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Influence of temperature gradients on the part properties for the simultaneous laser beam melting of polymers

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Abstract

By Laser Beam Melting of polymers (LBM), parts with almost any geometry can be built directly out of CAD files without the need for additional tools. Thus, prototypes or parts in small series production can be generated within short times. Up to now, no multi-material parts have been built by LBM, which is a major limitation of the technology. To realize multi-material parts, new mechanisms for depositing different polymer powders as well as new irradiation strategies are needed, by which polymers with different melting temperatures can be warmed to their specific preheating temperatures and be molten simultaneously. This is achieved by simultaneous laser beam melting (SLBM). In the process, two different materials are deposited next to each other and preheated a few degrees below their melting temperatures by infrared emitters and laser radiation ($\lambda = 10.60$ µm), before in the last step the two preheated powders are molten simultaneously by an additional laser ($\lambda = 1.94$ µm).

So far, multi-material tensile bars have been realized and analyzed regarding their boundary zone between both materials. The experiments showed that the temperature gradients in the boundary zone and along the building direction seem to be of great importance for the process stability and the resulting part properties. Therefore, a detailed analysis of the occurring temperature gradients during the process is needed to identify adequate process adjustments regarding the temperature controlling. To analyze the temperature gradients, thermocouples positioned inside the powder bed are used. By varying the temperature of the building platform, the influence of different temperature gradients on the resulting part properties is shown.

Keywords: Additive Manufacturing, Laser Beam Melting of Polymers, Multi-Material Parts

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1. Introduction

Additive manufacturing (AM) offers many advantages compared to other manufacturing technologies. By AM, incremental volume units are built layerwise to generate components with complex geometries and undercuts. Besides the realizable geometrical freedom of the parts, AM offers a high flexibility because no additional tools like molds are needed. At the moment, different AM technologies exist. One of them is Laser Beam Melting of Polymers (LBM), also called Laser Sintering. In the process, thermoplastic polymer powder is deposited in thin layers, preheated a few degrees below its melting temperature and molten by CO_2 laser radiation. By repeating the different process steps, three-dimensional parts are built. For the processing of polymers, LBM offers very high mechanical strengths of manufactured parts compared to other technologies like Fused Deposition Modelling (FDM), Stereolithography (SLA) or 3D Printing (3DP) and does not need any support structures [1]. Besides the advantages mentioned above, there are limitations which evolve from the increasing industrial need for complex multi-material parts. Currently, only single material parts can be built by LBM. Therefore, a new approach is needed by which different polymer powders can be additively processed to multi-material parts with different part properties like different stiffness or chemical resistances within a single building process.

Such a new AM technology is Simultaneous Laser Beam Melting (SLBM). In former works of the authors, multi-material specimens were generated by SLBM and analyzed regarding the connection stability in the boundary zone between the different polymers [2]. These investigations showed that the temperature gradients occurring in the process are of great importance for both the process controlling and the part properties. Depending on the temperature gradients, an inhomogeneous crystallization after melting the polymer powders can occur. This leads to an inhomogeneous shrinkage and results in the so called curling effect, which means that the solidifying polymer is bending towards the powder surface. Curling can lead to a build stop of the process because if the curling height is higher than the layer height, the part is torn away by the recoater during depositing a new powder layer. Other possible consequences are a decrease in connection stability of the boundary zone and shape and dimension deviations.

In order to prevent curling, a better process understanding regarding the temperature gradients is needed to improve the process adequately. Thus, the influence of different temperature gradients and the impact on the resulting part properties are analyzed in this paper.

