Limits of Loose Metal Powder Density in the Sinterstation

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Abstract

The possibility to increase the density of applied metal powder in the Sinterstation has been investigated by successive addition of smaller sized particle fractions to a base powder. The optimal composition for each blend was determined experimentally, and their apparent densities were compared to the densities after Sinterstation application. The principal limiting factors for the density of metal powder in the Sinterstation were found to be the interparticle friction and the small sized particles' tendency to form a suspension in the air. Finally, based on the evaluation of the different powder compositions' behavior, a practical upper limit of loose metal powder density was estimated to 86%, when applied by a Sinterstation. The highest applied powder density in this investigation was a binary blend with 68.60% relative density.

1. Introduction

Some of the major challenges facing the SFF technology today are concerned with the manufacturing of tools and other objects in metal. Several technologies have been developed, but despite rapid progress there are still many more challenges to meet and properties to be improved. The commercial direct tooling and metal applications of SFF technology are dominated by different powder based techniques, such as DTM's SLS, EOS' DMLS and ProMetal. In all these approaches, the materialization of objects starts by the spreading of loose powder layers over a processing area. The particles are then either fused by a melting fraction of the compound with the laser, and thus densify by pointwise liquid phase sintering (EOS), or glued together for sintering and infiltration in a separate furnace (DTM, ProMetal). Both strategies does somewhat limit the possible material systems and no manufacturer has yet marketed a hardenable tool steel material. However a combined approach, using liquid phase sintering of the whole object during a separate furnace cycle, could probably be used to overcome this gap [1], [2].

In all these approaches a crucial point is the density in the applied powder layers. For the furnace requiring approaches, 50% density is a minimum to avoid distortion during processing [3]. An increased loose powder density would, apart from improving green strength, also have a positive impact on precision and surface quality as well as decrease the need for liquid phase or infiltrant in order to reach the desirable density It is the purpose of this present work to investigate the practical possibility to increase the density and improve the quality of the loose powder mass applied by a Sinterstation, by the addition of finer powder grades. Thereby indicate what the main limiting factors are, and whether there is an upper practical limit, for the loose metal powder density in the current Sinterstation system.

2. Background

The packing of particles is a field of importance not only within the area of metals SFF, but in a vast range of areas. Needless to say, it has, in different forms been the subject of research several times before. In a fundamental work Mc Geary [4] investigated the basic scientific laws for the packing of spherical particles, and an arrangement with 95.1% of theoretical density was reported. To achieve this, a four component packing composition was used, where each finer grade was vibrated into the voids of a matrix formed by the larger sized grains. Powder densities of that magnitude are usually found only in compressed green bodies prior to sintering. Even though such reports are encouraging, the set up was idealized, and the results could probably not be duplicated on a practical SLS system.

In the field of SFF, Karapatis et. al. [5] used an idealized experimental set up to investigate the effect of relative size ratio on the density of a single applied layer of a binary powder blend, and thereby achieved binary layers with 63% relative density. However the size range used for the particles (up to 150 μm) was but a bit outside what could be used in a practical SFF system with acceptable precision. Furthermore, only single layers were used which gave a large influence to wall effects, which may not be as important in a real powder spreading process. In particular as the spreading procedure used was not similar to the counter-moving roller used in the Sinterstation.

This work takes a practical approach to powder application. The powders were experimentally applied in a Sinters station, and powder grades were primarily selected in respect to:

1. Size range; for optimal packing of a loose powder mass, it is necessary that the finer powder grains are filling the voids between the larger grains. From a theoretical analysis it has been found that suitable proportions would be 1:7:49:343…[6]. The largest grain size is determined by maximum acceptable layer thickness, which, in respect to precision, is 0.1 mm. The smaller grain sizes are limited by availability in the suitable size range.

2. Shape; smooth spherical shape is superior in respect to internal friction and packing properties [6],[4].

3. Availability; all powders used should be available as commercial grades.

4. Handling; since the current Sinterstation systems requires manual powder handling, it should be possible to handle the powders without damaging the equipment or the health of the operator.

