Influence of the particle size distribution on surface quality and mechanical properties in additive manufactured stainless steel parts

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Abstract:

A recent study confirmed that the particle size distribution of a metallic powder material has a major influence on the density of a part produced by SLM. Although it is possible to get high density values with different powder types, the processing parameters have to be adjusted accordingly, affecting the process productivity. However, the particle size distribution does not only affect the density but also the surface quality and the mechanical properties of the parts. Therefore, this study compares three different particle size distributions depending on the laser scan velocity and two layer thicknesses of 30μm and 45μm. By using an optimized powder material a low surface roughness can be obtained. A subsequent blasting process can further improve the surface roughness for all powder materials used in this study although this does not change the ranking of the powders with respect to the resulting surface quality.

Keywords:
Additive Manufacturing, selective laser melting, surface quality, powder materials

I. Introduction

Additive Manufacturing Technologies such as Selective Laser Sintering (SLS) and Selective Laser Melting (SLM) become more and more important for the fast production of industrial products. Typical fields of application for SLM are medical instruments and - in the future - implants, dental products such as e.g. caps and bridges and parts for diverse applications in mechanical engineering. Lightweight structures, e.g. for the aerospace industry, will be an important future field of application as the manufacture of very complex parts is possible with SLS and SLM. One of the first Rapid Manufacturing application was Conformal Cooling – the additive production of tools with complex internal cooling channels for plastic injection moulding [ ]. However, additive produced tools are typically conventionally surface finished in order to fulfil the requirements for a good surface. As Rapid Manufacturing is a growing technology for a wide range of products for diverse applications, the needs for a high part quality in the as-processed condition are growing. The technology can reach its maturity only when these needs can be fulfilled.

The quality evaluation of an additive produced part depends on several factors. Apart from geometrical restrictions, which may narrow down the range of possible geometries and which are process specific, the parts produced should generally fulfil the following requirements: The density

should be as high as possible, the materials' mechanical properties should be as close as possible to conventional materials and the surface quality should be as high as possible in order to minimize finishing operations. Furthermore, the parts have to be economic, which asks for a fast production process. These factors are the driving forces for the optimisation of the materials and the processes in order to meet the needs of the applications [1] and corresponding industries.

A lot of work has already been done to investigate the effect of different processing parameters like scan strategy, layer thickness, laser power and velocity on the resulting part properties [2-4]. However, the results are affected by the details of the equipment used and the fact that typically different powder materials have been used. Therefore, a direct comparison of the results is only possible on a qualitative basis.

The present paper investigates the effect of three different powder granulations on the resulting part density, the surface quality and the mechanical properties of the materials produced. For that purpose, for three powder grades and two layer thicknesses (30μm and 45μm), the scan velocity has been optimised with respect to the resulting part-density. It can be clearly shown that the particle size distribution plays an important role not only regarding the density [5] but also the surface quality and the resulting mechanical properties. These differences might at least partly explain some differences in the mechanical properties as stated in the material data sheet of the machine producers.

Furthermore, the current work points out that any standardisation initiative, such as ASTM-F42, would be well advised to take care of the granulation of powders for powder-bed based Additive Manufacturing processes.

II. Methods, Materials and Measurements

a) SLM Machine
The SLM machine, type Concept Laser M1, is equipped with a Nd: YAG solid state laser with a maximal laser power of about 105W. The scan strategy used to produce the samples in this study is a chess-board like structure, where 5x5 mm^2-squares are scanned. More details are described in [5, 6].

b) Materials
The samples for the measurement of the surface quality as well as the tensile bars were produced using the material grades according to Table 1. The particle size distributions were measured using a HELOS&RODOS R3 Laser Diffraction Sensor of Sympatec GmbH.

