Effect of Particle size of monomodal 316L powder on powder layer density in powder bed fusion

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Abstract

Powder layer density is an important measure for understanding the effect of powder on part quality in powder bed fusion. The density of thin layers, as they are deposited in powder bed fusion, differs from the density of powder in large containers. This study investigates this difference. Therefore, six monomodal powders with different particle size distributions, from coarse to fine, are spread in an 84.5 µm deep cavity to determine their powder layer densities for a single layer. A linear dependence of powder layer density on the D50 of powder is discovered for monomodal powders with good flowability. This dependence can be explained by the wall effect. Fine powders with low flowability show an increase in the standard deviation of the powder layer density. These findings suggest the existence of a particle size distribution that is sufficiently small to minimize the wall effect in a thin layer while still being sufficiently large to guarantee a good flowability of the powder.

Keywords LPBF · Powder layer density · Powder · Additive manufacturing · Laser powder bed fusion · Powder layer

1 Introduction

Powder significantly affects part quality in powder fusion bed (PBF) of metals [1, 2]. Meiners [3] demonstrated the effect of the particle size distribution (PSD) on part density, which was then confirmed in later studies [4, 5]. However, the effect of powder on final part properties is yet to be elucidated. A link between powder and part properties is the powder layer, most notably the powder layer density (PLD), as emphasized in several studies [2, 6–8]. The most frequently published method to measure powder bed density is to build cavities with a height of several powder layers during PBF and then measure the powder density in these cavities [6, 9–13].

However, Chen et al. [14], Wischeropp et al. [15], and Mahmoodkhani et al. [1] showed that the true PLD and the

effective thickness of a single powder layer differ from the values obtained by measuring several layers at once. Wischeropp et al. [15] measured relative powder layer densities, as a fraction of the solid material density, to be between 0.44 and 0.56 in an experiment on a PBF machine for the powders and layer heights listed in Table 1.

Chen et al. [14] investigated nine different powders, each at three different layer thicknesses, both experimentally and in a discrete element simulation. They obtained powder layer densities between 0.25 and 0.45 for layer thicknesses between 60 and 180 µm, for which they showed a decrease in the PLD for a decreasing layer height. Chen et al. [14] identified three effects that dominate the powder spreading process and affect the density of a single powder layer: the cohesion, wall, and percolation effect. Mindt et al. [16] numerically simulated the powder spreading process for PBF in a discrete element simulation. They numerically determined that the packing density for Ti6Al4V powder with particles between 15 and 75 µm in diameter for effective layer heights of 30 and 50 µm is 0.2 and 0.38, respectively. Muñiz-Lerma et al. [17] investigated the PLD of the three powders listed in Table 2. They spread a powder layer that was approximately 100 µm thick using a doctor blade and performed a computed tomography scan of the spread

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Table 1 PLD results of Wischeropp et al. [15]

	Powder 1, Build cycle 1	Powder 1, Build cycle 2	Powder 2	Powder 3
Alloy	SS 17-4 PH	SS 17-4 PH	Ti6Al4V	Ti6Al4V
PSD, D10–D90 (μm)	20–45	20–45	35–46	28–48
Leveling height of platform (μm)	30	50	30	30
Average powder layer height (μm)	165	225	132	130
Average relative PLD (–)	0.44	0.49	0.56	0.53

Table 2 PLD results of Muñiz-Lerma et al. [17]

	Powder A	Powder B	Powder C
Alloy	AlSi7Mg	AlSi7Mg	AlSi7Mg
D10, D50, D90 (μm)	48, 63, 83	54, 70, 91	14, 31, 58
Particle shape	Spherical	Spherical	Not spherical
powder layer height (as stated) (μm)	~ 100	~ 100	~ 100
Relative PLD (–)	0.547	0.536	0.414

powder. The measured relative densities were between 0.41 and 0.55.

Chen et al. [14] showed that coarser powders and smaller layer thicknesses result in lower powder layer densities; however, this relationship was discovered and quantified for only a few powders. Therefore, this study further investigates how PLD is affected by the PSD. In addition, a novel approach for measuring the PLD of a single powder layer is presented herein. In contrast to previously published methods, the PLD is not measured on a machine but ex-situ on a test bench in a cavity.

