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Thermal Conductivity and Mechanical Properties of Epoxy Composites With Hybridized PAN- and Pitch-Based Fiber Architecture

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Summary

A series of hybrid polyacrylonitrile- (PAN-) and pitch-based carbon fiber/epoxy composites were fabricated and tested to evaluate the influence of hybrid fiber configurations on composite strength, modulus, and through-thickness thermal conductivity. Two grades of pitch-based fiber were selected based on their thermal conductivity: YSH-60A and YSH-80A from Nippon Graphite. These pitch-based fibers were received in tow form and as plain weave to allow flexibility in the architecture of the multifiber laminate. Two hybridization approaches were followed: (1) interleave of pitch-based fiber weave within braided PAN/epoxy prepreg and (2) incorporation of pitch-based fiber tows within a three-dimensional braided preform. These showed considerable impact on overall composite mechanical properties and through-thickness thermal conductivity.

Introduction

To enable the next generation of fixed-wing and vertical-lift aircraft, NASA has set goals for aircraft efficiency, emissions, reliability, and noise (Ref. 1). In its recently updated aeronautics strategic plan, NASA noted the benefits of alternative propulsion, specifically electrified aircraft propulsion (EAP), as enabling for emerging small-aircraft markets (Refs. 1 and 2). As concepts evolve toward electric propulsion, a commonality among designs is needed for lightweight materials capable of thermal management, structural loading, and fatigue durability.

Because of their high strength-to-weight ratio, polymer matrix composites (PMCs) have been manufactured for an increasing number of structurally demanding rotorcraft propulsion components, including motor, gears, and shafts (Refs. 3 to 6). The specific demand on the PMC material characteristics will differ depending on the application, for example,

1. Magnet gear concepts for EAP require magnet retaining hoops that will see varying hoop stresses, with tension-tension fatigue driving the material performance metrics.
2. Shafts will experience torque and bending stresses, with the direct-drive case being the most severe for shaft bending.
3. Motor housings require high stiffness and high thermal conductivity.

Current state-of-the-art carbon fiber/resin composites address a number of these requirements. A well-known drawback of laminated composites, however, is the low out-of-plane thermal conductivity, which limits their application in structures requiring heat dissipation. Existing literature includes methods to increase the thermal conductivity of PMCs, most often through modification of the matrix resin, which has yielded moderate improvements in conductivity and often a negative impact on mechanical performance (Refs. 7 to 10). A less widely reported approach is derived from variation or hybridization of the structural carbon fiber, which generally comprises 50 to 60 vol% of the overall laminate.

PAN-based fibers have fueled the continued growth of the carbon fiber industry since 1970 and are currently used in a broad range of applications such as aircraft brakes, space structures, military and commercial planes, lithium batteries, sporting goods, and structural reinforcement in construction materials (Ref. 11). These fibers are manufactured by carbonization of a polyacrylonitrile precursor to yield a range of strength and stiffness properties (Refs. 12 to 14).

Mesophase pitch-based carbon fibers are produced from a coal tar pitch precursor that undergoes a process of stabilization, carbonization, and final heat treatment (Refs. 15 and 16). Because of their graphitic nature, pitch-based fibers are unique in their ability to achieve ultrahigh crystalline-lattice-dependent properties such as Young’s modulus and thermal conductivity and, therefore, are used in critical military and space applications (Refs. 11, 17, and 18). However, the high cost in addition to a low strain to failure has limited its widespread use in commercial aerospace structures.

