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Flexural Fatigue Testing and Qualification of Hybrid PAN-Pitch Composite Materials With High Through-Thickness Thermal Conductivity

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Summary

Carbon-fiber-reinforced polymers (CFRPs) considered for motor components within future electric vertical takeoff and landing (eVTOL) vehicles require not only a high strength-to-weight ratio but also a high through-thickness thermal conductivity (TC) to dissipate heat. Previously, CFRPs incorporating pitch CF interleaves demonstrated an increased through-thickness TC relative to the baseline composite, but the effect on flexural fatigue life was not evaluated. For this work, T700S/TC275–1 braided carbon fiber/epoxy prepreg baseline (no interleave), YSH–60A pitch interleave, and YS–80A pitch interleave configurations were tested for fatigue life. A combination of ASTM International Standards D7264 and D3479 were used to develop a reliable testing method. An Instron® (Illinois Tool Works Inc.) 8501 servohydraulic system with a fatigue test fixture was set up with a 16:1 span:thickness ratio, load ratio R set to 0.1, and frequency of 4 Hz. High-cycle fatigue testing was completed, and it was found that YSH–60A interleave performed slightly better than YS–80A below 100,000 cycles but showed no differences above 100,000 cycles. In all cases, the baseline CFRP had superior fatigue life performance both in terms of stress handled and total cycles to failure. These fatigue testing data will inform future use of hybrid CFRP materials by highlighting the tradeoff between increased through-thickness TC and fatigue life. Testing guidelines, equipment, and parameters for static and fatigue tests were successfully outlined for CFRPs.

Introduction

NASA has outlined a strategic plan to develop new technology that will meet growing needs for new vehicle concepts, markets, and applications, such as vertical takeoff and landing (VTOL) vehicles. Included in this mission is the need to reduce operating costs and noise while increasing accessibility and reliability without sacrificing performance (Ref. 1). One of the primary modes to reduce weight, noise, and operating costs while maintaining strength, durability, and flexibility of design is to replace metallic components with polymer matrix composites (PMCs) (Ref. 2). In fact, PMCs are unique and versatile in that their constituents, namely fiber reinforcement and matrix, and can be tailored based on specific application and design requirements.

For applications such as support or containment structures for electric propulsion, the PMC will have to dissipate heat generated within the engine (Ref. 3). Thermal management can be a challenge for PMCs, where the through-thickness thermal conductivity (TC) is generally one to two orders of magnitude lower than the in-plane TC because of a lack of conduction medium in this direction.

Various methods to increase the out-of-plane TC of carbon-fiber-reinforced polymers (CFRPs) have been reported in the literature. Ouyang, Rao, and Peng demonstrated that weaving highly conductive copper films into the composite increased composite through-thickness TC from 0.44 to 21.45 W/(m·K); however, the interlaminar shear strength (ILSS) decreased by approximately 20 percent (Ref. 4). This strength loss is not ideal for composite aircraft components such as motor housings, which require high strength and TC. Other applications, such as magnetic gear housings, see high hoop stress and should be resistant to tension-tension fatigue, whereas driveshafts need to be resilient to high bending stress and torque. Additional methods evaluated to increase through-thickness TC include deposition and integration into the matrix of a crushed pitch-based carbon fiber and graphene nanoplatelets mixture (Ref. 5), crushed diamond powder (Ref. 6), a highly conductive filler network of pitch-based carbon fiber and graphite flakes (Ref. 7), hexagonal boron nitride powder (Ref. 8), and alumina and boron nitride fillers (Ref. 9). Increases in TC up to 166 percent were reported, but these methods rely on complicated integration processes (Ref. 7). Zhang et al. report on the tradeoff between increased through-thickness TC and decreased tensile strength and modulus when using conductive fillers (Ref. 10), as do Yu et al., who demonstrated that z-pinning increased through-thickness TC of CRFP composites at the expense of tensile strength and modulus (Ref. 11). Therefore, a new method is needed to increase the TC of CFRPs without sacrificing strength.

Work has been done by Miller et al. (Ref. 12) to evaluate pitch fiber interleaves to increase TC without sacrificing strength. Hybrid polyacrylonitrile- (PAN-) pitch carbon fiber panels were fabricated using an interlayer method. It was found that the pitch interleaves greatly increased through-thickness TC at the tradeoff of lower tensile strength, as expected. However, the modulus increased and flexural strength was not significantly different. This demonstrates a potential solution for shafts and motor housings that require better thermal management while maintaining mechanical performance.

Building on the work reported in Reference 12, the viability of these new hybridized composite materials was studied through quantification of their flexural fatigue life, especially compared against the baseline braided prepreg T700S PAN-based carbon fiber with TC–275–1 toughened epoxy resin in a quasi-isotropic $[0/60/{-60}]_s$ configuration. The purpose of this work was to gather the flexural fatigue data for two particular interleave variations, YSH–60A and YS–80A, to better understand if pitch-based interleaves affect the fatigue life of the CFRP. YSH–60A and YS–80A are high-modulus, high-TC pitch-based graphite fiber fabrics.

