

## POWDER LIFE CYCLE ANALYSES FOR A NEW POLYPROPYLENE LASER SINTERING MATERIAL

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### Abstract

When processing polymers in laser sintering, material aging occurs. The consequences of these aging effects are changes of material and part properties. A reduction of surface quality and the occurrence of orange peel are often found when using a material of too high viscosity. These effects are well known when processing polyamide 12. For alternative materials there is only little knowledge on aging effects. Within the presented study effects of material aging and refreshing for a new developed polypropylene material are analyzed. Viscosity as well as powder flowability are characterized as material properties. Additionally, part properties in different orientations are studied for different aging states as well as refreshing levels. Tensile properties and part density are measured in order to analyze the influence of aging effects on part properties. Furthermore, the influence of different processing parameters on part quality is studied in order to establish fundamental process knowledge for the processing of the new polypropylene material.

### Introduction

Increasing competition, decreasing product life cycles, the wish for customized products and a shortage of resources cause the need for innovative manufacturing techniques for small series production, [1]. Since the transition from Rapid Prototyping to Rapid Manufacturing, Additive Manufacturing offers possibilities for small series production of customized products and an increased freedom of design due to the lack of tools, [2]. The laser sintering of plastic parts is, aside from beam melting of metal parts, one of only two AM-processes which have the capability to be used for Rapid Manufacturing in the near future, [3]. Laser sintered parts are built up layer by layer. The machine produces the parts by repeating four stages for each layer: First, the platform descends by the thickness of one layer. Secondly, powder is spread across the build platform by a leveling roller or coater. Thirdly, the layer is preheated to a temperature close to the material's melting point by a radiant heater. Then, a CO<sub>2</sub>-laser beam melts the powder by tracing the actual cross section line after line using a scanner system. These steps are repeated until the parts are completed, [4]. Due to high process temperatures, the material properties of the unmolten powder material surrounding the parts change. With PA 12, used powder shows an increase of melt's viscosity, a change of melting temperature and a reduction of powder flowability [5, 6]. In laser sintering these effects are called "aging". In order to avoid poor part quality, used powder therefore has to be refreshed with about 30 to 50 % virgin powder before processing it again.

Laser sintering has reached a high technical level within the past two decades. However, there is only a little number of available materials for the process. 90 to 95 % of today produced

laser sintering parts are made from polyamide 11 or polyamide 12 (PA 12), [5, 6]. Alternative materials are seldom used. The main reasons are high prices, a restricted availability, poor mechanical part properties, a more difficult processing or an insufficient understanding of the processing. The problems with other materials result from very complex processing conditions in laser sintering next to high requirements on the material characteristics. Therefore, developing new materials containing good processing behavior, good mechanical properties as well as a sufficient ductility of the parts is very difficult. Within this area, fundamental knowledge was established at the chair for manufacturing technology within the last years. Aim of the presented study is to analyze effects of material aging during laser sintering of a new polypropylene (PP) material.

### **State of the Art**

Technical literature contains several papers describing experiments on new materials. Rietzel analyzed polyoxymethylene (POM), polypropylene (PP), polybutylene terephthalate (PBT) and polyethylene (PE) [7]. Fiedler performed different experiments with PP, [8, 9]. Goodridge and Khalil studied UHMWPE, [10, 11]. Fraunhofer Umsicht and RPM evaluated the processing of PBT, [12, 13]. Drummer developed new PA12/PP- as well as POM/PP-blends for the laser sintering process, [14, 15]. Additionally, some studies consider the development of new powder production processes like the powder spraying using overcritical CO<sub>2</sub> [13], the use of immiscible blends to produce PA 12 powder [16] or the melt emulsification of polypropylene powder [17]. However, none of these studies led to new commercial materials.