Table 1: 316L powders used for producing the test cubes

Powder type	316L – Type M Type 1	316L – Type 16-45 Type 2	1.4404 – CL20 Type 3
Manufacturer	A	A	B
D_{10} (μm)	7.12	19.84	15.26
D_{50} (μm)	15.12	28.26	37.70
D_{90} (μm)	24.17	41.13	55.54

The powders can be characterised in the following way:

Powder type 1 and 2, which are produced by the same manufacturer, are characterised by a Gaussian like particle size distribution with a slight asymmetry towards coarser particles where as type 3 (Figure 1) is an asymmetric distribution with an in creased concentration of finer particles. More details are described in [5]. It is expected that a lognormal distribution could describe these powders more accurately than a Gaussian distribution.
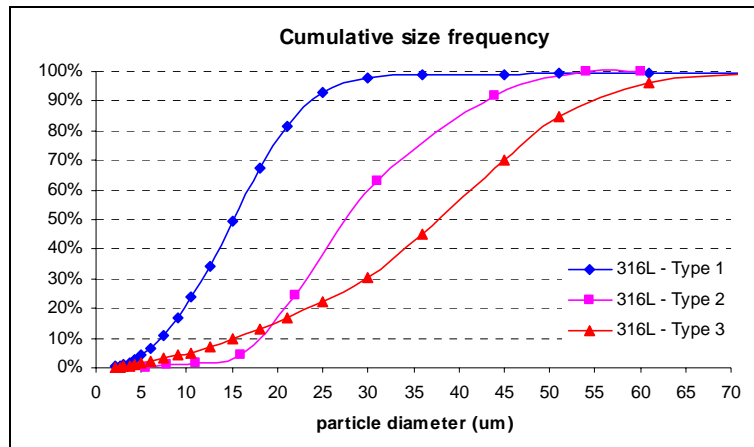
Figure 1: Cumulative size frequencies of powder type 1, 2 and 3

c) Surface quality measurements

Test samples for the measurement of the scan surface quality were produced using the processing parameters according to Table 2.

Table 2: Processing parameters for the production of test cubes for surface quality measurements

	316L – Type 1		316L – Type 2		316L – Type 3	
Layer thickness t_{Layer} (μm)	30	45	30	45	30	45
Laser Power[1] P_{Laser} (W)	104	104	104	104	104	104
Scanning speed[2] v_{Scan} ($^{mm}/_s$)	300 – 800[2]	175 – 300	250 - 500	175 - 275	250 - 500	175 - 275
Hatch distance s_{Hatch} (mm)	0.13	0.13	0.13	0.13	0.13	0.13

[1] Laser power approximately 104W $\pm$ 0.5W

[2] The scan speed was adjusted in steps of 25 $^{mm}/_s$ or 50 $^{mm}/_s$

Two layer thicknesses t_{Layer} of 30μm and 45μm were used, where the first is often used to optimize the surface quality of the parts produced, as stair effects are minimised [7]. The latter thickness can be used to reduce the number of layers that have to be generated and therefore to optimize the process productivity. For the same reason, the laser power P_{Laser} was set to the maximum possible power in order to maximise the scan velocity.

The measurement of the surface quality was conducted using a Perthometer type S3P measuring device of Feinprüf-Perthen GmbH. The sensing device was RFHTB-250. The measuring direction was 45° in relation to the orientation of the squares on the top layer of the test specimens. This is considered as a fair direction because a measuring direction parallel to the scan tracks would result in unrealistic low roughness values. In contrary, a perpendicular measurement would result in too high values. Therefore, 45° is a realistic direction and allows a measuring distance of 5.6 mm.

In order to analyse the effect of a simple surface treatment, the test cubes were measured both with and without micro blasting. A PEENMATIC-620S equipment with stainless steel particles SDR 0.4B of IEPCO, Switzerland was used. The blasting pressure was set to 5 bar.

c) Mechanical test bars

The test specimens were produced as a blank using SLM according to the processing parameters described in Table 3. The scan speeds used to produce these specimens were chosen as if each of the three powders would have been developed separately for the SLM process except for the powder type 3 / 30μm layer, where the recommendations of Concept Laser were used. Therefore, the scan speeds were selected to provide for a high productivity and a nearly fully dense material ($\approx$ 99.5%). After the SLM-production the blanks were drilled to the end geometry according to DIN 50125, form B, to avoid influences due to notch factors related to the rough part surface.

