MECHANICAL AND THERMAL PROPERTIES OF FDM PARTS MANUFACTURED WITH POLYAMIDE 12

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Abstract

Fused Deposition Modeling (FDM) is an Additive Manufacturing (AM) technology which is used for prototypes, single-part production and also small batch productions. For use as a final product, it is important that the parts have good mechanical properties, high dimensional accuracy and smooth surfaces. The knowledge of the mechanical properties is very important for the design engineer when it comes to the component design. End-use products out of the FDM process have to resist applied forces. In this paper, investigations were conducted with the polymer Polyamide 12 (FDM Nylon 12) from Stratasys Inc. This polymer can be processed with three different tip sizes resulting in different layer thicknesses from 178 µm to 330 µm. Thus, the mechanical properties were determined for these layer thicknesses and for different orientations on the build platform. In addition to the mechanical properties the thermal properties (e.g. with a DSC analysis) are also investigated.

Introduction

Fused Deposition Modeling (FDM) is an Additive Manufacturing (AM) technology which is used for prototypes, single-part-production and also small batch productions. It is one of the most used technologies in the AM market. Due to the layer by layer manufacturing, even very complex components can be built and no forming tool is needed [1]. The information for every layer is generated from a 3D CAD dataset. Therefore, only a few steps are necessary for the realization of a digital model to a physical component. For use as a final product, it is important that the parts have good mechanical properties, smooth surfaces and a high dimensional accuracy.

The FDM process is shown in Figure 1. The strand shaped raw material is heated up in the FDM head by two extrusion nozzles and then the molten material is deposited defined in X- and Y-direction on the build platform. After the completion of one layer, the building platform is lowered by one layer thickness and the next layer will be produced. The final part consists of model material and the support material is necessary to support overhangs and cavities. This support material can be removed in an additional post-processing step by breaking away or dissolving in an alkaline bath. The deposited material bonds with the layer beneath due to thermal fusion. This results in a strong and permanent bond between the layers [2].

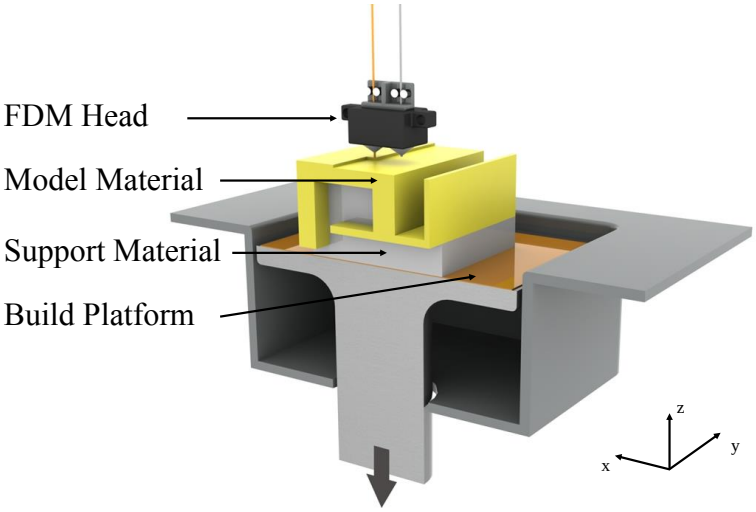

Figure 1: Fused Deposition Modeling (FDM) process

First of all the contour of one layer is deposited and then the raster fill of the inner geometry is completed. By default the filling is done at an angle of 45 ° to the X-axis. In the next layer, the raster angle is changing by 90 ° so that there is an alternating raster fill for the parts. As known from previous publications, there is an anisotropic material behavior for FDM components [2][3]. A filling of the layer with an angle of 45 ° should counteract an even stronger anisotropy. By using the Insight software from the manufacturer Stratasys, it is possible to change the filling strategy of every layer.

In this paper, mechanical and thermal properties of FDM components manufactured with the polymer Polyamide 12 (PA 12) are investigated. This material is distributed by Stratasys Inc. and the trade name for this material is FDM Nylon 12. Polyamide 12 is an engineering plastic and it is the first semi-crystalline polymer for the FDM process. The characteristics of this material are a low density of 1 g/cm³ and a high flexibility. Furthermore, it shows high fatigue and high impact resistance. PA 12 has a strong chemical resistance and the lowest water absorption of all polyamides (max. 1.5 %) [4][5]. Application areas include the industries of automotive, aerospace and consumer goods. All test specimens were manufactured with a Fortus 400mc from Stratasys. PA 12 can be processed with three different layer thicknesses, which are realized with different tip sizes. These tip sizes are T12, T16 and T20 and the number indicates the hole diameter in hundredths of an inch (T12 = diameter of 0.012 inch). With the use of these tip sizes layer thicknesses of 178 µm, 254 µm and 330 µm are realized [5].

Mechanical Properties

The investigations of the mechanical properties (tensile, bending and compressive) were carried out with the universal testing system Instron 5569 with a load cell of 5 kN at an ambient temperature of 23 °C and a relative humidity of 50 %. All test specimens were manufactured with the standard parameter of Insight 9.1 and the mechanical tests were done after a conditioning of the specimens at a minimum of 72 hours. The soluble support structures were removed in a warm water bath (60 °C) with an alkaline solution for a minimum of 4 h. As shown in Figure 2 the mechanical properties will be determined for three different build directions: X- (on its edge), Y- (flat lying) and Z-direction (upright). For the tensile tests eight specimens and for the bending

tests nine specimens in each direction and slice height were manufactured. The specimens were equally distributed over the entire build platform to avoid possible influences of the temperature distribution and belt tension.

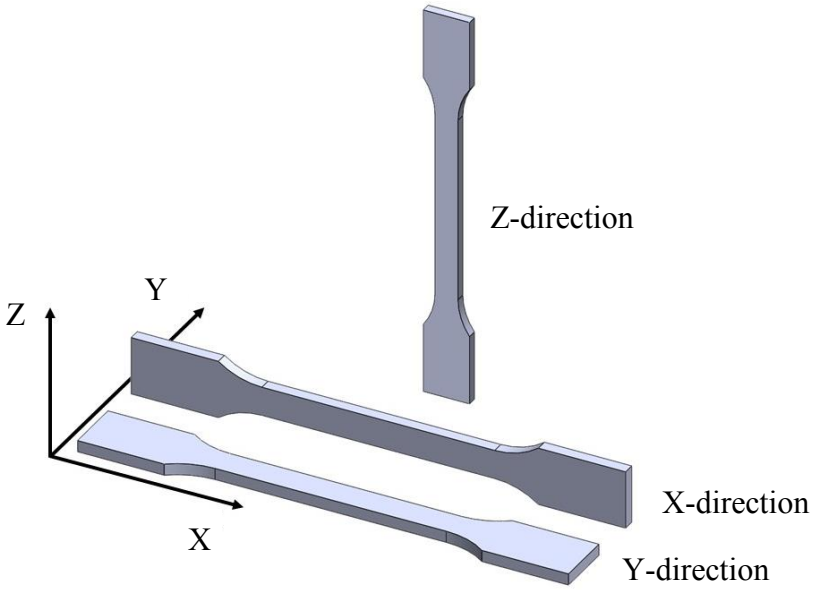

Figure 2: Illustration of different build directions of the test specimens for mechanical tests

