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A Round Robin study for Selective Laser Sintering of polyamide 12: Microstructural origin of the mechanical properties

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ABSTRACT

The mechanical and microstructural investigation of polymer parts (polyamide 12) fabricated by Selective Laser Sintering as part of a Round Robin initiative is presented. The paper focuses on the microstructural analysis of the Round Robin samples and their evaluation regarding their effect on mechanical properties with respect to each other. Therefore optical microscopy on microtomed samples, X-ray computed tomography and Differential Scanning Calorimetry is used to determine the morphology of residual particle cores and of internal pores.

The mechanical tensile testing revealed a high variability of the ductility of the samples among the used machines and a distinctive anisotropic mechanical response. Especially the quite brittle characteristic along the building direction has shown to be still a crucial challenge for the process. However, one machine delivered samples with outstanding ductility with total elongation values of about 21% along the building direction and of about 32% planar to the layer. This result was back traced to a distinctive pore and residual particle morphology which is characterized by low pore concentration, the absence of coplanar pore or residual particle arrangements and the highest degree of particle melting measured. Furthermore, the analysis depicts that both features, pores and residual particles, contribute to the mechanical properties significantly and that they are not necessarily linked since they can vary independently in a certain range depending on the machine configuration.

1. Introduction

Selective Laser Sintering (SLS) is one of the most widespread Additive Manufacturing processes that emerged in the last decades [1,2]. It allows the fabrication of three-dimensional polymer components with complex shapes according to CAD data directly out of powdery material without the need of tooling and setup [3]. Therefore parts are built up layer by layer. Three steps are repeated for each layer: First, the platform is lowered by the thickness of one layer. Second, powder is deposited on the building platform using a moving roller or blade device and preheated to a temperature close to the material melting point. And third, a laser beam scans the powder layer with arbitrary trajectories in order to melt specific powder layer areas. These steps are applied alternately until the part is completed. Before the parts can be extracted from the machine, homogeneous cooling has to take place whereas the viscose melted polymer crystallizes with minimal distortions.

The main advantage of SLS over other Rapid Prototyping or Additive Manufacturing technologies using plastics is the ability to produce parts which feature material properties that are very close to the material properties obtained by injection molding [4]. However, this is only achieved when a robust process and material routine is established leading to high quality and repeatable results. In many cases, SLS machine users observe minor reproducibility regarding to mechanical and geometrical properties [5], limiting the industrial application and cost-efficiency of SLS.

Especially the mechanical properties along the build direction have shown to be a challenge for the process. Ductility and strength are generally lower along the build direction than perpendicular to it and they are also very sensitive to varying process conditions and parameters. This can be explained by the stacking-layer-process which leads easily to insufficient interlayer connection, e. g., if energy input or

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the local process temperatures are too low for the complete melting of the layer section. Insufficient interlayer connection is related to distinctive microstructural features such as pore formation or residual particles (or cores) [6,7] which can occur with varying frequency and morphology (size, shape, orientation).

The occurrence of different morphologies of porosity inside polymer parts built by SLS is well known [6–8]. While it is affected by many material and process parameters [8], an evidentiary understanding of its morphology and impact on the mechanical properties is still missing. Very often the interlayer porosity is blamed for inferior mechanical properties along the building direction [6]. Besides pores, residual particle and particle core arrangements due to partial melting were also identified to affect the mechanical response of SLS processed samples. It was stated that an increase of the degree of particle melt (DPM) leads to an improvement of the mechanical properties and coplanar aligned residual particles and cores promote mechanical weakness along the building direction [6,9]. This is explained by the many short binding necks creating high stiffness which result in short elongation [10].

Nevertheless, a quantification of the influence of both effects (porosity and residual particles) on the mechanical properties in comparison has not been reached yet. Typically applied research methodology base on the variation of few parameters which leads to complementary changes in both features and thus do not allow the estimation of their respective significance for the mechanical properties. This can be exemplarily seen in [8] where the variation of the laser beam energy leads to congruent change of the crystal weight fraction and the porosity with strong impact on the elongation at break but with no clue of which effect is more important. Thus experimental methodologies should be applied which include the broad variation of parameters and different machines resulting in more versatile microstructural morphologies.