Table 3: Information on mechanical test specimens produced. Standard: DIN 50125 – Form B

t_{Layer}	Orientation[1]	Type 1		Type 2		Type 3[2]	
	0°	B10x50	/ 450 $^{mm}/_s$	B4x20	/ 250 $^{mm}/_s$	B10x50	/ 400 $^{mm}/_s$
30µm	45°	B10x50	/ 450 $^{mm}/_s$		-	B10x50	/ 400 $^{mm}/_s$
	90°	B10x50	/ 450 $^{mm}/_s$	B4x20	/ 250 $^{mm}/_s$	B10x50	/ 400 $^{mm}/_s$
45µm	0°	B4x20	/ 225 $^{mm}/_s$	B4x20	/ 225 $^{mm}/_s$	B4x20	/ 225 $^{mm}/_s$
	90°	B4x20	/ 225 $^{mm}/_s$	B4x20	/ 225 $^{mm}/_s$	B4x20	/ 225 $^{mm}/_s$

[1] 0° orientation: The test specimens were produced in a laying position
90° orientation: The test specimens were produced in a vertically standing position
[2] Scan speed for 30µm layers according to the recommendation of Concept Laser GmbH

Of each setup, at least 3 specimens were produced. In order to investigate the influence of different building orientations, the 30µm-test specimens were produced in the three orientations of 0°, 45° to the horizontal and in an upstanding position (90°) where as the 45µm specimens were only investigated in the 0° and the 90° orientation.

The mechanical parameters were measured with a materials testing machine Zwick Z100THW equipped with a 100 kN Xforce load cell and a high resolution non-contact optical strain measurement device (Zwick videoXtens) under control by the testXpert II software. The measurements and evaluations were performed using a strain rate of 0.001 s^{-1} in accordance to DIN EN ISO 6892-1.

III. Results and Discussion

a) Density

Density was measured using the Archimedes method. A comparison of different density measurement methods [8] showed that the repeatability of this method is < 0.1% for high part densities. The minimal scan energy density for high density parts depends on the powder characteristics and the layer thickness [5, 6], as shown in Figure 2. The processing parameters (Table 2) used for the production of tensile bars were selected based on these results, which have been published already earlier [5].
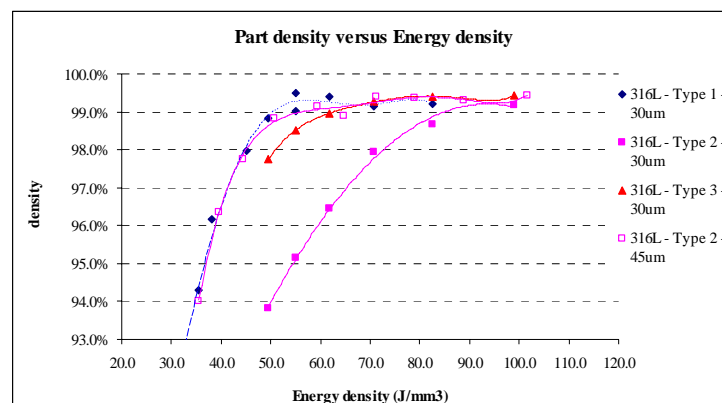

Figure 2: Part density versus Energy density for 30µm and 45µm layers

b) Surface quality

The effects of the different particle size distribution on the surface qualities are discussed by means of R_a values. Similar dependencies and differences can be observed even with other parameters like R_{max}, R_z and R_P.

The surface roughness values follow more or less a 2nd order polynomial equation over the scan speed or the specific energy density (Figure 3). Badrosamay [6] showed that the powder type influences the surface roughness in the "as processed" condition in a significant way. As expected, Figure 3 approves these findings that the processing of powders with coarser particles results in a

