

OPTIMIZATION OF POWDER LAYER DENSITY IN SELECTIVE LASER SINTERING

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Abstract

An important parameter for the overall quality of SLS parts is the density of powder layers before sintering. Previous studies have shown that the control of powder particle shape and size distribution can increase the density of non-packed powder beds. However, these studies concerned beds several orders of magnitude larger than the SLS layers. The purpose of this study is to determine if, and to what extent, the density of thin powder layers can be increased. Experiments show that the density of thin layers increases from 53% to 63% when adding 30% fine powder to the coarse powder, with a coarse-to-fine ratio of 1:10. Compared with the bulk experiments, this density improvement method is less efficient, because the particles do not arrange as efficiently, and the wall effects can become predominant.

1. INTRODUCTION

Metallic components made by selective laser sintering (SLS) for tooling applications generally exhibit high porosity and low surface quality that still impede a widespread application [1]. Two different SLS processes can be distinguished: the indirect and the direct sintering techniques. In the first case, the material is constituted of metallic particles (typically tool steel) coated with a specific polymer [1, 2]; the energy brought by the laser locally “remelts” the polymer, which then “glues” the metallic particles together. The polymer binder must be thermally removed. The obtained product is neither dense nor fully consolidated yet, and a third operation is necessary: the post-sintering, which, together with an infiltration with a lower melting point metal (e.g. copper), leads to the final part. Post-processing operations are required, which inevitably lead to an increased throughput time. Furthermore, due to the various thermally based operations, residual stresses and/or loss of accuracy are to be expected.

In the direct selective laser sintering route [1, 3], no post-processing is, theoretically, necessary. The material is a fully metallic powder which can contain two components: in that case, liquid phase sintering is used as the densification process. However, a single-component powder can also be selectively sintered, provided that only a fractional part of the powder is remelted under the laser. The molten phase propagates through the solid particles bed (which is in fact a thin layer), and the wetting properties of the liquid-solid couple lead to powder densification. Since the interaction times are usually very short in SLS processes compared to conventional liquid phase sintering (a few hundreds of milliseconds vs. several minutes), rearrangement is essentially the only operative densification process. After processing, the part density and surface quality are usually poor [4] and an infiltration (e.g. epoxy resin) is still required. However, the number of operations is limited and the thermal influence on the sintered components is reduced to a minimum.

In both SLS techniques, an extensive number of parameters control the quality of the sintered parts. Among them, the material properties play a major role: in particular, the characteristics of the powder bed before sintering are decisive for the quality of the finished component. More specifically, the relative density of the bed has a direct influence on the density of the sintered body. Therefore, powder materials and deposition process must be improved to insure the highest possible layer density before laser sintering.

2. THEORETICAL APPROACH

2.1. General considerations

Increasing the density of a powder requires the understanding of the mechanisms that dominate during particle motions. Such interactions have been described in detail in many other papers, such as [5]. A few simulation studies have been performed to reproduce these motions, and to identify and understand the phenomena [6, 7]. Basically, the most important factors are found among the particle shape and surface state, and the granulometry parameters of the powder.

The density of powder bulks can be increased by several methods [8]. A first approach is the optimization of the particle shape and surface state: smooth particles will move easily within the bed and will lead to a higher loose density. In addition, clean and dry surfaces prevent the formation of unwanted bonds between particles, so that particle motion is not prevented [5]. The shape of the particles will further influence the density of a powder: regular, equiaxed particles tend to arrange more efficiently (Figure 1); therefore, spherical grains are often preferred when available. Non-porous particles are evidently favored.

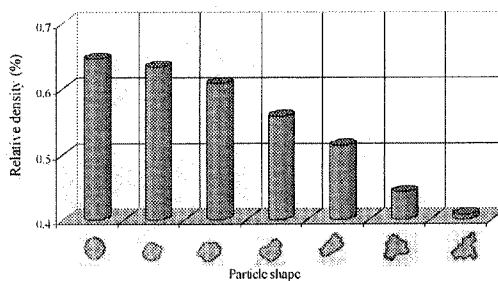


Figure 1: effect of particle shape on the achievable relative density (after R. M. German [8])

In general, the optimization approaches described above are closely related to the powder material and its production process. For instance, hard materials are generally manufactured by milling, producing angular, irregular particles, while most gas-atomized powders exhibit equiaxed, smooth spherical particles. Therefore, for a given material, there is usually little flexibility for modifying and optimizing the particle shape and quality.

