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LOW TEMPERATURE LASER SINTERING ON A STANDARD SYSTEM: FIRST ATTEMPTS AND RESULTS WITH PA12

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Abstract

The laser sintering process has been a well-established AM process for many years. Disadvantages of LS are the low material variety and the thermal damage of the unprocessed material. The low temperature laser sintering attacks at this point and processes powder material at a build chamber temperature lower than the recrystallization temperature. This drastic reduction in temperature results in significantly less thermal damage to the material. This work deals with the low temperature laser sintering of Polyamide 12 (PA12) on a commercial, unmodified laser sintering system to compare it to standard laser sintered PA12 and to create the basis for low temperature laser sintering of high temperature materials on such a system. First results by changing the exposure parameters and by fixing parts on a building platform show a processing of PA12 on an EOS P396 at a build chamber temperature less than 100 °C instead of standard approx. 175 °C.

Introduction

Laser sintering is one of the most used AM processes for polymers. It has a high productivity and a great design freedom but a significant disadvantage concerning the low material variety. The majority of all laser sintering components are still made of PA12 and PA11. For this reason, standard machines such as EOS P3 systems are mostly available on the market. High performance materials e.g. PA6, PPS, PEKK, etc. are appearing increasingly on the market, but they cannot be processed on standard systems due to the higher processing temperature. Standard systems are only designed and equipped for temperatures of up to around 200°C and high temperature systems, on the other hand, are very cost-intensive. A further issue and drawback of the laser sintering process is material aging of the surrounding and unused powder due to high process temperatures close to the melting point with long dwell times. As a result, the aged material cannot be completely reused and has to be refreshed with virgin powder which is not very sustainable and leads to increased costs.

Low temperature laser sintering addresses this problem by changing the process conditions, in particular by drastically reducing the process temperature, so that the material undergoes significantly less aging and damage. Thus, almost the complete unsintered material can be reused for the process and material costs can be reduced. In addition, low temperature laser sintering can create the possibility of processing high-performance polymers on standard laser sintering systems, which can have a high practical and cost significance for laser sintering system owners. However, the process-related changes in parameters, which include adjusted exposure parameters and strategies in addition to the reduced build chamber temperature, also create new challenges. The high temperature gradients between the melt and powder bed can lead to early recrystallization of the melt, which can result in the so-called "curling" of the component and have a significant negative impact on process stability. In addition, the components cool down more quickly after the process, which can lead to warpage of the component. “Curling” and warpage should be avoided by connecting the components to a building platform. In addition to the advantages already mentioned, low temperature laser sintering can bring further benefits such as new design possibilities or the reusability of previously non-reusable materials.

Within the scope of this work, feasible process parameters are to be developed for low temperature laser sintering on a system from the manufacturer EOS (type P396) with the material PA12. The process parameters are developed by empirically investigating the limits of process-relevant parameters. The main focus is on the connection of the components to the building platform, the build temperature as well as the exposure parameters (laser power, scan speed and hatch distance).

State of the Art

Low temperature laser sintering is not a completely unknown method. The companies Airbus, LSS, Lehmann&Voss and Rauch CNC developed the so-called ThermoMelt process [1], which is equivalent to the low temperature laser sintering considered in this work. The basic process flow is shown in Figure 1. In order to fix the components in the build job, a building platform inserted into the system is used, which in the case of ThermoMelt is made of plastic. Similar to other additive manufacturing technologies, components are built on this platform using support structures. The support structures serve to fix the components in place to counteract the warpage caused by the low temperatures. The laser locally introduces the required residual energy into the powder bed to melt the material at the desired area to create the component or support structures. Since the temperature in the powder bed is significantly lower than in the standard laser sintering process, the laser needs to apply comparatively more energy to the powder bed through adapted exposure parameters or strategies.

