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Effect in thermal conductivity due to compaction of powdered stainless-steel 316L used in additive manufacturing

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Abstract—Metallic and plastic powders have been used in additive manufacturing for many years. Unfortunately, in processes such as powder bed fusion, the mechanical properties of the parts are different than using traditional machining methods. Some of the deficits of printed parts are directly attributed to the layer by layer process, where the density of the printed parts is overall lower because of the voids generated between the powder particles. Such voids can be generated by insufficient material and/or melting energy. In the previous years, several parametric studies in 3d printing processes have been performed. At this moment, experimental studies using powders are limited because its complexity. The presented research studied on the different thermal distribution of the powder particles under different arrangements in order to improve their thermal conductivity. Our experiments show that compacting the powder helped to reduce the gradients in temperature under certain temperatures more than 50%.

Keywords-component; additive manufacturing; powder; stainless steel; thermal conductivity

I. INTRODUCTION

Nowadays, there is a big number of manufacturing processes[1]. One of the most recent process, which is gaining popularity in the last decades is the additive manufacturing. Now, additive manufacturing equipment is becoming more affordable and accessible. In the case of polymers, they are considered the most popular materials in additive manufacturing; moreover, there is a big community of people developing materials, models, and process optimizations[2]. On the other hand, metallic materials for additive manufacturing are still in development, many metals cannot be used due to their inadequate solidification [2]. The costs are still considered high, not only the final parts, but also the equipment and materials. Some of the industrial limitations are attributed to the complexity of the systems like number of materials available. Others are occasioned by the additive manufacturing concept, like slow mass scale production or properties challenges.

There are different processes to print metallic materials. Some of the most popular are based on powder bed fusion. In a brief words, powder bed processes share the same elements, which are as follows; at least one heat source (laser or electron beam), a movement controller for the heat source, and a system to control the powder's layers[3].

Some manufacturers use a roller or a blade to add more powder. In the case of EOS company, they use a hard-recoating technology to get a higher density. The coating blade is intended to be harder than the material processed to avoid contamination. However, comparing to soft recoating systems, some shear forces can be generated by hard recoating blades; hence, there is a risk that the printing process can fail [4].

When the laser is melting the material, the effects caused in the powder are complex due to the high thermal gradients in the particles. As a result, the material will expand and contract in different direction, generating a common problem called residual stress [4].

Other problems in the process are attributed to the lack of melted material. When the laser spot is in contact with the powder bed, some quantity of material is vaporized for the large amount of energy used in the laser. Moreover, the laser energy throw powder particles out of the path. Hence, the total of material available to be melted decreased, causing porosity[5].

In order to increase the properties of the printed parts, a characterization of the raw powder was performed. The thermal conductivity is an important factor to understand the thermal gradients in the material. One of the reasons is that it is a parameter that increases with temperature, then, knowing its behavior at sintering temperatures will help to have more accurate models. As a result, optimizing the thermal conductivity can help to improve the properties of the parts.

There is not a formal standard to measure the thermal conductivity in powders, however ASTM mentioned the *thermal hot plate* method as an approach to use [6]. However, the hot plate has limitations of the reachable temperature related to its setup. The higher temperature, the more complicated is to have a safe apparatus, due to the heat resistant of the elements. In contrast, furnaces can safely reach more than 1000°C, which is more suitable for the intended results. Furthermore, the cylindrical shape has the advantage of not having heat lose[7]. Finally, the tubular furnaces are suitable to be used in inert atmospheres and vacuum

II. METHODOLOGY

Measure the thermal conductivity of a single powder particle which a size of around 40 microns is not a simple task; moreover, the interaction with other particles make more challenging to determine the effective thermal conductivity between powder particles. Additionally, the variation of the radius and porosity generate many different configurations, changing the contact area between the particles. Therefore, the following experiment is not limited to an isolated sphere.

