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RESEARCH OF 316L METALLIC POWDER FOR USE IN SLM 3D PRINTING

ABSTRACT

3D metal printing is an increasingly popular production of steel parts. The most widespread and most accurate method is SLM (Selective Laser Melting), which uses metallic powder as the input material. The article is dedicated to researching the supplied powder from Renishaw. The powder is made by gas atomization and 3 phases of powder (virgin, sift and waste) that are present in the SLM process are examined. Powder morphology by SEM electron microscopy is investigated and the porosity of the powder is measured by optical method. Next, the powder grain size fraction is examined. In conclusion, there are recommendations and other directions of possible research. The main quantitative result from research is that, in general, small particles are reduced in the sift powder and the number of larger particles is increased, but the powder is still usable for further use.

Keywords: metallic powder; 3D printing; SLM; morphology; porosity

INTRODUCTION

Today's trend is to produce increasingly complex and quality components. There are greater demands on production speed, accuracy and overall quality of the final product. This unstoppable progress has given rise to a new progressive technology called additive production. Additive manufacturing is a technological process based on the gradual addition of material (layer-by-layer) under specified conditions [1]. One of the additive offshoots is the production of metal parts. For the production of metal parts, the SLM (Selective Laser Melting) method is the most accurate and affordable. Understanding the SLM process is extremely challenging, not only because of the large number of thermal, mechanical and chemical phenomena that take place here, but also in terms of metallurgy. The presence of three states (solid, liquid, gaseous) complicates the ability to analyze and formulate a model formula for proper simulation and prediction of part performance when printed. Since the SLM process operates on a powder basis, this process is more complicated by another factor compared to the use of other bulk material. The properties of the used printing powder define to a large extent the quality of the finished part [2]. To make a homogeneous powder layer
with a high density are important swaging the following parameters: particle shape, particle size and particle size distribution. In order to achieve the best packing density, it is advantageous to use the spherical shape of the powder particles (production by gas atomization) due to the flowability of the powder. It is also possible to improve the packing density by using very small powder particles, which, however, is not appropriate in terms of safety regulations and powder flow. The particle size cannot be greater than the layer thickness [3]. To make a smooth layer with maximum of packing density it is recommended use a bimodal (two-peak) powder distribution at a ratio of 1:7 [4]. However, a compromise is required, a large range of powder particle sizes leads to a higher packing density, while a smaller range results in better fluidity, presents Meiners in her dissertation [5].

The input material itself has a big impact on the final properties of the printed part using SLM. It is therefore important to carry out research on the powder delivered before printing and to know its properties and characteristics [6, 7]. Consolidation and final structure are determined by parameters such as morphology, particle size and distribution, flowability, and porosity of particles. It is also important to apply the powder evenly on the base plate [8]. ASTM International [9] also specifies what metal powder should have for use in additive technology.

MATERIAL AND EXPERIMENTAL METHODS

Three phases of metallic powder were investigated for research purposes. The first tested phase was the state, as the powder was supplied by the manufacturer, so-called virgin powder. The second phase of the test powder was about 30 times sieved, where have been predominantly observed impurities and other imperfections of the powder formed after laser melting process. The last stage of powder testing was waste that had settled in the sieve and was no longer being processed and disposed of.

Characterization of the supplied powder

It is a non-magnetic austenitic stainless steel, which contains a very low percentage of carbon and is alloyed with chromium, nickel, molybdenum and other negligible elements, see Table 1. Made by gas atomization method. The atomization process is characterized by the disintegration of the melt stream by nitrogen, helium or argon flowing from the nozzles under pressure, where a crucible with a charge is placed in the vessel above the atomization chamber. The charge is first heated and melted to the desired temperature above the melting point. The melt is then sprayed through the Laval nozzle through the clean gas into the atomization chamber. The gas is accelerated to the speed of sound in the nozzle, and when it comes into contact with the melt stream, it sprays it into fine drops and at the same time extracts heat from the melt, thereby solidifying it into a powder. The powder is then collected into a basket under an atomizer. The atomizer is able to powder up to 99.5% of the feed material. The remaining 0.5% is collected on the atomizer walls and in the filters. The advantage of this method lies in the resulting grain shape, which is regular and spherical [10]. The powder manufacturer and supplier also indicates the individual grain size of the powder of 45 ±15 µm and declares its properties, see Table 2.
Table 1. Chemical composition of AISI 316L powder declare by supplier [11]

| Element | Fe  | Cr | Ni | Mo | Mn | Si  | N   | O   | P   | C   | S   |
|---------|-----|----|----|----|----|-----|-----|-----|-----|-----|-----|
| Mass [%]| Balance | 18 | 14 | 3  | < 2 | < 1 | < 0.1 | < 0.1 | < 0.045 | < 0.03 | < 0.03 |