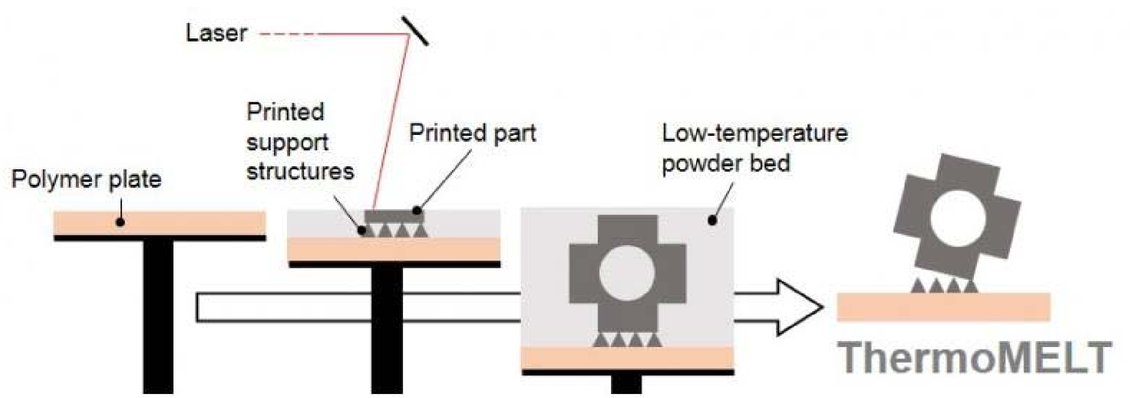


Figure 1: Process flow of ThermoMelt [1]

The company LSS offers in its portfolio a special ThermoMelt system (Raptor 84X-Q ThermoMelt^{TM}), which works among other things with four lasers and a multi-zone heater. The machine data sheet promises that components produced with ThermoMelt come with improved isotropy as well as improved elongation at break in z-direction. In addition, the reusability of the material and the surface finish of the components should improve and the cost per component should decrease, as shown in Figure 2. [2]

Low temperature laser sintering has also been considered in the research community. Publications on the subject can be found at the University of Tokyo by Niino et al [3] and at Virginia Tech by Chatman [4]. In Tokyo, PA12 was considered without preheating the powder bed and PEEK was studied in low temperature laser sintering. Both were performed on experimental laser sintering systems. Chatman examined PPS on a DTM Sinterstation 2500+. In the case of PA12, Niino et al. was able to show that 50% of the tensile strength and 67% of the impact strength could be achieved without preheating the powder bed compared to the standard laser sintering process. [3]

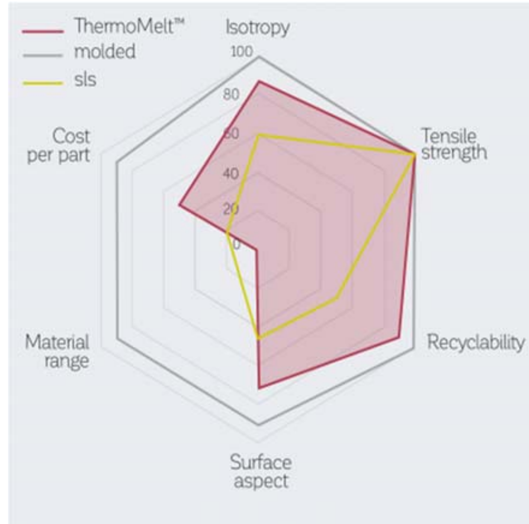


Figure 2: Comparison of ThermoMelt, Casting and Laser Sintering [2]

Test Set Up and Preliminary Investigations

Within the scope of this work, a standard laser sintering system of the type P396 from the company EOS is used. The system is not modified or optimized for the investigations. Only the building platform for fixing the components is inserted into the system. This means that the standard heating system and the standard blade recoater are used, even though an optimized multi-zone heating system for better temperature control as well as a roller recoater against possible “curling” would bring advantages in low-temperature laser sintering. Further, EOS PA12 (PA2200) is used in a mixing ratio of 50% virgin powder and 50% recycled powder. For the preliminary investigations, which were not aimed at optimizing the exposure parameters, initial exposure parameters that fundamentally create a connection to the platform were determined by simple experiments and used here as the starting value for the investigations. The scan speed is set to 5000 mm/s, the hatch distance to 0.14 mm and the laser power to 50 W. The build chamber temperature was set to 100 °C.

