Porous materials additively manufactured at low energy: Single-layer manufacturing and characterization

Davoud Jafari a,⁎, Koen J.H. van Alphen a, Bernard J. Geurts b, Wessel W. Wits c, Laura Cordova Gonzalez a, Tom H.J. Vaneker a, Naveed Ur Rahman a, Gert Willem Römer a, Ian Gibson a

a Faculty of Engineering Technology, University of Twente, P.O. Box 217, 7500 AE Enschede, the Netherlands
b Faculty EEMCS, Multiscale Modeling and Simulation, University of Twente, P.O. Box 217, 7500 AE Enschede, the Netherlands
c Thales Netherlands, P.O. Box 42, 7550 GD Hengelo, the Netherlands

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ABSTRACT

This paper presents an appropriate method to significantly reduce the pore size of high porosity porous stainless steel 316L structures fabricated by laser powder-bed fusion (LPBF) utilizing pulse wave emission (PW). PW deliberately avoids full-melt and applies low energy conditions to achieve single layer sintered porous material with controlled characteristics. Experimental approaches on a lab-scale setup equipped with a pulsed fiber laser system were developed to investigate the effect of laser scan settings. Properties of low-energy laser single sintered layers are studied experimentally, and the influence of laser power and pulse duration is discussed. A layer of sintered porous material was characterized in terms of the pore size, layer thickness, porosity and thermal conductivity. The results show that sintered porous layers can be fabricated by effectively connecting metal powder in the powder bed similar to a sintering process or partial melting. The porosity of fabricated structures was 51%–61% and the average pore radius ranged between 22 and 29 μm. We found that the thermal conductivity of a single powder particle is 31.5% of the sintered layer value and the thermal conductivity of the sintered layer is 4.8% of its solid material.

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1. Introduction

Porous materials are vital elements for a range of industrial applications, as they offer a number of special mechanical and thermal properties associated with their low density and large specific surface area. Various pore architectures and porosities can be engineered thanks to additive manufacturing (AM). Laser powder-bed fusion (LPBF) technology is a commercially available AM technology, in which parts are manufactured by depositing thin layers of powder particles repeatedly to form three-dimensional (3D) objects. An energy source is applied to particles deposited in a powder bed followed by selectively melting a pattern corresponding to the cross-section of the part that is being formed. LPBF enables the creation of metal components, using computer-controlled high-energy laser illumination, from 3D model data by incorporating material layer by layer. One of the main advantages of the LPBF system is the ability to manufacture complex freeform structures such as porous materials [1–3].

For two-phase devices, and potentially many other applications, e.g., in aerospace, chemical processes, petrochemical and semiconductor industries [3], porosity and average pore size are two key performance indicators. For the intended application, capillary driven two-
phase devices, the porous material serves as a pump for fluid flow by generating capillary pressure within the (micro) pores [4]. The desired characteristics of the porous materials include:

a) High permeability and low hydraulic resistance achieved by a high porosity. Permeability is a measure of the openness of the structure and often it is desirable to have a high permeability or porosity. The pressure drop \( dp/dx \) for flow through a porous media, governed by Darcy’s law, describes the relationship between flow resistance and permeability as \( dp/dx \) is proportional to \( 1/K \), where \( K \) is permeability.

b) High capillary pressure which is inversely proportional to the pore radius. Based on the Young-Laplace equation, the capillary pressure \( \Delta P_c \) is defined as \( \Delta P_c = 2\sigma \cos \theta / r_p \), where \( \sigma \) is the surface tension coefficient of the liquid, \( \theta \) is the equilibrium contact angle and \( r_p \) is the average pore radius.

The unavoidable trade-off between capillary pressure and flow resistance (i.e. permeability) defines the optimal working distance within the porous material for a given fluid flow rate – a desirable balance between these conflicting properties is critical. Therefore, the minimum resolution, defining small pores and high porosity, is very important in the layout of porous materials. Recently, much research has been carried out regarding AM of porous materials focusing on LPBF [1,2,6,7]. In this approach, a designed part through 3D modeling software is constructed from fully molten adjacent lines typically referred to as the hatch pattern. Similarly, layer to layer full melting is required to build parts. For such processes the minimum feature size is determined by the thinnest fully molten line the laser system can produce. Thus, a porous material can be constructed part through 3D modeling software is constructed from fully molten ad-

| Refs. | Laser system | Unit cell | Pore size | Porosity (%) | Material |
|-------|--------------|-----------|-----------|--------------|----------|
| [1,14] | MLab Cusing 90; yttrium fiber laser 100 W; \( \lambda = 1.07 \, \mu m; r = 40 \, \mu m \) | Not reported | 216 | 46 | Stainless steel 316L |
| [9] | M2 Cusing; yttrium fiber laser 200 W; \( \lambda = 1.07 \, \mu m; r = 100 \, \mu m \) | Not reported | 250 | 90 | Stainless steel 316L |
| [10] | In-house SLM machine; yttrium fiber laser 200 W; \( \lambda = 1.07 \, \mu m; r = 40 \, \mu m \) | 700–1000 | 68–82 | Ti-6Al-4V |
| [11] | SLM 280; \( \lambda = 1.07 \, \mu m; r = 40 \, \mu m \) | Not reported | 80 | 17 | Stainless steel 316L |
| [12] | MCP Realizer; yttrium fiber laser 200 W, \( \lambda = 1.07 \, \mu m; r = 50 \, \mu m \) | 300–500 | 20–60 | Al6061 |

The details of aforementioned studies regarding manufacturing metal porous materials via LPBF are summarized in Table 1.

