LONG-TERM AGEING EFFECTS ON FUSED DEPOSITION MODELING PARTS MANUFACTURED WITH ULTEM*9085

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<u>Abstract</u>

Relating to the direct manufacturing of end-use parts the knowledge about the effect of the long-term ageing of Fused Deposition Modeling (FDM) parts is of particular importance. For this, tensile specimens were stored for time periods of up to 52 weeks in two different conditions and the testing was conducted at different temperatures within a temperature range of -60°C to +160°C. Further tests were made after the exposure in multiple media. The parts were built up with the system "Fortus 400mc" from Stratasys with the material Ultem*9085 in two different build directions, the strongest direction X (on its side) and the weakest build direction Z (upright) and with the standard toolpath parameters of the Insight software version 7.0.

Introduction

Generative production techniques have the advantage of manufacturing parts via an additive process without needing a forming tool. One of these additive manufacturing technologies is "Fused Deposition Modeling" (FDM). It is one of the most used additive manufacturing processes to produce prototypes and end-use parts [1]. From a 3D-CAD data set, components and assemblies are manufactured out of thermoplastic material in only a few working steps. Native software automatically slices the data, calculates the support structures, and creates toolpaths. The parts are then built up layer by layer by means of an additive process. An extrusion head deposits the molten thermoplastic filament to create each layer with a particular toolpath. Due to the thermal fusion the material bonds with the layer beneath and solidifies. Thus a permanent bonding of two layers is formed [2].



Figure 1: FDM-Process

The FDM head processes in the coordinate directions x and y and by lowering the plate in the z-direction, manufacturing layer by layer is possible. If necessary, an additional support material is used to provide a build substrate if the component part shows an overhang, offset or cavity. This additional material prevents the component part from collapsing during the building process. The support material itself can easily be removed after the building process by breaking it off or dissolving it in a warm water bath.

The influence of the orientation and the inner part structure of parts manufactured with the material Ultem*9085 were already analyzed and published [3] [4]. Mechanical tests previously conducted were short-term tests; tensile test, compression test, three-point bending test and Izod impact test. Relating to the direct manufacturing

of end-use parts the knowledge about long-term behavior is of particular importance. No long-term environmental testing has been performed with FDM parts built with Ultem*9085, because it is unknown what effects chemical exposure may have upon the layer by layer manufacture process, i.e., the part may experience a delaminating effect.

Material Ultem*9085

The material Ultem*9085 is a trade name from the company SABIC Innovative Plastics IP BV and is created by blending polyetherimide (PEI) with polycarbonate (PC) [5]. Ultem*9058 was introduced for the FDM technology in 2009 and is desirable due to its mechanical properties and flame, smoke and toxicity properties that allow its use in aircraft cabins. Main features of the base polymer are a high heat resistance, and high strength and modules at elevated temperatures, a good chemical resistance and an inherent non-flammability with low formation of smoke, etc.. Beside its usage in the aircraft and automotive industry, this polymer can also used in many other fields; for example, tableware, medical gastronomy devices, car lighting engineering, heaters, ventilations, climate and liquid technique. [6]

Long-term Ageing Effects

Test specimens were built using the geometry as per ASTM D 638 [7] specifications in the directions X and Z (on its side and upright) with a contour and an inner part raster fill. The generation of the toolpath was made with the preset parameters of the native software "Insight 7.0" with a raster fill and an angle of 45° to the x-axis. Alternating layers are filled with a raster direction at 90° to one another. A T16 tip was used, that creates filament geometry of 0,508mm (0,02inch) in width and 0,254mm (0,01inch) in height.

The build directions of the specimens are presented in the following illustration. Furthermore, 10 specimen of each orientation were built up in one job. The Z-direction specimens are built with model material as a support in order to reduce the build time. The positions and placements of the specimens in a job are shown in the following illustration.



