The Issue of Regeneration of Metal Powder DLMS 3D Printing

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The subject of the article is a comparison of new and used powder for 3D metal printing. The powder is 316L stainless steel manufactured by Renishaw. The powder used was taken from the RENISHAW AM250 printer after use. Powder manufacturer Renishaw recommends using 15-45 micron powder in their 3D metal printers. An important parameter of monitoring is the chemical composition of the metal powder and its changes during the thermal treatment during laser sintering. Another important parameter of a metal powder is its mechanical properties, which determine the flowability, consistency and uniformity of powder application. By using an inert atmosphere for sintering and storing the powder, these chemical changes can be prevented, especially against the formation of nitrides and oxides at elevated temperatures.

Keywords: 3D printing, laser sintering, metal powder, recycling powder

1 Introduction

The powder and its reuse quality is an important area for the automation and economy of the entire 3D printing process. Powder container must be large enough to be able to fill the entire production area and therefore may occur uninterrupted printing. It is important to transport the correct amount of material to apply a new layer without large excesses. It must be ensured that the powder is evenly distributed in the layer, resulting in a thin, smooth and repeatable layer of material and the powder spreading process must not be applied with great force to prevent damage to the previous structure. During the construction of the part, the high temperature in the vicinity of the part to be formed can cause some particles to bond in the powder bed, and this is also related to the possibility of changing chemical properties. Depending on the material, these quality changes may be substantial or so small that the material may be considered infinitely recyclable. [1] [7][10]

2 Description of the RENISHAW AM250

The Renishaw machine has a printing area of 250x250x300 mm3 and can produce layers of 20 to 100 microns. High-performance fiber lasers serve to form metal parts from metal powder by laser sintering (Tab. 1). The powder reservoir has shut-off valves to replenish material during manufacture and is removable. The excess powder container is also provided with valves. Thus, unused metal material can be screened and reused for the construction of a new part. There are safe systems for the treatment of emissions and residual powder in the machine and the process is conducted in a controlled protective atmosphere. The loss of material in the process is minimal since most (about 98%) of the material surrounding the component during construction can be reused after processing in a Renishaw powder conditioning system. [2] [3]

During the construction, an inert atmosphere is created in the building space, which is very important to avoid contamination of the air molecules and to prevent the chemical and physical properties from being affected parts. First, a vacuum is created in the space and then filled with argon. In the process of building them the oxygen content is kept below 0.1%. [3]

Tab. 1 Some technical specifications of the RENISHAW AM 250 [3]

| Specification                        | Value                      |
|-------------------------------------|----------------------------|
| Speed of construction               | 5 up to 20 cm³/h           |
| Scan speed                          | Up to 2000 mm/s            |
| Positioning speed                   | 7000 mm/s                  |
| The diameter of the laser beam on the surface | 70 µm                      |
| Laser power                         | 200 or 400 W               |
| External dimensions of the machine  | 1700 x 800 x 2025 mm (LxWxH) |

3 Description of the manufacturing process

During the construction of the part begins the process by attaching the tray to a powder to the construction and subsequent insertion of the base plate to the workspace. CADU-modeled data is transferred to the machine system and converted into a different format in the software to be used in a 3D printer. [7] Recent studies have shown that 3D metal printing can offer excellent mechanical properties of processed material due to its rapid cooling and unique multidimensional heterogeneous or hierarchical
At the start of the process, a warning is displayed on the machine to see if the safety valves have been opened at the powder reservoir. After opening the safety valves, a vacuum is created in the production area by sucking air and moisture (Fig. 1a). And then the entire space is impregnated with argon, which has a protective atmosphere function (Figure 1b). [4]

After this step, the construction of the part can be started by applying a metal powder over thin layers, whereby the powder fuses by melting at the laser impact sites, e.g. more lasers (Fig. 2a). [4]

After the processing of the layer, the base plate is moved by the thickness of the layer and another unprocessed metal powder layer is applied. [8] After the part is printed, the base plate is returned with the upper halves (Fig. 2b) and the excess powder surrounding the entire part can be removed manually using tools (integrated gloves). Thanks to the integrated gloves, the protective atmosphere is not impaired when handling the powder. [4]

4 The issue of powder recycling

During laser sintering, heating is increased when some of the powder is transformed into gaseous condensate. To eliminate these phenomena, a moderate gas flow passes through the building space during construction. During rapid heating, there may be a risk of splashing from the melt pool when some particles are attached to the building. By setting appropriate parameters in the manufacturing process, this problem can be minimized. When sieving the powder through a sieve in the sifting station, the passage of agglomerates or sinters of the powder is prevented. Relative chemical stability is ensured by an inert environment. [5]