Reasons for that are the very complex processing conditions in laser sintering. These result in high requirements on the powder materials as discussed in [7, 18, 19]. Materials should have a wide processing window between the beginning of melting processes when heated up and the start of the crystallization process when cooled down again. Crystallization speed should be as low as possible in order to avoid warpage. The melt of the materials should have a suitable rheology and surface tension in order to form a flat film after laser exposure. The used powders need to have good flowing properties and preferable round particle shape in order to allow good powder spreading during the process. In addition to that, powders should have high bulk density and a suitable powder size distribution for the laser sintering process. Furthermore, the material should have a high absorption of the CO<sub>2</sub> laser beam wavelength of 10.6 μm.

Rietzel studied the thermal and powder properties, the processing conditions as well as the mechanical properties of different materials like PE, POM, PBT or PP. The analyzed PE and PP material was produced by precipitation while the POM and PBT powder was made by cryogenic grinding. The named powders had a relative bulk density fewer than 40 % oftentimes even under 35 % while standard laser sintering PA 12 has values over 40 %. Also the powder flowability showed worse values for the PP, PE and POM materials compared to PA 12 while the flowability of PBT is only a little bit lower than for PA 12. All materials considered were usable to produce parts in the laser sintering process. Only PBT was not tested within the study. However, all tested materials show low values for elongation at break (EAB). For none material more than 6 % EAB in the xy-plane was achieved. [7]

Fiedler published some papers on the development of a polypropylene laser sintering material [8, 9, 20, 21]. He studied different PP homopolymers and copolymers having varying material

properties. While doing so, he preferred materials which were available as powder. Basing on the different studied materials he developed PP compounds containing a homopolymer which was modified with a copolymer. He used contents of the copolymer between 8 and 50 %. With these materials he made laser sintering tests using a DTM Sinterstation 2500. Therefore, he used a grinded powder made from the developed compounds. Like in the study from Rietzel [7] also the results of Fiedler show a brittle part behavior with an elongation at break of under 5 %. Additionally, also tensile strength was found to be significantly under the values of injection molded parts made from the same material.

Reinhardt considered in [22] a commercial glass-filled PP material: Microfol Sinterplast PP. This material was available on the market for some years. However, it was withdrawn from the market due to unknown reasons. Reinhardt studied the effects of different processing parameters on different part properties considering also the part orientation using an EOS Formiga P100. He optimized the parameter settings in order to achieve optimal part properties. However, even for optimized parameters parts show a low elongation at break of under 7 % in x-direction. Kleijnen studied in [23] another commercial laser sintering material from Diamond Plastics PP CP 22. However, mechanical properties are very low even when considering different energy density levels. EAB in x-direction is under 1 % while ultimate tensile strength is under 12 MPa. Schmid analyses in [24] another PP material named iCoPP which shows superior part properties and very high elongation at break. The values are going up to over 400 % for reused material while virgin material shows values between 75 to 150 % EAB. Young's modulus is about 900 MPa while tensile strength is circa 21 MPa. However, material has a high price and a limited availability. Therefore, the use for laser sintering is restricted. The same low-isotacticity material from Trial Corporation was studied in [25, 26]. The authors analyzed different material properties and found that the powder particles are nearly ideal spherical. Additionally, they studied part density and mechanical properties as a function of energy input. High energy input over 0.03 J/mm<sup>2</sup> is necessary to achieve dense parts. A further study from Lexow and Drummer characterize two different LyndellBassell polypropylene materials which were converted to powder by grinding, [27]. They analyze the effect of antistatic agent as well as flow agent on the material properties and the processing of the produced powder materials. Results show good flow times. However, bulk density is under 39 %. The material modified with flow and antistatic agent has good processing conditions while mechanical properties are still low. Tensile strength is only 10 MPa and elongation at break is even under 1.3 %.

As shown, there are several studies on using polypropylene in laser sintering. However, there is only very little knowledge on aging effects when processing PP while there are several studies for PA 12, [28-31]. Fiedler, Schmid and Rietzel found that material aging result in an increase of PP's processing window, [7, 20, 24]. DSC runs showed in all three studies a significant increase especially of melting onset temperature. Only the study of Schmid [24] gives further information on the effects of material aging on powder and part properties. The studied iCoPP material showed even a slight improvement of powder properties after some runs, while no change of melt viscosity was found. Also tensile strength and young's modulus show no effects when parts were built by used powder. In contrast to that, elongation at break significantly increases from 75 to 150 % for virgin powder to over 350 % for used material.

