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Selective Laser Sintering of PPS

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Zusammenfassung

With specific additives, the high-performance engineering plastic PPS can be used in the form of powder material for laser sintering. In addition to modification of the material, it is necessary to adapt the laser sintering equipment.

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1. Introduction

With a market share of about 90%, polyamide 12 (PA12) is the predominant material used for laser sintering. Because of difficulties in processing, niche products such as PA6, PA11, PP or PEK/PEEK, for instance, are seldom used. This shortage of suitable materials restricts application of the process significantly. For this reason, investigations have been conducted at the IKD with the objective of optimizing a high-performance plastic to make it suitable for the laser sintering process. Polyphenylene sulfide (PPS) was selected because of its high thermal stability and chemical resistance. The PPS powder was characterized and modified with appropriate additives. In addition, a commercially available laser sintering system had to be optimized and adapted to meet the requirements of this material.

2. Use of flow modifiers

The function of flow modifiers is to reduce the adhesive forces between particles and in this way improve flow characteristics. They minimize clumping effects and also frequently serve as release agents by preventing the particle surfaces from coming into direct contact with one another. Course-grained, dry products with particle sizes greater than about 100 µm usually flow well, since the adhesion forces are relatively insignificant compared to gravitational forces. During laser sintering with a layer thickness of the 100 µm, however, the particles are considerably smaller. With these fine-grained products, the van der Waals forces are the primary reason for poor flow characteristics. Pyrogenic solicit acid is a frequently used flow-regulating agent (trade name: Aerosil, manufacturer: Evonik Industries AG). In addition, magnesium carbonate, magnesium stearate, lactose as well as titanium dioxide or aluminum oxide can be used to reduce the adhesion forces.

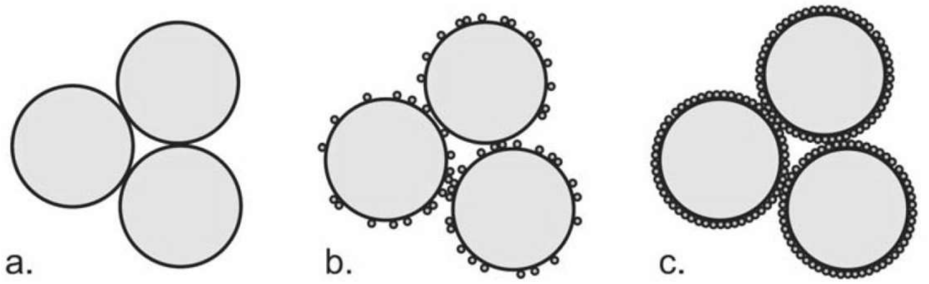

Fig. 1: Flow modifiers reduce the adhesion forces between particles and in this way improve flow characteristics. a: Particles without flow modifier. b: Particles with highly dispersed powder as flow modifier, c: Densely covered surface [1]

The flow modifier is added to the product in small quantities (typically: up to 1 wt. %). The particles of the flow modifier are then supposed to adhere to the outside of the particles and reduce the adhesion forces through their action as spacers (Fig. 1).

Homogeneous dispersion of all particles is important with all additives. This prevents agglomeration or nucleation effects during processing. For this reason, the proportion of additives should be kept as low as possible.

3. Material characterization

With laser sintering, the properties of the powdered starting material and the processing itself determine the quality of the finished parts. Whether the PPS powder investigated was suitable for laser sintering, was determined through analysis of the particle size and particle geometry, among other characteristics.

From the machinery standpoint, the particle size must not exceed the size of the gap between the fabrication platform and spreader, since it is otherwise not possible to apply the powder evenly. Furthermore, the particle size distribution affects the surface quality and resolution of the parts. An excessive proportion of coarse particles have an adverse effect on both criteria. If, on the other hand, the fines proportion is too high, the reduced flow and spreading characteristics cause problems during application.