The apparatus used in the test was a tubular furnace manufactured by Carbolite-Gero model EHA 12/450B, using the EUROTHERM 3216 PID controller. The maximum temperature that can be reached with such equipment is 1200 Celsius, which is an adequate temperature taking into account the sintering point of stainless steel. An important consideration using the furnace is that the ramping rate of temperature is limited to 5 Celsius per minute.

The powder used for the experiment was manufactured by EOS under the name of "EOS Stainless Steel 316L", a material that can be powdered and use in a significant number of metal 3d printers. EOS claims that the 316L class of stainless steel is appropriate for a large number of fields, for instance; automotive, aerospace, jewelry, food and chemical plants, and medicine. Some of the properties that they highlight includes the corrosion resistance, ductility, opportunity of post processing, and the possibility of been used for surgical implants. [8].

The recording of the temperature was performed using a thermocouple data logger OMEGA HH506RA with thermocouples type-K model OMEGACLAD XL connected, Figure XX thermocouple 1 and 2. The logging of the data had been recorded directly to the computer in steps of 3 seconds. In addition, a second pair of thermocouples were added to monitor the temperature of the water

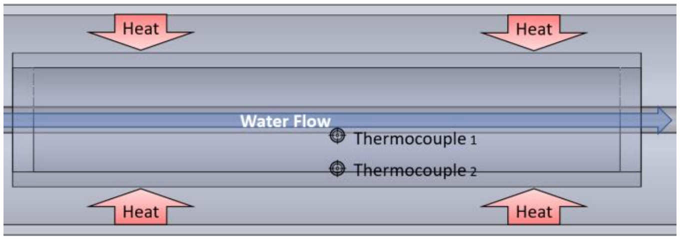

Figure 1, Experimental setup, showing the positions of thermocouples 1 and 2

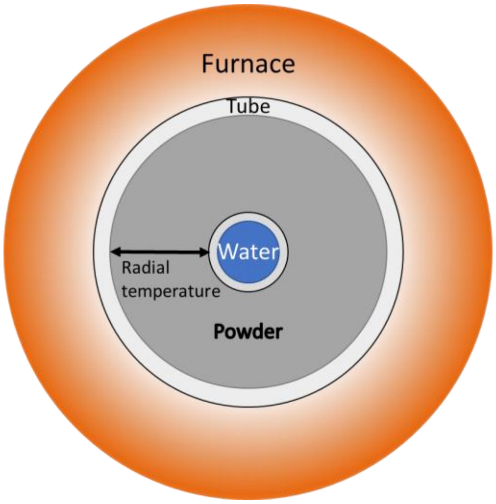

Figure 2, Sectional representation of the experimental setup

The follow experiments were performed to archive 2 goals while identifying the differences in thermal conductivity using different methods. The first one is to magnify the importance of pre-heating process. Secondly, test the thermal conductivity reaching the sintering point, were the particles starts to consolidate. Reaching the melting point of the material was not a part of the experiment, the portion of the sample being measured, would be changing drastically; therefore, the measurements will not be accurate.

The container of the powder was a stainless-steel tube, which was placed in the center of the tubular furnace. In order to get a uniform distribution of the heat, the sample was placed in a ceramic support that covered just a small part of the surface in the bottom.

The powder in the samples were prepared using 2 methods. The first case is just powder deposited without external factors, just the powder failing by gravity. On the other hand, for the second sample the powder was compressed 10% of its original size. The test expected a better thermal conductivity in the compressed sample tested.

