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Mass flow characterization of selective deposition of polymer powders with vibrating nozzles for laser beam melting of multi-material components

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Abstract

The generation of multi-material components by laser beam melting (LBM) is a challenge which requires the invention of new coating devices for preparation of arbitrary powder patterns. One solution is the usage of vibration-controlled nozzles for selective deposition of polymer powders. Powder flow can be initiated by vibration enabling a start-stop function without using any mechanical shutter. In this report, the delivery of polymer powder by vibrating nozzles is investigated with respect to their application in LBM machines. Therefore, a steel nozzle attached to a piezo actor and a weighing cell is used in order to measure the stability and time-dependence of the powder mass flow upon vibration excitation with the usage of different kind of powder formulations. The results show that precompression of the powder inside the nozzle by vibration excitation is essential to realize a reliable start-stop function with reproducible discharge cyles and to prevent a initial flush of powder flow. Moreover, the use of different powder materials showed that mass flow is even possible with powders which are not optimized regarding flowability, but is readily enhanced with a factor of 2 to 3 by admixing Aerosil® fumed silica.

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Keywords: Laser beam melting; polymer powder; multi-material deposition; vibration-controlled powder nozzle

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1. Introduction

These days, several freeform fabrication techniques are established in industry which enable the generation of unique three-dimensional design models or prototypes. While techniques as 3D-printing or Fused Deposition Modeling (FDM) yield parts which usually show a poorer mechanical stability, powder bed based techniques like laser beam melting (LBM) of polymers or metals can provide advanced mechanical properties (Kruth *et al.*, 2008). Due to that, LBM can also be used for the fabrication of real functional devices even for high performance applications including aerospace, tooling, medical devices or automotive (Levy *et al.*, 2003; Wohlers, 2014). However, commercial LBM systems are still restricted to the processing of a single material. The fabrication of plastic multi-material components which consist of different material zones locally separated by discrete or graded interfaces using LBM is still a subject of current research (Laumer *et al.,* 2014).

In order to realize multi-material polymer components with LBM, it is necessary to prepare powder layers which consist of arbitrary arrangements of different powder materials. Standard coating devices like blades or rollers typically used with LBM aren't capable of that. Thus, other deposition coating/deposition techniques have to be used which enable a fast powder handling with precise and reliable control over very small powder quantities.

One solution to achieve such multi-material powder layers is the application of hopper-nozzles which discretely deposit lines and dots next to each other to form patterned layers. In the recent years, reports were given which propose nozzles for micro dosing and deposition of powder materials for applications in additive manufacturing (Lu *et al.* 2006a; Stichel *et al.* 2014a) or in the pharmaceutical industry (Chen *et al.*, 2012a; Chen *et al.*, 2012b). As dosing mechanism different methods were investigated (Yang *et al.*, 2007), such as pneumatic, volumetric, screw/auger, electrostatic and vibratory methods. Among them the usage of vibration to initiate and control powder flow is one of the most promising methods and was studied by several research groups in the recent decades (Lu *et al.*, 2006a; Lu *et al.*, 2006b; Chen *et al.*, 2012a; Chen *et al.*, 2012b; Tolochko *et al.*, 2004; Jiang *et al.*, 2009; Qui *et al.*, 2011; Stichel *et al.* 2014a). It was shown that vibration excitation can improve the powders' flow behaviour by breaking down agglomerated powder clots and fluidizing it. By that continuous flow as well as a valve-like start and stop function could be achieved by vibration excitation without using any mechanical shutter (Chen *et al.*, 2012a; Jiang *et al.*, 2009; Stichel *et al.* 2014a).

In order to realize a dosing system basing on vibrating nozzles for LBM application, the temporal and spatial control accuracy of the powder mass flow is important for the repeatable generation of multi-material arrangements with defined powder zones. However, temporal and spatial accuracy is not necessarily given since the dry powder delivery is affected by the stability of the vibration mode and the current powder condition which is easily influenced by the environmental conditions including humidity and temperature (Rumpf, 1974). Moreover, the powders' flowability influences essentially the mass flow and depends on the cohesive forces between the particles (van der Waals, electrostatic interaction, etc.), which are different for different powders.