For upright standing specimen, a support structure is necessary because of the low ratio of base area to build height. This support structure should prevent tumbling of the specimen. In the first attempts to build upright specimen, it became visible that the surface was very bad, where contact with the support material (Stratasys SR-110) existed. Thus alternative support structures were necessary. In Figure 3, the different build-up styles are shown: A) is the standard support structure of Insight 9.1 with support style “surround”. Usually, the support extends to the upper end of the tensile bar but the last 24 mm were removed manually. The second build-up style B) is called “boxed”. In this case two boxes surround the tensile bar with a distance of 0.26 mm. The two boxes are built with the “sparse” style and with model material in order to save material and build time. The last build-up style is called “stabilized” C) because of the use of the “Stabilize wall” function of Insight 9.1. The stabilizing structure (compare Figure 3 – C) must be set at two points of the component and the supporting width can be modified. The structure consists of model material and contacts the tensile bar only every 10th layer. Another possibility to avoid support structures is to use bending bars for the tensile test with the dimensions 80 x 10 x 4 mm (L x W x H). These bars can be built upright without support. This build-up style is called “bars”. Furthermore, a sacrificial tower was used. This tower is the first part in each layer, where material will be deposited. This aims to increase the quality of manufactured components.

For the slice height of 254 µm the tensile specimens were also tempered. Tempering describes a process in which the specimens are subjected to a subsequent heat treatment. The heat treatment is done at 135 °C for 180 minutes in a convection oven. The temperature will be increased by 3 °C/min. Tempering should lead to a degradation of internal stress and a post-crystallization. Thus, the heat distortion temperature (HDT) will be increased [6]. This paper aims to clarify what influence the tempering has on the mechanical properties.

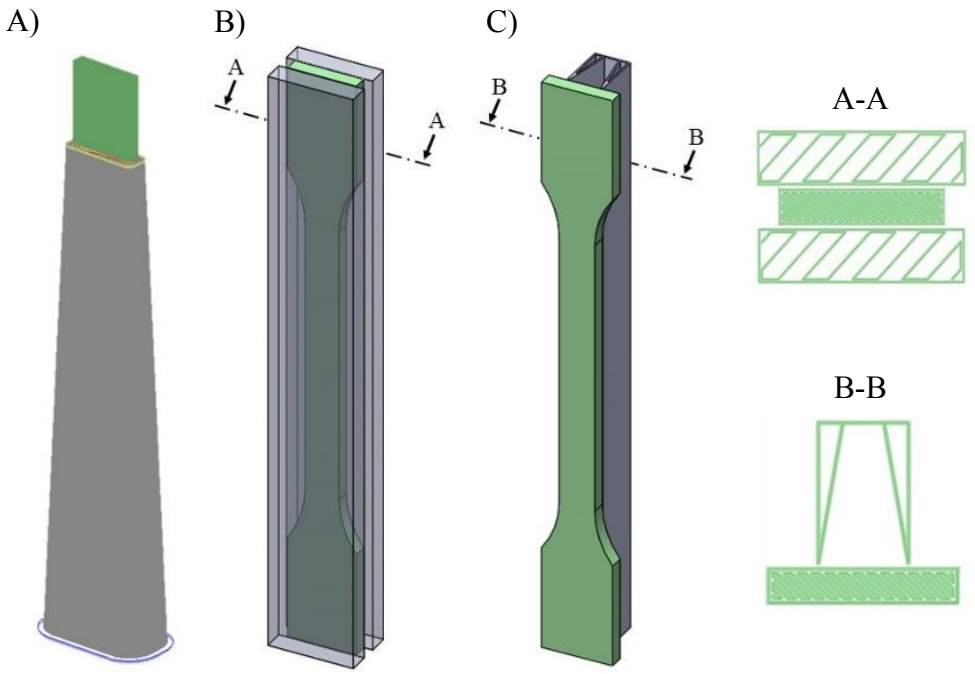

Figure 3: Different build-up styles for upright orientated specimens: A) surround B) boxed C) stabilized

Tensile Strength

The tensile strength for Polyamide 12 specimens was determined in accordance with DIN EN ISO 527 and referring to ASTM D638. The difference of these two testing standards is the geometry of the test specimen and the velocity of the tensile test. The velocity for the DIN standard is 1 mm/min for the determination of the Young's Modulus and 50 mm/min for the determination of the elongation at break. The ASTM standard has the same velocity for the Young's Modulus and for the elongation at break with 5 mm/min (~ 0.2 "/min).

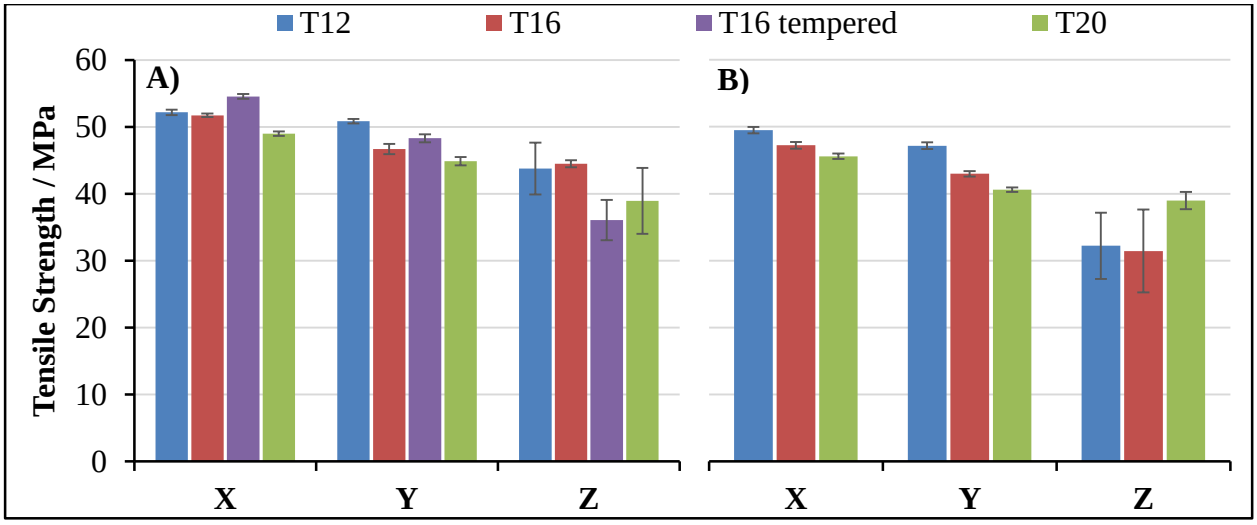

Figure 4: Maximum tensile strength for different build directions and layer thicknesses – A) DIN EN ISO 527 and B) ASTM D638