Methods

Materials

The materials used in this study are the same as those used in Miller's study (Ref. 12).

The mechanical properties of the raw fiber materials are summarized in Table I. The two grades of pitch-based carbon fiber, YSH–60A and YS–80A, were selected from Nippon Graphite Fiber Corporation because of their higher TCs and in-plane tensile moduli. An additional pitch-based material, YS–90A, is included in this table because it was used for width screening but not for flexural fatigue testing. This was done to conserve the YSH–60A and YS–80A material.

TABLE I.—FIBER MECHANICAL PROPERTIES SUMMARY (REF. 12)

Fiber	Fiber architecture	Tensile modulus, Msi	Tensile strength, ksi	Thermal conductivity, W/(m·K)	Tow size ($\times 10^3$)	Fiber areal weight, gsm
Pitch: YSH–60A	Plain weave	92	570	200	1.5	100
Pitch: YS–80A	Plain weave	114	530	320	1.5	140
Pitch: YS–90A	Plain weave	128	510	500	1.5	75
PAN: T700S	Triaxial braid [0/±60]	33	711	9	12 (bias) 24 (axial)	536

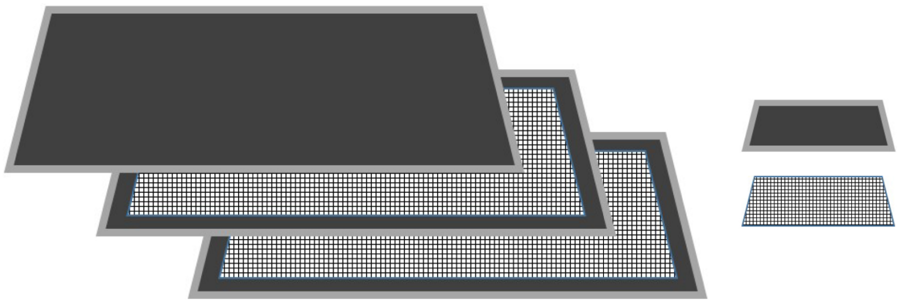

Figure 1.—Incorporation of dry pitch fiber weave into T700S/TC–275–1 prepreg (Ref. 12).

The prepreg was fabricated using braided T700S PAN-based carbon fiber and TC275–1 toughened epoxy resin. The T700S braid was manufactured by A&P Technologies Inc. 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.

Prepregging TC275–1 epoxy resin into the braided carbon fiber preform was performed at TenCate Advanced Composites (now Toray Advanced Composites). The resulting prepreg had an areal weight of 536 gsm and a 38 wt% resin content. The TC275–1 matrix was selected because preliminary tests demonstrated sufficient flow to wet-out the dry pitch fiber used in the interleave hybridization approach. Panels were fabricated by stacking six plies of braided prepreg with the axial tows aligned in the 0° direction, and pitch-based fiber plain weave was used as an interleave (see Figure 1). The laminated prepreg was vacuum-bagged and cured in an autoclave following the cure cycle recommended by Toray Advanced Composites for TC275–1.

The consolidated panels were shipped to Cincinnati Testing Laboratories (CTL) for coupon machining.

Coupons were received back from CTL; representative samples of each material system are shown in Figure 2.

Experimental Setup

An Instron® 8501 servohydraulic test frame was configured to run the static and fatigue tests. Static tests for each material were performed to characterize the flexural strength and were subsequently used to inform flexural fatigue loadings. A stainless-steel, three-point bend fixture (the Long Beam Flexure Fixture) was obtained from Wyoming Test Fixtures. Once installed, an antirotation device, shown in Figure 3, was needed to prevent the fixture from coming loose despite the use of shims and other means. This device prevented the bottom bend fixture from coming loose during the fatigue test and provided reliable and consistent force application.

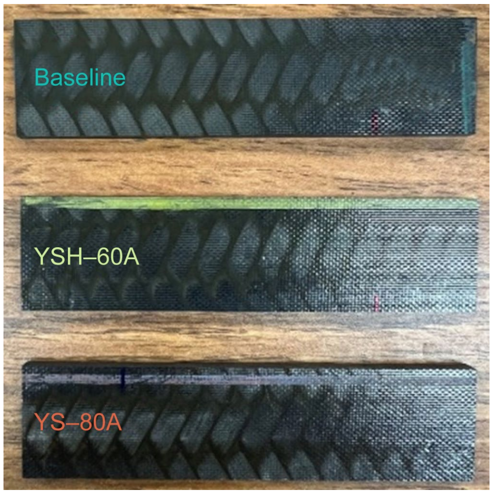

Figure 2.—Baseline, YSH–60A, and YS–80A hybrid PAN-pitch fatigue coupons received from CTL.