Approaches to utilize the best features of both polyacrylonitrile- (PAN-) and pitch-based fibers have been evaluated for applications requiring high strength, stiffness, durability, and thermal conductivity. Often, combination of the fibers has led to a reduction in composite tension, compression and bending strength, relative to a PAN-based laminate. Gowalia, et al. (Ref. 19) used a fiber-stitching approach to manufacture hybridized PMCs, where pitch-based fiber was incorporated in the through-thickness direction. The mixed fiber architecture led to a 1.3-fold increase of through-thickness thermal conductivity and a four-fold increase for the in-plane direction. However, the out-of-plane reinforcement led to a decrease in compression and bending strengths. Naito et al. (Ref. 20) reported tensile test data from carbon fiber/epoxy composites fabricated with high-strength PAN-based (IM600) and high-modulus pitch-based (K13D) fibers. A 50 percent reduction in tensile strength was observed following incorporation of pitch-based fibers into the PAN-based laminate. Waller et al. (Ref. 21) incorporated a dry, pitch-based fiber weave between plies of a PAN-based/bismaleimide prepreg. A 10-fold increase in thermal conductivity was measured, and an increase in both tensile strength and tensile modulus was observed. Additionally, Waller et al. demonstrated an improved fatigue resistance, attributed to reduced composite strain in the region of relatively stiffer pitch-based fibers.

This report details two approaches to incorporate pitch-based fiber into a PAN-based composite and evaluate the influence on both thermal conductivity and mechanical properties. The intent is a materials screening study to identify and enable applications for PMCs within EAP systems. Two basic approaches were taken to hybridization: (1) an interleave approach, where a pitch-based fiber weave was placed between plies of PAN-based fiber prepreg, and (2) the fabrication of a pitch/PAN co-braided preform, where pitch-based fiber was utilized in the axial tows of the braid, and PAN-based fiber was in the bias tow direction.

Materials

PAN-based fiber selected for this effort was T700S, an intermediate-modulus fiber purchased from Toray Industries, Inc. Pitch-based carbon fiber was procured from Nippon Graphite Fiber Corporation as a plain weave (PW) and in fiber tow form. Two grades of pitch were selected based on increasing thermal conductivity, YSH-60A and YSH-80A, and their fiber properties are summarized in Table I.

TABLE I.—SUMMARY OF FIBER PROPERTIES

Fiber	Fiber architecture	Tensile strength (MPa)	Tensile modulus (GPa)	Thermal conductivity (W/m·K)	Tow size (K)	Fiber areal weight (gsm)
Pitch: YSH-60A	Plain weave	3,930	634	200	1.5	100
YSH-80A	Plain weave	3,654	786	320	1.5	140
PAN: T700S	Triaxial braid (0, ±60)	4,902	228	9	12 (bias) 24 (axial)	536

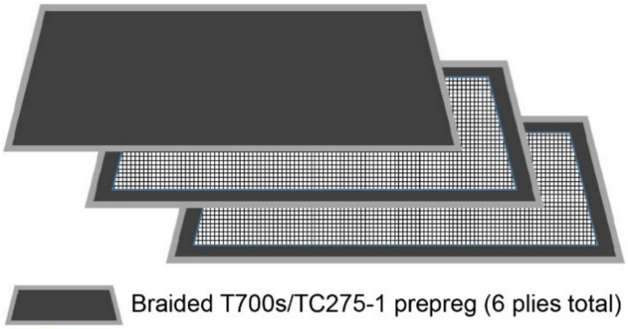

Figure 1.—Incorporation of dry pitch fiber weave into T700S/TC275-1 prepreg.

Dry carbon fiber (T700S) braid for the prepreg was manufactured by A&P Technologies Inc., Summerside, Ohio, and consisted of a quasi-isotropic $[0/+60/-60]_s$ braid (QISO® H-59) with 24K tows in the axial direction (0° tows) and 12K tows in the bias directions (±60° tows). The triaxially braided architecture provided nearly equal fiber volume fraction in each of the three fiber directions.

Prepreg fabricated with the braided T700S PAN-based carbon fiber and TC275-1 toughened epoxy resin was procured from Tencate Advanced Composites, Morgan Hill, CA, with an areal weight of 536 gsm and a 38 wt% resin content. The TC275-1 matrix resin was selected because preliminary tests demonstrated sufficient flow to wet-out the dry pitch fiber used in the interleave hybridization approach outlined below.

