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Manufacturing Process and Parameters Development for Water-atomized Zinc Powder for Selective Laser Melting Fabrication

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ABSTRACT

Biodegradability of metals is a desirable characteristic for medical implants. Metals like Fe, Mg, Zn and their alloys are usually preferred for this application, as their degradation rate has been shown to work on medical implants. The fast degradation rate of Mg may early compromise its structural performance for these components; while the slower degradation rate of Fe may also become a disadvantage. This leaves Zinc's degradation rate more suitable for this application. Vaporization temperatures make zinc a challenging material to use in conventional additive manufacturing systems. In this work, the process of developing parameters to print water atomized zinc powder is presented. This process was performed in a commercial SLM system, implying inconveniences for a powder not optimized for AM. Optical analysis of water-atomized powder was conducted for size and shapes measurement of precursor powder. This work includes density and microstructure analysis, followed up by conclusion and remarks.

INTRODUCTION

Biodegradable metals have a promising application for medical implants, as they are capable of degrading progressively inside the human body [1]. The use of such metals for implants eliminates the need of a second surgery to remove the previously installed implants, usually made from stainless steel and Ti-alloys. There are three main biodegradable metals used for this purpose: Fe, Mg, and Zn [2]. Fe is a simple material to use on additive manufacturing (AM) equipment due to its extensive use in research and has the highest mechanical strength of the three. Mg shows good biocompatibility and similar mechanical properties to Fe. These two metals share a shortcoming though, their degradation rate is not optimal for medical implants, as Fe degrades too slowly, and Mg degrades too quickly. This leaves Zn somewhere in the middle of the two previously mentioned metals. Zn has a possible application in small implants, like those found in cardiovascular stents, but its use for large implants should be researched more, due to toxicity risks generated by an extreme Zn intake [3][4][5].

Selective Laser Melting (SLM) is an AM technique that uses a high-power laser to melt and fuse metal powders together from a CAD file. The 3D CAD file is sliced into 2D images, which is how the layers are formed, and is how the machine produces parts. Depending on the file, prints can range from a few hundred layers up to thousands of layers, each layer being micrometers in height. Atomized metal is used under a controlled atmosphere of inert gas, with either nitrogen or argon being used, with the latter being one of the most used in AM, due to its natural abundance and relative low cost. The metal powder is laid by a recoater that moves back and forth over the build plate, or side to side depending on the machine used, and leaves a layer of powder. This layer of metal powder can range from 20 μm to 75 μm. After the powder is layered, the laser then fires at the powder and follows a pre-determined path in order to melt that layer and previous layers, which is what ensures adherence. SLM has been shown to be a useful tool for the development of medical implants, as some implants can have complex geometries.

Metal atomization is the process of manufacturing metal powder from a solid or molten state, depending on which process is used. Water atomization uses a falling stream of molten metal that is blasted by water jets [5]. This causes the metal to freeze rapidly and forms fine powder or particles that are larger than 1 mm. Pressure plays an important role on water atomization, as raising the pressure and changing jet angles on the melt metal can influence how fine the particles will be. Water atomization is one of the least expensive processes available in comparison to plasma atomization [6]. This process does not create the most clean and spherical particles, due to the faster cooling rate when in comparison to plasma atomized powders, cooling rates can range from 10 to 100 times faster [6]. This can turn problematic for its AM application, as spherical powders have been shown to produce higher density parts and can be easier to work with.

Process and parameter development constitute the primary focus in this investigation for water-atomized zinc powder, used to manufacture lattice structures on a commercial SLM machine. Challenges arise when expanding to non-developed materials for the AM industry. By developing lattice structures for water atomized zinc powder, progress is achieved in increasing the usage of AM technology in searching for new alloy materials and manufacturing capabilities. Parameter development for the manufacturing of zinc powder on commercial addictive manufacturing equipment adds to the current research of biodegradable metals for medical implants. Ideally, pure Zn is used when conducting any research for its development. Water-atomized Zn powder was used entirely for this experiment. A commercial SLM 125 HL was used for the development of parameters. Due to zinc's relative low melting point in comparison to other metals, custom zinc plates were needed to assure adhesion on the first layers.