The preliminary investigations first dealt with the connection of the components or possible support structures to the building platform so that warpage and “curling” can be counteracted and prevented. For this purpose, an adjustment platform (figure 3) is used so that the first powder layer can be applied as homogeneously as possible, since the standard platform is always tilted minimally in the machine, which would result in an uneven thickness of the first layer. For the component-platform-connection two different principles – mechanical adhesion and welding of plastics – are considered. For the principle of mechanical adhesion an aluminum platform is used as building platform. Test specimens should be built directly on this platform. A connection of the test specimen to the building platform was partially possible, but not reproducible (Table 1, left). For the principle of welding, which is in this case comparable to laser transmission welding of plastics, different settings were investigated. When welding plastics, only certain combinations of plastics can be welded together. PA12 is weldable with other polyamides such as PA6, PA66 and PA12 itself [5]. First, pure PA6 and PA12 platforms were used which were in case 1 non-bonded and in case 2 bonded to the adjustment platform. The investigations show that the platforms itself warped due to temperature impact which leads to a collision with the recoater (Table 1, left middle). Organo-Sheets with PA matrix and glass or carbon fibers should provide a remedy. In case of non-bonding the organo-sheet to the adjustment platform a low temperature process was possible but during the end of the build job and during the cooling phase the residual stresses led to distortion of the building platform and the test specimen itself – in some cases so strongly that during the last layers of the build job, test specimens warped so strongly that, similar to "curling", they stick out of the powder bed and were hit by the recoater (Table 1, right middle). Finally, organo-sheets bonded to the adjustment platform worked very well (Table 1, right) and test specimens could be built on the platform in a stable process. In the following, this option will be used.

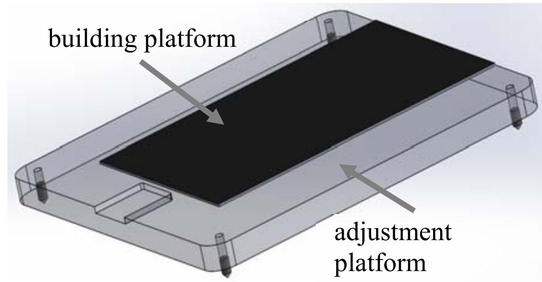
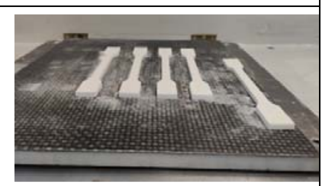


Figure 3: Adjustment platform

Table 1: Summary of build platform investigations

Mechanical Adhesion	**Welding of plastics**		
Aluminum platform	Pure PA6 / PA12 platform – bonded and non-bonded	Organo Sheets – non-bonded	Organo Sheets – bonded
			

In the second step the first layer bonding and exposure quantity and order was considered. The first layer needs to be applied as thin and homogeneously as possible (see Figure 4, left). These investigations showed that good adhesion or bonding of the first layers to the building platform, which is reproducible over several build jobs, results when the first layers are exposed several times. The lowest six layers are exposed five times and the following six layers three times before single exposure of the other layers is applied (see Figure 4, middle). Multiple exposures can result in long layer times, which is why the exposure sequence must also be considered for multiple components or test specimens. The melted layers should not be left open for too long to prevent early recrystallization. Therefore, the components in each layer are exposed one after the other in one exposure cycle before the following exposure cycle starts in the same layer. This means that a component is not directly exposed several times before the next component is exposed several times, but instead this is done one after the other. The first exposed layers of test specimens can be seen in Figure 4 on the right.

