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Selective deposition of polymer powder by vibrating nozzles for laser beam melting

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Abstract

In this report, the delivery of polyamide 12 (PA 12) powder and powder layer preparation by vibrating steel nozzles is investigated and discussed with respect to its application for laser beam melting. Therefore, a setup was realized which includes a steel nozzle attached to a piezo actor as well as a positioning system. In order to investigate the mass flow characteristics in dependency on the applied vibration state, a weighing cell is used enabling time-resolved mass flow measurements. Moreover, single-layer patterns consisting of colored and uncolored polyamide 12 were created and characterized regarding surface homogeneity and selectivity before as well as after the melting of the powder layers by a hot plate.

Keywords: laser beam melting, polyamide 12, vibrating nozzle, multi-material

1. Introduction

Laser Beam Melting (LBM) of polymer powders is one of the most popular additive manufacturing processes (Kruth, 1991; Levy et al., 2003). Even though LBM allows a high degree of freedom of design, usual LBM machines are still restricted to process single materials. The fabrication of components which consist of different material regions locally separated by interfaces using LBM is still a subject of current research. Progress has been made using Simultaneous Laser Beam Melting (SLBM) (Laumer et al., 2014; Laumer et al., 2015) which shall be able to process different powder materials even with different optical and thermal properties and thus shows great potential for the fabrication of multi-material components. By applying a simultaneous irradiation with adjustable intensity distribution on a large area, different polymer powders deposited next to each other within one layer can be transferred simultaneously from solid into melt.

However, the accurate preparation of arbitrary multi-material powder layers is still unsolved. Since standard coating devices of LBM machines basing on blades or rollers are not capable, new solutions are required which enable a fast powder handling with a spatial precision of a few dozens of micrometers. A possibility to realize such layers may be nozzle devices which discretely deposit dots and lines next to each other to form patterns. As regulatory mechanism for powder flow, vibration of the nozzle has been shown to be a promising method for adjusting the powder mass flow and thus was studied by 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). They revealed that vibration excitation can improve the powders' flow properties by breaking down agglomerated powder particles and fluidizing it which is especially beneficial for powders with low flowability. It was observed that continuous flow as well as a valve-like start and stop function can be achieved by vibration (Chen et al., 2012a; Jiang et al., 2009).

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 fabrication of multi-material arrangements with defined powder zones. This is important since 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 easily affected by the environmental conditions (Rumpf, 1974).

In this report, the mass flow and discharge characteristics of vibrating steel nozzles depending on the applied vibration state are investigated for the verification of stable process modes and mass flow influencing factors. Therefore a weighing cell is used enabling time-resolved mass flow measurements. Moreover, using a nozzle configuration including a positioning system, lines and patterns consisting of visually distinguishable coloured and uncoloured polyamide 12 were fabricated in order to demonstrate multi-material capability and potentials regarding deposition selectivity and homogeneity.

2. Experimental setup and material

A scheme of the experimental setup consisting of the vibrating nozzle and measurement devices as well as a photographic image of the nozzle assembled on a positioning system are displayed in Fig. 1.

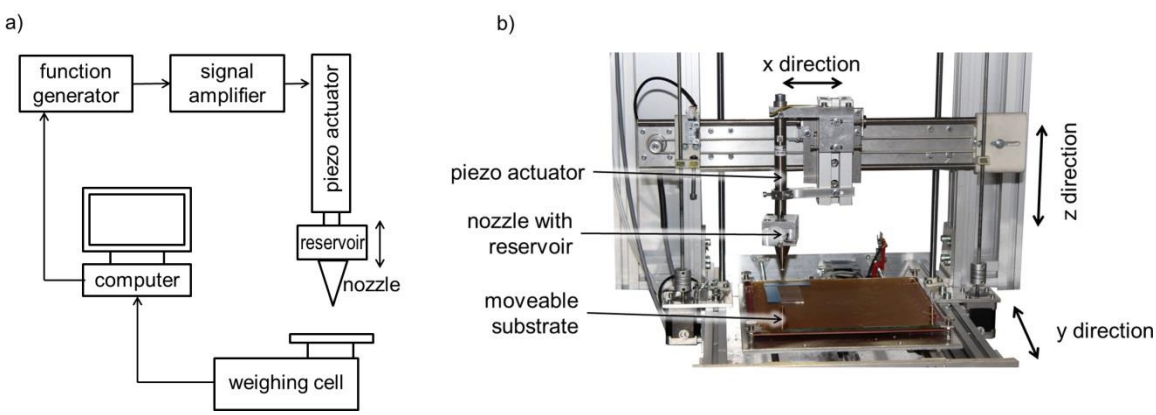

Fig. 1. (a) Scheme of the experimental setup for the mass flow measurements; (b) photographic image of the vibrating nozzle setup attached to a positioning system for the preparation of powder patterns.