rougher surface, even after a subsequent blasting operation. The minimal surface roughness R_a for a fine powder type 1 after the blasting operation is in the range of 5μm for both layer thicknesses. However, for higher layer thicknesses, the roughness values tend to conform to each other. Furthermore, for energy densities between about 50-90 $^J/_{mm}3$,
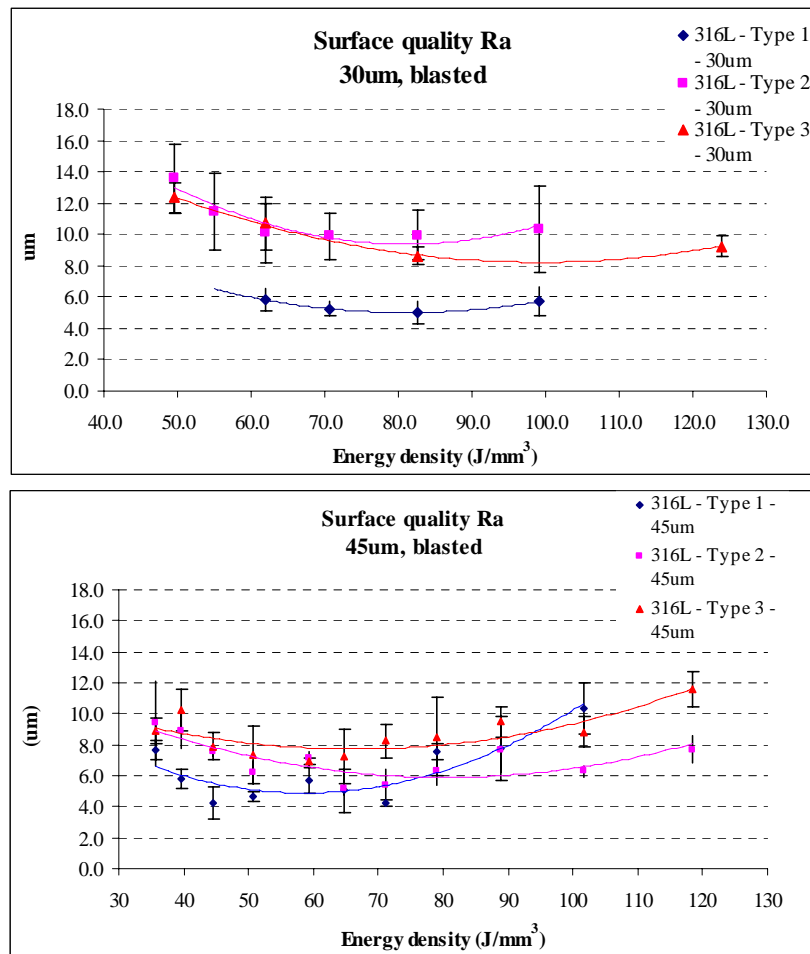
Figure 3: Blasted scan surface quality for 30μm and 45μm layer thicknesses

In order to remove adherent powder particles slightly fused to the scan- or sidewall-surfaces, blasting operations are widely used as this is a simple and economic treatment. The effect of a blasting operation is shown in Figure 4. Obviously the effect is dependent on the scan velocity as two significant different regions can be identified. For velocities > 300$^{mm}/_s$ a mean reduction over all powder types of 5.1±1.3μm (Ra) or 27.0±6.2μm (Rz) can be achieved.
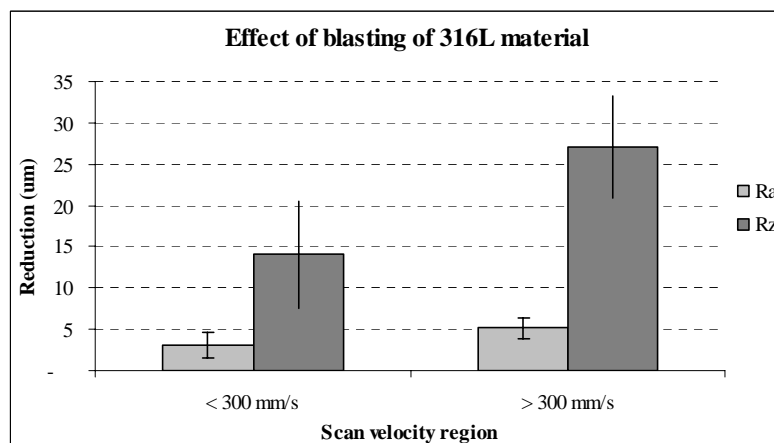
Figure 4 : Effect of a blasting operation on Ra for scan velocities of < and > 300 $^{mm}/_s$

c) Mechanical properties

It is widely known that the build orientation influences the mechanical properties of SLM-parts [9, 10]. In addition to the build orientation, present results show that powder granulation also influences the mechanical properties in a significant way.

The mechanical properties have to be compared to conventional material requirements[11]: tensile strength 490 – 690 MPa, fracture elongation 40%.