2. Laser Beam Melting process

In the SLBM process, three different energy sources are used to preheat and melt the powder materials. Besides infrared emitters, a CO_2 laser ($\lambda = 10.60$ µm) and a thulium laser ($\lambda = 1.94$ µm) are used. As first process step, two different powder materials are deposited next to each other by a two-chamber recoater. Infrared emitters and a heated building platform warm both powders to the preheating temperature of the polymer with the lower melting temperature, in this case polymer A (fig. 1). As next process step, polymer B, which represents the polymer with the higher melting temperature, is locally warmed to its preheating temperature by CO_2 laser radiation. To achieve a homogeneous energy and thus equally homogeneous temperature distribution on the powder surface, a diffractive optical element (DOE) is used. The DOE produces a rectangular beam with a homogeneous intensity profile on the powder bed. In the case of compatible thermoplastic polymers, both materials need to be in molten state at the same time. Only if this is ensured, the micro Brownian motion of the polymer molecules is sufficient to allow diffusion and the forming of a stable connection on a molecular level. To achieve simultaneous melting of complex layer geometries, the selective energy deposition by a scanner system has to be replaced by a beam shaping device enabling simultaneous and geometrical flexible energy deposition. Therefore, a Digital Light

Processing (DLP) chip is homogeneously irradiated by the second laser source and acts as flexible mask for beam shaping. Because CO_2 laser radiation is absorbed by the DLP chip and therefore would damage the device, a thulium laser with a wavelength of 1.94 µm is used. Each of the nearly two million micro mirrors of the chip 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. Up to now, a homogeneous intensity profile has been used to melt both materials. After both materials are in molten state, the building platform is lowered by 200 µm, a new powder layer is deposited and the process steps are repeated. The energy deposition and resulting temperatures in the x-y plane are controlled by a thermal imaging system with a resolution of 1280x1024. The system is calibrated with thermocouples in a temperature range between room temperature and the respective melting point of the materials. The calibration is necessary because temperature-dependent changes of the emissivity of the polymers would otherwise lead to a wrong temperature measurement.

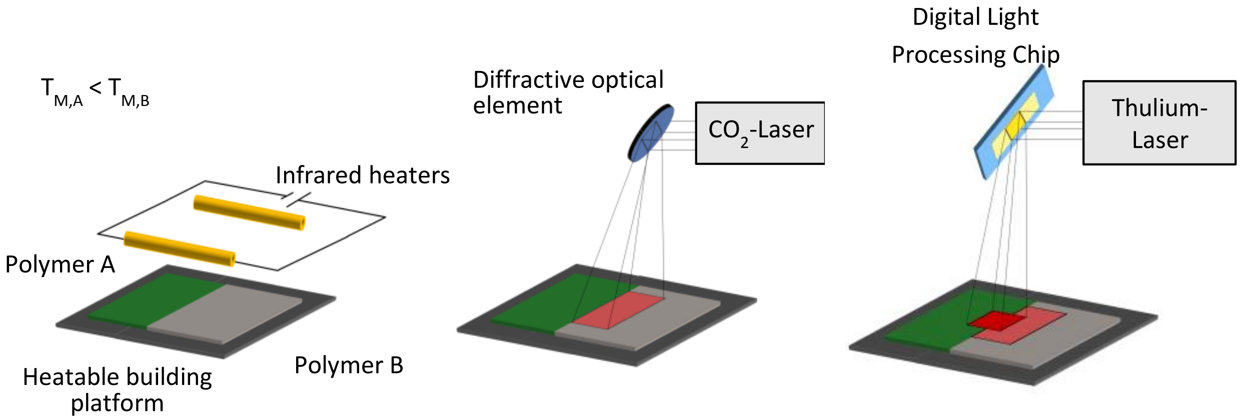

Fig. 1. Process steps of Simultaneous Laser Beam Melting

In the process, two important temperature gradients occur (fig. 2). One temperature gradient results of the different preheating temperatures and occurs in the boundary zone between both materials. With increasing difference in the preheating temperatures of both polymers, the temperature gradient increases. In the worst case, the temperature decrease of the higher melting polymer (polymer B) in the boundary zone is high enough that the crystallization temperature of the polymer is reached locally, leading to an inhomogeneous crystallization and occurring of curling. Due to curling, the solidifying polymer bends towards the powder surface, which can lead to a building stop or shape and dimension deviations of the part. The other gradient lies within the powder bed in building direction. The preheating temperature of both materials is provided by infrared emitters and laser radiation. Because of the limited penetration depth of both radiation sources, the preheating temperature is only reached near the powder surface but decreases towards the building platform. To minimize this decrease, the building platform can be additionally heated. Theoretically, the temperature of the platform should not be above the temperature of the lower melting polymer (polymer A) because otherwise the powder bed would melt completely. In conventional LBM machines, the temperature of the building platform can reach a maximum of 120 °C and thus is significantly below the preheating temperature of the standard polymer polyamide 12, which lies at around 165 °C. Thus, the temperature difference between the powder surface and deeper layers leads to a temperature gradient. In the case of SLBM, there are two different temperature gradients within the powder bed. Because of the different preheating temperatures of the powders provided by radiation and a constant temperature of the building platform, the temperature gradients are higher for the polymer with the higher melting temperature compared to the polymer with the lower melting temperature.