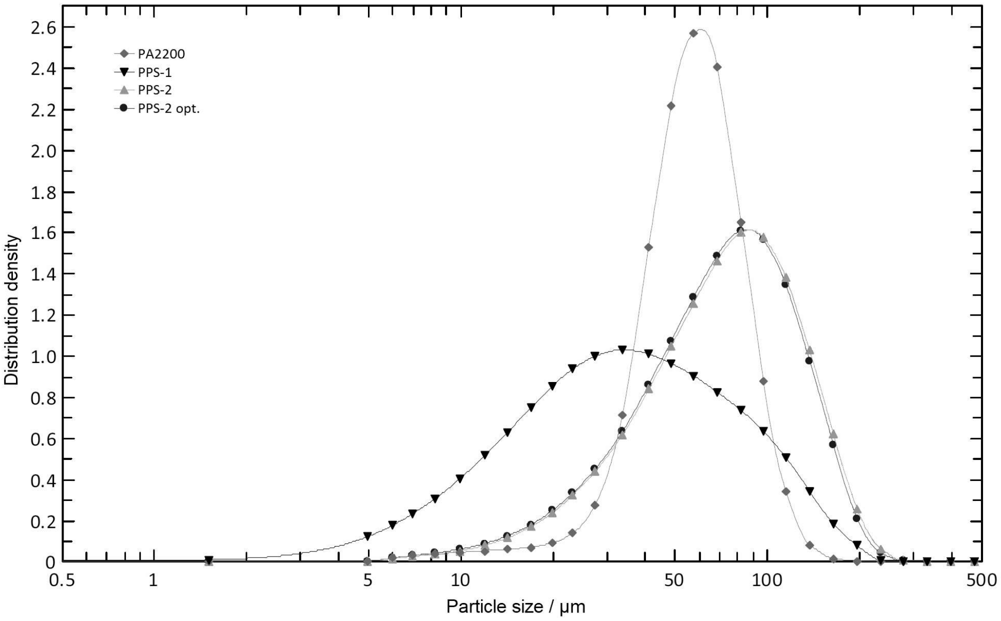

Fig. 2: The distribution density curves show that the particle size distributions of the PPS-2 and PPS-2 opt. samples are very similar. The PA2200 sample has a narrower, the PPS-1 sample a broader distribution

Both a cross-linked material (PPS-1) and a non-cross-linked material (PPS-2) were investigated. Additional modified versions were prepared using the non-cross-linked material (Table 1). The results of the tests performed show that the particle size distributions of the PPS-2 and PPS-2 opt. samples are very similar (Fig. 2). Sample PA2200 has a narrower distribution, that of sample PPS-1 is broader. For PA2200 a unimodal distribution is indicated, the same holds for PPS-1. The mean particle size is somewhat greater in PPS-2 that in PA2200, but both powders are in the range of 55 to 70

µm.

Material	Description
PA2200	Standard laser sintering material
PPS-1	Cross-linked PPS
PPS-2	Non-cross-linked PPS
PPS-2 opt.	Non-cross-linked PPS with additives

Table 1: Description of the materials investigated

Evaluation of the form factors in terms of sphericity as well as aspect ratio shows quite high agreement between the PPS-2 and PPS-2 opt. samples. PA2200 exhibits slightly lower values regarding aspect ratio. This indicates that the particles are, on average, somewhat more elongated than in the other samples. In both cumulative distributions, the PPS-1 sample shows slightly higher values in the lower range, which means it consists of a small fraction of more highly structured particles. At the same time, higher proportions of compact particles are indicated with regard to aspect ratio. This broad distribution indicates that the sample consists of both compact and structured particles (Fig. 3).

