

THE INFLUENCE OF CONTOUR SCANNING PARAMETERS AND STRATEGY ON SELECTIVE LASER SINTERING PA613 BUILD PART PROPERTIES

C. Kummert*, H.-J. Schmid*

*Direct Manufacturing Research Center (DMRC) and
*Particle Technology Group University of Paderborn, Germany

Abstract

Qualification of new materials for the laser sintering (SLS) process includes development of suitable parameters in terms of optimal part properties. Especially laser scanning parameters influence part porosity and therewith mechanical performance. In the present work tensile specimens were built of PA613 a new LS polyamide delivered by Evonik and was processed on an EOSINT P396. As build parameters have to be developed for the new material, scanning parameters and strategy of the PA613 specimen contour were varied in different ways. Resulting part properties were investigated by XCT-analysis as well as by tensile tests. The three-dimensional part porosity, pore density and arrangement were analyzed in relation to used laser scanning parameters and resulting mechanical properties. The investigations help to understand the existing correlations between laser energy input, part porosity and mechanical performance and therewith to find optimized build parameters for the new material.

Introduction

The material portfolio for the Laser Sintering Process (SLS) is still limited and therefore the potential to find new application fields for the Additive Manufacturing process is challenging. In particular, industries like electronics, aircraft and automotive demand high performance plastics showing high strength and temperature resistance. In this work, a new SLS material PA613, developed by Evonik, was investigated. This laser sintering powder may be implemented where PA6 is used in conventional manufacturing processes. Due to a higher melting temperature in comparison to standard laser sintering material PA12, PA613 may be applied in high temperature applications. Despite the higher melting temperature, PA613 is still processable robustly on an EOS P396 laser sintering system. However, process parameters in order to achieve high part quality have to be examined. X-ray Computer tomography (XCT) is a suitable non-destructive method to examine the build part microstructure, since high porosity and ragged surface appearance cause bad mechanical properties [1]. Therefore, a thorough investigation of these part properties in dependence on laser scanning parameters and strategy will allow for achieving improved part quality by application of optimal laser parameters. Since energy input into the part contour is supposed to be particularly important for the specimen quality, this study was mainly dealing with contour writing.

The porosity of build parts is defined as the ratio of volume of pores and the total build part volume [2]. Air pockets are also known from other polymer manufacturing processes like injection moulding, e.g. due to insufficient deaeration and shrinkage. The laser sintering process entails

several possibilities of pore formation. Besides incomplete coalescence of polymer particles, release of gases due to polymer degradation may be an additional reason for pore formation. Following, the avoidance of porosity requires a balanced energy input by the process temperature and laser, in correlation to the particular polymer and powder bulk. To find suitable laser sintering parameters for the PA613 material with particular consideration of the influence of the microstructure, parameters for the energy input into the build part contour are varied and correlated to porosity as well as mechanical properties.

State of the Art

In the laser sintering process different material properties, parameters and therewith physical and chemical phenomena influence the resulting build part properties. Every process step from powder layer recoating, heating up of the powder surface, laser exposure to solidification of the sinking layer is essential to generate a high density part with high strength and smooth surfaces. X-Ray Computed Tomography (XCT) has lately become an appropriate method to investigate porosity and the development of pores.

Dupin et al [3] investigated the microstructure of two PA12 build parts which exhibit different particle size distributions. The authors observed 2D sections and stated different porosities in X-ray tomography for different particle size distributions, morphologies and therewith lower powder bed density which led to different welding between two successive layers where pores were localized. It is explained by a hindered laser beam penetration. Further they mention bad coalescence processes of the polymer particles and diffusion of gases into the molten polymer during cooling stage as reasons for pore development. As a conclusion they highlighted particle size distribution, particle morphology, laser energy density and the crystallization temperature as the main influencing factors for residual porosity. On top of that crystallinity is investigated as another impact on mechanical properties.

Also Dewulf et al. [4] investigated PA12 build part porosity in dependence of laser scanning parameters with the help of X-ray computed tomography. Beside porosity and pores volume distribution, the XCT-model was sliced and attributed to the original STL-file to calculate an average porosity per slice. Building a cube sample they varied the laser parameters laser power, hatch distance and scanning speed and therewith the energy density in four steps. They correlated a mid-high laser energy density (Andrew Number) to minimized part porosity. Density increases with higher laser power because of higher fusion of polymer particles. Too high energy density leads to degradation of the material which causes gas emission. Furthermore, smaller pores show coalescence forming larger pores. A decrease of hatch distance leads to reduced porosity and magnitude of the porosity fluctuations. Furthermore, they investigated cylindrical pins built with different scanning patterns in different sizes, partly including contour and hatching scan lines. Exposure of only one spot causes voids in the center of the specimen. However, pins with contour scanning showed a ring of pores right inside the dense border. Likewise, the scanning order and beam offset of the contour influences the porosity of the pins.