The nozzle is mounted on a piezoelectric actuator in such way that a longitudinally vibration excitation could be performed 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 from PI (Karlsruhe, Germany) can be cooled by air which ensures a constant temperature during dynamic operation with high frequencies. The sinusoidal signal of a function generator was applied to the actuator. A weighing cell from Wipotec (Kaiserslautern, Germany) 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 1 g (N1) or 2 g (N2) powder. Then, the powder is precompressed by applying vibration with blocked nozzle in order to achieve a reproducible start condition. After unblocking the nozzle the powder flow is initiated by vibration and the mass accumulation is recorded 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.

In order to deposit arbitrary patterned powder layers the vibrating nozzle was attached to a positioning system. With it the nozzle can be moved along x direction while the substrate below the nozzle travels along the y direction.

The used polymer powder was polyamide 12 (PA12) which was obtained from EOS GmbH (Krailing, Germany) and thus is labeled PA2200. It features an average particle size of 58 µm according to manufacturers' data. 80 % of the particles are in the range between 40 and 90 µm. PA2200 is typically used with LBM applications. Before the measurements, the powder was sieved at 100 µm pore size in order to remove contaminations.

3. Mass flow characteristics

Using both nozzles (N1, N2) filled with PA12 powder without applying vibration, no mass flow can be detected. The powder develops an arching structure in the narrow end of the nozzle which prevents the powder from falling. By applying vibration the powder flow can be initiated by breaking down the arching structure blocking the narrow end of the nozzle. The resulting amount of mass flow is then defined by the flowability of the powder which is strongly manipulated by the vibration mode affecting the density state of powder. Here two kinds of effects 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 effect that predominates is determined by the powder properties (bulk density, particle size, etc.) and the nozzle geometry as well as by the vibration modes.

In order to prepare multi-material arrangements with defined powder zones precisely, the temporal control accuracy of the powder mass flow is essential. Since it is known that the mass flow is affected by the frequency, the amplitude, and the interaction between them (Stichel et al., 2014a; Stichel et al., 2014b), adapting the vibration modes seems to be predestinated for precise powder mass flow control. The easiest control can be expected if an ideal mass flow characteristic is achieved by a certain vibration mode which resembles a temporal constant value directly after vibration excitation and stays constant even with decreasing powder mass inside the nozzle. In that case the control could be confined to the start-stop-function of the vibration excitation.

However, since the flowability and thus the powder flow through a nozzle is affected easily by the experimental and environmental conditions an ideal mass flow is not expected necessarily. In order to point

out possible mass flow deviations und their origins the temporal mass flow is determined for different vibration modes and two nozzle sizes.

