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ADDITIVE MANUFACTURING AND CHARACTERIZATION OF ULTEM POLYMERS AND COMPOSITES

Kathy C. Chuang, Joseph E. Grady and Robert D. Draper
NASA Glenn Research Center, Cleveland, OH

Euy-Sik E. Shin
Ohio Aerospace Institute, Cleveland, OH

Clark Patterson and Thomas D. Santelle
Rapid Prototype Plus Manufacturing (rp+m), Avon Lake, OH

ABSTRACT

The objective of this project was to conduct additive manufacturing to produce aircraft engine components by Fused Deposition Modeling (FDM), using commercially available polyetherimides – Ultem 9085 and experimental Ultem 1000 mixed with 10% chopped carbon fiber. A property comparison between FDM-printed and injection-molded coupons for Ultem 9085, Ultem 1000 resin and the fiber-filled composite Ultem 1000 was carried out. Furthermore, an acoustic liner was printed from Ultem 9085 simulating conventional honeycomb structured liners and tested in a wind tunnel. Composite compressor inlet guide vanes were also printed using fiber-filled Ultem 1000 filaments and tested in a cascade rig. The fiber-filled Ultem 1000 filaments and composite vanes were characterized by scanning electron microscope (SEM) and acid digestion to determine the porosity of FDM-printed articles which ranged from 25-31%. Coupons of Ultem 9085 and experimental Ultem 1000 composites were tested at room temperature and 400°F to evaluate their corresponding mechanical properties.

1. INTRODUCTION

Additive manufacturing (AM) has gained considerable attention recently, because of the promise of being able to produce net shape 3D components layer by layer directly by automated machines. This is especially true for complex shape polymer parts and low production volume components, which are not economical to produce by injection molding. In addition, AM offers quick turn- around time for specialty parts and shortened production and testing cycle for components. This project concentrated on Fused Deposition Modeling (FDM) technique (Fig. 1) in which a polymer filament is melted and then deposited in successive layers to build a 3D component according to a computer-aided design (CAD) file [1]. The state-of-the art of FDM are populated with commercial ABS (acrylonitrile butadiene styrene), polycarbonate [2] and polyamides such as Nylons for use as prototyping at the temperature around 100-125 °C (212-257° F).

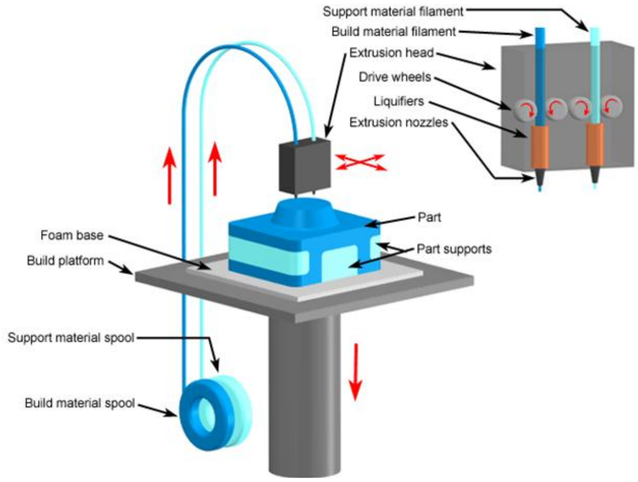
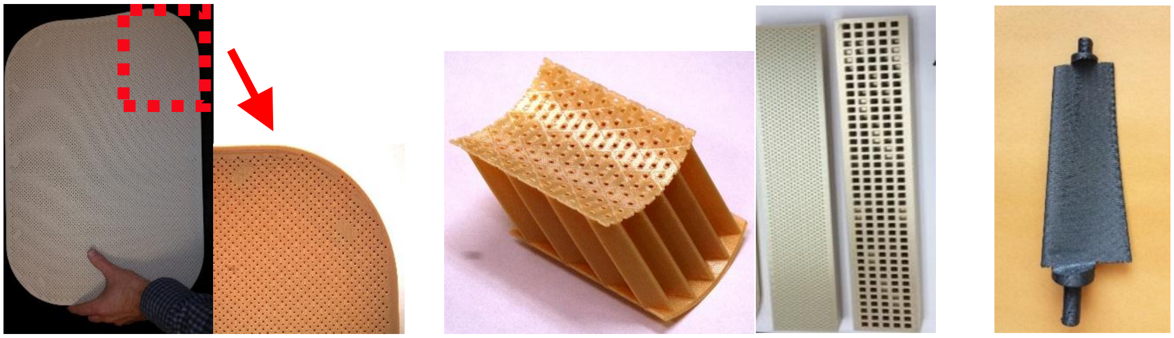


Figure 1. Schematic of Fused Deposition Modeling (FDM) process.

The objective of the project was to develop additive manufacturing approaches for polymeric aircraft engine components and conduct testing on coupons as well as built parts, such as acoustic testing in a wind tunnel. The Ultem 9085 polyetherimide filament is one of the commercial polymers marketed by Stratasys for use in FDM with a glass transition temperature (T_g) of 186 ºC (367 °F). Ultem 9085 is certified by FAA as flame retardant polymer for use in aircraft cabin. This project used Ultem 9085 as the baseline polymer for printing demonstration components, such as acoustic liners and a perforated engine access door. Furthermore, this project also strived to advance the FDM process into building polymer composites for aircraft engine parts. These additively manufactured components were tested in rigs and results have been presented in the first part of the report [3]. The Ultem 1000 with 10% AS4 carbon fiber was chosen as the candidate fiber-filled polymer filaments for this project, because Stratasys is making it available for the first time as an experimental filament under the State funded Ohio Third Frontier research project. Ultem 1000 is a homopolymer with higher T_g (217 °C, 423 °F) than that of Ultem 9085 which is a blend of polycarbonate and Ultem 1000 with lower viscosity and cost suitable for injection molding.

2. EXPERIMENTATION

All the FDM printing was performed at Rapid Prototype Plus Manufacturing (rp+m), using Stratasys' open source Fortus 400mc or 900mc FDM machines. The experimental XH6050 resin and carbon-fiber-filled Ultem 1000 filaments were supplied by Stratasys under the Ohio Third Frontier Program — Advanced Materials for Additive Manufacturing Maturation. The Ultem resins and composites were printed between 375-420 °C (707-788 °F). The specific engine components were selected by Honeywell Aerospace. Using Ultem 9085, a perforated engine door (Fig. 2a), an acoustic liner and its demonstration components (Fig. 2b) with 93 °C (200 °F) use temperature were printed by FDM at 375 °C in one piece, simulating the Aramid honeycomb structures bonded with epoxy composite face sheet. Additionally, composite vanes (Fig. 2c) with use temperature up to 204 °C (400°F) were printed at 420 °C, using Ultem 1000 filled with 10% chopped AS4 carbon fibers. Rig testing conditions and results of acoustic liner and composite inlet guide vane (IGV) are described in details in the first part of the report [3].