In addition, the limits of the build chamber temperature were determined on the basis of the exposure parameters which were used before. Starting at 100 °C, the build chamber temperature

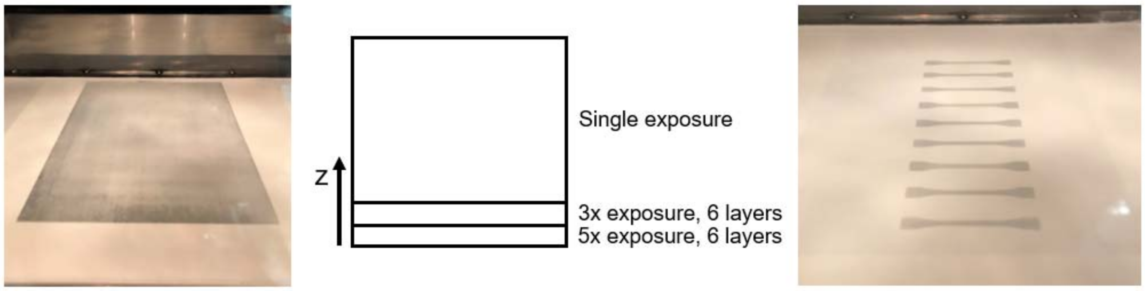


Figure 5: First layer application, exposure strategy and exposure areas of test specimens (from left to right)

was successively reduced for each build job. Reproducible results and a stable process are obtained down to a build chamber temperature of 80 °C, which is about 90 °C below the build chamber temperature when processing PA12 in standard laser sintering. Build chamber temperatures up to 60 °C were also possible, but the process was not well reproducible. Figure 5 shows the component densities for different temperatures. It can be seen that the density decreases with decreasing temperature, which was expected due to the constant exposure parameters. For 80 °C, the density is 7% below the density of the reference from the standard laser sintering process. For 80°C, it can be shown by the Melt Flow Rate (MFR) that the powder shows no significant aging and, in theory, can be completely reused since MFR value for powder “aged” at 80°C and powder, which was not exposed to temperature, are equal.

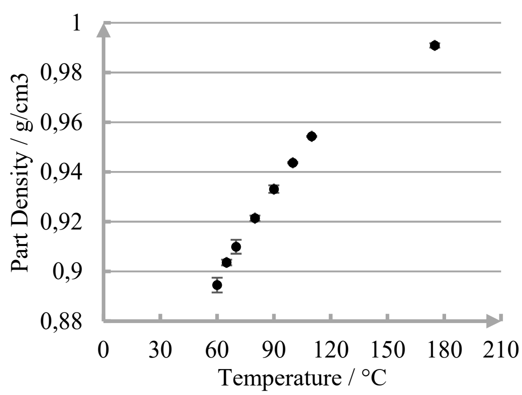


Figure 4: Part density for different build chamber temperatures

Development of Exposure Parameters

A full factorial experimental design is established to investigate the exposure parameters. The laser power (P), the hatch distance (d) and the scan speed (v) are varied based on the parameters used in the preliminary investigations. However, only the exposure parameters of the hatch lines are varied. The parameters of the contour lines (P = 30 W; d = 0.3 mm; v = 3000 mm/s), the layer thickness (120 µm) and the build chamber temperature (80 °C) are kept constant. The parameters of the hatch lines, which can be taken from table 2, are considered in each possible combination, resulting in 64 parameter sets. A typical parameter in laser sintering, which combines these values, is the energy density A_z. The energy density is given by following formula [6]:

$$A_z = \frac{P}{v * d} \qquad [\frac{J}{mm^2}]$$

Table 2: Exposure parameters for full factorial experimental design

Exposure Parameters	**Values**
Laser Power [W]	40 / 45 / 50 / 60
Hatch Distance [mm]	0,1 / 0,12 / 0,14 / 0,16
Scan Speed [mm/s]	3000 / 3500 / 4000 / 5000

The resulting energy densities of the experimental plan are between 0.05 J/mm^2 and 0.2 J/mm^2. At higher energy densities of the experimental plan, strong smoke emission occurs during the exposure process as can be seen in figure 6. Based on initial investigations, a maximum energy density of 0.12 J/mm^2 is determined as the threshold for acceptable smoke emission, which reduces the experimental design to 46 parameter sets. To evaluate the exposure parameters, the mechanical properties tensile strength, elongation at break and Young's modulus are considered. Tensile bars of type 1BA according to DIN EN ISO 527-1 manufactured in flat orientation with the respective parameters are used for this purpose.

