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Manufacturing and Mechanical Testing of TC1225/LM-PAEK and TC1200/PEEK Thermoplastic Composite Panels

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Summary

Development of thermoplastic composites (TPCs) for aerospace structures is experiencing renewed enthusiasm attributed to the availability of rapid manufacturing technology, ease of joining through fusion welding processes, and the successful utilization of the material in flight critical structures. Semicrystalline thermoplastics such as polyether ether ketone (PEEK), polyether ketone ketone (PEKK), and polyphenylene sulfide (PPS) have been successfully demonstrated as thermoplastic skins, keel beams, and angle brackets for commercial aircraft.

Composite specimens were fabricated from the Toray TC1225 and TC1200 unidirectional prepreg systems and tested to gain confidence with NASA in-house TPC processing as part of the Thermoplastics Development for Exploration Application (TDEA) project. TC1225 is a relatively new material system that uses T700GC fibers and low-melt polyaryl ether ketone (LM-PAEK) resin. TC1200 is the latest version of a material system that has been available for several decades with AS4 fibers and PEEK resin. Results for ultrasonic inspection, optical microscopy, acid digestion, and differential scanning calorimetry establish the quality of the panels. Mechanical test data for unnotched and notched laminate tension and compression as well as compression after impact provide data for equivalency assessment and support material selection in TDEA. Satisfactory equivalence of the TC1225 results and the National Center for Advanced Materials Performance (NCAMP) database was found. The results indicate high-quality manufacturing was achieved, and the resulting mechanical test data are in agreement with that in the literature.

*Summer Intern in Lewis' Educational and Research Collaborative Internship Project (LERCIP).

Introduction

Carbon-fiber-reinforced polymer composite materials enable NASA's science and exploration missions where lightweight, high-performance structural solutions are required. Current state-of-the-art materials for composite aerospace structures use a thermosetting matrix, where the resin material undergoes a chemical reaction (often requiring autoclave, press, or oven curing), and cannot be remolded after the initial forming. Joints and interfaces must often contain mechanical fasteners, which can adversely add mass and alter structural performance (Ref. 1). Bonded interfaces are highly sensitive to surface preparation techniques and the bonding processes. Programs are often not able to take advantage of the structural efficiencies offered by composite structures because of characterization and certification challenges, particularly with respect to joints (Ref. 2).

Alternatively, thermoplastics are fully formed polymers and processable with heat and pressure alone. They can be reheated, remolded, and cooled as necessary without causing chemical changes and offer several process options for joining. Unitizing structures with thermoplastics can increase structural efficiency, simplify characterization and certification, and reduce conservatism of the design. The Thermoplastics Development for Exploration Applications (TDEA) project is focused on using thermoplastics to create large monolithic structures that save on manufacturing time, part count, and weight. Ultimately, this could enable in-space manufacturing of large monolithic space structures.

The overarching goals of TDEA will focus on developing thermoplastic composite (TPC) joints for space structures by developing and maturing design capabilities, analysis tools and techniques, and manufacturing processes for TPCs. As part of this effort, TDEA will develop NASA's TPC processing and manufacturing capabilities; develop an understanding of advanced thermoplastic joining techniques relevant to space environments and applicable to unitized and/or reconfigurable composite structures; and advance structural analysis capabilities for design and analysis, including failure prediction, of TPCs and also joints.

This report addresses the processing and mechanical testing of TPC pathfinder panels manufactured to (1) demonstrate manufacturing equivalency between NASA composite manufacturing facilities and the NCAMP database and (2) provide baseline composite properties to be used for application-orientated material downselection within the TDEA project.

For structural materials, the temperature, strength, and stiffness requirements for aerospace structures often limits thermoplastic selection to semicrystalline materials including polyether ether ketone (PEEK), polyether ketone ketone (PEKK), low-melt polyaryl ether ketone (LM-PAEK), and polyphenylene sulfide (PPS), all of which offer a balance of strength, stiffness, and durability by optimizing the material's degree of crystallinity (DOC) (Ref. 3). Crystalline regions of the thermoplastic polymer provide stiffness, strength, reduced moisture absorption, and reduced creep, whereas amorphous regions provide toughness and damage tolerance. The DOC is a significant factor in material performance and is largely influenced by manufacturing and processing profiles where variables include heating rate, dwell time, dwell temperature, pressure, and cooldown rate.

LM-PAEK and PEEK were evaluated for this effort. These materials are structurally similar, and composites manufactured with these matrices provide comparable mechanical properties. The LM-PAEK has a lower processing temperature and melt viscosity relative to PEEK, which may facilitate out-of-autoclave processing and as well as welding and joint fabrication.