Influence of the build orientation on R_m and $R_{P0.2}$:

Regarding Figure 5, it can be stated that the powder types 1 and 2 show very much comparable mechanical parameters. The differences in the two powder types for 30μm layers in 0° orientation are only in the range of 3.7% (R_m) or almost 0% for $R_{p0.2}$. Even for 45μm layers, these differences are < 1.7%. For the 90° orientation, type 2 has a 3.7% higher mechanical strength (30μm layer) or -1.3% for the 45μm layers, respectively. In comparison, powder type 3 shows significant (p > 0.05) lower values. They are typically between -20% (R_m, 30μm, 0°) and -34% ($R_{p0.2}$, 30μm, 90°) lower than for powder type 1. The values for the 45μm layers are in the same range.

The decrease of R_m and $R_{P0.2}$ from the 0°- to the 90°-orientation is obvious. For the 30μm layers, these values are between -9.6% and -22.2% ($R_{P0.2}$), between -5.8% and -25.0% (R_m). For the 45μm layers, there is a significant decrease in R_m but not for $R_{P0.2}$, where powder type 1 and 2 show a slight increase of about 2.8% (mean).
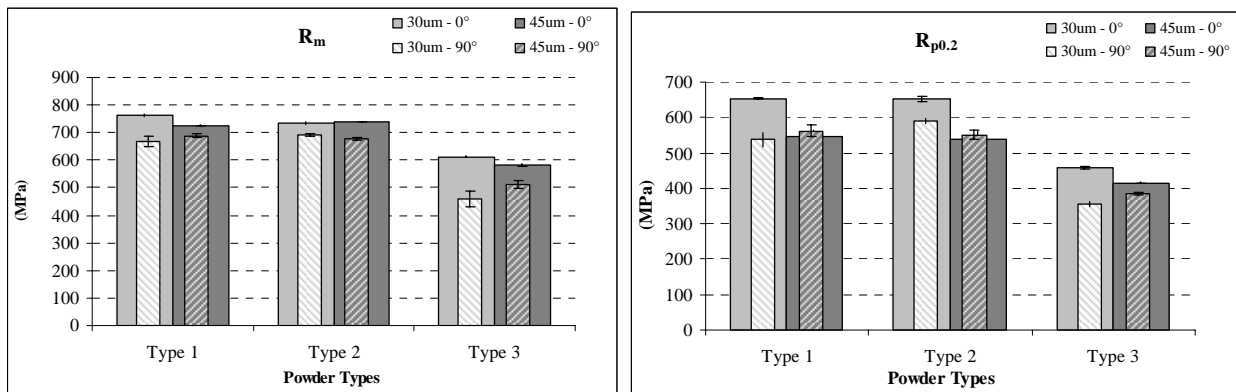

Figure 5: Ultmate tensile strength R_m and yield strength at 0.2% offset $R_{p0.2}$ for powder types 1, 2 and 3 for 30μm- and 45μm-layers and for the two specimen orientations 0° and 90°

The decrease of the mechanical parameters between 0° and 90° depends on the specific polar angle, as shown by Sehrt [3]. Thereby, within a polar angle between 0° and 45° there is only a slight decrease. Our results (Figure 6) fit very well to these data: -3.1% for powder type 1 and -0.5% for powder type 3. Thus, the big amount of the decrease takes place at higher angles, pointing out that the interlaminar bonding plays a significant role.
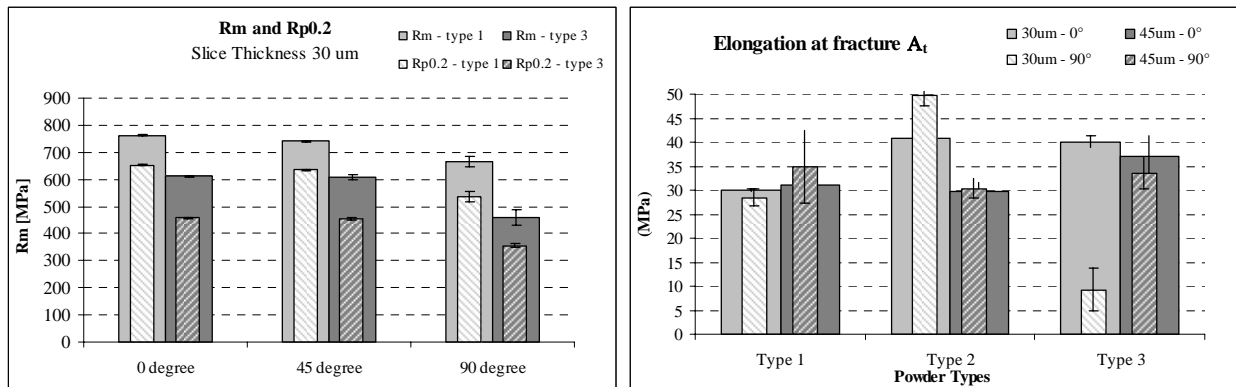

