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Evaluation of the Fire-retardancy of ULTEM 9085 Polymer Composites Processed by Fused Deposition Modelling

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In the paper, the results obtained for the fire-retardancy of ULTEM 9085 polymer composites manufactured by the fused deposition modelling (FDM) are summarized. The effects of processing parameters of FDM such as the percentage of infill, thickness of the sample, and the number of the solid layers on either side were experimentally evaluated against fire-retardancy parameters (burn length and heat release rate). Based on the results, all test samples of ULTEM 9085 were observed to have passed the test requirements specified in Federal Aviation Regulation (FAR) & EASA Certification Specifications (CS) Part 25 of Airworthiness standards for the aviation sector. The maximal burn length (approx. 80 mm) was registered for 30 % infill samples with zero solid layers on either side. Based on the results obtained it was concluded that the burn length was almost the same in all build directions. Moreover, inconsistent results were obtained for the heat release rate as a function of the thickness of the sample for different build directions. Though, certain clear effects were obtained regarding burn length as a function of infill percentage proving that fire-retardancy is the most effective at higher infill percentages.

Keywords: additive manufacturing, fire retardant polymers, aircraft industry, ULTEM, fused deposition modelling (FDM).

1. INTRODUCTION

The occurrence of fire in a confined environment surrounded by polymeric components can lead to disastrous consequences. This makes it essential to consider flame-retardant polymers in critical applications, such as aircraft, automobiles, train and building interior design components. Conventional flame-retardant methods are dependent on either adding halogen or phosphorous based chemicals to achieve the required attributes. There are also polymeric options that are intrinsically fire-resistant and can satisfy the fire safety regulatory standards such as the UL94 or FAR 25.853 [1]. However, the window of options is quite narrow, considering other aspects such as eco-friendliness, mechanical or chemical properties, and processing difficulties.

Flame retardant polymeric materials have been used extensively for the aircraft interior components, seats and furniture, insulations, interior panels, floor coverings, draperies, air ducting, linings, tubing, electrical components, and firewalls [1 – 3], meeting the fire safety requirements as per FAR 25.855 [4, 5]. Polyester, nylon, and vinyl blended with zirconium-based flame additives are used in upholsteries. Cushion foams are made of neoprene, silicone, and modified urethane. Synthetic fibres, such as polybenzimidazole (PBI), aromatic polyamides, and glass fibres are used to fabricate fire-blocking textiles. Plastic moulding on seats is normally made of polycarbonate, acrylonitrile-butadiene-styrene (ABS) and vinyl. Polyvinyl fluoride laminates are commonly used on the surfaces of the panels. Polyetherimide, polyphenylsulfone and polyimide are also used at various places as thermoplastic components. However, the never-ending urge to achieve high strength-to-weight ratios has constantly been pushing the boundaries for new fire-retardant polymeric material alternatives in the aerospace industries [6, 7].

While the actual fabrication methods based on the fire-retardant materials vary with the part shape, properties, and quantity, traditional methods typically include injection moulding [8, 9], hand lay-up, spray-up, compression moulding, filament winding, pultrusion, resin transfer moulding, vacuum-assisted resin transfer moulding, infusion, and continuous panel processing [10]. The honeycomb structures in the panels are made by a series of rolling and pressing cold works. All these methods have been widely used and well developed, but often suffer from the long supply-chain issues and lack the flexibility to accommodate design changes and customisation.

Additive manufacturing (AM) refers to a bunch of technologies that grew out of the 3D printing realm over time. With both material and process enhancements, some of the methods have emerged as acceptable manufacturing solutions for the direct production of end-use systems from digital data. Compared to traditional manufacturing techniques, AM allows a great degree of freedom of design, mass customisation and waste minimisation [11]. Selective laser sintering (SLS), selective laser melting (SLM), fused deposition modelling (FDM), and stereolithography (SLA) are the most common and popular additive manufacturing

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choices. Amongst these, SLS and FDM are the only methods used for processing flame retardant or resistant polymeric materials.

The additive manufacturing technologies have gained considerable ground in the meantime, gradually finding application potentials in both the automotive and aerospace industries [11].