Table 2. Powder properties indicated by the supplier [5]

| Property                      | Value            |
|-------------------------------|------------------|
| Density                       | 7.99 g·cm⁻³      |
| Thermal conductivity          | 16.2 W·mK⁻¹      |
| Melting range                 | 1371 °C - 1399 °C|
| Coefficient of thermal expansion | 1610⁶ K        |

Morphology of powder

Powder morphology is an important characteristic that affects the application of metal powder by laser in terms of fluidity and packing density. Characteristics of morphology depend on the method of powder production (gas atomization, plasma atomization, plasma rotating electrode). These methods produce predominantly spherical shapes of particles. It is generally stated that the plasma atomization method and the plasma rotating electrode form better and more spherical particles with smaller shape irregularities. These shape irregularities are called "satellites". Zhong et al. [12] claims that satellites arise because of the difference in solidification rate between smaller molten particles that adhere to partially melted particles of larger size.

The shape of the individual particles was measured and evaluated by SEM (Scanning Electron Microscope) on a JSM-6510 device. Visual quality evaluation was performed to determine the spherical quality of the powder and the content of the satellites. When working with SEM, a small amount of powder was applied to a transparent adhesive tape that was adhered to the sheet. The plate was then inserted into the SEM of the microscope where vacuum was formed and the electron beam was activated. The virgin powder, sifting powder, and the waste which did not pass through the sieve was investigated. Three phases of the powder were examined using a SEM microscope. The first phase consisted of virgin powder, the second phase was powdered several times (~ 30x) and the last phase was examined for powder which accumulates on the sieve, ie waste. The measurement of virgin powder (Fig. 1) reveals that the production of powder by gas atomization is not perfect and the shape of some particles is not perfectly spherical. It is also possible to observe satellites (small particles glued to larger ones, Fig. 2), which are again a defect of the production method.
The individual investigation of these phases revealed that the shape of the particles is not always isometric, and irregular, elongated and cylindrical shapes appear in both spherical and oversized powders in addition to spherical particles. Also, combined particles and particles with satellites appeared in both phases, but the number of particles with the satellites increased in the oversized powder and the number of irregular particles also increased. Another interesting phenomenon was manifested in the sieved powder, where particles with a smoother and more spherical surface were observed than the original particles. This is most likely due to the melting and solidification process that is specific to AM. Fig. 3 shows the results of observations of powder morphology.
Porosity of powder

To investigate the porosity of the 316L metallic powder, an optical method was used where the powder sample was embedded in a resin, after curing was a 1 mm layer abraded to cut the powder particles approximately in half to reveal the internal porosity, and then polish the prepared sample with diamond paste. In this time there is no standard to quantify the measurement to measure the intrinsic porosity of a metallic powder, so the available KEYENCE microscope and the supplied analysis software have been used. Several microscopic images were taken on random areas of varying magnification, then converted to black-and-white 8-bit depth. The images thus captured were inserted into the analysis software. Pore assessment is not easy because of satellites and irregular particle shapes. So it is a very individual decision. In general, pores must be closed from 3/4 of their circumference to be considered pores [13]. Particles that do not comply with this rule are automatically considered irregular particles. An example of open and closed pores that correspond to the rule is indicated by a red arrow in Fig. 4 on the left side, an example of open cavities that do not conform to the rule and has been excluded from measurement, is shown by the red arrow in Fig. 4 on right side. The software analysis of several images determined the total powder density of 99.785%.
Another interest in researching the porosity of the metallic powder was to measure the size of the individual pores. Measurements were taken on images with 1000x zoom and KEYENCE software was used again. Examples of measurements are shown in Fig. 5. The size of the longest axis was decisive for non-spherical pores. All pores starting at 5 µm were recorded and the arithmetic deviation was calculated. The limitation of this measurement lies in image resolution when measuring small particles. Based on this limitation, pore sizes of about 5-8 µm should be taken with some uncertainty.

