Flowability of SLS powders at elevated temperature

Report

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FLOWABILITY OF SLS POWDERS AT ELEVATED TEMPERATURE

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Abstract

The development cycle of a Selective Laser Sintering (SLS) material incorporates many aspects that must be fulfilled before it can be satisfactory processed. Among these properties the polymer flowing behavior in its powder state is of primary importance. A lack of a suitable powder spreading quality during SLS processing leads to inhomogeneity of part density and reduced mechanical properties. However, none of the available powder characterization methods attempts to simulate the powder flowability at effective SLS processing temperatures. Therefore this investigation characterizes the powder flowing dynamics at higher temperature for commercially available materials and research powders. A measuring system with a rotating drum emulates the stress state of the material during the SLS deposition. New insights regarding the spreading conditions are presented, particularly above the glass transition point were the viscous part of the polymer becomes more relevant. In particular the dynamic avalanche angle, volume expansion ratio and surface fractal distributions represent suitable parameters to characterize the different powders in terms of the flowing behavior and its correlation to the final sintered density. This system is proposed as a SLS quality control and screening tool during the early stages of powder development.

Introduction

According to Amado et al. [1] the development of new powdered materials suitable for the Selective Laser Sintering Process with the aim to broaden its application field constitutes one of the main research topics and challenges nowadays. Regarding the materials characterization for its SLS process suitability, most researchers concentrate their effort on intrinsic properties. However, non-intrinsic properties are normally barely reported or just left out. It is well known that good dispersion conditions are necessary to achieve a higher powder packing and a homogeneous layer spreading, but no quantitative information towards a prediction for SLS powders is available in literature. Thus, most researchers conduct a powder development cycle just by trial and error carried out on a full or scale SLS equipment, which increases the development costs in terms of a higher amount of initial powder production and larger processing times.

Several factors influence the powder flowing properties, e.g., particle size distribution, particle shape, inter-particle forces, moisture and temperature. It is a complex task to define theoretically the flow behavior of bulk solids in dependence of all of these parameters. Therefore it is necessary to determine the flow properties in

appropriate testing devices. An overview of most common characterization techniques and concepts is presented in Table 1.

Test Method Name	Measurement Concept	Characterization Parameters	Measurement Temperature	Measurement Procedure
Fluidimeter	Dynamic powder expansion under vertical fluid flow drag effect	Powder bed expansion height versus upstream fluid flow	Standard conditions (25ºC)	Not specified
Angle of Repose	Vertical powder deposition through a funnel / orifice under the gravity effect	Heap or pile angle of repose	Standard conditions (25ºC)	DIN ISO 4324
Ring Shear Cell	Quasi-static powder stability on an annular container under compression and shear	Failure locus of shear force versus normal pressure	Standard conditions (25ºC)	ASTM D6773
Bulk/Tap Density	Powder density ratio between a compacted and loose state under mechanical tapping	Tap and bulk apparent powder density	Standard conditions (25ºC)	ASTM D7481

Table 1: Most common powder characterization methods

In general, the information provided by each method is strongly dependent on the kinematic measurement condition and on the particular stress state of the sample. Thus the selection criterion of each method relies on the knowledge of the particular powder processing or handling system. In a previous work [1] a novel method based on a rotating drum was introduced and several SLS commercial and research powders were characterized. The advantage of this device is based on the emulation of the typical stress free turning powder wedge behaviour generated by any of the actual SLS spreading systems, i.e., the counter-clockwise rotating roller (3DSystems) or the concave blade coater (EOS). However, besides the selection of an adequate characterization device in terms of stress state and dynamic handling conditions, empirical evidence indicates that the chamber processing temperature constitutes a critical factor that influences the powder spreading conditions. According to the Hertz contact theory between two elastic bodies the particle-particle contact forces are strongly dependent on the effective elastic modulus [2].
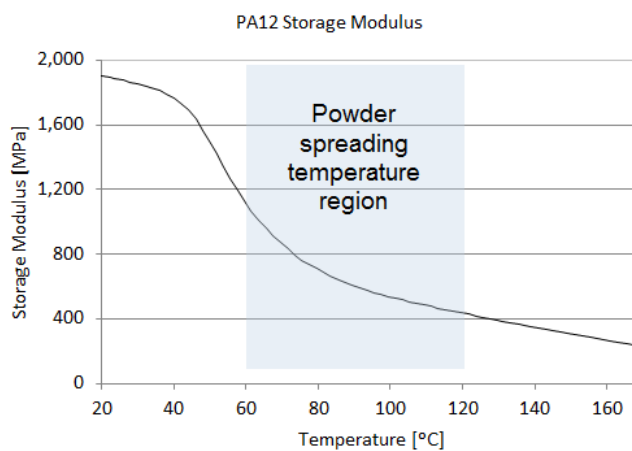
Figure 1: PA12 Storage Modulus obtained from a DMA test