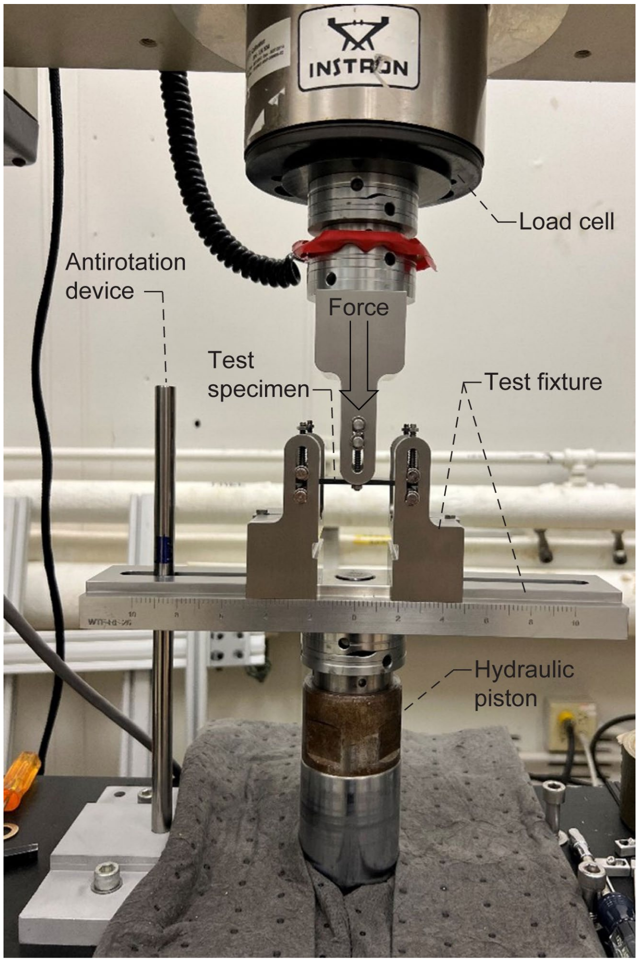

Figure 3.—Antirotation device installed on test frame to prevent flexural fatigue fixture from rotating during testing; test specimen under load with 2.5-in. span.

Because an explicit ASTM standard for flexural fatigue testing of PMCs does not exist, a combination of two relevant standards and the literature were used for setup purposes. ASTM Standard D7264 Flexural Properties of PMC Materials was used for three-point bend guidelines (Ref. 13) and ASTM Standard D3479 Tension-Tension Fatigue of PMC materials was used for fatigue standards such as load ratio *R* and minimum sample size (Ref. 14). As such, specimen thickness was measured and used to determine the appropriate span length of the fixture to maintain a 16:1 span:thickness ratio for testing. The fiber tows were aligned in the direction of the span to determine axial flexural properties.

Specimen Width Screening and Parameter Selection

Initially, multiple specimen widths of 1.91, 2.54, and 3.81 cm (0.75, 1.0, and 1.5 in.) were considered because the effect of width in accurately capturing the flexural mechanical properties was uncertain. Specifically, a sufficiently large coupon area was required to capture a full repeating unit cell of the prepreg fiber tow while minimizing the amount of material used to conserve limited manufactured panels. The results of this investigation and selection of a 1.91-cm specimen width are summarized in the Results section.

A practice specimen was loaded into the test fixture to tune the proportional-integral-derivative (PID) loop parameters for maintaining a consistent cyclic load. The optimized parameters used were proportional = 31.25 dB, integral = 0.167 1/s, and derivative = 0.8 ms. These parameters were slightly adjusted to optimize for each material system, but these values were a suggested starting point. A common loading ratio *R* in the literature is 0.1 (Ref. 15). This is defined as the ratio of the minimum to maximum force in cyclic loading. The frequency was ramped up in smaller steps from 1 Hz initially to 3 Hz, and finally to 4 Hz to reduce load overshoot. This is critical in fatigue tests to avoid inadvertent damage to the sample upon initial loading. Multiple practice tests were run to verify a reliable system setup, PID parameter settings, and frequency of testing. Demonstration of the minimal overshoot and reliable minimum and maximum loads between 100 and 1,000 N, respectively, are shown in Figure 4. Prior to this practice fatigue test, an identical specimen was statically loaded to failure, which helped inform the cyclic loading conditions. It is common to load a specimen to a specific percentage of the