In all the above reported research, fabrication of the porous material was limited by its minimum attainable feature size, which is at best around 100 \( \mu m \) [15]. Above works on LPBF consider laser beams with continuous wave (CW) emission. However, regulation of the heat input and therefore the kinetics of solidification and fusing of powder particles is required. A solution to offer increased control over the kinetics is the use of a pulsed wave (PW) laser system [16–18]. This is due to pulsed lasers having a smaller heat-affected zone that minimizes the region on the substrate that the laser is sintering. This allows greater control over the heat input and melting pools during the process and thus enables better spatial resolution and feature sizes [19]. In recent years, LPBF of metal particles using PW emissions has been studied using various materials. The focus has previously been set mainly on, thin-wall components [20–22], the modification of microstructures [18,23–25], fully dense components [26], and melting efficiency [27]. In all cases, full melting under PW conditions was aimed at. There is, however, very limited work out on the PW sintering to fabricate porous materials [28].

LPBF utilizing PW lasers offers the additional parameter of pulse power (pulse energy/pulse duration), which has a significant effect on the quality of manufactured components [17]. Very little work has been done to check the system requirements needed to fuse the metal particles. Therefore, a thorough understanding of the dependence of processing parameters is investigated here, with a wide range of pulse duration and pulse energy. The challenge when using this method for manufacturing a desired porous material with a right pore size and porosity, reliably and repeatably, is to apply the right amount of energy to the powder bed, thereby partially melting the powder particles or sintering, resulting in a packed non-fully molten powder that can have considerably smaller feature sizes compared to the CW mode. In this paper, LPBF utilizing PW emission is investigated for its use in producing highly porous materials with small pores exploiting partial melt conditions. The goal of this paper is to explore low-energy laser processing to achieve a high porosity in a porous material while reducing the pore size similar to that of a sintered structure.

Additively manufactured parts are constructed from multiple layers stacked on top of each other. Naturally, this would also hold for our envisioned porous materials. Therefore, before progressing to multi-

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Table 1: Summary of literature research for metallic porous materials manufactured via LPBF (CW emission, where \( \lambda \) is the wavelength and \( r \) is laser spot radius).
layer porous material manufacturing, it is critical to fully understand and solve problems within single-layer manufacturing. Hence, in this study we investigate porous materials within a single layer, taking fluence, which expresses the energy density at the peak of an ideal beam with a Gaussian distribution, as the process parameter to (1) discuss the relationship between fluence, porosity and pore size, (2) observe and analyse the forming characteristics of the porous material during laser sintering of metal powders, and (3) test the thermal conductivity of stainless steel 316L printed layers. In this study, both the thermal conductivity of stainless-steel powder and single-layer 3D-printed porous materials were measured and compared to available experimental data and correlations in the literature.

In order to document our proof-of-concept the following steps were taken. First, an experimental design and experimental apparatus were developed, presented in Section 2. The results and discussion from the experiment and specimen characterization are described in Section 3. Finally, conclusions are presented in Section 4.

2. Experimental development, specifications and characterization

In this section, the experimental setup is introduced in Subsection 2.1, the main variables are introduced in Subsection 2.2 and characterization details are given in Subsection 2.3.

2.1. Experimental setup

The powder feedstock used is gas atomized, stainless steel 316L (supplied by ConceptLaser). The powder was characterized using scanning electron microscopy (SEM). An image of the powder feedstock is shown in Fig. 1. Powder particles are generally spherical in shape and have a wide distribution of size; mostly particle diameters are within 30–47 μm range.

A purpose-build test set-up to characterize laser-powder interaction (see Fig. 2), was employed throughout this study with a maximum laser power of 100 W generated by a 1080 nm Ytterbium fiber laser. The experimental set-up consisted of a laser source (JK100FL laser), focussing mechanism, a laser positioning stage (Thorlabs KS1-Z8), a camera (STC-P635B), a processing chamber and a powder layer deposition system.

A layer of stainless steel 316L powder was deposited on a stainless-steel substrate. To deposit a layer of powder on the substrate, a manual powder deposition mechanism was used, capable of depositing a layer thickness of 200 μm. In this system a small amount of metal powder with a known weight is applied with an applicator strip along a ruler. As the layer thickness and track width are known, by measuring the track length, the layer density was determined by weighing the mass of the deposited powder layer.