Figure 6: Ultmate tensile strength R_m and yield strength at 0.2% offset $R_{p0.2}$ for 0°, 45° and 90° orientations (left) and elongation at fracture A_t (right) for powder type 1 and 3.

The elongation at fracture (ductility) is a highly sensible value resulting in high differences. However, Figure 6 (right) shows that the fine powder type 1 results only in minor variations between different part orientations and layer thicknesses. Powder type 2 shows very high breaking elongations for the 30μm layer thickness and almost the same values for 45μm as for powder type 1, where as powder type 3 has a very good breaking elongation only for the 0° orientation.

IV. Discussion

a) Part density and Surface quality

Typically, the scan velocity selected by the requirement for a good part density, leads to surface qualities in the region of the optimum. This indicates that the density of a powder layer with a specific granulation has to be as high as possible. Only in this case the effective layer thickness t_{eff} [5] remains at acceptable values, e.g. about 50μm for a powder layer density of 60% and a machine layer thickness of 30μm. However, if the powder consists of too many particles, which have to be classified as "coarse" in relation to the effective layer thickness [5] ($D_{90} <\approx 0.67 \bullet t_{eff}$) and if fine particles are absent, the density of the resulting single powder layer can be lower than the corresponding density of the powder itself. The lower the powder layer density, the higher t_{eff} typically resulting in a rougher surface. A comparison of the three powder densities is shown in Table 4. The tapped densities are close to 60%, which is a typical value for real powders [12, 13]. Obviously, there are only minor differences between the three powder grades regarding the tapped density, although the resulting part properties (surface quality, mechanical properties) differ significantly. It is expected that this is due to mentioned effects of the density of the effective powder layer, which is created by the layer device.

Therefore, the density of the effective powder layer will be significantly lower than the tapped density and more dependent of the material type. A further effect plays a significant role: The so called Container Wall effect [13, 14], describing the influence of the underlying plane (scanned surface) on the powder density. It is clear that this effect mainly play its role within the first few particle diameters [13] or at least within t_{eff}. Karapatis [14] showed that the additional amount of voids, caused by the platform, can cause as much as 40% of the total porosity of the powder bed. However, this might be true for flat surfaces. In the current case where the powder layers are produced on the last scan surface with a specific roughness, the additional porosity caused by the surface is reduced.

Powder type	Type 1	Type 2	Type 3
Apparent density (%)	39.4 ± 0.1%	39.7 ± 0.06%	39.4 ± 0.33%
Tapped density (%)	59.8 ± 1.5%	58.0 ± 1.2%	59.3 ± 0.2%

Table 4: Apparent and tapped densities for powder type 1, 2 and 3

As a consequence, the tapped density declaration for a powder material, as e.g. stated by the manufacturer, is not sufficient for the comparison of different powder grades regarding their usability for Additive Manufacturing Processes.

It can be stated that a fine powder granulation generally leads to better densities and surface qualities than a coarser material [6], even after a succeeding blasting operation (Figure 3). However, a particle size distribution has to be qualified against the background of the layer thickness used. Especially at higher layer thicknesses where the requirement for $D_{90} < 0.67\ t_{eff}$ is fulfilled for all investigated powder types, comparable surface roughness values are reached (Figure 3).

Beside the powder particle size distribution, the processing parameters, in particular the hatch distance, influence the resulting scan surface quality as well. For each given hatch distance, the roughness takes a specific level, as shown by Badrosamay [6] but the general dependency of a powder granulation remains the same.