Panels were fabricated by stacking six plies of braided prepreg with the axial tows aligned in the 0º-direction.

Two paths evaluated for manufacturing hybrid PAN/pitch fiber laminates were

1. Pitch-based fiber interleave
2. Co-braided

Pitch Fiber Interleave Method

Pitch-based fiber plain weave was applied as an interleave within the braided T700S/TC275-1 prepreg (Baseline INT, Figure 1). Based on the relative areal weights, interleaving pitch weave resulted in laminates of approximately 13 wt% of YSH-60A pitch fiber (INT-60A) and 18 wt% of YSH-80A pitch fiber (INT-80A).

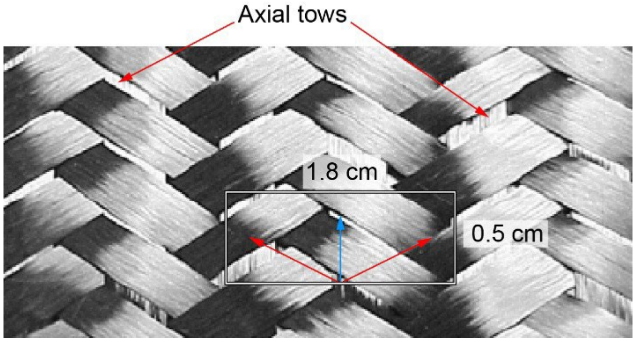

Figure 2.—Image of braided carbon fiber, calling out axial tows and unit cell size.

TABLE II.—TEST PANEL SUMMARY

Panel ID	Hybridization method and materials
---------------	Interleave material
Baseline INT	T700S fiber braid
INT-60A	Pitch: YSH-60A plain weave
INT-80A	Pitch: YSH-80A plain weave
---------------	Co-braided material
Baseline RTM	T700S fiber tow
RTM-60A	Pitch: YSH-60A tow
RTM-80A	Pitch: YSH-80A tow

Co-braided Approach

Dry T700S/pitch-based fiber hybrid braids were manufactured by A&P Technologies, Summerside, Ohio, and consisted of a quasi-isotropic $[0/+60/-60]_S$ braid architecture (QISO® H-59) with 24K YSH-60A or YSH-80A pitch fiber tows in the axial direction (0° tows) and 12K T700S fiber tows in the bias directions (±60° tows), resulting in an approximate 50 vol% pitch-based fiber preform. A schematic of the braid is provided in Figure 2.

Prepregging a small quantity of co-braided fibers was not cost effective for the material screening effort. Instead, RTM6 epoxy resin was selected for infusion: that material has been widely studied within the aerospace community (Refs. 22 and 23). RTM6 is a non-toughened, mono-component resin system that has been commercially developed by the Hexcel Corporation and is used for vacuum infusion and resin transfer molding (RTM) processes.

Braided preforms included a baseline T700S braid (Baseline RTM) and separate hybrids of T700S/YSH-60A (RTM-60A) and T700S/YSH-80A (RTM-80A). Table II provides a summary of panels made using both fabrication methods.

Manufacturing

Pitch Interleave Method

Flat panels (30.5 cm × 30.5 cm) were fabricated by hand lay-up of T700S/TC275-1 prepreg on a 1.3-cm-thick aluminum base plate. A 0.64-cm-thick caul plate was used to ensure uniform panel thickness. Braided prepreg plies were aligned with the axial tows oriented in the 0° direction, and dry, pitch fiber weave was placed between each layer, as illustrated in Figure 1. The laminated panels were vacuum bagged and autoclave cured following the Toray recommended cure process for TC275-1.

Co-braided Approach

Baseline and hybrid panels were fabricated by RTM of RTM6 into an aluminum tool housing. Panels were fabricated by A&P Technology using six plies, 30.5 cm × 30.5 cm, with the axial tows aligned in the 0°-direction. The average fiber volume fraction was 57 percent.