EXPERIMENTAL PROCEDURE

Water-atomized Zn was used in a commercial SLM 125 HL machine to develop parameters for its use in additive manufacturing. The SLM 125 HL is equipped with single (1x 400 W) IPG fiber laser. The laser has a beam focus diameter of 70 μm to 100 μm and a maximum scan speed of 10 m/s. The build plate dimensions are 125mm by 125 mm. The Zn powder used arrived in 4 different containers, with the particle size of each container being tested. Table 1 shows the size class of each container, while Fig. 1(a) - (d) shows the distribution size of each container. All of the particle size and shape characterization was conducted using dynamic image analysis (DIA) technology (CAMSIZER X2, Haan, Germany).

	Container A		Container B		Container C		Container D	
Size Class	P3 [%]	Q3[%]	P3 [%]	Q3[%]	P3 [%]	Q3[%]	P3 [%]	Q3[%]
0.0	.17	.17	.12	.12	.11	.11	.15	.15
10.0	1.96	2.13	1.95	2.07	1.97	2.08	2.62	2.77
20.0	18.04	20.17	17.33	19.40	17.50	19.58	18.72	21.49
30.0	50.31	70.48	48.30	67.70	48.71	68.29	48.30	69.79
40.0	28.43	98.91	30.95	98.65	30.98	99.27	29.27	99.06
50.0	1.00	99.91	1.27	99.92	.68	99.95	.88	99.94
> 60.0	.09	100.00	.08	100.00	.05	100.00	.06	100.00