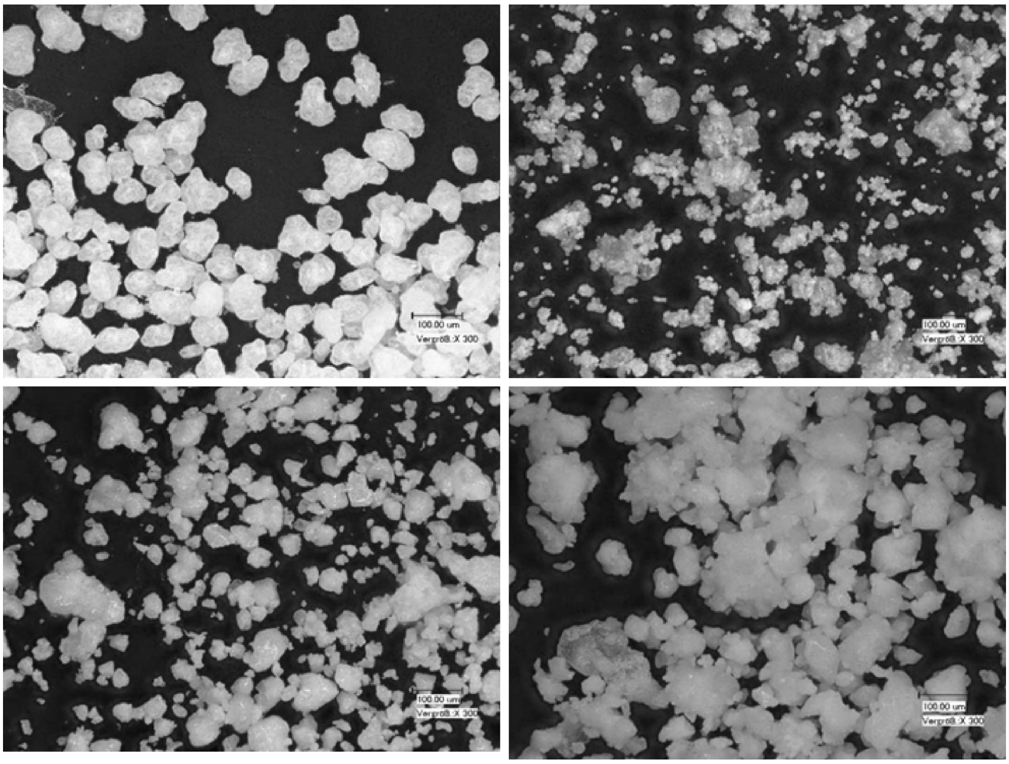

Fig. 3: Grain geometries of the materials investigated. Top left: PA2200, top right PPS 1, bottom left: PPS, bottom right: PPS opt. A digital microscope (manufacturer: Keyence, model: VHX-500F with VH-Z100R and VH-S30B) was used for the images

The decisive factor for laser sintering is primarily a homogeneous application of powder layers. By adding flow modifiers, the flow and spreading characteristics of the PPS powder can be enhanced. This was confirmed during the investigations by means of various analytical methods (determination of the avalanche angle, the Hausner factor, cohesion analysis). After successful application tests using a hand-held spreader with a gap width of 100 µm, the equipment was modified in the next step.

4. Modification of the laser sintering system

Modification of the system technology to meet the requirements for PPS was conducted on an already existing laser sintering system. It was modified and expanded with least amount of effort and expense order to meet the expanded requirements of the high-temperature thermoplastic. Ideally, it will be possible in the future to apply and adapt the concept to other systems as well.

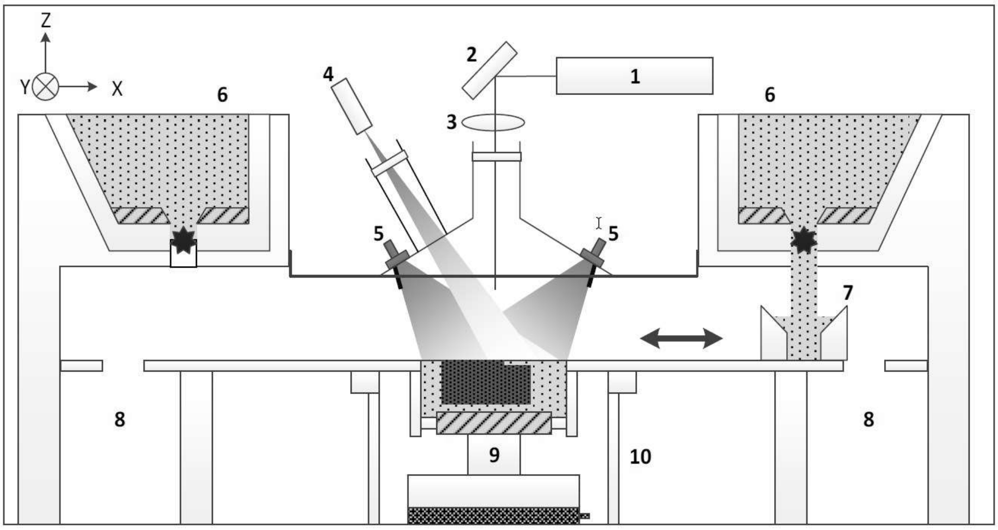

Fig. 4. Schematic layout of the modified laser sintering system with smaller chamber (1 Laser, 2 Scanner, 3 Focusing, 4 Infrared camera, 5 Infrared radiation source and thermal shielding, 6 Powder reservoir with dispensing device, 7 Layer former, 8 Residual powder shaft, 9 Smaller chamber, 10 Removal container)

Because of their material characteristics, high-temperature thermoplastics present considerably more demanding requirements in terms of the system technology for laser sintering then does the standard material PA 2200. According to the theory of quasi-isothermal laser sintering, the melting and crystallization behavior in particular must be taken into account. For PPS the melting point lies in the range of 280°C, for PA 2200, on the other hand, at 188°C. Accordingly, the system must be designed for an approximately 100°C higher temperature. For processing of PPS, it is necessary to control the preheat temperature up to 300°C. This makes it necessary to adapt the preheating system. Dependent variables for sizing a suitable preheating system include the power density of the thermal radiation and the time needed for through-heating of a newly applied powder layer. To prevent cooling of the already sintered regions of the part in the powder bed by the new, colder powder and thus formation of curl, the time for through-heating of the newly applied power layers must be as short as possible.