Perforated engine access door (Ultem 9085) b) Acoustic liner and components (Ultem 9085) c) Composite vanes Ultem 1000/fiber

Figure 2. FDM printed polymer components

3. RESULTS AND DISCUSSION

3.1 Property Comparison of FDM and Injection Molding

A mechanical property comparison between FDM-printed and injection-molded coupons of Ultem 9085, Ultem 1000 and the carbon-fiber-filled Ultem 1000 are shown in Table 1. These data indicated that Ultem 9085 (printed at 0° raster angle), displayed about 87% of tensile strength and 64% of modulus, as compared to the injection molded counter parts, due to the presence of inherent porosities within the FDM-printed test coupons. The porosity of FDM-printed Ultem 9085 was about 5-8%, depending on the orientation of the layup. The mechanical strength of FDM generated specimens also relied on the built direction, the thickness of the filaments, the tool path generation and the air gap between raster in the filled pattern. In general, the FDM generated structures are more brittle and have lower elongation than the injection molded counterparts [4].

Table 1. Property Comparison of Ultem 9085 and Ultem 1000 by Injection Molding *vs* FDM

esin type Properties	Ultem 9085 Injection Molded (Sabic data)	Ultem 9085 FDM printed (Stratasys data) 0°	Ultem 9085 FDM rp+m (GRC tested) ±45°	Ultem 1000 Injection Molded (Sabic data)	Ultem 1000+10wt% AS4 chopped C-fiber FDM rp+m (GRC tested) 0°/±45°
Tensile Strength (MPa)	83	72	62 ± 0.1	110	50±0.9/44±0.3
Tensile Modulus (MPa)	3,432	2,200	2,230 ± 12	3,579	2,901±48/2248±46
Flexural Strength (MPa)	137	115	92 ± 2	165	tbd
Flexural Modulus (MPa)	2,913	2,500	1,901 ± 41	3,511	tbd
Compression Strength (MPa)	n/a	104	tbd	n/a	tbd
Compression Modulus (MPa)	n/a	1,930	1,890 ± 32	n/a	tbd

*No Ultem 1000 filament for FDM is commercially available.

3.2 Initial Characterization of Carbon Fiber Filled Ultem 1000 Composites and Filaments

The initial tensile strength of 10% AS4 fiber-filled Ultem 1000 composites (first batch ever made from Stratasys) was only about 70% of Ultem 9085 resin as printed by FDM (Fig. 3), which was much lower than expected. The printing of ±45° raster angle reduced the strength by 80-86% as opposed to 0°. Further investigation by acid digestion indicated that the porosity of fiber-filler Ultem 1000 vanes ranged from 23-26%, which are unusually high (Table 2). The printing orientation did not exhibit much difference in terms of porosity. However, the porosity measurement based on the integration of optical microscope images (Fig. 4) ranged from 29-34% (Table 3), which was even higher than the 23-26% porosity obtained by acid digestion.

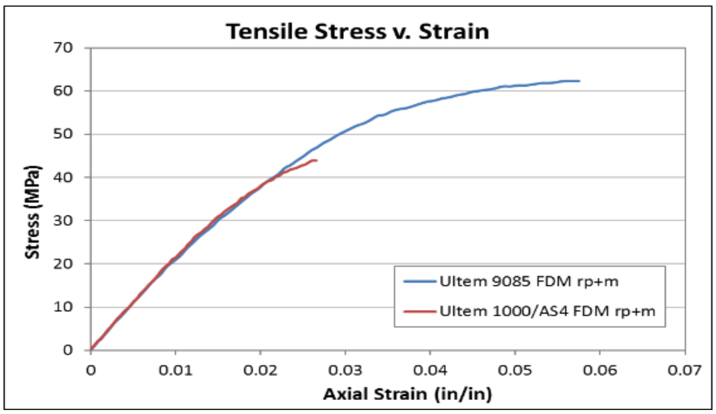


Figure 3. Tensile properties of Ultem 9085 and fiber-filled Ultem 1000 as received

Table 2. Porosity of Fiber-Filled Ultem 1000 Composite Vanes by Acid Digestion

Sample ID	After drying					From Theor. Density				FWF	FVF	porosity
	Balance	Pycnometer		Acid digestion								
	Mc, g	Vc, cc	ρc, g/cc	Mf, g	Mm, g	Vf, cc	Vm, cc	Vp, cc	Vp, cc	wt%	v%	v%
Ha1	0.7731	0.831	0.931	0.0712	0.702	0.0398	0.5527	0.2376	0.2385	9%	5%	28.7%
Ha2	0.3977	0.374	1.063	0.0498	0.348	0.0278	0.2739	0.0720	0.0722	13%	7%	19.3%
Ha3	0.9433	0.952	0.992	0.0894	0.854	0.0499	0.6724	0.2300	0.2297	9%	5%	24.1%
Ha4	0.6676	0.734	0.911	0.0753	0.592	0.0421	0.4664	0.2252	0.2256	11%	6%	30.7%
Ha5	1.1184	1.194	0.939	0.0627	1.056	0.0350	0.8313	0.3279	0.3277	6%	3%	27.4%
Avg										*10% ±3*	*5% ±2*	*26% ±4*
Va1	0.8706	0.929	0.938	n/a								
Va2	0.3349	0.347	0.966	0.0346	0.300	0.0193	0.2365	0.0911	0.0912	10%	6%	26.29%
Va3	0.5443	0.492	1.112	0.0413	0.503	0.0231	0.3961	0.0727	0.0729	8%	5%	14.81%
Va4	0.637	0.695	0.92	0.0632	0.574	0.0353	0.4518	0.2064	0.2079	10%	5%	29.91%
Va5	1.2997	1.25	1.037	0.0833	1.216	0.0465	0.9578	0.2462	0.2457	6%	4%	19.65%
Avg										*9% ±2*	*5% ±1*	*23% ±7*