These aspects taken in respect, suitable iron base alloyed powders were selected, Due to limited availability in the smaller size ranges, the number of components in the powder blends are limited to three. If the wall effects are a dominant limiting factor also in reality, then the applied powder density should in all cases be smaller compared to apparent density. To investigate this, each powder compound was prepared with an experimentally determined composition, and the apparent density prior to Sinterstation application was measured to be compared to the density of the applied powder mass.

3. Experimental

3.1 Experimental procedure

A Sinterstation 2000 was used in these experiments. The apparent density of each powder prior to Sinterstation application was measured by a simple procedure with a graduated cylinder and a precision scale. A generic build file was used to apply a sufficient amount of powder in the Sinterstation's part cylinder. After termination of the build, the powder in the build chamber was removed in level with the chamber floor, and the position of the part piston was noted (h_2). The powder in the part cylinder was removed by subsequently raising the piston 0.5 mm, and scraping off each layer with a straight edge blade. This was repeated until all the applied powder was collected in a container, and the second position of the part piston was noted (h_1). The powder mass in the container (m) was weighed on a precision scale and the density of the applied powder $(r_{app.})$ as well as relative density $(r_{rel.})$ was calculated by relation with part cylinder volume (V) and theoretical density of the massive material (r_m).

$$r_{rel} = \frac{r_{app}}{r_m}; \text{ where } r_{app} = \frac{m}{\left[\frac{d_{cyl.}}{2}\right]^2 p[h_1 - h_2]} \qquad \textbf{Eq. 1. \& 2.}$$

Each powder went through several experimental cycles in order to establish the powder mass' behavior in the Sinterstation under varying powder application conditions, such as feed rate and roller speed. Following this procedure the behavior of powders, consisting of one, two and three components were examined in respect to general powder behavior and applied powder density. To ensure comparability between the different powders, each composition was made by adding a smaller grained fraction to the previous powder compound.

3.2 One Component

3.2.1 The Powder

Large sized grains: For bulk material, a highly alloyed, gas atomized tool steel powder, Anval 60, from Dynamet Anval, was selected, see **Fig. 1.** The chemical composition and size range of this powder can be found in **Tables 1 & 2**.

Table 1

Element	C	Si	Mn	P	S	Cr	N	Mo	Co	V	W	N	Fe
%	2.39	0.54	0.47	0.022	0.047	4.29	0.19	6.86	10.2	6.52	6.6	0.053	Bal

Table 1. The chemical composition of Anval 60.

Table 2.

Micron	90	75	63	45
Mesh	170	200	230	325
%<	96	76	43	8

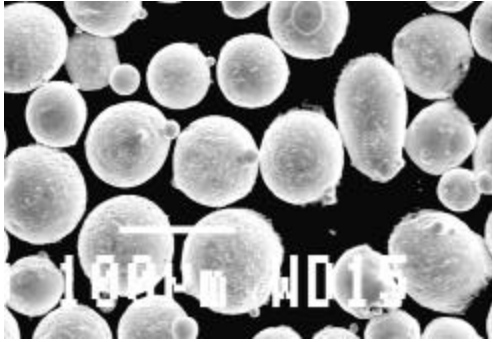*Table 2. The sieve analysis, (ASTM-E11) of Anval 60.*

Fig.1. Anval60. A highly alloyed tool steel material, with the characteristic spherical shape of gas atomized metal powder. The spherical shape is known to have the lowest interparticle friction and thus the best possible flowing behavior and packing. As in all commercial powders a size range interval is hardly avoidable. The apparent density was experimentally found to be 4.68 g/ cm^3 , or 57.16 % relative density. The density of solid Anval 60 alloy is calculated to 8.1874 g/ cm^3 .

3.2.2 In the Sinterstation

The powder was cycled twice through the experimental procedure, no visible differences in powder behavior was observed. The powder had excellent free flowing behavior. The applied powder densities found was $r_{rel.} = 60.82\%$, and $r_{rel.} = 60.09\%$.

3.2.3 Conclusion

The apparent density of this powder may seem rather low, but considering the size range interval, it is reasonable to find a lower density compared to more narrow size intervals. Still, the considerably higher relative density when applied by the Sinterstation indicates that the powder grains are being distributed in a favorable manner, and that the wall effects are not a significant limiting factor in the Sinterstation applied powder mass.