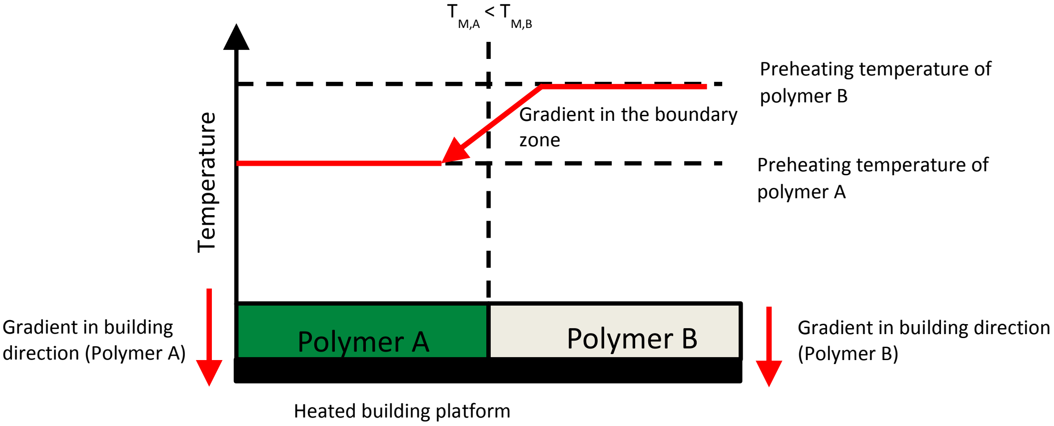

Fig. 2. Temperature gradients in the SLBM process

3. Materials and experimental setup

3.1. Materials

The materials used in this paper are polypropylene (PP, PD0580 Coathylene, DuPont) and polyamide 12 (PA12, PA 2200, EOS). The PA12 powder is a conventional LBM powder material and has a good flowability. To achieve a comparable flowability of the PP powder, 1.0 weight percent aerosil (Aerosil R106, Evonik) is admixed to the raw powder. For the mixing process, a turbula mixer is used with a mixing time of 60 minutes. After admixing aerosil, the deposition of 200 µm dense powder layers is possible by the recoater system. The particle size distributions of the powders are shown in table 1. Additionally, the melting point (T_M) and the crystallization point (T_C) of both materials, which are determined by a Differential Scanning Calorimetry measurement, are included in the table. The optical material properties, which are of great importance regarding the beam-matter-interaction between the particles and the different radiation sources, are determined by an integration sphere measurement setup [3]. By admixing carbon black particles (Orion) the low absorptance of the thulium laser radiation ($\lambda = 1.94$ µm) by the powders is increased. The carbon black particles are smaller than 100 nm and therefore have no measureable influence on the particle size distribution. The optical material properties of the later deposited layer thickness of 200 µm are also shown in table 1.

Table 1. Particle size distribution, thermal and optical material properties of used materials at a layer thickness of 200 µm

Particle size	d10 [µm]	d50 [µm]	d90 [µm]	T_m [°C]	T_c [°C]	Reflectance	Absorptance	Trans-mittance
Polypropylene + 1 wt. % Aerosil + 0.25 wt. % Carbon Black	60	100	150	165	129	0.11	0.76	0.13
Polyamide 12 + 0.25 wt. % Carbon Black	32	55	74	184	150	0.17	0.77	0.09

3.2. Temperature measurements

Following the model of quasi-simultaneous LBM, for an ideal process the crystallization temperature of the polymer should not be reached until all layers of the part are molten, leading to a homogeneous crystallization of the overall part after the melting process without the occurring of curling [4]. To determine if this model is adequate for the SLBM process or if crystallization occurs locally during the process, the development of the temperature gradients during the process needs to be analyzed.