Table 1. Particle size class characterization by container

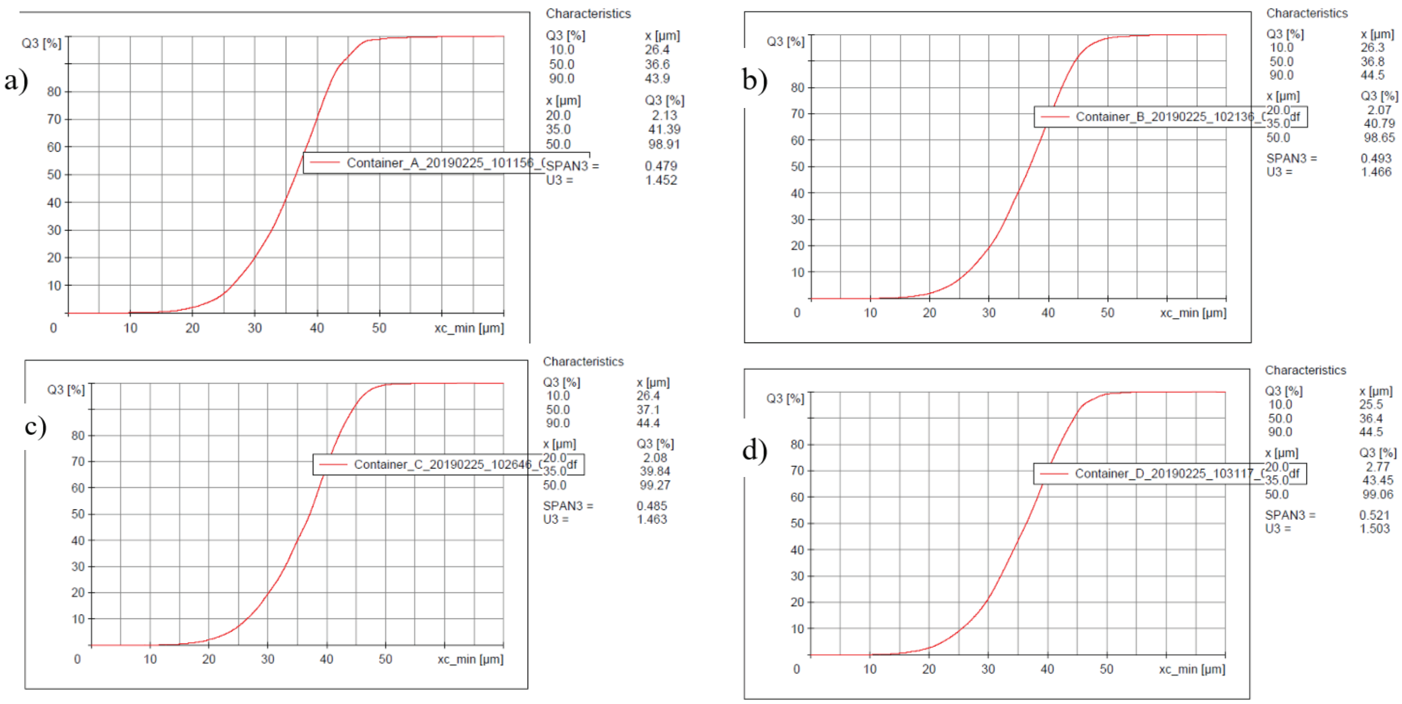

Fig. 1. (a)- (d) distrubution chart of ppwder for each container

Fig. 2 shows how non spherical and irregular the particles are. Although water atomization is one of the cheaper methods of atomization, the images show why most research is conducted on plasma atomized metal powders. The irregularity in shape can increase the failure possibilities to the experiments. Flowability also becomes an important factor when using water atomized metal powders. Not having a consistent spread of the powder onto the build plate will compromise the quality of each print. Due to the little research conducted on the use of Zn for the AM industry, custom Zn plates were manufactured for the use of this project. Previous research had stated that using water-atomized Zn was not ideal, and that it could not be done [7].

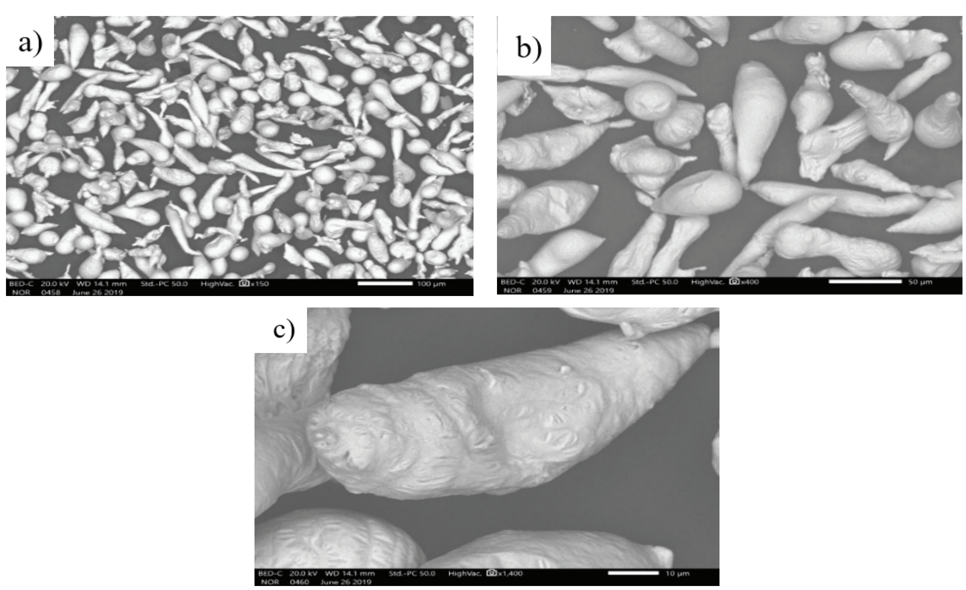

Fig. 2. Water-atomized Zn under a microscope at a) x150, b) x400 and c) x1400 magnification

Initial parameters were based on an experiment by Wen et al [7]. In this experiment, they developed a chart with parameters that plotted the laser power (W) used against the speed of the laser (mm/s). In this chart, they had higher density of printed parts as their goal. Fig. 3 shows the results of varying the laser power and scanning speed on pure Zn powder. For the purposes of this experiment, the initial parameters for tests were set to a laser power of 80 W, with a hatch speed of 400 m/s and a border speed of 100 mm/s, as this set of parameters yielded positive results from Wen et al [7].