Figure 2: Build direction of specimens for tensile test and position of these specimens in a build job

The specimens of one build job were tested after a specific conditioning time period and at a specific temperature. The specimens were stored at an ambient temperature of 23°C and a relative humidity of 50% according to the standards ASTM D 618 [8] / DIN EN ISO 291 [9]. Specimens stored at controlled conditionings were placed in an open box and specimens stored wet were totally immersed in distilled water. The conditioning time periods for the tensile specimens are 1, 4, 13, 26, 52 weeks. After the specific storage period the specimens were exposed at different temperatures for a minimum of 40h before testing at this temperature. The specific temperatures are -60° C, -20° C, $+20^{\circ}$ C, $+60^{\circ}$ C, $+100^{\circ}$ C, $+140^{\circ}$ C and $+160^{\circ}$ C.

Results

The tensile tests were conducted with the universal testing system Instron 5569. Tests were conducted according to the American standard ASTM D638 [7], at the specific temperature. The velocity was 5 mm/min and a load was applied to the specimens until they broke. A load cell with 5kN was used for this test.

The tensile properties of specimens build up in X-direction stored dry and wet are shown in Figure 3 against the exposure periods and for all testing temperatures. The following diagram shows the max. tensile strength of the specimens built up in X-direction.



Figure 3: Tensile strength of X-specimens against the exposure periods and the testing temperatures

The tensile strength obtains the highest data for the lower temperatures and is decreasing with rising test temperature. The highest average data is listed with σM =92,8MPa for specimens tested at a temperature of -60°C. The lowest average data is listed with σM =26,0MPa for specimens tested at a temperature of +160°C. The diagram shows constant average data for all conditioning periods with small deviations except for specimens tested at the lowest temperatures -60°c and -20°C. For these testing temperatures higher deviations are listed. However, no change of the tensile properties due to different conditionings and different conditioning time periods is noticeable for specimens built up in X-direction.

Figure 4 shows the Young's Modulus of the specimens built up in X-direction against the exposure periods for all testing temperatures and for the controlled and wet storage conditionings.



Figure 4: Young's Modulus of X-specimens against the exposure periods and the testing temperatures

The Young's Modulus has the highest data for the lower temperatures and is decreasing with rising test temperature. The highest average data is listed with E=3057MPa for specimens stored wet for short

conditioning time periods and tested at a temperature of -60° C. The lowest average data is listed with E=1535MPa for specimens tested at a temperature of $+160^{\circ}$ C and stored at wet conditionings. This diagram again shows nearly constant average data for all conditioning periods with small deviations.

Figure 5 shows the corresponding elongation at break of tensile specimens built up in X-direction against the exposure periods for all testing temperatures and for the controlled and wet storage conditionings.



Figure 5: Elongation at break of X-specimens against the exposure periods and the testing temperatures

The data for the elongation at break shows higher deviations and the data is not that constant over all conditioning periods. These deviations possibly can occur because of some process variation. Furthermore curves do overlap from different temperatures. The lowest average data is listed with ε =3,9% for specimens stored wet and tested at a temperature of -60°C followed by specimens tested at a temperature of +100°C. The highest average data is listed for specimens tested at a temperature of +20°C with ε =6,8%.

In Figure 6 the average data of all conditioning periods is shown to analyze the influence of the temperature and to compare the data of the wet and controlled conditioned specimens. The following diagram shows the average tensile strength against the temperature.



Figure 6: Average tensile strength of X-specimens against the testing temperatures

Again, the tensile strength obtains the highest data for the lower temperatures and decreases with increasing test temperature. The highest average data is listed for specimens tested at a temperature of -60° C and the lowest average data is listed for a testing temperature of $+160^{\circ}$ C. The diagram also shows higher standard deviations for the data of specimens tested at lower temperatures and very small standard deviations at higher testing temperatures. Furthermore the data for wet and controlled conditioned specimens obtain the same data. Thus, the conditioning has no influence on the mechanical data of specimens build up in X-direction.