The side wall roughness was not investigated in this study, as there is no obvious trend between roughness values and processing parameters and $R_{a, Side}$ is typically $< R_{a, Top}$ [6]. However, for

surfaces with an angle < about 45° to the horizontal, heat concentration effects lead to a partial fusing favoured of fine particles, reducing the surface quality. Therefore for such surfaces, which typically occur in industrial geometries, coarser powders are favourable.

The effect of a blasting operation on the top surface quality is dependent on the scan velocity or energy density, respectively, as shown in Figure 4. Low scan speeds are resulting in fully melted powder particles generating low roughness values; where as at high scan speeds especially coarse particles can be only partially melted. Such particles can be removed by a corresponding blasting operation.

b) Mechanical strength

Interestingly, powder type 1 and 2 show a comparable mechanical strength although the particle size distributions are quite different. In contrast, type 3 shows significant lower values. At first it was expected that the types 2 and 3 would be more comparable than the types 1 and 2. A reason for these differences in the mechanical parameters might be the fact that the powder type 3 results in bigger pores compared to type 1 (Figure 7). This effect could be due to the bigger particles and possibly a higher amount of hollow particles, which can be specific to the production process.
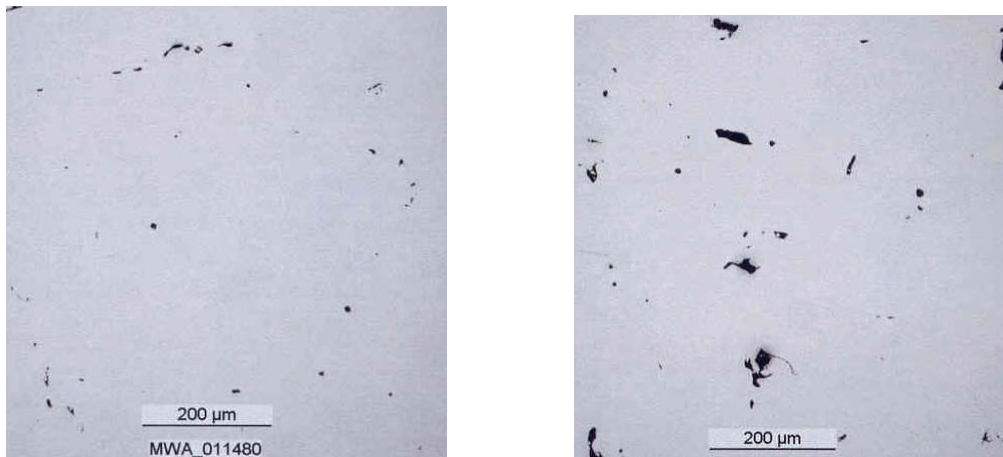

Figure 7: Pore sizes of test cubes for powder type 1 (left) and type 3 (right) for 30μm layers in 0° orientation

It is expected that the comparably higher scan speed (for 30μm layers) used for powder type 3 in comparison to type 2 is not the reason for the lower mechanical parameters as the same differences in the mechanical parameters can be seen for the 45μm layers although the scan speeds are the same (Table 3). Furthermore, the density of the powder type 3-material reached also values of about 99%, which is only slightly lower than the target value of 99.5%. Therefore, the higher scan speed used for powder type 3 can be at least partially explained by the fact that this powder consists of a specific amount of fine powder particles, allowing to fill the voids between them.

However, it is also expected that at least the high amount of big particles (in comparison to the effective powder layer thickness, Table 1) of powder type 3 plays a significant role. This is expressed by the fact that in comparison to the types 1 and 2, the decrease of the mechanical parameters (R_m, $R_{p0.2}$) between the 0° and the 90° orientation is about double the values of the latter two powders. We explain this at the moment by a reduced thermal penetration depth, which can be caused two effects:

a) A higher energy absorption by the bigger particles. This can be expressed by the ratio of the available to the required energy, needed to fully melt a powder particle. This ratio decreases with increasing particle radius:

$$\frac{E_{av}}{E_{need}} = \frac{A \cdot I_0 \cdot \pi \cdot r^2}{{4}/{3}\, \pi \cdot r^3 \cdot \rho \cdot (C_p \Delta T_m + L_m)} = \frac{1}{r} \cdot \frac{3\, A\, I_0}{4\, \rho\, (C_p \Delta T_m + L_m)} \qquad (1)$$