RESULTS AND DISCUSSION

The initial parts printed were a set of cubes that measured 10 mm all around. The first set of parameters managed to print successfully, but certain problems were evident. Both the hatch and the border of the cubes did not adhere to one another, plus the borders of some the cubes were peeled, as Fig. 3 shows. After the first print job, increasing the velocity of the cube borders seemed to fix the peeling issue. Changing the border speed to a range of 150 mm/s to 200 mm/s yielded better results for the cubes, as shown on Fig. 4. The first attempts of printing lattice structures can also be seen in the same image.

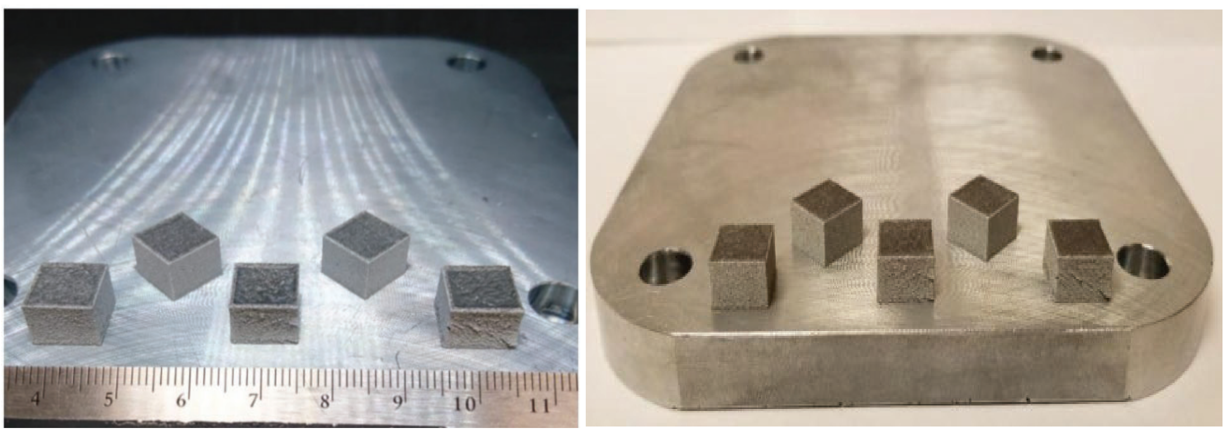

Fig. 3. First print of 10 mm cubes with hatch speed of 400 mm/s and a border contour speed of 100 mm/s

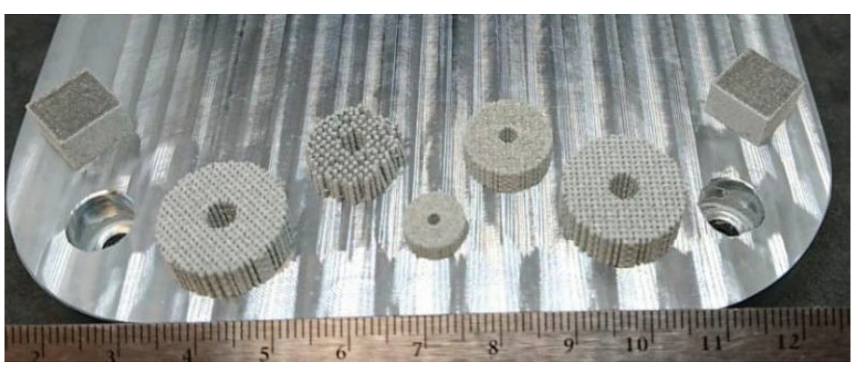

Fig. 4. Cubes printed with hatch and border speed of 400 m/s and 150 mm/s, respectively. First attempt of printing lattices are also shown

After being able to replicate cubes after each print, a set of 9 cubes was printed in order to test for density and analyze microstructure of each cube. Fig. 5 shows the set of parameters chosen for the experiment and their corresponding densities, tested by the Archimedes' Principle and Density Determination. Cubes located inside the gray zone had the highest density achieved of this experiment, between 95-97%. Any other cubes located outside of this gray zone are not ideal for dense parts, although a combination of parameters outside of this zone can be beneficial for other purposes. Fig. 6 compares the microstructure of multiple cubes printed in order to observe the effect of different parameter sets.