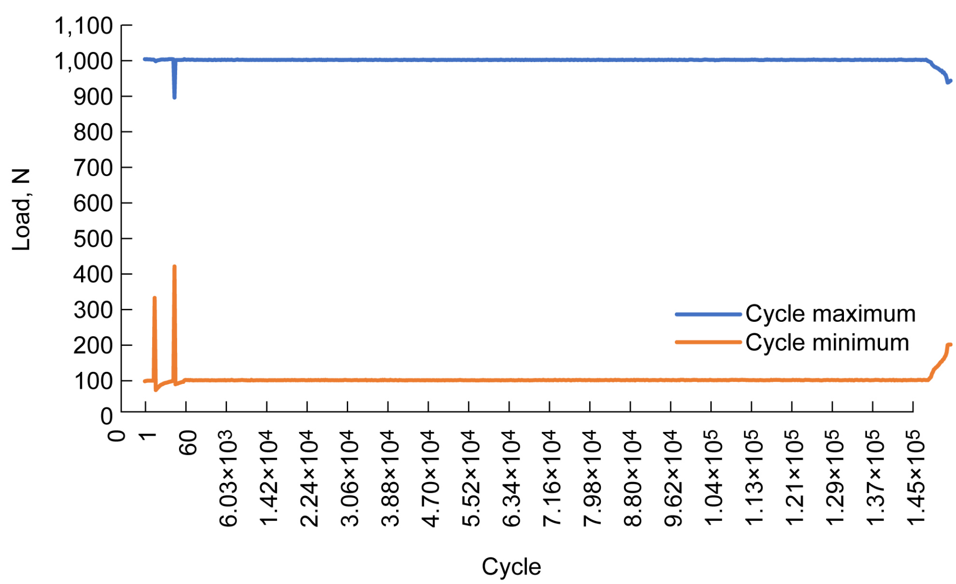

Figure 4.—Maximum and minimum load as function of cycles for practice fatigue test of YS–90A specimen with 0.75-in. nominal width. Load ratio *R* = 0.1; maximum load = 1,000 N nominal; minimum load = 100 N nominal; frequency = 5 Hz; cycles to failure = 153,000.

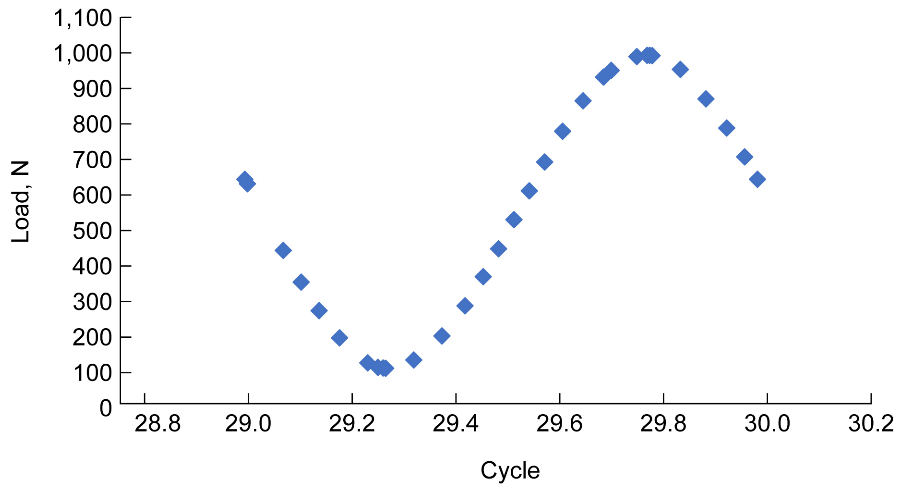

Figure 5.—Waveform of one cyclic load during fatigue testing. Load ratio R = 0.1.

maximum stress value for cyclic fatigue testing, ranging between 40 and 80 percent, depending on the material and geometry (Ref. 15). This was done for each material system to determine their material-specific maximum flexural stress. WaveMatrix™ (Illinois Tool Works Inc.) software was utilized to automate cyclic loading conditions, reduce data, store data, provide live readouts, and analyze peaks and trends throughout testing. An example of one loading cycle out of 153,000 cycles is shown in Figure 5.

Results

Width Screening and Selection

To determine if width played a significant factor in specimen strength and flexural modulus, three specimens of 7.62-cm length and 5.08-cm span with widths of 1.91, 2.54, and 3.81 cm were evaluated. Specimens were cut from the YS–90A hybrid PAN-pitch panel to conserve the YSH–60A and YS–80A materials, as the YS–90A had similar manufacturing and mechanical characteristics compared to both the YSH–60A and YS–80A pitch interleaves. Static flexural testing was performed according to ASTM Standard D7264 (Ref. 13) with a quantity of three specimens per width. Strength and flexural modulus for each width were calculated and are summarized in Table II with ±1 standard deviation. The results show that all values of strength and modulus are within one standard deviation. This is better visualized in Figure 6. Based on this data, a 1.91 cm width was selected for testing.