After manual measurement, a histogram was generated showing that the 15 µm pore size was most present in the metallic powder particles. The largest measured leek was 30 µm. Table 3 shows a histogram and individual pore sizes.
Table 3. Measured values of porosity of powder particles

| Statistics [μm] |          |
|----------------|----------|
| Mean Ø         | 13.48    |
| St. Dev.       | 5.640    |
| N              | 44 [pcs] |

Powder grain diameter fraction

The optical method using the Keyence VHX-5000 microscope was chosen to measure the grain size distribution. The measured data were mainly compared for the virgin powder phase and the powdered phase (~ 30x). Each powder sample was coated with a thin layer on a glass substrate that was illuminated coaxially and underneath. Measurements were always made at five random locations with 200x magnification. Each area evaluated contained about 200 particles from which static analysis was performed. Image evaluation was done using software supplied by Keyence see Fig. 6.
cut-off limit within which the size falls to 10, 50, 90% of the measured particles. The distribution range or width of distribution $Span$ is determined by calculation (1):

$$Span = \frac{d_{90} - d_{10}}{d_{50}}$$ \quad (1)

as shown by optical image analysis, by repeatedly sieving the powder, the average particle size increases only slightly. The sieving station is trying to separate agglomerated or molten particles larger than 45 μm, but due to irregular particles that are longer in one direction and shorter in the other, they still fall through the mesh of the sieve. From the results (Table 4 and Table 5), it can be seen that, in general, small particles (<10 μm) are reduced in the sift powder and the larger particles are increased. Distribution has the character of Gaussian distribution.

Table 4. Distribution of particles in virgin powder

| Statistics [μm] | 
|----------------|
| Mean $\phi$ | 26,30 |
| St. Dev. | 9,025 |
| $d_{10}$ | 14,78 |
| $d_{50}$ | 26,29 |
| $d_{90}$ | 37,81 |
| Span | 0,876 |

Table 5. Distribution of particles in sift powder

| Statistics [μm] | 
|----------------|
| Mean $\phi$ | 31,73 |
| St. Dev. | 8,335 |
| $d_{10}$ | 18,18 |
| $d_{50}$ | 29,05 |
| $d_{90}$ | 39,93 |
| Span | 0,748 |
The sift powder showed an increase in particle volume and surface area while circularity decreased, indicating that virgin powder generally has a higher sphericity. Much more larger particles were measured in the waste measurement, as this sample contained particles that were already partially oxidized or sintered together or otherwise thermally affected. Table 6 shows the distribution of these particles and in Table 7 a summary of all the results is shown.

**Table 6. Distribution of particles in waste powder**

| Sample   | Mean diameter [μm] | $d_{10}$ [μm] | $d_{50}$ [μm] | $d_{90}$ [μm] | Distribution Range [-] | Circularity [-] |
|----------|--------------------|---------------|---------------|---------------|------------------------|-----------------|
| Virgin   | 26,30 ± 9,025      | 14,78         | 26,29         | 37,81         | 0,876                  | 0,8449          |
| Sift     | 31,73 ± 8,335      | 18,18         | 29,05         | 39,93         | 0,748                  | 0,79            |
| Waste    | 61,98 ± 19,37      | 37,88         | 61,98         | 86,09         | 0,779                  | 0,8454          |

**Table 7. Summary of measured values**

| Sample   | Mean Ø [μm] | St. Dev. [μm] | $d_{10}$ [μm] | $d_{50}$ [μm] | $d_{90}$ [μm] | Span [-] |
|----------|-------------|---------------|---------------|---------------|---------------|----------|
| Virgin   | 61,98       | 19,37         | 37,88         | 61,98         | 86,09         | 0,779    |

**CONCLUSIONS**

Research on powdered metal is a necessary basis for further research into additive technology. General knowledge of powder morphology, which is largely dependent on the powder manufacturing method, has been confirmed [15, 16]. The investigated AISI 316L stainless steel powder was produced by gas atomization and showed numerous particles with satellites. Before investigating the distribution and defects of the metallic powder, the porosity of the particles was investigated. The results revealed a porosity density of 99.785% of the particles, which is due to the powder production itself, and the individual pores of the particles were measured. Three phases of powder were examined - virgin powder, sift powder and waste. Certain defects such as particulate fusions, gas impurities, agglomeration, etc. have been observed at all stages, despite the number of sieves (~ 30x). It can be stated that, in general, small particles are reduced in the sift powder and the number of larger particles is increased, but the powder is still usable for further use. This makes SLM ecological and...
economic technology [17]. It is, however, necessary to take into account that the powder may lose quality over time due to oxygen absorption and related oxide formation in the microstructure. This can adversely affect mechanical, chemical and physical properties.

From the research conducted and the results obtained using experimental investigation we can recommend the following measures to achieve the best possible consolidation:

- as spherical particles as possible
- fine surface
- as few satellites as possible (satellite - surface irregularity, smaller particles adhered to the surface of a larger particle),
- low internal porosity
- as few surface pores as possible,
- tight distribution of particles,
- high purity.

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