2 Materials and methods

2.1 Powder characterization methods and powders

All powders were characterized before their PLD was measured. The PSD was measured via laser diffraction using an LS230 laser diffractometer from Beckman Coulter. The particle circularity was measured using a Leica DM6 light microscope from Leica Microsystems GmbH. Images were captured via transmitted light microscopy and then processed in MATLAB, where all agglomerates were manually

removed until 3000 particles per powder were identified. The circularity f_{circ} was then calculated according to the formula used by Bouwman [18] as

$$f_{\text{circ}} = \frac{4 \times \pi \times A}{P^2}, \quad (1)$$

where A is the area and P perimeter of the projected particle. The apparent and tap densities of each powder were measured three times according to ASTM B417-18 [19] and ASTM B527-15 [20], respectively. Subsequently, the Hausner ratios were calculated as the ratios of the tap to apparent density. The powder flowability was also measured using a REVOLUTION powder analyzer by Mercury Scientific Inc. The avalanche angle and surface fractal were measured according to the procedure described by Spierings et al. [21].

Six monomodal, gas atomized, 316L-powders with approximately the same distribution width (D90–D10), high circularity, and different D50 (10–60 μm) were used. The PSD and distribution width of all powders are shown in Table 3. The scanning electron microscopy images of the powders are shown in Fig. 1. The designation of all powders was selected to reflect their D50.

2.2 Layer density measurement

The PLD was measured by filling the cavity shown in Fig. 2a using the coating blade shown in Fig. 2b. Subsequently, the density was calculated from the powder mass and the known volume of the cavity. Coating blades from softer materials, e.g. rubber, cannot be used in this setup as they deform during coating and deliver inconsistent results. The cavity was made from hardened 1.2379 steel with an outer cylinder diameter of 44.0 mm, a total height of 15.2 mm, and a mass of 133.993 g. The inner cavity with a diameter of 40 mm was

Table 3 D10, D50, D90 and distribution-width (D90–D10) of P10–P60, volume fraction

	P10	P20	P30	P40	P50	P60
D10 (μm)	4.0	13.6	21.9	31.2	37.8	50.5
D50 (μm)	9.5	20.2	28.3	37.9	46.7	59.3
D90 (μm)	19.3	29.7	35.8	47.3	57.3	71.9
D90–D10 (μm)	15.2	16.1	13.8	16.1	19.5	21.4

Fig. 1 Scanning electron microscopy images of **a** P10, **b** P20, **c** P30, **d** P40, **e** P50, and **f** P60

Fig. 2 Cavity **(a)** and coating blade **(b)** made from steel

created via electrical discharge machining; its average depth was $84.5 \mu\text{m}$ and its bottom surface roughness $R_a = 5.09 \mu\text{m}$, as measured using a GelSight Benchtop system by GelSight Inc. The volume of the cavity was 106.2 mm^3 , as calculated using the diameter and average depth.

The resulting top surface roughness of the cavity, $R_a = 5.09 \mu\text{m}$, is approximately the roughness of a

PBF surface, which ranges from $R_a = 4.25 \mu\text{m}$ for 316L [22], $R_a = 4.25 \mu\text{m}$ and $R_a = 15 \mu\text{m}$ for 1.2709 [23], and $R_a = 20.925 \mu\text{m}$ for AISi10Mg [24]. Lower end values of effective layer thicknesses reported in the literature were selected for the cavity depth such that the wall effect, as described in [25], for the deposition of thin powder layers becomes more pronounced.

The cavity was coated on a test bench, which was propelled with a toothed belt drive on the coater axis and a spindle screw drive on the z-axis. The cavity was placed on the build platform and leveled such that the tip of the coating blade lightly touched the rim of the cavity along the entire movement on top of the cavity. Subsequently, the build platform was raised by 50 μm to create pressure between the coating blade and the cavity rim. This pressure ensured that no powder remained on the cavity rim after coating. The position of the cavity in coating direction was secured using two mechanical stops on the build platform.

The PLD of all powders was measured five times for each powder. Therefore, the coating blade was moved to its starting position in front of the build platform. The cavity was then placed on the build platform and the powder placement platform was placed around the cavity, as shown in Fig. 3. Powder with a volume of 12 cm^3 at apparent density was positioned in front of the cavity on the powder placement platform. Subsequently, the coater was moved across the cavity at a speed of 100 mm/s. Finally, the cavity was removed, cleaned from the outside, and weighed on an AX205 Delta Range scale (Mettler Toledo).

3 Results

The PLD is affected by the particle shape [17]. Therefore, the particle circularity was measured, and the values for all powders are shown in Table 4. The finer powders have a slightly higher circularity than the coarse powders;

however, the difference is insignificant and will be disregarded hereinafter.

