

Discontinuous Fiber Thermoplastic Composite Qualification Framework Development

John Tomblin, Rachael Andrulonis, Royal Lovingfoss & Brandon Saathoff

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Federal Aviation Administration



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Presented by:

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JAMS 2023 Technical Review – Seattle, WA



DFC Research Team

NIAR

- John Tomblin, PhD •
- **Rachael Andrulonis**
- **Royal Lovingfoss**
- **Brandon Saathoff**



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Research Partners:



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WSU-NIAR & University of Washington Collaboration

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DFC Background and Research Motivation

DFCs are attractive for manufacturing low ulletcost complex parts.

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- Thermoset and thermoplastic polymers have been used in discontinuous fiber composite applications. Thermoplastic polymers are the most attractive due to:
 - Reduction of manufacturing cycle time
 - Increased performance (fracture toughness, chemical resistance, etc.)
- Widespread adoption of discontinuous fiber composites for structural applications is limited:
 - Inherent challenges with predicting and characterizing the material properties
 - Lack of statistically meaningful material property data used for design especially with various flow behaviors
- JAAS CECAM
- Level of conservatism in design unknown



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Project Overview

TORAY

Overall Goals

- Develop a framework for the qualification of new and innovative • thermoplastic composite material systems including guidelines and recommendations for their characterization, testing, design, and utilization.
- Second goal: To transition the test data and guidelines generated in this • program into shared databases, such as CMH-17.

Research Outputs

- **Trial tests** provide valuable lessons learned on temperature effects of key mechanical properties, test methods best suited for thermoplastic composites, and effects of key processing parameters
- First public qualification of a continuous fiber thermoplastic **composite** with material and process specifications
- Lessons learned, guidelines, and data made available to CMH-17
- Qualification framework for chopped (discontinuous) fiber thermoplastic composites



Continuous Fiber Thermoplastic Qualification

(T700/TC1225) & Equivalency Toray Advanced Composite



Establishing

Committee

Steering

CMH17

COMPOSITE MATERIALS HANDBOOK



Discontinuous Fiber Thermoplastic Trials » Qualification





Development of Qualification Framework for DFCs

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Other (pl

40% 50% 60% 70% 80% 90% 100

 Evaluate appropriate test methods, specimen sizes, environments, and failure modes

High Flow Panel Size Description – 13.5"x1.5"

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High Flow Panel Size Description – 7.0"x6.5"

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High Flow vs. Low Flow Panel Visual Comparison

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Trial Test Matrix – <u>Square</u> Platelet

- Solvay APC PEKK-FC AS4 Unitape Prepreg Material
 - Areal Weight: 145 gsm
 - Resin Content: 34%
- Panel Fabrication at Sekisui Aerospace
 - Qforge[™] Process

Platelet Size	Flow Behavior	Thickness
Square	High Flow	Thickness 1 (0.15")
(0.5"x0.5")	Low Flow	Thickness 2 (0.25")
	Square Platelet 0.5" x 0.5"	

