

AGGLOMERATION TENDENCY ANALYSIS OF STAINLESS STEEL POWDERS FOR DMLS PRODUCTION USING SEM IMAGING

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ABSTRACT

Direct Metal Laser Sintering (DMLS) technology is gaining ground in the automotive industry, especially in areas where a high strength-to-weight ratio is of paramount importance, such as racing cars and limited production supercars. It has the advantage of being able to create complex, weight-reduced components. In this research, we present a scanning electron microscopy study of a powder feedstock of a given particle size. The state of the new feedstock and the effect of cyclic reuse are investigated and the changes are analysed. DMLS technology enables virtually waste-free production, as the powder can be reused after sieving, with the exception of the supports used for undercuts. The aim is to investigate whether the particles tend to agglomerate and thus form larger irregular clusters. It is well known in the literature that clusters adversely affect powder fluidity, spreadability and laser energy absorption, suggesting that the quality of the powder is a fundamental determinant of the properties of the final product. Clusters should be identified and investigated from both a scientific and a technological point of view. The investigated powder material is Oerlikon MetcoAdd 17-4PH-A, a martensitic stainless steel feedstock produced by gas atomization.

KEYWORDS

3D metal printing, reused powder, agglomerate, SEM - Scanning Electron Microscope, DMLS

1 INTRODUCTION

Nowadays, additive manufacturing technologies are also rapidly advancing in the field of metal 3D printing. The 3D metal printing technologies with the highest potential are selective laser melting (SLM) or direct metal laser sintering (DMLS) [Jhinkwan 2023].

Both terms are accepted in the scientific literature, as in the early stages of the technology, users used one or the other depending on their geographical location. The term "SLM" was commonly used in Europe, while "DMLS" was more common in the United States. Thus, today these terms are used synonymously [Gibson 2021].

In DMLS technology, a laser selectively melts and fuses the contours of fine metal powder layers, typically 20–100 µm thick, to build up the part layer by layer [Vinoth 2019]. The process involves repeated melting and solidification cycles, which affect both the microstructure and the dimensional accuracy of the parts [A. Simchi 2006]. The cyclically repeated

thermal effects also influence the properties of the powder feedstock, for example, they can affect the morphology of the particles, their size distribution, and their tendency to form agglomerates. Thus, they can play a decisive role in determining the final quality of DMLS products. Due to the repeated recycling of the powder, it is therefore justified to map its behavior [Swain 2019].

In addition, DMLS technology offers numerous advantages that make it suitable for the direct production of end-use parts. It allows for the use of a wide range of metal materials and the creation of lightweight, geometrically optimized parts. With virtually zero waste and the possibility of reusing sieved powder, DMLS can contribute to a sustainable manufacturing structure by minimizing material waste while maintaining mechanical strength and design flexibility [Simchi 2003].

The (DMLS) technology is increasingly used by industry, specifically in areas where the weight/strength ratio is of high importance and where non high volume production is required. The advantage of DMLS over conventional manufacturing technologies is that it is considered a constant manufacturing cost, and the technology has gained traction in areas such as prototype vehicle manufacturing, motorsport and the world of limited production supercars [Duda 2016].

The present research focuses on the investigation of a stainless steel powder of a given particle size, as the properties of the powder have a great influence on the final mechanical properties of the finished product [Gao 2020]. The size and distribution of the grain sizes essentially determine the thickness of the construction layer. The thinner the powder layer, the stronger the bonding between the layers, thus achieving a higher final density, but if the value is too low, the speed of the manufacturing process becomes too slow. In DMLS technology, the intensity of the laser beam is adjusted so that the new powder layer melts and bonds to the previous layer during re-melting, thus creating the right bond between the layers [Buchbinder 2014].

