ASSESSMENT OF MOISTURE CONTENT AND ITS INFLUENCE ON LASER BEAM MELTING FEEDSTOCK

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ABSTRACT

Additive Manufacturing (AM) techniques are known for building functional parts by adding layers of material. This layer-wise fabrication of metal parts yields freedom of design, weight reduction and product customization. Most of the metal AM processes use powder as feedstock, as small particles give freedom to the process to create all kind of complex structures with high tolerances. However, their (relatively) large surface can be affected by environmental conditions, such as moisture and elevated temperature, and by contamination. In the present research, Laser Beam Melting (LBM) powders are investigated to determine and quantify (i) the typical moisture content in an as-received batch, (ii) how to prevent this kind of contamination by proposing pre-treatments for the powders and (iii) its impact on the LBM process and products. In general, avoiding contamination during the production leads to a better quality and prolongs the product lifetime.

1. INTRODUCTION

Additive Manufacturing (AM) encompasses a group of techniques that build complex functional parts by adding material layer-by-layer. From the industry point of view, the most relevant metal AM processes are Laser Beam Melting (LBM), Electron Beam Melting (EBM) and Directed Energy Deposition (i.e. cladding)[1, 2]. The first two belong to a group called Powder Bed Fusion systems. They use metal powder as feedstock which is smoothly deposited onto the build plate using a wiper mechanism [3, 4]. Powder bed-based systems are sealed and work in a shielded (usually inert) environment. Nevertheless, there is always a risk of contamination within the powders that can be brought to the process. This contamination, namely oxygen and nitrogen pick up, moisture or other external materials, can highly compromise the properties of the final part [5, 6].

Metal powders used in the Powder Bed Fusion processes are in general produced by gas atomization [7]. By this production method the particles normally acquire a rounded and homogeneous shape with a narrow Particle Size Distribution (PSD). Nonetheless, the smaller the particle size is, the larger the relative surface is which yields interaction with gases present in the atmosphere resulting in a highly reactive surface. Figure 1 illustrates the powders life-cycle from its production to the final application: a metal AM process. Handling and storing conditions are a key step to avoid contamination and aging of the material. Some powder distributors deliver the material in HDPE containers very well sealed with inert gas and even silica bags are placed inside the containers to maintain the dry conditions. When the feedstock will be used, the containers are opened, losing the sealing gas, and the silica bags are removed. Therefore, if the (remaining) powder is deposited back to the storage after use in the AM process, the ageing could start due to exposure to the atmosphere. Moreover, the building chamber is sealed and protected with inert gas (i.e. Argon) to avoid any kind of external contamination during the building process. But the powder is melted by a high power source (e.g. laser or electron beam), so there is still a risk of gases pick up, e.g. oxygen or hydrogen due to trapped moisture in the material, or nitrogen.

Figure 1. Metal powders life cycle before becoming an AM part

This paper studies the influence of external conditions leading to moisture and oxygen pickup in the metal powders used for LBM. The influence will be quantified in changes of both composition and morphology. Additionally, the impact of these conditions on the LBM process will be estimated by analyzing the powder flowability. Finally, two drying procedures are proposed as measures to prevent moisture contamination in the feedstock to enter the building chamber.

2. MATERIALS AND METHODS

2.1. Experimental set-up

The aim of this paper is to quantify the moisture impact in four LBM powders: Inconel 718, Ti6Al4V, AlSi10Mg, Scalmalloy, acquired for building Additive Manufactured parts from the following suppliers: Oerlikon for Inconel 718, LPW Technology for Ti6Al4V and AlSi10Mg, and Airbus APWorks for Scalmalloy.