	0.5" x 0.5" Platelet Test Matrices																			
Test Matri	x F	low	Thick- ness	Platelet Size	Coupon Group	Test Type	Geometry	Test Method	CTA -65F	RTA	ETA 180F	ETW 180F	ETA 350F	ETW 350F	Panel Size	Total # of Panel <u>s</u>	Panels Receiv <u>ed</u>			
					1	Tension	12" x 1.5"	ASTM D3039 (DIC at RT)	6	6	6	6	6	6	13" x 1.5"	36	36			
					2	Tension (L)	7" x 1.5"	ASTM D3039 (DIC at RT)	6	6	6	6	6	6	7" x 6.5"	9	9			
					2*	Tension (T)	6.5" x 1.5"	ASTM D3039 (DIC at RT)		6					7" x 6.5"					
					3	Compression	12" x 1.5"	ASTM D6484 with no hole	6	6	6	6	6	6	13" x 1.5"	36	36			
					4	Compression (L)	7" x 1.5"	ASTM D6484 with no hole	6	6	6	6	6	6	7" x 6.5"	9	9			
					4*	Compression (T)	6.5" x 1.5"	ASTM D6484 with no hole		6					7" x 6.5"					
					5	Shear	3" x 1.5 " (mod)	ASTM D7078 with DIC		6				00000	13" x 1.5"	2	2			
					6	Shear (L)	3" x 2.2 "	ASTM D7078 with DIC		6					7" x 6.5"	2	2			
					6*	Shear (T)	3" x 2.2 "	ASTM D7078 with DIC		6					7" x 6.5"					
					7	Flex	5.5" x 0.5"	ASTM D790, 3 pt bend		6					13" x 1.5"	3	3			
					8	Flex (Wide)	5.5" x 1.5"	ASTM D790, 3 pt bend		6					13" x 1.5"					
					8	Flex	5.5" x 0.5"	ASTM D790, 3 pt bend		6					7" x 6.5"	1	1			
Iteratio	n 4	High	0.15"	0.5" x 0.5"	8*	Flex (Wide)	5.5" x 1.5"	ASTM D790, 3 pt bend	11111	6					7" x 6.5"					
					9	OHT	12" x 1.5"	ASTM D5766, hole: 0.25"		6					13" x 1.5"	6	6			
					10	OHT	7" x 1.5"	ASTM D5766, hole: 0.25"	111116	6					7" x 6.5"	2	2			
					11	OHT	12" x 1.5"	ASTM D5766, hole: 0.75"		6					13" x 1.5"	6	6			
					12	OHT	7" x 4.5"	ASTM D5766, hole: 0.75"		6					7" x 6.5"	6	6			
					13	OHT	12" x 1.5"	ASTM D5766, hole: 1"		6					13" x 1.5"	6	6			
					14	OHT	7" x 6"	ASTM D5766, hole: 1"	VIIIII	6					7" x 6.5"	6	6			
					15	OHC	12" x 1.5"	ASTM D6484, hole: 0.25"	VIIIII	6					13" x 1.5"	6	6			
					16	OHC	7" x 1.5"	ASTM D6484, hole: 0.25"		6					7" x 6.5"	3	3			
					19	SSB	5.5" x 1.5"	ASTM D5961 pr. C, 0.25"d		6					13" x 1.5"	4	4			
							20	SSB	5.5" x 1.5"	ASTM D5961 pr. C, 0.25"d		6					7" x 6.5"	4	4	
						21	CAI	6" x 4"	ASTM D7136/D7137	<i>Millin</i>	6					7" x 6.5"	6	6* (6)		
					22	CAI	6" x 4"	ASTM D7136/D7137		6					7" x 6.5"	6	6			
				0.5" x 0.5"	1	Tension	12" x 1.5"	ASTM D3039 (DIC at RT)		6	6		6		12"x12"	3	3			
Iteratio	n 6	Low	0.25"		2	Compression	12" x 1.5"	ASTM D6484 with no hole		6					12"x12"	1	1			
liorado			0.20		3	Shear	3" x 2.2 "	ASTM D7078		6					12"x12"	1	1			
					4	OHT	12" x 1.5"	ASTM D5766, hole: 0.25"	00000	6					12"x12"	1	1			
					1	Tension	12" x 1.5"	ASTM D3039 (DIC at RT)	6	6	6	6	6	6	12"x12"	6	3 (3)			
									3	Compression	12" x 1.5"	ASTM D6484 with no hole	6	6	6	6	6	6	12"x12"	6
					5	Shear	3" x 2.2 "	ASTM D7078		6					12"x12"	1	1			
					6	Flex	5.5" x 0.5"	ASTM D790		6					12"x12"	1	1			
	-				7	Flex (Wide)	5.5" x 1.5"	ASTM D791		6					12"x12"					
Iteratio	n 8	Low	0.15"	0.5" x 0.5	7	OHT	12" x 1.5"	ASTM D5766, hole: 0.25"		6					12"x12"	1	1			
					8	OHT	12" x 4.5"	ASTM D5766, hole: 0.75"	VIIII	6					12"x12"	3	3			
					9	OHT	12" x 6"	ASTM D5766, hole: 1"		6					<u>12"x12"</u>	3	3			
					10		12" x 1.5"	ASTM D6484, hole: 0.25"		6					<u>12"x12"</u>		1			
ALL CAR					12	SSB	5.5" x 1.5"	ASTM D5961 pr. C, 0.25"d		6					12"x12"	1	1			
					13	ICAI	6" x 4"	ASTM D7136/D7137	111111	6			<u>VIIIIII</u>	<i>V////////////////////////////////////</i>	12"x12"	3	3			
										Т	est Con	pleted			Marc	h Shipment	51			
											In-Prog	ress			Ар	ril Shipment	68			
										On-Ho	ld / Pen	ding Re	view		Ма	y Shipment	50			
											Not Sta	arted			Jur	e Shipment	18			
									4	Additiona	l Testin	g Outlin	ed Blue	9	Augu	st Shipment	6			
															High Flow E	xtra Panels	3			
															Low Flow E	xtra Panels	2			