The laser was focussed at the top of the powder layer and pulsed with a defined pulse duration. The laser fiber entered the optical unit, which has a mirror that redirects the beam to the lens as described in detail by [29]. The mirror is reflective for the 1080 nm wavelength of the laser, but transparent for visible light. A camera was mounted behind the mirror to observe the laser spot and process interaction. The laser beam progresses through the optical unit to the tilting mirror. The lateral position of the optical unit was changed using a Thorlabs MTS50/M-Z8 linear actuator [29]. This provided the possibility of changing the focal length of the laser light from the lens to the powder bed to change the focal position. The actuator has a range of motion of 50 mm with an accuracy of 6 mm. The mirror, a Thorlabs PF10-03-P01, was tilted along one axis using a KS1-Z8 setup [29]. This allowed for a range of motion of 8°. The mirror was used to reflect the laser beam to a certain point on the substrate. Detailed information on the focusing mechanism is discussed in [29]. Finally, the laser beam passes through a transparent window into the processing chamber. This chamber was filled with argon gas as a shielding gas. The build platform could be moved along one axis. With the rotating axis of the mirror oriented across, a 2D surface could be laser scanned. All experiments were carried out in an air-conditioned laboratory environment at 22 °C ambient temperature.

2.2. Theory – variable definition

The LPBF process using PW emission to sinter a layer of powder particles is influenced by a number of parameters, which together determine the properties of the printed artefact. This includes, but is not limited to, environmental parameters, material specifications, and laser (scanning) parameters [30]. The fluence parameter (F in J/m²), which expresses the energy density at the peak of an ideal beam with a Gaussian distribution is considered in this study. Controlling the fluence on the surface is key to affect the pore size and porosity/density of 3D-printed parts [31]. The fluence determines the state of the molten powder (i.e., state of vaporization, state of fusion, state of sintering or state of heating-up). Therefore, fluence is taken as the main parameter to describe the effect of the laser conditions on the pore size and porosity of the sintered single-layer experiments of the present study. The fluence is determined by laser power (P), pulse duration (τ), and laser spot radius (r) as [32]:

\[ F = \frac{2Pr}{\pi r^2} \]  

(1)

Sintered layers were fabricated as a grid of laser dots (51 × 21). The following process parameters were selected according to a preliminary series of single spot/line scanning experiments (see Fig. 3): laser spot radius (r) of 200 μm, a point distance (dx) of 100 μm, the distance between two consecutive illuminated points, a hatch spacing of 100 μm. The jump speed was kept at s = 0.625 mm/s, describing the speed of the movement when the laser moves from point to point. Since a pulsed laser is used, the laser is fired discretely rather than continuously. In this case, the overall scanning speed is determined by point distance (dx), pulse duration (τ), and jump speed (s) as follows

\[ v = \frac{dx}{\tau + \frac{dx}{s}} \]  

(2)

In order to reliably manufacture porous materials through LPBF, several phenomena need to be investigated [33]. The main parameters include laser power and laser scan speed. Material parameters as well as laser-specific parameters, determine the amount of energy that must be deposited into the material. To estimate the energy required to
produce a desired porous layer of stainless steel 316L powder in the powder bed using the pulsed laser, the following dimensionless groups of process variables are considered:

\[ P^* = \frac{\alpha P}{Rk(T_m - T_0)} \]  \hspace{1cm} (3)

\[ v^* = \frac{vR}{\alpha} \] \hspace{1cm} (4)

where, characteristics of the beam include power \((P)\), scan speed \((v)\) and beam radius \((R)\); and the material properties include absorptivity \((\alpha)\), thermal conductivity \((k)\), and thermal diffusivity \((\alpha)\). \(T_m\) and \(T_0\) are the melt and initial temperatures, respectively. \(P^*\) and \(v^*\) regulate the material’s thermal cycle maximum temperature and heating rate, respectively. The material and process constant values used to calculate \(P^*\) and \(v^*\) include [34]: average thermal properties (conductivity and diffusivity) of 0.6 \(T_m\) and a surface absorptivity of 0.5.

According to the dimensionless process diagram presented in Fig. 4 [34], laser processing is characterized according to three regimes: heating (including necking between particles), melting and vaporization. Hence, the process parameter set of Table 2 was selected, resulting in energy thresholds required to sinter/partially melt particles (a further discussion is presented in the Subsection 3.1):

(i) varying pulse duration in the range of 0.75–14 ms while keeping the laser power at a constant level (20 W), thereby varying the fluence;
(ii) constant fluence by varying both laser power (20–100 W) and pulse duration (0.8–4 ms) simultaneously;
(iii) constant laser power (20 W) and pulse duration (4 ms), for repeatability testing.
The porosity of the specimen was measured using an analytical balance with an accuracy of 0.0001 g (Mettler Toledo). The specimen's mass was measured under atmospheric conditions and the outer dimensions were measured to determine the specimen's volume. Cross-sectional images were made using an optical light microscope. From the images and differential scanning calorimetry (DSC) test, the resulting structure. The second goal of this experiment is to collect experimental data in the literature [34]. Data collection for the three main classes: heating, melting, and vaporization. Red symbols denote failed specimens. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

The first goal is to investigate whether it is possible to create a highly porous layer using a LPBF process by evaluating the effect of fluence on the resulting structure. The second goal of this experiment is to collect statistical data and establish that the process is repeatable.