Several studies have observed that finer grains can absorb a greater amount of laser energy, which can result in higher sintering rates. The laser energy output optimised for fine grains is not able to melt the larger, clumped grains properly, impairing porosity and thus the final mechanical properties [Simchi 2006]. However, prior to the manufacturing process, it is necessary to set the grain size to the appropriate layer thickness for the production process, which can be achieved by using different types of sieving equipment. DMLS technology can enable near-waste-free production, as the powder can be reused after sieving, except for the supports used for the undercuts [Brandl 2012].

Manfredi and his colleagues observed that the powder can clump, even after screening processes between prints, if the material is prone to it [Manfredi 2013]. The size, surface area and volume of the agglomerated particles can be significantly different from the optimum, resulting in adverse porosity and reduced final part properties. In this case, the laser power optimized for the finer powder particles is not able to fuse the layers properly [Manfredi 2013].

In this study, we examined Oerlikon MetcoAdd 17-4PH-A stainless steel powder to assess its agglomeration behavior during reuse. To this end, we examine the powder used to print our DMLS test samples. Using SEM examinations, we compare the properties of virgin powder with those of used powder, examine the characteristic differences, and perform analyses [Quinn 2019]. We observe the composition and characteristics of the new material and compare it with the properties of the used powder, looking for characteristic changes, paying particular attention to the search for agglomerates [Gu 2008].

The aim is to investigate whether the particles are prone to agglomeration, subsequent sticking together, and thus the formation of larger, irregular clusters after sieving [Huang 2022]. There are scientific and technological reasons for screening and testing clusters. Several studies conclude that it is not sufficient to examine the relationship between technological parameters and the final product, as the effects of powder, storage, and reuse are significant and influence the final results, and therefore these must also be examined [Fotovvati 2018]. Contaldi et al. have investigated the behaviour of stainless powder and observed that the reusability of powder varies, some materials are well tolerant to cyclic physical and chemical effects, while others show significant variations after a period of time and have a large influence on the properties of the final product, so it is justified to investigate the virgin powder and the reused powder [Contaldi 2019]. The agglomeration of metal grains can occur due to several factors, including electrostatic attraction, surface oxidation, air humidity, and partial melting during laser processing or powder handling. Repeated thermal cycles and mechanical effects during powder reuse can further promote cluster formation, especially for fine particles smaller than 20 μm , which adhere to larger grains [Szabó 2024]. The designation "-45 + 15 μm " refers to the particle size range of the powder, which means that the powder consists mainly of particles with a diameter greater than 15 μm but less than 45 μm . This particle size range is commonly used in DMLS technology because it provides a good balance between powder flowability and layer density. For powders with this distribution, the recommended layer thickness is typically 20–40 μm , which is about half to two-thirds of the average particle diameter, ensuring uniform distribution and melting. Based on our practical experience, the 45 μm vibrating screen initially used became clogged very quickly, probably due to stuck or partially agglomerated particles. Based on the manufacturer's technical recommendations and our industrial experience, we therefore used a 63 μm screen, which effectively removed oversized or agglomerated particles while ensuring adequate powder flow at a layer thickness of 25 μm . These practical observations led directly to our current research, which investigates the agglomeration tendency of the powder and its behavior during reuse.

2 MATERIALS AND METHODS

In this chapter, we present the virgin powder and equipment used in the research.

2.1 Virgin powder

The virgin powder used in this study is Oerlikon MetcoAdd 17-4PH-A. Its main properties are summarized in Table 1.

Properties	
Nominal Chemistry	Fe 17Cr 4.5Ni 4Cu 0.3(Nb / Ta) 0.07C
Material "Norm" (Similar Chemistry)	AMS 5643
Nom. Particle Size Distr.(μm)	-45 +15
Manufacturing Method	Gas Atomization
Product Form	Powder
Recommended Process Technology	Powder Bed Fusion - Laser Beam

Table 1. Oerlikon MetcoAdd 17-4PH-A important properties

The virgin powder, as described by the manufacturer, is a martensitic stainless steel powder with a chemical composition similar to AMS 5643. The production method of the virgin powder is gas atomization, which results in an almost perfectly regular spherical virgin powder. The grain size of the feedstock corresponds to the thickness of the layer used in our production. The feedstock is sieved through a 63 μm sieve to obtain the appropriate layer thickness.