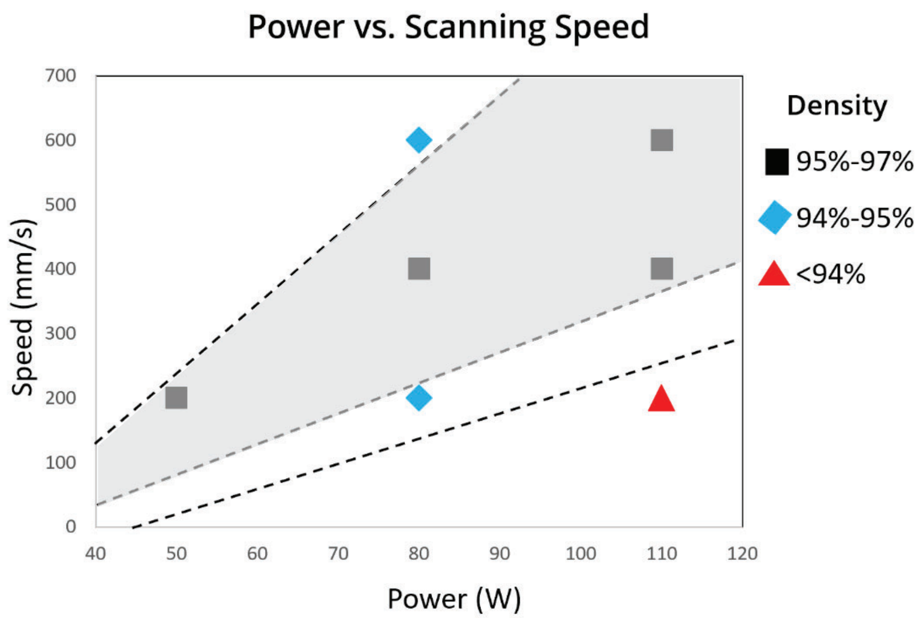

Fig. 5. Relationship of density under different power (W) and laser speed (mm/s)

After being able to successfully replicate cubes and obtaining data, the focus shifted to being able to create lattice structures. Using the same parameters for the lattices that were used for the cubes did not yield expected results, as the lattice structures came out as solid cylinders, as Fig. 7 shows. This outcome was hyphothesized to be due to the high power of the laser, as the small features of the lattices were being fused together. This led to the lowering in power laser to a range of 20 to 40 W and increasing the scanning speed to a range of 700 to 900 mm/s. These two sets of ranges have different effects on lattice structures when created with water atomized Zn powder. If both the laser power and scanning speed are on the lower spectrum of the previously mentioned ranges, the lattices can be destroyed by simply touching them, which renders them useless, and vice versa, if the both parameters were set too high, solid cylinders would be the result.