In case of semi-crystalline polymers, the behavior of the elastic component is strongly dependent on the temperature and presents a considerable reduction above the glass transition point. As an example, Figure 1 depicts the storage modulus of a commercial SLS PA12 material between 20ºC and 170ºC. Indicated is also the typical powder pre-heating temperature region previous spreading which depends on the SLS system employed (i.e., EOS or 3DSystems). As observed, variations of more than 100% are obtained which indicates the influence of the temperature factor. Based on these arguments the aim of the current study is to expand the previous powder characterization work towards elevated SLS processing temperatures.

Testing Equipment & Experimental Set Up

The measurement device consists of a modified Revolution Powder Analyzer manufactured by Mercury Scientific Inc. It consists of a rotating and an image acquisition system as shown in Figure 2. The rotating drum is machined in aluminum with an inner diameter of 50 mm and 35 mm width. The lateral sides are covered with transparent glass to allow the powder behavior inside be captured by the image acquisition system. Additionally, for this study the aluminum rotating drum was modified and equipped with heated core cylinder to adjust the temperature ranging from 25ºC up to 120ºC inside the powder cavity.
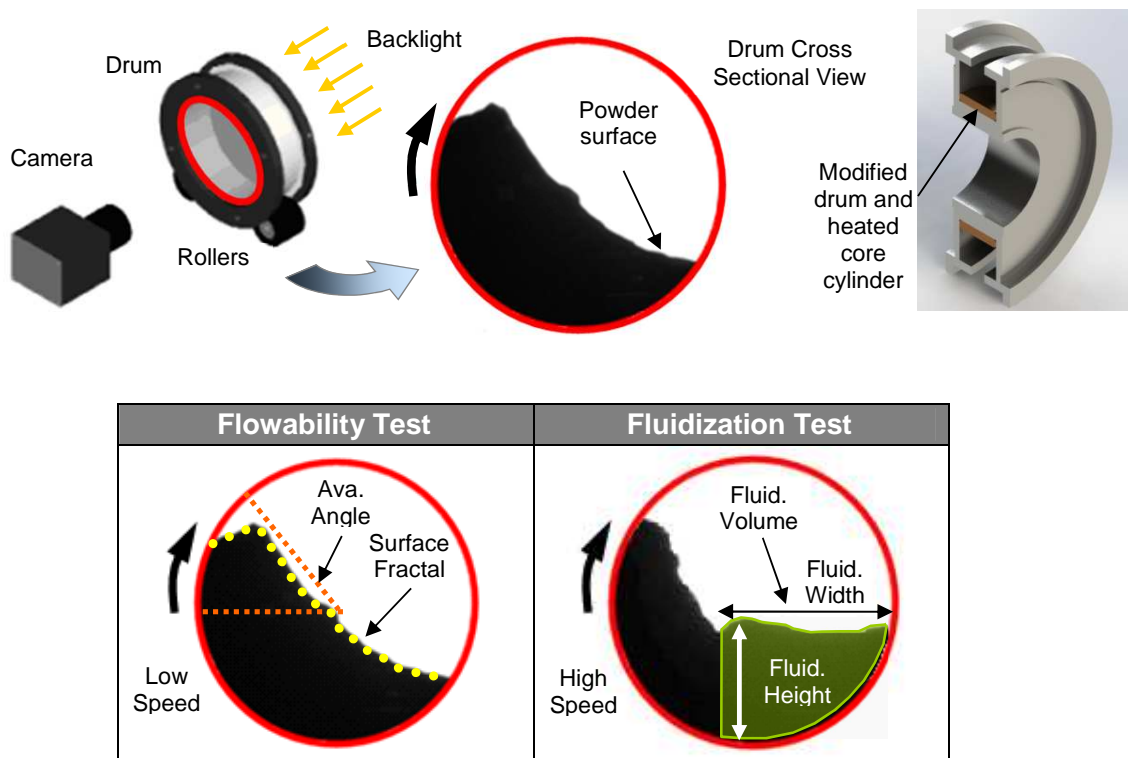