Fatigue Testing

Prior to fatigue testing, static testing of each material system was performed to understand the ultimate flexural strength. For the baseline sample, three specimens were tested and averaged to have an ultimate strength of 710.3 MPa. The same was done for YSH–60A and YS–80A with ultimate strengths of 536.7 and 426.2 MPa, respectively, as summarized in Table III.

This was crucial to calculate, because all specimen loadings were back-calculated and normalized as percent of maximum stress relative to baseline. This means that relative to the maximum strength of baseline samples, a load was calculated for each unique YSH–60A and YS–80A specimen based on its geometry; that is, a 60-percent maximum stress relative to baseline meant the necessary load was used to reach a maximum force applied on each respective specimen to reach 710.3∗0.6 = 426.2 MPa.

TABLE II.—SUMMARY OF MECHANICAL PROPERTIES FOR HYBRID PAN-PITCH YS–90A COMPOSITES OF DIFFERENT WIDTHS
[Standard deviation ±1 MPa.]

Width, cm	Strength, MPa	Flexural modulus, MPa
1.91	471.9±49.2	28.7±2.5
2.54	427.0±26.5	32.2±1.1
3.81	449.7±32.7	30.9±0.7

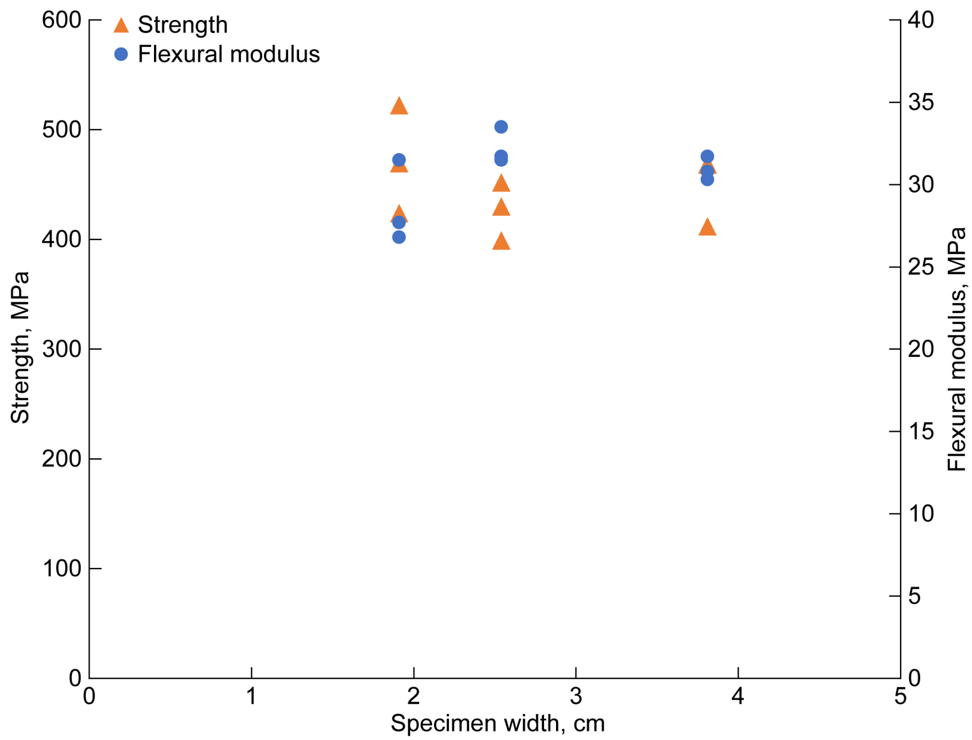

Figure 6.—Average strength and flexural modulus of practice bend specimens with YS–90A pitch-based graphite fiber interleave at nominal widths of 1.91, 2.54, and 3.81 cm.

TABLE III.—AVERAGE ULTIMATE STRENGTHS FOR EACH MATERIAL SYSTEM SUBJECTED TO STATIC FLEXURAL TESTING
[Standard deviation ±1 MPa.]

	Baseline	YSH–60A	YS–80A
Average ultimate strength, MPa	710.3	536.7	426.2
Standard deviation MPa	30.4	73.7	22.4

In total, 14 fatigue tests were completed for YSH–60A (including one runout), 13 tests were completed for YS–80A, and 14 tests were completed for baseline. The resulting fatigue curves are shown as a normalized maximum stress relative to baseline in Figure 7 and as raw maximum stress in MPa in Figure 8.

The plots indicate that YSH–60A performs slightly better than YS–80A at cycles less than 100,000. However, after 100,000 cycles they have similar fatigue life. In all cases, the baseline samples with no pitch interleave performed the best in terms of both maximum stress handled and cycles to failure for flexural fatigue.