In Figure 4, the maximum tensile strength for different layer thicknesses and build directions (compare Figure 2) is shown. In the left part of the figure (A) the results of the measurement according to DIN standard are presented. The results which have been determined in accordance with the ASTM standard, can be seen on the right side (B). The specimens which are built in X-direction show the highest strength properties (DIN standard). Slightly lower values (on average about 10 %, except T12) can be recognized for the Y-direction. The lowest tensile strength can be identified for the upright built specimen (Z-direction). Thus, PA 12 has an FDM typical anisotropy. However, this is not so much distinctive like Ultem 9085 [3]. The results in Z-direction are 14 to 20 % lower than in X-direction. The values which have been determined in reference to the ASTM standard are consistently slightly below the DIN values. The observed trends for the DIN results are also visible for the ASTM results. Furthermore, it is remarkable that higher values in X- and Y-direction can be achieved with a heat treatment. As already mentioned, the build-up in Z-direction has been realized with various styles (compare Figure 3). For the evaluation in accordance with DIN standard, the highest values were used which have been determined in the investigation of the different build-up styles.

The measurement results of the maximum tensile strength in Z-direction with different build-up styles are shown in Figure 5. The lowest tensile strength is recorded for the surround support style (standard) and the T12 tip. An increase of the tensile strength up to 60 to 70 % is realized with the build-up style “boxed” and “bars”. The maximum tensile strength is 46.6 MPa for the style “bars”. Furthermore, the standard deviation is reduced. Also for the T16 tip there is an increase in strength by the styles “stabilized” and “boxed” recognizable. The increase amounts to 29 % to a value of about 44.5 MPa.

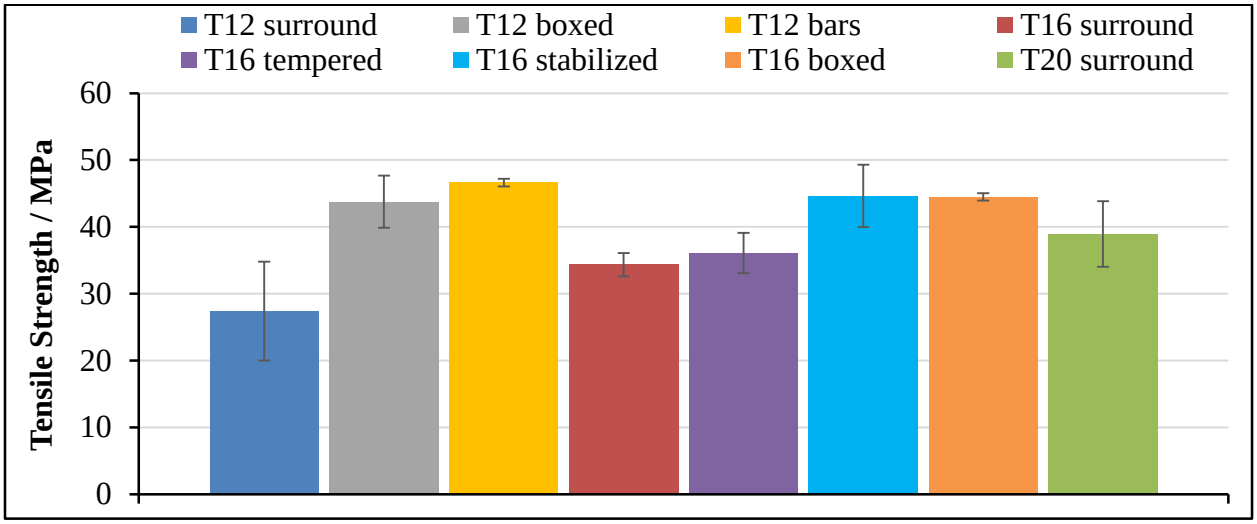

Figure 5: Ultimate tensile strength for the Z-direction with various build-up styles and different layer thicknesses – DIN EN ISO 527

The results for Young’s Modulus (according to DIN standard) show that there is no anisotropy for the three different layer thicknesses (see Figure 6 – A). For the T12 tip, the values are at one similar level with about 1320 MPa. The averages for the T16 and T20 tip are slightly above this value. The heat treated specimens of the T16 tip achieve a higher value in Y- and Z-direction than the non-treated specimens. The maximum value for the Young’s Modulus with 1535 MPa is measured with a T16 tip in X-direction, but there is also a high standard deviation

identifiable. In determining the Young's Modulus in reference to the ASTM standard (Figure 6 – B), no differences for the T12 and T16 tip are obtained. The average value amounts about 1420 MPa. Thus, the values are about 100 MPa higher than the DIN standard. For the T20 tip, the X- and Z-direction are at a value of 1310 MPa. The flat built specimens with a layer thickness of 330 µm achieve a higher value of 1450 MPa.

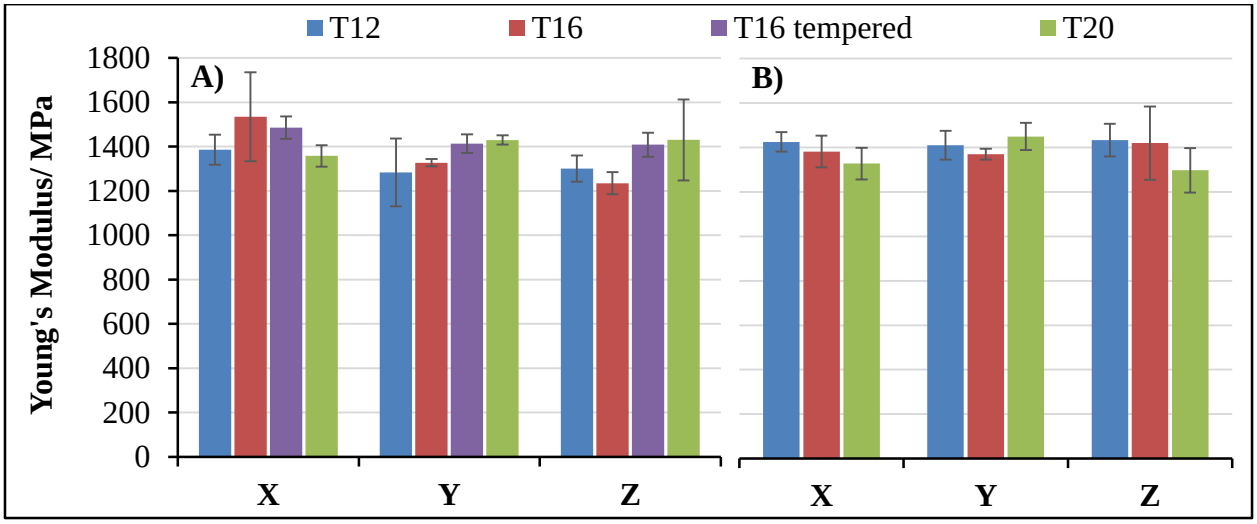

Figure 6: Young's Modulus for different build directions and layer thicknesses – A) DIN EN ISO 527 and B) ASTM D638