Materials and Processing

Both the LM-PAEK matrix and PEEK matrix prepreg materials were received from Toray Advanced Composites in Morgan Hill, CA, with the following specifications:

- LM-PAEK: T700GC carbon fiber/TC1225 (LM-PAEK) unidirectional prepreg tape. Fiber areal weight of 145 g/m^2 (4.28 oz/yd^2), 34 percent resin content, and consolidated ply thickness (CPT) of 0.137 mm (0.0054 in.). Panels were fabricated from a single batch of prepreg.
- PEEK: AS4 carbon fiber/TC1200 (PEEK) unidirectional prepreg tape. Fiber areal weight of 146 g/m^2, 34 percent resin content, and CPT of 0.142 mm (0.0056 in.). Panels were fabricated from a single batch of prepreg.
- Mold release: Loctite Frekote NC-770 mold release was applied to Kapton® (DuPont) film for easy removal of consolidated panels from the steel tooling.
- Manufacturing: Test coupons were machined from 30.5- by 30.5-cm (12- by 12-in.) compression-molded panels, where panel fabrication followed the National Center for Advanced Materials Performance (NCAMP) processing specification for TC1225, as required for the equivalency test campaign (Ref. 4).

Panels were compression molded in a steel picture frame tool, with the Kapton film and mold release placed on both panel surfaces (Figure 1).

The compression molding procedure for TC1225 (LM-PAEK) follows:

1. Place ply stack into press.
2. Apply 207 to 345 kPa (30 to 50 psi) of pressure to the laminate.
3. Heat to 371 °C (700 °F).
4. Increase pressure to 1,551 kPa (225 psi).
5. Hold at 371 °C (700 °F) for 60 min.
6. Cool to lower than 71 °C (160 °F) at a rate between 2.8 and 5.6 °C/min (5 and 10 °F/min).

The procedure was maintained when manufacturing TC1200 (PEEK) panels, except the hold temperature was raised to 393 °C (740 °F).

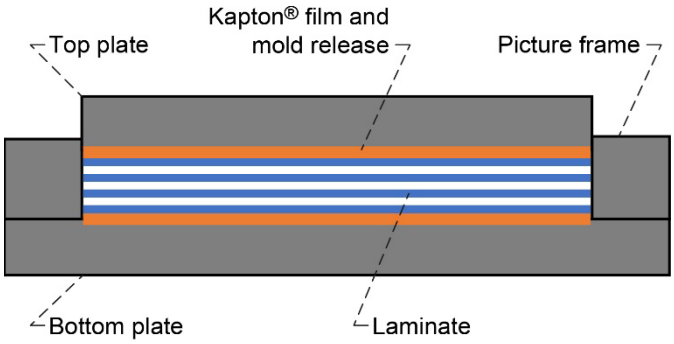

Figure 1.—Panel layup in picture frame tool.

TABLE I.—LAMINATE TEST MATRIX FOR LM-PAEK AND PEEK COMPOSITE

Abbreviation	Test	ASTM standard	Layup	Size, 0° by 90°, in. (cm)
UNT	Unnotched laminate tension	D3039	[45/0/–45/90]2s	10.0 by 1.0 (25.4 by 2.5)
UNC	Unnotched laminate compression	D6641	[45/0/–45/90]3s	5.50 by 0.5 (14.0 by 1)
OHT	Open-hole tension	D5766	[45/0/–45/90]2s	12.0 by 1.5 (30.5 by 3.8)
OHC	Open-hole compression	D6484	[45/0/–45/90]4s	12.0 by 1.5 (30.5 by 3.8)
CAI	Compression after impact	D7136 + D7137	[45/0/–45/90]4s	6.0 by 4.0 (15 by 10)

The mechanical test matrix is defined in Table I. Ply configurations matched those used to generate the NCAMP database (Ref. 5). Six coupons were used per test, and in most cases all coupons came from the same panel. Because of their size, the six CAI (compression after impact) coupons were cut from three separate panels. The test condition was limited to room temperature ambient (RTA).

Consolidated panels were characterized by the following standard methods:

- Inspection: Laminate consolidation was characterized by ultrasonic C-scan using a 5 MHz transmission frequency. In addition, representative sections of each panel were sectioned and then polished for optical microscopy.
- Acid digestion: The void volume and fiber content of each flat laminate panel was calculated following ASTM 3171–76, with six samples tested per material. The weight of an approximately 0.5- by 0.5-cm laminate piece was measured in air and water. The matrix material was digested in hot sulfuric acid, and the remaining carbon fibers were filtered through a fine mesh screen. The fibers were flushed with water followed by an acetone rinse. The acetone was evaporated overnight in a fume hood and the fibers were then dried in an oven at 100 °C prior to weighing.
- Differential scanning calorimetry (DSC): A modulated DSC Q1000 (TA Instruments) was used to evaluate the DOC of each manufactured panel. The laminate was cut, and 10 to 15 mg was weighed into a crimped aluminum DSC pan. The tests were performed under nitrogen, with a ramp rate of 10 °C/min from room temperature to 400 °C.