3.3 Two Components

3.3.1 The Powder

Intermediate sized grains: To fit into the voids between the larger particles, the powder of intermediate particle size should not be larger than 1/7th of that grain size. Since a particle size interval for both coarse and fine powders seems unavoidable, a fine powder where the larger part of the grains could fit into the voids of the larger part of the coarser grain mass was selected, in this case Carbonyl Iron CL from BASF, see **Fig. 2**. The chemical composition and size range of this powder can be seen in **Tables 3** & **4**.

Table 3.

Element	Fe	C	N	O	Ni	Cr	Mo
Unit	g/100 g	g/100 g	g/100 g	g/100 g	mg/kg	mg/kg	mg/kg
Limit	Min. 99.5	Max. 0.05	Max. 0.01	Max. 0.2	Max. 50	Max. 50	Max. 50

Table 3. The chemical composition of BASF Carbonyl Iron Powder CL.

Table 4.

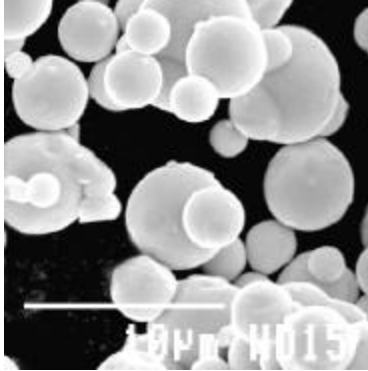| Size Distr.	D10	D50	D90
Unit	μm	μm	μm
Limit	<5	<10	<30

Table 4. The particle size distribution (Microtrac X100) of BASF Carbonyl Iron Powder CL.

Fig.2. Carbonyl Iron CL. Almost pure iron, with the spherical shape typical of carbonyl metal powder. In this size range the interparticle friction associated with Van der Waals and electrostatic forces are of increasing importance. Nevertheless the spherical shape still is the best possible considering flow and packing properties. As in all commercial powders a size range interval is unavoidable. Tap density, given by the manufacturer, is 3.8–4.5 g/cm^3, ASTM B527 (analog). The chemical composition suggests that it is reasonable to estimate the density of the solid material to be the same as pure iron: 7.86 g/cm^3.

Optimal composition in terms of weight fraction large particles X^* can, according to [6] be calculated by: $X^* = f_L / f^*$, **Eq. 3.**, where f_L is the fractional packing of large particles. f^* is the packing density at optimal composition, given as $f^* = f_L + f_S(1 - f_L)$, **Eq. 4.**, where f_S is the fractional packing of small particles. f_L has been determined experimentally to 0.60, and the tap density interval was used to find f_S. Tap density: $r_{CL} = 3.8 - 4.5$ g/cm^3 ; $r_{Fe} = 7.86$ g/cm^3 $=> r_{rel.} = 48.35\%$-57.25%, thus, $f_S = 0.4835 - 5725$. **Eq. 3.** & **Eq. 4.** => $X^* = 0.7245 - 0.7569$. Compensated for the difference in specific mass this gives that the optimal composition would include 25.30-28.65 weight % Carbonyl Iron Powder CL. Such a compound could give a theoretical maximum packing density, f^*, of 79.39-82.94% of the solid material. Several compositions with weight fraction Carbonyl Iron CL varying from 21.8% to 32.24% was mixed in a horizontally rotary type-blender, and the densities were measured. With higher fraction fine particles, the free flowing properties decreased, as the interparticle friction and the tendency to form agglomerates in the powder blend increased. This made the measurement of apparent densities more uncertain. The variation of the achieved densities can be seen in **Fig. 3.**
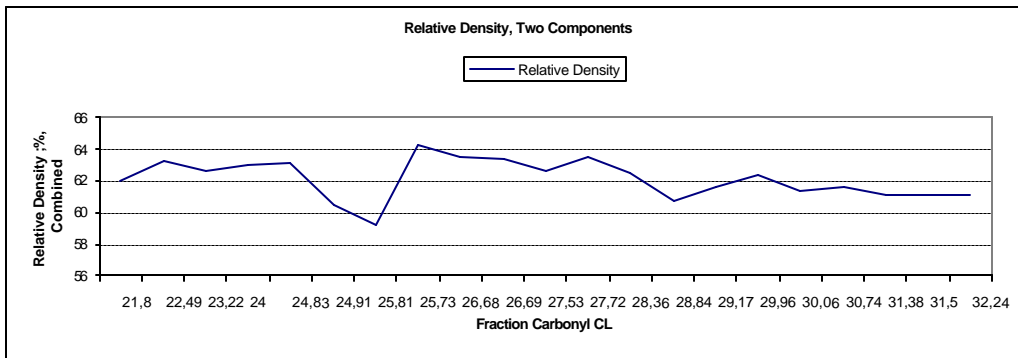
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Fig. 3. *Relative densities of the binary blend of Anval60 and BASF Carbonyl Iron Powder CL.*