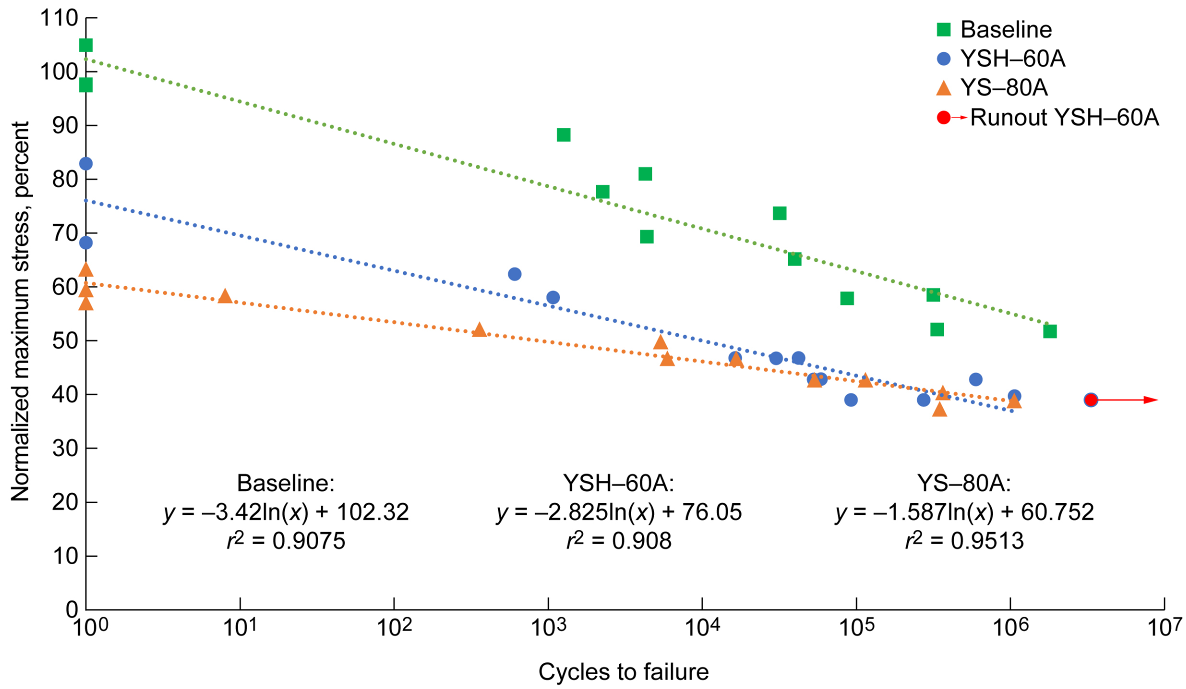

Figure 7.—Fatigue life curve, maximum stress (normalized relative to baseline) versus cycles to failure, for hybrid PAN-pitch YSH–60A, YS–80A, and baseline composite materials. Stress ratio R = 0.1; frequency = 4 Hz; data fit to semilogarithmic trend line. The correlation coefficient r^2 of best fit is provided for each line.