2.3. Characterization

The fabricated sintered layers were subjected to various characterization methods, including geometric and thermal analyses. The analyses performed are summarized here: optical microscopy analysis using an optical light microscope of model VK 9700 Keyence, SEM analysis and differential scanning calorimetry (DSC) test.

In view of the importance of accurately measuring the porosity of a sintered layer, three porosity measurement techniques were employed. From the top and side surfaces of the as-fabricated sintered layers, images were made using an optical light microscope. From the images the outer dimensions were measured to determine the specimen's volume. The specimen's mass was measured under atmospheric conditions using an analytical balance with an accuracy of 0.0001 g (Mettler Toledo). Thereafter, the porosity of the specimen (ε) was computed by dividing the actual mass, obtained from dry weighing (m), by the volume (V) of the parts, obtained from dimension measurements and the bulk density (ρ) of stainless steel 316L (8.0 g/cm³). The specimen porosity from direct mass measurement, so-called volumetric mass porosity, is obtained by:

\[
\frac{1}{\rho} = 1 + \frac{m_p}{m} \rho
\]  

(5)

The porosity values for each specimen were also characterized using Archimedes method. In this approach the sintered layer was fully saturated with fluid, following the same procedure of our recent work [1]. The mass of each sintered layer was recorded before and after saturating it with methanol and the porosity is measured by weighing the saturated fluid by:

\[
\varepsilon = 1 + \frac{m_p}{m} \rho
\]  

(6)

where \(m_p\) and \(\rho\) are the methanol liquid mass needed to saturate the specimen, and mass density, respectively. To complete the geometric characterization, image analysis from an optical light microscope and SEM were also used for determining the porosity level and pore size directly. Different cross-sectional images were captured. These images were then converted into de-noise black-and-white micrographs using ImageJ software and a porosity analysis tool [35]. Fig. 5 shows a cross-sectional image of an actual specimen, used for image processing.

Thermal conductivity measurements include measuring the temperature gradient and heat flow through a specimen of known geometry, using DSC. The details of the measurement process are reported in [36,37]. In this system, in the contact area between the sintered layer and the sensor, both temperature and heat flow are measured. The temperature on the opposite side of the specimen cannot be measured. The temperature of that opposite side is known during the melting of a known melting point material, i.e., indium (known melting temperature of 156.6°C), placed on top of the specimen. From the temperature of both sides and the recorded heat flow, the thermal conductivity can be determined (see Fig. 6).

We performed the experiments as follows. The sintered layer was placed in the pan and indium was placed on top of the pan while the reference furnace remained empty. The temperature must be constant during the melting of the indium; thus the top of the specimen remains at constant temperature, while the temperature of the bottom side of the specimen rises at a constant rate. To measure the differential power produced during the indium melting, a scan was performed. The curve obtained during the melting was decreasing and approximately linear and increased rapidly after completion of the melting. The slope measurement of the decreasing part of the curve makes it possible to evaluate the specimen's thermal conductivity:

\[
\frac{q}{\Delta T} = S
\]  

(7)

where \(S\) is the slope of the linear side of the melting curve. The thermal resistance \(R_t\) to heat flow \(q\) through the specimen under the difference in temperature between the heater and the melting indium can be expressed as

\[
R_t = \frac{1}{S}
\]  

(8)

The thermal resistance of the specimen is determined by the difference between the measurement with the Indium at the top and without as

\[
R_t = R_t' - R_t
\]  

(9)

where, \(R_t\) is the thermal resistance of calorimeter and sensor material, and \(R_t'\) is the thermal resistance of calorimeter and sensor material with an Indium layer on top of the specimen (Fig. 6). Compared to the
sensor’s thermal resistance and the crucible, the total thermal resistance obtained yields directly the thermal conductivity (k) of the specimen as:

\[ k = \frac{L}{AR_s} \]

where, \( A \) is the apparent cross-sectional area of the sintered specimen, and \( L \) is the specimen length. For the thermal conductivity measurement of porous materials, 2 × 2 mm\(^2\) specimens were prepared and the diameter of the sensor specimen (indium) was 1.9 mm. Similarly, a crucible was filled with metal powder and the reference material was partly sunk in it and the thermal resistance between the metal and the crucible from the DSC melting peak slope was obtained. This approach was successfully used in metal powder characterization in [38].