State of the art gives several information on the processing of different experimental and commercial polypropylene laser sintering materials. However, there is only little information on

aging effects when PP is processed in laser sintering. Within the work presented the aging behavior of a new polypropylene powder development is studied. Firstly, the influence of material aging on the most important material and powder properties is analyzed. Secondly, effects of recycled or refreshed powder on part properties are studied.

### Experimental Setup

Due to LS process conditions, i.e. powder bed temperature and inert atmosphere, powder is subjected to a material aging process during the production of parts. The effect of material property changes on processing and part quality was investigated using used and refreshed powder. Within the study, a precommercial version of ROWAK Rolaserit PP was used. The material was processed in virgin, used as well as in refreshed state on an EOS Formiga P100. Refresh rate was chosen to 50 % of virgin powder for the tests. A test build job containing tensile bars, density cubes, powder boxes and some other parts was designed having a build length of 13.5 h and a height of 164 mm, Fig. 1. These setup was built with all three material states, while for virgin powder the job was built twice in order to achieve enough used powder material for a second processing and refreshing with virgin powder. The build job contains four different sets of specimen in order to vary energy input. This was varied between 0.2 and 0.35 J/mm<sup>3</sup> using steps of 0.05 J/mm<sup>3</sup>. The detailed parameter sets are shown in Table 1. Layer thickness was set to 0.1 mm. Processing temperature was optimized before the processes by some test jobs.

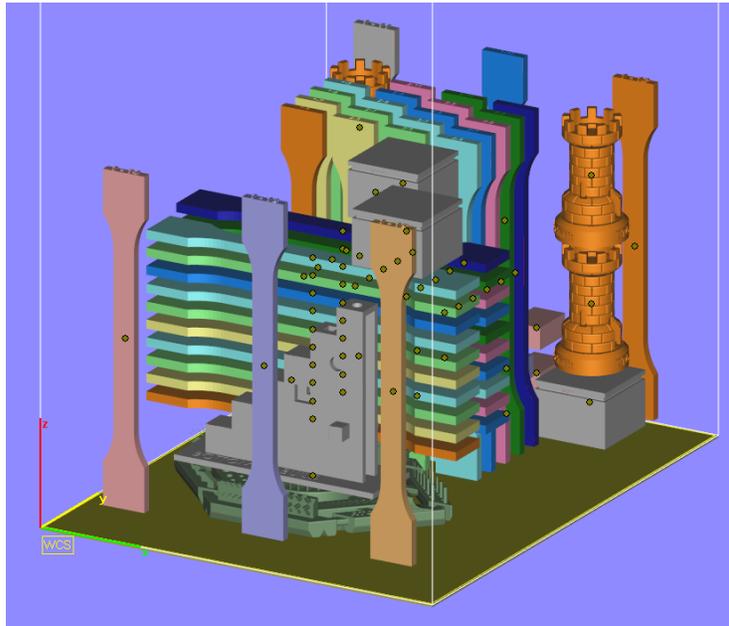


Fig. 1: Layout of the build process

Table 1: Processing parameters

	Laser power [W]	Hatch distance [mm]	Scan speed [mm/s]	Volume energy density [J/mm <sup>3</sup> ]
Parameter 1	16	0.2	4000	0.20
Parameter 2	20	0.2	4000	0.25
Parameter 3	24	0.2	4000	0.30
Parameter 4	24.5	0.2	3500	0.35

The most important material properties in laser sintering were measured for the three materials states. Melt Volume Rate (MVR) measurements were performed according to DIN EN ISO 1133 using a Karg MeltFlow@on. MVR measurement is performed by extrusion of the molten polymer out of a cylinder through a defined capillary die (inner diameter 2.095 mm, length 8 mm) forced by an extrusion piston loaded with a weight of up to 21.6 kg. Measuring conditions were chosen for PP to 230 °C and 2.16 kg.

