Understanding the influence of energy-density on the layer dependent part properties in laser-sintering of PA12

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Abstract

As the demand for individualization and complex parts is continuously growing, laser-sintering of polymers is on the edge from a pure prototyping technology to manufacturing parts for applications in series production. The influences on resulting parts and layer depending part properties are well known in the literature but the understanding of the interaction between process parameters and layer dependent properties is missing and limiting the dimensioning. Within this study, tensile bars with different amounts of layers and energy densities were produced and investigated for the resulting mechanical properties, roughness, density and the degree of particle melt. The results showed a strong interaction between the energy density and amount of layers, which results in differences in the fracture behavior as well as the mechanical properties. Therefore, the presented results enable the prediction of necessary part thickness for dimensioning thin parts with laser-sintering.

Introduction

Additive manufacturing techniques like laser-sintering of polymers (LS) have gained increasing interest for producing individualized parts and small lots for industrial applications [1]. LS manufactures parts by layer-wise fusing of mainly polyamide powders using a CO₂-laser [2]. The resulting part properties are generally influenced by the bonding of the layers and the used process parameters like the energy density (ED) [3] as well as the temperature of the building chamber [4]. The parts possess specific properties like a high surface roughness [5], an anisotropic mechanical behavior and a remaining porosity [2]. Different investigations [6, 7] have been performed to characterize the effect of the used ED on the resulting part properties and how the layer-wise manufacturing influences the development of the part properties [8, 9]. The missing link is the interaction between those influences and thus presented in this paper.

State of the Art

The laser-sintering process is divided in 3 main steps [2]. First, a layer of powder is spread across the building platform, heated just below the melting temperature of the used material and molten by a CO₂-laser. Afterwards the building platform lowers by a layer thickness and the steps repeat until the part is fully generated [10]. Due to the layer-wise processing the resulting parts have specific characteristics which divide them from conventional processes like injection molding [11]. Due to the high temperatures during processing and a slow cooling, the parts have a high degree of crystallinity and a homogenous morphology [2]. The surface structure is determined by attaching or semi molten powder particles and lead to a high roughness [5]. Inside the parts a porosity typically between 3-5 % remains, with pores mainly orientated along the layers, which reduce the cross section of the parts. These pores may lead to increased notch effects inside the parts and therefore influence the resulting mechanical properties [2, 12]. In dependency of the used process parameters, unmelted particles can remain in the parts due to inhomogeneous melting resulting of mostly under dosed energy input [13].

The resulting LS-parts exhibit higher mechanical properties and densities with increasing part thickness [8] as well as influences on the dimensional accuracy, which are also affected by the used hatching strategy [9]. The properties are mainly determined by the used process parameters, which show decreasing anisotropy with increasing energy density [14] until a maximum is reached which is limited by material degradation [15]. Besides the energy the used temperatures inside the process chamber [16], the orientation of the parts during processing as well as the scanning strategy [17] influence the size and shape of the melt pool and therefore the temperature dependent material properties like viscosity [15, 18] and surface tension [19]. The layer attachment mainly influences the resulting part properties and the layer-wise manufacturing of the parts [8, 9]. Still there is a remaining anisotropy mainly for the elongation at break, which leads to higher mechanical properties along the building platform [10].
Summing up the state of art, it shows that within the laser-sintering of polymers the influence of the process parameters and especially the ED on the resulting part properties have been investigated in many approaches. Furthermore, the layer-wise manufacturing has exhibited a strong influence on the development of the part properties. The missing link is the interaction between those influences, as they tend to interact and have a great effect on the part properties, which is the aim of the presented results.

**Experimental Setup**

The investigations were performed using a polyamide 12 (PA12) powder of the type PA2200 (EOS GmbH, Krailing Germany) with a refreshing rate (ratio between new and used powder) of 50 %. Powder mixing was performed using a mixer of the type MP-20 (Somakon UG, Lünen Germany) for 30 min with a rotational speed of 200 min⁻¹. To ensure a good powder quality for processing, the viscosity number (Vn) was determined according to DIN EN ISO 307 [20] and showed a value of 61 ml/g. Additionally, the packing density was measured following DIN EN ISO 60 [21] and showed a density of 0.45 g/cm³. The used powder has a melting peak temperature of 185 °C and a crystallization peak temperature of 150 °C.