Panel Characterization and Test

Ultrasonic C-scan and optical microscopy were used to evaluate panel consolidation, void content, and wet-out of the dry fiber interleave. C-scans were collected using a 5-MHz transmission frequency, and the scans were evaluated on homogeneity of transmission and acceptable decibel loss.

Tensile tests coupons were machined to 25.4 cm × 3.5 cm, and tests were performed in accordance with ASTM standard D3039-17. All tests were performed at room temperature.

Flexural test coupons were machined to 7.6 cm × 1.9 cm, and tests were performed in accordance with ASTM D7264-21, Procedure B. All tests were performed at room temperature.

Thermal conductivity was measured using a steady-state approach. The test sample was placed between two control materials of known thermal conductivity. Then cooling and heating plates were attached, one on each side (Figure 3). Each plate was connected to a PolyScience Refrigerated Circulator (model number AD15R-30). The entire apparatus was surrounded by a Styrofoam insulation, and thermal grease was applied to all mating surfaces to minimize contact resistance.

The hot end was held constant at 40 °C, while the cold end was held constant at 20 °C. Assuming the heat flux is one dimensional, Fourier's Law can be used to calculate the approximate thermal conductivity. Using known thermal conductivities of the reference materials, along with known thicknesses and temperature measurements, the heat flux on each side of the test sample was calculated. Theoretically, these values should be equal, but this is not always the case, as heat is inevitably lost to the environment. After taking the average of the two heat fluxes, Fourier's Law was used to calculate the thermal conductivity of the test sample.

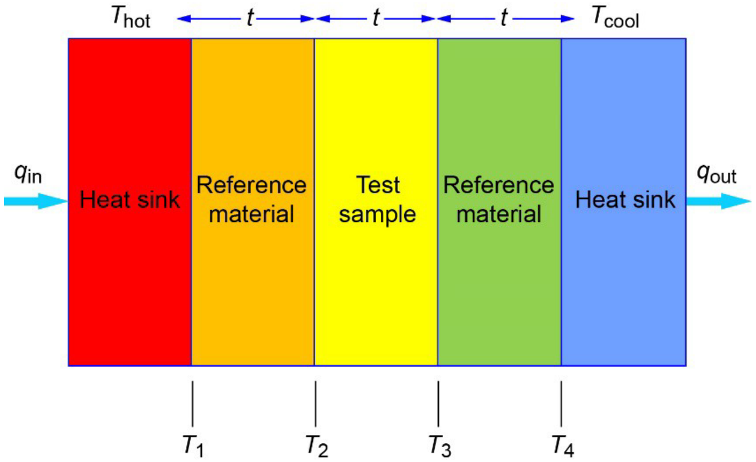

Figure 3.—Schematic depiction showing the configuration used for thermal conductivity measurements for temperature T, time t, and thermal flux q.

Results and Discussion

Photomicrographs of representative laminate cross sections are shown in Figure 4 for interleave panels and in Figure 5 for the co-braided hybrids. All images reflect well-consolidated panels, lacking significant void content, which was also confirmed by C-scan. Resin pockets are typical in the braided architecture, which facilitated wet-out of the interleave pitch fiber when used. The contour of the interleave follows undulations inherent to the braided preform.

Tensile strength and modulus data from interleave coupons are summarized in Table III. A 30 to 40 percent increase in Young’s modulus was recorded for laminates containing pitch-based fiber and is appropriate because of the relatively greater stiffness of the pitch-based fiber. The modulus of PAN T700S fiber is reported as 33 Msi compared to 92 and 114 Msi for YSH-60A and YSH-80A; respectively. The 2- to 3-fold greater stiffness of the pitch-based fiber does not directly translate to the composite stiffness because of (1) the low volume percentage of pitch-based fiber relative to PAN-based ones, and (2) more importantly, the PAN-based axial tows of the braided prepreg carry the primary load for this test.