Figure 2: Schematic diagram of the system, modified rotating drum & test condition parameters

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With the aid of a backlight source, the powder free surface and cross sectional area of powder inside the drum can be recorded. Depending on the turning speed, two different tests can be performed: At low values, a discrete behavior is achieved based on a sequence of avalanches; at higher speeds, a continuous operational mode is reached, characterized by a steady state regime. Each behavior is called Flowability and Fluidization respectively. For the present research, the initial set up and the different parameters employed for each method are summarized in the following table:

Flowability Test		Fluidization Test	
Parameter	Value	Parameter	Value
Sample Volume	25 cc (tap density)	Sample Volume	25 cc (tap density)
Rotating Speed	10 rpm	Prep. Rotating Speed	90 rpm
Preparation Time	30 s	Preparation Time	30 s
Avalanche Threshold	0,65 %	Initial Rotating Speed	50 rpm
Angle Calculation	Half	Final Rotating Speed	90 rpm
N° Avalanches to record	512	Rotating speed incremen t	10 rpm
Image capturing rate	15 fps	Image capturing rate	30 fps

Table 2: Flowability and Fluidization test set up main parameters

The materials considered for both measurements are a commercial PA12 (PA2200) [3] and a random co-polypropylene (icoPP) [4]. For each material three different temperatures were selected. For PA2200 30ºC, 70ºC and 110ºC were employed. For icoPP 30ºC, 45ºC and 60ºC were defined. Additionally, for each powder tested, 3 consecutive measurements were considered, to determine any possible variations regarding the repeatability of the results.

Based on the previous work, the following characterization indexes presented in Table 3 were employed. Further details about their definition can be found in reference [1]:

Index	Test	Definition
Avalanche Angle	Flowability	Angle obtained from a linear regression of the free surface at the maximum potential energy prior to the start of the powder avalanche occurrence
Surface Fractal	Flowability	Fractal dimension D obtained from the free surface of the powder. D corresponds to a dimensionless parameter based on the self-similarity concept and constitutes a powder rearrangement indicator
Total Volume Expansion Ratio	Fluidization	Ratio between the volume measured inside the drum (expanded volume) and the volume occupied by the powder in the preparation sample container (tap density)

Table 3: Characterization indexes for Flowability and Fluidization test conditions

Among the parameters presented, the ratio between the fluidized volume expansion and the sample volume can be correlated to a certain extent to the so called Hausner Ratio (HR) or Carr index (CI=1-HR^{-1}) (ASTM D7481) derived from compressibility studies. However in this case, several values are obtained depending on the rotational speed. The results for the Fluidization test will be reported on an extended version of this paper.

