Multi-Material Deposition of Polymer Powders with Vibrating Nozzles for a New Approach of Laser Sintering

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Conventional Selective Laser Sintering of polymers is restricted to the processing of single materials. The fabrication of components consisting of different material regions is a challenge which cannot be realized by standard coating devices basing on blades or rollers. Thus, advanced coating and deposition techniques are needed which enable the precise and reliable control over very small powder quantities in order to prepare arbitrary powder patterns with high accuracy and repeatability. In this report, the delivery of polymer powder by vibrating nozzles inside laser sintering machines is investigated. Therefore, a steel nozzle attached to a piezo actuator is integrated into a machine, whereas the nozzle itself features internal channels which allow the precise control over the powder temperature using heat transfer oil. The setup is used to study the influence of different system configurations on the powder deposition characteristics as resolution and layer surface roughness. The results show that temperature influences the mass flow depending on the material used and that a precise deposition of powder pattern with microscale resolution is possible with optimized parameters. Finally, the multi-material powder pattern is fused by a new illumination strategy of laser sintering which is called Simultaneous Intensity-Selective Laser Sintering in order to demonstrate the potential of this new approach.

DOI: 10.2961/jlmn.2018.02.0002

Keywords: Selective Laser Sintering, vibration-controlled powder nozzle, selective powder deposition, multi-material, polypropylene, thermoplastic elastomer, polymer powder

1. Introduction

Several freeform fabrication techniques are established in industry, which enable the direct generation of three-dimensional design models or prototypes. While techniques as 3D-printing or Fused Deposition Modeling (FDM) yield samples which usually show a poor mechanical stability, powder bed based freeform fabrication techniques like Selective Laser Sintering (SLS) of polymers or Laser Beam Melting (LBM) of metals can provide advanced mechanical properties [1]. Thus, powder bed based technologies are useful for the fabrication of functional devices even for high performance applications including aerospace, tooling for injection moulding, medical devices or automobiles [2, 3]. However, commercial systems are still restricted to the processing of a single material. The fabrication of multi-material components which consist of different materials locally separated by discrete or graded interfaces using SLS or LBM is still a subject of current research [4, 5].

In order to realize multi-material polymer components with SLS, it is necessary to coat powder layers consisting of arbitrary arrangements of different powder materials. Typically used coating devices like blades or rollers cannot fulfill this requirement. Thus, other deposition techniques have to be developed, which enable a fast powder handling with precise and reliable control of 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 recent years, frequent reports were given which propose nozzles for micro dosing and deposition of fine powder materials for applications in additive manufacturing [5, 6] or in the pharmaceutical industry [7, 8]. As dosing mechanism different methods were investigated [9], 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 [5-13]. It was shown that vibration excitation can improve the powders’ flow properties by breaking down agglomerated powder clots and fluidizing it which is especially beneficial for fine powders with low flowability. Moreover, it was observed that continuous flow as well as a valve-like start and stop function could be achieved by vibration excitation [5, 7, 12].

With respect to the application of vibrating nozzles inside SLS machines, the temperature dependency of the powder discharge of polyamide 12 was studied by Stichel et. al. recently [14]. They discovered that temperature significantly influences the mass flow of PA12 powder through a nozzle which was related to the changing humidity of the powder affecting the particle-particle-adhesion forces [14]. Thus, the control of temperature is a fundamental demand in order to assure constant mass flows which is important for the repeatable generation of multi-material arrangements with defined powder zones.
For the application of multi-material components, the dimensional stability and selectivity of the different material regions are also very essential. Thus, the minimum dimension of discharged powder dots and lines is of high interest since they define the spatial control accuracy if we assume perfect positioning of the nozzle.

In this paper, the delivery of polypropylene (PP) and thermoplastic elastomer (TPE) powder by vibrating nozzles inside a laser sintering machine is investigated. Therefore, a steel nozzle attached to a piezo actor is integrated into the machine, whereas the nozzle itself features internal channels which allow the precise control over the powder temperature using heat transfer oil. The setup is used to study the influence of temperature on mass flow and the powder deposition characteristics resolution and layer surface roughness. Finally, a multi material pattern of PP and TPE is prepared and fused using a new exposure strategy which is called Simultaneous Intensity-Selective Laser Sintering (SI-SLS).