The experiment captures the changes in gradients of temperature every 100°Celsius, starting at 100° at ending at 1000°.After reaching a study point in the furnace, the temperature is hold in order to reach a steady state in the system. For that steady state, the gradient of temperature of the water between thermocouple 1 and 2 is measured. Then it is possible to get the heat flux in the system using Equation (1)[9].

$$Q = m * C_p (T_b - T_a) \quad (1)$$

Where:
Q = Heat flux (J/s) (W)
m = mass flow (kg/s)
Cp = specific heat (J/kg*K)
Ta = temperature before furnace (K)
Tb = temperature after furnace (K)

The temperature of the water should be monitored to avoid reaching 100°Celsuis. However, to measure the temperature it is better to have a laminar flow in the system. The number off Reynolds determine if the flow is a laminar, or turbulent. In our case it is important to get a number under 2300 to get a laminar flow. The number of Reynolds is calculated with Equation (2)[10]. Note that some of the properties of the water change with the temperature, it was considered as part of the calculations.

$$Re = (\rho\ V\_average * D)/\mu = (V\_average * D)/v \qquad (2)$$

Where:
Re = Reynolds number
ρ = density of the fluid
V_ average = average velocity
D = diameter
μ = dynamic viscosity
v = average viscosity

Finally, we got all the variables to get the thermal conductivity using Equation (3)[10]. As the heat flux must change for all the cases in the different samples.

$$Q = k * S * \Delta T / L \qquad (3)$$

Where:
Q = heat flux (W) (J/s)
k = thermal conductivity (W/(m*K))
ΔT = thermal gradient between thermocouple 1 and 2 (K)
L = distance between thermocouples 1 and 2 (m)
S= surface contact (m²)

III. Results

First, the temperature in the water during the steady state remain in almost constant value as it is shown in Figure 3. When the water flow was established at 30LPH to get a laminar flow; the calculated Reynolds number was 1890 using Equation (2). The temperature of that segment corresponds to the steady state indicated in Figure 4

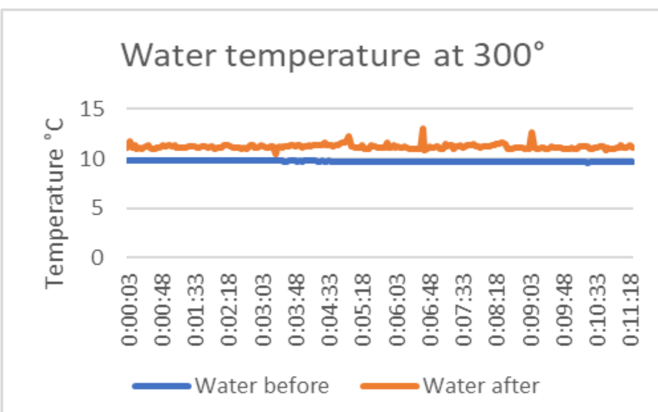

Figure 3, Example of water temperature in the system. The water was measured before and after going through the furnace

Having the measurements of the water, it is possible to calculate the heat flow in the system, using Equation (1). The results show the amount of heat moving in the system, Figure 5. As it can be seen the amount of heat is re

Preliminary results can be observed in Figure 6, when the gradients of temperature are compared. Compressed powder shown a more compacted distribution in temperature.

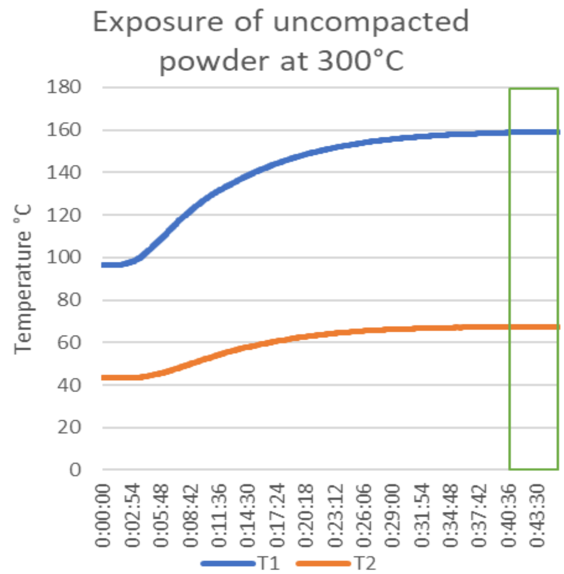

Figure 4, Example of results obtained from the powder sample in thermocouples 1 and 2, denoting the data for analysis in steady state