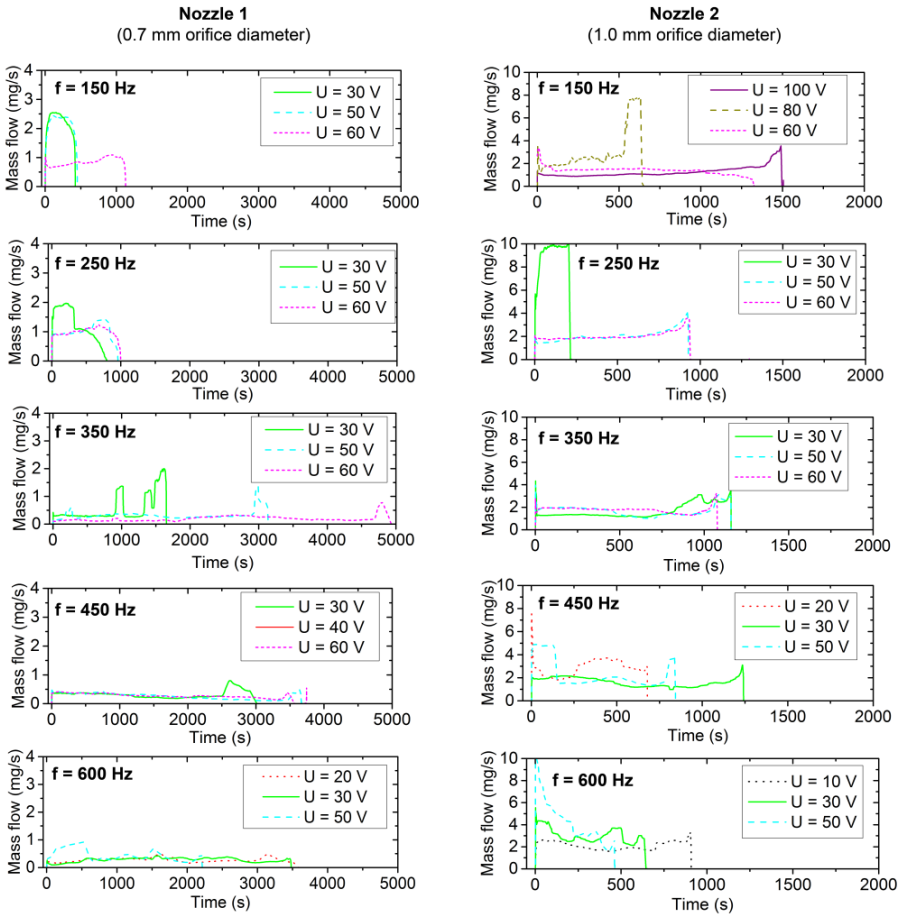

Fig. 2. Temporal mass flow measurements in dependency on the different vibration modes for two different nozzle sizes: Vibration modes are parameterized by amplitude voltage of the piezo actuator and the vibration frequency.

3.1. Influence of vibration frequency and piezo voltage on the mass flow

The vibration mode is resembled by the vibration frequency and the amplitudes of the vibration. Despite the piezo actuator is built for translations along the longitudinal direction, parasitic oscillations along the lateral direction cannot be excluded for this setup (Stichel, 2014). That means that even if we assume perfect sinusoidal oscillation shapes, seven parameters resemble the vibration modes - the frequency, the amplitudes along each direction (x, y, z), and the phases of each component. A change of the voltage would not affect the frequency, but might lead to an unpredictable response of the other parameters.

Nevertheless, it can be expected that increasing voltage leads to increasing absolute vibration amplitudes in most cases (Stichel, 2014). This brings about higher acceleration forces and thus stronger mechanical

stress upon the particles inside the nozzle, which may drag the flowability due to enhanced powder density, electrostatic charging or interlocking.

The results of Fig. 2 show that frequency and voltage affect the mass flow strongly. In dependency on them, mass flows up to 2.5 mg/s for nozzle 1 and 10 mg/s for nozzle 2 were achieved. While the mass flow characteristic differs strongly with the chosen frequencies, it is remarkable that even small changes in voltage can lead to strong variations of the mass flow which shows the sensitivity of the powder state inside the nozzle regarding to the vibration mode. However, it can be seen that at each frequency the mass flow is mostly reduced with increased voltage signal. This confirms the expected theory that increasing voltage leads to higher acceleration forces and thus the drag of the mass flow due to enhanced powder density, electrostatic charging or interlocking.

Moreover, the results show that severe deviations from the ideal mass flow characteristic (mass flow constant in time) occur. Nearly all parameters result in mass flow curves with a deviation at their beginning and end. At their beginning the particle systems needs time to relax into a certain density state with a more or less constant mass flow. The longest time spans with around 50 s are found with the lowest frequency of 150 Hz which indicates that the relaxation time depends on the vibration frequency. An exception to this is the mass flow with 600 Hz and nozzle 2 which shows a decreasing for ca. 200 s at the beginning in a highly unstable manner. The reason for this is unclear. Maybe electrostatic charging increases with increasing time which affects the powder's flowability by promoting agglomerations.