2.2 The DMLS machine and test pieces

We use the Orlas Creator DMLS machine to produce the parts. The recommended settings of the Oerlikon MetcoAdd 17-4PH-A material were used for the production of the test specimens and the material for the material testing was taken from this machine after the production process. The test pieces produced were circular rings 5 mm high, with an outer diameter of 90 mm and an inner diameter of 80 mm. The main manufacturing parameters are shown in Table 2.

ORLAS Creator	
Laser Type / Power	Yb Fibre Laser / 250W
Beam Diameter Expander	40 μm
Laser Percentage Power	50%
Mark Speed	1200 mm/s
Scanner Overlap	80%
Hatching Base Angle	45°
Boundary Offset	80 μm
Layer Height	25 μm

Table 2. Main production parameters of Orlas Creator machine

The workpiece of the DMLS machine is shown in Figure 1. It is characterised by a circular construction platform and work area. The powder dispenser moves in a circular path, feeding the virgin powder or reused powder from the first roller and building the part on the middle roller. The powder overflow is located downstream.



Figure 1. Orlas Creator DMLS machine workbench

2.3 Scanning Elektron Microscope (SEM)

The SEM images were taken with a ZEISS Sigma 300 VP. The ZEISS Sigma 300 VP Gemini is a scanning electron microscope

(SEM) capable of high-resolution image processing and material analysis. The central unit of the microscope is Zeiss' proprietary Gemini electron optics system, which provides excellent image quality even at low accelerating voltages. The microscope can be equipped with multiple detectors, even for material composition analysis. The resolution is up to 1.3 nm. This microscope is ideal for materials science research and analysis, such as microstructural analysis of metals, alloys or additive manufacturing powders. The machine is shown in Figure 2.

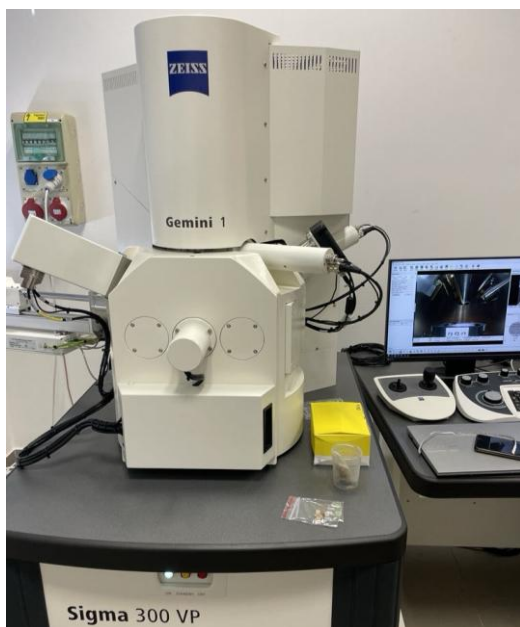


Figure 2. Zeiss Sigma 300 VP

The measuring equipment has a special sliding table. This is shown in Figure 3.

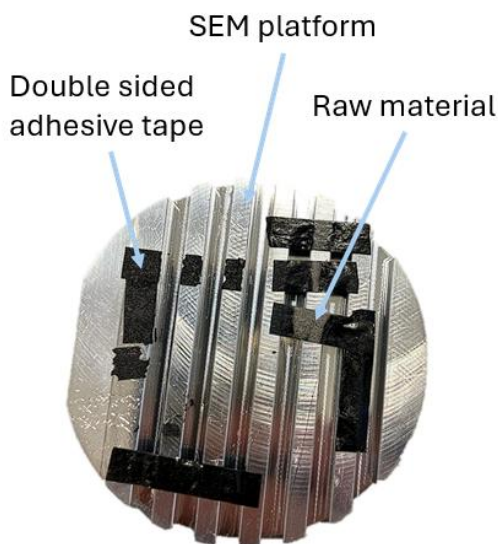


Figure 3. SEM machine's baseplate is useful during measurements with dust

Measurement samples can be attached to this with the help of various instrument components, but in our case the fine grain powder could only be properly attached with special double-sided adhesive. Thus, the powder to be measured was sprinkled on the adhesive strip, and the amount that did not

bind to the adhesive was removed, so that the particles tested during the measurement remained stable.