In this report, the mass flow characteristics upon vibration excitation for two nozzles with different orifice diameters is investigated in order to determine the temporal accuracy of the mass flow and to estimate the reliability of the start-stop function. Therefore a weighing cell is used enabling time-resolved mass flow measurements. Moreover, different powder materials were used to relate powder material specifications to the mass flow behaviour.

2. Experimental

2.1. Vibrating Nozzle and Characterization Setup

A scheme of the experimental setup consisting of the vibrating nozzle and measurement devices as well as a photographic image of the nozzles used are displayed in Fig. 1. The nozzles are mounted on a piezoelectric actuator in such way that a longitudinally vibration could be excited predominantly. Two nozzles (N1, N2) with different orifice diameters are used which are made out of steel by means of spark-erosion sinking. The orifice diameters are 705 ± 4 µm (N1) and 1041 ± 8 µm (N2) and the interior incident angles are 27.8° (N1) and 27.0° (N2). The piezoelectric actuator bases on a parallelogram design which ensures minimal lateral oscillations during dynamic operation with high frequencies. The sinusoidal signal of a function generator was applied to the actuator. A weighing cell allows the time-resolved measurement of the mass accumulation with a resolution of 0.001 mg and a frequency of 0.1 Hz. The measurement procedure is established as follows. First, the nozzle's reservoir is filled with a certain amount of powder. Then, the powder flow is initiated by vibration and the mass accumulation is recorded for a certain time span or till the nozzle's reservoir is empty. Finally, by the derivative of the measurement signal with respect to time, the temporal mass flow is obtained. The average mass flow resembles the average mass flow over the entire time span. Therefore, the mass flow curve was fitted with a horizontal line yielding also the standard deviation.

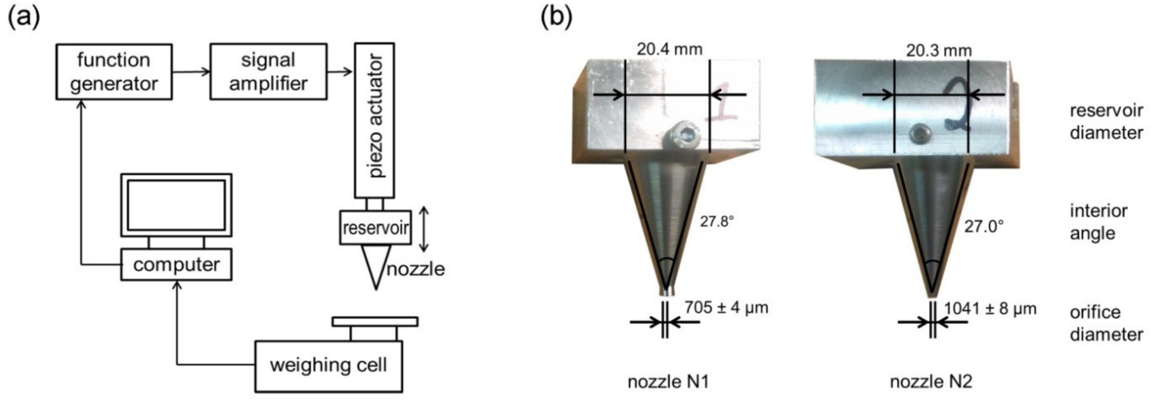

Fig. 1. (a) Scheme of the experimental setup for the mass flow measurements; (b) photographic images of the stainless steel nozzles N1 and N2 with different orifice diameters.

2.2. Powder Materials

The utilized polymer powders are listed in Tab. 1 including particle size data; SEM images of the respective powders are displayed in Fig. 2. Polyamide 12 (PA12) powder which is labeled as PA2200 was obtained from EOS GmbH (Krailing, Germany) and is mostly used with LBM applications. PA2200 is confected by precipitation process leading to potato-shaped particles with a low amount of fine particles and thus an outstanding flowability (Schmid *et al.*, 2014). Instead, polypropelene (PP) and high-density polyethylene (PE-HD) used were obtained from cryogenic milling, causing spattered particle shapes with a large amount of fine particles. However, the resulting low flowability can be improved easily by admixing Aerosil® fumed silica at a concentration of 1 wt. % (Blümel *et al.,* 2015). Moreover, carbon black at a concentration of 0.25 wt. % was added which is typically used in Simultaneous Laser Beam Melting (SLBM) of multi-materials in order to improve the absorption behaviour of the powder (Laumer *et al.,* 2014). Finally, the powder formulations were sieved to remove all particles larger than 100 µm to ensure a faultless powder delivery thourough the nozzles.