The granulometry of a powder, which is not directly related to a selected material is an important parameter for density optimization. In a monomodal, monodisperse granulometry distribution, the particles will assume a stable position with the highest possible density, if sufficient energy is brought in for rearrangement. However, it is evident that a significant number of voids exist between particles: for a compact packing (fcc), the relative density is about 74%. In a monomodal but polydisperse particle size distribution, with a given peak width, some of the particles on the low diameter side of the peak can fill the holes left between the coarser particles [8]. The maximum density can be obtained when the particle size distribution follows Anderson's equation:

$$W = A \cdot D^q$$

where W is the cumulative weight fraction, D is the class diameter, A and q are empirical constants. The best results are obtained for values of q between 0.5 and 0.67, corresponding to a large fraction of fine particles ("fines") [8].

Although not fully efficient, the peak width method can increase the density of a powder, in a relatively straightforward way. A more effective means would be to determine the size or size distribution of the available voids in a starting, coarse powder and then to add suitable fine particles to fill them (multimodal granulometry). Evidently, an energy input into the system, e.g. by vibration, can further increase the final density. Further improvements can be accomplished by adding finer particles with suitable size ratios to fill in the remaining voids.

2.2. Previous studies

McGeary [9] applied the multi-modal approach to powder bulks. His experimental setup was composed of vertical transparent cylinders and vibratory equipment. The test procedure consisted in pouring a coarse powder into the tank, vibrating it until no more volume variation was noticeable; then, the first finer powder was poured, vibrated and the process was repeated for the subsequent fine fractions. The experiments were performed with various materials and powder absolute sizes, but always with particles as spherical as possible and with narrow size distributions. Blends with different size ratios and fines content were prepared and the densities were measured. Before the addition of the fine powder, McGeary observed that the coarse particles assume an arrangement close to orthorhombic.

The results showed that a size ratio about 1:7 with about 30 % of fine particles constitutes the approximate optimum achievable density increase. Higher size ratios do not present significant improvements and are usually less favored because of availability and handling problems.

The determination of the void size, and thus of the ideal coarse-to-fine ratio can be made on the basis of an orthorhombic arrangement. Two types of voids can be identified in this particular packing configuration: a triangular pore and a "square", larger passage. Since the compaction is made by allowing the fine particles to percolate through the bed of the coarse particles, the limiting

factor is the smaller diameter. The value of the small particle radius r can be determined geometrically; it is given by the relation:

$$r = \left(\frac{2}{\sqrt{3}} - 1 \right) \cdot R \cong \frac{1}{7} R$$

which is in good agreement with the experimentally determined value.

The optimum amount of fine particles X_{fines} to be added to the coarse is determined by:

$$X_{\text{fines}} = 1 - \frac{\rho_{\text{coarse}}}{\rho^*} = 1 - \frac{\rho_{\text{coarse}}}{\rho_{\text{coarse}} + (1 - \rho_{\text{coarse}}) \cdot \rho_{\text{fines}}},$$

where ρ_{coarse} is the relative density of the coarse particles and ρ_{fines} the relative density of the fine particles arrangement. Taking a relative density of about 60 % for both coarse and fine powders, the composition should be about 30 % of fine particles (with a 1:7 ratio with the coarse particles). The maximum density achievable with such a composition is $\rho^* \approx 0.84$.

The equations above can be easily extended to a larger number of size mixtures. This way, a second fine fraction with a size ratio 1:49 can be added to further improve the density. However, finer particles induce handling problems (due to aggregation). They may also be difficult to obtain. Both last equations admit implicitly that the whole space between the coarse particles can be filled with fines. However, the calculated maximum density is consistent with the data obtained by McGeary [9]. Although this study does not consider a void size distribution, it has been shown that adding 30 % of fine particles, with a size ratio of 1:7, can increase the density of a powder bulk by about 30 %.

A remarkable side conclusion of McGeary's study is that the ideal ratio and the achievable density do not depend on the particles absolute size. This is probably true only for particles larger than ca. 1 μm , i.e. such that they are not influenced by electrostatic, Van der Waals or other joining forces.