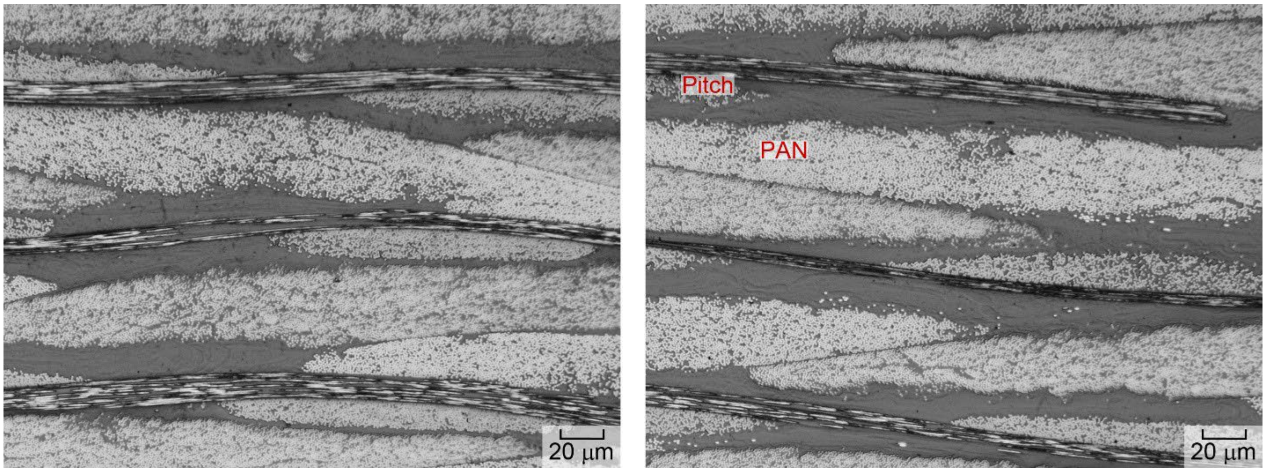

Figure 4.—Photomicrographs of representative panel cross sections showing pitch-based fiber interleave.

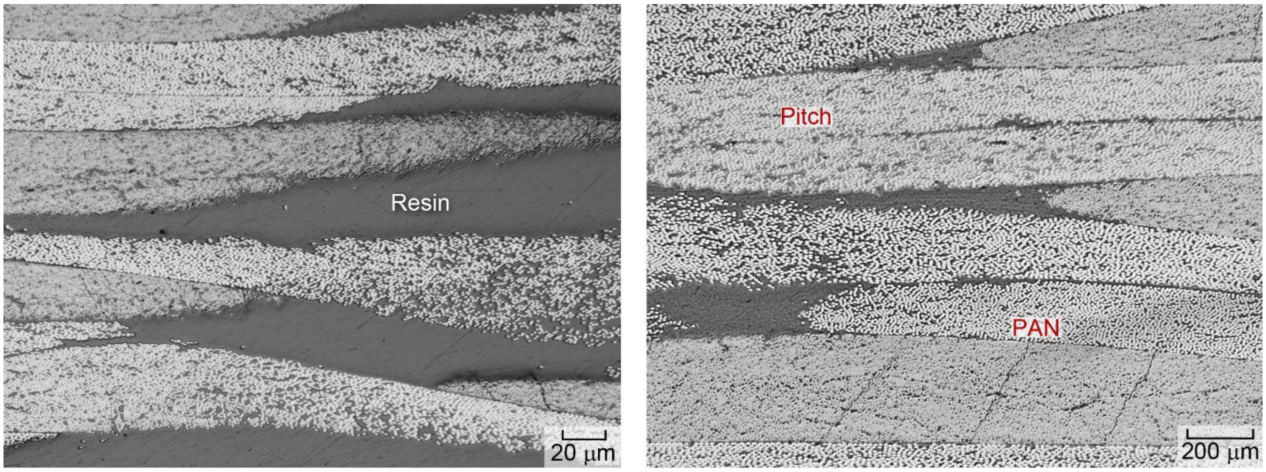

Figure 5.—Photomicrographs of representative panel cross sections manufactured by the cobraiding approach.