With compensation for some divergent values the trend is obvious in **Fig. 4.**

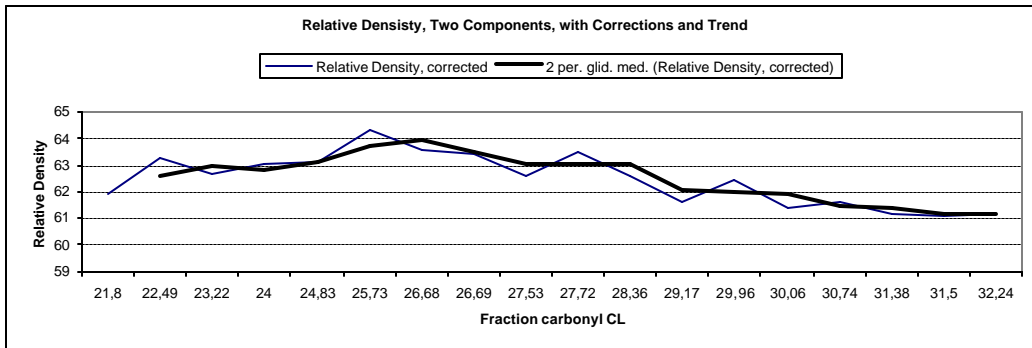**Relative Densisty, Two Components, with Corrections and Trend**

Fig. 4. Corrected relative densities of the binary blend with trend inserted.

In conclusion of these experiments, a powder compound was composed by blending 26% Carbonyl Iron CL into a base of Anval 60. This binary blend has a relative apparent density experimentally found to be 63.88%. The solid density is calculated to 8.1 g/cm^3.

3.3.2 *In the Sinterstation*

The powder properties were examined according to the experimental procedure. The powder body's consistency was similar to moist sand, and apart from a tendency to form agglomerates and a general decreased free flowing behavior, the powder showed a disposition to raise dust in the air during handling. Still, the powder spread well and showed no visible signs of powder segregation during the application of layers in the part cylinder. Obtained densities related to roller speed and feed rate can be seen in **Fig. 5.** & **6.**
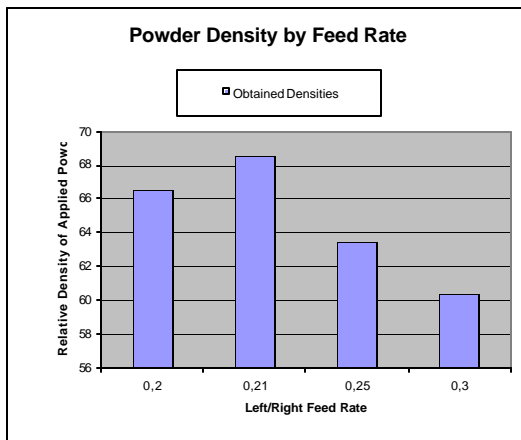

Fig. 5. Powder densities obtained by variation of feed rate. At feed rate 0.20 the amount of powder was insufficient to fill the layer of the part cylinder. Roller speed is set to 100.
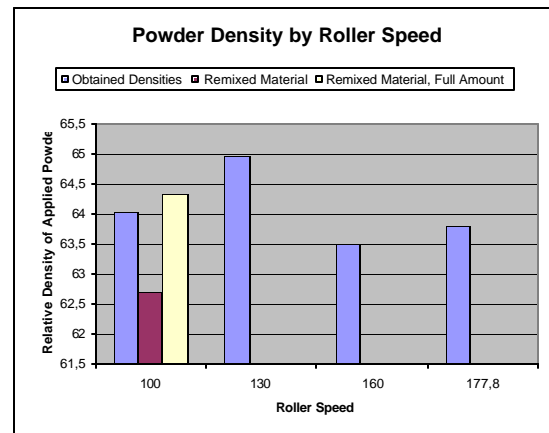
Fig. 6. Powder densities obtained by variation of roller speed. The two extra runs at roller speed 100, was made to find out whether the lower densities in this test series was caused by powder segregation. Left/right feed rate is set to 0.21.