2. Experimental

2.1 Vibrating nozzle module

A heatable vibrating nozzle module was integrated into a modified laser sintering machine for selective powder deposition during the laser sintering process. A photographic and rendered image of the vibrating nozzle module integrated into the SLS machine is shown in Fig. 1.

The nozzle including a powder reservoir is made of stainless steel using LBM technology. During post processing, the nozzle’s interior surface was smoothed by spark erosion and a polishing step. The cone-shaped nozzle with a cone angle of 40° and an orifice diameter of 0.7 mm possesses internal channels which can be filled with tempered oil. Using an oil circulation heater, the nozzle temperature and thus the powder temperature can be adjusted in limited temperature range. The oil temperature can be varied between 30 and 150 °C.

The nozzle is mounted on a temperature-resistant piezoelectric actuator, enabling a longitudinally vibration excitation. The vibration mode (frequency, amplitude, shape) is adjusted by a function generator. A scheme of the setup is shown in Fig. 2.

2.2 Simultaneous Intensity-Selective Laser Sintering

The machine is equipped with a Thulium laser (wavelength of 1.94 µm) with a controllable micro mirror array in addition to the typical CO₂ laser (10.6 µm), enabling the processing of powder patterns of different powder materials by simultaneous intensity-selective illumination [4]. It allows the application of the quasi-isothermal laser sintering process route for multi-material powder patterns, whereas locally different preheating temperatures are established for different material regions with different melting temperatures. The principle of SI-SLS is shown in Fig. 3.
The process starts with the deposition of two different powder materials next to each other by vibrating nozzles. The whole building chamber and thus both powders are warmed by infrared emitters to the preheating temperature of the powder with the lower melting temperature. At the same time, the second polymer, which has the higher melting temperature, is locally warmed to its preheating temperature by CO₂ laser radiation. To achieve a homogeneous energy and thus homogeneous temperature distribution on the powder surface, a high-speed scanner with a scanning speed up to 25 m/s is used for quasi-simultaneous preheating of the higher melting polymer. After both polymers are warmed to their preheating temperatures, the specific layer geometry is molten simultaneously by the Thulium laser and the micro-mirror array. Each of the nearly two million micro-mirrors can be tilted between two angles with an individual tilting frequency. One tilting angle guides the incident beam onto the powder bed, whereas by the other tilting angle the beam is guided into a beam trap. By varying the tilting frequency, locally graded intensity profiles and therefore temperature distributions can be realized in the powder bed. After both materials are in molten state, the building platform is lowered, before the process steps are repeated. The energy deposition and resulting temperatures on the powder surface are controlled by a thermal imaging system.

### 2.3 Powder materials

The utilized polymer powders are polypropylene (PP, PD0580 Coathylene, DuPont) and a polyamide based thermoplastic elastomer powder (TPE, TPE 210-S, Advanced Laser Materials). PP and TPE feature average particle sizes of 100 and 85 µm respectively, according to manufacturers’ data. Since both raw powders possess a low flowability, 1.0 wt. % Aerosil® (R106, Evonik), nano-scaled silicon dioxide particles, is admixed to the powders. The absorbance of the powders is improved by admixing 0.25 wt. % nano-scaled carbon absorption intensifier. The powders and additives are mixed for 60 minutes by using a turbula mixer [4]. The melting temperatures of the powders are 132 °C for TPE and 165 °C and for PP.

### 2.4 Characterization of the mass flow

A weighing cell from Wipotec (Kaiserslautern, Germany) is placed below the nozzle, allowing 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 powder. After the temperature of the powder inside the nozzle is adjusted using the oil circulation heater, the powder is precompressed by applying vibration with blocked nozzle. After unblocking the nozzle the powder flow is initiated by vibration and the mass accumulation is recorded. Finally, the temporal mass flow is obtained by smoothing and derivation of the measurement signal with respect to time.