In Rösenbergs [5] work, thin sections were analyzed by XCT-scans and showed a denser outer area in comparison to the inner build part. This effect was also observed by Rouholamin and Hopkinson [6] who varied the laser power in in order to examine its effect on build part density, morphology and mechanical properties as well. The presence of denser structures at the edges

compared to the centers may result in improved mechanical properties, as the averaged pore diameter was not affected by increasing laser power.

Pavan et al. [7] investigated the dimensional accuracy of laser sintered PA12 parts with XCT varying the beam offset and laser power of the contour vector. The 3D voxel model was compared with the original CAD file and dimensional deviations were analyzed for holes in vertical and horizontal walls with different thicknesses. While the number of slices affects the dimensions of holes along the z- build direction, the xy- direction dimensions are affected more by the beam offset. It was found to be difficult to find one beam offset, which results in similar dimensions in all orientations and placements of holes within a test specimen.

Ghita et al. [8] used Micro-Computer Tomography (μ CT) to investigate tensile specimens of virgin and mixed PEK powder . The usage of recycling material led to higher porosity in the part and therewith to worse mechanical properties. Coalescence of polymer particles is hindered by increased particle sizes and higher melt viscosity of the aged material, with the result of higher porosity. The latter might be compensated by higher laser energy input.

On one hand material properties like particle size distribution, particle morphology and ageing stage have an impact on pore development during laser sintering and on the other hand process parameters like laser energy input and temperature are influencing factors. However, the found correlations are not linear and suitable process parameters have to be found for each new material with individual polymer and powder / particle properties.

Experimental methods

Within the present work PA613, a new polyamide for the laser sintering process, delivered by Evonik is investigated. Virgin powder is processed on the laser sintering system EOS P 396 to build zx-direction tensile specimens according to DIN EN ISO 527 (1A). Within preliminary examinations of laser scan parameters of the filling lines the material showed quite constant mechanical properties over a wide range of energy input (0.027-0.045 J/mm² areal energy density). Where the areal energy density (ED) also called Andrew Number is calculated by the following equation:

$$ED = \frac{P}{v * d}$$

Where P is the laser power, v the scan speed and d the hatch distance between two laser scan lines. To improve the anisotropy between xy- and zx- build directions as well as the build part surface, the laser parameters and strategy of the contour is varied, whereas the filling is kept constant in the present work: Using a constant layer thickness of 120 μ m the laser power to exposure the filling lines was 30 W, the scan speed 4750 mm/s and the hatch distance 0.16 mm, resulting in an area energy density (ED) of 0.0395 J/mm². Furthermore, the contour line, shown in red in Figure 1, was varied as outlined in Table 1. The first contour parameter set “REF” serves as a reference. In comparison to that, energy density was increased by a reduced scan speed (“SS1”), higher laser power (“LP”) or a double exposure (“DE”). For parameter set “BO” the beam offset

was increased and therewith energy density decreased. On top of that, further specimens were built with no contour exposure (“NC”) at all. To calculate the energy density of the contour exposure the hatch distance is replaced by the beam-offset.

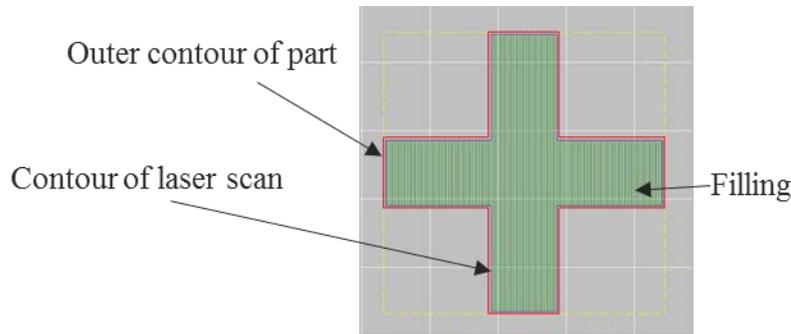


Figure 1: Laser scanning lines of the build part filling (green) and contour (red)