In addition to the MVR measurements, Hausner ratio, relative bulk density (bulk density/material density) and relative packing density within the powder bed were determined for all powder states in order to obtain information on the powder's flow characteristics. The so-called Hausner ratio describes the ratio between bulk density and tap density. Both values were measured according to DIN EN ISO 60 and DIN EN ISO 61 using a measuring glass with a volume of 100 ml. This was filled with 100 ml of powder and the mass of the sample was weighed using an analytical balance. Thereafter, the powder in the cylinder was compacted by knocking the glass on a board with a frequency of 0.5 Hz. Tap density is determined when no more compaction occurs.

Packing density in the powder bed was measured by producing four hollow cuboids inclosing packed powder. The cuboids were first weighed after removal of the attached powder. Then the cuboids were opened using a scalpel and the enclosed powder was also removed. Afterwards, the empty cuboids were weighed again and the packing density was calculated from the difference in weight and the measured cavity volume.

Tensile specimens according to DIN EN ISO 3167 and density cubes were produced for testing. Specimens for tensile tests were produced in two different part orientations (x-direction and z-direction) with three specimens each. The tensile tests in compliance with DIN EN ISO 527-1 and DIN EN ISO 10350-1 were performed using a Zwick Z020 M (MutiXtense) and a testing speed of 5 mm/min. Part's density was measured for cubes of ca. 20 x 20 x 10 mm size using the Archimedes method according to DIN 1183. Three parts were produced for each parameter set.

## **Results**

Different material properties recommended by the VDI Guideline 3405 Part 1 were measured in order to evaluate aging effects of the processed polypropylene material. Fig. 2 shows the MVR-value as a function of aging state. The results show only little effects compared to PA 12. MVR value decreases only by 3 % when material is reused for laser sintering. Used material refreshed with 50 % virgin powder show the same MVR-value of ca. 146 cm<sup>3</sup>/10 min as the once used material. Even when material is used twice MVR-value decreases only by ca. 6 %. The viscosity of studied polypropylene material is in contrast to PA 12 only less affected by the long process times at high processing temperatures.

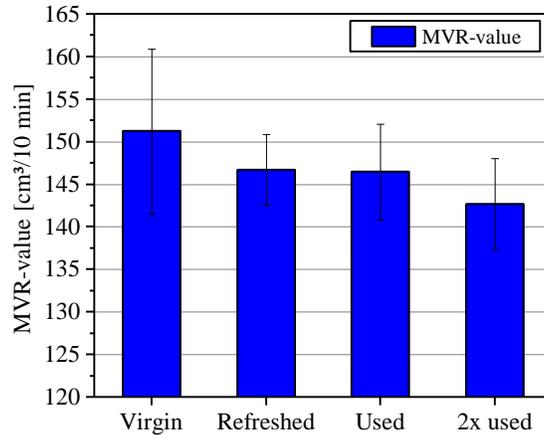


Fig. 2: MVR-value as a function of material state

Besides MVR measurement, the VDI 3405 Part 1 recommends the determination of the Hausner ratio for quality control of powder flowability. Fig. 3 shows the measured Hausner ratio and the relative bulk density of the different powder states. Bulk density of the virgin material is about 45 % which is even higher than for standard PA 12 powder, [28]. The virgin material shows also a low Hausner-ratio of 1.18 which is comparable to most commercial materials. When powder is processed the relative bulk density decreases while Hausner-ratio is not affected when the material was used once. Bulk density is reduced by 6 % when used once and 10 % compared to the virgin state when used twice. The refreshed material has a bulk density which is only 2 % lower than in virgin state. The Hausner-ratio of virgin, refreshed and once used powder is almost identical. However, twice used powder has a significant lower flowability with a Hausner-ratio of 1.24 which is near to the reduced flowability limit of 1.25. When these results are compared to PA 12 [28] it can be found that reduction of flowability is even worse for PA 12. In contrast to the PP, the Hausner-ratio of once used PA 12 is close to 1.25. Therefore, also flowability of the analyzed PP powder is less affected by aging effects, while bulk density is influenced.