### 3. Results and discussion

In this section, we first present the laser material procession diagrams to determine a suitable process parameter range within which laser sintering of stainless steel powder should yield a desired porous layer (Subsection 3.1). This follows from the results of experiments to identify the effect of laser power and pulse duration on the microstructures and porosity/pore size of sintered layers (Subsection 3.2). Then, the thermal analysis is provided for both the metal powder and manufactured specimens (Subsection 3.3).

#### 3.1. Laser material procession diagrams

To characterize the sintered single-layers, they were manually extracted from the powder bed. In relation with our results, published data in the literature [34] is compared as a laser processing diagram in Fig. 4 on logarithmic axes of \( P^* \) and \( v^* \). According to Fig. 4, the nature of laser processing is categorized as heating, melting and vaporization (sketched boundaries). Table 3 shows the combination of parameters for which built specimen could not successfully be lifted from the bed. Interestingly, we observed that failed single layers are in the ‘only’ heating region or ‘only’ melting region. Thus, the powder sintering/melting mechanisms corresponding to the process diagram are defined as follows:

1. No sintering/no melting – failed specimen (Fig. 7a), ‘only’ heating region in Fig. 4: the delivered laser energy is insufficient, resulting in either non-fused (melted) specimens or poor specimens due to reduced necking that broke when lifted from the bed. This occurs at a (constant) low laser power, \( P = 20 \text{ W} \) which is equivalent to \( P^* = 3.2 \), and applying a lower pulse duration, \( \tau < 3 \text{ ms} \) which is equivalent to \( v^* < 3.3 \).  

2. Sintering/partial melting – desired layers (Fig. 7c), common heating and melting regions in Fig. 4: (i) at a (constant) low laser power, \( P = 20 \text{ W} \) which is equivalent to \( P^* = 3 \), and applying a higher pulse duration, \( \tau \geq 3 \text{ ms} \) which is equivalent to \( v^* < 3.3 \); or (ii) at \( 20 \text{ W} \leq P \leq 40 \text{ W} \) which is equivalent to \( 4 \leq P^* \leq 8 \), and applying a pulse duration between 2 and 4 ms, which is equivalent to \( 2 \leq v^* \leq 5 \), allows more time for heat to be absorbed compared to the case of lower pulse duration.

3. Melting – failed specimens (Fig. 7b), ‘only’ melting region in Fig. 4: at a lower pulse duration \( \tau < 2 \text{ ms} \) which is equivalent to \( v^* > 5 \), and a higher laser power \( P > 40 \text{ W} \) which is equivalent to \( P^* > 8 \), results in an excessive (partial) melting which was not appropriate as it was almost impossible to extract specimens.

In summary, it was clearly found that a minimum fluence to fabricate a consistent specimen is in a range between 9.5 and 44.5 J/cm\(^2\). At low fluence, higher pulse duration is preferable, e.g., fluence of 9.5 J/cm\(^2\) (laser power of 20 W and \( \tau = 3 \text{ ms} \) as observed in Fig. 7c) or fluence of 44.5 J/cm\(^2\) (laser power of 40 W and \( \tau = 2 \text{ ms} \)) to have a consistent specimen. Further discussion on microstructure evaluation is presented in Subsection 3.2.
Table 3

Combination of parameters resulting in failed specimens.

| Fluence | Pulse Duration | Interaction Time |
|---------|----------------|------------------|
| 2.4 J/cm² | 0.75 ms | 0.3 ms |
| 4.0 J/cm² | 1.25 ms | 0.75 ms |
| 6.3 J/cm² | 2.0 ms | 1.4 ms |
| 12.7 J/cm² | 10.0 ms | 80 ms |
| 12.7 J/cm² | 10.0 ms | 80 ms |
| 12.7 J/cm² | 10.0 ms | 0.89 ms |

3.2. Microstructure, porosity level and pore size evaluation

The first set of experiments allows the comparison of properties obtained from the successfully removed sintered single layer formation at a constant laser power (20 W). SEM micrographs of the surface of sintered layers obtained by using different pulse durations are shown in Fig. 8. Porosity created by particle sintering can be observed in all the specimen surfaces, the pore size is dependent on the pulse duration. SEM analysis reveals that in none of the specimens the sintering is uniform and therefore the pores have an unusual size and morphology compared to traditional sintering.

In the range of cases included in this study, the pulse exposure period ranges between 3 and 14 ms while the radial thermal diffusion time (\(R^2/\alpha\)) for a \((0.5–1) \times 10^{-6} \text{ m}^2/\text{s}\) thermal diffusivity (\(\alpha\)) powder bed [39] is about 40–80 ms. Thus, based on these time scales, during the interaction time the heat flow distance is less than the particle size particularly at low pulse durations, resulting in very rapid heating of the particle surface [31]. Thus, the exposed powder particle temperature can easily exceed the temperature of melting, leading to melting of the particles rather than necking as evidenced in Fig. 8.