Right before the end of the mass flow measurement, we can observe an increase or a decrease of the mass flow. This happens because of the powder mass inside the nozzle approaches a zero value which leads to dilution of the powders density affecting the resulting mass flow. The different behavior (increase or decrease of the mass flow) reveals that two different discharge mechanisms are existent, whereas the predominant effect determines if mass flow decreases or increase before the end. First mechanism is resembled by a gravity-driven mass flow. This is provided by the combination of vibration and increasing dilution which leads to a powder state with increasing flowability and thus to an increasing mass flow through the nozzle. Instead, if mass flow is driven by vibration acceleration, a dilution of the powder state leads to discharge packages with lower dense resulting in a decreasing mass flow at the end. The reason for which effect predominates cannot be excluded from the experiments since the vibration characteristics are unknown for the experiments.

While the varying powder mass effects clearly the mass flow at its end, it can be suspected that an influence during the whole discharge time is existent. In order to clarify that influence, it is important to sort out the kind of affection of the powder mass onto the system. Since we know that the powder density state is affected by the powder mass which leads to the deviation towards the end of mass flow, an influence of the powder mass in the earlier and middle parts of the mass flow might be reasoned by the powder mass affecting the vibration mode of the system. This is yet unclear and requires an online-observation system of the vibration characteristic of the system, which was not performed yet. The affection of the vibration mode might also be a reason for the unstable mass flow characteristics of, for example, the parameter sets with 600 Hz.

Despite the unanswered questions regarding the affection of the powder mass variation on the vibration and mass flow behavior, results show that some vibration modes yield mass flow with quite constant values over time and thus could be useful for successful powder layer preparation. While the mass flow deviations at the end can easily be excluded by an incremental addition of powder for example by a screw extruder, possible mass flow variation at the beginning (after initiating the mass flow) have to be compensated by adjusting the vibration mode or adapting the deposition velocity during pattern preparation.

3.2. Influence of the orifice diameter

Comparing the mass flow results of the two nozzles (N1, N2), the influence of the orifice diameter can be derived. The measurements show that the nozzle N2 with the larger orifice diameter enables larger mass flows than nozzle N1 at the same frequencies and voltages. Hereby increases by factors between 2 and 5 can be roughly estimated.

When interpreting the mass flow measurement results for both nozzles, one should be aware that the vibration response of the nozzle might be totally different even at the same amplitude voltage. There are slight geometrical differences between the nozzles at their reservoirs which also result in different masses. While N1 possesses a mass of 82 g and N2 weighs 77 g, the different mass inertias may lead to the different vibration amplitudes with the same excitation force (defined by the amplitude voltage).

The general mass flow behaviors of the nozzles seem to differ systematically. The mass flows for nozzle 2 with the larger orifice diameter are more stable which indicates that the system or the discharge mode is less sensitive to unmeant variations like vibration mode, density state or electrostatic charge variations. This hints to gravity-driven mass flow which can be expected to be less sensitive to vibration variations since the vibration just breaks down the powder arches and enhances the flowability, but not induces directly the discharge of powder packages. A second hint for the gravity being the dominant driving force is that the mass flow deviation at the end of curves usually increases with nozzle 2, while the mass flow decreases with nozzle 1.

The generally higher mass flows of nozzle 2 allow much faster preparation of layers which is important since time consumption affects the cost-effectiveness in additive manufacturing. Choosing the mass flows 2.5 mg/s for nozzle 1 at f = 150 Hz and U = 30 V and 10 mg/s for nozzle 2 at f = 250 Hz and U = 30 V, we estimate a preparation time of 5.8 min (N1) and 1.5 min (N2) for a layer with an area of 100 x 100 mm^2 and a height of 0.2 mm when assuming a bulk density of 0.435 g/cm^3. Since these preparation times are up to two orders higher than the preparation times which are usually achieved by common blade or roller devices, solutions like parallelization techniques should be considered in the future (e. g. nozzle arrays).