3 RESULT AND DISCUSSIONS

Based on our SEM examinations, the new, unused 17-4PH powder predominantly exhibits a nearly spherical morphology, with minor surface irregularities in places. According to our observations, fine particles that are an order of magnitude smaller adhere to the large particles, but their effect is negligible. No significant agglomeration tendency was observed. This was followed by DMLS production, during which we produced three workpieces. The virgin powder was recycled during production and sieved at the end. After each production cycle, we filtered the dust and only refilled the material from the construction area and the overflow compartment. At the end of the process, we identified three characteristic agglomeration groups in the recycled powder: (i) partial agglomerates (a few medium-sized particles adhering to larger base particles), (ii) completely fused, irregular large particles, and (iii) transitional, partially fused groups. Their size generally exceeds the sieving limit, so they can be removed by appropriate sieving. Manfredi's research came to a similar conclusion, namely that particles with a diameter of less than $10\text{ }\mu\text{m}$ tend to agglomerate, leading to the formation of $60\text{--}80\text{ }\mu\text{m}$ clusters [Manfredi 2013]. This can impair the flowability of the powder and adversely affect the final density achievable with the DMLS process, especially at the $25\text{ }\mu\text{m}$ layer thickness used in the study. Figure 4 shows a critical agglomeration and the manufacturing defect it caused.

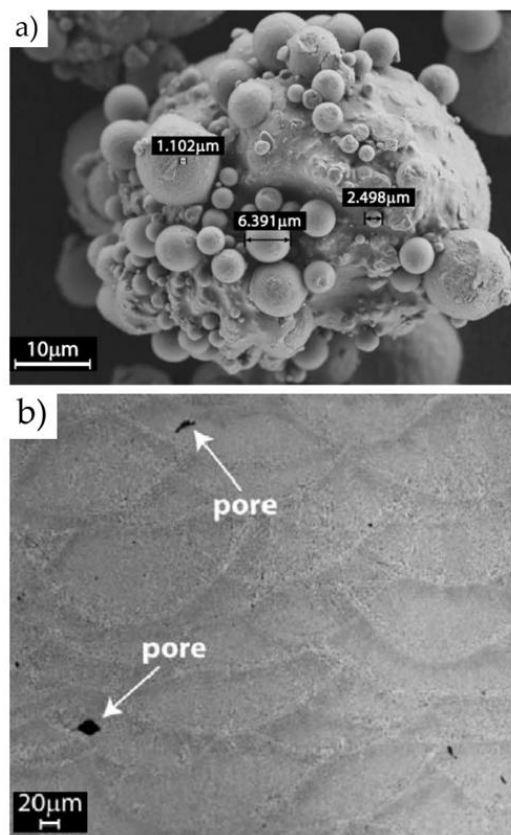


Figure 4. a) SEM image of the agglomerated grain [Manfredi 2013]
b) The material defect caused [Manfredi 2013]

Contaldi and colleagues investigated the reusability of stainless steel powders and found different properties for similar types of materials [Contaldi 2019]. They worked with two types of

stainless steel powder: martensitic (PH1) and austenitic (GP1), and compared their chemical and physical properties, as well as the mechanical properties of parts made from them, over ten DMLS cycles. After each manufacturing cycle, the powders were sieved through a 60 μm sieve, and only the material from the build chamber and overflow tray was refilled. The particle size distribution of PH1 shifted to the right during the cycles, indicating agglomeration of smaller particles. This was not the case for GP1 [Contaldi 2019]. This research also confirms the importance of material testing and highlights that reusability is a material-dependent factor. Our goal was to observe the agglomeration tendency of the virgin powder and the reused powder due to reuse cycles. For us, the aim was to observe the propensity of the feedstock to agglomerate.