Table 1. Particle size distribution according to manufacturas' data.

Powder Material	PA 12 (EOS GmbH)	PP (DuPont)	PE-HD (DuPont)
d10 [µm]	32	60	35
d50 [µm]	55	100	57
d90 [µm]	74	150	92

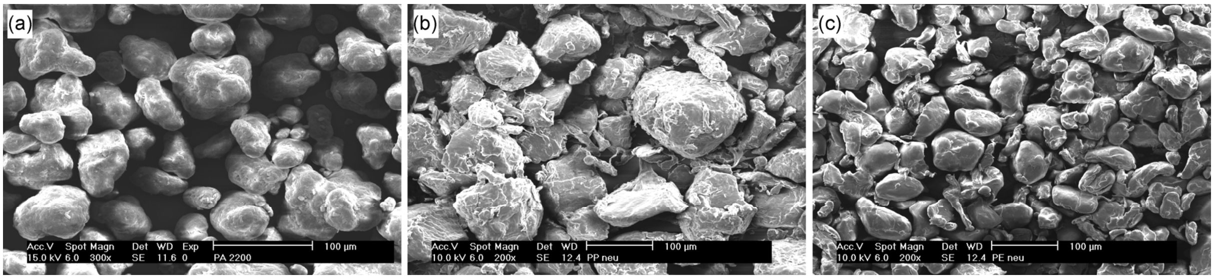

Fig. 2. SEM images of (a) PA12, (b) PP and (c) PE-HD powder.

3. Results and Discussion

3.1. Suitable Vibration Modes for Powder Dispensing

Without vibration no powder mass flow could be detected. The powder develops an arching structure in the narrow end of the nozzle which prevents the powder from falling. With applying vibration the powder flow can be initiated by breaking the powder arch. Here two kinds of effects on the powder flow have to be taken into account: compaction and dilation. While the latter results in reduced friction between the particles which increases the flowability and thus enhances mass flow, compaction reinforces friction and drags the mass flow. The predominating effect is determined by the powders' properties (morphology, mechanical properties, etc.) and the nozzle's geometry (orifice diameter, interior angle) as well as by the vibration modes which are defined by the vibration frequency, amplitude, orientation, and shape.

Not all vibration modes which result in sufficient mass flow are suitable for selective powder dispensing. A narrow discharge characteristic is also important for good dosing performance which is not necessarily given at high mass flows. Unsuited vibration modes can lead to strong dispersed or even eruption-like discharge which cannot be used for high-selective powder deposition [Stichel *et al.* 2014b].

Since the vibration characteristic can be quite complicated and strongly depends on the piezo actuator design used as well as on the nozzle setup geometry and mass, a detailed characterization and discussion of different vibration modes is spared. However, prelimariy tests revealed that a stable and reproduceable vibration mode with a minimum of parasitic oscillations as transversal oscillations or higher order modes are realized for a frequency of 420 Hz with a piezo voltage of 50 V. Moreover, this parameter set leads to a quite narrow discharge characteristic with a sufficient mass flow and thus is applied in the following experiments.

3.2. Start-Stop Function

In order to qualify the start-stop function and mass flow regarding time stability, the piezo was activated for 20 seconds and paused again for 5 seconds, while the decharged mass was measured using the time-resolving weighing cell. The development of the mass over time is differentiated to determine the mass flow. Since the weighing cell is highly sensitive to environmental influences, curves of the mass flow need to be smoothed using adjacent averaging.

A phenomenon which could be observed at the different parameter sets is excessive mass flow after the piezo was actuated for the first time. In both diagrams of Fig. 3, each belonging to a different nozzle, a significant peak can be remarked which is circled in red. For nozzle N 1, the initial mass flow peak is two times higher than the mass flow levelling off over time, whereas the initial peak for nozzle N 2 at the given vibration mode is up to six times higher. Obviously, the significant difference in the peaks as well as in average mass flow results from the wider orifice diameter of nozzle 2. The reason for the initial peak is the relatively low bulk density of the powder inside the nozzle before vibration. When the vibration is activated for the first time the powder arch in the narrow end of the nozzle breaks and an entire flush of powder with low bulk density is discharged. This effect, however, is less intense when the vibration is activiated for the second and following times since the powder is already compacted due to the prior vibration step. As a consequence, the peaks are very small and can be misjudged for background noise.