### 2.5 Characterization of powder lines and layers

The surface topography of prepared powder lines and layers is determined by a laser stripe sensor from Micro-Epsilon Messtechnik (Ortenburg, Germany). It enables 2D profile measurements with a sampling frequency of 200 Hz and a resolution of 40 µm at a stripe length of 50 mm. In order to obtain three-dimensional surface topography data from experiments, the sensor is mounted on a positioning system in such a way that the laser stripe is aligned perpendicular to the moving direction of the positioning system. During the measurement, the substrate moves with a speed of 1.25 mm/s, enabling a resolution of 50 µm at the chosen sampling frequency of 25 Hz.

### 3. Results and Discussion

#### 3.1 Characterization of the system’s temperature profile

The temperature of the powder in the nozzle at different oil temperatures was determined by measuring the temperature of the powder inside the nozzle by three thermocouples for different oil temperatures with a sampling rate of 0.1 s. Thermocouple I was placed directly inside the nozzle tip while thermocouple II and thermocouple III were arranged above thermocouple I in a distance of 20 and 40 mm respectively. A description and the measurement results are shown Fig. 4.

![Fig. 4 Temporal measurement of the temperature at three different places inside the nozzle at different oil temperatures.](image)
placed inside the powder near the powder surface with the largest distance from the nozzle’s body, significant lower temperature levels are reached compared to the other measurement places.

In the following sections, all presented temperature values are the values measured using thermocouple II.

3.2 Suitable vibration mode for powder dispensing

In case of an established start-stop configuration, no powder mass flow can be detected without applying vibration. 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 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 [5].

Since the vibration characteristic can be quite complicated and depends strongly on the piezo actuator design used as well as on the nozzle geometry and mass, a detailed characterization and discussion of different vibration modes is difficult.

However, preliminary tests using PP and TPE powder revealed that suitable vibration modes exist, enabling stable and reproducible mass flows and a reliable start-stop function for the nozzle used. For PP and TPE, these criteria were fulfilled for a piezo voltage of 50 V and frequencies of 300 Hz and 150 Hz respectively when using a sinusoidal oscillation shape. A more detailed discussion of the influence of vibration mode on the mass flow is given in [5].

3.3 Influence of temperature on mass flow

It can be expected that the amount of temperature influences the dosing with nozzles. In order to quantify the influence, mass flow measurements were done with PP at different temperatures between 30 and 130 °C. Therefore, the nozzle was filled with a defined amount of powder, heated till equilibrium, and excited by a frequency of 300 Hz and voltage amplitude of 30 V. The temporal mass flows were determined using the weighing cell and the results are shown in Fig. 5.

For nearly all temperatures quite constant mass flows in time and reliable start-stop functions were achieved. The highest mass flows between 2.5 and 3.0 mg/s were realized for temperatures between 58 and 122 °C while for 30 °C a lower value of about 1.7 mg/s was measured. At 130 °C the mass flow stopped abruptly after 130 s. This and the lowest mass flow of about 1.0 mg/s detected indicate a reduced flowability of the powder.

The most constant mass flow was achieved using an oil temperature of 92 °C. The corresponding mean value with standard deviation extracted from the measurement is 2.92 ± 0.07 mg/s.

3.4 Mass flow of TPE powder

Analogous to section 3.3 the powder temperature was varied from 30 °C to 130 °C during the mass flow measurements using TPE powder. The results are shown in Fig. 6. In contrary to PP, no powder mass flow could be detected for all temperatures in the range of 58 °C to 130 °C. At 30 °C a quite constant mass flow of 1.10 ± 0.05 mg/s was achieved.

The observed change in mass flow with increasing temperature is most likely caused by changing humidity [14]. When getting drier, the particles’ adherent water vanishes leading to reduced liquid bridges and thus to weaker capillary forces, better flowability and to the higher mass flows observed. However, at even higher temperatures, softening of the powder particles occurs which alleviates viscoelastic and viscoplastic deformations of the particles during vibration leading to larger contact areas and thus to enhanced cohesive forces (van der Waals, interlocking, etc.). This leads to ceasing of the mass flow at 130 °C.
In order to understand the failed powder delivery at higher temperatures, a detailed knowledge of the TPE formulation is necessary. The TPE is a multi-block copolymer which consists of stiff polyamide 12 segments and polytetrahydrofuran soft blocks. Both components exist alternately in the macro molecules leading to a characteristic (more or less high or low) elastic modulus depending on the amount of each component. Since it is known that the soft component polytetrahydrofuran exhibits a very low melting temperature at about 46 °C [15], an increase of the temperature of the TPE above this temperature leads to additional softening of the TPE macromolecules. As discussed in the previous section, softening alleviates viscoelastic and viscoplastic deformations of the particles and thus can lead to higher cohesive forces and to reduced flowability.