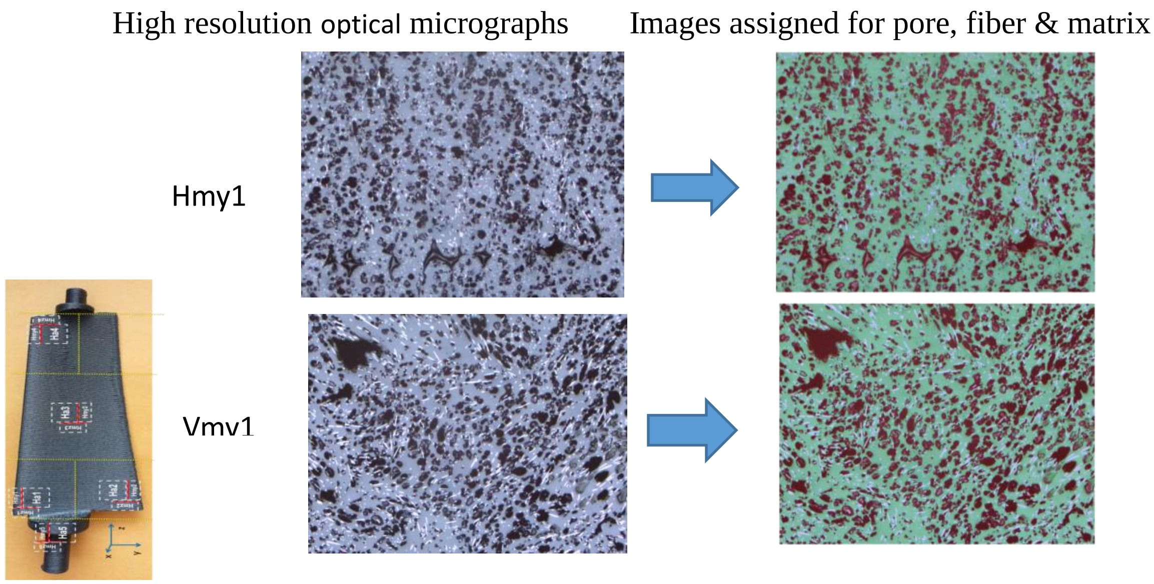
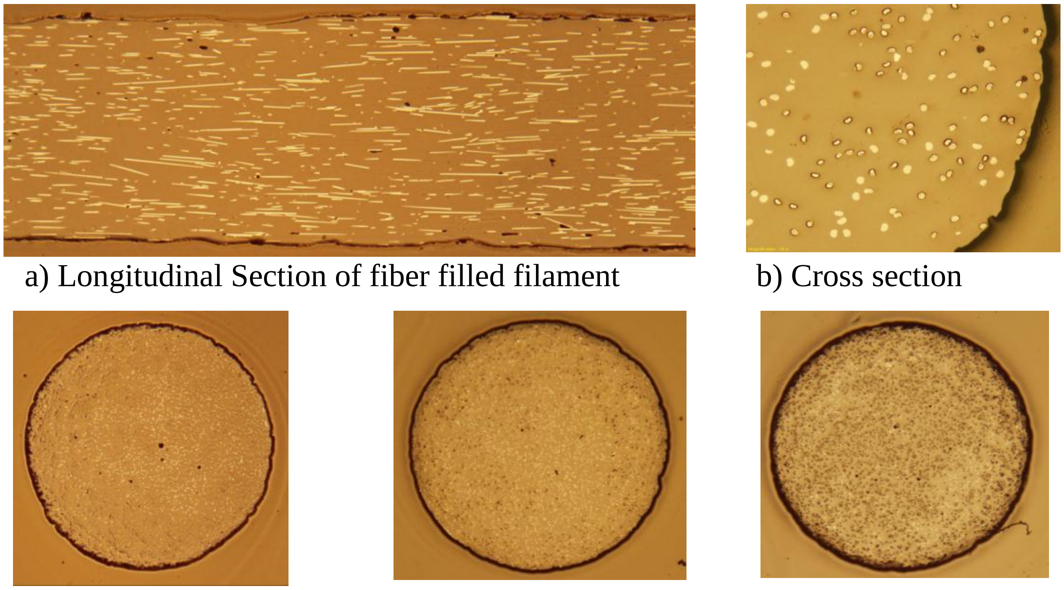


Figure 4. High Resolution of optical micrographs (left) and images of pore, fiber and matrix (right) of a FDM-printed composite vane

Table 3. Porosity of composite vanes based on Optical Microscope Images

Sample ID	Average* ± S.D.		Sample ID	Average* ± S.D.	
	FVF, v%	Porosity, v%		FVF, v%	Porosity, v%
Hmy1	5.7% ± 0.3%	33.2% ± 0.3%	Vmy1	7.5% ± 0.3%	29.9% ± 2.6%
Hmy4	5.0% ± 0.3%	34.8% ± 1.7%	Vmy5	7.3% ± 0.6%	32.5% ± 5.0%
Hmy5	6.4% ± 0.3%	32.3% ± 1.5%	Vmz1	5.1% ± 0.3%	39.2% ± 1.6%
Hmz1	7.1% ± 0.3%	33.4% ± 3.2%	Vmz3	6.8% ± 0.4%	31.2% ± 2.8%
Hmz3	6.8% ± 0.6%	33.3% ± 5.2%	Vmz4	7.0% ± 0.3%	33.8% ± 0.8%
Hmz4	7.5% ± 0.4%	28.9% ± 3.8%	Vmz5	6.5% ± 0.4%	31.6% ± 2.4%
Hmz5	6.3% ± 0.4%	34.4% ± 3.0%			
Overall Avg ± SD	**6.4% ± 0.7%**	**33.2% ± 3.2%**	**Overall Avg ± SD**	**6.7% ± 0.8%**	**32.9% ± 4.1%**

To understand the origin of high porosity in the FDM-printed fiber-filled Ultem 1000 composites, an effort was initiated to investigate the as-received thick Ultem 1000 filament filled with 10% chopped AS4 carbon fibers, which was fed into the FDM machine. The fiber-filled Ultem 1000 filaments were produced by mixing 6 mm AS4 chopped fibers with Ultem 1000 in an extruder, cut into pellets, and then re-extruded into filaments at Stratasys. As shown in Fig. 5, the longitudinal section (a) revealed that the chopped fibers were aligned with the filament axis, and significant amounts of the fibers were further chopped into average length of 2-3 mm during the extrusion process. The cross section (b-e) indicated that there were little voids present in the as-received filaments in this segment of initial investigation.



a) Longitudinal Section of fiber filled filament

b) Cross section

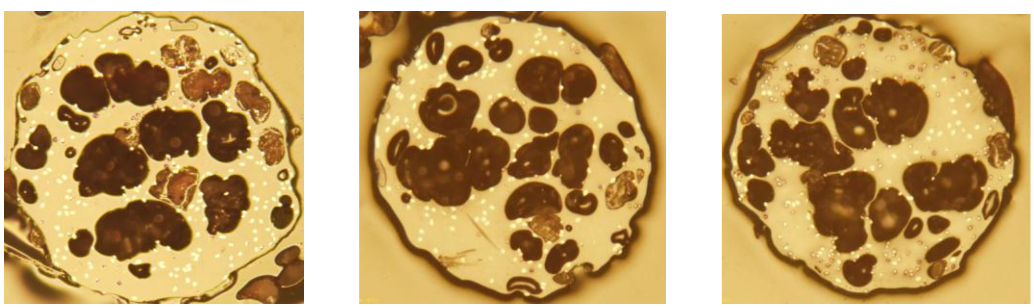
c) Cross section #1

d) Cross section #2 after removing ~1 mm

e) Cross section #3 after removing ~2 mm

Figure 5. Photomicrographs of 10% AS4 fiber filled Ultem 1000 filaments (as received-thick)

Separately, thin filaments of fiber-filled Ultem 1000 extruded from the liquefier of Stratasys' Fortus 400mc FDM machine at 420 ºC were collected and analyzed, since these thin filaments were used directly for FDM printing. As shown in Fig. 6, the FDM extruded thin filaments were full of voids in the form of blisters, due to the sudden exposure to extreme high heat.



a) Cross section#1

b) Cross section #2 after removing ~1 mm

c) Cross section #3 after removing ~2 mm

Figure 6. Photomicrographs of 10% AS4 fiber filled Ultem 1000 filaments (thin)

The acid digestion values in Table 4 confirmed that this segment of as-received thick fiber-filled filament analyzed had no porosity as confirmed by the photomicrographs in Fig. 5. The fiber content was 9% by weight which was very close to the original formulation of 10% chopped fibers. However, the thin FDM-extruded filaments were found to have about 31% of porosity, which was closer to the image analysis result of 33% porosity than that of 24-26% porosity by acid digestion of the printed vane.