Since the initial mass flow flush can be easily prevented by precompression by preceding vibration application with blocked nozzle, we can now judge the start-stop function to be functional for both nozzles. Of course the larger orifice diameter of nozzle N2 leads to a higher mass flow of around 2.6 mg/s during vibration activation compared to 1.3 mg/s achieved with nozzle N1. However, the mass flow level of the activation regimes seems to be slightly decreasing over time for both nozzles which can be attributed to the decreasing powder level inside the nozzle leading to slightly varying bulk density condition over time. But this can easily be solved by continuous leveling off the powder amount inside the nozzle using screw/auger methods.

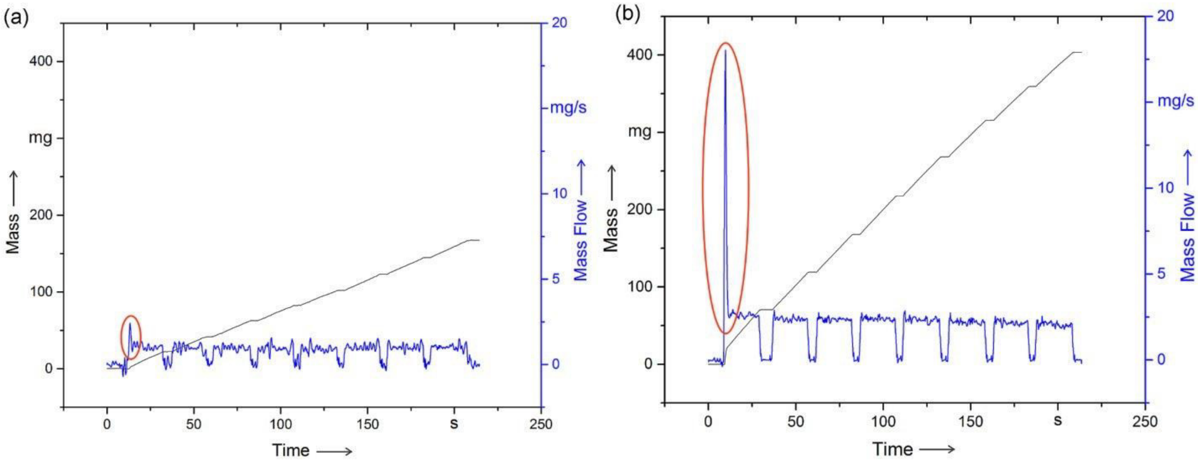

Fig. 3. Development of mass and mass flow over time of nozzle 1 at 420 Hz, 50 V (left) and nozzle 2 at 420 Hz, 50 V (right) using PA 12 powder. The initial excesive mass flow (peak) is circled with red color.

3.3. Mass Flow of different Powder Materials

The average mass flow of different powder materials using nozzle N2 can be seen from Fig. 4a. When the raw powders without additives are compared, the results confirm the expectations of PA 12 being the powder with the best flowability. Due to the potato-shape and the small amount of small particles the powder flows with a average mass flow of 3 mg/s with very small fluctuations at a relative standard deviation of 8 %. PE and PP are not as beneficially shaped, but spattered with a high amount of small particles leading to agglomeration and thus to a low flowability. Consequently, PE flows at a rate of only 0.9 mg/s while PP at 1.3 mg/s. Moreover, PE in particular shows a large standard deviation indicating an irregular powder output resulting from excessive agglomeration due to interlocking of the spattered particles. In Fig. 4cd, deposited mass of PA 12 is compared to mass of PE. The pictures clearly demonstrate the tendency of PE (PP likewise) to agglomerate. While PA 12 spreads evenly over the vessel, PE forms a heap with steep slopes out of agglomerated units.