The specimen’s geometry of the type 1A according to DIN EN ISO 527 [22] was used with different number of layers (1, 2, 3, 4, 5, 10, 15, 20, 25, 30, 35, 40) with constant layer thickness of 100 μm and hence resulting varying part thickness. The parts were produced using a LS-manufacturing system of the type P110 (EOS GmbH, Krailing Germany) with a building chamber temperature of 167 °C and a scanning speed of 2500 mm/s. A hatchdistance (hd) of 0.25 mm was used with 4 different energy densities (ED) (0.25, 0.30, 0.35 and 0.40 J/mm³). To ensure a constant thermal history of the parts, 4 building jobs were performed with 8 parts of the same number of layers in the building cross section. Beginning with the highest amount of layers in the first building layer.

The part’s dimensions were analyzed using a micrometer for the thickness and a coordinate measuring machine for the width. The characterization of the morphological properties was performed using thin cuts through the cross-section of the specimen’s middle section and analyzed by transmitted light microscopy. The surface roughness was measured using a tactile stylus instrument Waveline 20 (Jenoptik, Jena Germany) with a stylus tip of 2 μm and an attaching force of 0.8 mN. The roughness Ra was determined according to DIN EN ISO 4287 [23] in 3 parallel lines in the middle of the test specimen along the longest axis for the bottom and the top surface with a measuring distance of 15 mm. The density measurement was performed volumetric. To investigate the melting behavior, the degree of particle melt (DPM) was determined according to Zarringhalam [24] by performing DSC-measurement of the specimen’s cross-section following 11357 [25] with a heating ratio of 10 K/s and evaluation the peak areas.

The specimens for mechanical testing were conditioned to a humid state until reaching weight consistency. The weight of the specimens was measured repeatedly by means of a precision balance in order to ensure the weight consistency. The mechanical properties were measured following the DIN EN ISO 527 [22] with a crosshead speed of 1 mm/min for the young’s modulus using a testing setup of the type 5948 (Instron, Darmstadt Germany) and 50 mm/min for the elongation at break and the tensile strength with a tensile testing machine of the type 1484 (Zwick Roell, Ulm Germany). The fractured surface was analyzed by scanning electron microscopy to characterize the breaking behavior of the parts.

**Results and Discussion**

The dimensional accuracy of the parts is strongly influenced by the interaction of the ED and the number of layers as shown in Figure 1 and exhibits an oversize of the thickness. Mainly the parts with a low amount of layers display a high deviation of the thickness, which increases with a higher ED especially for the first two layers. The resulting thickness consists of two main factors. The surface structure with attaching or semi molten powder particles and the core of the parts, which consist of the molten structure and form in combination with the surface the thickness of the specimens. Higher EDs lead to deeper penetration of the powder bed in the first layers and therefore a higher deviation at the beginning, which is slowly negotiated with increasing layers as a constant layer thickness of 100 μm is added and develops the resulting thickness of the parts.
The differences in the thickness are shown in the morphological structure in Figure 2, which shows the cross section of 1 layer parts with an ED of 0.25 and 0.40 J/mm$^3$. As the surface of both specimens tend to show a similar structure, the higher ED exhibits a thicker molten cross section occurring from a deeper penetration of the laser beam into the powder bed and therefore developing a higher dimensional deviation. The oversize decreases with an increasing amount of layers independent from the used ED. By reaching 5 layers the deviation negotiates and is mainly influenced by the cross section, as the laser does not penetrate the powder beneath the parts and hence increases with a constant layer thickness independent from the ED.

As the morphology (Figure 2) exhibits segments with unmolten particles for the lower EDs, the density and DPM development with increasing number of layers is important for the part properties. As seen in Figure 3 left, the density increases with higher amounts of layers. The density for the specimens with a low amount of layers is higher for the ED 0.35 and 0.40 in comparison to the two lower ones. After reaching 10 layers, the increase of the density decreases and slowly increases to 40 layers. In comparison, the DPM also shows an increase with a higher amount of layers as well as higher EDs. As the density is nearly constant with the
changing EDs, the morphology (Figure 2) showed unmolten particles the DPM is lower for the ED of 0.25. As the particles have a higher density than the molten parts [2], the density measurement across the whole parts show nearly no difference but these particles lead to inhomogeneity and therefore may lead to stress peaks during mechanical strain.