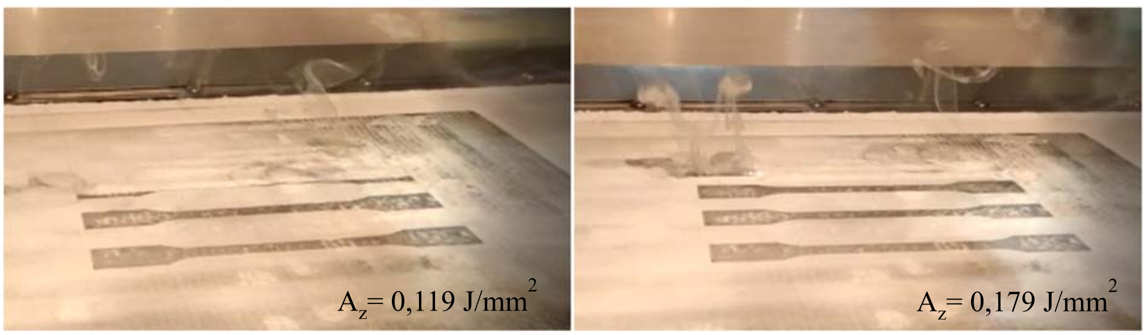


Figure 6: Acceptable (left) and unacceptable (right) smoke emission at different energy densities

All 46 parameter sets could be manufactured without process abort. An exemplary build job is shown in Figure 7. The results of the mechanical properties study are shown in Figures 8-10. In Figure 8, tensile strength is plotted over energy density. It can be seen that the tensile strength increases with increasing energy density. For energy densities above approx. 0.085 J/mm^2, a plateau around approx. 32.5 MPa is reached. The maximum tensile strength is 34.42 MPa at an energy density of 0.104 J/mm^2, which is approx. 25.3 % below the reference sample (46 MPa) produced by standard laser sintering. Furthermore, it can be seen that the tensile strength varies between 24.7 MPa and 32.3 MPa at the same energy density, e.g. at 0.083 J/mm^2.