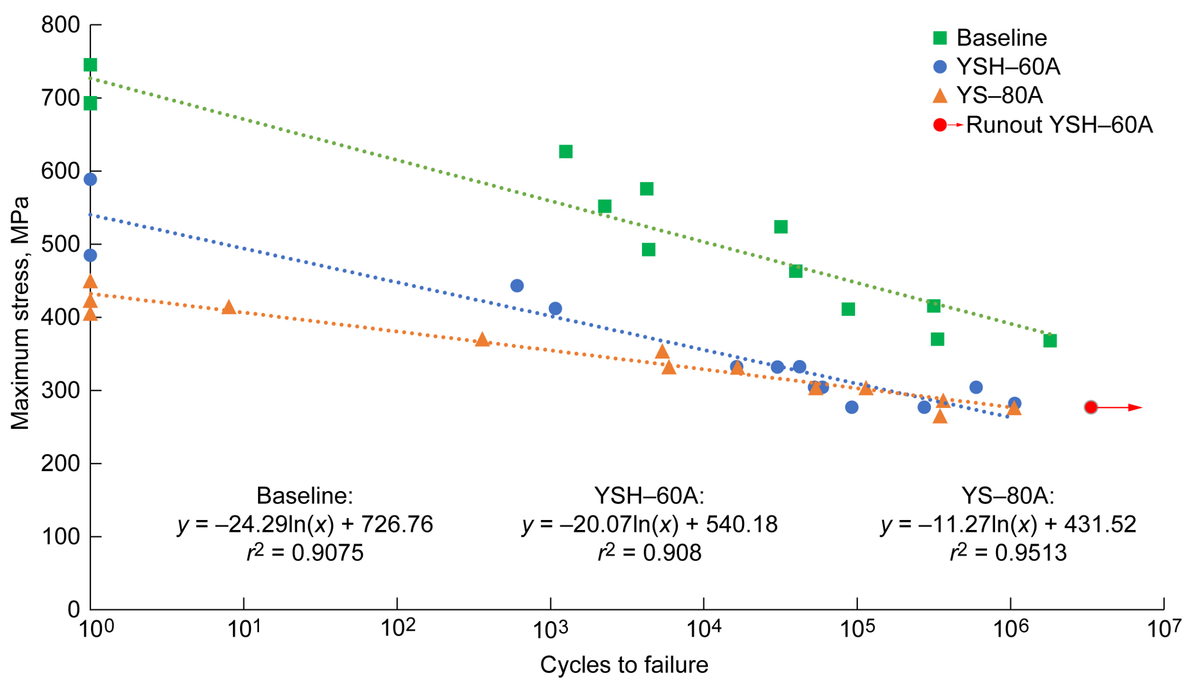

Figure 8.—Fatigue life curve, maximum stress versus cycles to failure, for hybrid PAN-pitch YSH–60A, YS–80A, and baseline composite materials. Stress ratio R = 0.1; frequency = 4 Hz; data fit to semilogarithmic trend line.

Failure Modes

Optical microscopy of the fractured baseline coupons shows matrix cracking on the tension (bottom) side along tow edges over a wide area along with fine matrix cracks across the tows. On the compression (top) side, matrix cracking along the tows occurred over a much smaller region of the specimen. However, a few tows have delaminated, broken, and popped out near the load pin, as shown in Figure 9. Delamination through the section is limited to the surface regions.

The YSH–60A coupons exhibit similar damage and fracture patterns, but delamination is more pronounced through the section from tension to compression sides, as shown in Figure 10.

For the YS–80A coupons, the matrix cracking on the tension surface is not as widespread, but much more delamination is exhibited through the section, as shown in Figure 11. The YS–80A specimens that were statically loaded to failure do not show much delamination but rather, more compression side cracking, which is different from the behavior of the fatigue samples.

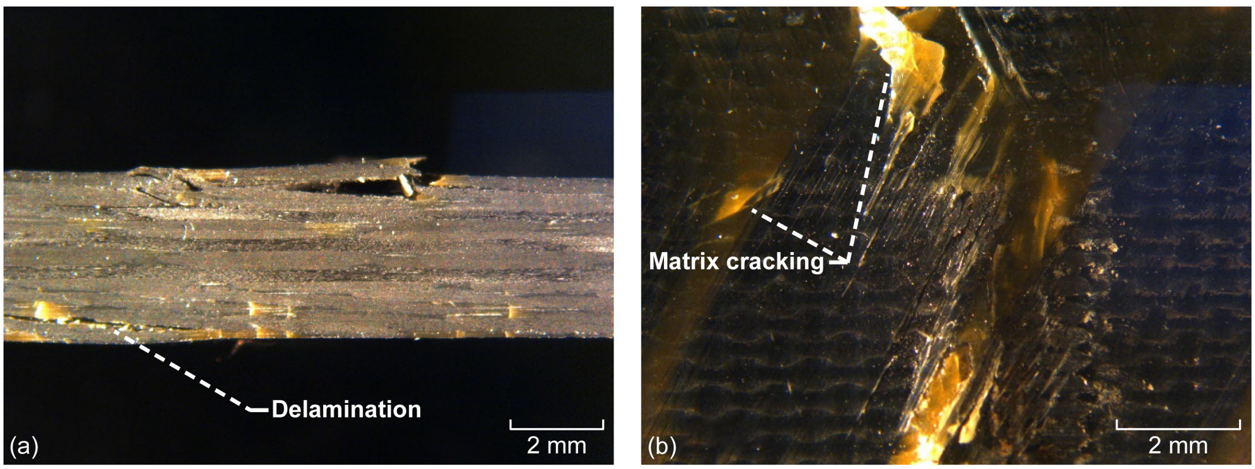

Figure 9.—Representative baseline sample after failure. (a) Side view showing delamination. (b) Compression surface view showing epoxy matrix cracking.