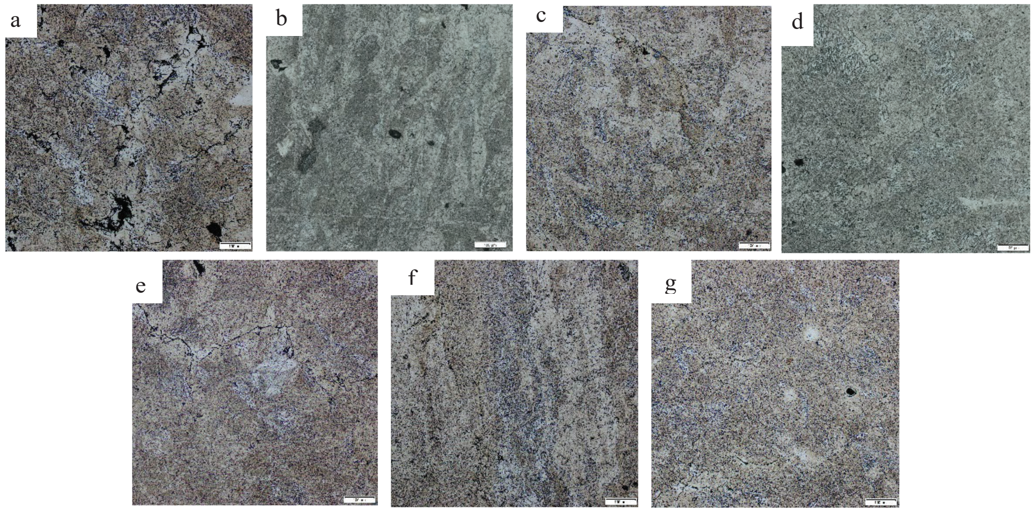

Fig. 6. Microstructures of cubes from least (a) to greatest (g) density achieved shown

Modifying the parameters would still yield solid cylinders, so the focus was shifted to the design of the lattice structure itself. Since the lattice itself was a CAD, any modifications made would take a long time to render. This led to a different approach, creating lattices from support structures inside Materialise Magics software, as Fig. 8 shows. This approach allowed for the modification of the design to be easier.

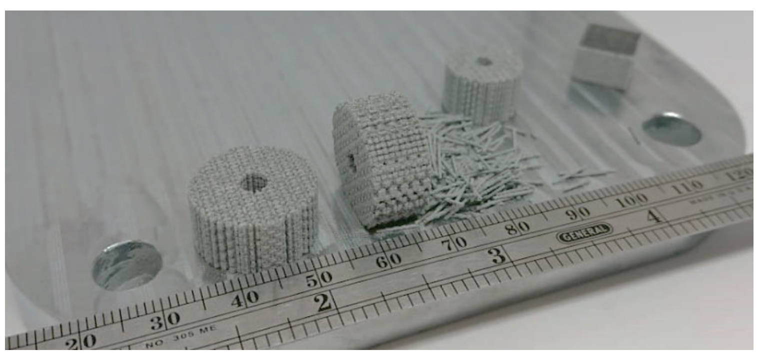

Fig. 7. Initial set of lattice structures printed

After using this approach, lattices began to have better resolution when printed, as Fig. 9 shows. This allowed for the creation of lattices with smaller features, until a minimum feature size was reached, anything smaller than 50 µm in size could not be printed, at least in our experiments.