At a low pulse duration, from 3 to 9 ms when fluence values are equivalent or over 9.5 J/cm² a 'balling' effect of molten stainless steel powder is observed. Because of insufficient fluence, the presence of such a balling area is characterized by the agglomeration of a collection of ball-like particles to form large melt pools [40,41]. In the specific range of 9.5–28.6 J/cm², molten material tends to form a ball. At a pulse duration of 9 ms balling formation finds its minimum amount among all the procedures.

The layer thickness (\(L\)) of specimens is measured (see Fig. 9) and plotted in Fig. 10 as a function of pulse duration (and fluence). The layer thickness decreases with increasing pulse duration from 6 to 9 ms. Due to the formation of balling and lumps of partially melted particles, the large layer thickness in specimens at a low pulse duration is observed (see Fig. 8). It varies between 159 and 165 \(\mu\)m by increasing pulse duration.

As expected, increasing the pulse duration, i.e., higher fluence, results in a larger melting volume of powder. It was observed that neighbouring particles combined during the sintering process and partially melted particles are clearly visible. With increasing pulse duration to 10 ms, the specimen surface has a smoother porous appearance and larger connecting features than what was observed at shorter pulse duration, as observed in Fig. 8.

To compare the present results with literature, a dimensionless exposure time (\(\tau^*\)) is considered and defined according to \(\tau^* = cR^2/\alpha\) to characterize laser-sintered microstructures. Accordingly, we observed fewer sintering necks between particles or insufficiently bonded results at \(\tau^* < 0.3\), balling formation at \(0.3 < \tau^* < 1\) while at \(1 < \tau^* < 1.4\), the energy input is more evenly distributed causing the formation of a uniformly melted sintered stainless-steel surface. This agrees with reported results in [42,43], in which a laser sintered 316L stainless steel structure was evaluated. In [42] a balling formation was observed at \(\tau^* < 1\) while a uniformly melted surface at \(1 < \tau^* < 2\) when using a laser scan speed between 20 and 40 m/s and a laser power of 100 W. Stašić and Božič [43] observed balling formation for shorter pulse duration (\(\tau^* = 1\)) under laser power of 70 W. Based on microstructural characterization, we conclude that a minimum fluence of 9.5 J/cm² is necessary to achieve necking/partial melting of powder particles - however, if balling formation is considered - a fluence above ~28.6 J/cm² is necessary for obtaining a smoother sintered layer.

Fig. 11 shows the effect of fluence on the porosity determined by either of the three methods available: the volumetric measurement, Archimedes method and SEM image analysis. It is evident that an increase in pulse duration results in a decrease in porosity, due to the increasing degree of melting. Fewer pores are evident after 7 ms pulse durations at fixed power of 20 W. The plot in Fig. 11 shows a downward trend – a negative linear relationship – when the energetic input...
increases above 19.1 J/cm². Between 9.5 J/cm² and 22.2 J/cm² there is a porosity variation of only 1%. However, when fluence increases from 22.2 J/cm² to 28.6 J/cm², a noticeable 9% decrease in porosity is observed. With increasing pulse duration, the porosity decreases with a maximum porosity of ~50% (determined by the volumetric measurement) obtained at a fluence of 12.7 J/cm².

The porosity measurement results by volumetric measurement, Archimedes method and image processing show an identical trend. For the volumetric approach, Archimedes method and image processing trend lines were fitted with linear regression yielding R² values of 0.95, 0.88 and 0.96, respectively. The image analysis method consistently recorded a lower value compared to the volumetric measurement and Archimedes method. The porosities as determined by the Archimedes method are 13% (average) lower than the direct mass measurements and 13% (average) higher than those determined by image analysis. The possible reasons for this difference are discussed next.

A difference between the volumetric and Archimedes porosity measurements may highlight that during the saturation, not all of the air was replaced by liquid. A difference between Archimedes method and image analysis may arise due to fact that an image analysis can reflect the effect of un-melted powder on the results of porosity, as the cavities may contain un-melted powder. These cavities can especially show a significant influence on the results of structures with a high porosity. The results reported in this study are consistent with data reported in [44–47]. A comparison of the Archimedes method and image analysis showed that for low-porosity parts (porosity of approximately 2%), the differences are within about 1%. However, the Archimedes method shows 8% higher porosity compared to image analysis at a porosity of around 10% [44]. Similarly, Bai et al. [45] observed that the results of Archimedes method and image analysis are similar when the porosity is near 0%, however, the porosity values using the image analysis are obviously lower than those using the Archimedes method for higher porosity specimens. Damon et al. [46] observed higher porosity values recorded by Archimedes method compared to microscopic image analysis by 25% at porosity of around 3%. Thus, from comparison of different porosity measurement approaches, it can be concluded that the results using the three different methods differ from each other to a degree already observed in literature before. The advantage of the image analysis is that more information about the distribution, size and form of pores in the part is determined. The characterization of a porous material by image analysis is however directly influenced by software capabilities. It seems that the Archimedes method determines more reliable results as the whole specimen volume (in 3D) is considered instead of considering just the upper surface (in 2D), which might not be representative for the entire specimen.