The apparent density, tap density, and Hausner ratio of all powders are shown in Fig. 4. For P30–P60, those values are similar. However, P10 and P20 exhibit a decrease in apparent density, an increase in tap density, and a resulting increase in Hausner ratio for a decreasing D50.

Figure 5 shows the avalanche angle and surface fractal measured using the REVOLUTION powder analyzer. The mean and standard deviation of the avalanche angle are similar for P40–P60 and increase with decreasing particle diameter. Meanwhile, the mean and standard deviation of the surface fractal are similar for P30–P60. Both P10 and P20 indicate a higher standard deviation of the surface fractal; however, only P10 indicates a higher mean surface fractal than the other powders. This dependency of the Hausner ratio, avalanche angle, and surface fractal on the D50 is caused by interparticle forces, primarily the Van der Waals forces [14], which decrease the powder flowability.

The PLD of all powders is shown in Fig. 6, where the relative PLD is plotted over the ratio of the D50 of a powder to the cavity depth $h = 84.5 \mu\text{m}$. The data show the dependency of the PLD on the D50 of a powder as the PLD increases with decreasing D50. Furthermore, the results indicate that P30–P60 exhibit a relatively low standard deviation of the PLD, whereas P10 and P20 exhibit a relatively large standard deviation.

Because the data in Fig. 6 show a linear dependency, a least-squares regression was fitted to the data to quantify the dependency and determine the goodness-of-fit to a

Fig. 3 **a** Powder (12 cm^3) placed in front of the cavity before the coating process; **b** powder in the cavity after the coating process

Table 4 Circularity of P10–P60

	P10	P20	P30	P40	P50	P60
Circularity, mean (–)	0.95	0.90	0.90	0.89	0.84	0.88
Circularity, SD (–)	0.07	0.09	0.10	0.11	0.14	0.11

Fig. 4 Mean and SD of Hausner ratio, tap density, and apparent density of P10–P60 **Fig. 5** Mean and SD of avalanche angle and surface fractal of P10–P60 **Fig. 6** Mean and SD of PLD of P10–P60 with least-squares linear regression (LR) line for P30–P60 from Eq. 2

linear model. The homoscedasticity of the data was verified, as it is a precondition for linear regression. Levene's test for equal variance shows that the PLD data of P10–P60 ($p < 0.001$) and P20–P60 ($p = 0.024$) are not homoscedastic. The same test indicates homoscedasticity for a dataset comprising the densities of P30–P60 ($p = 0.424$). Therefore, the least-squares linear regression in Eq. 2 was fitted to the density data of P30–P60 as

$$\rho_{pl} = \rho_0 - \Phi_{reg} \left(\frac{d}{h} \right) = 0.398 - 0.186 \left(\frac{d}{h} \right), \quad (2)$$

where ρ_{pl} (between 0 and 1) is the relative PLD as a fraction of the solid material density, ρ_0 the intersection of the regression line with the y-axis, Φ_{reg} the particle size-dependent powder layer porosity, d the particle diameter (in this case approximated with the D50), and $h = 84.5 \mu\text{m}$ the cavity depth. The regression shows a good fit with the coefficient of determination R^2 of 0.951. The high R^2 indicates a linear relationship between the D50 and PLD of powders P30–P60.

4 Discussion

4.1 Wall effect

All the measured PLDs are lower than the apparent densities and within the range of experimental values reported in the literature for a single powder layer: 0.44–0.56 [15]; 0.25–0.45 [14]. Furthermore, the PLD decreases with the increasing ratio of the particle diameter to the layer thickness. The dependency of the layer density on the particle diameter can be explained by the wall effect, which is the reduction of packing density through vacant sites in a packed powder in the presence of a wall, as illustrated in Fig. 7.

Fig. 7 Wall effect of a thin powder layer between two walls for face-centered cubic packing; lost particle volume shown by dashed semi-circles

The wall effect for a single powder layer in PBF is often discussed [5, 14, 15, 26]; however, only one analytical estimation has been reported, i.e., that by Karapatis et al. [25]. Karapatis approximated the wall effect based on an orthorhombic arrangement with a bulk porosity $\Phi_{\text{bulk}} = 0.4$ of monodisperse particles with a diameter d in a disk layer of diameter D and height h as

$$\frac{\Phi_{\text{wall}}}{\Phi_{\text{total}}} = \frac{\frac{1}{6}\pi^2 dD\left(\frac{D}{4} + h\right)}{0.4\pi\frac{D^2}{4}h + \frac{1}{6}\pi^2 dD\left(\frac{D}{4} + h\right)} \approx \frac{d\left(\frac{1}{h} + \frac{4}{D}\right)}{0.8 + d\left(\frac{1}{h} + \frac{4}{D}\right)}, \quad (3)$$

where Φ_{wall} is the layer porosity due to the wall effect and $\Phi_{\text{total}} = \Phi_{\text{bulk}} + \Phi_{\text{wall}}$ is the total porosity of the powder layer. This equation was simplified by Chen et al. [14] for $D \gg d$ as

$$\frac{\Phi_{\text{wall}}}{\Phi_{\text{total}}} = \frac{1}{0.8\frac{h}{d} + 1}. \quad (4)$$