To achieve the required temperature, the temperature control of the system was modified to allow operation beyond the usual shutdown point. In turn, appropriate additional measures were taken to protect endangered components. This involved primarily the optics via which the laser beam enters the process chamber. The lens was equipped with additional cooling to ensure the usual operating temperatures. The nitrogen flow at the lens was also increased in order to achieve greater cooling.

Furthermore, other components such as solenoid valves and critical actuators close to the process chamber were equipped with additional cooling to prevent overheating here as well. Installation of shielding for the infrared radiation sources is an additional step. This fulfills two functions: first of all, it protects the layer former and other electronic and mechanical peripherals that are installed in the lower part of the system from direct exposure to the IR radiation sources and the associated increased thermal load. Secondly, it can be assumed that heat is introduced into and distributed within the smaller compartment uniformly in the smaller compartment, since the best overlap of the infrared radiation from the individual radiation sources occurs here. The schematic layout of the modified laser sintering system is shown in Fig. 4.

An infrared camera that provides data on the powder bed temperature measured was directed at the smaller chamber. The camera is located next to the optics via which the laser beam enters the process chamber. It is housed in a cooled enclosure to combat overheating by the higher process temperatures. During the sintering process, the optics of the infrared camera and the sensor chip must be shielded by a shutter from direct exposure to laser radiation, for instance, due to reflection of the laser beam by the powder bed.

Following calibration and setting of the emission coefficient, the camera provides very exact monitoring and supervision of the process. Thanks to the camera-assisted monitoring and very accurate process control, the temperature can be adapted optimally to the material. The smaller chamber provides a uniform temperature field.

5. Mechanical properties of the test specimens

To test the mechanical properties, tensile test specimens were produced by means of laser sintering from the PA2200 and PPS test materials. In addition, cubes and pyramids with an edge length of 10 mm were fabricated; these correspond to 100 individual layers (Fig. 5). It can be seen from the pyramids that PPS-2 exhibits very good dimensional stability.

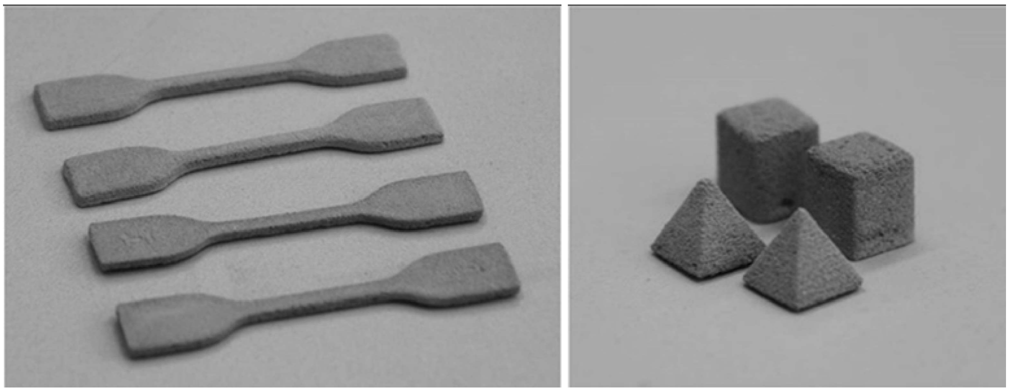

Fig. 5: Test specimens produced by means of laser sintering from the PPS test materials. Left: Tensile test specimen of PPS-2 to DIN EN ISO 527-2:2012-06, right: geometry for evaluating the dimensional stability of PPS-2

The sintered PPS test specimens exhibited the expected behavior (relatively brittle) in tensile testing. Closer examination of the fracture surfaces on the test specimen showed very high porosity. This is probably attributable to the incomplete melting of the powder particles. Future investigations call for determining the potential for further optimization of the process conditions.

6. Conclusion

The knowledge gained in the course of the investigations is useful for developing new materials for the selective laser sintering market. This will open up new opportunities for generative methods that could not be used to date because of the limited selection of materials.

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9. Literature

[1] Schulze, Dietmar: Pulver und Schüttgüter: Fließeigenschaften und Handhabung. 3rd Ed., 2014. Berlin: Springer Vieweg, 2014

Volltext

Lizenz

Empfohlene Zitierweise

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