It is clear from the SEM image analysis (Fig. 8) that at a higher pulse duration the surface is much smoother and uniform, and the porosity decreases. The average pore-size at different fluences is illustrated in Fig. 12. At different energy inputs, by changing pulse duration, the average pores size is between 22.3 and 29.3 μm from high to low pulse duration. We observed some tunnel like pores that go into the sintered layers. It was also observed that <7% of the pores were between 40 and 50 μm in size.

The porosity is found to be dependent on the pulse duration as well as on the value of porosity in the sintered layer. As evidenced in Fig. 11, at pulse durations of 12–14 ms, approximately 27% of the sintered layers refers to porosity with average pore size of 22–24 μm (Fig. 12), based on image analysis. While at the pulse duration of 3–4 ms approximately 39% of the specimens has a porosity with average pore size of 27–29 μm. It means that the porosity decreases by around 30% and accordingly the pore size decreases by approximately 24%. Thus, a relation is observed to describe the pore size variation with porosity as a function of pulse duration at constant laser power.

The next set of experiments concerns the comparison of the results obtained at constant fluence. The effects of laser power and pulse duration on the layer thickness and porosity are plotted in Fig. 13, for the maximum laser power of 40 W and the pulse duration ranging from 2 to 8 ms. As discussed in Section 2, at laser powers higher than 40 W, the specimen was stuck to the substrate and could not be lifted from the bed for characterization. As evidenced in Fig. 13, the layer thickness clearly increases by increasing laser power (decreasing pulse duration). It is clear that even though fluence was kept constant, the change in laser power and pulse duration has an effect on the layer thickness of the specimens.

Fig. 13 shows a clear rise in porosity with increasing laser power. The overall porosity level is below 61%. A noticeable drop in specimens porosity (17%) is observed from specimen with a P = 40 W and τ = 2 ms to P = 30 W and τ = 2.66 ms. Both are at an identical fluence level (12.7 J/cm²). A specimen with high laser power and low pulse duration achieves higher porosities. This result suggests that fluence is not a good indicator for porosity level of LPBF manufactured porous materials when processing at low laser powers/pulse durations, as also described in [13]. Previously, it was reported that porosity in stainless steel specimens can vary if the laser power or the speed of scanning varies at the same fluences due to, e.g., balling or poor wetting characteristics [48,49].

Lastly, a total of 6 specimens has been produced, extracted and measured using the same settings. These specimens were produced with an energetic input of 12.7 J/cm². The height, area and weight of the specimens have been measured and the porosity has been computed. The results of these measurements are presented in Fig. 14. The porosity and layer thickness of the specimens is on average 50% and 167 μm, respectively.

From the experimental results presented in this section several conclusions can be drawn. Firstly, it is possible to create highly porous (50–60%) materials using printing based on a pulsed LPBF process.
Secondly, fabricating porous materials is repeatable using the current setup, although the structures are random in nature. Thirdly, the porosity can be controlled within a certain bandwidth by changing laser power and pulse duration. The minimum pulse duration is found to be highly important. In this study, we observed a minimum pulse duration of 2 ms and 3 ms at laser power of 40 W and 20 W, respectively, to have a consistent porous material.

3.3. Characterizing metal powder and sintered single-layer specimens in terms of thermal conductivity

For using additively manufactured porous materials in heat transfer devices, it is very important to know its thermal conductivity, and consider the pore/porosity effect on the heat flow. Moreover, the thermal conductivity of powder within a powder bed and the first layer that is printed are essential properties in modeling LPBF processes and to improve process parameters. In this subsection, the thermal conductivity of the printed single-layers is analysed. Table 4 presents the results of measurements of slopes and thermal conductivities obtained for each specimen. Accordingly, thermal conductivity of sintered layers related to fluence and pulse duration is presented in Fig. 15. Each point presents an average of 3 measurement results. The thermal conductivities of specimens vary between 0.37 and 0.73 W/mK according to pulse duration range and it can be clearly observed that the conductivity increases as fluence is increased. This is expected as higher fluence results in a higher sintering temperature and thus better fusion of powder particles. Solid–solid contact regions expand by two mechanisms: sintering and partially melted particles. These regions provide heat conducting pathways to improve thermal conductivity. It can be seen that over the range of pulse duration, thermal conductivity almost doubles (1.95 times) when determined at the pulse duration of 7 and 12 ms. However, the
porosity changes by about 30%, which implies that both morphology and sinter densification changes affect the thermal conductivity. Similar results have been reported in literature for powder bed fusion using electron beam melting [50].