C_p and L_m are the specific or latent heat of fusion, respectively. A is the absorption coefficient of the powder material and I_0 the intensity of the laser beam.

b) A lower powder layer density (coarse powders) leads to generally higher effective powder layer thicknesses [5], which reduces the amount of energy reaching the underlying surface. The reduced thermal penetration depth prevents a fast (and possibly deep enough) re-melting of the underlying surface, which would allow a reliable connection of the layers, potentially resulting in more inhomogeneous regions like cracks and incomplete fusion in the material. Such cracks have their long axis in parallel to the layer orientation so that external loads perpendicular to the layers open the cracks, resulting in a decrease of the K_{IC} value. Consequently, the strength in the vertical direction (90°) is reduced, especially for coarse powders.

Regarding the breaking elongation (Figure 6, right) there is a tendency that an amount of coarser particles is generally helpful to increase the ductility, although this effect is less pronounced for higher layer thicknesses. This can be an evidence for a weaker bonding of the different layers. The differences should be less distinct for laser sources with a higher laser beam quality M^2, as for such beams deep penetration welding occurs. However, the size range of particles has to remain limited as for broader size distributions the mechanical strength is reduced (e.g. powder type 3). However, in order to assure that coarser powders lead to a reduced re-melting of the underlying layers, additional investigations have to be undertaken.

General considerations

The general comparison of the dependency of the mechanical parameters for different part orientations (0°, 45°, 90°) shows clearly that there is a anisotropy in Additive Manufactured metallic parts [2, 4, 10].

We have to point out that at the moment, the real reasons for the anisotropy and the differences in the mechanical behaviour due to different powder granulations are not well understood. Further aspects have to be taken into consideration such as e.g. the shape and the sizes of the metallic grains. Therefore, the Petch-Hall relationship shows that finer grains result in an increased mechanical strength. This is in fact the case for the 0° tensile specimens, where the external load / stress is perpendicular to the orientation of the very fine and thin dendrite grains. In contrast, for the 90° tensile specimens, the external load is in parallel to their orientation. In this case, the long dendrites act principally as bigger grains resulting in a lower mechanical strength than for the 0° specimens.

V. Conclusion

The powder material does affect the properties of Additive Manufactured metallic parts. Beside the productivity, the scan surface quality as well as the mechanical properties are dependent on the particle size distribution used. In order to optimise a powder material for SLM processes, the following aspects have to be taken into account.

- Resulting part density in relation to the scan speed (productivity)
- Scan surface quality, affecting the quality of the powder layers and part itself
- Mechanical parameters

In all cases, an amount of fine particles is needed in order to optimise the part properties (density, scan surface quality and mechanical strength), where as the amount of big particles has to remain limited in relation to the effective powder layer thickness. Generally, fine particles can easily be melted and therefore are beneficial for high part densities, process productivity and scan surface quality. As a consequence, a high mechanical strength can be expected. In contrast, bigger particles are beneficial for higher breaking elongations (Figure 6) nearly reaching the values of conventional material in the case of the investigated stainless steel 316L. Therefore, the selection of suitable particle size distributions has to take care of these contrary effects. Some guidelines for the selection of suitable particle size distribution are given in Spierings [5]. Furthermore, current results clearly show that the particle size distribution of powder materials should be covered by a coming

standard for Additive Manufacturing Processes.

VI. Outlook

Additional measurements have to be undertaken in order to investigate the real powder layer thickness and its density. The effect of different particle size distributions on the thermal behaviour of the effective powder layer thickness has to be investigated in detail using specific simulation analysis.

It is expected that fracture mechanical effects highly affect the mechanical behaviour of the material. Therefore, additional investigations into the dependency of K_{IC} on the layer orientation are needed. This has to be aligned with analysis of the effect of grain size (Petch-Hall) in order to differentiate between these contributions.

In order to complement the comparison of different powder grades, further analysis of the dynamic mechanical behaviour are needed.

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