Thus, PP and PA12 powders are deposited next to each other by the two-chamber recoater in a layer height of 16 mm. This height is constant for all experiments and acts as thermal buffer between the building platform and the first layer of the part. In a depth of 1 mm beneath the powder surface two thermocouples (typ K) are positioned with a distance of 3 mm from the middle of the boundary zone. After several hours of preheating to achieve a homogeneous temperature distribution, the SLBM process starts and a rectangular shaped (30 mm times 40 mm) five layer thick specimen is built. The preheating temperature of PP is 150 °C, whereas the preheating temperature of PA12 is 165 °C. The intensity of the CO_2 laser radiation used for providing the temperature difference between both polymers is 3 mJ/mm²s. The irradiation time of the thulium laser for melting is 20 s with an intensity of 6 mJ/mm²s. After irradiation, a new powder layer is deposited with a height of is 200 µm. Between the deposition of a new powder layer and the melting of the next layer, the powders are preheated for 40 s. These parameters are constant for all experiments. The temperature of the building platform is varied in the measurements to achieve different temperature gradients and analyze the influence on the part properties. The SLBM machine is modified to allow higher heating temperatures up to 160°C compared to 120 °C of conventional LBM machines. For the measurements, building platform temperatures of 120 and 140 °C are used. For each temperature the experiments are repeated three times to allow a statistical evaluation. The schematic measurement setup can be seen in figure 3.

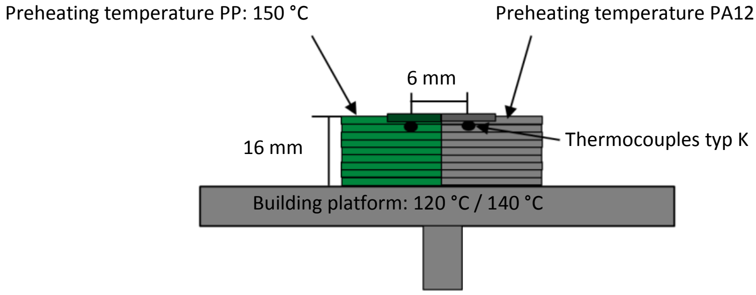

Fig. 3. Measurement setup for determining the temperature development during the SLBM process

To analyze the connection stability between the materials, tensile bars are cut out of the rectangular specimens. As specimen geometry, 1:5 scaled Campus tensile bars are chosen. For statistical interpretation, nine tensile bars are realized for each parameter set. The thickness is defined by the height of five layers. The bars are tested with a tensile testing machine. Their cross-section area is measured at the position with the minimal cross section. By dividing the breaking force by the cross-section area, the tensile strengths of the tensile bars is determined. Additionally, cross sections of the boundary zone are prepared.

4. Results and Discussion

The temperature measurement for a building platform temperature of 140 °C can be seen in figure 4. It shows a typical temperature development for a LBM process [5, 6]. Due to the energy deposition of the thulium laser for melting, the temperature of both polymers is steeply increased during melting until a new powder layer is deposited. Because the powder in the recoater is not directly preheated by the infrared heaters, its temperature is below the preheating temperature of PP. As a consequence, both polymers show a strong decrease of their temperature during powder deposition. Afterwards, both polymers are preheated for 40 seconds and the thulium laser melts the newly deposited powder layers again. This is repeated until all five layers are built and the cooling phase begins.