As the mechanical properties of the parts are influenced by both the inner and outer properties, the surface (Figure 4) was characterized on the up and down skin of the parts. It showed that independent from the amount of layers and used ED nearly equal surface roughness developed. Also the top and bottom exhibited a comparable roughness which results from the attached powder particles as Figure 2 showed, that the resulting roughness is mainly influenced by attaching than semi molten particles on the outer surface of the parts and shows no influence of the ED variation.

After characterizing the inner and outer properties of the specimens in dependency of the amount of layers and ED the mechanical properties were determined. Figure 5 shows the young’s modulus and the tensile strength of the specimens. It exhibits that both increase with higher amounts of layers. The increase of the mechanical properties in dependency of the layers is higher than the influence of the used ED. The first 5 layers show higher properties for the increased EDs. This may result from a more homogenous melting of the cross sections, which has been shown in the DPM as well as the morphology of the parts. The resulting unmolten particles in the cross section may have a bigger influence on these small cross sections and therefore lead to earlier failure of the parts. During the first layers, the influence of the rough surface may decrease the
mechanical properties due to notching effects, as the cross section of the parts is not capable of deforming the material in dependency of the mechanical load especially for the tensile strength.

Besides the young’s modulus and the tensile strength, elongation at break is more sensitive to changes in the morphology and inhomogeneity across the specimens. As Figure 6 shows, the elongation depends on the amount of layers as well as the used ED. The specimens with a low number of layers exhibit a low elongation at break, which occurs due to the ratio between the cross section of the specimens and the surface roughness as well as unmolten particles inside the parts, which may result in stress concentration, and therefore earlier breaking. The higher elongation at break for increased ED at lower amounts of layers may occur due to a more homogenous melting of the cross section as well as thicker specimens because of increased melting as shown in Figure 1, which leads to a larger cross-section to deform under the load during testing. With increasing amount of layers for the specimens, the differences in the dimensions decrease and the inhomogeneous melting with unmolten powder particles remaining have a greater influence on the mechanical properties and therefore decrease the elongation at break in comparison to the higher EDs.

Great differences occur during the mechanical testing for the variation in the amount of layers as well as the ED. It shows that with increasing number of layers a yield strength is formed beginning with 5 layers and slightly increasing with a higher amount of layers for the ED of 0.25. In comparison, the higher ED exhibits a small yield strength already for parts with 1 layer and increase with higher amount of layers. The yield strength is higher, broader for the same amount of layers, and lead to a higher deformation of the specimens in comparison to the lower ED, which is influenced by the reduced density and especially the
unmolten particles in the cross section. With the higher ED and amount of layers the highest elongation and strain is reached.

![Figure 7: stress-strain curve for different amount of layers and varying ED]

The differences in the stress-strain curve is reflected in the surfaces of the specimens after breaking. With higher energy, a small ductile breaking zone is formed already for a low amount of layers, which increases with a higher number of layers as exhibited for an ED of 0.40, which develops a ductile breaking behavior across the whole specimens and correlates with the higher stress-strain curve and the formed yield strength of the specimens.

![Figure 8: fractured surface in dependency of the used ED and amount of layers]

**Summary and Outlook**

Producing parts with LS for series application needs the possibility to predict the part properties in the dependency of the process influences to fully enable the process advantages. As it is a well-known factor that LS-parts show a strong dependency of the layer-wise manufacturing of the parts and also the used ED, the
presented results exhibit the interaction of both influences on the resulting part properties. Therefore, parts were produced with a constant layer thickness and varying process parameters as well as different amount of layers. The resulting properties showed a strong dependency of the number of layers in combination with the used ED. It exhibited a reduction of the mechanical properties for small part cross sections, which results from a high influence of the surface and is further reduced by lower EDs due to unmolten particles across the specimens. Higher EDs exhibited increased mechanical properties, especially the elongation at break, which may result from larger cross-sections due to the higher energy input during processing. The stress-strain-curve showed an increase in the yield strength as well as the elongation and the young’s modulus with increasing number of layers. The main influence of the ED is determined in the elongation at break, where the reduction of energy reduces the elongation due to unmolten particles across the parts. The higher ED leads to a more homogenous cross section and therefore increased mechanical properties due to a higher deformation of the cross section during the mechanical testing, which is reflected in a more ductile breaking behavior of the parts.

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