As it was described in the introduction section, the LBM process is very sensitive to contamination and especially to humidity during the printing process. For this reason, a sample of each material was aged in a climate chamber for 72 h under 80% humidity and 50°C. These conditions were chosen as they resemble a usual case where there is a high humidity environment and the build chamber temperature during a regular build job reaches ~50°C. In a previous study for AlSi10Mg by Bauer [8] potassium-chloride and magnesium-chloride solutions were used to age the powder, according to ASTM E104, resulting in morphological changes due to the extreme salty and moisturized conditions.
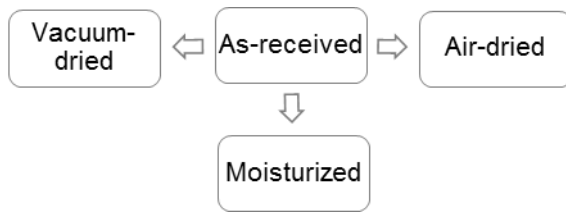
Figure 2. Experimental route to achieve four powder states

The scheme in Figure 2 illustrates the methodology followed for the experimental part. Firstly, a sample of as-received powder was separated to conduct the reference measurements. Next, two samples were dried from the as-received state. The first sample under vacuum conditions at 85°C for 6 h and the second was dried in air at 150°C for 20 min. Finally, a fourth sample was placed in the climate chamber. This methodology resulted in measurements of four different states for all four powders (in total sixteen measurements). The results presented in section 3 are based on 5 or more measurements for each data point.

Figure 3 shows Inconel 718, Ti6Al4V, AlSi10Mg and Scalmalloy powders in the containers after being exposed to moisture in the climate chamber for 72 h. There is a clear relation between the materials condition and its affinity to moisture. The visual inspection reveals that humidity has a strong influence on AlSi10Mg, which shows a light gray powder on the upper layer, while the material on the bottom has a darker gray color, resembling sand at the beach when digging deep. The other three materials do not show this effect, but do form agglomerations that were easily separated with the Turbula: a shaker/mixer.
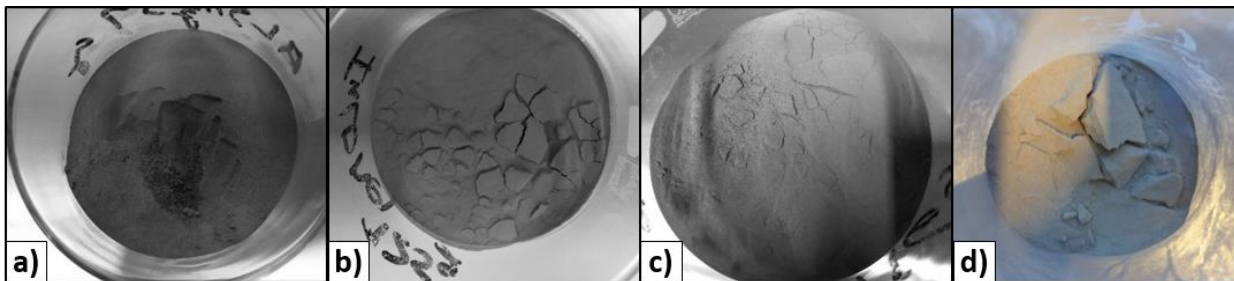
Figure 3. a) Inconel 718, b) Ti6Al4V, c) AlSi10Mg, d) Scalmalloy

2.2. Morphology and composition

Firstly, the morphology (i.e. particle shape) is analyzed with the Scanning Electron Microscope (SEM). Secondly, the chemical composition was estimated with Energy-dispersive X-ray spectroscopy (EDX) for all the states: as-received, vacuum-dried, air-dried and moisturized. The aim of these measurements was to observe the particles surface and irregularities and to detect any potential change that could have taken place due to the drying/aging procedures. Especially the oxygen content in the powders might change.

2.3. Particle size distribution

The particle size distribution of the powders was obtained with the Mastersizer 2000 (according to ASTM B 822-02) [9]. These experiments were carried out in a water based wet dispersion. In addition, to improve the sample dispersion and avoid agglomeration of powders, samples were placed in ultrasonic vibration for about 5-10 min

before the measurements. Moreover, deflocculants as Fluicer PD 96/F and Dolapix CE64 were added to the suspension in order to improve the dispersion of Ti6Al4V and Inconel 718, respectively.

2.4. Specific surface area

There is an inverse relation between particle size and specific surface area of a powder. The finer the particle, the more surface is exposed for a given volume of powder. Furthermore, particles with satellites and surface irregularities such as cracks, porosity and roughness have a greater surface than the theoretically calculated geometrical figure (i.e. assuming perfect spheres) [10].

The Brunauer–Emmett–Teller (BET) measurement estimates the total specific surface area for a powder [11]. Before the measurement started, a degasification process was carried out by heating the sample up to 90°C for 60 min and then to 250°C for 120 min under a Nitrogen gas flow. Afterwards the BET measurement was set up with two test tubes. The first empty tube is compared to the second tube which contains the powder sample. The test tubes were submersed in liquid Nitrogen to cool them down to 77 K. A Nitrogen and Helium gas mix was simultaneously injected in the tubes. A gas like Nitrogen is adsorbed upon clean solid surfaces at low temperatures, which means that the powder particles are covered by multiple Nitrogen layers as if a painting process was executed. The measurement is based on equation (1) to estimate how much Nitrogen is adsorbed by the particle surface.

$$\frac{P}{V_a(P_0 - P)} = \frac{1}{V_m C} + \frac{C-1}{V_m C}\left(\frac{P}{P_0}\right)$$

(1)

where, V_a is the adsorbed gas volume at pressure P, V_m is the adsorbed gas volume when the surface is covered by a monomolecular gas layer, C is a constant and P_0 is the saturation pressure. Measuring the pressures and adsorbed gas volumes (by comparing the filled and empty tubes), the value of V_m can be derived, and that quantity is directly proportional to the (specific) surface area.

The volume was kept constant for measuring the different powder materials. The reason for this is that the particles surface would be the same in the event that all alloys have the same morphology, regardless the difference in density. Several properties are correlated with the specific surface area such as the morphology, particle size distribution and flowing behavior.

2.5. Moisture content and Flowability

The moisture content was estimated following a *mass loss on drying* principle with the Moisture Analyzer, model MS-70 from A&D Company Limited. The sample was placed on a heating plate for 20 min at 150°C with a heating rate of 10°C /min. The samples from the four states shown in Figure 2 (as-received, vacuum-dried, air-dried, moisturized) were dried in the Moisture Analyzer and the weight was tracked during the measurement. Consequently, with the measured mass loss the moisture content M was estimated with

$$M(\%) = \frac{m_w - m_d}{m_w}$$

(2)

where m_w represents the initial sample weight or "wet mass" and m_d the "dried mass"

During the measurements, the samples were taken approximately with the same volume in order to be able to compare the water loss. Therefore, to ensure the same volume in all materials, despite the differences in density, a correctional factor was applied to M (%) to correct for the constant error reported by the manufacturer (0.01 g).

Using the standard Hall flowmeter method (ASTM B213) [12] the flow rate Φ_{Hall} was determined for all four powders in the four states.

3. RESULTS AND DISCUSSION

3.1. Morphology

Figure 4 shows the morphology of all four as-received powders. The first two materials, Inconel 718 and Ti6Al4V, present an homogenous and rounded surface with few satellites. Alternatively, AlSi10Mg shows an irregular shape with satellites. The same investigation was carried out with the moisturized and dried powders, but there were no detectable changes on the morphology due to these processes.
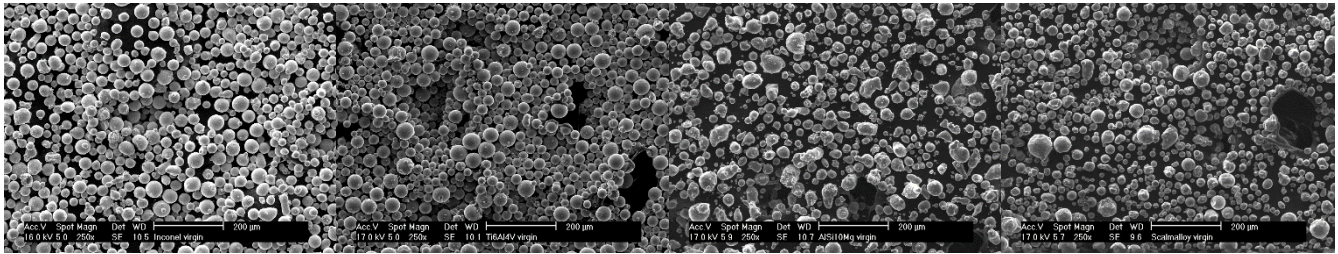
Figure 4. From left to right: Inconel 718, Ti6Al4V, AlSi10Mg, Scalmalloy