3.5 Line thickness at different nozzle travel speeds

During dispensing, the powder falls on a substrate and spreads due to the collision. In order to minimize the dispersion and achieve a highly selective deposition, it is reasonable to assemble the nozzle close to the substrate. Hence, the distance between the narrow nozzle end and the substrate was set to 1 mm. This ensures a minimum dispersion as well as contact-free deposition.

For the experiments, a frequency of 300 Hz and an amplitude voltage value of 50 V were chosen. Lines were produced at different nozzle travel speeds using PP powder at a nozzle temperature of 30 °C and investigated by optical microscopy. The results are shown in Fig. 7.

The microscopic images from Fig. 7 show that the smallest lines widths are achieved with the highest nozzle travel speeds. Thus, for a speed of 45 mm/s line width of around 0.7 mm are reached, which is the size of the orifice diameter of the nozzle. However, at such high speeds the powder line morphology is quite inhomogeneous including unsteady lines and frazzled line edges which hinder the precise preparation of homogenous powder pattern. Instead, lines with a quite homogeneous appearance are produced with speeds between 15 and 25 mm/s, which feature also still quite small line widths between 0.9 and 1.1 mm. This shows that the best result is a tradeoff between resolution and geometric homogeneity. Lines deposited with a speed of 5 or 10 mm/s are much larger with line widths between 1.5 and 2.0 mm due to powder accumulation.

3.6 Influence of powder material on line deposition

A frequency of 150 Hz and 300 Hz respectively and an amplitude voltage value of 50 V were chosen to fabricate powder lines with TPE and PP powder at a temperature of 30 °C at different nozzle travel speeds. The laser stripe sensor was used to evaluate the three-dimensional geometric data of the lines, which are displayed in Fig. 8. The mean values and standard deviations of line thicknesses and line heights extracted from the data are plotted in the graphs of Fig. 9. Therefore, the dimensions were measured at different line sections with n=10.

As discussed in the previous section, the best result is a tradeoff of smallest dimensions and geometric homogeneity of the line whereas the latter corresponds to standard deviations of geometric measurements. Figure 8 and 9 show that line dimension for both materials are different at same nozzle travel speeds which can be related to the different mass flows of PP and TPE (see section 3.3 and 3.4). For PP the best lines were produced with a nozzle travel speed of 20 mm/s resulting in a line width of $1.13 \pm 0.09$ mm and height of $0.24 \pm 0.03$ mm. Similar line characteristics were achieved with a nozzle travel speed of 10 mm/s using TPE powder but with larger standard deviation of 0.16 mm for the width and 0.05 mm for the height. It is remarkable that the standard deviation of lines produced with TPE seems to be generally larger than the
standard deviation of PP lines which is obviously related to a higher tendency of TPE to form agglomerations during nozzle delivery. This can possibly be explained by the low elastic modulus of TPE which alleviates deformations of the particles during vibration and thus interlocking during powder discharge.

3.7 Influence of temperature on powder line deposition

In order to estimate the influence of temperature on powder pattern preparation using vibrating nozzle, a powder temperature of 92 °C was used to fabricate powder lines using PP powder. This temperature was chosen because very constant mass flows could be achieved at that temperature (see section 3.2). For fabrication a frequency of 300 Hz and an amplitude voltage of 50 V were applied. The nozzle travel speeds were varied between 25 and 40 mm/s. The measurements of the laser stripe sensor are shown in Fig. 10. The corresponding line dimensions are plotted and compared to the line dimensions of PP lines produced at 30 °C in Fig. 11.