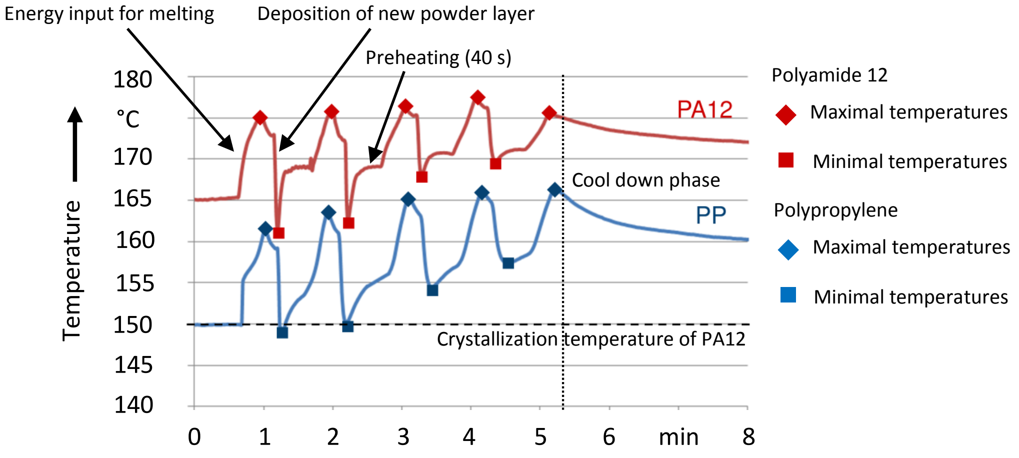

Fig. 4. Temperature development during SLBM process for a building platform temperature of 140 °C

The results of the temperature measurements for the two different building platform temperatures are shown in figure 5. In order to improve comparability, only the minimal and maximal temperatures according to figure 4 are presented and divided into two different polymers.

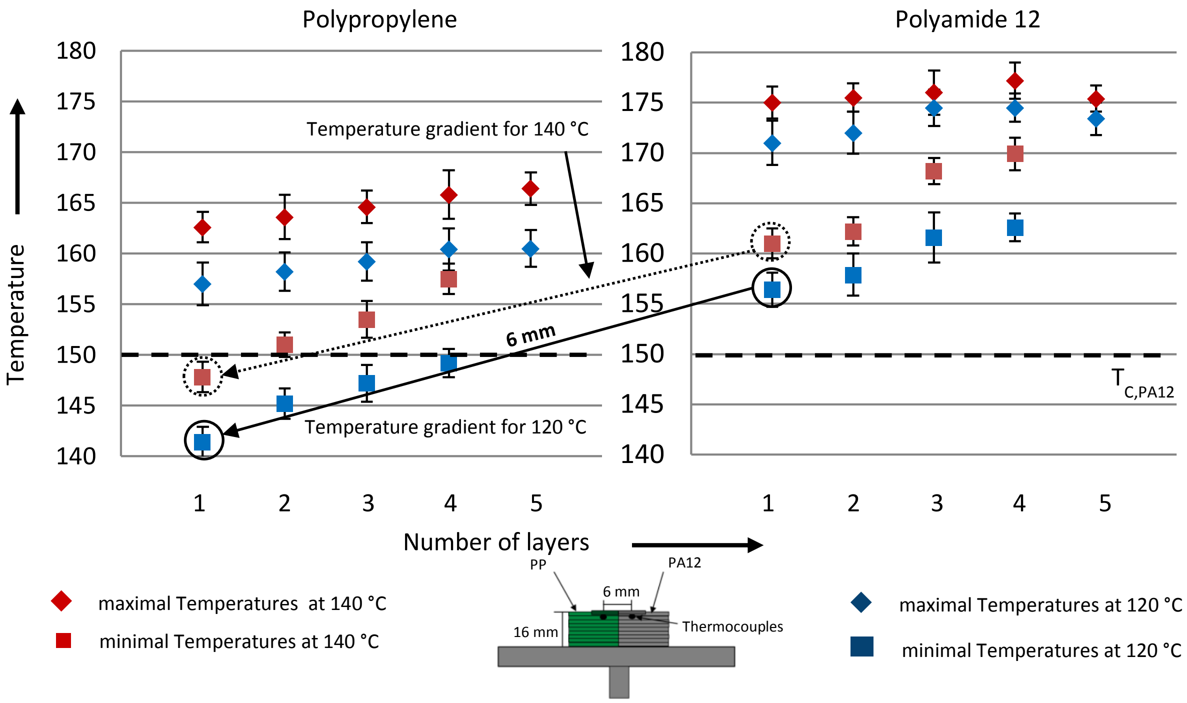

Fig. 5. Temperature development during SLBM process (a) in the polypropylene side; (b) in the polyamide 12 side