Results & Analysis

Figure 3 depicts the results for the Avalanche Angle parameter obtained for each material. On the left side each curve corresponds to the averaged cumulative distribution of 3 consecutive measurements with their respective standard deviations measured at different drum temperatures. Additionally on the right side the box plot of each curve is presented. The tops and bottoms of each "box" are the 25th and 75th percentiles of the samples, respectively. The line in the middle of each box corresponds to the sample median and the notch displays the variability of the median between samples. The width of a notch is computed so that box plots whose notches do not overlap have different medians at the 5% significance level. The significance level is based on a normal distribution assumption, but comparisons of medians are reasonably robust for other distributions [5]. In case of icoPP, the median at a temperature of 30°C presents an Avalanche Angle value of 44 degrees and remains constant for the higher temperatures. Due to the overlap of the notches at different temperatures no statistical differences can be stated. A similar trend can be observed for PA2200 with a slight increase of the 75th percentile at 110°C. Despite that the median value is higher in compassion to icoPP no clear variations of the avalanche angle with the temperature increase can be stated for none of the materials.
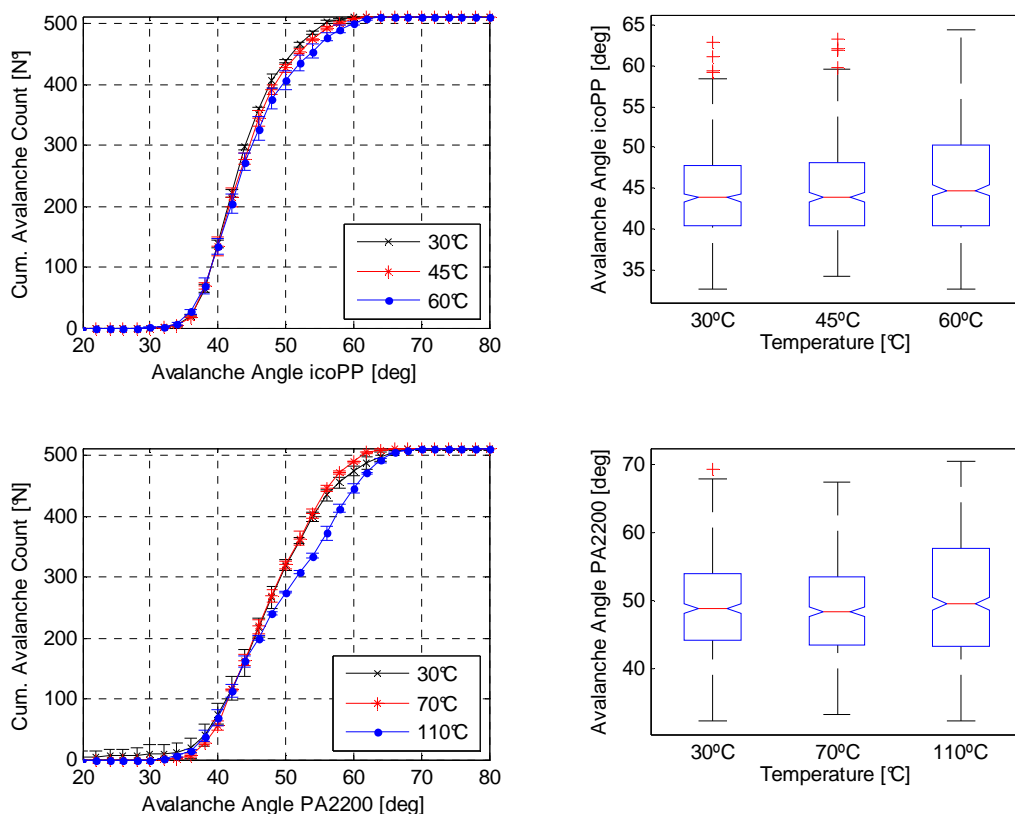
Figure 3: Avalanche Angle Distribution versus pre-heating temperature (icoPP & PA2200)

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Figure 4 depicts the results for the Surface Fractal parameter at each temperature. In this case clear statistical differences can be observed. In case of icoPP, as the temperature increases from 30℃ up to 60℃ a contin uous increase of the mean Surface Fractal value is obtained from 2.01 to 2.36 and also a broadening of the interquartile range. In case of PA2200 and interesting behavior can be observed. As the temperature increases from 30℃ to 70℃ a considera ble reduction of the Surface Fractal value is obtained. This effect correlates with the transition above the glass temperature (51℃ for PA2200), where a reduction of the storage modulus (elastic component) is present. Afterwards, as the temperature is raised again from 70℃ up to 110℃ an increase of the Surface Fractal mean is ob served. In case of icoPP this effect is not visible and an explanation can be related to the lower glass transition point of co-polypropylene, which is below the measurement range considered and even below standard room temperature (25℃).