Results and Discussion

C-scan data and panel thickness measurements were used to confirm panel consolidation and guide coupon cut plans. Calipers were used for thickness measurements. The average CPT of LM-PAEK panels was 0.137 mm (0.0054 in.) and average CPT of PEEK panels was 0.142 mm (0.0056 in.). Both values met vendor specifications. Cut plans were drawn for each panel and submitted to the National Institute of Aviation Research (NIAR) for machining and mechanical testing, ensuring defect regions were excluded from the test coupon. A common defect observed in most panels was reduced consolidation in the corners due to uneven pressure distribution (Figure 2).

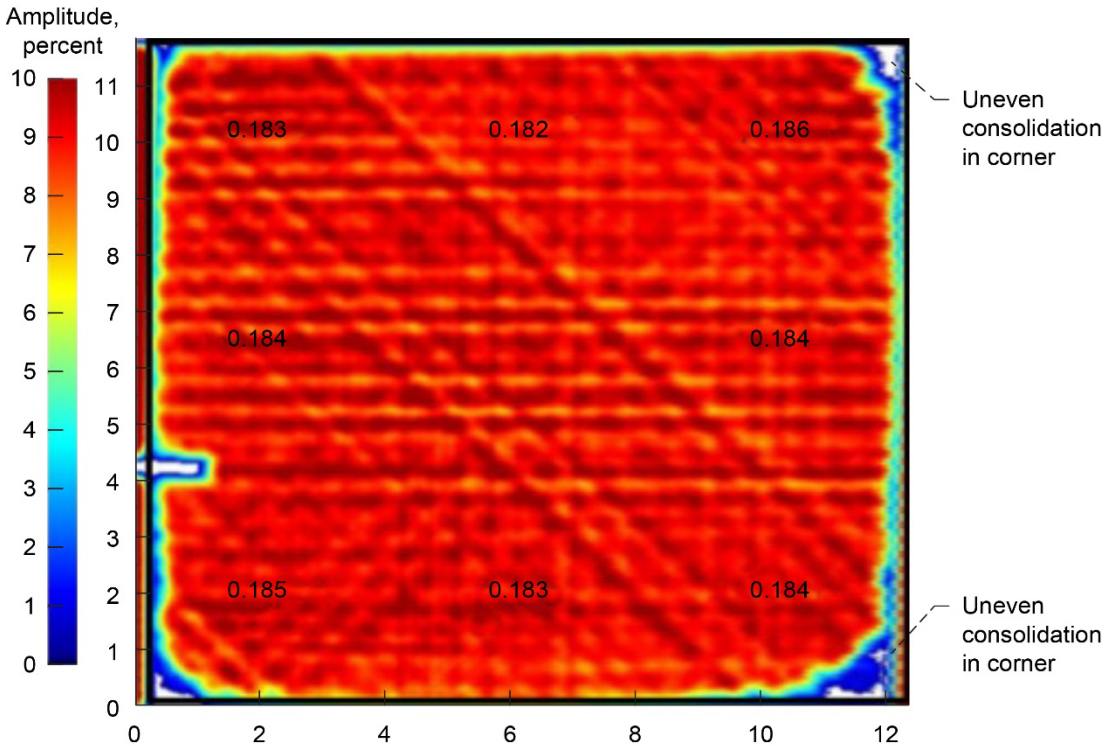

Figure 2.—C-scan and measured panel thickness (in inches).