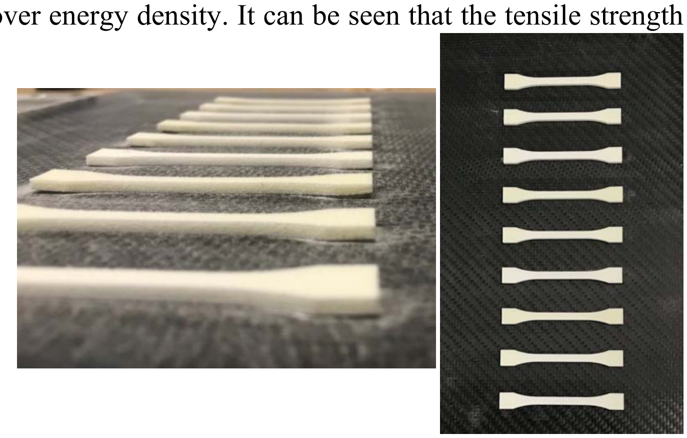


Figure 7: Exemplary build job of tensile bars

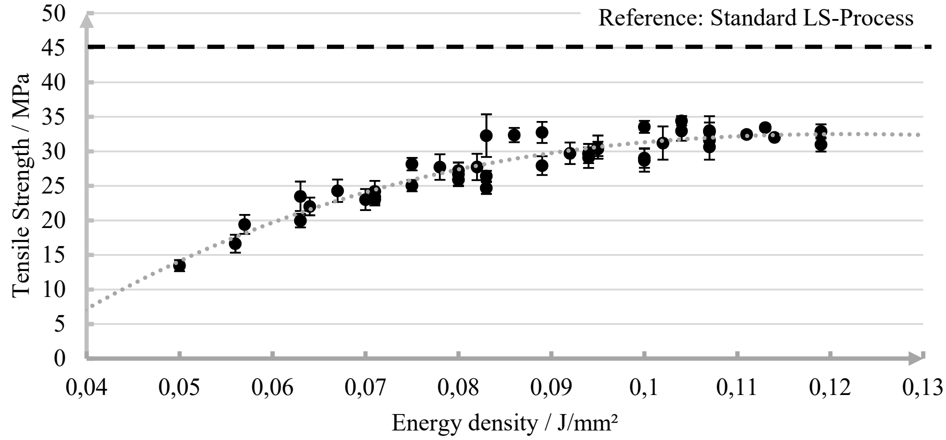


Figure 8: Tensile strength for different energy densities

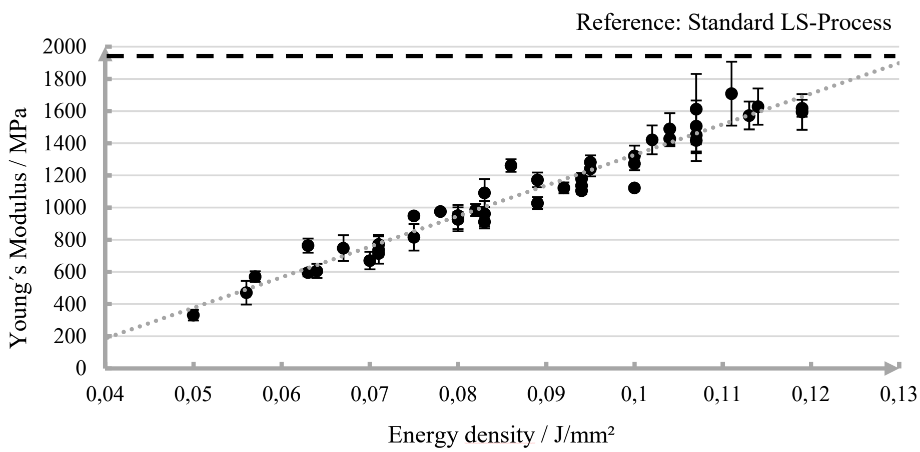


Figure 9: Young´s modulus for different energy densities

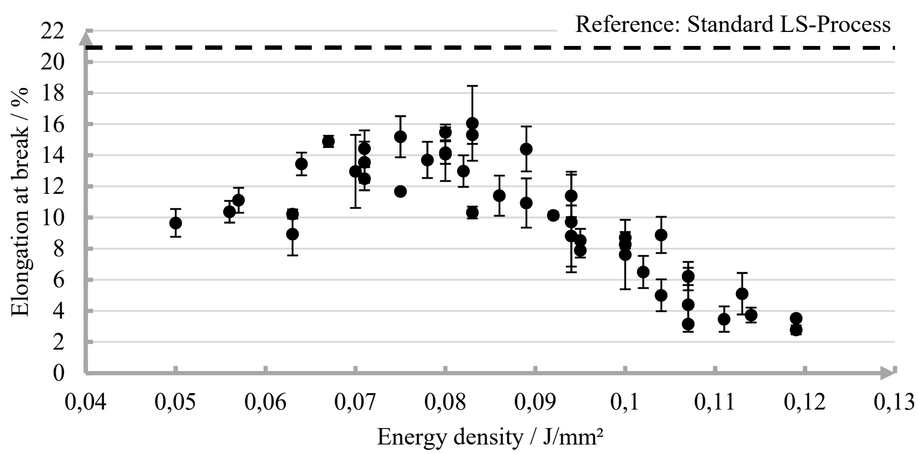


Figure 10: Elongation at break for different energy densities

Thus, it is not only the energy density that is decisive, but also the combination of the underlying exposure parameters. In this case, higher laser power (50 W), higher scan speed (5000 mm/s) and low hatch distance (0.12 mm) lead to higher tensile strength. Figure 9 shows Young's modulus over energy density. Young's modulus increases with increasing energy density and follows an approximately linear trend. At an energy density of 0.111 J/mm^2, the maximum is reached at about 1707 MPa, which is about 9.3 % lower than the reference manufactured by standard laser sintering (1883 MPa). The curve of elongation at break, plotted over energy density (Figure 10), is less straightforward. First, up to an energy density of approx. 0.08 J/mm^2, an increase in the elongation at break of approx. 9 % up to the maximum of 16 % at an energy density of 0.083 J/mm^2 can be observed. After that, the components become brittle and the elongation at break drops to a minimum of approx. 3 % as the energy density increases further. The maximum elongation at break is approx. 22.4 % below the elongation at break of the reference sample produced by standard laser sintering.