3.1 SEM analysis of the virgin powder

We started by SEM testing of the brand new, never-used base core. The results are shown in Figure 5.

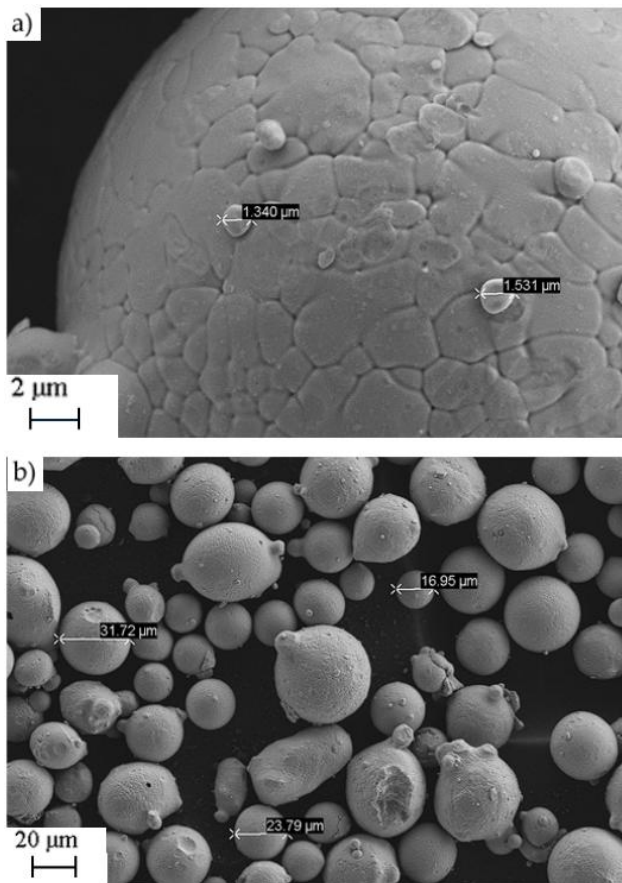


Figure 5. a) SEM image of the surface of the virgin powder
b) SEM image of the virgin powder [Szabó 2024]

The figure above shows that the virgin powder meets the requirement, although the fresh powder granules are not perfectly spherical. The structure of the powder can be described as largely homogeneous and no major lesions were observed. However, the shape of the powder deviates from the ideal spherical shape, with a high number of oval grains and irregularly shaped grains. These grain types do not favour the production process. In the higher resolution image, the grain surface is observed to be not perfectly flat, and the smaller grains of a few μm adhere to the larger ones, showing agglomeration tendencies. The range of powder grain sizes corresponds to the thickness of the print layer we used. This

agglomeration tendency seems to be negligible for the new powder.

3.2 SEM analysis of the reused powder

There are several steps to testing the reused powder. After printing, this powder was lifted out of the used powder container and tested. The first step was to take remote SEM images, looking for agglomerated, larger particles, and then to analyse these particles and classify the lesions.

The first step is shown in Figure 6. This is a more distant image with the outliers marked. These are then examined one by one at the appropriate magnification.