The biggest differences between the two testing standards can be seen in the elongation at break (see Figure 7). Due to the 10 times higher testing velocity there are lower values for the DIN standard with a maximum of 16.6 % elongation at break (Figure 7 – A). Conspicuous are the high standard deviations that indicate a high variance of the measured values. Equally significant is the low elongation at break for the Z-direction (on average only 3.2 %). In X-direction the measured values are at about 15 % (except T12). The results in Y-direction are about 13.7 %, the low value for the T16 tip may be considered as an outlier. In determining the elongation at break referring to the ASTM standard (Figure 7 – B), very high values were measured for the T16 and especially the T20 tip. The highest elongation at break averages 88 % in X-direction. The measurement results in Y- and Z-direction are equivalent to the DIN results. The very high elongation at break can be explained by the deposition of the strands. Sideways manufactured specimens have many strands which lay in the load direction. Furthermore, the low testing velocity leads to higher elongations because cracks cannot grow as fast as at higher velocity.

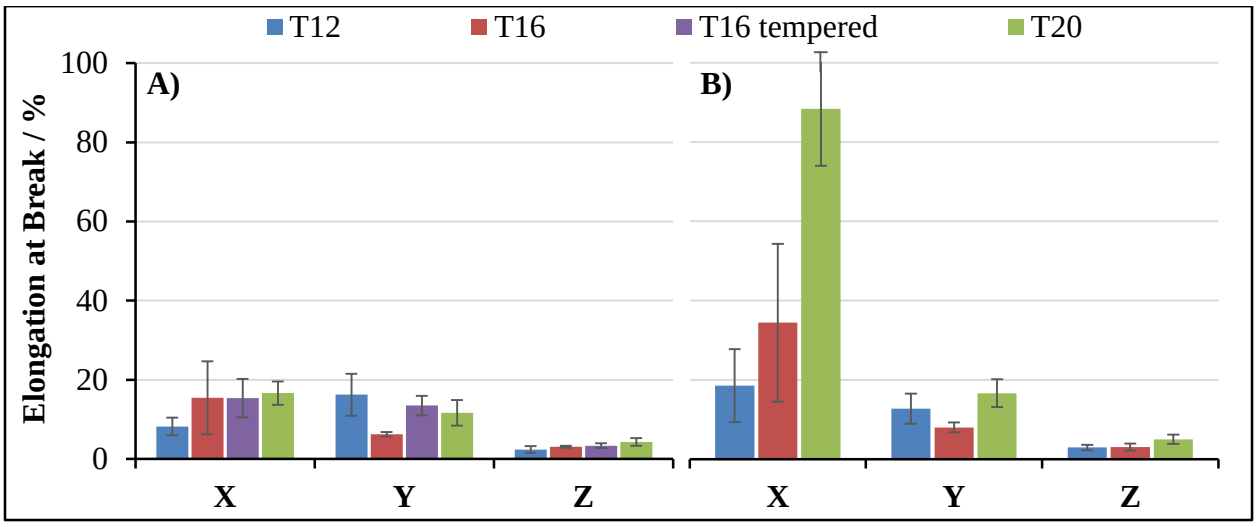

Figure 7: Elongation at break for different build directions and layer thicknesses – A) DIN EN ISO 527 and B) ASTM D638

In Figure 8, a comparison of different build-up styles with focus on the elongation at break is made. The results were determined in accordance with DIN standard. By default the build-up is made with support material (surround). However, this leads to rough and defective surfaces. Those surface defects act as starting points for cracks that lead to early failure and therefore to breakage of the specimen. The result is a lower elongation at break than the styles "boxed", "stabilized" and "bars". For the layer thickness of 178 µm, a doubling of the elongation at break was reached by the "boxed" style. In the tensile test with simple bars the highest values in Z-direction with 6.5 % could be achieved. One possible explanation is that here no notch effect occurs due to taper of the structure's cross-section. Through the use of the "stabilize wall"-function an elongation at break of 6.5 % can be achieved for the T16 tip as well. However, a higher standard deviation is noticeable. The elongation at break can possibly be further increased if the interval of the contact points between the support structure and component is increased.

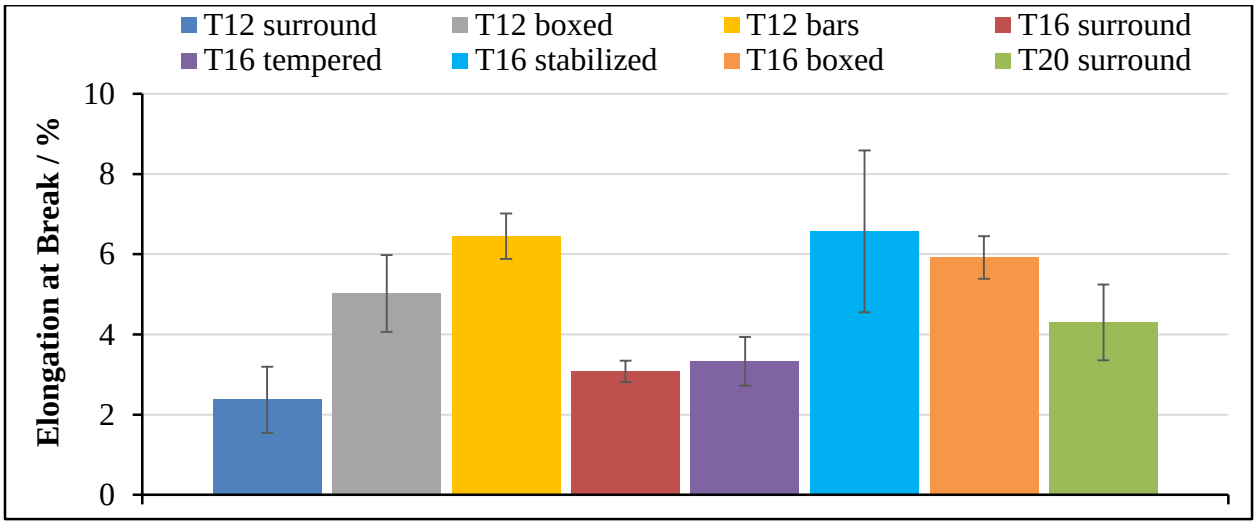

Figure 8 Elongation at break for specimens in Z-direction with various build-up styles and different layer thicknesses – DIN EN ISO 527

