graduate school applications, and much focus was placed on this experience during interviews with Hitachi High Technologies America Inc., for whom I have recently been employed as an Applications Engineer in the Nanotechnology Systems Division based in Clarksburg, MD.

I would like to express my sincere gratitude for providing me the opportunity to earn this valuable research experience on such an interesting process. The work I have done, and will do, enables me to further solidify the fundamental and more advanced concepts in materials science & engineering through hands-on laboratory work and collaboration with my supervisors and peers. Thank you.
References:


