Characterizing the Bulk & Flow Behaviour of LS Polymer Powders

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Abstract

The properties of laser sintering (LS) powders affect processability and the quality of parts manufactured. This study compared three different methods used to quantify both the static and dynamic powder properties – a Revolution Powder Analyzer, FT-4 Powder Rheometer and Hausner Ratio. The aim of the work was to identify the most reliable method to characterize powder properties in correlation to the dynamic conditions that occur during LS. The experiments focused on different particle size distributions of a cryogenically ground polyurethane powder compared to a standard polyamide 12 LS material, PA2200. The results have led to a deeper understanding regarding powder interactions and therefore serve as input for material design and quality assurance.

Keywords: Powder Bed Fusion, Polyurethane, Powders, Laser Sintering, Flow Properties, Bulk Behaviour.

1. Introduction

The term Additive Manufacturing (AM) covers a variety of technologies used to build parts directly from a three-dimensional digital design data. Powder bed fusion (PBF) of polymers also known as Laser Sintering is one of the main additive processes that can be used to produce functional parts for testing or low series production [1]. However, the performance of components, mainly built from Polyamide 12, is limited concerning their near series properties. Since there are many different requirements for polymer consumer products, it is clear that the current repertoire of available polymers in PBF is no longer sufficient. Thus one main aim in AM research is to gain deeper understanding of the requirements of new laser sintering polymers. The most common polymers used in PBF and therefore the focus for the majority of current research, are semi-crystalline polymers predominantly polyamides (PA 12 or PA 11) or polyamide-based compounds as well as some filled varieties [2, 3]. To broaden the field of application, the research presented in this work introduces a thermoplastic polyurethane powder, a type of polymer which has not been well reported in AM literature up to now. In addition, the majority of methods to characterize PBF materials that are described were originally established for conventional manufacturing processes, for example injection moulding or the storage of powders in hoppers [4, 5]. The complex interactions over the whole process chain in PBF demand a deeper understanding of the requirements for various powder qualities, the interactions of process parameters between the non-intrinsic (particle shape and size, flowability), as well as the intrinsic (melting point, melt flow) properties of the polymers used. As PBF powders are stored statically in a reservoir before being applied in

a dynamic manner, it is important to understand the particle size distribution (PSD) and shape coupled with its ability to flow under the roll spreading conditions present during PBF. Most researchers report on particle shape or morphology, size distribution and tap or bulk densities of the powders investigated, but do not take the dynamic flow properties into account [6, 7]. In addition the methods used to quantify the packing efficiency of the PBF powders are highly dependent on the user and do not necessarily lead to reliable data. In order to set up criteria for quality assurance it is essential to have analysis methods in place that work independently from its user. Amado *et al.* introduced a powder characterization method and found that its dynamic quantification of PBF powder properties lead to a more sensitive differentiation in characterization in comparing different commercially available powders [8]. In correlating a standard PA2200 with a cryogenically ground thermoplastic elastomer this study also takes the effects of different shapes and PSDs into account.

2. Objective

Due to the lack of quantitative information on the dynamic properties of PBF powders, the development of a new polymer powder coincides with high efforts in trial and error cycles. The aim of this paper therefore is to compare three different powder characterization methods giving information on the packing as well as dynamic behaviour of different PBF powders and to identify the most reliable. In complementing the existing knowledge on powder characterization approaches it is aimed to find methods that help to reduce the effort in qualifying new powders for PBF.

3. Experimental approach and methods

The experiments focused on different PSDs of a cryogenically ground polyurethane powder compared to a standard polyamide 12 PBF material, namely PA2200.The different fractions for the ground powders were achieved within an air classification process leading to a sharp separation between the size distributions. F0 therefore refers to the standard distribution out of the manufacturing process, F25 and F45 respectively indicates that no fine particles below 25 µm or 45 µm are enclosed. Commercially available powders showing good flowability consist of spherical particles with a narrow distribution in particle size around D50=50-60 µm and a volumetrically low amount of fine particles at around 10 µm [9, 10]. PA2200 therefore serves as the benchmark material.

Size distribution and particle shape were initially analysed, as described in 3.1. The three different methods used to quantify static and dynamic powder properties were a FT-4 Powder Rheometer (3.2), a Revolution Powder Analyzer (3.3), and Hausner Ratio (3.4). In order to obtain comparable results, all the experiments were completed in the same environmental conditions with an ambient temperature of 22°C and a relative humidity of 50 %. The goal of this study is to compare these methods with respect to 4 different process steps during laser sintering before the exposure of the powder bed as follows below:

- **Cohesion** of powder affecting **packing** and **flow efficiency**
  - BFE (FT-4)
  - Avalanche Angle (RPA)
  - Δ Density ($\rho_{bulk} - \rho_{tapped}$) (HR)

- **Flowability** of powder during **powder layer application**
  - SE (FT-4)
  - Avalanche Angle (RPA)
  - HR

- **Packing efficiency** of the powder inside the **feeders** and **build chamber**
  - CBD (FT-4)
  - Density (RPA)
  - Bulk (poured) Density (HR)

The cohesion between the powder particles plays a significant role on the packing efficiency within the build chamber and the flow efficiency of powders during powder layer application [3, 11-13]. Previous studies which analyze powder properties show that cohesion and flowability are two properties that are not clearly distinguishable and most of the times they are considered to describe the same characteristic or at least characteristics that are similar in terms of their definition [5, 14, 15]. However, the decision in this study was to present the results for cohesion separately from flowability characteristics to correlate them to a certain process step in PBF. Therefore, cohesion was considered to characterize more the powder behaviour within the static storage in the reservoir of the laser sintering system, while on the other hand, flowability relates to the materials dynamic characteristics during application of a powder layer.

3.1 Size distribution and particle shape

To identify the physical characteristics of the powder particles (size and shape), a Camsizer XT was employed. Using a high resolution camera system consisting of two digital cameras, one camera detects the size and characteristics of fine particles, while the second camera is optimized for the analysis of large particles [16, 17]. The PSD was identified by the frequency distribution $\mathbf{q_3(x)}$ [%/µm] based on volume. **Sphericity** and **Width/Length Ratio** were two shape factors chosen to describe the shape of the particles. For a sphere or a circle, the SPHT and the Width/Length Ratio shows a value = 1.

3.2 FT-4 Powder Rheometer

An FT-4 Powder Rheometer was used to identify the dynamic **flow and bulk properties** of the powders. The operating principle of the FT-4 standard Stability Test is based on a reproducible dynamic flow pattern. The test assesses the powder sample's tendency to change its bulk and flow properties when it is forced to flow by an helix rotating through its bulk both downwards (anticlockwise) and upwards (clockwise) [18, 19]. The values obtained by the stability test describe how *stable* and *flowable* the powder is during the measurement. As the energies and densities measured are directly dependent on inter-particulate forces the effects of the powders' shape and size can be detected [14, 19].

3.2.1 FT-4 Characterization Indexes

The **Basic Flowability Energy (BFE)** [mJ] is the energy required to establish a compressive high stress flow pattern in a conditioned, precise volume of powder. The BFE is calculated

from the energy consumed by moving the blade downwards through the powder bulk from the top of the vessel to the bottom. The shape of the blade is such that its movement is highly compressive and because the powder is "confined" at the bottom of the vessel, the compressibility of the powder plays a major role in the BFE [18]. Cohesive powders consist of fine particles that include several air voids inside the bulk. During the downward movement of the blade, the particles are forced to flow at the blade face and cover the air gaps that exist in between the particles creating a relatively short and localized stress transmission zone. On the other hand, non-cohesive powders are generally packed more efficient and the air gaps inside the bulk are reduced. Therefore the stress transmission zone and as a result the flow zone is extended further inside the bulk raising the value of BFE due to friction and high inter-particulate contact stresses. In this case, high flow efficiency is a result of high BFE for powders with different PSDs [14, 20, 21].

The **Specific Energy (SE) [**mJ/g] is a measure of how powder will flow in an unconfined stress environment and defines the mechanical interlocking of the powder particles. It is calculated in the same way as BFE but during the upwards movement of the blade, where the sample is not confined and can be lifted without restrictions. While powder compressibility can be very significant for the BFE, the SE is most dependent on the shear forces and inter-particulate mechanical interlocking. Due to the low stress environment, cohesion, particles shape and surface texture are the most influential properties [22].

Conditioned Bulk Density (CBD) [g/ml] is the density of a precise volume of powder measured inside the vessel of the FT-4 after the initial conditioning cycle of the bulk. Bulk density is dependent on many physical properties, such as particle size and distribution, particle shape, particle surface texture and cohesive/adhesive forces. As a result of these properties the particles are arranged inside the bulk [14, 19, 23].

3.3 Revolution Powder Analyzer (RPA) – Flowability Test

The Revolution Powder Analyzer is a measurement device to detect the dynamic flow properties of powders, consisting of a rotating drum and a digital camera system. The rotating drum is manufactured from aluminum and has an inner diameter of 110 mm and 35mm width. It is connected by two glass sides allowing the system to capture images of the rotating bulk. The camera detects the surface and the area of the rotating bulk in predefined time steps [24].

3.3.1 RPA Characterization Indexes

Avalanche Angle [°] is defined by the maximum angle obtained on the free powder bulk surface before an avalanche occurs. The average value of all avalanche angles is then being calculated. The RPA software calculates the flowability angle from the center point on the powder edge to the top of the powder edge. As a general rule it could be stated that, the higher the avalanche angle the poorer is the flowability and the more cohesive is the examined powder sample [8, 24].

Volume Expansion Ratio (VER) defines the ratio between the expanded bulk volume (bulk density) inside the drum and the tapped bulk volume in the vessel, before filling the drum during the preparation stage (tap density). The preparation step consists of filling a 100 ml

cylinder under manual tapping until the maximum powder compaction is achieved. The volume of the sample is exactly the same for every powder. The expanded volume is measured by image capturing as the sum of the area of every image pixel occupied by the powder multiplied by the width of the drum [8, 25].

Density: The bulk density inside the drum is calculated by the mass of the powder sample divided by the expanded volume inside the drum [24].

3.4 Hausner ratio

The Hausner Ratio (HR) is a well established method to characterize bulk and flow properties of powders [26, 27]. To determine the Hausner Ratio only two laboratory accessories are needed, a measuring cylinder and a powder funnel. The cylinder that was used for the HR measurements was 100 ml and the tapping of the powder was performed manually.

3.4.1 Hausner Ratio Characterization Indexes

The **HR** describes the ratio of tapped and bulk densities and therefore provides a statement on the flowability of powders. It is calculated as follows [28, 29]:

$$Hausner\ ratio = \frac{bulk\ volume}{tapped\ volume} = \frac{\rho_{tapped}}{\rho_{bulk}}$$

The **Δ Densities** ($\rho_{bulk} - \rho_{tapped}$) describes the difference between the bulk and tap density and served to characterize the cohesiveness of powders within this study.

4. Results & Discussion

4.1 Powders Physical Characteristics

Table 1, overleaf, provides an overview of the physical properties of the materials observed, including the particle size, shape, moisture content, the base polymer as well as their manufacturing processes. These properties represent the most influential magnitudes on the PBF materials non-intrinsic properties.

Characteristic/Powder	PA 2200	TPU F0	TPU F25	TPU F45
D_{10} [µm]	36.5	17.9	31.1	47.7
D_{50} [µm]	50.9	45.7	52.2	62.8
D_{90} [µm]	66.3	74.1	76.2	84.2
Sphericity	0.891	0.855	0.879	0.888
b/l	0.763	0.762	0.77	0.775
Base Material	Polyamide 12	Thermoplastic Polyurethane		
Manufacturing Process	Precipitation	Cryogenic Milling		

Table 1: PBF powder samples characteristics

In Figure 1 the SEM images for these materials can be seen to indicate the morphology and shape characteristics.

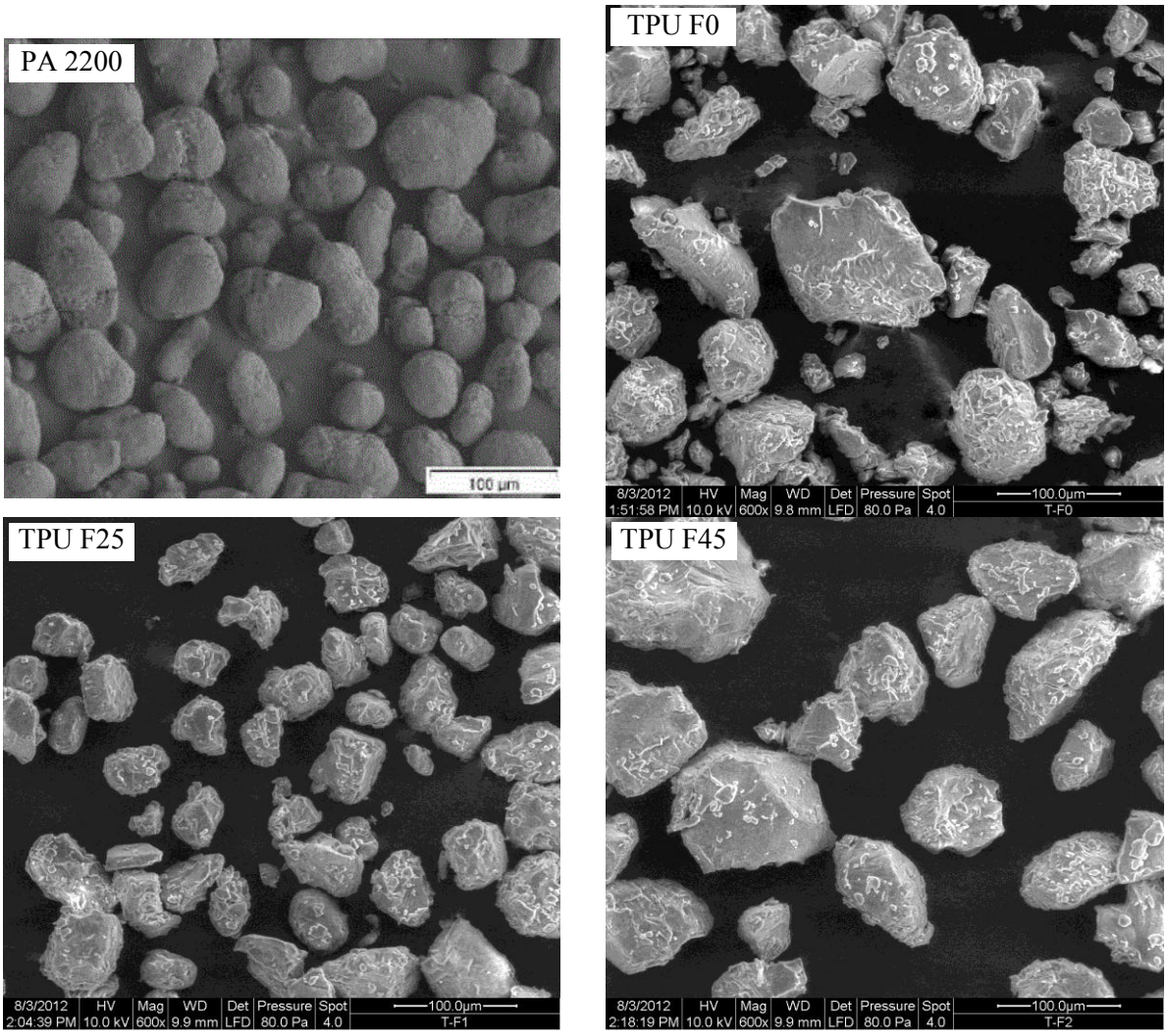

Figure 1: SEM images (resolution: 100µm for all) of PA 2200 [11] and the 3 TPU fractions [BMW]

4.2 Particles size and shape characteristics

Apart from the moisture absorption, other characteristics which form the powders bulk and flow properties are the PSD and the particle shape characteristics. As SPHT leads to a higher differentiation between the shapes of the powders observed, only the results for this value are shown. In Figures 3 and 4, the PSD and the shape characteristics (SPHT) plotted against the PSD are presented. Figure 3 represents the PSD vs. inner width x_{cmin} of the particles.

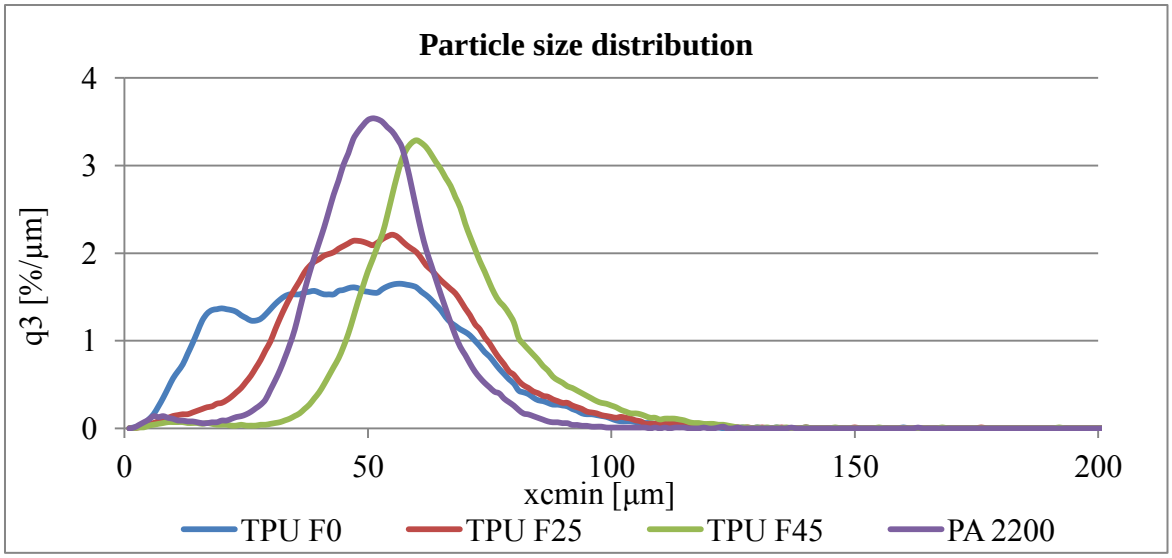

Figure 3: PSDs of powder samples over inner width

The PSD for TPU F0 is broader than it is for the other powders as a result of the milling process. After sifting the powders they align more and more to the aimed PSD of the benchmark material, the PA 2200. TPU F45 shows a D10–D50 span of 36.5 µm (D_{10} +23.48 % / D_{50} +18.95 % / D_{90} + 21.26 %) compared to PA 2200 with a span of 29.8 µm. The SEM images (in Figure 1) were used as input for differences in the surface morphology. As a general remark, TPU fractions have edgy particles with non spherical shape, especially the fractions that include finer particles meaning F0 and F25, whereas PA 2200 particles are closer to an ellipsoid or circular shape with a smoother surface texture. The difference in shape can also be seen in the SPHT-Q_3 graph (see Figure 4**)**. The powder with the most spherical particles (SPHT<1 cf. Section 3.1) was the PA 2200 with an average SPHT = 0.891 followed by TPU F45 (SPHT = 0.888), TPU F25 (SPHT = 0.879) and TPU F0 (SPHT = 0.855) respectively, indicating that the fine particles represent the most irregular shaped.

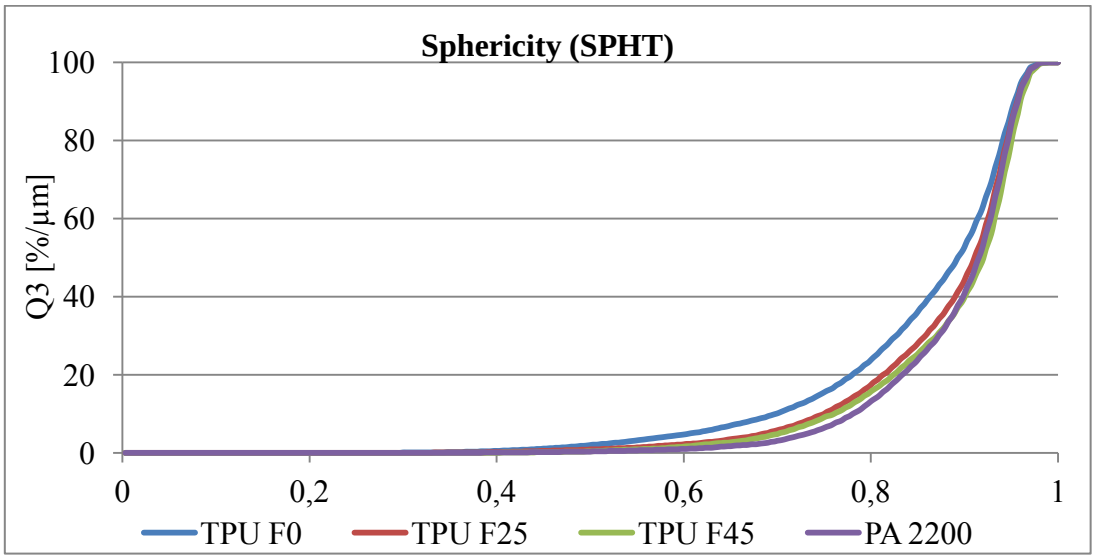

Figure 4: Cumulative PSD over particles sphericity (SPHT)

In addition, the SEM images support the results regarding the shape of the powder particles. As a result of the above mentioned, it can be noted that the fractions of TPU with larger particles have rounder or more compact particles and that the precipitated PA 2200 generates more spherical particles with a smoother surface, while cryogenic milling creates particles with a jagged edged shape.

4.3 Comparison of characterization methods

According to the objective introduced, this section provides a comparison of the results from the analysis with respect to the powders' bulk and flow properties.

4.3.1 Cohesion results

Figure 5 summarizes the results from the cohesion measurements using the three different methods introduced.

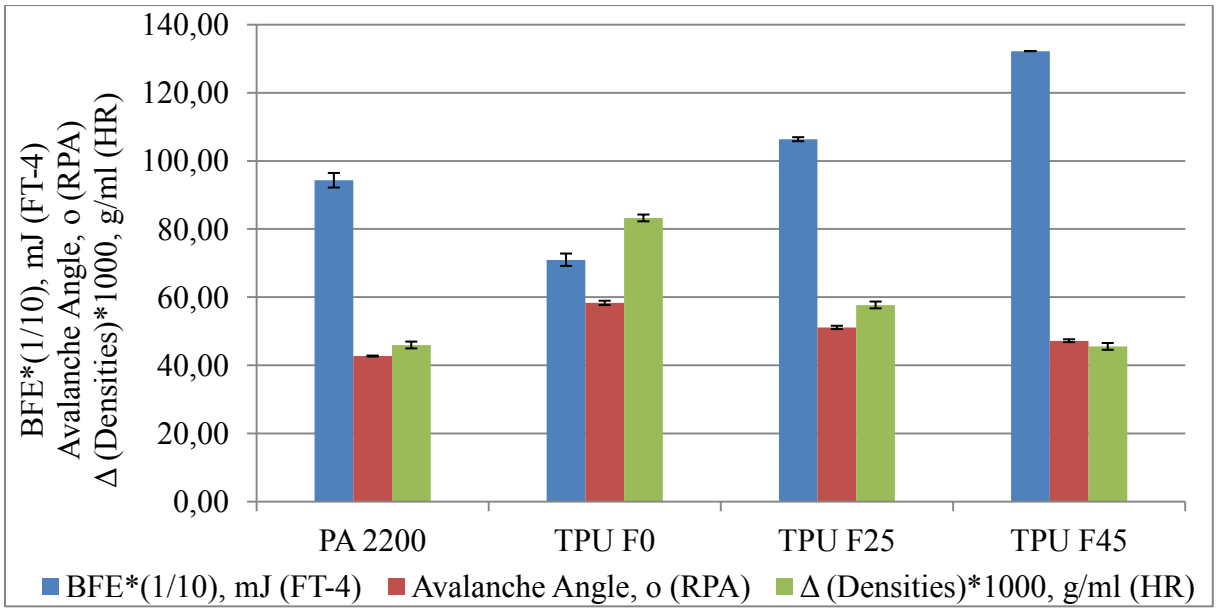

Figure 5: Comparison (materials and methods) of cohesion results

In order to interpret the results depicted in Figure 5, the important conclusion is that the results for the BFE, Avalanche Angle and the Δ Densities show the same trends in terms of cohesiveness. The higher the Avalanche Angle and the Δ Densities (see Section 3.4.1) are the more cohesive a powder behaves and vice versa for the BFE [8, 15, 24, 30]. The results show that with a decrease in fine particles within the TPU fractions the cohesive character of the powder bulk decreases (BFE TPU F0 = 709.72 mJ / TPU F25 + 33.26 % / TPU F45 + 46.31 %). TPU F25 with its lower amount of fine particles already gets close to the cohesion characteristics of PA 2200. The BFE even indicates a lower cohesiveness. Here the index of BFE presents a misleading trend when comparing PA 2200 and TPU fractions. It is known from empirical data during processing the materials in the PBF systems that the TPUs act more cohesively than the PA 2200 material. The Avalanche Angle as well as the Δ Densities show a straight forward trend compared to the processing characteristics of the powders within the PBF process. TPU F45 with the lowest amount of fine particles shows the same value for Δ Densities compared to PA 2200 and a 9.53 % higher Avalanche Angle indicating

a negligibly higher cohesiveness. This could be due to van-der-Waals interactions that represent the dominating adhesive forces at lower particle sizes up to 50 µm [5]. With a further increase in particle size the influence of these forces decrease due to the increasing influence of the weight forces of bigger particles [11, 31].

4.3.2 Flowability results

Flowability is the exact opposite value of cohesion. The more cohesive a material behaves the lower its flowability and vice versa. Figure 6 depicts the comparison of the flowability results again using the three methods introduced within the methodologies section. Since it is known from literature and the results out of this study, that the VER does not help to distinguish between powders with different shapes and PSDs, the flowability results of the RPA refer to the Avalanche Angle as well [8]. This value shows the same trend as the VER with a lower standard deviation and therefore delivers a more significant differentiation between varying powders. Furthermore, in terms of cohesion, the higher the Avalanche Angle is, the higher the cohesiveness of the powder gets. As a consequence a high Avalanche Angle results in poorer flowability.

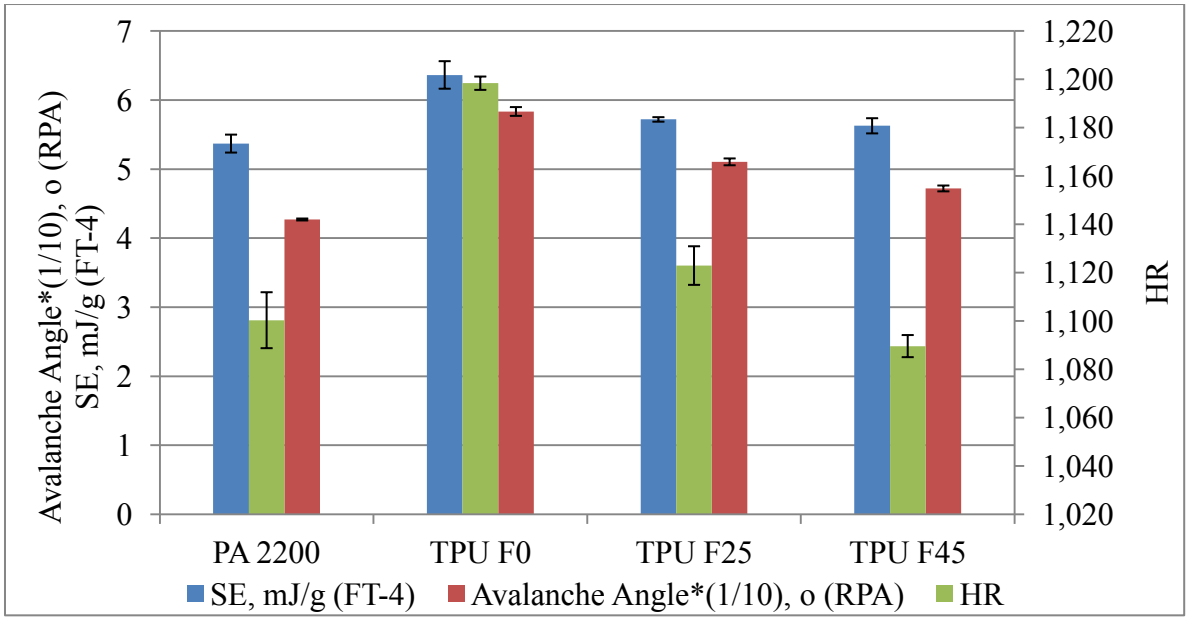

Figure 6: Comparison (materials and methods) of flowability results

Conversely, the Specific Energy is an indication of the particles' mechanical interlocking as it detects the amount of energy that the helix needs during its upwards movement in order to break the inter-particulate bonds of the powder bulk. The bulk is not confined on the top of the FT-4 vessel, so no compressibility forces are present. The higher the Specific Energy is the stronger is the mechanical interlocking and the inter-particulate bonds of the powder that result in a poorer flowability. Figure 6 depicts that Avalanche Angle and Specific Energy show the same trends within the TPU fractions. TPU F0 which has finer particles (F0 D_{10}= 17.9 µm *c.f.* F45 D_{10}= 47.7 µm) shows the highest values in Avalanche Angle and SE resulting in the poorest flowability of all the samples. This is due to the changes in the dominating adhesive forces within the powder bulk described in Section 4.3.1, as with an increase in particle size the weight forces lower the influence of van-der-Waals interactions

and therefore allow the particles to slide along each other easier. TPU F25 shows higher values of Avalanche Angle and SE than PA 2200 and TPU F45, implying also poorer flowability in comparison. As PA 2200 and TPU F45 show similar PSDs (Section 4.2) the higher Avalanche Angle and SE of TPU can be attributed to the irregular shapes and rougher surface of its particles and the higher mechanical interlocking while flowing past each other.

The HR results show a similar trend compared to the Avalanche Angle and SE results, with a higher standard deviation though the measurements where repeated three times to get a statistically resilient statement. The deviation results out of the fact that the procedure is highly user dependent due to the manual tapping and consolidation phenomena between powder particles influence the repeatability.

4.3.3 Packing efficiency results (Feeders and Build Chamber)

Figure 7 shows the results from the density measurements indicating that for the TPU fractions a decrease in the fine particles leads to an increase in packing density. Although it is known from literature that a certain amount of fine particles is needed for an efficient packing to fill in the gaps between the larger particles, this does not apply for the jagged edged shaped TPU particles [11, 14, 32].

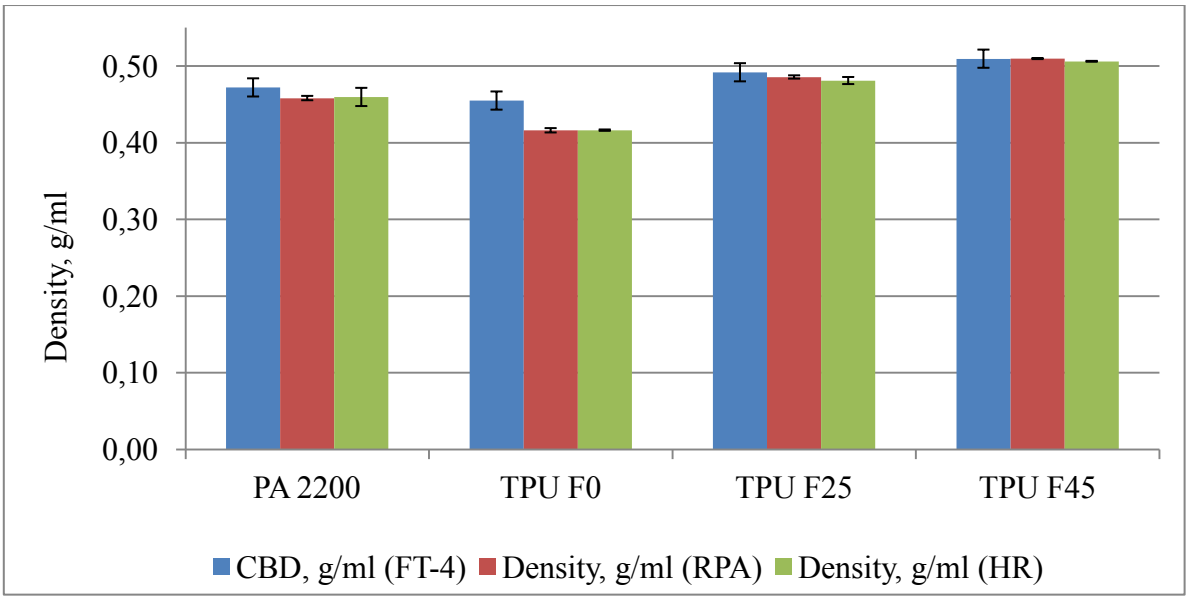

Figure 7: Comparison (materials and methods) of packing density results

In addition the positive effect of a certain amount of fine particles filling in the gaps between the larger ones might be hindered due to the dominating adhesive forces between them (i.e. TPU F0) and may not distribute homogenously within the overall powder bulk (see Figure 8).

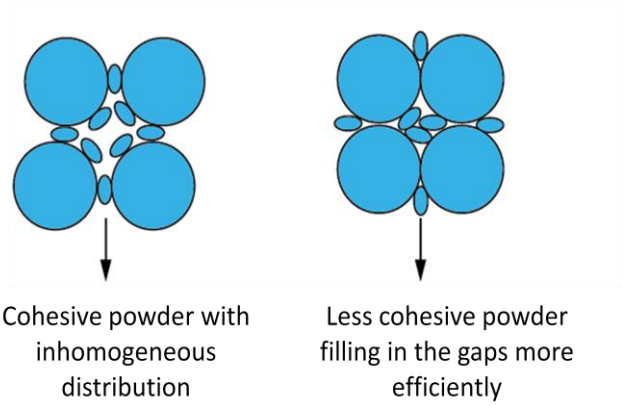

Figure 8: Schematic view of effects on packing efficiency between cohesive and non cohesive powders following [32]

The higher packing density of F45 (+ 9.8 % in RPA) compared to PA 2200 therefore results out of larger contact areas between the less spherical particles. RPA and HR almost show similar values for density and align more to the real density to be expected. This can be aligned to the fact that the bulk density in the FT-4 is measured after one conditioning cycle where the helix went through the bulk performing rearrangements in the bulk and particles collisions and therefore leads to a higher density in general. This effect is even more significant for the powders consisting of fine particles (i.e. TPU F0) as the CBD density reduces the adhesive effects within the powder bulk due to the external compressive force that is applied [13, 14].

6. Conclusions

In designing new powders for PBF the analysis methods introduced could bring added value in predicting the static as well as the dynamic powder properties. It was shown that it is important to also take the particle shape and size distribution into account, as an improvement in the bulk properties could be reached in modifying these properties especially for cryogenically ground materials. For TPU F25 an improvement of the flow efficiency as well as its packing behaviour was shown in sifting out a low amount of fines below 25 µm, while keeping material wastage low.

- **Cohesion:**

The **BFE** (FT-4) is an accurate value to detect cohesion behaviour but showed some drawbacks when it is about comparing powders with significantly different shapes as well as PSDs. Detecting cohesiveness the Avalanche Angle of the RPA leads to reliable and reproducible trends (low standard deviation) within this study. The Δ Densities (HR) did not lead to a clear differentiation between the cohesiveness of TPU F45 compared to PA 2200. The more the analyzed powders differ in their particle shape and surface morphology the less sensitive the differentiation between them might be.

- **Flowability**:

In terms of quantifying flowability or mechanical interlocking and therefore an indication of the behaviour of a powder during the application of layer in PBF, the results show that for

powders with very fine particles as TPU F0, **SE** (FT-4) is an accurate value. However, in the comparison between TPU F25 and TPU F45 the **SE** values are very close and do not distinguish between powders with a similar shape sensitively enough within the materials observed. The RPA again showed reproducible and reliable results in detecting flowability. The deviations of the HR results were higher than all the other methods applied. HR therefore is not the method of choice to obtain reproducible results.

- **Packing efficiency**

The **CBD** (FT-4) indicates changes in density but does not necessarily lead to a statement on real bulk density and only serves as a method to compare changes attributed to varying PSDs (see TPU fractions). The **Densities** in the RPA and the HR showed low deviations and correlate more with the real density of the powder bulk within the feeder of a PBF system.

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