Essential features of the different types of materials used in AM exist. Nylons are widely used in SLS, it is low cost, and the strength of the material is decent [12]. However, it is flammable and cannot be used in an environment with stringent fire safety regulations. The current solution is to add halogen and phosphorus-based compounds into the nylon powders to modify their flame retardancy, to meet the requirements.

PAEK (polyaryle ether ketones) is a family of thermoplastics, it includes PEEK (poly ether ether ketone), there is PEK (poly ether ketone), PEKK, and PEKEKK (poly ether ketone ether ketone ketone). All these materials have excellent chemical and thermal resistance and also good strength. PEEK is inherently resistant to combustion and when forced to burn, they produce very few toxic gases, unlike other polymers [13].

ULTEM polymer family such as ULTEM 9085 is an amorphous polyetherimide thermoplastic blend that was synthesised for injection moulding. Recently, these materials were used for FDM processing. The main component of these materials is polyetherimide (PEI) with a polycarbonate copolymer blend, similar to the related PEEK. ULTEM polymers, like PEEK, are chemical and heat resistant, inherently flame retardant and emit lower smoke and pass the majority of the fire safety regulation tests. They also have excellent dimensional stability and strength at elevated temperatures [14]. ULTEM resins are widely used in aircraft applications because of their properties which are compliant with aviation regulations. The ULTEM 9000 resin series is compliant with aircraft regulations such as FAR 25.853, ABD 0031, OSU 65/65 tests and NBS smoke tests for being used as aircraft interior materials. All the additive manufacturing processing for ULTEM materials is based on the FDM technique [15].

Modification of design and geometry such as printing sandwich structure, the mixture of different materials, and printing part with entrapped water can also provide flame retardancy, however, more research and data are needed to prove these methods can produce certified parts [16, 17]. Horizontal and vertical flame retardant tests could be successfully applied for checking the thermal resistance and stability of polymers and polymer-based composites [18].

Processing parameters for FDM such as printing orientations, printing strategies, percentage of infill, nozzle diameters, layer thickness, printing speed, bed temperature, filling structure, and filling angle need to be experimentally evaluated against flame retardancy. The test information needs to be compiled into a database for designers and end-users to use for certification [18]. To the best of the authors' knowledge, no such or similar results for ULTEM materials regarding the effect of processing parameters on flame-retardant properties were previously discussed in the literature. Therefore, the novelty of the paper is to reveal these effects and check if all tested samples could pass the requirements formulated in FAR 25.853 and FAR 25.855 for the materials used in the compartment interiors and cargo or baggage compartment in the aviation sector.

Thus, the current paper aims to evaluate the effects of several FDM processing parameters, i.e., percentage of infill, number of solid layers, and thickness of the samples on the fire-retardant properties, burn length and heat release rate, of ULTEM 9085.

2. EXPERIMENTAL DETAILS

2.1. Experimental plan

Stratasys Fortus 450mc and F900 machines are widely available at Baltic3D factory and were used for the manufacturing of all test samples. From the technical considerations, both machines operate with the same manufacturing settings and produce identical quality 3D printed samples. The dimensions of the samples printed for different tests are provided in Table 1.

The material used was ULTEM CG (Certified Grade). ULTEM 9085 is PEI (polyetherimide) thermoplastic FDM material. It features a high strength-to-weight ratio, high thermal and chemical resistance, and meets multiple aerospace and railway industry standards for flame, smoke and toxicity (FST) characteristics. The material filament was stored in a canister, which is vacuum-sealed in a protective bag filled with dry packs. The raw material was stored at a temperature of 13 – 24 °C and humidity < 60 %. The material used for the sample manufacturing was stored in the same conditions, therefore it preserves the same properties before and during the manufacturing, and there is no variation between the material cartridges.

The purpose of this test plan outlines the flammability test requirements and methods required for the testing of the 3D printed test samples. This is to demonstrate compliance with the airworthiness requirements of JAR/FAR/CS 25.853 as mandated by the air regulatory authority. Fig. 1 shows the 3-printing direction, in *x*, *y* and *z*. The printed samples had a varying thickness (1 – 5 mm), and fixed dimensions that meet the requirement for standard vertical burn and heat release tests (see Table 1).