In Figure 7 the average data of the Young's Modulus for all conditioning periods is shown against the testing temperature and for the wet and controlled conditioned specimens.



Figure 7: Average Young's Modulus of X-specimens against the testing temperatures

The highest data for the Young's Modulus is obtained for the lower temperatures and the data is decreasing with rising test temperature. Same as the tensile strength, the data show higher standard deviations for specimens tested at lower temperatures and the strengths for wet and controlled conditioned specimens obtain the same data.

The average data of the elongation at break for all conditioning periods is shown in Figure 8 against the testing temperature and for the wet and controlled conditioned specimens.



Figure 8: Average elongation at break of X-specimens against the testing temperatures

Again, the elongations at break obtain the same data for wet and controlled conditioned specimens. The highest average data is listed for specimens tested at a temperature of $+20^{\circ}$ C, followed by specimens tested at a temperature of -20° C and $+160^{\circ}$ C. The lowest average data is listed for specimens tested at a temperature of -60° C followed by specimens tested at a temperature of $+100^{\circ}$ C.

The tensile properties of specimens built up in Z-direction stored controlled and wet are shown in the next diagrams against the exposure periods and for all testing temperatures. Figure 9 shows the max. tensile strength of the specimens built up in X-direction.



Figure 9: Tensile strength of Z-specimens against the exposure periods and the testing temperatures

Like for specimens built up in X-direction, the tensile strength of Z-specimens obtain the highest data for the lower temperatures and is decreasing with rising test temperature. The highest average data is listed with $\sigma M=56,7MPa$ for specimens tested at a temperature of -60°C. The lowest average data is listed with $\sigma M=12,6MPa$ for specimens tested at a temperature of +160°C. The diagram shows constant average data for all conditioning periods with a few deviations. The tensile strength values for specimens tested at the lowest temperature (-60°C) have higher data for the longest exposure time (26 and 52 weeks). The data at higher testing temperatures show small deviations. However, overall no change of the tensile properties due to different conditionings and different conditioning time periods is noticeable for specimens built up in Z-direction.

Figure 10 shows the Young's Modulus of the specimens built up in Z-direction against the exposure periods for all testing temperatures and for the controlled and wet storage conditionings.



Figure 10: Young's Modulus of Z-specimens against the exposure periods and the testing temperatures

The Young's Modulus also has the highest data for the lower temperatures and is decreasing with rising test temperature. The highest average data is listed here with E=2990MPa for specimens stored wet and tested at a temperature of -60°C. The lowest average data is listed with E=1328MPa for specimens tested at a temperature of +160°C. This diagram again shows nearly constant average data for all conditioning periods with some deviations.

Figure 11 shows the corresponding elongation at break of tensile specimens built up in Z-direction against the exposure periods for all testing temperatures and for the controlled and wet storage conditionings.



Figure 11: Elongation at break of Z-specimens against the exposure periods and the testing temperatures

The data for the elongation at break shows nearly constant values for all conditioning periods with some bigger deviations with curves overlapping. The lowest average data is listed with $\varepsilon = 1,1\%$ for specimens stored wet and tested at a temperature of +160°C and the highest average data is listed for specimens tested at a temperature of -60°C, +20°C and +60°C with $\varepsilon = 2,6\%$.

In Figure 12 the average data of all conditioning periods is shown to analyze the influence of the temperature and to compare the data of the wet and controlled conditioned specimens. The following diagram shows the average tensile strength of Z-specimens against the testing temperature.



Figure 12: Average tensile strength of Z-specimens against the testing temperatures

Again, the tensile strength obtains the highest data for the lower temperatures and decreases with increasing test temperature. The highest average data is listed for specimens tested at a temperature of -60°C and the lowest average data is listed for a testing temperature of +160°C. The diagram also shows higher standard deviations for the data of specimens tested at lower temperatures and smaller standard deviations at higher testing temperatures. Furthermore the data for wet is lower for nearly all testing temperatures, but within the standard deviation. Thus, the conditioning has no influence on the mechanical data of specimens build up in Z-direction. In Figure 13 the average data of the Young's Modulus for all conditioning periods is shown against the testing temperature and for the wet and controlled conditioned specimens.