Table 4. Porosity of Fiber-Filled Ultem 1000 Filaments

Sample ID	After drying					From Theor. Density			FWF, wt%	FVF, v%	porosity, v%
	Balance	Pycnometer		Acid digestion							
	Mc, g	Vc, cc	ρc, g/cc	Mf, g	Mm, g	Vf, cc	Vm, cc	Vp, cc			
Filament, thick	0.2753	0.2084	1.3209	0.0254	0.250	0.014	0.1968	-0.003	**9%**	**7%**	**-1.2%**
Filament, thin 1	0.0582	0.0645	0.9029	0.0054	0.053	0.003	0.0416	0.02	**9%**	**5%**	**30.9%**
thin 2	0.0583	0.0653	0.8924	0.0054	0.053	0.003	0.0417	0.021	**9%**	**5%**	**31.6%**

* Thick filament—as received and fed into FDM machine; thin filament — extruded from FDM machine

Thermogravimetric analysis (TGA) with Fourier Transform Infrared Spectroscopy (FTIR) was carried out to investigate the origin of blistering in Ultem 1000. As shown in Fig. 7, the major weight loss shown in TGA up to 300 °C (572 °F) corresponded to water shown in FTIR, which indicated that some water was trapped inside the filament, which was more difficult to remove than the surface water. Additionally, Fig. 8 indicated that Ultem 1000 resin pellet contained 0.375% of water, and the fiber-filled Ultem 1000 filament had 0.593% of water whereas separate TGA analysis had indicated that the chopped fiber contained 0.25% of water. These two curves indicated that other than water loss, the Ultem resin and filament are very stable until about 500 °C (932 °F). However, the thin fiber-filled filament showed not only the loss of surface water around 100 ºC (212 °F), but also some other weight loss due to degradation, as it had been exposed to the sudden high temperature of 420 °C (788 °F) at the liquefier in the FDM machine during the melting process. This first lot of the fiber-filled Ultem 1000 filament seemed to be solid but brittle whereas the thin filament appeared to be fragile and extremely porous.

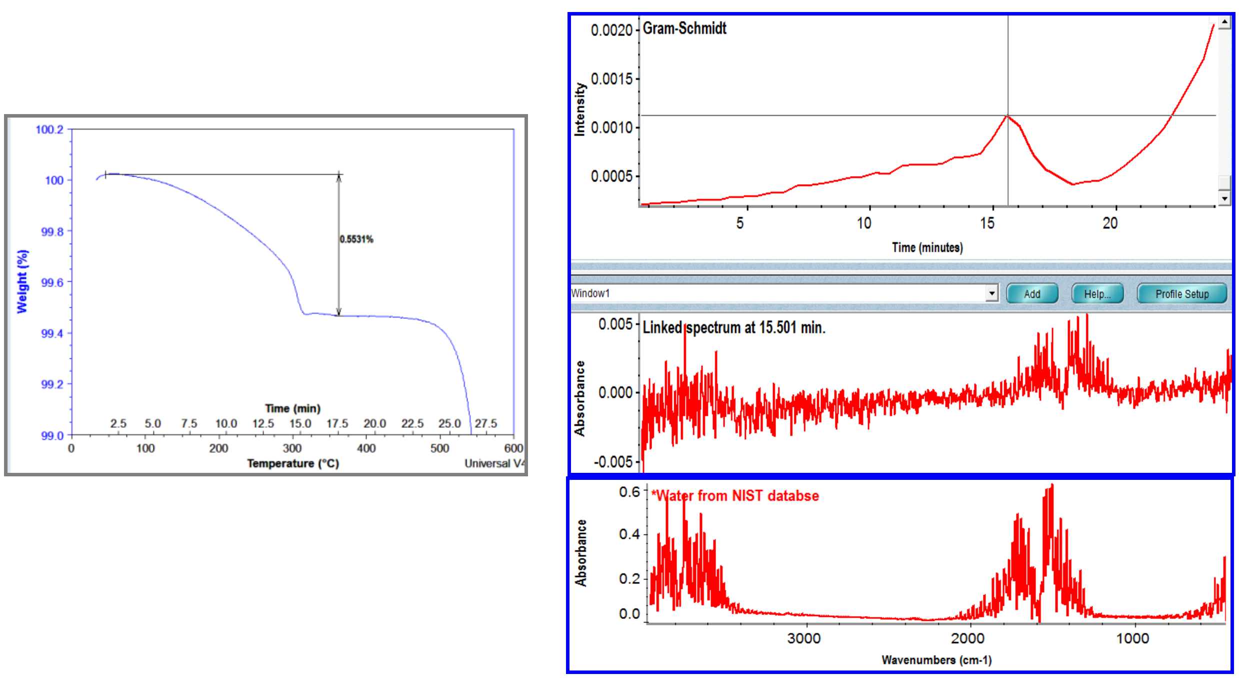


Figure 7. TGA-FTIR analysis of the fiber-filled Ultem 1000 filament

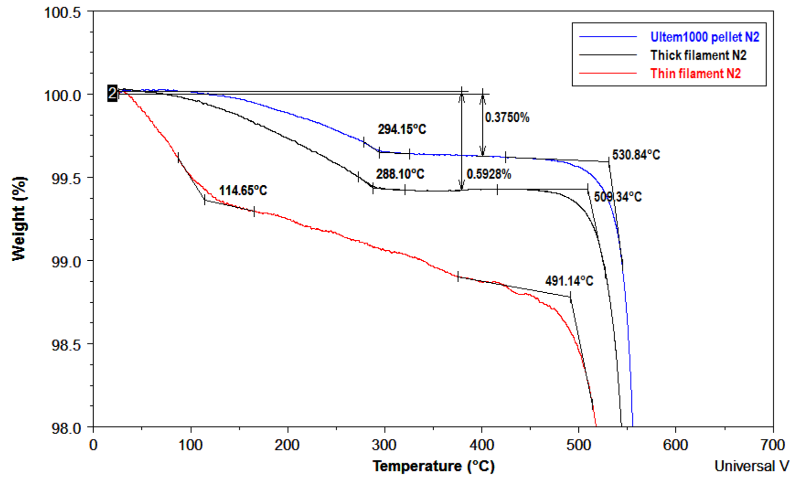


Figure 8. Thermogravimetric analysis of and fiber-filled Ultem 1000 filament and resin pellet

3. 3 Evaluation of Drying Processes for Fiber-Filled Ultem 1000 Filaments

1) To improve the quality and mechanical properties of FDM-printed composites, an initial drying of as-received filament at 185 °C (365 °F) was carried out to remove the water from fiber-filled Ultem 1000 in an air-circulation oven for 12 hours; this was followed by the printing two cubes by FDM (Fig. 9). The dried thick filaments and FDM-spun thin fibers seemed shrunken and more ductile than the corresponding as-received filaments and un-dried FDM-spun thin filaments. The resulting printed cubes contained much lower porosity (13.6-17.4%) than that of the vanes (23-26%) as indicted in Table 2.