Since different mass flows are achieved with different temperatures (see section 3.2), line dimensions for both materials differ strongly at same nozzle travel speeds. While 25 mm/s leads to a line width of 0.93 ± 0.13 mm and line height of 0.18 ± 0.04 mm for 30 °C, a far larger width of 1.42 ± 0.12 mm and line height of 0.39 ± 0.03 mm is reached at 100 °C. Hence, 25 mm/s is a much too small travel speed at 92 °C and thus not usable for powder pattern preparation for SLS. Here also an accumulation effect takes place, leading to increased line height at same width when comparing the line produced with 25 and 30 mm/s. At a temperature of 92 °C, the best result is achieved with a speed of 40 mm/s, leading to a width of 1.05 ± 0.15 mm and height of 0.17 ± 0.02 mm which is comparable to the values measured for a speed of 25 mm/s at 30 °C.

The results show that for PP powder a systematic change of powder deposition characteristics with the different temperatures used is just related to the different mass flows. Since the standard deviations are roughly in the same range, temperature does not necessarily impact the accuracy of the powder deposition. However, this should be only valid for temperatures where a stable mass flow is observed. The limit for PP seems to be close to 130 °C which leads to ceasing of the mass flow as detected in section 3.3.

3.8 Multi-material powder layer preparation

Conventional SLS machines follow a quasi-isothermal process route to prevent process errors as curling or incomplete melting of particles. This means that powder is preheated inside the machine before the deposition by blade or roller to a temperature between 100 and 160 °C depending on the powder material used in order to maintain the thermal process stability. Consequently, using vibrating nozzles, powder should also be heated similarly in order to prevent the mentioned process errors. However, the mass flow results show that heated powder cannot necessarily delivered by nozzles at higher temperature. While PP can be deposited till a temperature of around 130 °C which is close to typical preheating temperatures of PP around 150 °C, TPE can be just delivered for temperatures around 30 °C which is far below typical preheating temperatures or the crystallization temperature. Thus, curling may occur during the deposition of TPE powder at a temperature of
30 °C which should be prevented by certain deposition strategies where only small dosages are deposited and additional preheating is applied.

Nevertheless, as simple demonstration of the potential of nozzle for the preparation of multi-material layers, PP at a temperature of 92 °C and TPE at a temperature of 30 °C are used for the preparation of simple single-layered powder patterns. Therefore, the powder is deposited using typical hatching of straight powder lines (snake lines) with different distances. The powders pattern just consists of two rectangles deposited next to each other while all corresponding lines are in parallel order. The prepared powder layers are characterized regarding layer surface roughness depending on the hatch distance between the lines. For PP and TPE a nozzle travel speed of 35 and 10 mm/s respectively was chosen in order to provide matching powder line dimensions with a line width between 1.0 and 1.2 mm and a line height of between 200 and 250 µm. The measurement results of layer height and surface roughness (RMS: root-mean-square) depending on the hatch distance are shown in Fig. 12.

The results show that hatch distance clearly affects the layer thickness as well as the surface roughness. The hatch distance of 1.0 mm is obviously too large for both materials, resulting in a high RMS value of over 60 µm due to clear valleys between deposited lines. Logically, the corresponding layer thickness values are the lowest values and similar to the line heights measurement from the previous sections.

A hatch distance of 0.6 mm results in the lowest RMS values but also in high layer thicknesses of nearly 300 µm exceeding the single line heights due to overlap of the parallel deposited lines. Instead, the hatch distance of 0.8 mm seem to be an attractive compromise which leads to clearly lower layer thicknesses around 200 µm and also to relatively low RMS values between 50 and 60 µm. This configuration was also used to prepare a multi-material pattern shown in Fig. 13.

In comparison to the layers prepared by the nozzle setup, the layer specifications achieved with a typical SLS using a blade for layer preparation are advantageous. On the one hand thinner layers with heights from 100 to 150 µm can be prepared, which is nearly half as low as the layer thickness achieved with the nozzle setup. And on the other hand the powders’ surface topography is more homogeneous with a low RMS value of 27 µm [5].

3.9 SI-SLS of multi-material powder layer

The multi-material-powder layer of Fig. 13 was molten using SI-SLS. The process was recorded using a thermal camera in order to clarify the single process steps. The measurements are shown in Fig. 14.