Table 1: Laser parameters for contour line variations

Parameter set	Laser power [W]	Scan speed [mm/s]	Frequency [-]	Beam-Offset [mm]	Energy density [J/mm ²]
REF (reference)	25	3000	1	0.2	0.042
SS1 (decreased scan speed)	25	1000	1	0.2	0.125
SS2 (destroyed by test)	25	1000	1	0.2	0.125
LP (increased laser power)	40	3000	1	0.2	0.067
DE (Double exposure)	25	3000	2	0.2	2 x 0.042
BO (increased Beam-Offset)	25	3000	1	0.35	0.024
NC (no contour)	-	-	0	-	-

For each parameter set five tensile specimens were built, whereof four were tested according to DIN EN ISO 527 on an INSTRON 5569 EH universal testing machine. The fifth of each set was used for XCT-Scans. To analyze the effect of tensile destruction on the microstructure also a tested specimen built with parameter set SS1 was scanned and is called SS2 in the following. The XCT-scan (computed tomography) was performed with GE Phoenix Nanotom S with 70 kV

150 μA and 1440 projections. As it is not possible to measure the whole tensile specimen, a cut was taken from the samples as shown in Figure 2. Furthermore, the data are analyzed using the “Defect Analysis” of the software VGstudio MAX 2.2 from Volume Graphics GmbH. The algorithm compares the gray scale value with a threshold, so that only defined defect sizes are taken into account. Hereby the voxel size is 10 μm . Furthermore, the Region-Of-Interest (ROI), which is analyzed in detail, was restricted to the contour area. It has to be noted that this region might differ between the specimens as dimensions are different due to varied contour energy input. However, evaluated characteristics like porosity, averaged pore volume and pore density are relative values so the information is still representative.

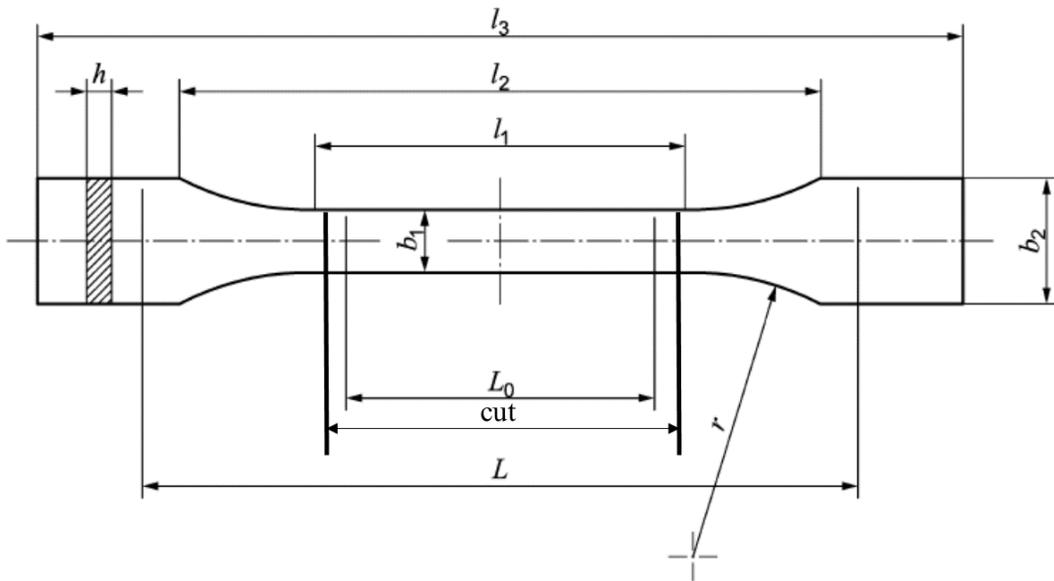


Figure 2: Cut-out of tensile specimen (DIN EN ISO 527 1A) for XCT-scans

Results and discussion

Tensile specimens were built with different contour laser parameters as described before and tensile tests as well as XCT-scans were conducted. If not mentioned differently, evaluated results refer to the contour regions as the porosity of the inner specimen body was in average 4.39 ± 0.26 % for the seven investigated specimens. However, the contour region showed lower porosity in comparison to the inner build part for all investigated specimens as visible in Table 2. This effect was already stated by Rouholamin et al. [6] and Rösenberg et al. [5] for PA12, who assumed that the thickness of the laser sintered part and the cooling rate might have a strong influence on the outer solid area and the porosity.