The results of the measurements show that the crystallization temperature of PP (129 °C) and PA12 (150 °C) are not reached within the specific side of the powder bed during melting. Therefore, within both parts of the powder bed the molten polymers solidify after the building process, and thus no curling needs to be expected. However, regarding the minimal temperatures after melting the first layer, a temperature gradient exists between both materials in the boundary zone (see fig. 5). This gradient leads to a temperature decrease of the molten PA12 towards the PP side of the powder bed within the boundary zone. If the temperature decrease is high enough, the crystallization temperature of PA12 is reached locally in the PA12 side of the boundary zone during the building process, which leads to curling of the solidifying PA12. In the case of higher building platform temperatures, the temperature gradient can be reduced and thus, the crystallization temperature is not reached in the PA12 side of the boundary zone but in the PP side. Because this temperature is still above the crystallization temperature of PP, no inhomogeneous crystallization in the overall boundary zone occurs and curling is prevented.

To verify this consideration, two cross sections of the boundary zone of the multi-material tensile bars are shown in figure 6. On the top, the specimen built with a building platform temperature of 120 °C and beneath, the specimen built with a building platform temperature of 140 °C. The left side of the bars is PP, the right side PA12.

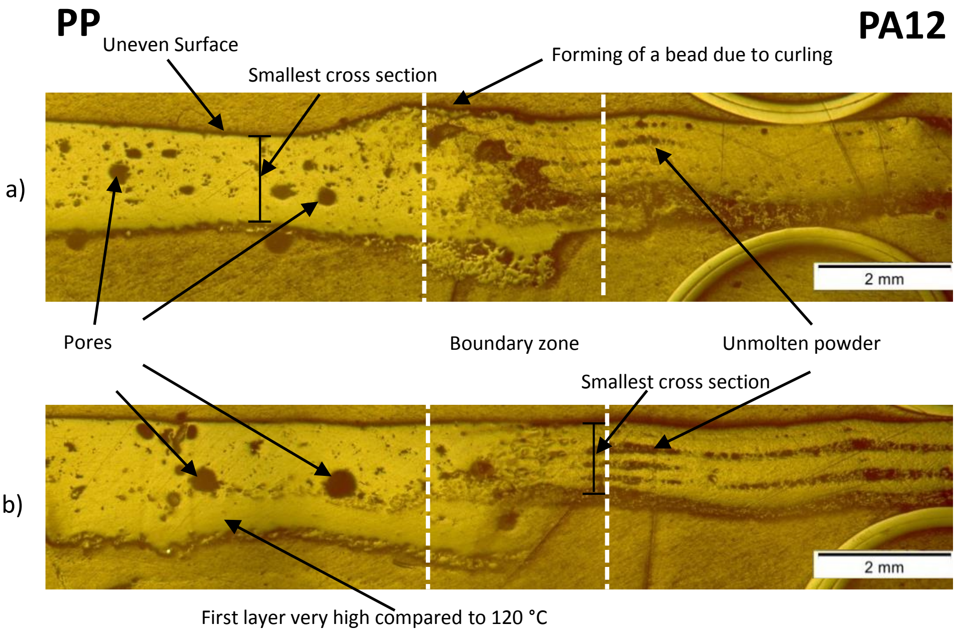

Fig. 6. Cross section of the boundary zone of the multi-material tensile bars for a building platform temperature of (a) 120 °C and (b) 140 °C

Regarding the boundary zones, no clear separation between both materials can be seen. Because the separation blade of the two-chamber recoater creates turbulences, the powders are mixed during the powder deposition. This leads to a boundary zone with a width of approximately 2 mm in which both polymer powders are randomly mixed. But to allow an interpretation of the occuring of curling, the surface and geometry of both specimens need to be examined. The surface of the specimen built with 120 °C is very curly and uneven. The explanation for this can be attributed to curling, which can be seen by the orientation of the single layers in the PA12 side. The curling forms a bead and leads to strong shape deviations not only in the boundary zone but also in the overall specimen. Additionally, there are pores within the boundary zone. If these pores occur due to the material properties of PP (like can be seen in the PP side of the specimen) or due to curling needs to be analyzed in future works. For 140°C, the surface is very flat which means that curling is strongly minimized or does not occur at all. The not completely molten PA12 powder particles in both cross sections indicate that the energy provided by the thulium laser was not high enough to achieve a complete melting of the polymer.