McGeary's work has been extended by B. M. Zivanovic [10] to the compaction of monodisperse, but non-spherical particles. Basically, the same results are obtained: a similar density increase can be expected, although the values of relative density are lower than those obtained with spherical particles. In this case however, the size ratio is higher than 1:7 (over 1:10), since the passages follow a size distribution. Increasing the size ratio may lead to better results due to an enhanced percolation through the coarse bed, but fine particles will fill a given volume less efficiently than a single, dense particle. Another study on particle arrangement by simulation was presented by G. T. Nolan and P. E. Kavanagh [11], considering a random arrangement of log-normal distributed particles. The influence of the peak width on the void size, number and distribution was studied: when the peak width increases, the voids size expands, their number decreases and the apparent density increases.

2.3. Application to Selective Laser Sintering

The studies presented above are valid for powder bulks, i. e. powder particles arranged in containers several orders of magnitude larger than an average particle size. In Selective Laser Sintering, the situation is drastically different. Although particles can be very spherical, their granulometry is usually not ideal (in terms of size ratios). Most importantly, the dimensional characteristics of the “container” (the powder layer) are much closer to the size of the particles: 50 μm layers can be prepared with a 30 μm mode size powder.

Therefore, “wall effects” (Figure 2) [12] will be more relevant in such layers, inducing lower densities. The wall effects can be estimated with the relation:

$$V_{\text{walls}} \approx N_s \cdot V_s \cdot S_w$$

where N_s is the number of vacant sites per unit area, V_s is the void volume per vacant site and S_w is the total wall surface area.

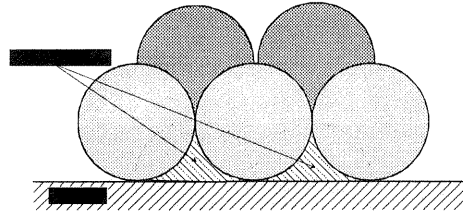


Figure 2: vacant sites created by the presence of a wall. The dotted circles represent spheres that would be present if the wall was not there. Therefore, the hatched volumes are not filled and constitute the wall voids.

Here, the wall effect is approximated by considering an orthorhombic arrangement of particles with a monodisperse radius R (bulk porosity of 40 %) deposited in a disk layer of diameter D and height h . The influence of the wall effect is:

$$\frac{V_{\text{walls}}}{V_{\text{voids}}} = \frac{\frac{1}{3} \pi^2 R \cdot D \cdot \left(\frac{D}{4} + h\right)}{0.4 \cdot \pi \frac{D^2}{4} \cdot h + \frac{1}{3} \pi^2 R \cdot D \cdot \left(\frac{D}{4} + h\right)} \approx \frac{R \left(\frac{D}{4} + h\right)}{0.4 \frac{D}{4} \cdot h + R \left(\frac{D}{4} + h\right)}$$

where V_{voids} is the total void volume in the powder layer.

In the above equation, the void volume due to the walls is the volume of the “missing” half sphere that is absent at the powder/wall interface, as shown in Figure 2. Figure 3 shows the importance of the wall effect on the powder porosity for a powder bed: with a cavity height of 100 μm, about 40 % of the porosity is due to wall voids. This value may be underestimated, since the upper surface will also be locally porous. However, it must be born in mind that subsequent layers will be sintered on a rougher surface, with a probably lesser influence of the walls.

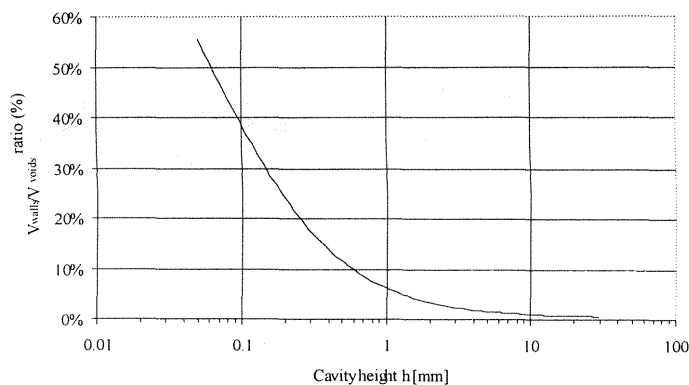


Figure 3: influence of the wall effects on the powder density (cavity/layer diameter = 60 mm; particle diameter 25 μm).