4. Powder deposition and spatial control of the mass flow

Next to the temporal control, the spatial control accuracy of the powders' mass flow is also important for the repeatable realization of multi-material arrangements with arbitrary powder zones. In this regard, the characteristic (width, height, contours) of deposited powder lines resemble the system's potential concerning selectivity and homogeneity. Using the setup shown in Fig. 1b with both nozzles, lines and patterns were produced at room temperature with different deposition velocities and characterized before and after melting. Therefore, the vibration mode with a frequency of 450 Hz and an amplitude voltage of 30 V was used since it yields quite temporal constant mass flow values for both nozzles. Melting was simply achieved by using a hot plate. For the investigation a laser scanning microscope (LSM) was used. The graphical analysis of a single line made from PA 12 is exemplary displayed in Fig. 3.

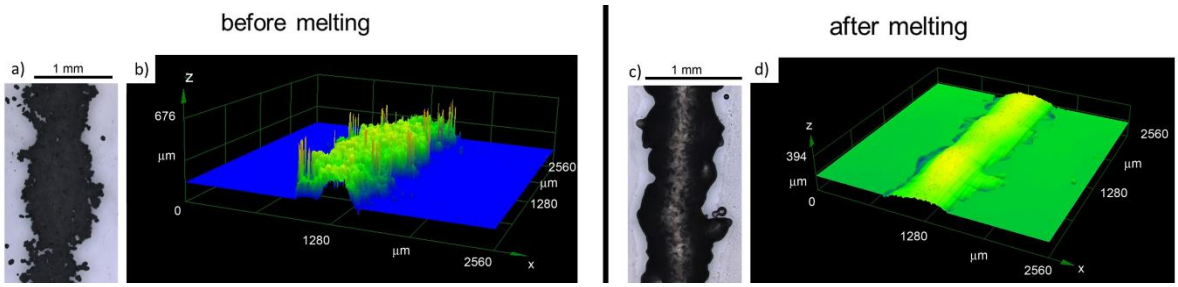

Fig. 3. (a, c) microscopic top-view, and (b, d) three-dimensional laser scanning microscope images of polyamide 12 powder line (a, b) before and (c, d) after melting by a hot plate. Powder line is generated with nozzle 2 at deposition velocity of 13 mm/s, a vibration frequency of 450 Hz, and a voltage amplitude of 30 V.

Before melting, the line shown in Fig. 3 produced with a deposition velocity of 13 mm/s possesses a maximum width of 875 µm, a maximum height of 221 µm, and an average cross section of 0.113 mm^2. It is remarkable that the line width is smaller than the orifice diameter which shows that a very narrow discharge characteristic is achieved with the vibration mode used. During melting, the dimensions shrink clearly due to the coalescence of the polymer particles. While the width of the line is reduced by about 20 % to 704 µm, the height shrinks to 110 µm which means a reduction of 50 % regarding to the original value. Consequently, the cross section is reduced by about 60 % to 0.045 mm^2.

Varying the velocity between 3 mm/s and 67 mm/s, the dimensions of the powder lines can be adjusted in a certain range. That way, line widths between 0.5 and 2.5 mm and heights between 0.05 and 2.00 mm were realized with nozzle 1 and line widths between 0.7 and 3.5 mm and heights between 0.09 and 3.00 mm with nozzle 2. Of course heights and widths are decreasing with increasing velocity, but at higher velocity the powder lines' edges tend to be more frazzled than with lower velocity. However, if the velocity is too low, powder lines reach heights beyond 500 µm which are not useable for LBM application. Very good homogeneous line appearance (no frazzles) was achieved when using a velocity of 13 mm/s.

In order to study the quality of interfaces of the powder deposition, parallel lines with visually distinguishable powders are prepared. In Fig. 4 images are presented which show the fusion of two powder lines aligned with different hatch distances, consisting of pure PA12 and PA12 formulated with 1 wt. % chrome oxide particles, respectively. Nozzle 2, a deposition velocity of 13 mm/s, a frequency of 450 Hz and a voltage of 50 V was used for this experiment resulting in powder line widths of about 1300 µm (PA12) and 1150 µm (PA12 with chrome oxide).

The results show that the fusion zone as well as the line topography is strongly affected by the hatch distance. A hatch distance of 0.6 µm provides a curved surface even after melting which indicates a too small hatch distance. Moreover, the cross section image reveals a broad fusion zone which is reasoned by the large overlap of the deposited lines. Since the lines are prepared successively, the doped PA12 powder is predominately present in the upper region of the fusion zone while the pure PA12 is settled beneath it. Best results at the used parameters are realized with a hatch distance of about 1.0 mm. Here, the narrowest fusion zone, an even surface above the fusion zone, and a homogeneous layer thickness of about 80 µm are achieved. Instead, when applying a larger hatch distance of 1.2 mm, a dip in the area of the fusion zone occurs due to the marginal overlap.

