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A Round Robin study for selective laser sintering of polymers: Back tracing of the pore morphology to the process parameters

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ABSTRACT

The mechanical properties of polymer parts built by Selective Laser Sintering are strongly related to the internal microstructure which differs with the applied production parameters. The paper focuses on the back tracing of the pore morphology of laser sintered polyamide-12 samples to the process parameters. Therefore, a data base is used which is supplied by a Round Robin initiative and includes mechanical tensile tests and the microstructural analysis of the pore morphology of several different sample charges built with different machines. The pore morphologies (porosity, pore density, pore shape and pore arrangement) measured by X-ray computed tomography are compared and discussed regarding the employed parameters and the resulting mechanical properties.

The investigations point out that pore density is a superior indicator than porosity for mechanical issues. This is especially valid along the build direction since pore morphology has shown to be strongly anisotropic. Moreover, the analysis revealed that pore density is strongly affected by the process temperature, which is proved to be essential for the fabrication of mechanical robust parts using Selective Laser Sintering.

1. Introduction

Selective Laser Sintering (SLS) using polymer powder is one of the classical Additive Manufacturing processes on the market (Kruth, 1991). This layer-based technology is able to produce functional plastic parts for design approval, trial and test, or even end-use in true working environments. Fabricated directly from 3D CAD data, complex designs and incorporated features can be achieved more easily with AM technologies, which make SLS predestinated for high performance applications including aerospace, tooling for injection molding, medical or automotive devices (Levy et al., 2003).

A typical SLS process includes three steps which are repeated for each layer. In the first step, the building platform is lowered by the thickness of one layer and powder is spread on the platform using a recoater system like a roller or blade. Secondly, the powder bed is heated just below the melting temperature to minimize the required laser energy. And thirdly, a laser beam scans the powder layer in order to melt defined powder layer areas. These steps are applied alternately for each layer until the part is completed (Laumer et al., 2014). Then, the heaters are turned off and the powder bed cools gradually. Once the powder in the part chamber, or the "part cake", is cooled below the glass transition temperature, it is removed from the chamber and the loose powder is detached from the part (Bourell et al., 2014).

The key benefit of SLS compared to other polymer-based Additive Manufacturing technologies is the ability to fabricate parts which feature mechanical properties that are close to the mechanical properties obtained by injection molding (IM) (Kruth et al., 2008). However, while similar yield strengths are achieved, the maximum elongations of SLS parts are typically an order of magnitude lower as shown by Ajoku et al. (2006) and Griessbach et al. (2010). Morerover, DuraForm (2014) specifies the DuraForm™ PA with an elongation at break of 14% which is well below the 200–300% elongation achieved by IM parts made from polyamide. Bourell et al. (2014) explains this on the one hand by the different crystallinity of SLS and IM parts which is related to the different cooling cycles of the processes, and on the other hand by a different microstructure provided by the SLS process, which includes pore formation and the presence of non-molten residual particles (and cores).

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SLS process parameters are e. g. area energy density of the laser beam (Leigh, 2012), scan speed (Drummer et al., 2014), temperature of the powder bed (Bourell et al., 2014), powder bed density (Drummer et al., 2012) and layer thickness (Monzon et al., 2009). They affect the microstructure of the part and thus the mechanical properties. The results of Bourell et al. (2014) showed that the mechanical properties along the building direction are very sensitive to process deficiencies, leading easily to very brittle material behavior along the building direction. As discussed by Bourell et al. (2014), such behavior is typically attributed to weak interlayer connection due to residual particles and cores, periodic porosity and interlayer porosity which arise when the energy input is insufficient for the complete melting of the layer section and the partial melting of the previous melted layer.

Porosity is known to be affected by the laser energy input. While too low laser energy results in enhanced porosity due to incomplete melting as it was discussed by Dupin et al. (2012), exceeding laser energies lead to degradation and vaporization and thus to increased pore formation too. Lowest porosity values around 3% are typically achieved with area energy densities between 2.0 J/cm^2 and 3.5 J/cm^2 as reported by Dupin et al. (2012). However, it is not fully clear how other parameters in comparison like process temperature influence the porosity formation. Thus more extensive studies are needed which do not only include porosity or density measurements but also methods for the determination of the pore size distribution, pore shapes and arrangements.