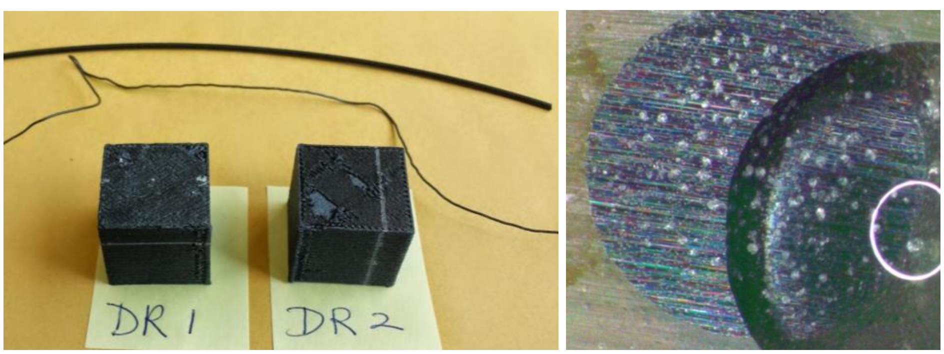
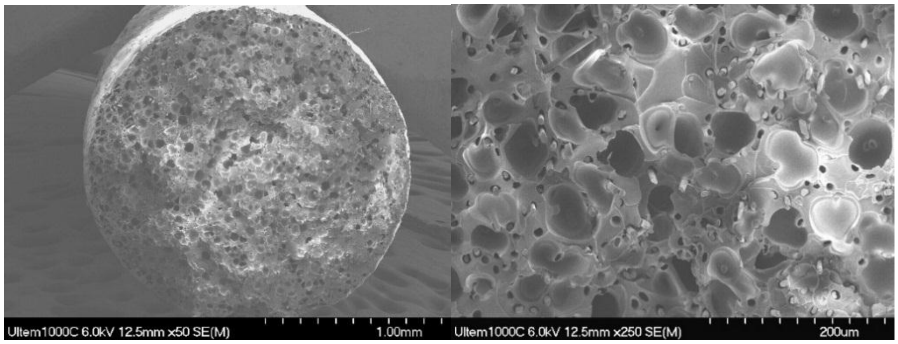
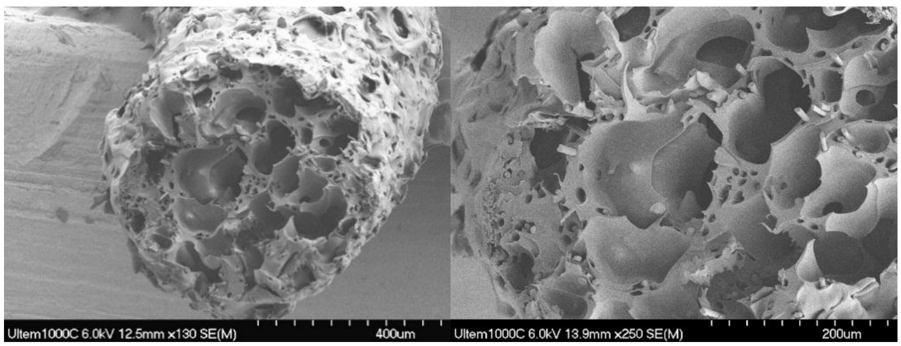


Figure 9. Picture and micrograph of fiber-filled Ultem 1000 filaments and printed cubes

2) A second drying trial for fiber-filled Ultem 1000 was conducted at 204 °C (400 ºF) for 22 hours. SEM micrographs in Fig. 10 showed that the dried thick filaments still had large pores of voids which were formed by the trapped water, air or other gases within the filaments during extrusion and drying (Fig. 10a), as well as the small pores of fiber pull-out at the fracture surface. Furthermore, Fig. 10b revealed that the porosity of FDM-spun thin filaments was much higher than the thick filaments as confirmed by acid digestion values listed in Table 5. The severe porosity of thin filaments were the results of volume expansion of trapped water vapors, air bubbles or other gases generated from the degradation of Ultem 1000 resin exposed to the sudden high liquefying temperature of 420 °C (788 °F) used to spin it within the FDM machine.



a) Thick filament after drying at 204 ºC (400 ºF) for 22 hours



b) FDM-spun thin filaments derived from 204 ºC dried thick filaments

Figure 10. SEM of fiber-filled Ultem 1000 filament after drying at 204 °C for 22 hours

Table 5. Porosity of Various Dried Fiber-Filled Ultem 1000 Filaments

Sample ID	FWF, wt%	FVF, v%	Porosity,v%
1st Gen Composite Vanes (received @ 6/18/14)			
Horizontal Vane	*9.6%*	*5.2%*	*26.1%*
Vertical Vane	*8.6%*	*4.8%*	*22.7%*
As-rec Filament, thick	9.2%	6.8%	-1.2%
FDM extruded, thin	9.3%	4.7%	30.9%
185 °C dried filament and cubes (samples received @ 8/25/14)			
As-dried Filament, thick	10.1%	6.7%	8.5%
Dried & extruded, thin	10.0%	5.3%	28.3%
DR1 Cube	9.6%	6.0%	13.6%
DR2 Cube	9.5%	5.7%	17.4%
204.4 °C (400 °F) dried filament (samples received @ 10/17/14)			
As-dried Filament, thick	8.5%	5.2%	16.5%
Dried & extruded, thin	5.2%	2.8%	24.5%
149 °C (300 °F) dried filaments but in a desiccant system, received @ 12/8/14			
As-received from Stratasys	9.6%	5.9%	15.3%
As-dried Filament, thick	9.5%	5.9%	15.0%
Dried & extruded, thin	10.2%	5.5%	25.6%

3) A third drying process was conducted in a desiccant system at 149 °C (300 °F) for 12 hours and characterized along with another section of as-received thick carbon-filled Ultem 1000 filaments as a repeat to investigate the uniformity and porosity of the experimental composite filaments supplied by Stratasys. As shown in Table 5, the porosity of the fiber-filled Ultem 1000 thick filaments as-received for the second trial was 15.3% which is much higher than the void-free in the initial as-received filaments. This clearly indicated that the porosity of experimental fiber-filled Ultem 1000 filaments varied considerably from 0-15.3%, and the porosity remained at 15% even after drying at 149 °C for 12 hours (Fig. 11). The photomicrographs in Fig. 10 also confirmed that as-received fiber-filled Ultem 1000 filaments exhibited some porosity this time, in contrast to the void-free segment shown in Fig. 4 previously. Since the moisture content of the as-received Ultem 1000 composite filaments was only 0.6 %, including 0.2 % moisture from as-received chopped fibers, the voids shown in the as-received fiber-filled Ultem 1000 thick filaments could either come from moisture trapped inside or the air bubbles introduced during the extrusion process. Nevertheless, after drying at various conditions, all the FDM-extruded thin filaments still displayed consistent porosity of ~25%, which is similar to porosity of printed composite vanes, but lower than the 30% porosity detected in the FDM-extruded thin filaments derived from the undried thick filaments (Table 5). This fact clearly indicated that once the moisture pore or air bubbles formed within the filaments, they are much more difficult to remove than the surface water