Table 2: Result of defect analysis of contour regions

Sample	Porosity [%]	Averaged pore volume [μm^3]	Pore number concentration [$1/\text{mm}^3$]
REF	1.86	51408	362
SS1	3.08	119458	261
SS2	3.58	118015	300
LP	0.84	42155	199
DE	1,61	52978	304
BO	2,09	56872	368
NC	2,17	72351	300

Figure 3 shows a section of a XCT-scan of specimen “REF”, where the contour region is marked by the blue frame. In the contour area the pores have been evaluated (yellow). In comparison to the inner part (black pores) the number of pores seems to be decreased. The evaluated values confirm the visual appearance. The reference contour laser exposure REF shows a porosity of 1.86 % with a pore-density of 362 $1/\text{mm}^3$ and an averaged pore volume of 51408 μm^3 .

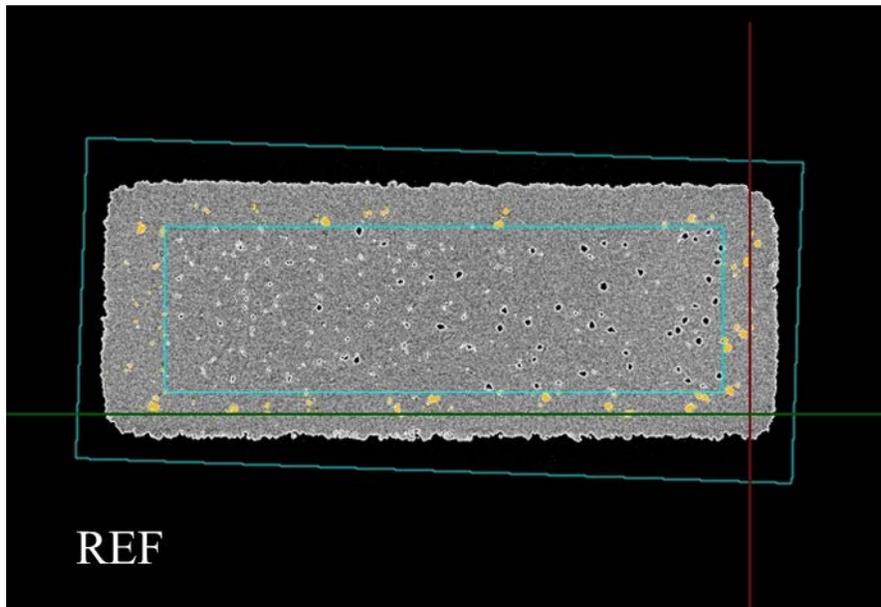


Figure 3: XCT-scan picture of specimen “REF”

The specimen LP was built with increased contour laser power (40 W instead of 25 W) and consequently with higher energy density of 0.067 J/mm². The contour region shows the lowest porosity of all specimens due to both, lower number concentration and averaged pore volume. This effect leads to the assumption, that polymer particles show superior melt and coalescence behaviour by the higher energy input. Presumably as a consequence of the low porosity elongation at break and tensile strength are slightly improved in comparison to REF (see Table 3).

Table 3: Tensile properties for different contour build parameter sets

Parameter set	Elongation at break [%]	Modulus of elasticity [MPa]	Tensile strength [MPa]
REF	13.59	2090	57.84
SS2 (SS1)	10.45	2132	56.56
LP	17.77	1906	58.78
DE	21.46	2076	59.51
BO	14.06	1968	58.86
NC	6.85	1868	54.16

However, the highest mechanical properties show specimens built with double contour exposure (DE). Even though the specimens built with higher energy input by double contour exposure show only slightly lower porosity than the REF samples. Here another effect may play the decisive role, but has to be investigated in the future.

A further reason for better mechanical performance might be a smoother part surface. In Figure 4 XCT-Scan sections of specimens LP, DE and NC are shown. No contour laser exposure NC shows worst surface appearance and worst mechanical properties as well compared to all other cases. The specimen surface is ragged caused by the alternating laser scan strategy in x- and y-build direction and lead like notches to earlier material failure. Although a comparison of the contour regions of NC to the other specimens is difficult due to different part microstructure, a qualitative comparison shows that porosity is quite high for NC due to a high average pore size volume.