In order to improve powder discharge, flowability enhancing additives such as Aerosil® fumed silica can be admixed. This clearly enhances mass flow to about 3 mg/s for PE and PP which is then similar to the mass flow of pure PA12. But for PA12 no increase of mass flow could be detected when using Aerosil® which shows that the improvement of the flowability using Aerosil® is limited to a certain range. The additional admixing of carbon black is not changing the mass flow significantly. However, all values including powder with Aerosil® admixed, possess quite high standard deviations between 25 and 35 %. With carbon black it goes further up to over 40 %. A possible explanation could be an inhomogeneous mixing process. Since the powder were mixed manually, Aerosil® and carbon black might segregate partially resulting in powder zones of vastly varying flowability.

Finally, pure PA12 has shown to be most suitable for selective powder deposition using vibrating nozzle. High mass flows of about 3 mg/s were achieved with low standard deviation. The addition of Aerosol® which is essential for PE and PE to achieve mass flows around 3 mg/s comes along with large standard deviation indicating a inhomogeneous powder mixture condition.

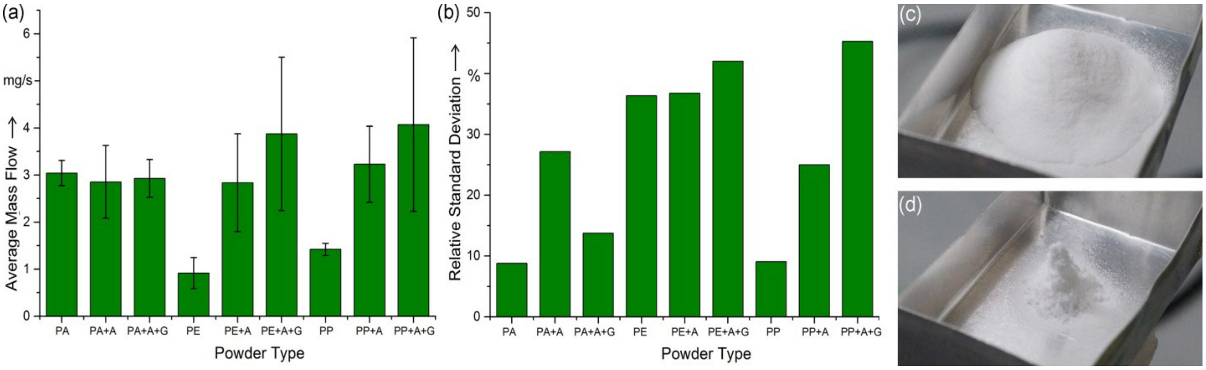

Fig. 4. (a) Average mass flow using nozzle N2 and (b) corresponding standard deviation of different powder formulations including the powder materials polyamide 12 (PA), high-density polyethylene (PE) and propylene (PP) as well as the additives Aerosil® (A) and carbon black (G); Photographic image of dispensed (c) polyamide 12 and (d) polyethelene powder. The standard deviation is calculated from the temporal mass flow curve with respect to an ideal constant temporal mass flow.

4. Conclusion

In this report, the delivery of polymer powder by vibrating nozzles was investigated with respect to their application in LBM machines. Therefore, steel nozzles attached to a piezo actor and a weighing cell were used in order to investigate the stability and time-dependence of the powder mass flow upon vibration excitation with the usage of different kind of powder formulations.

For the mass flow measurements a certain vibration mode at 420 Hz and a piezo voltage of 50 V was applied. The results show that precompression of the powder inside the nozzle by vibration excitation is essential to realize a reliable start-stop function with reproducible discharge cyles and to prevent a initial flush of powder flow which was shown to be very strong for the nozzle with the larger orifice diameter. Generally, a slight decrease of mass flow level of the activation regimes over time for both nozzles was detected which was attributed to the decreasing powder level inside the nozzles leading to slightly varying bulk density over time due to mass reduction. This might be solved by continuous leveling off the powder amount inside the nozzle using screw/auger methods.

Moreover, the use of different powder materials revealed that pure PA12 (PA2200, EOS GmbH) is most suited for selective powder deposition using vibrating nozzle resulting in a mass flow of 3 mg/s with a low standard deviation of 8 % . Nevertheless, mass flow is even possible with powders which are not optimized regarding flowability basing on PE or PP, but is readily enhanced with a factor of 2 to 3 by admixing 1 wt. % Aerosil®. However, the standard deviation also increases using additives which is most likely caused by inhomogeneous mixture resulting in powder zones of vastly varying flowability.

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