TABLE III.—TENSILE TEST DATA FOR PANELS INCORPORATING PITCH-BASED FIBER INTERLEAVE

Hybrid architecture	Tensile strength (MPa)	Standard deviation	Tensile modulus (GPa)	Standard deviation
Baseline INT	671	11.56	41.4	0.32
INT-60A	384	3.62	55.8	0.19
INT-80A	325	1.66	69.6	0.41

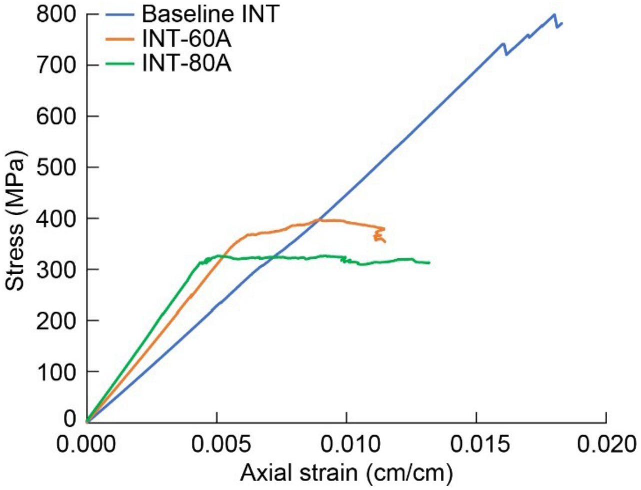

Figure 6.—Representative tensile stress-strain curves for interleave panels.

The increase in modulus from pitch-based fiber interleave correlates to a 50 percent reduction in both tensile strength and strain to failure. Representative tensile stress-strain curves for the baseline and hybrid coupons are plotted in Figure 6. In general, stress applied to the material was linearly proportional to strain until failure. Fracture surfaces of the TC275-1 revealed resin infiltration throughout the laminate thickness.

Tensile stress of the baseline material shows a typical, linear response to tensile load. Stress-strain data from the hybrid, interleave panels have an increased slope at the early stages of loading due to the high modulus pitch interleave and early failure where stress levels off. This is due to failure of the pitch-based fiber. However, failure of the pitch interleave does not instantaneously fail the coupon; rather, the load is supported by (transferred or shed to) the plies of PAN-based T700 fiber, thereby allowing additional strain.

Tensile data from co-braided materials yielded a similar trend (Figure 7). In this case the pitch-based fiber replaced PAN, as the axial tow of the braid and panels were manufactured by RTM with epoxy resin RTM6. Therefore, the baseline strength of the materials is expected to be different from the interleave series, but the trend in strength, strain, and modulus was of interest to this study.

Tensile test data of the co-braided material were reduced to a greater extent than that of the interleave coupons. Using a co-braided approach for hybridization directs tensile load to the pitch-based axial tows, significantly influencing strength and modulus (Table IV). The slope of the curves associated with the pitch-based fiber reveals a pronounced increase in modulus: 90 percent for YSH-60A and 150 percent for the YSH-80A. Tensile strength of the pitch-based hybrid panels dropped by 60 percent with the YSH-60A and 70 percent for YSH-80A.

In contrast to the interleave specimen, failure of the pitch-based fiber of co-braided hybrid coupons led to instantaneous coupon failure because the pitch-based fibers carry the primary tensile load.