Table 1. Dimension requirements for the tests

Description	Criteria/Procedure	Specimen dimensions, mm	Number of tests
Vertical burn test, 60 s	EASA CS 25.853 Appendix F part I (a)(1)(i)	75 × 305	20
Heat release test	Heat release rate FAR/CS 25.853, PART IV	150 × 150	20

For the vertical burn test (60 s), the specimens were prepared by Baltic3D using an FDM printing method made from ULTEM 9085 CG material. Each specimen set is prepared in different thicknesses and various infills: 30, 50, 70, 90 and 100 %. Each separate set of specimens prepared for testing consisted of at least five specimens. In Table 2, the list of manufactured test samples is provided showing the thickness, infill percentage, and number of solid layers for verical burn and heat release tests. The manufacturing of the test samples was performed by using layer-to-layer

FDM technology. An example of a solid 2 mm infill sample is shown in Fig. 2 and Fig. 3.

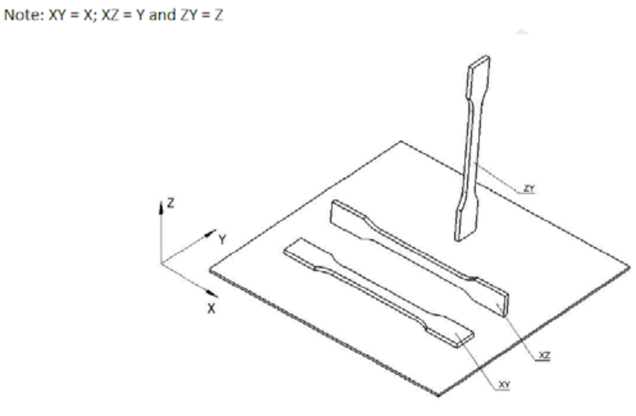

Fig. 1. The sample printing orientations

Table 2. Test samples manufactured for vertical burn tests (VBT) and heat release tests (HRT)

Thickness, mm	Print direction	Number of solid layers	Infill percentage, %
1.00	X, Y, Z	1	100 % (HRT)
1.25	X, Y, Z	0, 1	30 %, 50 %, 70 %, 90 % (VBT)
1.50	X, Y, Z	0, 1	30 %, 50 %, 70 %, 90 % (VBT)
	X, Y	1	100 % (HRT)
1.75	X, Y, Z	0, 1	30 %, 50 %, 70 %, 90 % (VBT)
2.00	X, Y, Z	0, 1	30 %, 50 %, 70 %, 90 % (VBT)
	X	0, 1, 2	50 % (VBT)
	X, Y, Z	1	100 % (VBT, HRT)
2.50	X, Z	1	100 % (HRT)
5.00	X	1	100 % (HRT)

The manufacturing of the test samples was performed by using layer-to-layer FDM technology. An example of a solid 2 mm infill sample is shown in Fig. 2 and Fig. 3.

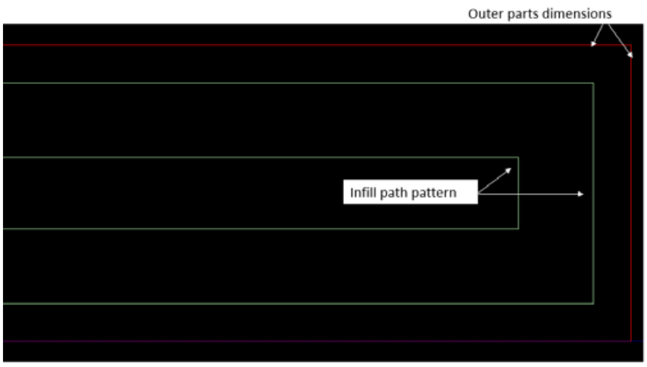

Fig. 2. Raw infill pattern view from the top

For the samples manufactured in the Y and Z direction (see Fig. 1), the sample infill was performed with a straight line pattern as shown in Fig. 2 and Fig. 3. For samples printed in the X direction (see Fig. 1), the infill was performed with a 45° infill pattern. For samples with a thickness of 5 mm, where a partial infill was used, an example of such sample infill for a 5 mm sample is shown in Fig. 4. The standard infill pattern was at +45° for the first layer and -45° for the following layer and so it continued with the start angle of 45° and delta angle of 90°.