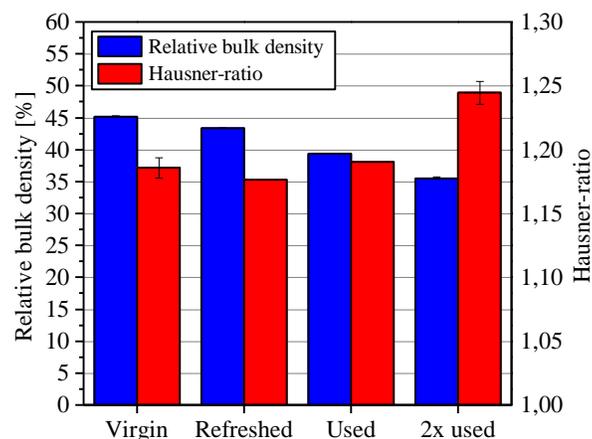


Fig. 3: Relative bulk density and Hausner-ratio as a function of material state

In laser sintering the powder is spread in case of the EOS Formiga by a blade on the build platform, which result in a slight compression of the powder bed during powder spreading. This effect is very essential to achieve high packing densities in the powder bed which enable for high part densities. Therefore the powder packing density in the partcake was determined by building hollow cubes. Fig. 4 shows the measured relative packing densities for the different processed powders. The data for virgin powder shows a very high packing density of 52.5 % which is 10 % higher than found for virgin PA 12 with 42.6 % in [28]. When processing once used powder, packing density is reduced to 45.3 % which is still higher than for PA 12. The refreshed material has a relative packing density between virgin and used powder. Value is about 49.3 %. Therefore, the new developed PP material offers a very good packing behavior. However, aging affects the powder packing in the process.

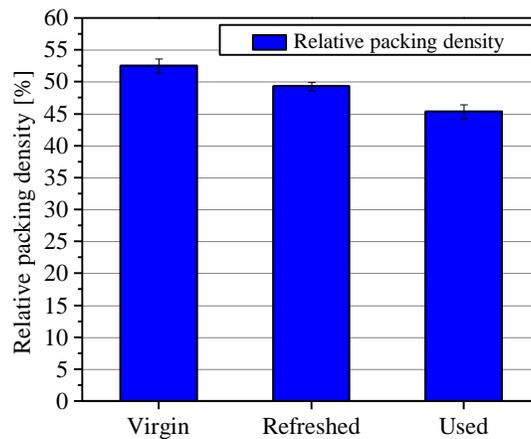


Fig. 4: Relative packing density in the partcake as a function of processed material state

Besides, powder properties also part properties were considered within the study. The effect of material reuse and refreshing on part density as well as tensile properties was analyzed. Additionally, different energy density levels were compared. In contrast to PA 12 no orange peel was found during the experiments.

Fig. 5 gives the measured relative density values in dependence of used powder quality and energy input. Results show differences in the effects between the different energy densities. For the lowest energy input of  $0.2 \text{ J/mm}^3$  a visible effect of material aging is considered. A density of nearly 98 % is achieved for the virgin material at that energy level. However, density is reduced by 1.3 % when used material is processed. Even for refreshed material a slight decrease is found. For none of the other energy densities the aging effect has a similar size. Therefore, at a suitable energy density level part density is only little affected by aging effects. For energy densities over  $0.25 \text{ J/mm}^3$  part density show in all cases and independent from the material state values between 97.5 and 98.3 % which are very similar to values achieved for PA 12.