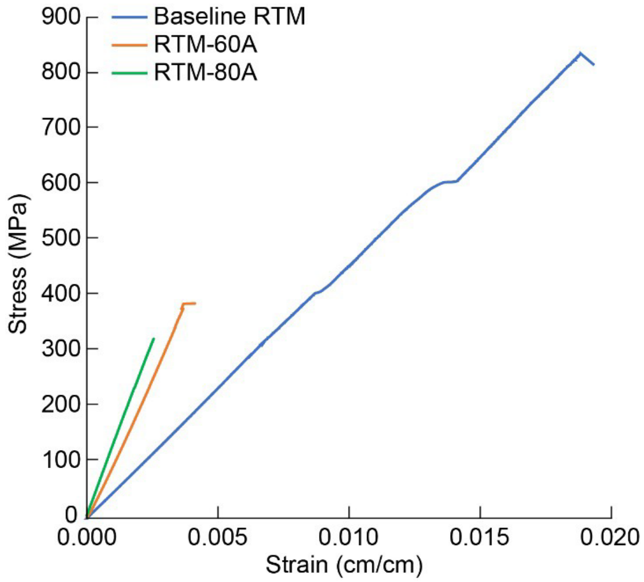

Figure 7.—Representative tensile stress-strain curves for co-braided panels.

TABLE IV.—TENSILE TEST DATA FOR PANELS INCORPORATING PITCH-BASED FIBER VIA CO-BRAIDED METHOD

Co-braided hybrid architecture	Tensile strength (MPa)	Standard deviation	Tensile modulus (GPa)	Standard deviation
Baseline RTM	811	4.43	46.9	0.08
RTM-60A	330	14.40	88.9	1.69
RTM-80A	245	19.66	119.3	1.66

TABLE V.—FLEXURAL TEST DATA FOR PANELS INCORPORATING A PITCH-BASED FIBER INTERLEAVE

Interleave hybrid architecture	Flexural strength (MPa)	Standard deviation	Flexural modulus (MPa)	Standard deviation
Baseline INT	555	4.08	37	0.11
INT-60A	570	4.52	44	0.30
INT-80A	530	4.86	46	0.28

TABLE VI.—FLEXURAL TEST DATA FOR PANELS INCORPORATING A PITCH-BASED FIBER VIA THE CO-BRAIDED METHOD

Co-braided hybrid architecture	Flexural strength (MPa)	Standard deviation	Flexural modulus (GPa)	Standard deviation
Baseline RTM	698	4.57	42.7	0.21
RTM-60A	570	1.53	70.3	0.68
RTM-80A	277	5.13	76.5	1.45

Flexural strength and modulus per ASTM D7264-21 in 4-point flexure data is summarized in Table V and Table VI for interleave and co-braided hybrids. The interleave approach yielded a negligible change in flexural strength for either grade of pitch-based fiber, indicating good interfacial strength between the dry pitch weave and the surrounding prepreg. An average 20 percent increase was measured in flexural modulus measured.

A greater impact was observed with the co-braided hybrid architecture where both tensile and compressive loads are carried by the pitch-based axial fiber. In these specimens, fiber hybridization reduced flexural strength of 54 and 60 percent for the YSH-60A and YSH-80A respectively; flexural modulus increased by 40 and 45 percent, respectively.

Representative stress-strain curves highlight the increased modulus attained through both hybridization methods (Figure 8 and Figure 9). The co-braided method places the low-strain pitch-based fiber in the load path, leading to early failure of the hybrid material.

The purpose of this work was to balance the thermal conductivity benefits from hybridization with pitch-based fiber to the expected reduction in composite strength. The results of thermal conductivity testing are detailed in Table VII with data from interleave coupons measuring a 16 and 46 percent increase in conductivity with YSH-60A and YSH-80A pitch fiber interleave, respectively. Data from panels with pitch incorporated along the axial tows exhibited an opposite trend, which cannot be explained at this time. Two measurements were taken per sample to confirm repeatability.