Since the second test series concerning roller speed gave a considerable lower density at conditions identical to the most favorable, it was suspected that a segregation in the powder mass had occurred. However, after recycling the powder in the mixer, a smaller sample of powder obtained an even lower density, and the full amount of powder showed only a slight increase. Evidently segregation was not the problem.

3.3.3 Conclusion

The addition of a second, smaller sized powder gave a distinct increase in powder density. The densities obtained were however, not in the range of the theoretically achievable. Increased roller speed had, if any, a small, and possibly negative effect on the powder packing. Feed rates above the necessary to complete the new powder layer showed a distinct negative effect on the obtainable powder densities. The apparently increased interparticle friction in the powder compound could explain this. A free flowing powder forms a rolling wave in front of the roller during the spreading of each new layer. With the interparticle friction, the free flowing behavior is reduced and the powder partially forms a "wedge" of agglomerates that is being pushed along by the roller. An increase in mass of such a wedge would induce a higher pressure, and thus higher frictional shear in the applied powder bed below, causing cracks and poor packing in the applied layer. The counter rotation of the roller does lift and transport some powder, but only grains in direct contact with the rollers moving surface. There is only a limited rolling motion in this powder blend during the application of new layers.

Densities above the measured apparent density of this powder composition were obtained in the Sinterstation. This could be explained by the strong effect friction has on the used method for the measurement of apparent density, but in the Sinterstation does the powder motion probably also have an influence. The counter rotating motion of the roller is likely to have a vibratory effect on the upper surface of the applied layer, as well as exerting slight downward pressure on the last thin "edge" as the powder is being moved ahead. These conditions are not entirely unlike those idealized conditions used in McGeary's experiments, [4], mechanically inducing percolation of fine powder in each layer. These results confirm that the influence of the powders' interparticle friction dominates over the influence of wall effects on the density of Sinterstation applied powders.

The lower densities obtained by the repetition of early experimental runs was a mystery with separation ruled out as an explanation. However by re-weighing the powder mass it was found that a significant amount of powder had been lost during handling. There are no obvious leaks, except for the powder's tendency to raise dust in the air. Given a moderate estimation, that 75% of thereby lost powder is fine sized Carbonyl Iron CL, that would leave a blend with only 23.75% Carbonyl Iron CL. Such a composition had a relative apparent density of approximately 63% (**Fig. 4.**). In that case, the densities obtained in the last experimental runs follow the pattern of the earlier in respect to the obtained densities. Therefore this assumption is being made for the continued experiments.

3.4 Three Components

3.4.1 The Powder

Smallest sized grains: The third size range is supposed to fit into the voids between the grains in the binary powder blend. As both previous components show a size range interval, the prediction of the actual sizes of these voids is hardly possible. However, by selecting the smallest available powder, with an average particle size, within the range to fit into the matrix of the intermediate sized powder, it was estimated that this addition would give an increase in loose powder density. The selected powder was Carbonyl Iron Powder HQ from BASF, see **Fig. 7.** The chemical composition and grain size distribution can be seen in **Tables 5** & **6**.

Fig.7. Carbonyl Iron HQ. Almost pure iron, with the spherical shape typical of carbonyl metal powder. In this size range, the friction is associated with Van der Waals and electrostatic forces, and is thus increasingly independent of particle shape. Nevertheless, the spherical shape still is the best possible considering the achievable packing properties. As in all commercial powders a size range interval is hardly avoidable, and with smaller particle size, the relative size interval increases.

Tap density, is given by the manufacturer, to $3.8-4.5$ g/cm^3, ASTM B527 (analog). The chemical composition suggests that it is reasonable to estimate the density of the solid material to be the same as pure iron: 7.86 g/cm^3.