It can be seen that the exposure parameters represented by the energy density in this evaluation have a direct, major influence on the mechanical properties. Trends can be seen for tensile strength and Young's modulus. The characteristic values increase with increasing energy density. The course of elongation at break shows a maximum value in the middle of the range of energy densities considered. No clear trend is detectable. In the present state of the investigations, it is therefore not possible to clearly identify an optimal exposure parameter set and an application specific exposure parameter set would have to be selected. Furthermore, the partly large standard deviations have to be critically questioned and the number of test specimens, which was limited to three within the scope of this work for capacity reasons, could be extended in order to verify the results. In addition, a detailed analysis would have to be carried out to evaluate how strong the influence of the respective exposure parameter (laser power, scan speed or hatch distance) is on the mechanical properties at the same energy density.

Conclusion and Outlook

Within the scope of this work, it was shown that PA12 could be processed with significantly reduced build chamber temperature by low temperature laser sintering on a standard system (EOS P396). To avoid "curling" and warpage, in order to make the process stably feasible, the components in low temperature laser sintering are built directly on a building platform and fixed in this way. The investigations showed that organosheets with carbon fibers and polyamide matrix bonded on an adjustment platform are particularly suitable for fixing, in this case welding. The welding of the first layers of the components to the building platform plays a decisive role. The first powder layer must be applied as thin and homogenous as possible. Exposure of the component areas takes place several times in the bottom layers in order to realize a strong connection, and is reduced to a single exposure in the further stages of the layer buildup. Compared to standard laser sintering (~175°C), the build chamber temperatures could be drastically reduced to 80°C. Furthermore, a comprehensive, full-factorial test plan was carried out for the exposure parameters (laser power, scan speed and hatch distance) of the hatch lines and evaluated on the basis of the mechanical properties of the test specimens. It can be seen that no optimal parameter set has been identified for all mechanical properties (tensile strength, Young's modulus and elongation at break). A specific set has to be selected for each application. In general, the mechanical properties show lower values than in standard laser sintering. The maximum tensile strength of the tests is approx.

25%, the maximum Young's modulus approx. 9% and the maximum elongation at break approx. 22% lower than the properties in standard laser sintering.

Following this work, a wide variety of investigations are still on the agenda for this research topic. The exposure parameters or the exposure strategy, for example, should be further investigated in the form of multiple exposures and also contour exposures. The influence of the layer times should also be considered. Furthermore, the crystallinity of the components and the coalescence of the particles can be investigated. In addition, the investigation has so far been limited to the flat orientation of the test specimens, so that other orientations should also be investigated, especially in the buildup direction. In order to implement this, however, a focus must first be placed on the use of support structures. In addition, new design possibilities can be considered and investigated. Finally, the results of the investigations will be transferred to high-temperature materials.

Acknowledgement

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References

[1] Lehmann&Voss&Co., Press release, Hamburg, 2016
[2] www.lss-europe.com: Machine brochure LSS RAPTOR 84X-Q (last call: 27.08.2021)
[3] T. Niino, H. Haraguchi, Y. Itagaki: Feasibility study on plastic laser sintering without powder bed preheating, Proceedings of the Annual International Solid Freeform Fabrication Symposium, Austin, TX, 2011
[4] C. A. Chatman, T. E. Long, C. B. Williams: Powder bed fusion of poly(phenylene sulfide) at bed temperatures significantly below melting, Additive Manufacturing, vol. 28, pp.505-516, 2019
[5] R. Klein: Laser Welding of Plastics, Wiley, 2012
[6] Schmid, M.: Selektives Lasersintern (SLS) mit Kunststoffen – Technologie, Prozesse und Werkstoffe, Carl Hanser Verlag, 2015