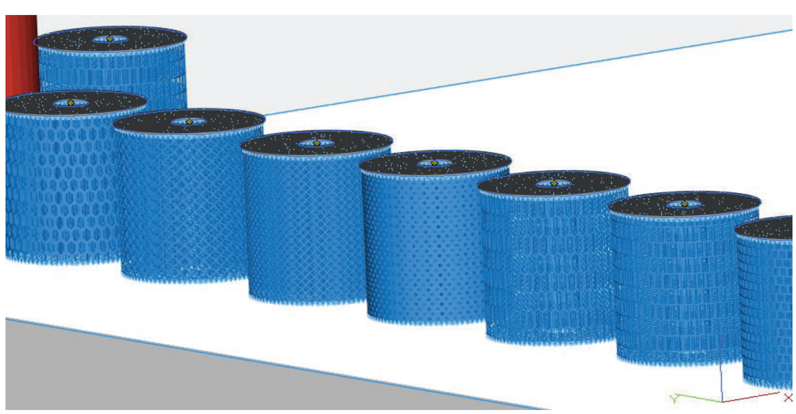

Fig. 8. Materialise Magics support structures concept

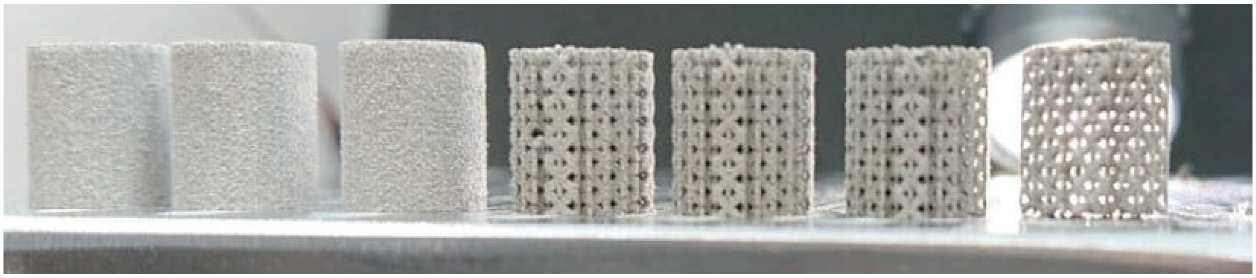

Fig. 9. Lattice structures made from support structures inside Materialise Magics software

This software allowed for simple modifications to already modeled lattices. Besides developing parameters for the use of water atomized Zn in AM, getting a high level of detail on prints was also desired. After more lattices were printed, it was determined that using a scanning speed within a range of 600-800 mm/s and a laser power of 20-40W yielded the best lattices for our experiment, as Fig. 10 demonstrates.

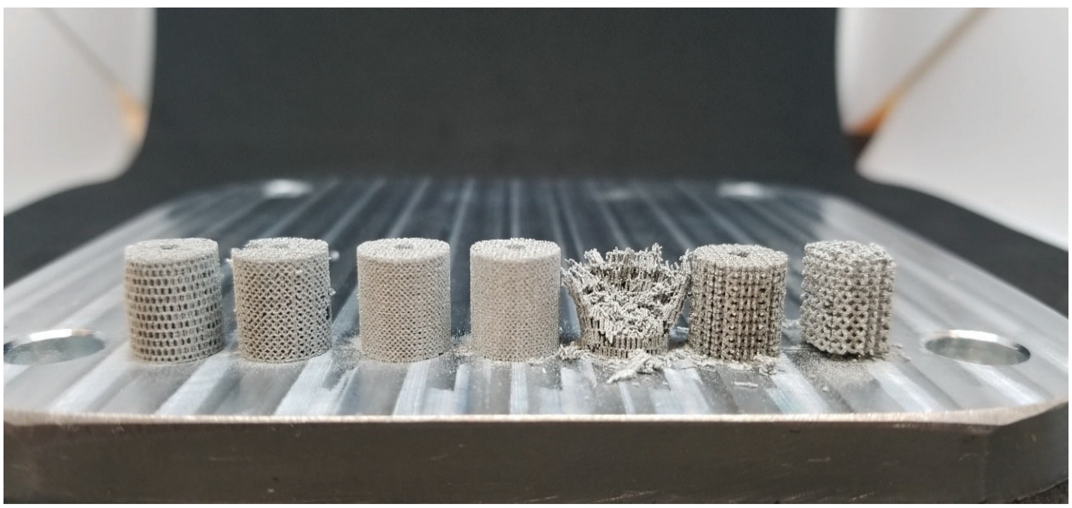

Fig. 10. Lattice structures with different designs with a laser power of 30 W and scanning speed of 800 mm/s

Conclusion

Lattice structures have been produced in a commercial SLM machine using water-atomized Zn powder. The main focus of this research was to utilize water-atomized Zn powder and produce parts, in this case, lattice structures with small sized features. This contributes to the on-going research to expand AM use for biodegradable medical implants. Water-atomized powder is less desirable than other forms of powders used in the AM industry due to its irregularity in shape. Further research will be conducted in order to explore more uses for water-atomized Zn powder for additive manufacturing, which may include implant candidate designs, such as cervical fusion implants and coronary stents. Microcracking, mechanical performance, and biodegradability testing will also be conducted in the future.

Acknowledgements

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