The reasons for the different elongations at break can be explained on the basis of break patterns (see Figure 9). For specimens in Z-direction, the force is applied perpendicular to the layers. Hence the elongation at break is critically dependent on the bonding between the layers. In Figure 9 – C, it is visible that the breakage is exactly between two layers. For the specimens in X- and Y-direction, the force is applied parallel to the deposited filaments. Therefore, the material properties of the polymer are more relevant for the elongation at break. The fracture occurs at an angle across the test specimen (compare Figure 9 – A & B). The specimens in Y-direction are built rotated by 90 ° to the X-direction, hence the break pattern is also rotated by 90 °.

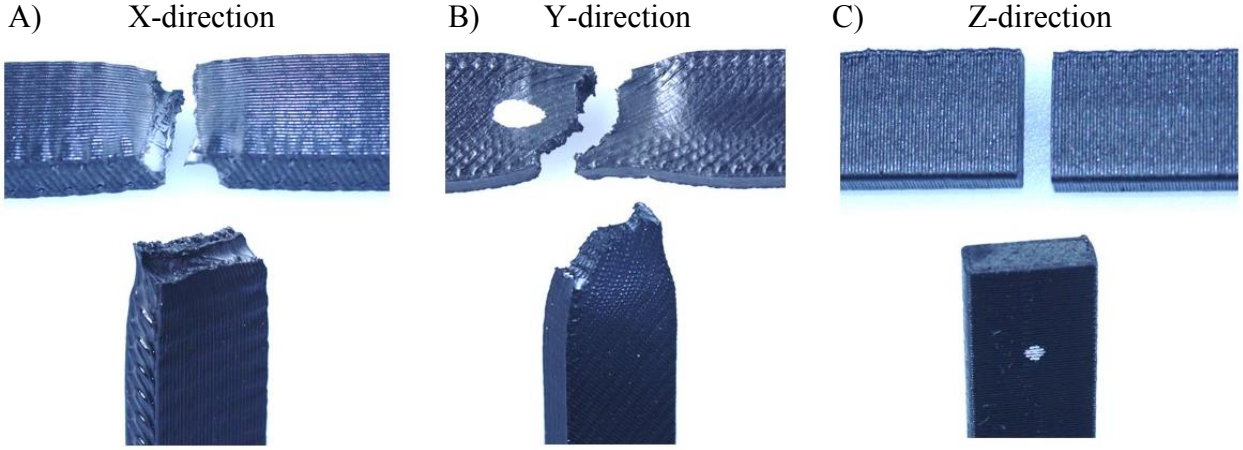

Figure 9 Break patterns of tensile specimens for different build directions – T12

Flexural Strength

The bending test is used to analyze the mechanical and deformation properties of specimens by applying a three point load. The tests were conducted according to DIN EN ISO 178. The velocity was 2 mm/min and a load was applied to the specimens until they broke. The span distance was 64 mm and the radius of the loading noses and the supports were 5 mm. The used test specimen is a bar with the dimension 80 x 10 x 4 mm. The bar graph in Figure 10 illustrates the results for the flexural strength and the line graph shows the results for the flexural modulus.

The maximum flexural strength for all build directions is very identical for the tip sizes T12 and T16. The highest values are measured for the X-direction and amount 67 MPa. The measured values in Y-direction are only slightly lower. There is no real anisotropy as in the tensile test. Thus, the values in Z-direction are not significant lower than the other build directions (61.5 MPa for T12 and T16). In comparison to this, the results for the T20 tip are slightly lower in X-, Y- and Z-direction. It is obvious that the flexural strength for the T16 tip can be enhanced by a heat treatment. Thus, the maximum flexural strength in X-direction is reached with 72.9 MPa for the annealed specimens. The heat treatment has also a positive influence on the flexural modulus. The tempered specimens show the highest stiffness for all build directions. In X-direction the maximum value amounts 1425 MPa. Identical to the values of flexural strength there are no differences between the T12 and T16 tip size. The T20 tip shows the lowest values for the flexural modulus. It is also identifiable that the stiffness of X- decreases to Z-direction. The standard deviation is very low for all measured values.

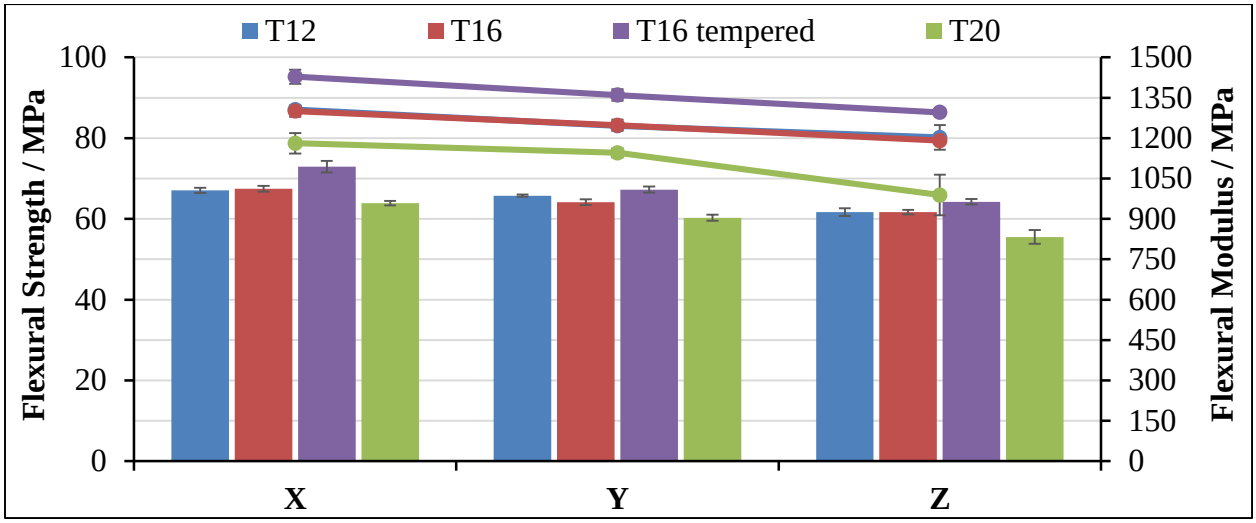

Figure 10 Results of the bending test as a function of the build direction and layer thickness – DIN EN ISO 178