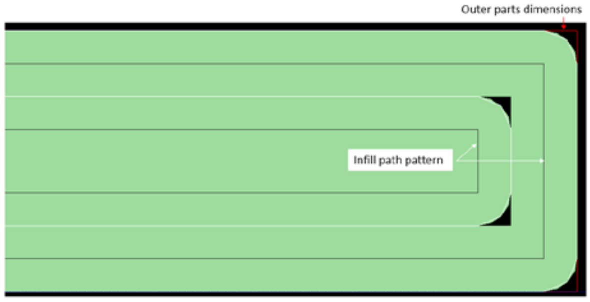

Fig. 3. Infill pattern view from the top with shaded toolpath

Depending on the samples infill percentage there is a variation in distance between the toolpath path, it remains in each layer exactly the same.

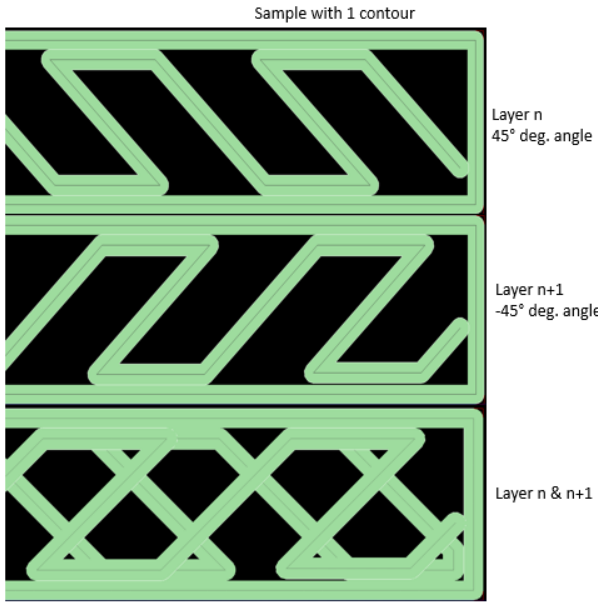

Fig. 4. Infill pattern view from the top for a partial infill with shaded toolpath

2.2. Vertical burn tests for cabin and cargo compartment materials

The vertical burn tests were carried out at Lantal Textiles AG (Langenthal, Switzerland) according to FAR/CS 25 Appendix F Part I of aviation standard.

The scope of this test method is intended for use in determining the resistance of materials to a flame when tested according to a 60-second vertical burn test (V60). There are several definitions in this test:

1. ignition time: length of time the burner flame is applied to the specimen. It is 60 seconds for the V60 vertical burn test;
2. flame time: time in seconds that the specimen continues to flame after the burner flame is removed from beneath the specimen. Noted, the surface burning results in a glow but not in a flame is not included;
3. drip flame time: drip flame time is the time in seconds that any flaming material continues to flame after falling from the specimen to the floor of the chamber. If no material falls from the specimen, the drip flame time is reported to be 0 seconds, and the notation “no

drip" is also reported. If there is more than one drip, the drip flame time reported is that of the longest flaming drip. If succeeding flaming drips reignite earlier drips that flamed, the drip flame time reported is the total of all flaming drips;

4. burn length: the distance from the original specimen edge to the farthest evidence of damage to the test specimen due to that area's combustion including areas of partial consumption, charring, or embrittlement but not including areas soothed, stained, warped, or discoloured nor areas where the material has shrunk or melted away from the heat.

The requirements for passing the test are:

1. flame time: the average flame time for all of the specimens tested will not exceed 15 seconds for the 60-second vertical test;
2. drip flame time: the average drip extinguishing time for all of the specimens tested will not exceed 3 seconds for the 60-second vertical test;
3. burn length: the average burn length for all of the specimens tested will not exceed 152 mm for the 60-second vertical test.

2.3. Heat release rate tests for cabin materials

The heat release rate tests were performed at Lantal Textiles AG (Langenthal, Switzerland) according to FAR/CS 25 Appendix F Part IV of aviation standard.