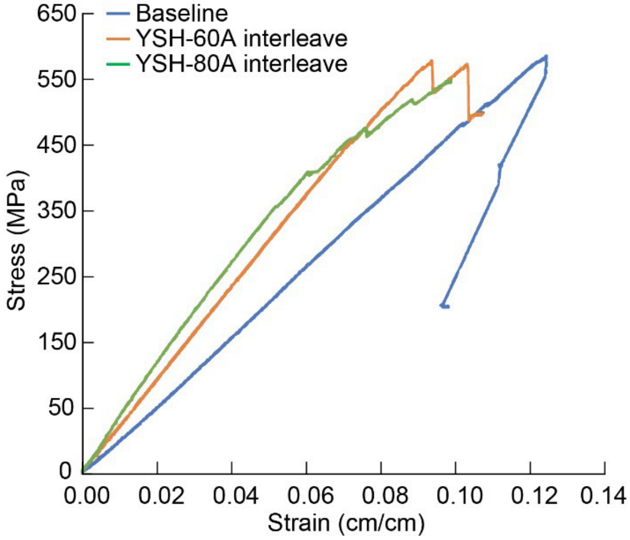

Figure 8.—Representative flexural stress-strain curves for interleave panels.

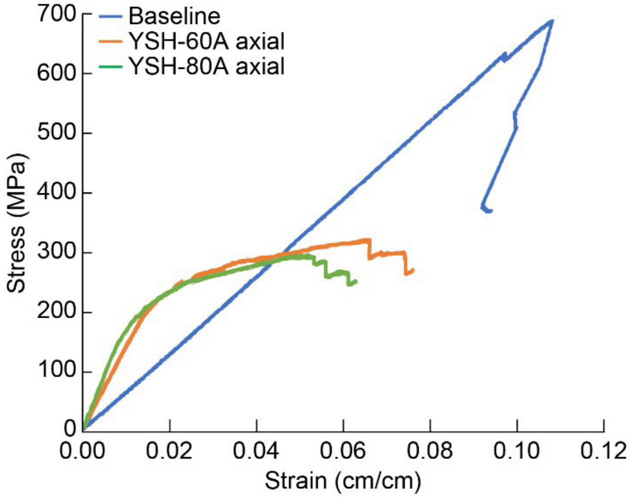

Figure 9.—Representative stress-strain curves for co-braided panels.

TABLE VII.—RESULTS OF THERMAL CONDUCTIVITY MEASUREMENTS

Material		Change relative to baseline, percent	Notes
Epoxy	Hybridization method		
TC275-1	Baseline INT	0.00	Baseline conductivity measured as 0.984 W/m·K
	INT-60A	16.25	
	INT-80A	46.55	
RTM6	Baseline RTM	0.00	Baseline conductivity measured as 0.974 W/m·K
	RTM-60A	48.40	
	RTM-80A	19.14	

As reported in Table I, the thermal conductivity of YSH-60A and YSH-80A is 200 and 320 W/m·K, respectively. Therefore, it was anticipated that hybridization with YSH-80A would yield a greater overall composite thermal conductivity. Both methods of pitch-based fiber incorporation yielded an increase in thermal conductivity, despite the co-braided method incorporating the pitch-based fiber at a greater volume than the interleave approach. This is due to the placement of the interleave material at the interlayer. In addition, results from the interleave approach followed a predictable trend based on the conductivity of the pitch-based fiber.

Conclusions

The method of PAN/pitch-based fiber hybridization had considerable impact on composite mechanical properties. The incorporation of pitch-based fibers via two approaches increased Young's modulus and through-thickness thermal conductivity at the expense of tensile strength. The co-braided approach resulted in larger decreases in strength than the interleave approach. Co-braiding better directs tensile and flexural loads to the weaker fiber. Interleaving allows load shedding from the weaker fiber to the stronger fibers. Laminate thermal conductivity increased following the incorporation of pitch-based fiber, and the data from the interleave method followed a predictable trend.

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