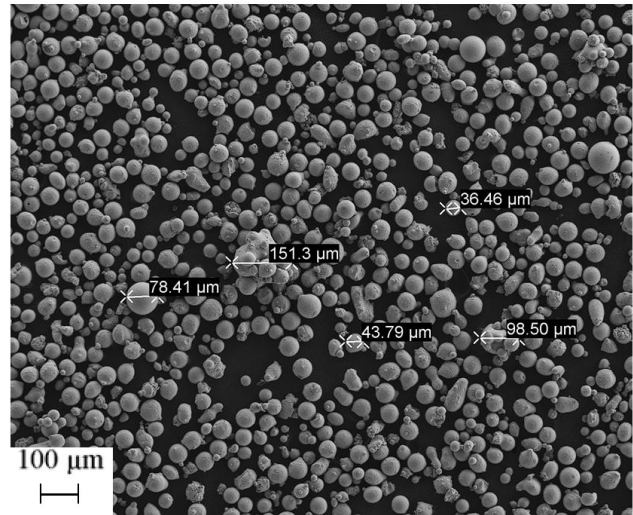


Figure 6. Remote SEM image of the reused powder

The first group we observed consists of a large basic particle and adhering smaller particles. These are partially agglomerated grains. The base particle has a regular spherical shape, but its size exceeds (92.98 μm) both the sieving and printing sizes. The two smaller grains adhering to it still fit within the powder tolerance (45.32 and 34.04 μm), but their shape is irregular, one oval and the other two fused elements of average grain size. This is illustrated in Figure 7. The basic grain of this partial agglomeration can be filtered out by sieving (the adhering grains alone would probably not hang on the sieve).

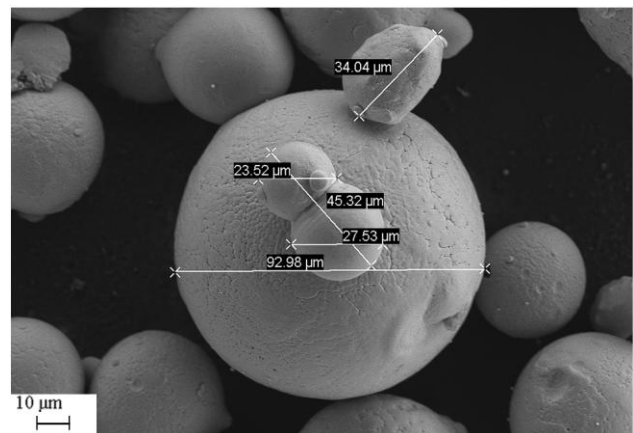


Figure 7. Partially agglomerated large particles [Szabó 2024]

The next group is a large purely fused particle with a geometry that is not a regular shape. However, here again a regular spherical base body is observed, which is large and has a

regular profile. Associated with this are several smaller grains which are clearly fused to the parent grain. Together they form a large irregular grain (149.3 μm). This grain does not meet the dimensions required for production and can be filtered out during sieving. This is shown in the following figure.

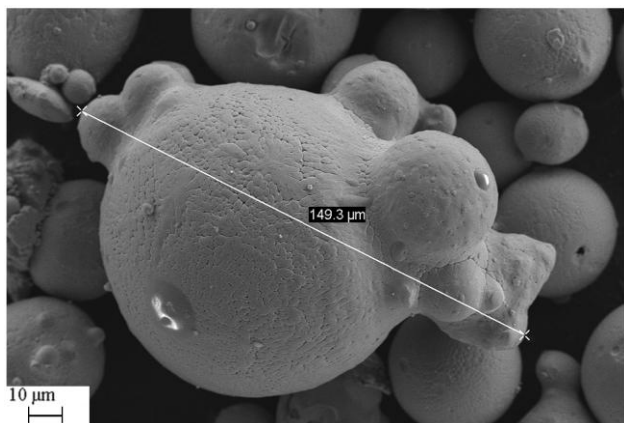


Figure 8. Large completely fused particles [Szabó 2024]

The third group observed is the partially fused and clumped group. They are in a transitional phase. They are shown in Figure 9.