3.2. Composition

Figure 5 shows two EDX spectra for AlSi10Mg in as-received and moisturized state. The X-ray energy for Oxygen is $K_\alpha = 0.525$ KeV. For the as-received state Oxygen content is not detected with the EDX. On the contrary, for the moisturized state there is a small peak corresponding to Oxygen element around 0.5. This measurement was carried out for all materials in both as received and moisturized state and results of Oxygen element are showed in Table 1.
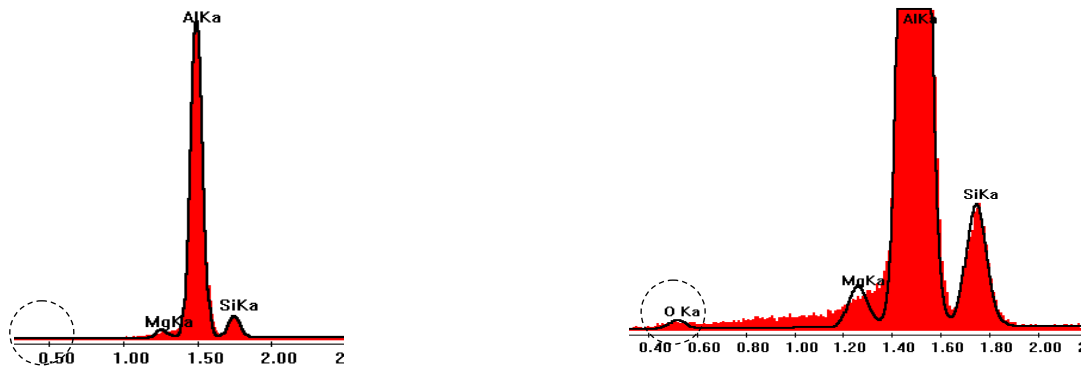

Figure 5. EDX spectra for AlSi10Mg in as-receive (left) and moisturized (right) state

Table 1 lists the Oxygen of each material for both above-mentioned states. Nevertheless, the EDX method is not 100% accurate in determining the Oxygen content of the powders. Besides the technique accuracy, there are variations in the measurements from particle to particle. Therefore, it is recommendable to measure the oxygen content additionally with a different technique in order to check the presently obtained results.

Table 1. Oxygen content increases (wt%) estimated with EDX in as-received state and in moisturized state

Material	[Moisturized/As received]
Inconel 718	2,2
Ti6Al4V	1,5
AlSi10Mg	3,1
Scalmalloy	1,6

3.3. Specific surface area and particle size distribution

The specific surface area is estimated by measuring the amount of Nitrogen gas adsorbed as a mono layer over the particle using the BET technique. Therefore, morphology and other external powder features are expected to be visible in the values of this parameter. The graph in Figure 6 shows the relation between the specific surface area and the average particle size *d50* (50% PSD).
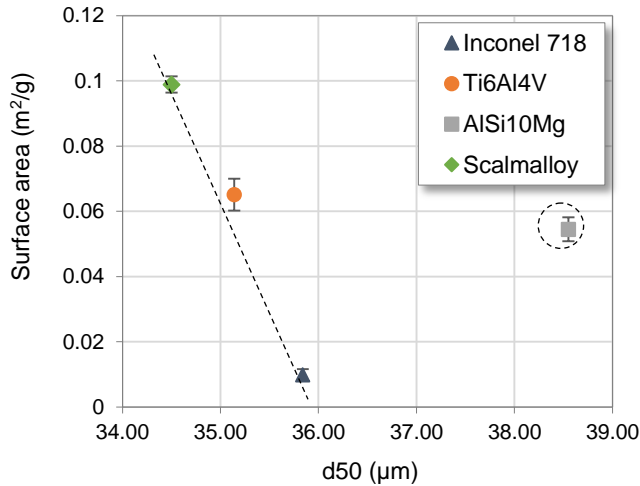
Figure 6. Specific surface area as a function of particle size.

As it was mentioned in section 2.5, the presented parameters (surface area and *d50*) should be inversely proportional. Therefore, the finer the powder is, the greater the surface area. This means that more moisture and other contamination in the atmosphere can be deposited on the surface. Following this argument Scalmalloy, Ti6Al4V and Inconel 718 data points follow the dashed line with a negative slope, indicating that they follow the expected trend. However, AlSi10Mg seems to stand out of the line, having a much higher surface value as expected by its size. In section 3.1, where morphology was discussed, AlSi10Mg proved to have the most irregular shape, with roughness and satellites, which could explain the high surface area. However, AlSi10Mg also has a quite broad particle size distribution and is the coarser powder. That might also affect its position in Figure 6.