August Shipment

High Flow Extra Panels

Low Flow Extra Panels

Trial Test Matrix – <u>Narrow</u> Platelet

- Solvay APC PEKK-FC AS4 Unitape Prepreg Material
 - Areal Weight: 145 gsm
 - Resin Content: 34%
- Panel Fabrication at Sekisui Aerospace
 - Qforge[™] Process

	0.5" x 0.0625" Platelet Test Matrices																
Test Matrix	Flow	Thick- ness	Platelet Coupon Size Group Test Type		Geometry	Test Method	CTA -65F	RTA	ETA 180F	ETW 180F	ETA 350F	ETW 350F	Panel Size	Total # of Panels	Panels Received		
				1	Tension	12" x 1.5"	ASTM D3039 (DIC at RT)		6	6		6		13" x 1.5"	19	21	
				2	Tension (T)	6.5" x 1.5"	ASTM D3039 (DIC at RT)		6	6		6		7" x 6.5"	5	5	
				2*	Tension (L)	7" x 1.5"	ASTM D3039 (DIC at RT)		4					7" x 6.5"	1	1	
				3	Compression	12" x 1.5"	ASTM D6484 with no hole		6					13" x 1.5"	6	6	
Iteration 3	High	0.15"	0.5" x	4	Compression (T)	6.5" x 1.5"	ASTM D6484 with no hole		6					7" x 6.5"	2	2	
iteration 5	lingu	0.13	0.0625"	4*	Compression (L)	7" x 1.5"	ASTM D6484 with no hole		4					7" x 6.5"	2	2	
				5	Shear	3" x 1.5 " (mod)	ASTM D7078 with DIC		6					13" x 1.5"	2	6	
				6	Shear	3" x 2.2 "	ASTM D7078 with DIC		6					7" x 6.5"	2	2	
					7	OHT	13" x 1.5"	ASTM D5766, hole size 1 (0.25")		6					13" x 1.5"	6	6
				8	OHT (Tranv.)	6.5" x 1.5"	ASTM D5766, hole size 1 (0.25")		6					7" x 6.5"	2	2	
					1	Tension	12" x 1.5"	ASTM D3039 (DIC at RT)		6	6		6		12"x12"	3	3
Iteration 5		0.25"	0.5" x	2	Compression	12" x 1.5"	ASTM D6484 with no hole		6					12"x12"	1	1	
iteration 5		0.25	0.0625"	3	Shear	3" x 2.2 "	ASTM D7078 with DIC		6					12"x12"	1	1	
				4	OHT	12" x 1.5"	ASTM D5766, hole size 1 (0.25")		6					12"x12"	1	1	
				1	Tension	12" x 1.5"	ASTM D3039 (DIC at RT)		6	6		6		12"x12"	3	4	
Iteration 7		0.15"	0.5" x	2	Compression	12" x 1.5"	ASTM D6484 with no hole		6					12"x12"	1	1	
		0.15	0.0625"	3	Shear	3" x 2.2 "	ASTM D7078		6					12"x12"	1	1	
				4	OHT	12" x 1.5"	ASTM D5766, hole size 1 (0.25")		6					12"x12"	1	1	

Test Completed

In-Progress

On-Hold / Pending Review

Not Started

Additional Testing Outlined Blue





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Non-Destructive Inspection Techniques

Through-Transmission Ultrasonic Inspections

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Summary of TTU C-Scan Results

















Square Platelet (0.5" x 0.5") – XRCT Process Comparisons



Square and Narrow Platelet High Flow Micrograph Comparison





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Fiber alignment in flow direction at edge of specimen



Square and Narrow Platelet Low Flow Micrograph Comparison







Thermal Analysis – Glass Transition Temperature

• **ASTM D7028** – "Standard Test Method for Glass Transition Temperature (DMA Tg) of Polymer Matrix Composites by Dynamic Mechanical Analysis (DMA)"