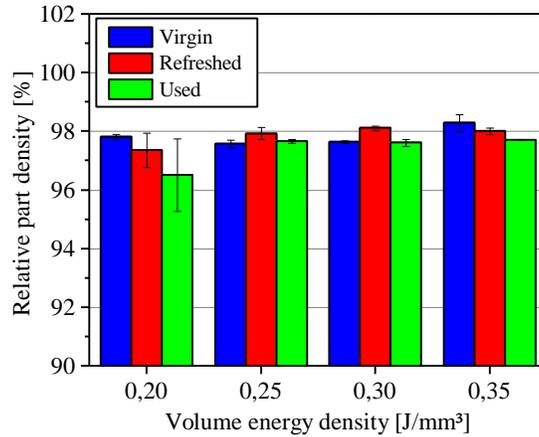


Fig. 5: Relative part density as a function of energy density and powder state

In addition to part's density, tensile properties in x- and z-direction were evaluated. Fig. 6 to Fig. 8 show the results of the tensile tests for the different powder states.

Fig. 6a. summarizes the results of young's modulus values measured for tensile bars oriented in x-direction, while Fig. 6b. shows the values for z-direction. For the x-direction similar effects like for part density are found. Again lowest energy density show highest effect of powder state and a significant reduction of young's modulus by 6 % when used powder is processed. In all other cases young's modulus varies within the small range from 825 to 875 MPa with no obvious effect of material state. The found trend for the lowest energy density enlarges when the young's modulus in building direction is analyzed, Fig 6b. The values in z-direction at an energy density of 0.2 J/mm³ show a bigger drop by ca. 20 % when processing of virgin and used powder is compared. However, even for the refreshed powder a drop of young's modulus by 9 % is measured. A drop of properties for the used powder is also found at an energy density level of 0.25 J/mm³. Young's modulus drop in this case by 7 % while no reduction is determined for the refreshed material. For higher energy input, young's modulus is only little affected by the material state. Values vary between 800 and 850 MPa and show therefore also little deviation to the x-direction. Same values are found at 0.25 J/mm³ for the virgin and refreshed powder state.

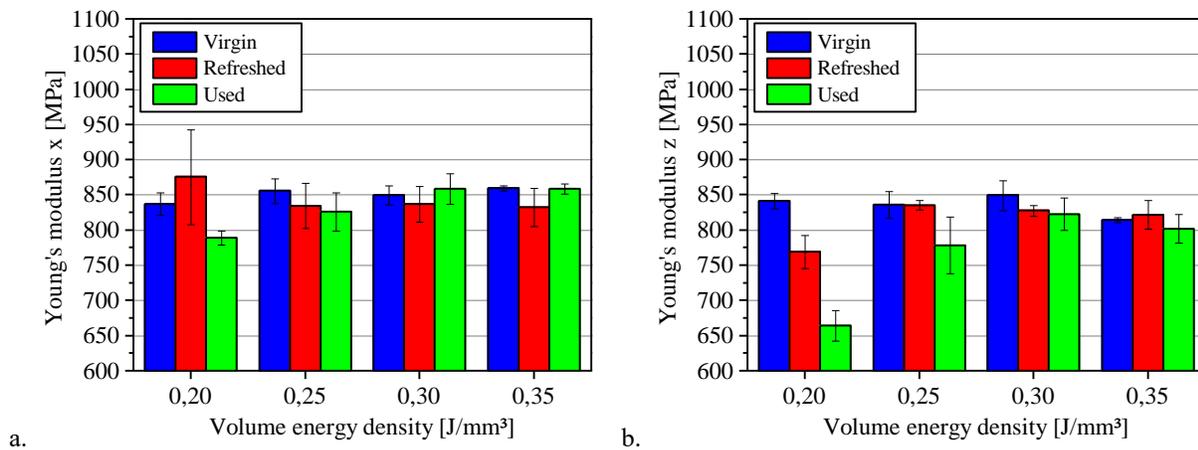


Fig. 6: Young's modulus as a function of energy density and powder state; a. x-direction, b. z-direction