Pore morphology of additively manufactured samples can be effectively measured using X-ray computed tomography as it was used by several research groups. Dupin et al. (2012) used this technology in order to estimate the volume and frequency distribution of the pores volume of different polyamide-12 samples built with SLS. Dewulf et al. (2016) investigated the influence of different laser scanning parameters on the porosity of laser sintered polyamide-12 samples using x-ray computed tomography. For metal samples built with Selective Laser Melting (SLM) Alsalla et al. (2016) used this technique for the determination of the density of cellular structure components. However, the huge potential of X-ray computed tomography is seldom fully utilized. Besides densities, volume and frequency distribution, the technology offers also the possibility to visualize three-dimensional pore shapes and arrangements. A thorough investigation and discussion of such features could help to understand varying mechanical properties of laser sintered polymer samples.

Typically applied research methodologies base on the variation of few parameters using a single machine and thus are not immune to systematic errors induced by the respective machine characteristics. As consequence revealed processing-structure-property-relationships may be machine-dependent which can lead to uncertain argumentation for explaining them and even to contradictory conclusions. For example, Caulfield et al. (2007) observed that tensile testing samples exhibit higher ductility if they are built with vertical orientation (z axis) compared to samples built with horizontal orientation (x and y axis), which contradicts Leigh (2012) stating that the z- axis will yield a significantly lower elongation at break than the x and y axis specimens. Instead, experimental methodologies which include the broad variation of parameters and different machines exclude systematic errors and thus can be advantageous to achieve a more profound understanding of processing-structure-property-relationships.

The paper focuses on the back tracing of the pore morphology of polyamide-12 (PA12) samples to the process parameters. Therefore, a Round Robin initiative supplies several different sample charges built with different machines and parameters. These samples are mechanically tested and characterized regarding their pore morphology using X-ray computed tomography. The concluding structure-property relationships are discussed and compared with the process parameters to clarify correlations between process parameters, mechanical properties and pore morphology.

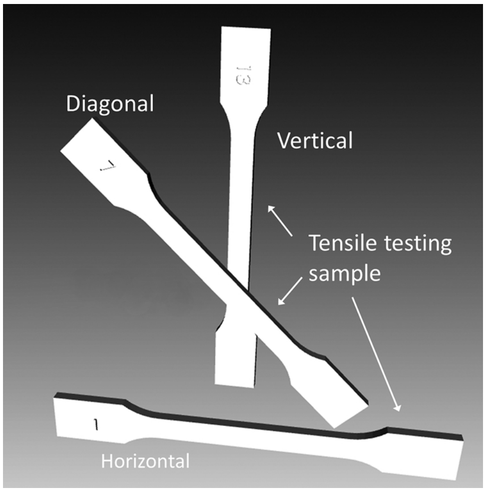

Fig. 1. CAD design of a testing sample set consisting of three differently orientated tensile bars: Horizontal orientation corresponds to in-plane direction (x and y axis), vertical orientation to build direction (z axis).

2. Experiments

2.1. Design of the Round Robin study

The Round Robin includes six different SLS machines (M1-M6) from different manufacturers (EOS GmbH, 3D Systems, DTM) which were used to produce a range of tensile samples (DIN EN ISO 3167, Type A) with different build orientation (H: horizontal; D: diagonal, V: vertical) (see Fig. 1). For each machine different parameter sets (laser power, scanning speed or layer thickness, etc.) were applied, which were specified to be the optimum for the respective machine. The samples were made from the SLS standard material polyamide-12 Duraform PA (3D Systems) or PA2200 (EOS) which base on the same powder VESTOSINT produced by Evonik.

The most relevant parameters are displayed in the parameter protocol of Table 1. It represents the fundament of the following back tracing analysis.

In the following discussion, the laser energy input will be represented by the area energy density (AED) which is (Beaman et al., 1997)

$$AED = \frac{P}{\pi\left(\frac{d}{2}\right)^2}\frac{d}{v}\frac{d}{h} = \frac{4P}{\pi v h} \tag{1}$$

with the laser powder P, the scanning speed v and the hatch distance h. Since the scanning speed is unknown for M3, no respective value could be calculated in this case. The values will be used for the discussion in the following chapters.

2.2. Tensile testing

Tensile tests were performed to quantify the applicability of the samples. They are carried out using the *Zwick/Roell 100 Allround-Line* machine with a test speed of 5 mm/min. The values that are evaluated and compared are the ultimate tensile strength (UTS) and the total elongation (TE) which is also known as elongation at break. The yield point is defined for plastics as the first point on the stress-strain curve at which an increase in strain occurs without an increase in stress (ASTM D638-02a, 2017).

2.3. X-ray computed tomography

The samples were scanned with the Werth TomoCheck 200 3D system with a CT sensor. The detector possesses 1024 × 1024 pixels at