If Φ_{total} is substituted with $\Phi_{\text{total}} = \Phi_{\text{bulk}} + \Phi_{\text{wall}}$ = $0.4 + \Phi_{\text{wall}}$, the layer porosity due to the wall effect can be estimated as

$$\Phi_{\text{wall}} = 0.5\left(\frac{d}{h}\right). \quad (5)$$

This value overestimates the measured wall effect Φ_{reg} of the linear regression in Eq. 2 by more than a factor of 2.5. This is because several assumptions of the Karapatis model are violated. The particles in the experiment were not arranged in an orthorhombic packing. Furthermore, polydisperse particles were used for the experiment, and the equation was simplified. However, the model predicts a linear dependency of the wall effect based on the ratio of the particle diameter to layer thickness, as observed in the experiments. The cause of linearity in the experiment is yet to be elucidated.

The numerical results of the layer densities and the contribution of the wall effect during the experiment must be

Fig. 8 SD of P10–P60 plotted against mean Hausner ratio

handled with caution, as the cavity depth of $h = 84.5 \mu\text{m}$ is lower than the effective layer thickness in PBF, which was measured to be more than $150 \mu\text{m}$ in some cases [1, 15].

4.2 Cohesion of powder

The Hausner ratio is a widely used indicator of the flowability of powders [21]. Spierings et al. [21] proposed the avalanche angle and surface fractal as flowability indicators for powders in PBF. Figures 4 and 5 show that the flowability, measured using the Hausner ratio, avalanche angle, and surface fractal, increases with the particle diameter for P10 and P20, whereas the improvement in flowability is only slight for P30–P60. This finding is consistent with that of Abdullah and Geldart [27], who reported that flowability of powder generally increases with the particle size up to a critical range above which the flowability does not show any improvement. The relationship between flowability of powder and particle size is mainly governed by interparticle forces [28], of which the Van der Waals forces dominate the deposition of powder in a powder bed [29].

The effect of the interparticle forces measured by the Hausner ratio on the PLD is shown in Fig. 8, where the Hausner ratio is plotted against the standard deviation of the PLD for all six powders. The standard deviation of the PLD of P10 and P20 increases as the flowability of the powders decreases. All the other powders, with a Hausner ratio below 1.15, exhibit similar standard deviations of the PLD. This effect is qualitatively similar for the avalanche angle and surface fractal; therefore, it is omitted herein.

Similar powder behaviors have been reported previously. Spierings [30] observed that for powders with a low flowability, the layer became less smooth and agglomerates tended to form in the layer. These findings are consistent with the results of Ma et al. [29], who demonstrated that powder flowability decreased consistently with the increase in the fraction of fine powder until the particles agglomerated and

were pushed across the powder bed instead of spreading out. These agglomerates are likely responsible for the effect shown in Fig. 8. Chen et al. [14] termed this effect as the cohesion effect, which denotes the reduction in the powder layer packing density owing to cohesive forces, e.g., van der Waals forces, which impede the spreading process.

5 Conclusion and outlook

In this study, a cavity was used to measure the density of a single powder layer for monomodal, spherical powders. The results indicate that the density of a single powder layer is significantly smaller than the apparent density and is governed by the wall effect and powder cohesion.

The linear relationship between the PLD and D50 of monomodal powders with a D50 of 30 μm and above show that fine powders result in higher powder layer densities in PBF. The increased standard deviation of the PLD for monomodal powders with a D50 of 20 μm and below indicates the existence of a threshold for the PSD, below which the spreadability is impeded. The Hausner ratio, avalanche angle, and surface fractal are suitable indicators for this effect. The cause of the linear dependency of the PLD on the PSD is still unclear; hence, further research is necessitated. Other influences, such as coating material, coater geometry, and coater speed, were not investigated in this study and will be examined with a modified setup for other recoating technologies in the future.

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Data availability All Data are available.

Compliance with ethical standards

Conflict of interest The authors declare that there is no conflict of interest regarding the publication of this article.

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