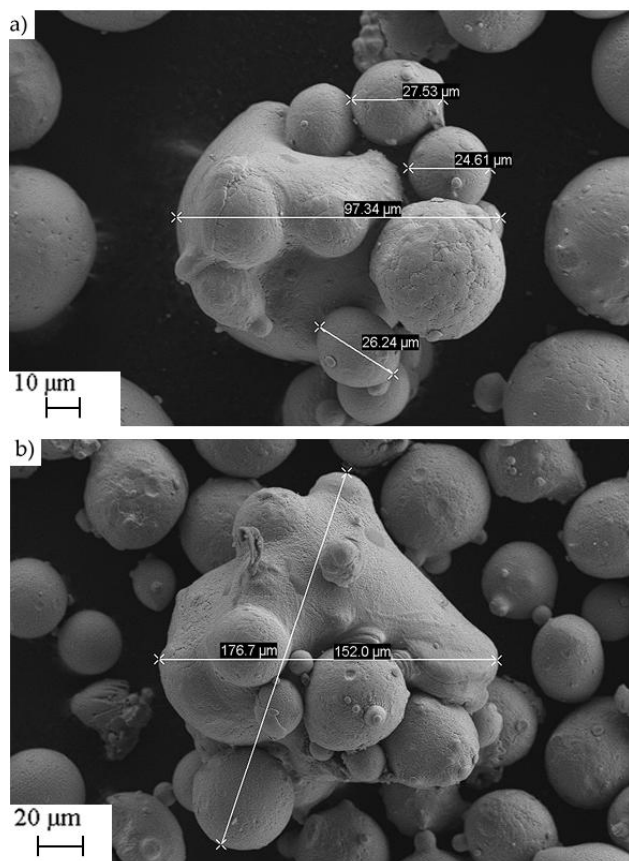


Figure 9. a) Partially fused and agglomerated granules in a state of transition
b) Partially fused granules of large size

In Figure 9(a), we observe a fused basic particle with smaller, average-sized particles attached. The irregularly shaped grain formed is 97.34 μm and can be filtered out by sieving. The adhering grains are less than 30 μm and do not hang on the sieve by themselves. Figure 9(b) shows a partially fused grain

with a large size and irregular shape. The associated grains are largely fused to it. This particle can be filtered out by sieving due to its large size (dimensions above 150 μm).

4 CONCLUSIONS

In this study, we examined the composition and behavior of Oerlikon MetcoAdd 17-4PH-A, which was our base material for research purposes. We took samples from both virgin powder and reused powder. In the DMLS process, the powder has a decisive influence on the properties of the end product. We used SEM images for a detailed, high-resolution analysis of the virgin powder and reused powder. Our goal was to examine the agglomeration tendency of virgin powder and reused powder. The reused powder is exposed to physical and chemical effects during the manufacturing process (heat, physical impact, moisture, etc.), which can change its reuse properties.

The results show that the grains of the virgin powder are not perfectly spherical and contain a large number of irregularly shaped and oval grains. However, the vast majority of the particles fall within the desired size range, but the irregularity of the powder surface and the adhesion of smaller particles to larger ones is already observed in this state, but the effect is negligible, because only particles of a few μm adhere to larger ones.

We identified three agglomeration groups for the reused powder:

- In the partially agglomerated case, a few average-sized particles (approximately 30 μm) adhere to the large base particle and together form a large, irregularly shaped particle that exceeds both the upper size range of the powder and the sieving size. The number of agglomerated particles is not exceptionally high and is only observed in the powder used, but even in small numbers, these particles can significantly weaken the mechanical load-bearing capacity of the component. This phenomenon can be eliminated by sieving, and is unlikely to occur on its own, but long-term tests are required to investigate this.
- In the case of complete fusion, a large, irregularly shaped particle is formed that is significantly larger than the sieve size, so this can also be avoided and does not occur on its own.
- Partial agglomeration and coalescence result in completely irregularly shaped particles in which transitional states develop. The particles are large and do not occur on their own, they can be filtered out by sieving.

Overall, the virgin powder meets the expected requirements. In its new state, it does not show any significant agglomeration tendency, only minimal clumping can be observed, which does not exceed the upper limit of the powder. During reuse cycles, we observed different types of agglomeration that could negatively affect the final strength or dimensional accuracy of DMLS parts. Most of these were partially fused particle groups, which is a natural process in DMLS manufacturing. Of the groups we found, the first group was the partially agglomerated group, where medium-sized particles stuck together. This process did not show any significant change during the three reuse cycles we examined, so a long-term series of experiments

involving multiple reuse cycles is necessary to investigate this tendency.

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