Compressive Strength

In real applications, components are not only stressed at tensile or bending, but also on pressure. By using the compression test according to DIN EN ISO 604 the strength and stiffness under pressure load for PA 12 specimens can be determined. For the investigation of these characteristic values two different test specimen are necessary. The specimen for the determination of the compressive modulus has the dimensions 50 x 10 x 4 mm and for the compressive strength 10 x 10 x 4 mm. The test specimens are pressed together by defined punch at a velocity of 1 mm/min. Due to the cuboid shape, the strength values in X- and Y-direction do not distinguish. In Figure 11, the compressive strength is plotted in a line graph and the compressive modulus as a bar graph. The resulting compressive strength is independent of the build direction. Furthermore, it is visible that the strength values are identical for the layer thicknesses 178 and 254 µm. The compressive strength amounts about 58 MPa. Only the largest layer thickness of 330 µm is slightly below those values with an average of about 53.8 MPa. For the compressive modulus the highest measured values are recognizable in Z-direction. The maximum is visible for the T16 tip with 940 MPa. The lowest values are recorded for the X-direction.

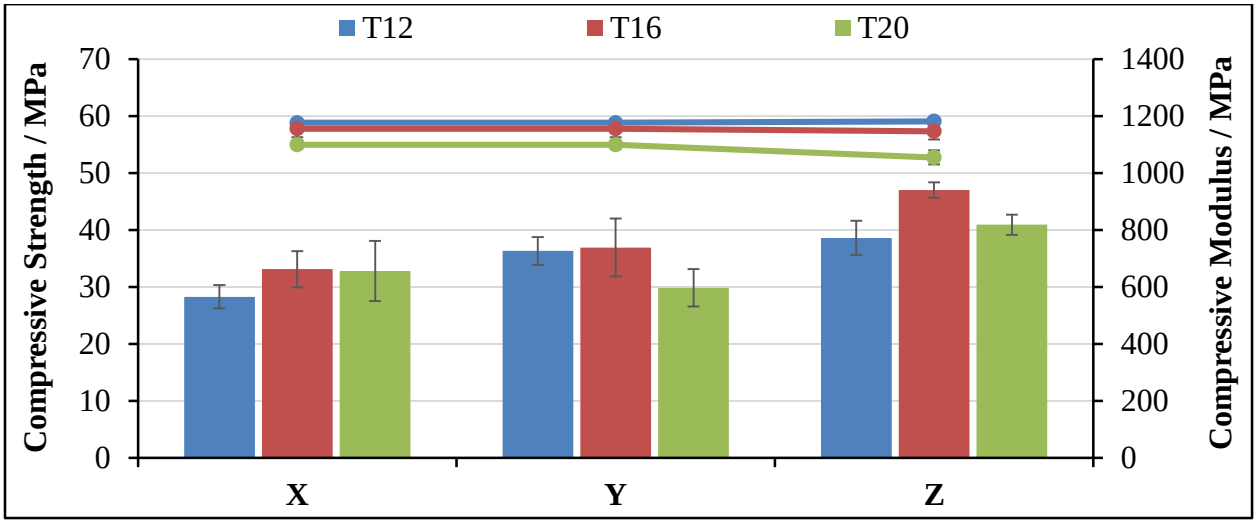

Figure 11 Results of the compression test as a function of the build direction and layer thickness – DIN EN ISO 604

Thermal Properties

In this subchapter, the thermal properties of Polyamide 12 for the FDM process are investigated. From investigations on PA 12 for the AM process Laser Sintering (LS) is known, that there is an aging of the material due to thermal load in the process. This implies an increase of molecular chain length of the powdery material PA2200. This effect is characterized by a reduced Melt Volume Rate (MVR) [7]. With the determination of the MVR it should be clarified if similar effects can be observed for the PA 12 in FDM process as well. The Differential Scanning Calorimetry (DSC) will be used to analyze the effect of the heat treatment within the polymer. Furthermore, the DSC provides knowledge about the melting behavior of PA 12. This can be used to determine chemical and physical properties including but not limited to glass transition temperature (TG) or melting point of the polymer.

Melt Volume Rate

The Melt Volume Rate (MVR) is determined according to DIN EN ISO 1133 and is used to characterize the flow behavior of polymers. This characteristic value describes a volume flow which flows through a defined capillary under certain environmental conditions. The MVR is inversely proportional to the viscosity of the polymer [7]. Thus, the molecular weight can be calculated as well. If the density of the material is known, the MVR can be converted into the MFI (g/10 min). The PA 12 has to be dried before the MVR can be determined. The material is dried at 105 °C for 30 minutes in a convection oven. This is important to remove water out of the material to get a reliable MVR. Subsequently, the PA 12 is heated up to 235 °C and 275 °C for 300 s in the testing device. The material will be loaded with a test weight of 5 kg. The temperature of 235 °C is chosen to achieve a comparison to the MVR in the LS process. The increased temperature of 275 °C is selected because the material is extruded at a heating element temperature of 355 °C in the FDM process. Both temperatures are permissible according to the standard. The test weight generates a pressure which is necessary for the polymer to flow through the capillary. This test is done with the machine “Mflow” by Zwick / Roell. Finally, the MVR can be calculated by using this formula:

$$MVR = \frac{A \cdot 600 \cdot L}{t} \quad (1)$$

By measuring the piston travel L, time t and with the sectional cross area A of the piston, the MVR can be calculated in the unit of cm³/10 min. The values which are shown in Figure 12 are resulting from the average of three individual measurements. In addition to the virgin material, which has been extracted out of the strand material, processed PA 12 is used. This material is removed from the T16 tensile specimens. Furthermore, tempered material is examined to determine the influence of the heat treatment on the molecular weight.