Platelets	Flow	Thick- ness	Panel Size	Sample ID	Onset Storage Modulus Tg [°F]	Peak of Tangent Delta Tg [°F]
				4-1-1	289.11	331.86
			13" x 1.5"	4-1-2	290.21	332.22
	Link	0.45"		4-1-3	288.72	330.94
	High	0.15	7" x 6.5"	8-13-1	286.52	331.70
Square 0.5" x 0.5"				8-13-2	278.98	326.79
				8-13-3	284.65	331.93
		0.25"		6-1-1	289.35	333.43
	1	0.15"	12" x 12"	8-1-1	288.57	332.62
	LOW			8-1-2	289.67	333.68
				8-1-3	286.93	331.74
Narrow		0.45	13" x 1.5"	3-5-1	278.47	326.17
0.5" x	High	0.15"	7" x 6.5"	3-6-2	283.73	325.54
0.0625"	Low	0.15"	12" x 12"	7-3-1	282.27	324.36
	•		Ave	Average [°F]		330.23
			Stadard	Deviation	4.02	3.25
AC				C.V. [%]	1.41%	0.99%









Thermal Analysis – Crystallinity

ASTM D3418 – "Standard Test Method for Transition Temperatures and Enthalpies of Fusion and Crystallization of Polymers by Differential Scanning Calorimetry" •

Platelets	Flow	Thick- ness	Panel Size	Sample ID	Melting Peak Temperature [°F]	Enthalpy of Melting ΔHm [J/g]	Crystallizatio n Peak Temperature [°F]	Enthalpy of Crystallization ΔHc [J/g]	Degree of Crystallinity [%]
				4-1-1	648.64	9.686	559.17	12.794	21.91
			13" x 1.5"	4-1-2	645.04	8.557	560.48	12.095	19.36
	Lliab	0.15"		4-1-3	646.27	11.297	559.71	11.368	25.56
	Figh	0.15		8-13-1	644.74	11.794	550.72	12.331	26.68
Square 0.5" x 0.5"			7" x 6.5"	8-13-2	651.54	13.586	550.24	12.766	30.74
				8-13-3	650.14	12.194	552.83	12.962	27.59
		0.25"	12" x 12"	6-1-1	649.51	10.873	552.40	13.528	24.60
	Low	0.15"		8-1-1	650.86	13.159	553.95	13.856	29.77
	LOW			8-1-2	651.20	12.577	553.89	12.997	28.45
				8-1-3	650.26	12.897	553.66	13.259	29.18
				3-5-1	643.59	11.228	562.33	13.441	25.40
			13" x 1.5"	3-5-2	648.07	11.732	562.59	13.235	26.54
	Lligh	0.15"		3-5-3	651.02	10.345	561.83	14.720	23.40
Narrow	Figh	0.15		3-6-1	651.76	13.873	565.38	14.707	31.39
0.5" x			7" x 6.5"	3-6-2	653.29	13.032	565.32	13.435	29.48
0.0625"				3-6-3	653.00	14.560	565.63	15.142	32.94
				7-3-1	650.86	13.159	553.95	13.858	29.77
	Low	0.15"	12" x 12"	7-3-2	651.20	12.577	553.89	12.997	28.45
				7-3-3	650.26	12.897	553.66	13.259	29.18
								Average [%]	27 39



- ΔH_f : Enthalpy of Fusion / Melting
- ΔH_{cc} : Enthalpy of Cold Crystallization
- ΔH_f^{o} : Theoretical Maximum Enthalpy of Fusion for Fully
- Crystalline Sample (130 J/g)
- R_c : Resin Content (**34%**)

Average [/0] Stadard Deviation 3.41





Summary of Panel Fiber Volume & Void Content

Fiber & Void Content

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- ≈1" x 0.5" samples (*target≈1.5-grams*) extracted from each panel (locations documented for each panel)
 - ASTM D792-13 "Standard Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement"
 - ASTM D3171-15 "Standard Test Methods for Constituent Content of Composite Materials" (Method I, Procedure B)
 - ASTM D2734-16 "Standard Test Methods for Void Content of Reinforced Plastics"
- Density:
 - Resin: 1.31 g/cc (%crystalline=30%)
 - Fiber: 1.79 g/cc

Platelet	Flow	Property	Average	Specimen Qty						
	Lliab	Fiber Volume [%]	56.863	100						
$S_{\text{cutors}} \left(0 \in \mathbb{N} \setminus 0 \in \mathbb{N} \right)$	пıgri	Void Content Volume [%]	2.249	123						
	Low	Fiber Volume [%]	57.216	20						
	LOW	Void Content Volume [%]	1.378	20						
	Lliab	Fiber Volume [%]	56.175	4.4						
Norrow $(0.5" \times 0.0625")$	nign	Void Content Volume [%]	2.294	44						
Natiow (0.5 X 0.0625)		Fiber Volume [%]	58.097	11						
	LOW	Void Content Volume [%]	1.575	11						
				1						