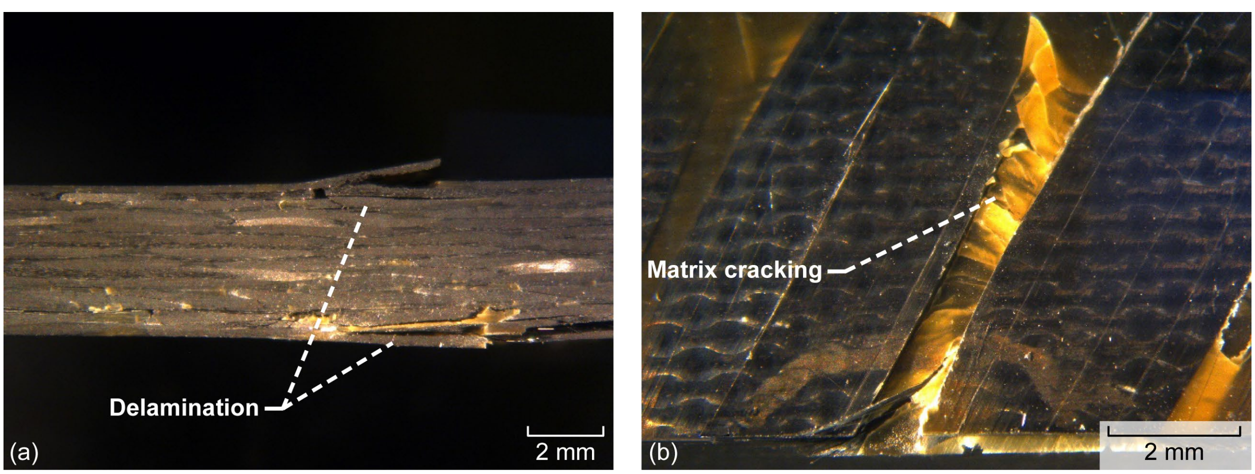

Figure 10.—Representative YSH–60A sample. (a) Side view showing delamination. (b) Tension surface view showing epoxy and carbon fiber cracking.

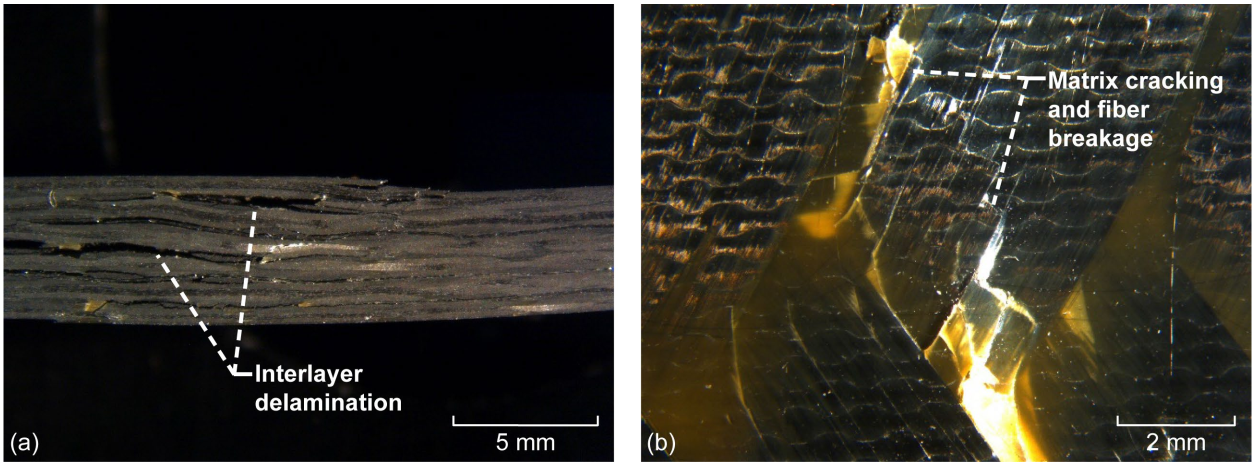

Figure 11.—Representative YS–80A fatigue sample. (a) Side view showing prominent interlayer delamination. (b) Compression surface view showing cracking within fiber tows and epoxy.

Conclusions

To increase the through-thickness thermal conductivity (TC) of carbon-fiber-reinforced polymers (CFRPs), hybrid polyacrylonitrile- (PAN-) pitch composites were manufactured using a pitch interleave approach. The effect of fiber hybridization on flexural fatigue performance was evaluated. A standard test method had to be developed, as none currently exists for flexural fatigue of polymer matrix composites. The method used for this work is based on ASTM Standards D7264 and D3479. A specimen size of 1.91 by 7.62 cm (0.75 by 3.0 in.) was chosen, and an Instron® 8501 servohydraulic machine was configured with a flexural fatigue fixture. The tests maintained a 16:1 span:thickness ratio, load ratio R of 0.1, and frequency of oscillation at 4 Hz. High-cycle fatigue and static testing was completed for YSH–60A interleave, YS–80A interleave, and baseline composite material systems. The key findings were that the YSH–60A samples performed slightly better than the YS–80A samples at cycles less than 100,000, but similarly after that level. Most notably, the flexural fatigue life of baseline samples with no interleave and lower TC had superior performance in terms of maximum stress handled and cycles to failure. This highlights the trade-off between the increase in through-thickness TC and reduction in flexural cyclic fatigue life. Testing guidelines, equipment, and parameters for long-cycle flexural fatigue testing of CFRPs were successfully outlined in this work.

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