Fig. 7a. and 7b. show the results measured for tensile strength in x-direction respectively in z-direction. The correlations on energy density and material state are similar as found for young's modulus. In x-direction a significant effect with a drop by 12 % is measured for energy input of 0.2 J/mm<sup>3</sup>. Additionally, the refreshed state shows a slight decrease. Values between 17 and 18 MPa are achieved for the x-direction for energy densities between 0.25 and 0.35 J/mm<sup>3</sup>. The higher measured values are found for use of virgin and refreshed powder while the lower value results from the used powder. In z-direction effect at 0.2 J/mm<sup>3</sup> is even worse. The use of used powder leads to a drop of tensile strength by nearly 40 %. However, even the virgin state results in worse values compared to higher energy densities. In case of tensile strength in z-direction also for an energy input of 0.25 J/mm<sup>3</sup> lower values of maximum 15 MPa are achieved for the virgin state. Highest tensile strength values in z-direction are found for 0.30 and 0.35 J/mm<sup>3</sup> ranging from 15 to 17 MPa. However, also for these energy density levels the reuse of powder lead to a decrease of tensile strength by up to 12 %. Compared to young's modulus additionally higher anisotropy of values is found. Percentage deviation between the orientations is in the optimal energy density range about 6 % for the virgin state and increases to 12 % for the used state. However, deviations are similar to values found for PA 12, [32].

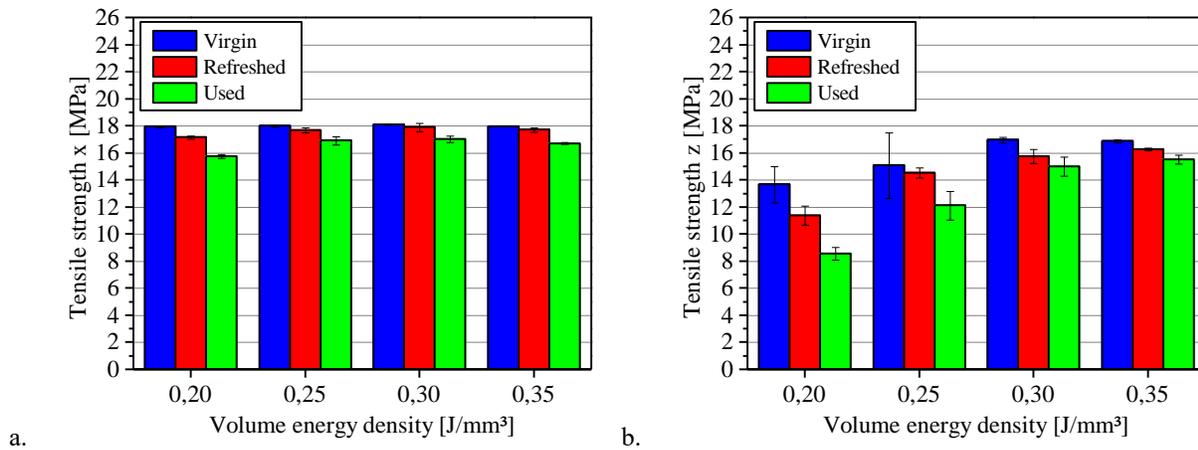


Fig. 7: Tensile strength as a function of energy density and powder state; a. x-direction, b. z-direction

Elongation at break (EAB) shows highest influence of material state. The measured values are shown in Fig. 8a. for the x-direction and Fig. 8b. for the z-direction as a function of the energy density and the material state. For the virgin state values for elongation of break in x-direction lie between 11.5 and 13.3 %. Same range of values is found for refreshed powder in case of energy densities over 0.25 J/mm<sup>3</sup> while EAB for 0.2 J/mm<sup>3</sup> is only 9 %. A reduction of EAB by 20 to 29 % occurs for all energy inputs when used powder is processed. Values in x-direction then vary between 8 and 10 %. The highest value is achieved for an energy density of 0.35 J/mm<sup>3</sup>. The EAB measured in z-direction shows other correlations. The values significantly increase with an increase of energy density. Therefore, energy input has a strong influence on the layer to layer bonding of the processed PP material. Lowest values are found for 0.2 J/mm<sup>3</sup> with an EAB of 2 to 3.2 % depending on the powder state. Reduction of EAB due to aging is in this case about 36 %. Highest values are achieved for 0.35 J/mm<sup>3</sup> with 6.3 to 8.1 % while the values of the other two energy inputs lay between both ranges. In case of 0.35 J/mm<sup>3</sup> the aging based