The infrared preheaters are used to provide the preheating temperature of 115 °C in the whole building chamber, which is also the typical preheating temperature of the TPE material used. The building platform temperature was set to 100 °C. By using the CO₂ Gaussian laser beam with a diameter of 2.4 mm, a laser power of 10 W with a scan speed of 6 m/s and a hatch distance of 1.2 mm, the rectangular shaped area of the PP powder is heated to a temperature of about 150 °C. Finally the complete powder pattern is fused by the simultaneous illumination for 80 s using the thulium laser. The intensity was adjusted to be 8 mJ/mm²s for the TPE material and 2.5 mJ/mm²s for the PP material, considering the different properties of both powder materials. The lower intensity for PP material was chosen in order to prevent possible oxidation of the surface of the material since the heat conductivity and the melting enthalpy of PP is significantly lower than the corresponding values of the TPE material [16]. The process results in a simple multi-material component made from TPE and PP material which is shown in Fig. 15.

The multi-material component features a relatively stable joint (not quantified) between the different material zones. Since it is known that PP and TPE are not soluble and therefore cannot form a common boundary zone by diffusion processes of the macromolecules, it seems that the forming of a common boundary zone bases on microscopic undercuts (mechanical adhesion). This was also ob-

![Fig. 12 Height and RMS value of layers prepared with different hatch distances: PP was deposited with a temperature of 100 °C and a nozzle travel speed of 35 mm/s; TPE was used with a temperature of 30 °C and a nozzle travel speed of 10 mm/s.](image)

**Fig. 12** Height and RMS value of layers prepared with different hatch distances: PP was deposited with a temperature of 100 °C and a nozzle travel speed of 35 mm/s; TPE was used with a temperature of 30 °C and a nozzle travel speed of 10 mm/s.

**Fig. 13** Photographic image of a multi-material powder layer consisting of PP and TPE powder material.

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served by Laumer et al. when using a two-chamber recoater design in combination with the SI-SLS process [16].

However, more experiments are needed in order to quantify the influence of the border zone characteristics (width, degree of mixture, shape of undercuts) on the mechanical properties of the joint.

4. Conclusion

In this report, the selective deposition of polypropylene and thermoplastic elastomer powder for new approach of laser sintering application was investigated by means of an experimental setup which consists of a heatable (up to 150 °C) steel nozzle assembled on a piezoelectric actuator. By applying sinusoidal voltage amplitude to the actuator, the nozzle was set into vibration, which enables the control of the powder mass flow involving a valve-like start-stop function.

Mass flow measurement at different nozzle temperatures revealed that temperature has a strong effect on the resulting mass flow of polypropylene powder. This is caused by moisture variations in the powder which affect cohesive forces as liquid bridges and van der Waals forces. In contrast to polypropylene, thermoplastic elastomer powder could not be delivered by nozzle with temperatures from 58 °C. This is caused obviously by softening of the TPE macromolecules which alleviates viscoelastic and viscoplastic deformations of the particles, leading to higher cohesive forces and thus to reduced flowability.

Both materials were used for the deposition of powder lines and layers. Experiments show that line widths of down to 700 µm, layers with a thickness of about 200 to 300 µm, and a surface roughness (RMS) of down to 50 µm can be achieved using both powders and a nozzle with an orifice diameter of about 700 µm. It was shown that the hatch distance has to be adapted to the line width in order to achieve homogeneous layer surfaces, which is important for the application in laser sintering technology. Nevertheless, the layers achieved with blades used with typical SLS devices are still advantageous compared to the layers prepared by the nozzle setup.

The multi-material layer was fused using a new process called Simultaneous Intensity-Selective Laser Sintering. This technology bases on the application of a Thulium laser with a wavelength of 1.94 µm with a controllable micro mirror array in addition to a typical CO₂ laser. It allows the application of the quasi-isothermal laser sintering process model for the use of multiple powder materials with different melting temperature next to each other. By this process a stable interconnection between the different materials zone was established, basing on mechanical adhesion.

Acknowledgments and Appendixes
The authors want to thank the German Research Foundation (DFG) for funding the Collaborative Research Center 814 (CRC 814) – Additive Manufacturing, sub-project B6.

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(Received: October 12, 2017, Accepted: May 13, 2018)