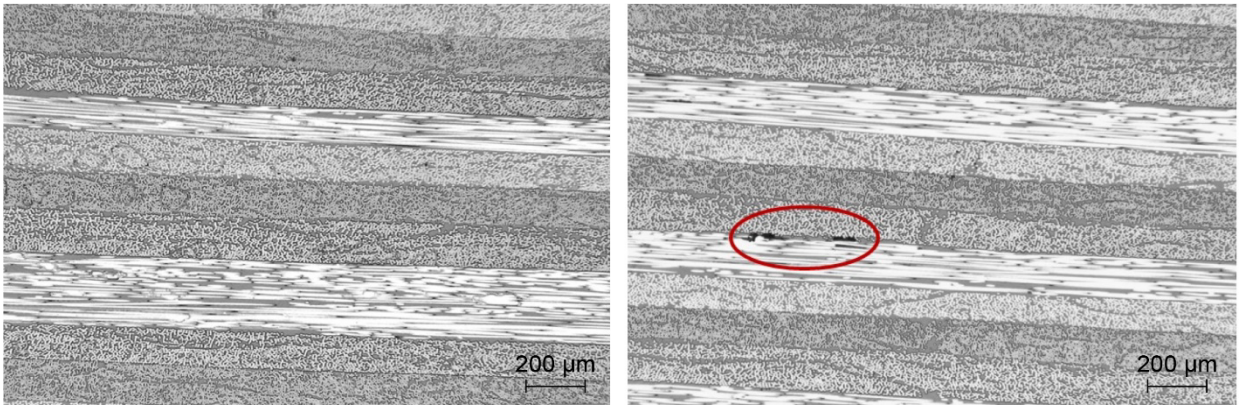

Figure 3.—Optical microscopy of representative TPC cross section.

Optical microscopy was used to confirm consolidation of all panels. Representative images from panel cross sections (Figure 3) illustrate very little void content and uniform ply thickness. Localized interlaminar voids are circled on the right-hand image.

Acid digestion data provided further validation of void-free laminates with the average void content being 0.7 percent across all LM-PAEK panels and 0.6 percent across all PEEK matrix panels. The average fiber volume fraction for the LM-PAEK and PEEK panels was 57 and 55 percent, respectively, and the resin was measured as 35 and 37 wt%, respectively. The 35 percent resin content of the LM-PAEK is consistent with the material specification. The higher resin content of the PEEK panel is within the vendor specification range.

DSC was used to determine the DOC for representative panels. The DOC is calculated from the enthalpy of melt and enthalpy of cold crystallization using Equation (1). An exotherm related to cold crystallization results from incomplete crystal development during cooling from the melt. When reheated

to a temperature higher than the glass transition temperature (T_g), increased polymer chain mobility enables crystallization. The absence of a cold crystallization peak higher than T_g is indicative of a fully crystallized material.

$$X_{mc} = \frac{H_m - H_{cc}}{H_f\,(1 - x_{mr})} \tag{1}$$

where

X_{mc} = mass fraction of crystallinity
H_m = heat of fusion at T_m, measured as the area of the melt endotherm
H_{cc} = heat of fusion for cold crystallization, measured as the area of the crystallization exotherm. Cold crystallization is identified as occurring higher than T_g on heating
H_f = theoretical heat of fusion for a pure crystalline phase; 130 J/g was used, per the prepreg supplier
x_{mr} = mass fraction of carbon fiber reinforcement

DSC thermograms are presented in Figure 4, representative of those for LM-PAEK and PEEK matrix panels. The LM-PAEK panels averaged 20 to 25 percent crystallinity, and the PEEK panels averaged 25 to 30 percent crystallinity, which is appropriate for each material. The DSC data generated did not include a peak for cold crystallization, confirming full crystallization.

The equivalency test matrix outlined in Table II reflects a subset of the data available within the NCAMP database for LM-PAEK (TC1225). This set of coupon tests was identified to provide confidence in NASA Glenn's in-TPC manufacturing and provide baseline data for material downselection.

Equivalency is based on a 95 percent confidence interval. Overall, the Glenn-manufactured panels performed well, passing the equivalency metric for all strength tests, however, the small number of coupons tested was noted as insufficient for equivalency. The modulus values from Glenn-manufactured panels were greater than the maximum allowed value for equivalency. This was attributed to two factors: (1) fiber sizing on the Glenn material had changed from that of the qualification panels, and an increased modulus was predicted and (2) all panels were normalized to 0.137 mm (0.0054 in.), although the measured thickness of qualification panels was 0.0129 mm (0.0051 in.).

The successful equivalency of data tested provides confidence in the accuracy of data generated on other materials of interest within the TDEA project. Mechanical property data for the PEEK matrix composites are provided in Table III.