The scope of this test is intended for use in determining heat release rates to show compliance with the requirements of FAR 25.853. The heat release rate is measured for the duration of the test from the moment the specimen is injected into the controlled exposure chamber and encompasses the period of ignition and progressive flame involvement of the surface.

Heat release is a measure of the amount of heat energy evolved by a material when burned. It is expressed in terms of energy per unit area (kW·min/m^2). The heat release rate is a measure of the rate at which heat energy is evolved by a material when burned. It is expressed in terms of power per unit area (kW/m^2). The maximum heat release rate occurs when the material is burning most intensely.

The requirements for passing the test are:

1. the average maximum heat release rate during the 5 minutes tests will not exceed 65 kW/m^2;
2. the average total heat released during the first 2 minutes will not exceed 65 kW·min/m^2.

3. RESULTS AND DISCUSSION

3.1. Vertical burn test

The experimental setup for the vertical burn tests and the test sample before and after the test are shown in Fig. 5. Fig. 6 shows the vertical burn test results for samples printed in the X direction with various thicknesses and infill percentages. The graphs presented in Fig. 6 show that the burn length is increased with the decrease of infill percentage to 30 %. For infill percentages 50 %, 70 % and 90 %, the burn lengths are relatively stable as evident in Fig. 5. Moreover, according to Fig. 6 with the increase in thickness, the burn length generally decreases. It could be explained by the fact that the more the material was contained in any sample, the lesser was the burn length. Similar results that more infill percentage caused less burning and that thinner samples had larger burn length than thicker ones were obtained for Ultem 9085 by Federal Aviation Administration [19].

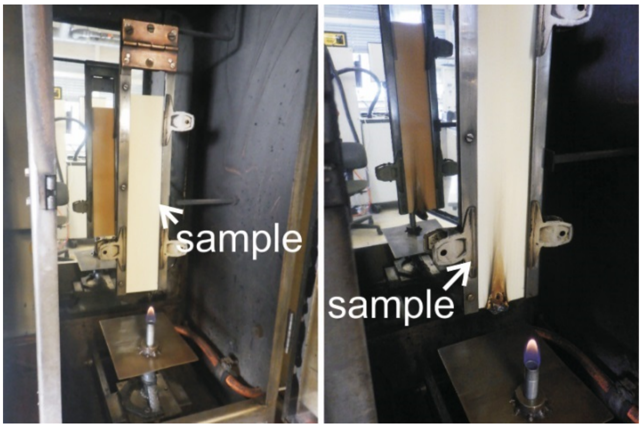

Fig. 5. Experimental setup used for vertical burn tests at LANTAL labs. The test sample before (to the left) and after the test (to the right)

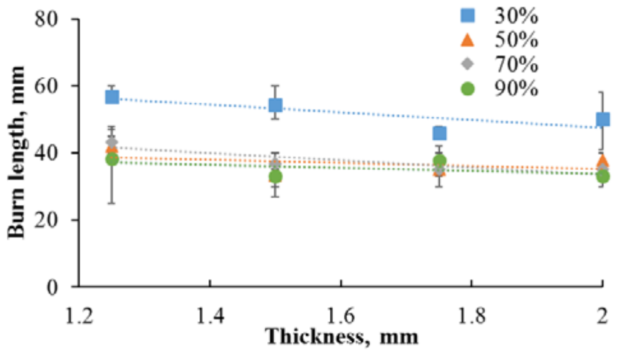

Fig. 6. Vertical burn length vs thickness with 1 solid layer on either side for different infill percentages (as indicated on the graph). Dots – experimental data, lines – linear approximations

Fig. 7 summarizes the vertical burn test results for samples printed in the X direction with zero (0) solid layers on either side.