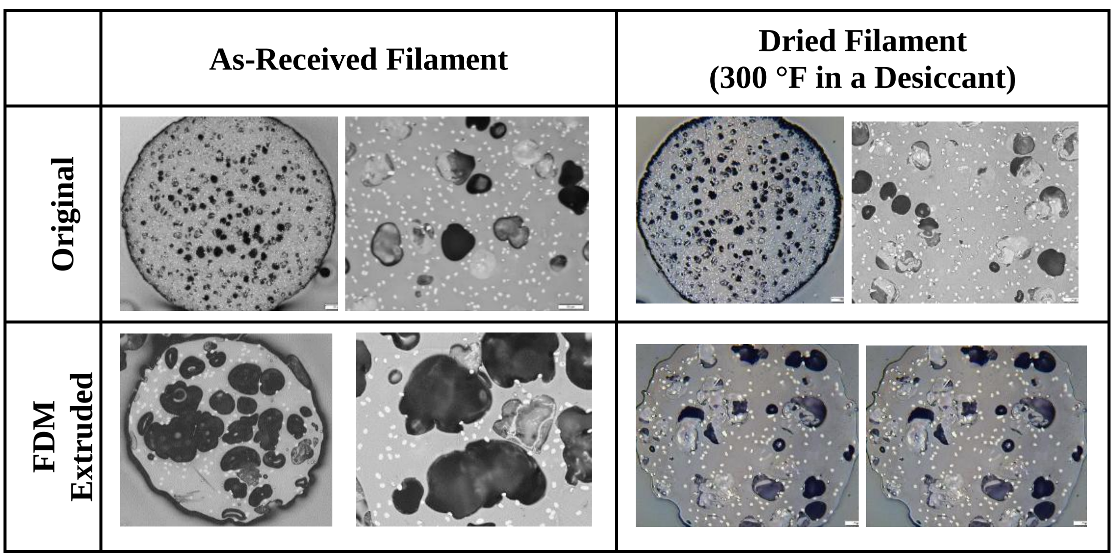


Figure. 11. Optical Micrographs of as-received and dried fiber-filled Ultem 1000 filaments

3.4 Mechanical Properties of FDM-Printed Ultem Resin and Ultem 1000 Composites

Tensile tests of FDM-printed Ultem 9085, Ultem 1000, XH6050 and fiber-filled Ultem 1000 coupons were conducted at room temperature and 204 °C (400 °F) as listed in Table 6. Fig. 12 showed that Ultem 1000/C-fiber composite printed with the dried FDM filament showed the highest modulus while Ultem 9085 resin showed the highest toughness and strength at room temperature. The carbon fiber reinforcement Ultem 1000 was estimated to increase the tensile strength by 23% and modulus by 38% while the strain-to-failure ration dropped by 55%, backcalculated based on XH6050 data of injection-molded versus FDM-printed. Regardless of test temperature, Ultem XH6050 showed inferior properties than either Ultem 1000 composites or Ultem 9085 (T_g =186 °C), despite of its higher T_g (245 °C). XH6050 also showed significant losses in toughness and strength at 204 °C (400 °F). Thermal analysis results in Table 7 showed that substantial moisture still trapped within the composites even after drying.

Table 6. Mechanical Properties of Ultem 9085, Ultem 1000, XH6500, Fiber-filler Ultem 1000

Material	Process	Printing Orientation	Tensile Properties at RT (23 °C)				
			Modulus MPa	Strength MPa	Strain-to-Failure, %	Poisson's Ratio	Data Source
ULTEM 9085	Injection Molded	n/a	3,432 ± n/a	83 ± n/a	72 ± n/a	n/a ±	Sabic
	FDM by Stratasys	0°	2,200 ± n/a	72 ± n/a	6.0 ± n/a	n/a ±	Stratasys
	FDM by rp+m	± 45°	2,230 ± 12	62 ± 0.1	6.1 ± 0.4	0.38 ± 0.02	GRC
ULTEM 1000	Injection Molded	n/a	3,579 ± n/a	110 ± n/a	60 ± n/a	n/a ±	Sabic
ULTEM 1000 + 10wt% AS4 C-fiber: As-received filament	FDM by rp+m	0°	2,901 ± 48	50 ± 0.9	2.4 ± 0.1	0.33 ± 0.01	GRC*
	FDM by rp+m	± 45°	2,248 ± 46	44 ± 0.3	2.8 ± 0.1	0.39 ± 0.01	GRC*
ULTEM 1000 + 10wt% AS4 C-fiber: Dried filament at 300°F	FDM by rp+m	0°	3,132 ± 20	52 ± 2.0	2.1 ± 0.0	0.35 ± 0.02	GRC*
	FDM by rp+m	± 45°	2,835 ± 177	53 ± 1.8	2.7 ± 0.1	0.34 ± 0.02	GRC*
ULTEM XH6050	Injection Molded	n/a	3,511 ± n/a	96 ± n/a	25 ± n/a	n/a ±	Sabic
	FDM by rp+m	0°	2,069 ± 190	36 ± 4.7	2.2 ± 0.3	0.33 ± 0	GRC*
	FDM by rp+m	± 45°	1,938 ± 105	35 ± 4.8	2.2 ± 0.5	0.38 ± 0	GRC*
			Tensile Properties at 400 °F (204 °C)				
ULTEM 1000 + 10wt% AS4 C-fiber: As-received filament	FDM by rp+m	0°	1,920 ± 94	11.4 ± 0.5	5.2 ± 3.4	0.32 ± 0.08	GRC*
	FDM by rp+m	± 45°	1,456 ± 143	9.3 ± 0.5	5.2 ± 3.7	0.33 ± 0.04	GRC*
ULTEM 1000 + 10wt% AS4 C-fiber: Dried filament at 300°F	FDM by rp+m	0°	1,951 ± 119	11.4 ± 1.3	9.2 ± 3.7	0.30 ± 0.06	GRC*
	FDM by rp+m	± 45°	1,197 ± 82	5.8 ± 2.3	43.5 ± 7.8	0.35 ± 0.03	GRC*
ULTEM XH6050	FDM by rp+m	0°	1,497 ± 26	9.4 ± 0.6	0.65 ± 0.1	0.34 ± 0.1	GRC*
	FDM by rp+m	± 45°	1,367 ± 123	8.2 ± 1.1	0.6 ± 0.1	0.41 ± 0.1	GRC*

** All GRC testing used 0.2 in/min CHS and averaged out of three repeat runs.*

- No Ultem 1000 filament for FDM is commercially available currently.