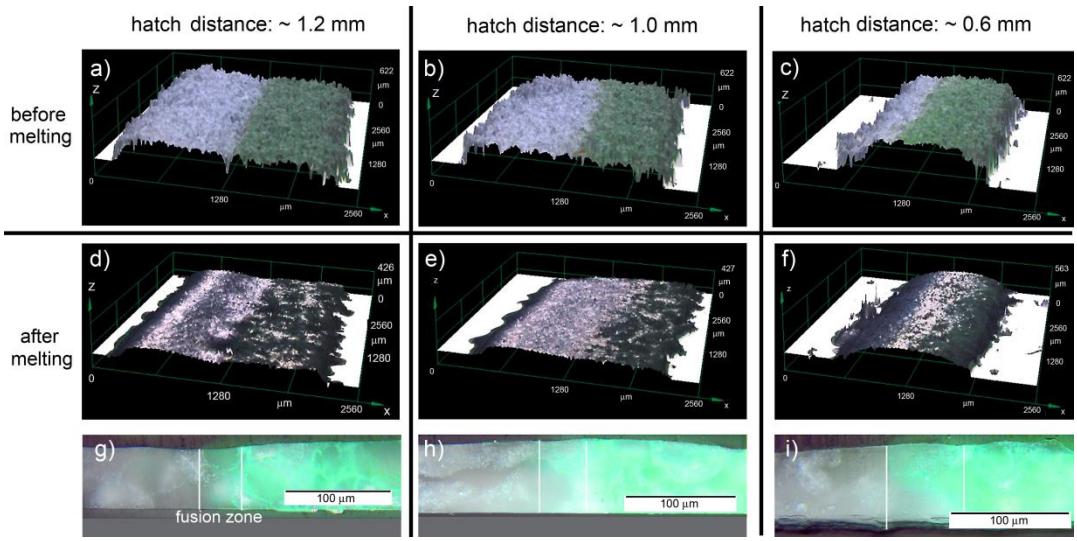

Fig. 4. (a-f) Laser scanning microscope images and (g-i) cross sections of two parallel overlapping lines produced with different hatch distances. The materials used is polyamide 12 and polyamide 12 formulated with 1 wt. % chrome oxide particles.

5. Conclusion

In this report, the powder discharge control and selective deposition of polyamide 12 (PA2200) using a vibration-controlled steel nozzle was investigated with respect to its application in laser beam melting. The temporal mass flows were determined using a weighing cell with two nozzle sizes and different vibration modes in order to identify the potential for accurate powder deposition and possible control strategies. The results show that systematic temporal variations of the mass flow are existent which depend on the vibration modes and nozzle orifice diameters. Mass flows with quite constant values were detected for 150, 250, 350 and 450 Hz at distinct amplitude voltage.

A typical systematic variation is found directly after initiation the powder flow at which the particle system needs several seconds to relax into a certain density state with a more or less constant mass flow. In order to establish a deposition strategy for the precise deposition of powder lines or patterns on that base, it is suggested to compensate this behavior by adapting the deposition velocity regarding to the mass flow variation.

However, generally, there are temporal mass flow variations whose origins are unclear so far. Hence, it is necessary to establish a more detailed understanding of the influencing factors including vibration mode and electrostatic influences. Therefore, an online-observation system will be built up to track the vibration during mass flow as well as a Faraday cup electrometer for the measurement of the charge condition of the deposited powder.

Next to the temporal control, the spatial control accuracy of the powders' mass flow is also important for the repeatable realization of multi-material arrangements with arbitrary powder zones. Experiments show the current reachable specifications of the vibrating nozzle setup with respect to spatial resolution, selectivity, and homogeneity. In this regard, analyses identify the hatch distance of the powder lines as an important determining factor for powder layer preparation and show that line widths between 0.5 and 3.5 mm and heights between 0.05 and 2.00 mm could be achieved with the presented setup.

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