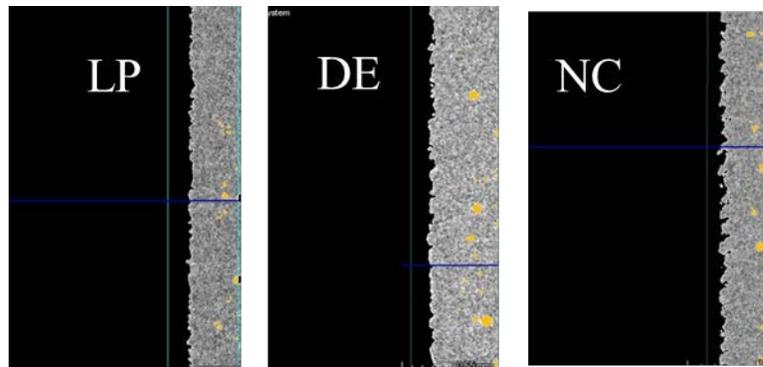


Figure 4: XCT-Scan sections of specimens LP, DE and NC to regard the surfaces

As the specimens SS1 and SS2 were manufactured with the same laser parameters, experimental data are examined together. Comparing the results of the “Defect Analysis” in Table 2, values are in the same range. SS2, which was destructed in a tensile test shows slightly higher porosity due to a higher number of pores. It was presumed that the tensile test leads to a stretch and an enlargement of the pores. However, the visual appearance does not substantiate this assumption, comparing in Figure 5. The cross section of the specimen is smaller as it is expected for tested specimens. In comparison to the reference specimen REF both of them show a distinct increased

porosity due to higher averaged pore volume. The number of pores is even higher for the reference. These bigger pores become clearly visible in the XCT-scan pictures where the pores are arranged like a ring which is visible in Figure 5. Possibly the local higher energy input by low scan speed leads to material degradation or at least condensation and therewith to gas inclusion. Such a pore ring was also detected by Dewulf et al. [4] for PA12 LS parts. Furthermore, mechanical properties are slightly lower in comparison to REF probably caused by the higher porosity within the contour region of the specimen.

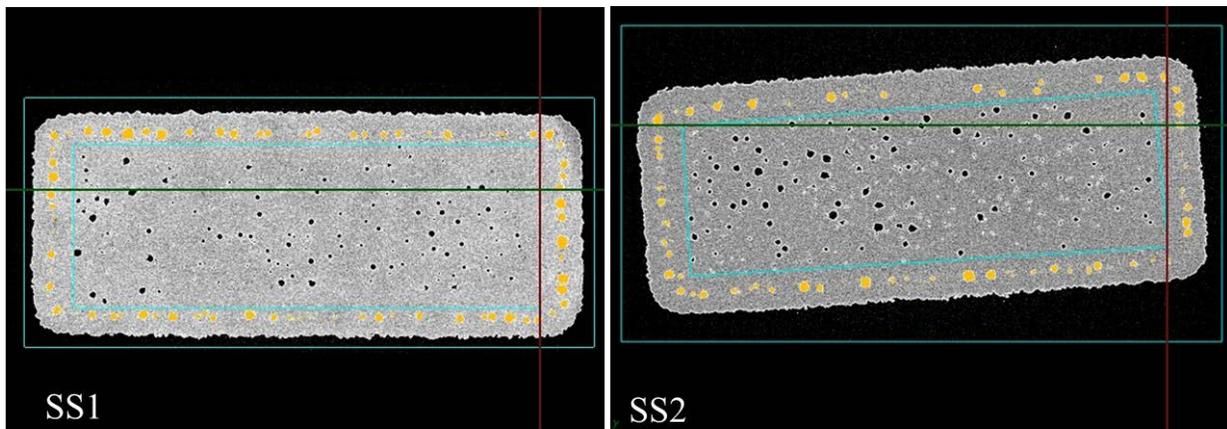


Figure 5: XCT-scan of SS1 and SS2 specimen

The last parameter which is varied, is the beam offset of the contour scan line. Instead of 0.2 mm the distance was set to 0.35 mm. Pores of the contour area of this specimen are bigger and result together with a higher number of pores to higher porosity in comparison to REF. Due to the higher distance of the contour to the filling scan lines, polymer particle coalescence is hindered. Again a ring of pores is visible in Figure 6 but not that obvious as seen with SS2. However, mechanical properties are not significantly affected by the higher porosity.