3.4. Moisture content and flowability

The moisture content is illustrated in the graph on the left side of Figure 7. The represented values correspond to all powders states: as-received, vacuum-dried, air-dried and moisturized. The most outstanding data point is AlSi10Mg after being aged under humid conditions, suggesting that AlSi10Mg is the most sensitive for moisture pick-up. The results for Inconel and Ti are somewhat unexpected, as the moisture content in the air-dried state is higher than in the moisturized state. This might be due to the inaccuracy of the measurement.

On the right side of Figure 7 the flowability is shown. It represents the time (in seconds) that 50 g of powder takes to flow through the Hall meter. The fastest powder to flow is Inconel 718 followed by Ti6Al4V, both influenced by their density (relatively high) and morphology (regular). Further, Ti6Al4V slightly increases its flowability after applying further treatments to the powder such as drying and moisturizing. Inconel shows a more unpredictable behavior, the highest flowing time is registered after drying the powder in air. Alternatively, Scalmalloy and AlSi10Mg are light-weight alloys. Therefore, their flowability behavior is very easily influenced. In this case Scalmalloy presents difficulties to flow or it doesn't flow at all after drying under vacuum and being moisturized. Finally, AlSi10Mg shows the most expected result. The flowability improves when drying under vacuum and it doesn't flow after acquiring humidity from the climate chamber.
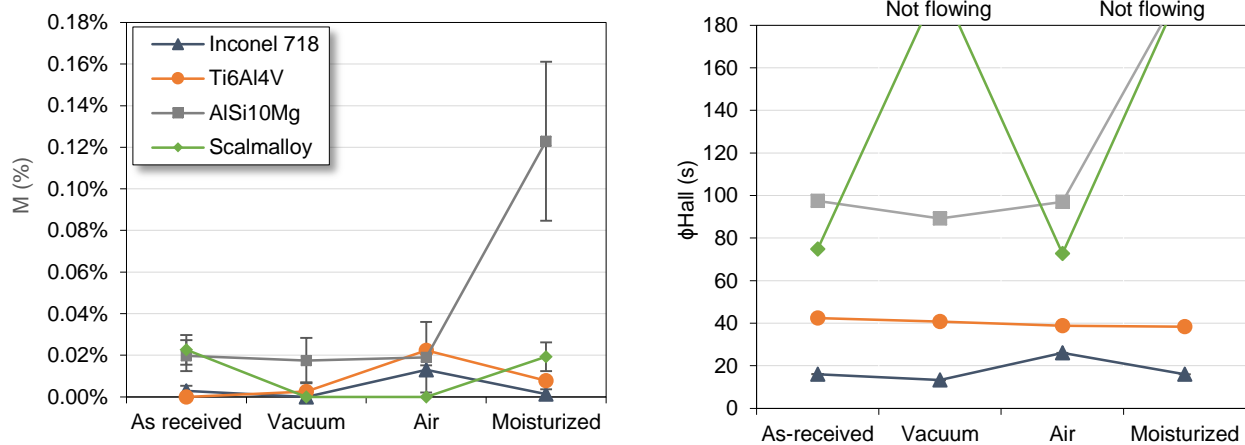

Figure 7. (left) Moisture content and (right) powder flowability

4. CONCLUSIONS

This study assessed the impact of an aging treatment and proposed solutions through drying processes for four different LBM alloys. Changes on the morphology, composition and ability to flow were quantified. Due to the robustness of Inconel and Ti6Al4V both in density, composition and morphology for this process, they showed low affinity to moisture and low impact of drying/aging treatments. On the other hand, Aluminum alloys seem to have difficulties to flow, especially under extreme conditions due to their irregular morphology, size and density. The conclusion of this work is that Aluminum powders (Scalmalloy and AlSi10Mg) are the most affected of the four materials by any treatment. In addition, further experiments should be conducted in order to deeper determine the impact of aging conditions on the Aluminum alloys used in LBM processes.

ACKNOWLEDGMENTS

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