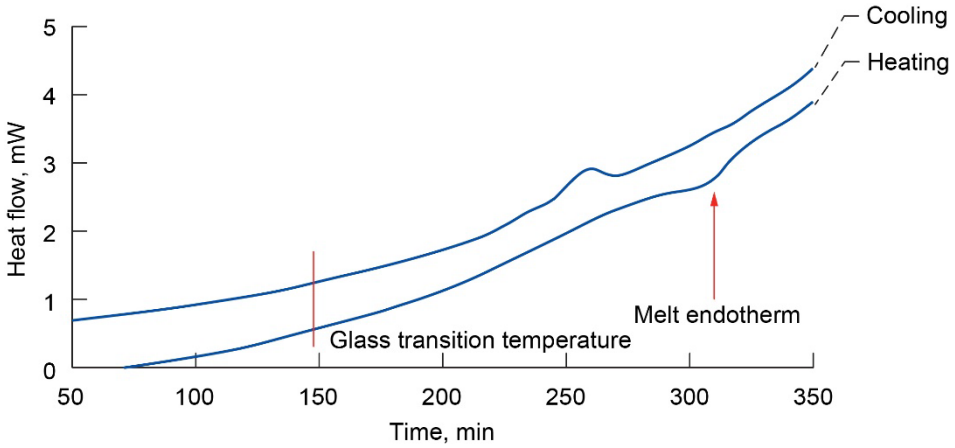

Figure 4.—DSC thermograms of coupons from representative LM-PAEK panels.

TABLE II.—MECHANICAL TEST DATA FROM NASA GLENN AND NCAMP QUALIFICATION PANELS

TC1225/T700[a] LM-PAEK	NASA data		NCAMP database	
	Average	Standard deviation	Average	Standard deviation
Unnotched laminate tension, UNT				
Tensile strength, ksi (MPa)	150.8 (1,040)	3.622 (24.97)	141 (972)	8.172 (56.34)
Tensile modulus, Msi (GPa)	7.265 (50.1)	0.143 (986)	6.651 (45,860)	0.163 (1,120)
Unnotched laminate compression, UNC				
Compressive strength, ksi (MPa)	80.62 (555.9)	4.616 (31.83)	77.07 (531.4)	3.18 (21.9)
Compressive modulus, Msi (GPa)	6.893 (47.53)	0.123 (848)	6.18 (42,600)	0.136 (938)
Open-hole tension, OHT				
Tensile strength, ksi (MPa)	70.56 (486.5)	1.93 (13.3)	68.52 (472.4)	3.227 (22.25)
Open-hole compression, OHC				
Compressive strength, ksi (MPa)	47.56 (327.9)	0.3824 (2.637)	45.79 (315.7)	1.38 (9.52)
Compression after impact, CAI				
Impact strength, ksi (MPa)	50.58 (348.7)	1.834 (12.64)	45.38 (312.9)	1.609 (11.09)

[a]LM-PAEK unidirectional prepreg tape from Toray Advanced Composites.

TABLE III.—MECHANICAL TEST DATA FROM NASA GLENN PEEK PANELS

TC1200/AS4[a] (PEEK)	NASA data (as measured)		Toray datasheet[b]
	Average	Standard deviation	Average
Unnotched laminate tension, UNT			
Tensile strength, ksi (MPa)	118.75 (818.75)	1.81	Not available
Tensile modulus, Msi (GPa)	6.78 (46.7)	0.12	Not available
Unnotched laminate compression, UNC			
Compressive strength, ksi (MPa)	86.14 (593.9)	2.14	Not available
Compressive modulus, Msi (GPa)	6.63 (45.7)	0.1	Not available
Open-hole tension, OHT			
Tensile strength, ksi (MPa)	54.31 (374.5)	1.43	56
Open-hole compression, OHC			
Compressive strength, ksi (MPa)	47.27 (325.9)	1.15	46
Compression after impact, CAI			
Impact strength, ksi (MPa)	42.08 (290.1)	2.79	44

[a]PEEK unidirectional prepreg tape from Toray Advanced Composites.

[b]Reference 6.

The purpose of this study was to demonstrate process equivalence and collect baseline data. The TC1225 and TC1200 prepreg materials varied not only in matrix resin but also fiber type, which will have a significant influence on the material properties. The tensile strength and modulus of 12K-tow AS4 fibers are 650 ksi (4,500 MPa) and 33.5 Msi (231 GPa), respectively. The T700G fiber properties are a tensile strength of 711 ksi (4,900 MPa) and a tensile modulus of 34.8 Msi (581 GPa).

The full set of materials screened within TDEA will include PPS and polyethylenimine (PEI). PPS is of interest due to its lower processing temperature relative to LM-PAEK or PEEK. PEI is the only amorphous material considered and is of interest for its potential benefits to welding. Test data from these materials will be published in a future report.

Summary of Results

This report described the in-house manufacturing, characterization, and mechanical test data for a series of thermoplastic matrix composites. Standard composite characterization confirmed a low void content in each panel and that full crystallization was reached during the manufacturing process. Equivalency tests confirmed that the quality of panels fabricated at NASA Glenn Research Center was equivalent to industry-supplied database properties. This provides confidence in further data generated from panels fabricated at this facility.

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