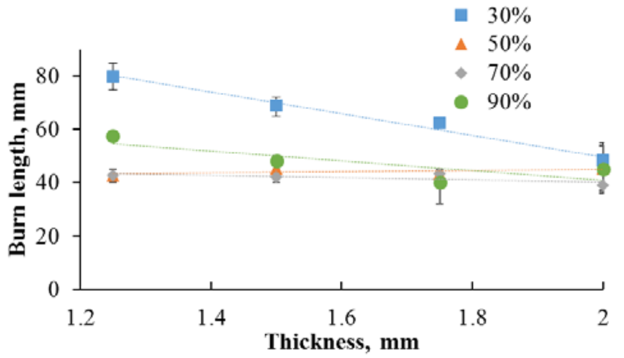

Fig. 7. Vertical burn length vs thickness with 0 solid layers on either side for different infill percentages (as indicated on the graph). Dots – experimental data, lines – linear approximations

It may be observed from Fig. 7 that the 30 % infill samples have shown relatively higher burn lengths at all the thickness levels, compared to the other samples with higher percentage filling. This is expected as the total material content is the least in the 30 % infill case. Further, the burn length almost linearly reduced with an increase in the thickness, which is also expected due to the gradually increasing mass to burn. The 50 % and 70 % infill samples have shown almost the same burn length at all the thickness levels tested. The 90 % infill samples have resulted in slightly higher burn lengths compared to the 50 % and 70 % infill samples, particularly at lower thickness. Other than these variations, the results, in general, follow the trend that the more the mass of the printed material, the higher is the fire resistance, reflected in the lower burn lengths.

Fig. 8 shows the vertical burn test results in the X direction with 50 % infill and 2 mm thickness with varying numbers of solid layers. Obviously, the burn length slightly reduces with the increase in the number of solid layers from 0 to 1, and then mostly remains stable as the number of solid layers increases from 1 to 2.

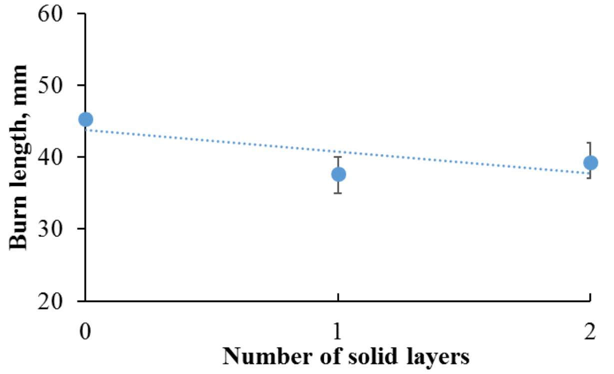

Fig. 8. Vertical burn test vs various number of solid layers with 50 % infill and 2 mm thickness

The results shown in Fig. 9 are comparing the burn length against various infill percentages with a constant thickness of 1.5 mm and one solid layer on both sides. From the graph, it can be seen the burn length is quite high in the 30 % infill samples compared to the 50 %, 70 % and 90 % infill samples. The general trend in the burn length reducing with increasing infill percentage from 30 to 90 % is understandable from the material content point of view. Also, it should be mentioned that the range of the experimental errors was significantly larger in all cases, possibly indicating a lack of control on the data collecting metrics.

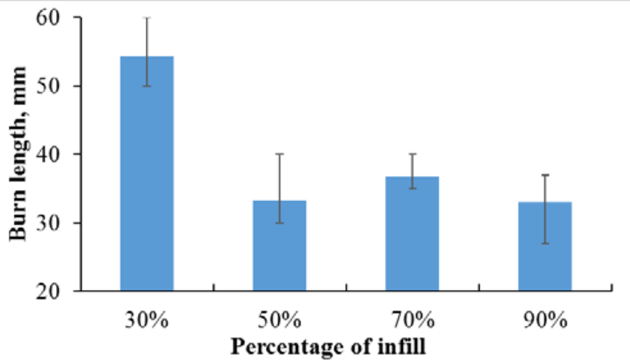

Fig. 9. Vertical burn test vs various infill percentages (indicated on the graph) with 1.5 mm thickness and 1 solid layer on top and bottom