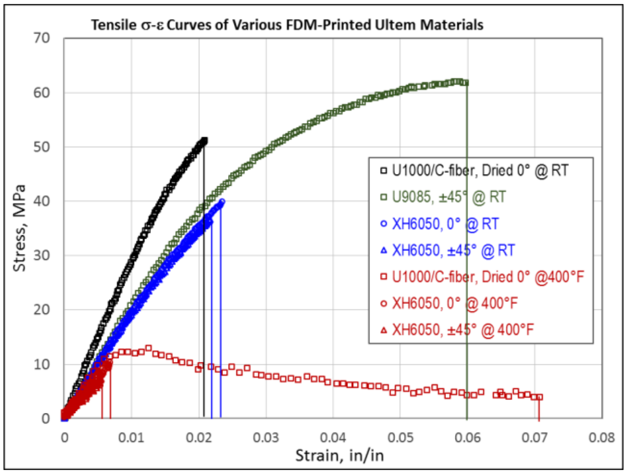


Figure 12. Tensile Properties of FDM-printed Ultem resins and fiber-filled Ultem 1000

Table 7. Thermal analysis of fiber-filled Ultem 1000 Composites

Sample ID	mDSC Tg, °C Total Heat	mDSC Tg, °C Rev. Heat	TGA (under N2 gas) Td, °C	ΔWt% RT-100 °C	ΔWt% 100 - 300 °C	ΔWt% @ 750 °C	Char yield
1st Gen Composite Vanes (received @ 6/18/14)							
Horizonal vane, top	214	217	556	0.298	0.389	42	58
Horizonal vane, bottom	213	213	560	0.326	0.340	42	58
Vertical vane, top	213	217	558	0.332	0.303	42	58
Vertical vane, bottom	213	217	556	0.261	0.479	41	59
As-received Filament			563	0.074	0.580	40	60
FDM-spun Filament			550	0.500	0.574	40	60
185°C dried filament and cubes (samples received @ 8/25/14)							
As-dried Filament	214	217	563	0.110	0.471	41	60
FDM-spun Filament	213	215	560	0.403	0.178	37	63
DR 1	213	216	559	0.325	0.078	44	56
204.4 °C (400 °F) dried filament (samples received @ 10/17/14)							
As-dried Filament			565	0.050	0.199	43	57
FDM-spun Filament			557	0.313	0.387	43	57
149 °C (300 °F) dried filaments but in a desiccant system, received @ 12/8/14							
As-received Filament	215	220	554	0.094	0.331	42.5	57.5
As-dried Filament	214	216	554	0.062	0.374	42.2	57.8
As-dried & extruded		215	546	0.154	0.438	45.9	54.1

More specifically, drying FDM filament prior to FDM-printing improved the room temperature (RT) properties of Ultem 1000 composites considerably, as indicated by the reduced porosities of FDM-extruded thin filaments from 30% to 24% after various drying conditions (Table 5), especially ±45° samples, even though its residual moisture content was still high. As shown in Fig.13, the 0° sample, showed ~ 8% increase in modulus, ~ 3% increase in strength, but ~ 11% decrease in strain-to-failure. The ±45° sample displayed ~26% increase in modulus, ~ 20% increase in strength, but ~ 2.4% decrease in strain-to-failure. At 204°C which is near Ultem1000's T_g (217°C), all the properties decreased considerably due to the softening of the resin.

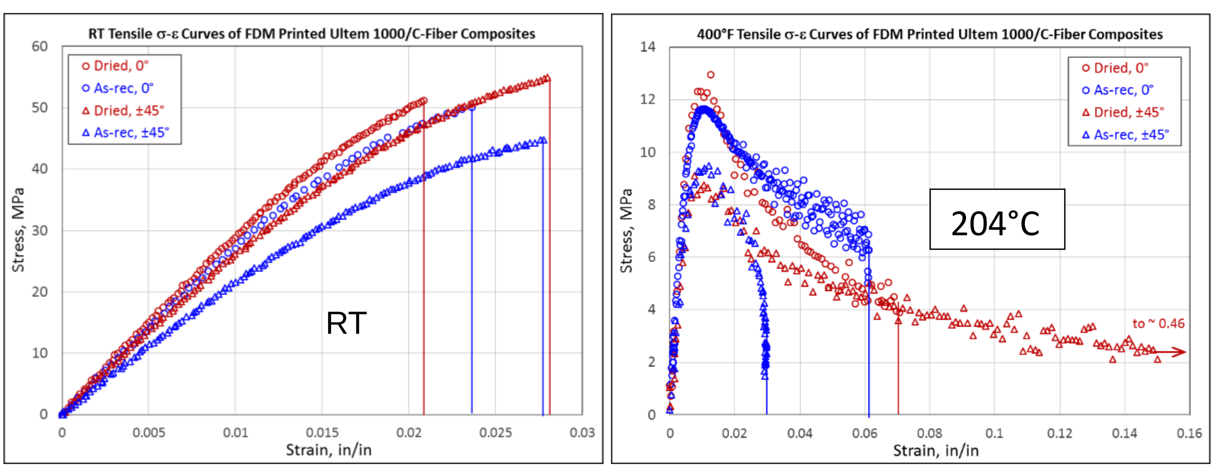


Figure 13. Tensile properties of as-received and dried fiber-filled Ultem 1000 composites

3.5 Characterization of Ultem 9085 versus Ultem 1000

An effort was undertaken to understand why FDM-printed fiber-filled Ultem 1000 composites exhibited an average of 25% porosity whereas Ultem 9085 displayed only 5-8% porosity associated with the inherent FDM process. Acid digestion of the as-received Ultem 9085 neat resin filaments showed 1.8-3.5% porosity and optical micrograms in Fig. 14 indicated that both as-received Ultem 9085 filaments and thin filaments extruded at 375 °C by FDM exhibited no porosity. The discrepancy between two methods depends on the segments of the filaments analyzed in each technique, and subjected to variable porosity. Thermal analysis of Ultem 9085 revealed that there was 0.3-0.4 % weight loss between 100-300 °C due to moisture presence in the as received filaments, which is common among all the moisture sensitive polyetherimides.