5 Powder characteristics SS 316L-0407 according to manufacturer

316L metal powder is an austenitic stainless steel alloy that contains up to 18% chromium by weight, 14% nickel and 3% molybdenum along with other elements. Carbon content is very low against standard alloy 316L, therefore, steel is particularly resistant to carbide precipitation at the grain boundary and has good welding properties characteristics. Another feature is the high hardness and stiffness, high corrosion resistance and good machinability. This alloy can be used to make plastic injection molds, molds for casting, extrusion matrices, surgical instruments, spindles and screws and various industrial ones included. Other alloy characteristics are listed in Tab. 2 and the manufacturer’s chemical composition the data sheet is in Tab. 3. [3]

| Characteristic                        | Value                  |
|--------------------------------------|------------------------|
| Density                             | 7.99 g/cm³             |
| Thermal conductivity                 | 16.2 W/mK              |
| Temperature range for melting        | 1371 °C up to 1399 °C   |
| Coefficient of thermal expansion     | 16*10⁶ K⁻¹             |
6 Metal powder testing

Powders of 0 to 15 µm and 15 to 45 µm were used for testing. Sample 1 is a powder used for about 2 years in a Renishaw 3D printer. The dimensions of Sample 2 are from 0 to 15 µm and the dimensions of Sample 3 are from 15 to 45 µm.

For each sample, two shots of particles were taken at different scales and then EDS chemical composition analysis from the surface. The particle shape evaluation is made from the images and the chemical composition is compared with the composition given by the manufacturer in the datasheet. Some elements have been excluded from chemical analysis, eg carbon, which is detected in larger quantities because of its content in the powdered tape. Furthermore, fluorine, which is erroneously detected, in the energy proximity of iron. Another element whose detection may be distorted during the test is oxygen, so it is also excluded from analysis. It is to be understood that EDS analysis does not distinguish between sulfur and molybdenum. Detection of increased amounts of silicon may be due to sample contamination. Therefore, only the chromium, nickel and manganese values found are compared. The results of the EDS analysis are shown graphically for each element where the x-axis is the energy of radiation in keV and the number of pulses is plotted on the y-axis. The weight percent elements are also included in the graph.

Sample 1

Electron microscope

Sample 1 was collected behind a sieve with a nominal mesh diameter of 61 µm. Since the material is behind the sieve and also a returnable material, the expected value of the particles is less than 61 µm, which will be relatively homogeneous in shape. It is clear from the images that these are primarily spherical particles, which may have some very small particles welded on them (Fig. 3). The chemical composition of the EDS analysis is shown in Fig. 4.

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**Tab. 3 Chemical composition of powder SS 316L-0407 [3]**

| Chemical symbol | Element name | Weight [%] |
|-----------------|--------------|------------|
| Fe              | Iron         | Recalculation into a whole |
| Cr              | Chrome       | 16.00 up to 18.00 |
| Ni              | Nickel       | 10.00 up to 14.00 |
| Mo              | Molybdenum   | 2.00 up to 3.00 |
| Mn              | Manganese    | ≤ 2.00     |
| Si              | Silicon      | ≤ 1.00     |
| N               | Nitrogen     | ≤ 0.10     |
| O               | Oxygen       | ≤ 0.10     |
| P               | Phosphorus   | ≤ 0.045    |
| C               | Carbon       | ≤ 0.03     |
| S               | Sulfur       | ≤ 0.03     |

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![Fig. 3 Images of scanning microscope](image-url)
Sample 2 and Sample 3

Electron microscope

The dimensions of Sample 2 are from 0 to 15 µm (Fig. 5) and the dimensions of Sample 3 are from 15 to 45 µm (Fig. 6). In both cases, samples were taken from new powders. The results of EDS analysis are shown in Fig. 7 for Sample 2 and Fig. 8 for Sample 3.
Fig. 6 Scanning microscope images 15 to 45 (Sample 3)

Fig. 7 EDS analysis 0 to 15 (Sample 2)
7 Conclusion

In addition to the used powder, the material that is not used backwards (deposited on the filter, spatter, etc.) was also evaluated. For all these samples, after two years of monitoring, it can be concluded that the proportion of Ni and Fe in the powder used gradually decreases as a result of the burn, and the proportion of Cr increases. Mixing with a new powder does not help here, as the guaranteed chemical composition does not have to come out. For this reason, it is recommended that after a certain number of cycles, or after two years, complete powder replacement is performed with a new one with guaranteed chemical composition.

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