Table 5.

Element	Fe	C	N	O
Unit	g / 100 g	g / 100 g	g / 100 g	g / 100 g
Limit	Min. 97.5	0.7-1.0	0.7-1.0	0.3-0.5

Table 5. *The chemical composition of BASF Carbonyl Iron Powder HQ.*

Table 6.

Size Distr.	D10	D50	D90
Unit	μm	μm	μm
Limit	1.2	2.5	4.0

Table 6. *The particle size distribution of BASF Carbonyl Iron Powder HQ.*

To find a suitable composition for the ternary powder an experimental approach was used. Different blends with varying composition was mixed and the apparent density was measured. The variation of the achieved densities for respective composition can be seen if **Fig. 8.**

Considering that the increased interparticle friction in the ternary blends, made the difficulties of apparent density measurement increase with the fraction Carbonyl Iron HQ, and that a different measuring cylinder was used for blends 12 and 13 the difference in density are in general small among these compositions. Nevertheless in conclusion of this experimental series, a powder compound was composed by with 73.35% Anval 60, 14.10% Carbonyl Iron CL, and 12.55% Carbonyl Iron HQ. This ternary blend has a measured relative apparent density of 68.40%. The solid density is calculated to 8.1 g/cm^3.

Relative Density, Three Components

Relative Density

Relative Density, %

Three Component Blends
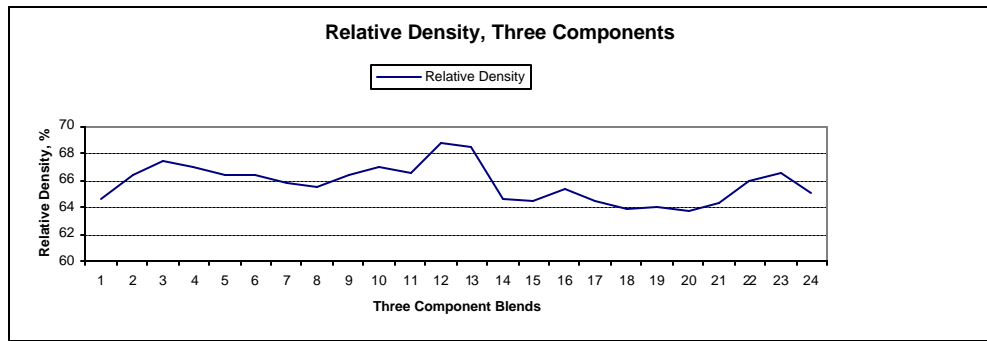
Blend	Anval 60	CL	HQ		Blend	Anval 60	CL	HQ
1	84,27	15,23	0		13	74,86	14,08	11,06
2	83,24	15,54	1,23		14	74,96	14,08	11,06
3	82,23	15,35	2,42		15	74,09	14,51	11,4
4	81,24	15,17	3,59		16	73,27	14,97	11,76
5	80,28	14,99	4,73		17	72,4	15,46	12,14
6	79,34	14,81	5,85		18	71,48	15,98	12,55
7	78,43	14,64	6,93		19	70,48	16,53	12,98
8	77,53	14,47	8		20	69,42	17,13	13,45
9	76,65	14,31	9,04		21	68,28	17,77	13,95
10	75,8	14,15	9,04		22	67,04	18,46	14,5
11	74,96	13,99	9,04		23	65,71	19,21	15,08
12	74,96	13,99	9,04		24	64,26	20,02	15,72