3. EXPERIMENTAL METHODS

3.1. Powders

The powders used for the experimental determination of layer density were spherical, smooth particles of nickel-base alloy (Figure 4). These materials were provided by Castolin+Eutectic SA. The powder was separated in size classes and blends with given size ratios were prepared (Table I and Table II).

Table I: available size classes.

Class	Sizes [μm]
B	150 – 200
C	100 – 150
E	50 – 75
G	25 – 40
H	< 20

Table II: composition of blends

Size ratio	Blended classes
1:2	C + E
1:5	B + G
1:10	C + H
1:18	B + H

3.2. Measurements

Conventional granulometry measurement equipment was used for the characterization of the base materials. The density of the powder layers was determined by weight measurement of a powder filled cavity (500 μm depth, 65 mm diameter). In some experiments, a deeper cavity (1500 μm) was used, because of the difficulty to deposit regular layers with the coarser powders. The container height was 5 mm and its external diameter was 80 mm. The weight of the empty container was about 57 g. The powder was deposited using a metallic ruler, taking care that as little powder as possible remained outside of the cavity.

The measurements were performed with a precision scale (± 0.02 mg). Although powder layers in SLS are usually below $100\ \mu\text{m}$ ($50\ \mu\text{m}$), the cavity used in this study was deeper in order to minimize measurement errors due to irregular layers.

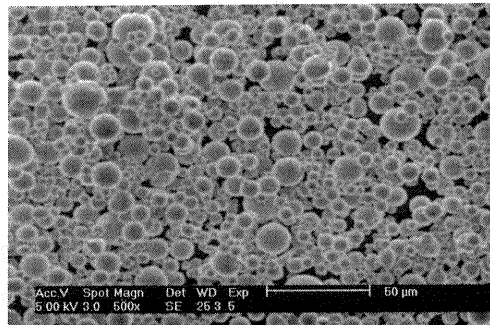


Figure 4: SEM image of the nickel model powder.

4. RESULTS AND DISCUSSION

Figure 5 presents the results obtained for different size ratios at different fines compositions. The first observation is that the 1:2 ratio, corresponding to the square void in the orthorhombic arrangement, does not bring significant improvements. Although the powders are mixed before deposition, percolation is still required and the fine particles cannot rearrange in the square voids.

The 1:5 ratio leads to a slight improvement at a rather high composition, as observed by McGeary. Due to the void size distribution (and to the locally disordered arrangement), the fine particles can still percolate through the coarse powder. With higher ratios (over 1:10), the density increase is more significant: at 30 % fines, the layer density has increased from about 55 % up to 63 %, which amounts to an over 15 % relative increase.

At higher fines content, the density decreases, probably because the passage voids are saturated and the fines tends to pull the coarse particles apart. Interestingly, the curves for bulks and for layers are quite similar, indicating that the same processes occur, although with less efficiency in the layer case. The differences observed between the pure coarse and pure fine densities are due to the different classes used, which exhibited an agglomerate structure for the finest powders.

Figure 6 compares the results of the layer density with the data obtained by McGeary for powder bulks, for the same fine content (30 %). Again, the tendency is clearly similar, but the apparent densities are more than 15 % below that of powder bulks, although the particles are also spherical (Figure 4).

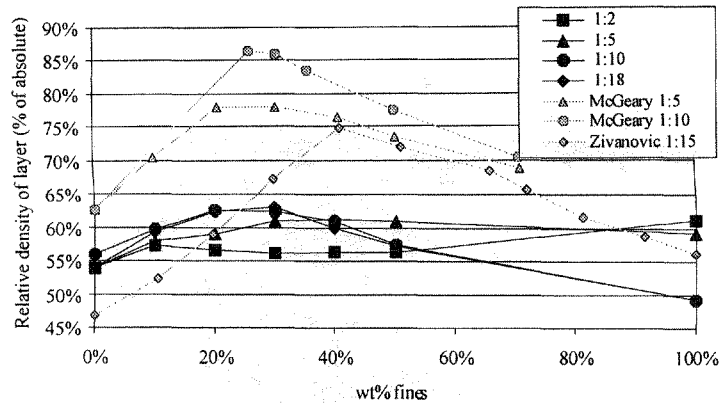


Figure 5: relative density curves for thin layer as a function of the amount of added fine fraction, for several size ratios. For comparison, data for bulks are also represented.