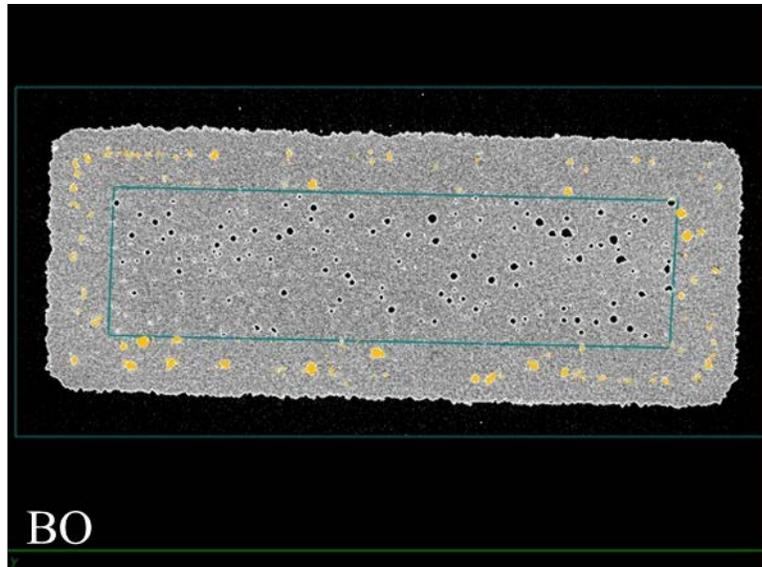


Figure 6: XCT-scan of BO specimen

As a result, higher energy input into contour regions of the specimens leads until a certain degree to lower porosity in the particular area and further to higher mechanical properties and smoother surfaces. However, too high energy input causes pore development with higher average pore volume. Overall, the microstructure of the contour region appears to be an important factor for the build part performance.

Conclusion and outlook

In the present work the contour region of laser sintered parts built with different exposure parameters and strategies were investigated by three dimensional X-Ray Computed Tomography (XCT). It was shown, that a variation of energy input gets visible in the microstructure level and helps to find reasons for mechanical failures or improved properties. Apparently, the microstructure of the contour region of the specimen is crucial for mechanical performance as filling exposure parameters were kept constant, resulting in quite constant porosity in the bulk region. Not only porosity but also surfaces can be regarded by the XCT-analysis. As the notch effect is a known mechanism to cause build part failure, considering the specimen surface is an important part of the investigations. Considering the small sample size, interesting influences of different laser contour parameters became visible. While a moderate increase of energy density leads to lower porosity in the contour region and therewith to improved mechanical properties. Too high energy led to a porous ring, possibly due to material degradation and therewith to reduced mechanical properties. In the future, sample size and design of experiment of the conducted investigations have to be enlarged, to verify the found results and to get a better understanding about porosity development as well as build part surface appearance. This will narrow down the influences and limits of contour exposure for high build part quality.

References

References

- [1] J. P. Kruth, P. Mercelis, L. Froyen et al., “Binding Mechanisms in Selective Laser Sintering and Selective Laser Melting,”.
- [2] S. Lowell and J. Shields, *Powder Surface Area and Porosity*, Chapman and Hall, London, New York, 1984.
- [3] S. Dupin, O. Lame, C. Barrès et al., “Microstructural origin of physical and mechanical properties of polyamide 12 processed by laser sintering,” *European Polymer Journal*, vol. 48, no. 9, pp. 1611–1621, 2012.
- [4] W. Dewulf, M. Pavan, T. Craeghs et al., “Using X-ray computed tomography to improve the porosity level of polyamide-12 laser sintered parts,” *CIRP Annals*, vol. 65, no. 1, pp. 205–208, 2016.
- [5] S. Rüsenberg, ed., *Mechanical and Physical Properties – A Way to assess quality of Laser Sintered Parts*, 2011.
- [6] D. Rouholamin and N. Hopkinson, “An investigation on the suitability of micro-computed tomography as a non-destructive technique to assess the morphology of laser sintered nylon 12 parts,” *Proceedings of the Institution of Mechanical Engineers, Part B: Journal of Engineering Manufacture*, vol. 228, no. 12, pp. 1529–1542, 2014.
- [7] M. Pavan, M. Sinnaeve, S. Leysens et al., “Determining the dimensional accuracy limits of laser sintered PA12-parts: from artefact design to dimensional characterization by X-Ray Computed Tomography,” *euspen*, pp. 149–153, 2017.
- [8] O. R. Ghita, E. James, R. Trimble et al., “Physico-chemical behaviour of Poly (Ether Ketone) (PEK) in High Temperature Laser Sintering (HT-LS),” *Journal of Materials Processing Technology*, vol. 214, no. 4, pp. 969–978, 2014.