Figure 13: Average Young's Modulus of Z-specimens against the testing temperatures

The highest data for the Young's Modulus is obtained for the lower temperatures and the data is decreasing with rising test temperature. The data for wet and controlled conditioned specimens obtain the same values. The average data of the elongation at break for all conditioning periods is shown in Figure 14 against the testing temperature and for the wet and controlled conditioned specimens.



Figure 14: Average elongation at break of Z-specimens against the testing temperatures

The data for the elongations at break obtain smaller values for wet than for controlled conditioned specimens, but are within the standard deviations. The highest average data is listed for specimens tested at a temperature of $+20^{\circ}$ C, followed by specimens tested at a temperature of $+60^{\circ}$ C and -20° C. The lowest average data is listed for specimens tested at a temperature of $+160^{\circ}$ C.

Geometry and weight

The geometry and weight of all stored specimens was measured before and after the exposure to analyze any change. In his section only the differences of the width, height and weight of specimens after the longest storage period is shown, (52 weeks). After this storage period the most changes should be visible. For this analysis the average data with its standard deviations of all specimens stored for this time period is used. Figure 15 shows the results.



Figure 15: Comparison of the average width of specimens before and after the exposure

The average widths and heights of all specimens, for both build directions and for both conditionings, show a slightly smaller data after the storage period of 52 weeks. But all data are within the standard deviations. Thus, no change in the geometry after 52 week storage at controlled and wet conditionings is noticeable.

Figure 16 shows the average weight of the specimen after the exposure in comparison to the weight before the exposure.



Figure 16: Comparison of the average weight of specimens before and after the exposure

The weights of specimens stored controlled show higher data after the storage period of 52 weeks for both build directions and these values are not within the standard deviation. One reason could be that the moisture content of the material Ultem*9085 is higher after the storage at room temperature (RT) with a relative humidity of 50%. This higher data is noticeable for all storage periods, even after 1 week.

Media Exposure

In this section the mechanical data of FDM specimens are shown after an exposure in multiple media relevant for the airplane industry. Specimens were exposed for a one week time period at RT and for a period of 90 days at different temperatures. For this analysis tensile specimens again were built up in the two build directions X and Z (on its side and upright) with the standard parameters (green flag) of the Insight software.

One week media exposure

The one media exposure was done according to the standards ASTM D 543 [10] / DIN EN ISO 175 [11]. All specimens were totally immersed in the medium and stored in a dark chamber at RT.

Media used for this exposure were two different de-icers with the trade name Killfrost TKS 80, that is used for airplane in-flight de-icing and anti-freeze protection (without alcohol), and Killfrost RDF, that is used for a rapid defrosting (with alcohol). Furthermore an alkaline cleaner with the trade name Rhobaclean 303 STU was used for this exposure. This is a degreasing and cleaning concentrate.

The tensile tests were conducted at RT, with a velocity of 5 mm/min and a load cell with 5kN.

Relating to the geometry of the specimens after this exposure, no change was measured.

Figure 17 shows the data of the tensile strengths with the standard deviations of FDM specimens built up in different directions against the stored media in comparison to the standard strength value known for the respective build styles.



For all build directions and build styles the tensile strength properties are lower after the media exposure, compared to the standard values. Also the strength values after the exposure in the alkaline cleaner Rhobaclean are the lowest. Thus, this media does influence the Ultern material and increases the strength properties. Figure 18 shows the data of the Young's Modulus with the standard deviations of all built up specimens against the stored media in comparison to the standard strength values.



No change of the data for the Young's Modulus is noticeable for all specimens after a media exposure of one week. The data show very small variances, but they are within the standard deviations.