decrease of EAB is reduced to only 15 % which is half the value found for 0.2 J/mm<sup>3</sup>. Specimen produced by used and refreshed powder show independent from the orientation and the energy input a significant reduction of EAB compared to the virgin state. Therefore, EAB is the most sensitive value regarding aging effects. One reason for that might be the reduction of packing density in the powder bed. This may lead to the observed reduction when processing especially used material. However, EAB values found for this new PP material are very similar to values found for PA 12. Especially in z-direction PA 12 values are often not better than the data reported in this study for PP. For the two highest energy densities the deviation between the orientations show also similar values as found for PA 12, compare [32]. Percentage deviation varies between 28 and 50 %.

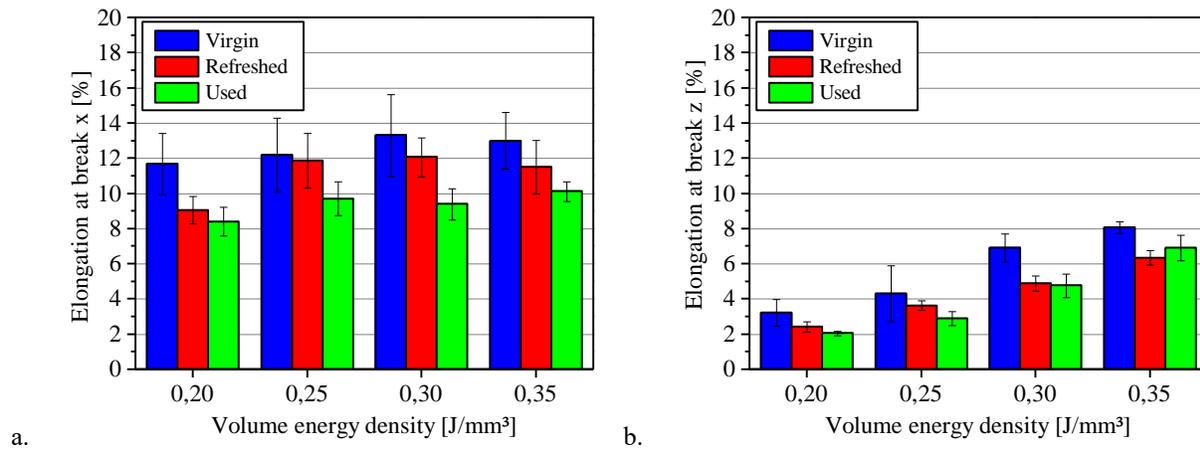


Fig. 8: Elongation at break as a function of energy density and powder state; a. x-direction, b. z-direction

## Conclusions

Within this study the influence of material aging on material and part properties of a new developed polypropylene laser sintering material was studied. This is one of the first studies on material aging effect in laser sintering which do not consider PA 12 but an alternative material.

Several material and part properties were studied within the performed analyses. Materials viscosity as well as Hausner-ratio are only little affected by aging effects. On the other hand bulk and packing density is reduced by reusing the material. Results show that for robust processing an energy density of at least 0.3 J/mm<sup>3</sup> should be used. Then aging has only little effect on the measured values of part density, young's modulus and tensile strength. However, elongation at break is always influenced by aging effects and shows for all studied energy density levels a significant reduction of values due to the observed reduction in packing density. In summary, the new developed material show very good powder properties and also good part properties especially high values for elongation at break. These are comparable to values of PA 12 and much higher than for most other commercial materials which are not PA 11 and PA 12.

Future work should consider the effect of aging on further material properties like particle size or melting behavior. Additionally, processing conditions of the studied material should be

improved in order to commercialize it in near future. Aims are especially the establishment of better reusability. Therefore, the packing behavior of used material should be improved.

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