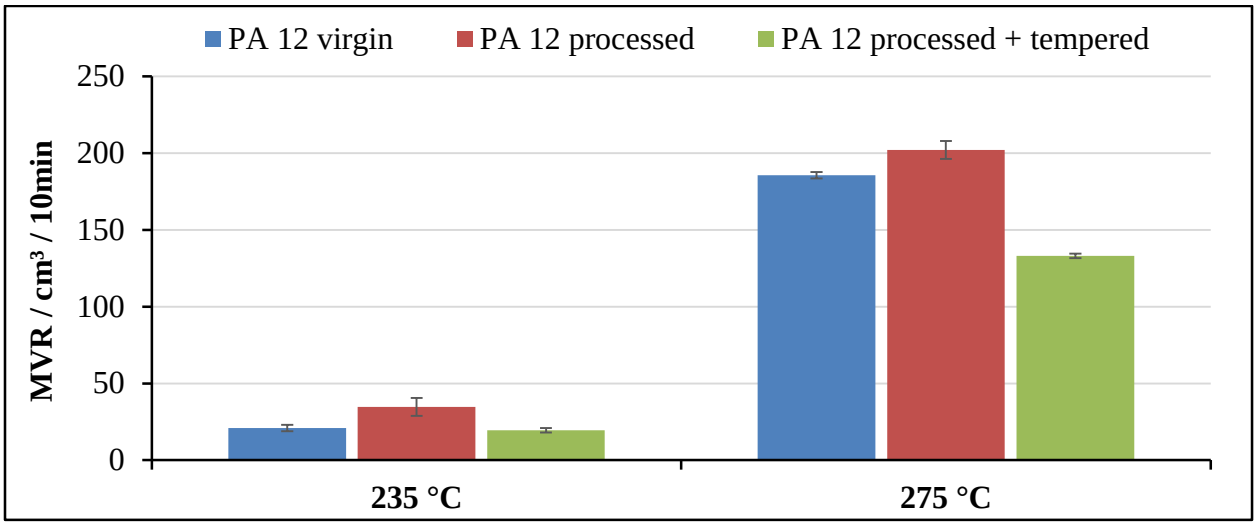

Figure 12 Melt Volume Rate (MVR) of PA 12 at different temperatures after different processing steps – DIN EN ISO 1133

In Figure 12, the results for the MVR measurement for both temperatures can be seen. The virgin material has an MVR of 21 cm³/10 min at 235 °C and for 5 kg load. At the raised temperature of 275 °C the flowability increases and reaches a value of 185.5 cm³/10 min. This is a typical behavior for shear thinning materials that show at elevated temperature a decreasing viscosity. The rad bar indicates the MVR for processed material. It is obvious that the MVR increases to 34.7 cm³/10 min (235 °C) and 202 cm³/10 min. The higher values can be explained by a degradation and damage to the molecules, resulting in shorter molecular chains. This degradation of the molecular chain length is indicated by the thermal load in the FDM process. This results in fewer entanglements and the material can flow faster through the capillary. The viscosity changes and the material is thinner. The subsequent heat treatment is reflected in a reduced MVR and therefore in a higher viscosity. The measured value amounts 19.4 cm³/10 min (235 °C) respectively 133 cm³/10 min (275 °C) which is below the initial value for virgin material. The heat treatment might lead to longer molecular chains and / or to a reorganization of the semi-crystalline sections. More specific information can be possibly obtained by DSC measurement.

Differential Scanning Calorimetry

The differential scanning calorimetry (DSC) is used to analyze the thermal behavior of polymers. The differences of the heat flow from a sample to a reference sample are determined at a specific temperature profile. These data provide information on properties for example crystallization temperature (T_K) or the specific heat capacity (c_p) of the material. For evaluation,

the heat flow is plotted against the temperature (see Figure 13). The sample is located in a sealed aluminum crucible. When applying a specific temperature profile, the temperature of each sample is measured and the heat flow is calculated internally. If both samples are identical, the result is no heat flow difference. However, since the reference crucible is empty, a heat flow difference is always measured. In case of a phase transformation, the heat flow changes and this will be visible as a peak in the diagram [9]. In this paper, the measurements start at a temperature of -30 °C and reach a maximum temperature of 280 °C. After this first heating, a defined cooling to -30 °C is conducted. Afterwards, a second heating up to 280 °C is applied. At each maximum / minimum temperature is held for 5 minutes. The heating respectively cooling rate is constant at 10 K/min and is conducted in a N_2 atmosphere. A sample weight of 5 to 6 mg was chosen. For these measurements, a DSC "1-STARe" testing system from the manufacturer Mettler-Toledo was used. The measurement showed a glass transition temperature (T_G) of 40.37 °C for the raw material. This limits the operating temperature range of PA 12.

Figure 13 shows the result of the DSC measurement for virgin PA 12 from the FDM filament. The black curve represents the first heating and the red curve the second heating. Due to the heating two endothermic peaks occur at 177 °C and 228 °C. The melting energy is normalized at 14.31 J/g for the first peak and 9.31 J/g for the second peak. The reason for the double endotherm can be of various kinds. *Roller* describes the following reasons: bimodal crystallite size, crystallites of different modification or different components in the material (e.g. PA 12 and PA 6,12) [8]. It is assumed that this PA 12 is composed of crystallites of different modification (α and γ). However, this requires further DSC measurements to confirm this assumption. After cooling, the second heating is conducted. This is done to remove the thermal history of the material. The two endothermic peaks in the second heating have a reduced melting energy and they are at slightly lower temperatures (172 °C and 222 °C). The reason for this could be the cooling rate. The cooling rate of 10 K/min could be too fast, so the molecules do not have enough time to organize themselves and to form larger crystallites. This results in small, poor ordered crystallites [8].