As expected, the value for non-spherical particles (given by Zivanovic [10]) is situated below the McGeary's curve but over that of layer density. This indicates that several effects act jointly in layer deposition. First, since the powder is not monodisperse, the voids follow a size distribution, which is a sign that probably finer particles would be required. The percolation process is also less efficient, since little energy, except gravity and friction, is provided to the powder. More importantly, the wall effects are probably very effective in this case.

Figure 3 shows that for thin layers (below 100 μm), over 40 % of the voids are generated by the wall effects. This ratio decreases rapidly when the layer height increases. The wall effects could be advantageously reduced by increasing the roughness of the walls. This way, some of the wall voids can be filled by the wall peaks. Since the layer thickness used in this study was higher than in common SLS, it can be expected that in the real case, the density and its improvement will be even lower than in Figure 6.

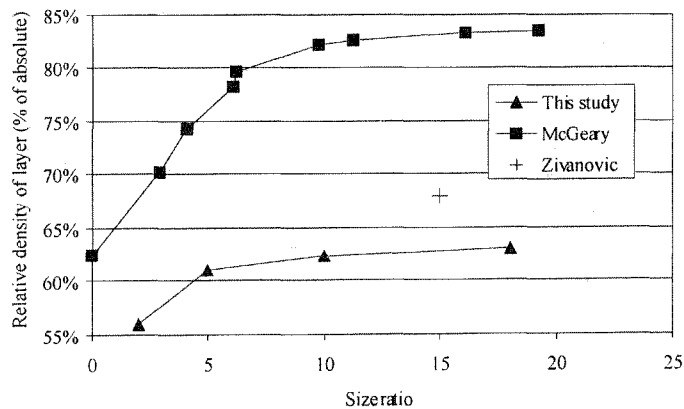


Figure 6: relative density of thin layers and bulks with 30 wt% fines as a function of size ratios.

5. CONCLUSIONS

In this paper, it has been shown that the density of powder layers can be improved by using multimodal powders. With a suitable size ratio between coarse and fine particles (over 1:10) and adequate composition (about 30 %) of fines, a density increase of about 15 % can be expected. Wall effects were approximated, showing a central influence on layer density: for layers less than 200 μm thick, the influence of the wall voids can be higher than 20 % of the total porosity in the powder. This effect may be reduced by an increased roughness of the support; the control of the roughness between layers may also be beneficial. The density values presented in this paper should be considered as an upper, achievable density, since the experiments were performed on thick layers (500 μm) compared with the thickness in the SLS process. It is to be expected that the density improvement will be less efficient in the real situation, with thinner layers.

6. REFERENCES

- 1 N.P. Karapatis ; J.-P.S. van Griethuysen ; R. Glardon, "Direct rapid tooling : a review of current research", *Rapid prototyping J.*, Vol. 4, No 2, 1997, pp.77-89
- 2 Girouard D., "Molds for Low Temperature Molding Processes using Rapid Prototyping", *Proceedings of the 4th International Conference on Rapid Prototyping*, 1993
- 3 Lohner A., "Laser sintering ushers in new route to PM parts", *MPR*, February 1997, pp. 24-30
- 4 N.P. Karapatis ; Y. Guidoux, P. E. Gygax, R. Glardon, "Thermal Behavior of Parts Made by Direct Metal Laser Sintering", 9th SFF Symposium, Austin, 1998, pp. 79-87
- 5 R. M. German, *Liquid Phase Sintering*, Plenum Press (1985)
- 6 S. Tamura et al., *J. Mat. Process. Techn.*, Vol. 42, 1994, pp. 197-207
- 7 Ferrez J.A., Müller D., Liebling Th.M., "Parallel Implementation of a Distinct Element Method for Granular Media Simulation on the Cray T3D", *EPFL Supercomputing Review No. 8*, 1996
- 8 R. M. German, *Powder Metallurgy Science*, Metal Powder Industries Federation, 1984
- 9 R.K. McGeary, *J. of The American Ceramic Society*, Vol. 44, No 10, 1961, pp.513-22
- 10 B.M. Zivanovic, *Proc. 1st Int.Conf. Compaction and Consolidation of Particulate Matter*, Brighton, 1974, pp.37-40,
- 11 G.T. Nolan, P.E. Kavanagh, *Powder Technology*, Vol. 74, 1994, pp.231-238
- 12 F. Zok, F. F. Lange, *J. Am. Ceram. Soc.*, Vol. 74, No 8, 1991, pp. 1880-1885