In order to measure the influence of pore formation and residual particle (cores) on mechanical properties with respect to each other, a comprehensive microstructural analysis is performed upon tensile test samples built during a Round Robin initiative based on six different machines and parameter sets. The internal pore morphology of the samples is measured by X-ray computed tomography while the residual particles (and particle cores) are investigated using microscopy on thin sections and Differential Scanning Calorimetry (DSC). The results are used to trace back conspicuous features of the tensile testing outcome to the source in order to value the significance of pore and residual particle morphology.

2. Round Robin methodology and tensile test samples

The Round Robin includes six different SLS machines and users (M1-M6) which produced a range of tensile samples (ISO 3167, Type A) with different build orientations (see Fig. 1). For each machine different parameter sets (laser power, scanning speed or layer thickness, etc.) were used, which were specified by the applicants to be the optimum for the respective machine. The samples were made from the standard material polyamide 12 Duraform PA (3D Systems) or PA2200 (EOS) which base on the same basic powder VESTOSINT produced by Evonik. An overview of the production parameters are displayed in Table 1.

Six sample sets were produced in machine M1, M3, M4 and M6, three sets in machine M2 and one set in machine M5. The nominal and measured linear dimensions of the samples as well as the classification details according to ISO 2768-1 are displayed in Fig. 2. The samples of M6 could not be measured due to strong deformations and displacements and are beyond of any fabrication tolerances. The samples fabricated with machine M5 cannot be classified by the norm since the sample length is exceeding the defined deviation of ± 2.5 mm for the "very coarse" category by far. Instead, the samples of the machines M1, M3 and M4 can be categorized as "coarse", the samples of M2 as "medium".

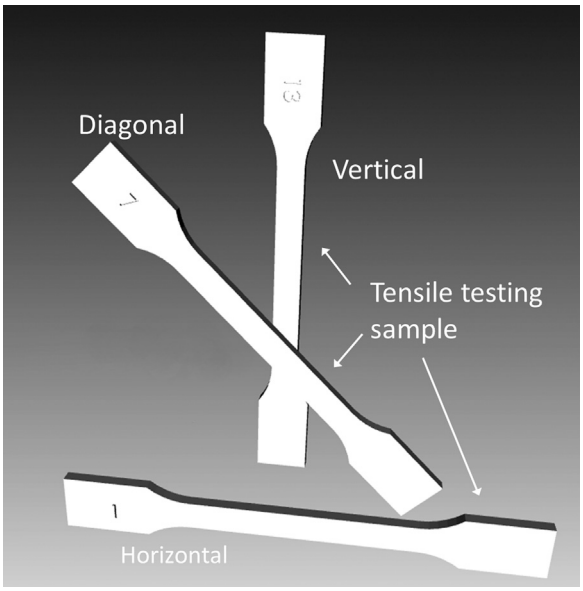

Fig. 1. CAD design of a testing sample set consisting of three differently orientated tensile bars: Horizontal orientation corresponds to in-plane direction, vertical orientation to build direction.

3. Characterization methods

In Fig. 3 an overview of the performed characterization methods is given. The mechanical properties which resemble the functionality of the samples are determined via tensile testing according to ISO 3167. Microstructural features which are represented by the residual particle (core) arrangements and the porosity can help to explain the appearance of functionality (mechanical) issues. While arrangement of residual particles and cores are verified by optical analysis of thin sections, the general amount of residual particle is resembled by the degree of particle melt which is determined using Differential Scanning Calorimetry. In order to analysis the pore morphology of the samples comprehensively, X-ray computed tomography is used which delivers information about porosity, pore density, pore arrangement, pore orientation and pore size distribution. All respective determination methods are commented in detail in the following sections.

3.1. Mechanical testing

Tensile tests 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 [11].

3.2. Thin section analysis

Optical analysis was performed on thin sections with a thickness of 50 µm prepared by a microtome. Polarized light was used in order to distinguish between non-molten, partly molten and fully molten particles in the sample inside. The preparation of the section was realized in that way that they display the layer stack.

Already mechanically tested tensile test samples were picked and prepared by the microtome. The thin sections were extracted from the furthest clamping areas of the tensile bars where no plastic deformation affecting the samples' microstructure is introduced upon tensile and clamping force. This was verified by controlling the geometry of the