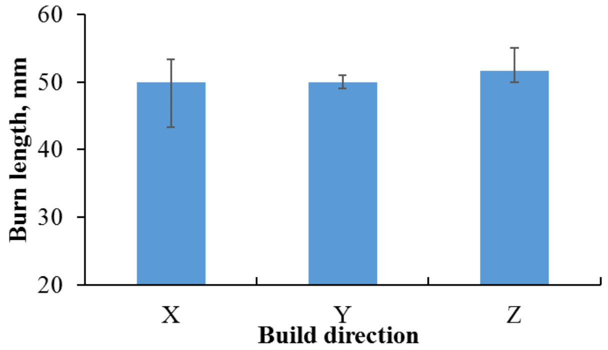

Fig. 10. Vertical burn test result vs the build direction with 100 % infill and 1 solid layer with 2 mm thickness

Efforts were being made to compare the vertical burn lengths against three build directions, X, Y, and Z with 100 % infill and one solid layer on either side, and Fig. 10 shows the results obtained with 2 mm thick samples, respectively. It can be seen from Fig. 10 that the burn length was almost the same in all build directions. Similar results were obtained for the rest of the sample thicknesses at 100% of infill proving that the direction of printing generally was not affecting the burn length.

3.2. Heat release rate tests

Fig. 11 shows an example of a testing chamber (a) and an operational burner (b) for the heat release test [5], as well as the 15 × 150 mm sample before (c) and after (d) testing.

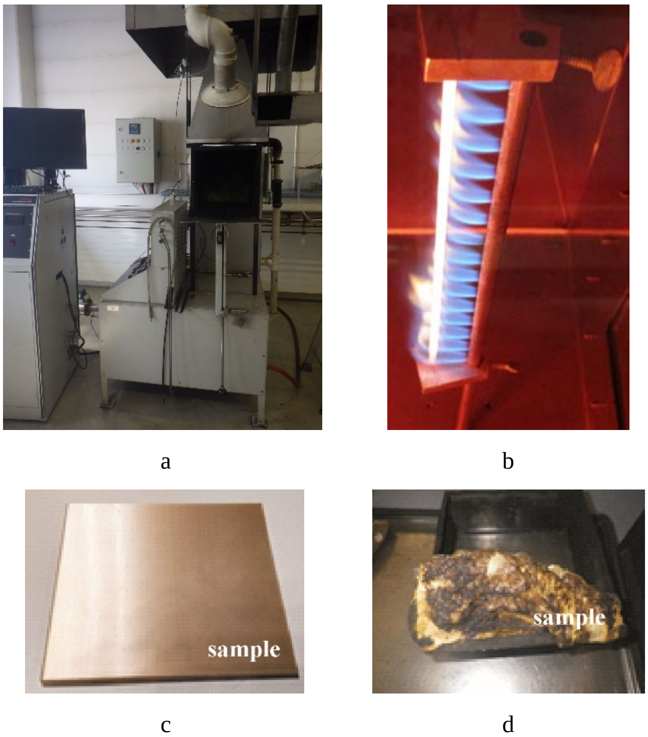

Fig. 11. Test chamber of heat release apparatus: a – with upper pilot burner; b – operation for heat release test by FAR/CS 25.853; testing samples c – before test completion; d – after the test completion

Fig. 12 shows the variation of the heat release rate plotted against various parameters drawn. As evident in Fig. 12, in the X build direction, the HRR can be seen to decrease with the increase of thickness until 2 mm, then increase slightly up to 2.5 mm thickness and then stays the same up to 5 mm thickness. A similar trend was observed in the Y build direction, HRR was decreased and then increased with the increase of thickness from 1 to 1.5 mm and 1.5 to 2.00 mm respectively. Finally, for the Z build direction, the HRR was increased with increasing thickness. Also, it should be mentioned that the scattering of data results was rather high for all build directions indicating the possible improvement of the testing procedure and/or samples' quantity and quality.

Finally, the graphs shown in Fig. 13 revealed the variation of the peak and total values of the heat release rate against the mass as recorded during the first 2 minutes of burning.