Figure 19 shows the data of the elongations at break with the standard deviations of all built up specimens against the stored media in comparison to the standard strength value of these parts.



After the exposure to both de-icers, no changes of the elongation at break are noticeable for all build directions and build styles. All values are within the standard deviations. But after the exposure in the alkaline cleaner Rhobaclean the mechanical strength properties did decrease.

Media exposure for 90 days

Further analyses were done on FDM specimen exposure in different media for a time period of 90 days. Media used for this analysis were lubricating oil (exposure temperature 143°C), hydraulic fluid (exposure temperature 82°C) and jet fuel (exposure temperature RT / 23°C and 50% relative humidity).

The strength properties of FDM parts after the exposure to both oils show slightly lower data and the tensile strength of these specimens decreases more after an exposure in jet fuel. The average data for the Young's Modulus after the exposure also show slightly lower values after the exposure in all media, but are within the standard deviation. Thus, no change of the Young's Modulus of FDM specimens after this exposure is noticeable. The data of the elongation at break for the FDM specimens decreases after the exposure to lubricating oil and jet fuel. The average data after the exposure to hydraulic oil is also smaller, but within the standard deviation with reference to the standard values of these specimens.

Summary

In this paper the long-term ageing effects on FDM parts manufactured with the material Ultem*9085 are shown after a storage of up to 52 weeks in two different conditions at RT and after the exposure in multiple media relevant for the airplane industry. Furthermore, specimens were tested at different temperatures.

After the storage of the FDM specimens at RT in controlled and wet conditionings up to 52 weeks no change of the tensile properties due to different conditioning and time periods is noticeable. Only the elongation at break shows some higher deviations. Furthermore strength properties obtain the highest data for the lower temperatures and are decreasing with rising test temperature. Moreover no change in the geometry after 52 weeks storage at controlled and wet conditionings is noticeable. The weight of these specimens show higher data, also for specimens stored controlled. A reason could be that the moisture content of the material Ultem*9085 is higher after the storage at RT with a relative humidity of 50%.

The tensile strength properties after a media exposure show small decreases for most of the tested media. The highest influence on the strength properties of the FDM parts manufactured with Ultem*9085 have the alkaline cleaner with the trade name Rhobaclean and the jet fuel.

References

[1] T. Wohlers: "Wohlers Report 2011 – Additive Manufacturing and 3D Printing State of the Industry", Annual Worldwide Progress Report, 2011

[2] B. Wendel: "Prozessuntersuchung des "Fused Deposition Modeling"", Dissertation, Friedrich-Alexander-University of Erlangen-Nuernberg, 2009

[3] A. Bagsik, V. Schoeppner, E. Klemp: "FDM Part Quality Manufactured with Ultem*9085", 14th International Scientific Conference on Polymeric Materials 2010, Halle (Saale) 2010

[4] A. Bagsik, V. Schoeppner, E: Klemp: "Schicht für Schicht", Kunststoffe 10/2011, pp. 178-182, Carl Hanser Verlag, Muenchen, 2011

[5] B. Graybill: "Development of a Predictive Model for the Design of Parts Fabricated by Fused Deposition Modeling", Master Thesis, University of Missouri-Columbia, 2010

[6] GE Plastics - Ultem Profil

http://www.plastoplan.com/download/ge_plastics_pdf_downloads/gep_Ultem.pdf.

[7] ASTM D 638: Standard Test Method for Tensile Properties of Plastics

[8] ASTM D 618: Standard Practice for Conditioning Plastics for Testing

[9] DIN EN ISO 291: Normalklimate für Konditionierung und Prüfung

[10] ASTM D 543: Standard Practices for Evaluating the Resistance of Plastics to Chemical Regents

[11] DIN EN ISO 175: Prüfverfahren zur Bestimmung des Verhaltens gegen flüssige Chemikalien