Table 8. Analysis of Ultem 9085 Resin Filaments by Acid Digestion

Sample ID	Sample conditions	Drying		Mass (g)	Pycnometer	Density (g/cc)	Porosity (%)
		T (°C)	t (hr)		Volume (cc)		
Ultem 9085	**As-received Filament**	120	24	0.3972	0.302	1.3152	1.85%
		165	24	0.3963	0.3066	1.2926	3.54%
	Extruded Filament	120	24	0.0743	0.0505	1.4713	-9.80%
		165	24	0.0742	0.0506	1.4664	-9.43%

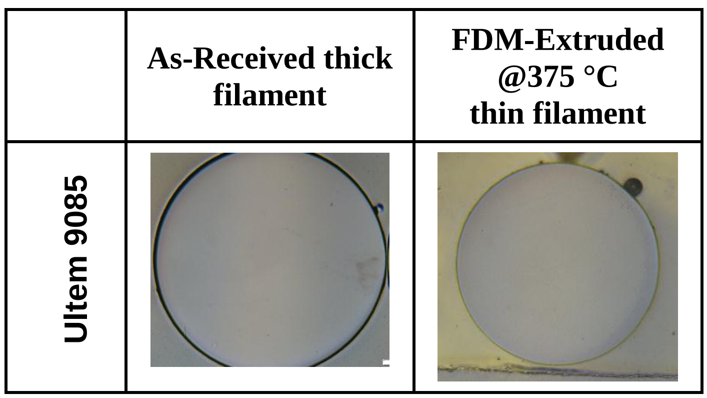


Figure 14. Optical micrographs of Ultem 9085 resin filaments

As shown in Fig. 15, the complex viscosity of Ultem 1000 is ~3 fold higher than that of Ultem 9085 at 350 °C. Basically, the high viscosity of Ultem 1000 raised the printing temperature of fiber-filled Ultem 1000 to 420°C, causing the volume expansion of trapped moisture and degradation gases, which in terms increased the porosity of the FDM product, as compared to Ultem 9085 that generally printed at 375 °C with lower porosity (5-8%). The as-received fiber-filled Ultem 1000 composite filaments with varied porosity of 0-15% clearly warranted more process improvement. One possible solution is to produce Ultem 1000 with controlled molecular weight that exhibits similar viscosity profile as that of Ultem 9085 to enable printing at 375-380 °C; thus, avoiding polymer degradation and ensuring production of high quality 3D objects with low porosity.

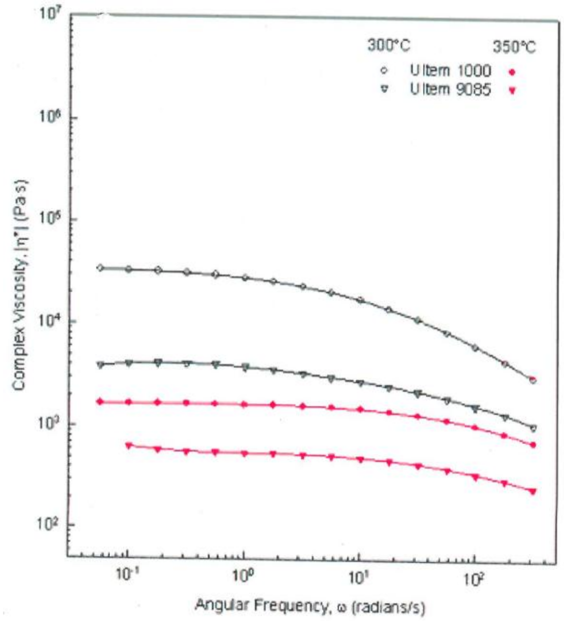


Figure 15. Complex viscosity of Ultem 9085 and Ultem 1000
[Adapted from Fig. 7 in Ref. 5]

4. CONCLUSIONS

To advance the state-of-the-art in additive manufacturing via Fused Deposition Modeling (FDM) beyond the commonly used ABS, polycarbonate and Nylons as prototyping for 100-125 °C use, this project aimed at producing aircraft engine components by FDM, using Ultem 9085 and cabon fiber-filled Ultem 1000 composite filaments with higher temperature (130-175 °C) capability.

A perforated access engine door and acoustic liners, simulating conventional honeycomb structures, were printed with Ultem 9085 to modulate the sound wave and reduce noises. Composite engine inlet guide vanes were printed using Ultem 1000 filled with 10% AS4 chopped fibers as a reinforcement to eliminate the need for machining when using conventional polymer prepregs to make vanes. Preliminary data indicated that the FDM-printed Ultem 9085 exhibited about 84% of its original strength and 64% of its original modulus as compared to its injection-molded counter parts. The incorporation of 10% chopped fiber into Ultem 1000 increased the tensile strength by 23% and modulus by 38%, but also made the resulting composites more brittle. The experimental fiber-filled Ultem 1000 filaments (as received) contained 0-15% varied porosity. However, the FDM extruded thin filaments and FDM printed Ultem 1000 composite vanes exhibited ~25% porosity, due to the volume expansion of trapped moisture, air or other gases generated form degradation at elevated printing temperature of 420 °C by FDM. In contrast, the Ultem 9085 resin filament is a high quality commercial product that manufactures 3D objects with only 5-8% porosity inherently associated with the FDM process when printed at 375 °C.

In summary, this project proved the feasibility of printing integrated complex aircraft parts with polymers by FDM. FDM printing compared favorable to bonded honeycomb structures with face sheets in acoustic liners. However, printing composite parts by FDM is still considered experimental, as in the case of this effort to print Ultem 1000 composite vanes. Incorporation of 10% of chopped fibers into Ultem 1000 raised the viscosity significantly that affected the compounding efficiency in the extruder, resulting in high porosity in the extruded filaments and FDM-printed composite objects. In light of conventional polymer composites with 65% fiber content, additive manufacturing only looks favorable for printing intricate parts that are difficult to manufacture by conventional methods. In order to increase the fiber content and reduce porosity in polymer composites, it might be worthwhile to look into printing composite structures using thermoset polyimides with higher temperature performance and lower viscosity by selective laser sintering (SLS) for future works.

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6. REFERENCES

1. Ludmila Novakova-Marcincinova, Ivan Kuric: “Basic and Advanced Materials for Fused Deposition Modeling Rapid Prototyping Technology”, Manuf. and Ind. Eng., <u>11(1)</u>, 25-27 (2012), ISSN 1338-6549.
2. Water C. Smith and Richard W. Dean: “Structural Characteristics of Fused Deposition Modeling Polycarbonate Material”, Polymer Testing, <u>32(8)</u>, 1306-12 (2013).
3. Joseph E. Grady, William J. Haller, Phil Poinsatte, Michael C. Halbig, Sydney Schulo, M. Singh, Don Weir, Natalie Wali, Michael Vinup, Michael G. Jones, Clark Patterson, and Tom Santelle, “A Fully Non-Metallic Gas Turbine Engine Enabled by Additive Manufacturing: *Part I: System Analysis, Component Identification, Additive Manufacturing, and Testing of Polymer Composites”,* NASA TM-2015-218748, Glenn Research Center, Cleveland, OH (2015).
4. Angnes Bagsik and Volker Shoppner: “Mechanical Properties of Fused Deposition Modeling Parts Manufactured with Ultem 9085”, Proceedings of ANTEC Conference, May 1-5, Boston, MA (2011).
5. Thomas W. Hughes, Roger Avakian, Ling Hu and Kathy C. Chuang: “Reactive Extrusion of High Temperature Resins for Additive Manufacturing”, Proceedings of SAMPE Technical Conference, June 2-4, Seattle, WA (2014).