Fig. 8. *Compositions and relative densities of the ternary blends.*

3.4.2 In the Sinterstation

The powder was examined in the Sinterstation according to the experimental procedure. With a body consistency quite similar to wet sand, the powder showed much interparticle friction and was also difficult to handle without raising smoke-like dust. The compound hardly had anything like free flowing behavior and was more likely to form agglomerates than not. Since the powder tended to stick to the roller, scrapers were installed. Several cycles were preformed to investigate the powder compound's spreading behavior and find suitable process conditions. The obtained densities can be seen in **Fig. 9.** In difference from the powder in the part cylinder, the powder distributed around the building area settled to a stable and apparently very dense surface, whereas the powder mass in the part cylinder, remained unstable and shifted with every pass of the roller.
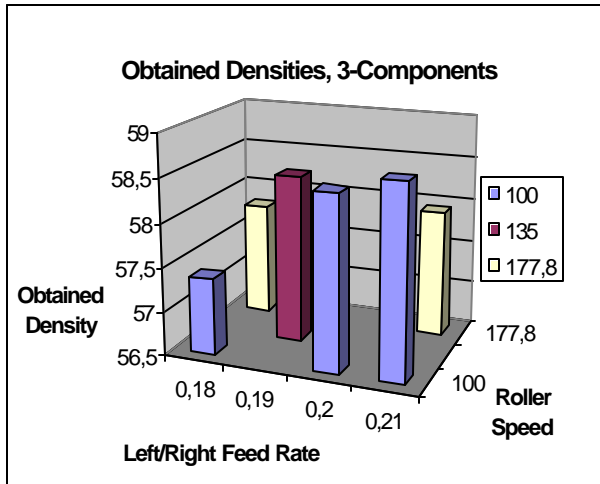
	100	135	177,8
0,18	57,37		57,81
0,19		58,42	
0,2	58,48		
0,2	57,45		

Fig. 9. *Powder densities obtained by variation of feed rates and roller speeds. The powder showed unstable behaviour and the applied layers were uneven and cracked. As the different cycles at 0.20 feed rate/100 roller speed shows, the results are of dubious reproducibility.*

3.4.3 Conclusion

The addition of a third component did increase the apparent density of the powder mass, but in difference to the binary blend that increase could not be observed in the Sinterstation. The obtained densities were on the contrary significantly lower than those obtained with the binary blend. Despite variation in both roller speed and feed rate, a stable behavior in the applied powder mass could not be found. This is explained by the strong disposition to form agglomerates and is in analogy with the explanation of the behavior of the binary powder. A higher interparticle friction in the powder mass causes a stronger frictional shear in the underlying layers. When the shear is too big the powder mass starts to shift. The frictional shear depends on both the inner friction in the powder mass and the pressure applied by the mass of transported powder during spreading.

These phenomena in combination with the general problems of handling extremely fine powder, makes metal powder compositions containing grain sizes in the range of one micron or smaller, unlikely as practical SLS materials.

5. Conclusions

It has been established that the addition of particles of a smaller size range can increase the relative density of a powder mass applied in the Sinterstation. It is furthermore concluded that the principal limiting factors for such an approach to densification of the powder mass are: the availability of suitable powder grades, the inner friction of the powders with smaller particle sizes, and the dusting of fine sized powder grades.

1. Availability: Each additional smaller sized powder component should be of a narrow size interval, and maximum 1/7th of the diameter of the larger particles to increase the density of a powder mass. It is obvious that there are limits to how fine powders and how narrow grain size ranges can be obtained at a reasonable cost.

2. Inner friction of fine powders: As particle sizes reach the range of 1-2 microns or smaller, the pure powder shows a dominant tendency to agglomerate, which counteracts the densification as well as separation. This effect could possibly be reduced by addition of some sort of lubricant to the powder. However this lubrication would in it self reduce the powder density, and it is most uncertain how such an addition would influence the behavior during the further processing.

3. Dusting of fine powders: A suspension of metal particles is not an ideal atmosphere for electronic equipment, which must include SFF equipment. Even less so considered that such suspensions are deemed highly flammable and hazardous to the human health. Iron, for example, is probably harmless to ingest, but ill-advised to inhale. Even if the previous limits could be overcome, the dusting of fine powders is hardly possible to address without an entirely new powder handling system.

These points taken in consideration, size range compositions beyond binary are unlikely to become of practical use. The highest theoretical density that thus could be expected to come of use is 86%. Still considering the practical difficulties, any density in a stable powder mass above 70% should be considered an achievement. In this work, the highest achieved applied powder density was 68.60%.

These limits of applied loose powder density, makes further densification a necessity to build full density objects. This, in turn, will have a limiting effect on the material choice, achievable green strength, and also influence the process precision. It is unlikely that these issues could be fully addressed successfully to their full extent without designing an entirely new machine.

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