In correlation to the SLS process these differences in the rearrangement of the powder in dependence of the temperature after an avalanche can also influence the spreading conditions and packing of the powder previous laser scanning, particularly considering which kind of SLS equipment is employed.
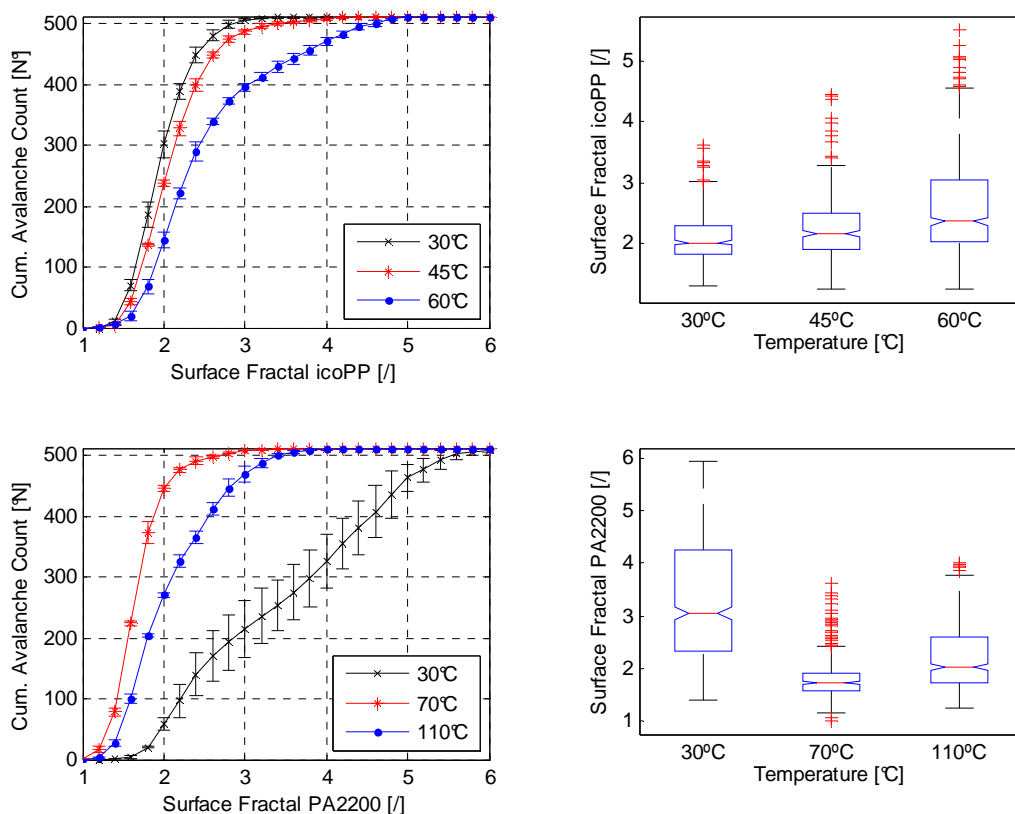

Figure 4: Surface Fractal Distribution versus pre-heating temperature (icoPP & PA2200)

In case of EOS systems a controlled pre-heating stage of the powder is not available. However, due to a longer residence time in the hopper feeds previous spreading the material reaches an average temperature of 60°C whe n PA2200 is processed. In case of 3DSystem machines this temperature is operator controlled and a standard production value is set at 120°C. For these two dif ferent temperature set up considered two different powder spreading conditions are obtained, which certainly influence the packing density variations and its effect on the final sintered density as reported by Wegner & Witt [6].

Summary

The dynamic characterization of two SLS semi-crystalline powders could be analyzed at elevated temperatures by means of a novel measurement device. New insights regarding the behavior of SLS powders are introduced in correlation with the pre-heating temperature of the material previous its spreading step on SLS machines. As a main observation the particular temperature set up has a clear effect on the powders flowability, where the glass transition point defines a remarkable transition between different behaviors. In case of PA2200 a reduction of the Surface Fractal value above the vitreous temperature indicates a better flowing condition and thus suggests an increased and homogenous powder packing deposition. Additionally this method can also be used to define the maximum pre-heating temperature allowed to avoid the formation of clumps during the spreading stage. More details about the Fluidization results and the correlation with the packing and sintering density will be covered on an extended version of this paper.

References

[1]   Amado, A., Schmid, M., Levy, G. & Wegener, K., 2011. „Advances in SLS Powder Characterization". *Proceedings SFF Symposium*, Austin, USA, pp. 438-452.

[2]   Johnson, K.L., Kendall, K. & Roberts, A.D., 1971. „Surface Energy and the Contact of Elastic Solids". Proceedings. R. Soc. Lond., vol. (324), pp. 301-313.

[3]   Datasheet PA2200 EOS Gmbh, http://eos.materialdatacenter.com/eo/de, visited on 26.03.2013.

[4]   Datasheet   icoPP   Inspire   AG,   https://www.inspire.ethz.ch/irpd/RP RM/mat/ds_iCoPP_deutsch, visited on 26.03.2013.

[5]   MATLAB Software R2010b, The MathWorks Inc., Natick, MA, USA.

[6]   Wegner, A. & Witt, G, 2012. „Betrachtung zur Pulvernutzungsdauer beim Laser-Sintern   und   Einfluss   der   Prozessführung   auf   die   Entstehung   von Ausschussbauteilen". *Proceedings of the Rapid Tech. Conference*, Erfurt, Germany.