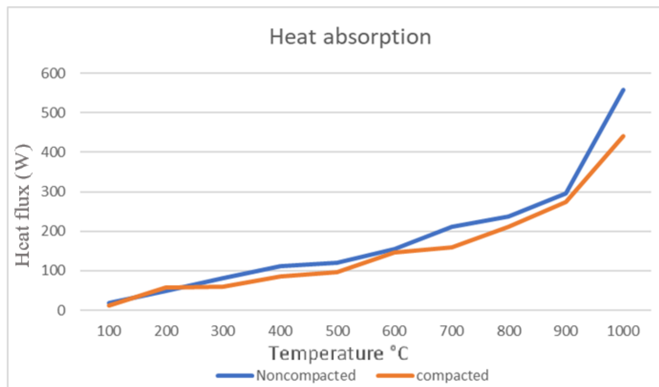

Figure 5, Heat flux absorbed by the system at different temperature points

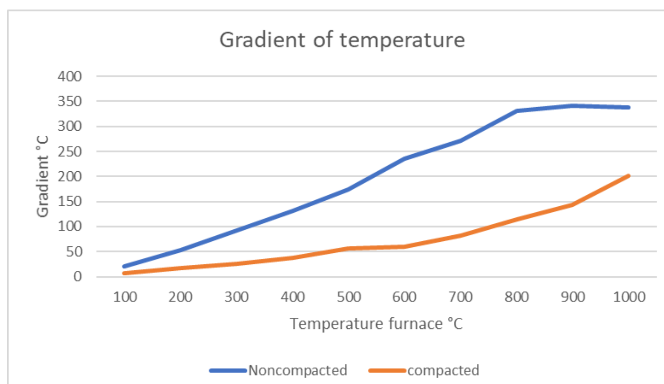

Figure 6, Comparing gradients of temperature at the different stages of the experiment.

IV. DISCUSION

In the calculations of the heat flux, note that the temperature in the water is not increasing drastically with the temperature inside the furnace. The reason is related to the nature of the heat source. The tubular furnace radiates to the surface of the tubular container. As a result, the powder sample is interacting with 2 different types of conduction; a conduction flow of water causing convection, and radiation emitted from the furnace. Hence, the water flow will have a greater impact, as it can be identified in Figure 5 where the greater temperature in the powder is just 53% of the temperature in the furnace

After comparing and interpreting the results from the gradients in temperature using Equation (3), we can assume that the bigger gradient has a significance of a lower thermal conductivity. Then the thermal conductivity was calculated using Equation (3) and was compared with the kwon values from the literature, Table 1. The calculated thermal conductivity of powder is only a fraction of the bulk material. Such findings have been attributed to the nature of the samples, the area of contact and voids between the particles significantly affects the thermal conductivity. However, the compressed sample got a higher thermal conductivity.

TABLE I. CALCULATED RESULS THERMAL CONDUCTIVITY

Stainless steel	**Thermal conductivity (W/m K)**		
	Bulk Material	***Compressed***	***Non-compressed***
Minimum (100ºC)	15.87	0.3612	0.1736
Maximum (1000ºC)	27.3	0.6676	0.2822

V. CONCLUSION

Using an alternative experiment to the hot plate, it is possible measure thermal conductivity under different circumstances. The experimental setup has the possibility to change with minor changes the controlled atmosphere. More experiment should be conducted with different materials and under different conditions.

Compacting the powder as a part of the printing process play an important role. The thermal conductivity in the compacted structure resulted in a higher thermal conductivity. 10% of the volume compacted revealed a high difference, following experiments can compared that value with other compression rates.

The experiment can be considered as a base to make deeper studies of the impact of powder structures in the thermal conductivity. Likewise, optimizing the powder structures would be beneficial to reduce previously mentioned problems in the printing process.

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