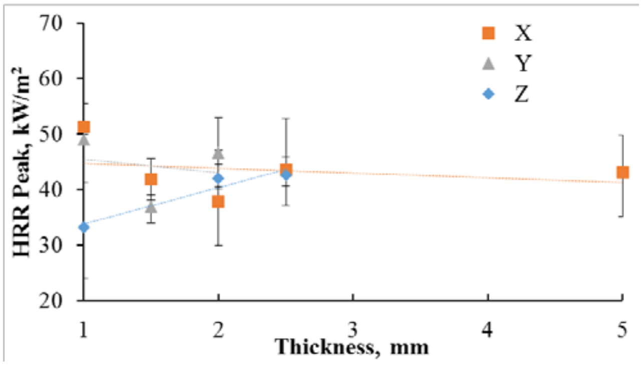

Fig. 12. Heat release rate vs thickness in different build directions X, Y, Z as shown in Fig. 1

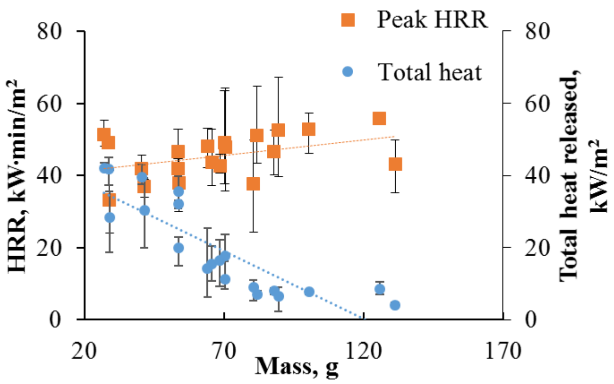

Fig. 13. Peak heat release rate (left axis) and total heat released (right axis) vs mass of the samples

Neglecting the scatter in the points and the relatively large experimental errors, the general trend in Fig. 13 is that the heat release rate is relatively stable with varying mass, though there is a slight increase towards the end for the peak value. Contrarily, there is a clear trend in the variation of the total heat release rate showing an almost linear decrease with the increasing mass of the samples. Once again, the error bars are quite large with specific experimental points.

4. CONCLUSIONS

The results obtained in the research could be summarized as follows:

1. The test samples of ULTEM 9085 have all passed a 60-second vertical burn test (V60) that is specified in FAR 25.853 and FAR 25.855 at all infill percentages, all numbers of solid layers, and at all thicknesses. The requirement to pass the test is set to 152 mm burn length for the 60-second vertical test. The maximal burn length (approx. 80 mm) was registered for 30 % infill samples with zero (0) solid layers on either side. Nevertheless, a certain relationship was obtained regarding the effect of thickness on burn length showing clear evidence that the more material was contained in any sample, the lesser was the burn length. The 30 % infill samples have shown relatively higher burn lengths at all the thickness levels, compared to the other samples with higher percentage filling (50 – 100 %). This was expected as the total material content is the least in the 30 % infill case.
2. The burn length was slightly reduced with the increase in the number of solid layers from 0 to 1, and then mostly remained stable as the number of solid layers increased from 1 to 2. Moreover, based on the results obtained for the comparison of the vertical burn lengths against three build directions, X, Y, and Z with 100 % infill and one solid layer on either side, it was concluded that the burn length was almost the same in all build directions proving that the direction of printing generally was not affecting the burn length.
3. The test samples of ULTEM 9085 at all infill percentages and numbers of solid layers have also passed the 65/65 acceptance criteria requirements for the heat release rate test, defined in FAR/CS 25: the average maximum heat release rate during the 5 minutes tests did not exceed 65 kW/m^2 and the average total heat released during the first 2 minutes did not exceed 65 kW min/m^2. Though the scatter in the data was rather high, a clear trend in the variation of the total heat released was revealed showing an almost linear decrease with the increasing mass of the samples. While the general conclusion is that the heat release rate is relatively stable with varying mass for all test samples of ULTEM 9085.
4. To summarize, the samples were manufactured in all three directions, X, Y and Z, with various thicknesses, and different percentages of infill. The manufactured samples have all passed the flammability aviation requirements as per FAR/CS 25.853 and FAR/CS 25.855 for the materials used in the Compartment interiors and Cargo or baggage compartment. With this in mind, it is clear to say that samples made from ULTEM 9085 material, using FDM printing technology are suitable for use in aircraft interior compartments. The manufacture of those parts can have partial infill and therefore the design of the future parts can be minimized to the lowest manufacturing technology requirements for additional weight reduction.

Acknowledgments

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