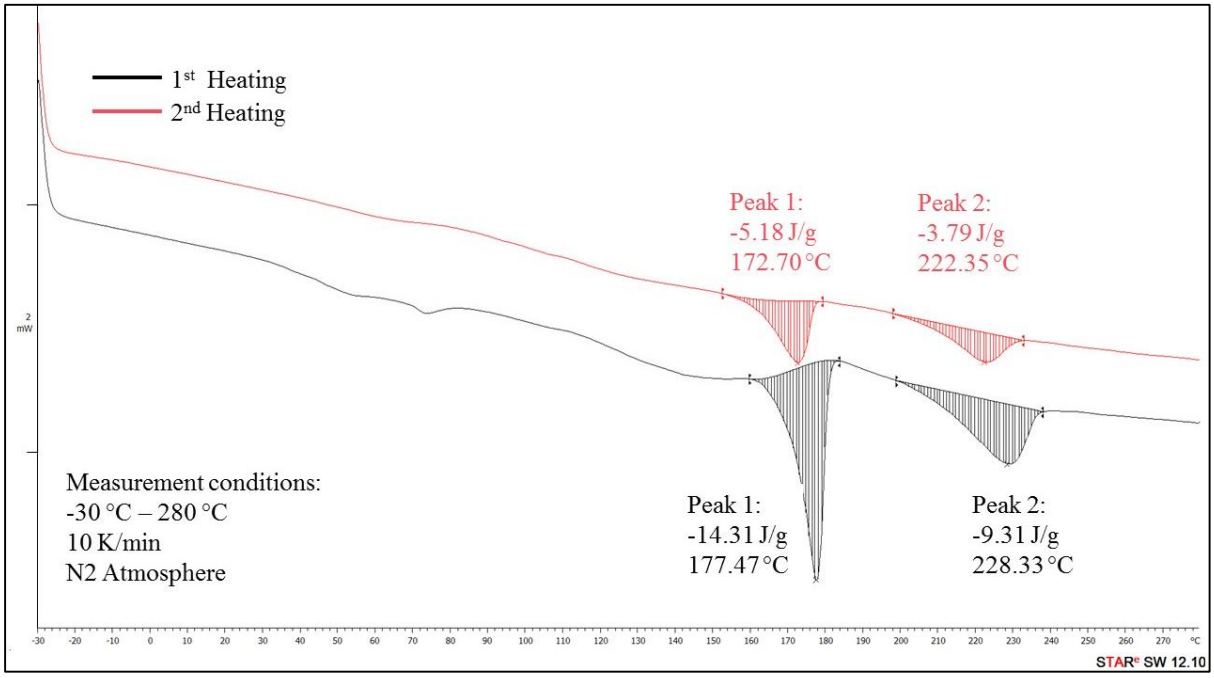

Figure 13 DSC measurement of Polyamide 12 – virgin material

After the virgin PA 12 has been investigated by means of DSC, the focus is now on the tempered PA 12. The heat treatment at 135 °C (for 180 min) should lead to a reduction of internal stress and to a recrystallization. The DSC curve for the tempered PA 12 is shown in Figure 14 and a total of three peaks are recognizable. It is obvious that the first peak is now at a lower temperature of 152 °C (instead of 177 °C). The second peak is quite similar to the first peak of the curve for the virgin material. The same applies for the third peak in this measurement, this third peak is identical to the second peak in Figure 13. Overall, the energy that is required for melting of this sample increases by 50 % to 35.54 J/g. The heat treatment has led to larger, better ordered crystallites. Due to the arrangement in ordered structures, higher forces are acting between the molecular chains. In the second heating, it can be seen that the heating rate of 10 K/min means that almost all crystallite sections have been removed and only very small peaks are identifiable.

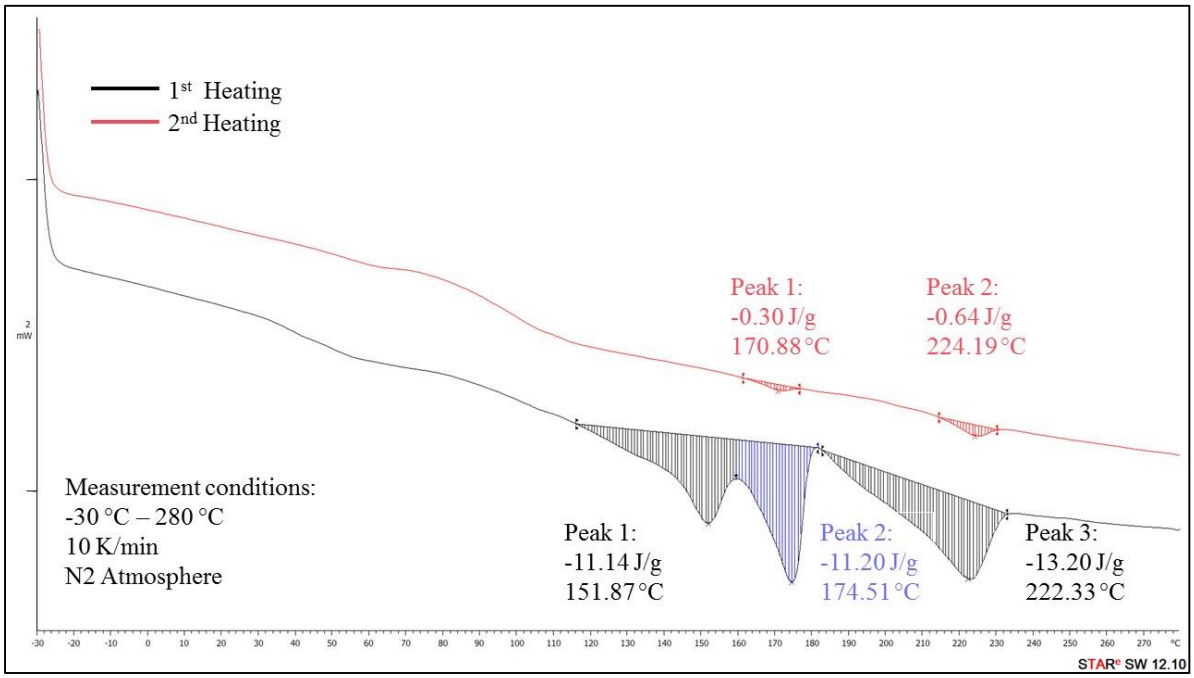

Figure 14 DSC measurement of Polyamide 12 – processed and tempered material

Summary and Outlook

In this paper, the mechanical properties of test specimens manufactured with Polyamide 12 (FDM Nylon 12) were analyzed. Therefore, tensile, flexural and compression tests were conducted according to the European DIN and referring to the American ASTM standard. The test specimens were manufactured with a Fortus 400mc FDM machine and with three different layer thicknesses: 178 µm, 256 µm and 330 µm. For the layer thickness of 256 µm, a thermal treatment (tempering) was carried out. This treatment should improve the heat resistance. Due to the known anisotropy of FDM test specimens, the mechanical properties were determined in the direction in space X, Y and Z. The results show that even with the PA 12 anisotropy can be seen. However, this anisotropy is not as strong as for other FDM materials. The highest tensile strength was recorded in X-direction and the upright specimens have 14 to 20 % lower properties. For upright specimens, it is important, that the surface has a good quality. For that reason different build-up styles were tested and the style “boxed” has been found as the most suitable. The difference between the testing standards is especially noticeable in the elongation at break. According to ASTM standard a lower testing

velocity is used, which results in high values for the elongation at break up to 88%. No anisotropy was visible for the flexural strength but a slight tendency for the flexural modulus. The highest compressive modulus is noticeable in Z-direction with a layer thickness of 254 µm. For the compressive strength, there are only slight differences recognizable. As thermal properties of the Polyamide 12, the flowability is determined (MVR) and the melting behavior by means of DSC analyze. The Melt Volume Rate (MVR) indicates an increase of the flowability due to the processing in the FDM process. The reason for this might be a reduction of the chain lengths of the polymer by the thermal load. With a subsequent heat treatment the MVR is reduced to a value below the virgin material. So the tempering leads to a reorganization and/or recrystallization of the molecular chains. The exact reason has to be determined in further investigations. The results of the DSC analyze showed that there are two endothermic peaks for the first heating. This might have different reasons which have to be clarified in further investigations. The maximum temperature could be reduced to a temperature between the two peaks. Furthermore, the DSC analyze has to be done with a lower heating rate e.g. 5 K/min to see which influence the heating rate has on the crystallization. Finally, there should also be some investigations with processed PA 12 material.

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