The first layer of the PP side is thicker for a building platform temperature of 140 °C compared to a temperature of 120 °C. Due to the higher temperature of the building platform the temperature gradient towards the building platform is lower and the powder layers near the surface are warmer (see fig. 5). Thus, less energy is needed to achieve a phase transition of the powder particles near the surface and the particles are molten shortly after the start of irradiation. During the remaining irradiation time the laser radiation can penetrate also in deeper layers because of a higher transmission of the molten polymer layer compared to powder, resulting in an overall higher melting depth compared to a temperature of the building platform of 120 °C [7].

To allow also an analysis of the resulting mechanical properties of the specimens, the results of the tensile tests are shown in figure 7. All tensile bars failed in the boundary zone between both polymers. The mean value of the tensile strength of the samples built at a building platform temperature of 120 °C is 8.7 MPa and therefore, is significantly lower compared to a tensile strength of 12.9 MPa for samples built at a building platform temperature of 140 °C. Considering the cross sections in figure 6, the defects in the boundary zone lead to an overall lower connection stability and thus lower tensile strength. Also the elongation of break is higher for a temperature of 140 °C but the difference is not as distinct as for the tensile strength.

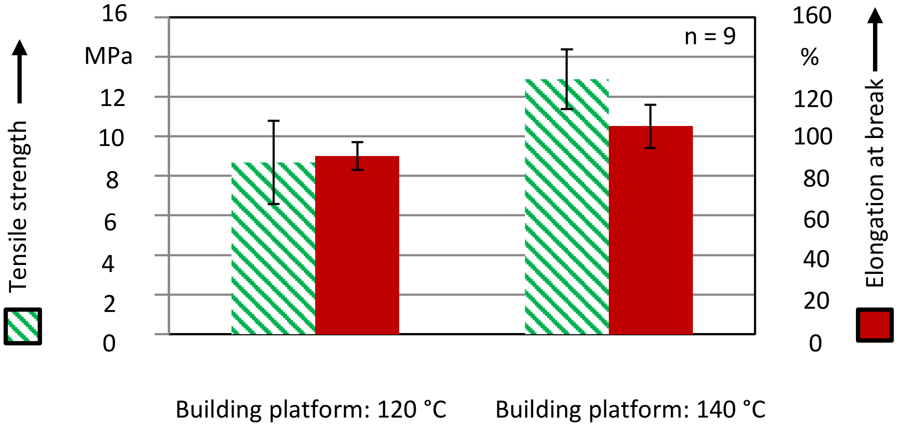

Fig. 7. Tensile strength and elongation at break of 1:5 scaled Campus tensile bars for differing building platform temperatures

5. Conclusion

In this work, the influence of different temperature gradients on the part properties of multi-material parts realized by Simultaneous Laser Beam Melting is presented. The different temperature gradients are achieved by varying the temperature of the building platform (120 °C / 140 °C). To analyze the temperature development within the powder bed, thermocouples are used during building specimens consisting of PP and PA12. The measurements show that for a building platform temperature of 120 °C the crystallization temperature of PA12 is locally reached within the boundary zone between both materials during the building process. This leads to curling and results in significant shape and geometry deviations which can be seen in the prepared cross sections. By using a higher temperature of the building platform, curling can be prevented because the crystallization temperature of PA12 is not reached before the building process is finished and the molten layers solidify together during the cool down phase. Additionally, tensile bars are generated and tested. Due to curling, the mechanical part properties are lower for a building temperature of 120 °C compared to 140 °C as could be expected by the cross sections. Altogether, the importance of an adequate temperature controlling for the SLBM process, which needs to be suited for the specific material combination, is shown by expanding the existing process knowledge.

Acknowledgements

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