Accordingly, a ratio of thermal conductivity of the powder particle and sintered layers to its bulk thermal conductivity ($k/k_s$) related to porosity is presented in Fig. 16 and compared to available experimental data and correlations in the literature. The thermal conductivity of the sintered layer is found to be 0.38 W/m K which is higher than that of powder particles (non-sintered) (0.12 W/m K) at porosity of approximately 50%. In the literature, there are many models to predict the thermal conductivity of sintered powder particles, within a liquid, gas, or vacuum. Examples of such investigations include the works of Alexander [51] and Hadley [52]. Alexander’s model [51] was developed for

![Fig. 11. The measured porosity of specimens using different approaches at constant laser power of 20 W and varied pulse duration (τ).](image1)

![Fig. 12. The measured pore size of specimens by SEM at constant laser power of 20 W and varied pulse duration.](image2)

![Fig. 13. The measured layer thickness (top) and porosity (bottom) of specimens at constant fluence of 12.7 J/cm².](image3)

![Fig. 14. Porosity and layer thickness results for specimens in the repeatability experiment.](image4)

![Table 4](table)

| Specimen | Porosity (%) | Slope (mW/K) | K (W/m K) |
|----------|--------------|--------------|-----------|
| 1        | 0.46         | 2.30         | 0.37      |
| 2        | 0.41         | 2.48         | 0.44      |
| 3        | 0.37         | 3.00         | 0.55      |
| 4        | 0.32         | 3.95         | 0.73      |
sintered powders and unconsolidated beads, given by the thermal conductivities of the solid \(k_s\) and the fluid phase \(k_f\) as:

\[
k = \frac{k_f (k_s / k_f)^{1 - \varepsilon}}{1 - \varepsilon (k_s / k_f)}
\]  

(11)

In addition, the Maxwell–Eucken lower bound correlations [52], derived for a dilute spherical particle suspension in a uniform liquid as:

\[
k = \frac{2\varepsilon + (k_s / k_f) (3 - 2\varepsilon)}{3 - \varepsilon + (k_s / k_f) \varepsilon}
\]  

(12)

We have compared our experimental data with the previous data on non-sintered powder particles, sintered powder particles and model predictions in the literature. As evidenced in Fig. 16, an excellent agreement is observed for the normalized experimental values of powder particles in this study and Alexander’s model while an over prediction of the Maxwell–Eucken model is observed. Experimental data determined in the present study for a powder particle are in good agreement with the independent experimental results of Rombouts et al. [53], Alkahari et al. [54] and Agapiou and DeVries [55]. Therefore, to estimate the thermal conductivity of a powder bed, Alexander’s model can be proposed.

As evidenced in Fig. 16, the thermal conductivity of a powder particle differs from a sintered layer of the present study and sintered porous materials reported by Biceroglu et al. [56], Thewsey and Zhao [57] and Smith et al. [50]. We observed that the thermal conductivity of a single 316L stainless steel powder particle is \(-0.17\) W/mK, which is close to reported particle thermal conductivity of \(0.156\) W/mK whereas the corresponding bulk material value is \(15\) W/mK [48]. Hence, the thermal conductivity of the powder particle is \(31.5\)% of the sintered layer and the thermal conductivity of the first sintered layer is \(4.8\)% of its bulk material. Similarly, Zhang et al. [58] showed that the Ti64 powder conductivity is approximately \(4-5\)% of the bulk Ti64 conductivity and the results of Inconel 625 powder, conductivity is \(4-7\)% of its bulk material.

In summary, we conclude that:

1. thermal conductivity not only depends on the porosity but also the necking between powder particles;
2. due to the growth of the contact area during sintering, the thermal conductivity of a powder bed changes significantly after high-temperature heating.

Therefore, the powder bed thermal conductivity should be stimulated taking into consideration the powder bed density and fusion of particles at the first layer. This is because of the fact that for a low thermal conductivity, heat in the first layer(s) cannot be easily transmitted to the underlying layers/material. Hence, the exposed powder particle temperature reaches the melting temperature faster. An incremental change in temperature results in material evaporation. Our findings with regards to thermal conductivity of a first layer would help to better understanding and simulating of the complex melt pool physics in LPBF.

4. Summary and conclusions

In this paper, a single layer of sintered porous material was manufactured using a lab-scale setup, considering laser power and pulse duration. 316L stainless steel powder was used as powder...
The thermal conductivity of sintered layers and powder particles were also evaluated. The main results obtained are as follows:

- An excellent model was observed for the experimental data, and the thermal conductivity values of powder particles and sintered layers were characterized in terms of porosity, pore size, and feedstock material. The formed microstructures were discussed and also 13% higher than those determined by image analysis in references.

- The porosity decreases by around 30% and the pore size distribution widens with increased pulse duration. A relation was also obtained from high to low pulse duration and the pore size distribution as determined by the Archimedes method.

- The porosity as determined by the Archimedes method is 13% lower than those determined by direct mass measurements of heat pipes made by additive manufacturing. 2017 23rd International Workshop on Thermal Investigation of Cryo- and System Technologies (THERMICA 2017). 2020, (Pisa, Italy).

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