Square and Narrow Platelet Comparisons



Panel ID: 8-1-3





MIST



Mechanical Test Methods





Mechanical Testing – Key Takeaways

• General:

• Specimen Sizing

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- Cross sectional area must be sized considering platelet size
- Tension/Compression sized with 3X platelet size (0.5" platelet size \rightarrow 1.5" coupon width)
- Strain Measurement Techniques
 - Large strain gradients associated with DFCs
 - Strain Gauge grid length of the strain gauge must be significantly larger than the inhomogeneity present in the strain field
 - All work focused on DIC and Extensometers
 - Maximize gage length when using extensometers
- Flow behavior largely drives fiber orientation within part/panel and significantly effects mechanical performance
 - Varies for each mold







Mechanical Testing – Key Takeaways Cont.

Tension

- In the majority of the alike configurations tested, the narrow platelet material resulted in higher (average) strengths and moduli in comparison to the square platelet material
 - Most significant change in tensile strength and modulus for low flow panels

Open Hole Tension

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- Standard hole diameter of 0.25" (ASTM D5766) insignificant as stress concentrations inherent to the material were higher than stress concentration at hole
 - As hole diameter is increased beyond fiber length » stress concertation around hole more significant
- Bearing

Significant change in strength and failure mode between flow behaviors











Single-Shear Bearing Testing – (Square Platelet)





SSB Failure Mode Summary









Program Status & Summary

- Pre-Qualification Trial Testing
 - Completed trial test matrices for square and narrow platelet DFC material
 - Load/Strain rate sensitivity testing in-progress (DFC versus continuous fiber composite laminate)
 - Facilitating the transfer of test data to University of Washington. A shared file transfer portal was created and has been used for transferring test data.
- Qualification
 - Currently outlining test matrices and specifications to move toward qualification program
- Benefit to Aviation
 - Publicly available DFC data linked to M&P Specifications.
 - Lessons learned, guidelines for characterization, and data made available to CMH-17.



Room: 337

SAMPE 2023 Seattle Convention Center Summit Conference: April 17-20, 2023 Exhibition: April 18-19, 2023

Track: Composites Certification and Qualification

Title: Characterization Approach for Compression Molded Discontinuous



CECAM

Fiber Thermoplastic Composites Date: Thursday, 4/20/2023 Time: 11:30 AM - 11:55 AM

Questions/Comments: Contact Rachael – <u>Rachael.Andrulonis@idp.wichita.edu</u>

CHARACTERIZATION APPROACH FOR COMPRESSION MOLDED DISCONTINUOUS FIBER THERMOPLASTIC COMPOSITES

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ABSTRACT

Thermoplastic composites show potential in increasing the manufacturing production rate of composite aerospace structures. This is largely due to their ability to be consolidated quickly using automated processes. A variety of reinforced thermoplastic material forms are offered that can be processed multiple ways in order to meet structural performance requirements at the necessary production volumes without substantial compromise. Intrinsically, this requires generating a significant amount of statistically-based material property data for each unique material and process combination. Currently, the National Institute for Aviation Research (NIAR) and the Federal Aviation Administration (FAA) are developing a material qualification framework for compression molded discontinuous fiber thermoplastic composites in consensus with industry experts. To aid in the development of the qualification framework, a screening test matrix was formed to identify the key processing parameters and evaluate the appropriate test methods and specimen sizes. Three main variables were considered in the trial testing: reinforcement size, material flow behavior and panel thickness. The effect of these key processing parameters on the mechanical properties are discussed along with guidelines for testing and characterization.

Keywords: Discontinuous Fiber Composites, Thermoplastics, Material Testing and Characterization Corresponding author: Brandon L. Saathoff (bsaathoff@niar.wichita.edu)

1. INTRODUCTION

Increased global demand for lightweight aircraft structures has driven the need to accelerate the manufacturing production rates associated with composite aircraft. This has motivated manufactures to make significant investments integrating new materials and manufacturing methods into legacy factories to remain commercially viable and meet aggressive production rate goals. In the early stages of considering composite materials in structural components, a mix of testing and analysis is generally used to substantiate the structural performance and prove the material and manufacturing process selected are viable options for the application. The reliability and efficiency of this process is enhanced with a greater understanding of the material *Construction 2023 Uted the Societae of the Manufacture and the Societae and the Societae and the Societae and the Manufacture and the Societae and the Manufacture and the Societae and the Amountation and the societae and the Amountation and the societae and the Societae and the Amountation and the societae and the Manufacture and the Societae and the Manufactu*

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(DOI will be added by SAMPE)

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