DURABILITY OF CARBON/CYANATE ESTER PULTRUDED COMPOSITES IN GAS TURBINE ENVIRONMENT

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Abstract

The effect of gas turbine lubricant oil soaking, thermal cycling and their combination on fiber-matrix interfacial morphology and mechanical properties of pultruded composites was studied. Two types of lubricant oils were used: Mil-L-7808 and Mil-L-23699. Composite samples were soaked for 720 hours. The composite samples were thermal cycled for 600 cycles between RT and 315°C (600 °F) at a rate of 1 hour/cycle with 28 minutes hold at elevated temperature. A special furnace was designed and built for this purpose. As received and all conditioned samples were mechanically tested to measure the loss of modulus, strength and fracture strain in tension and the loss of stiffness and fracture load in flexure. Oil soaking had minimal effect whereas thermal cycling had significant impact on measured properties.

1 Background

High-temperature polymeric composite materials have the potential to reduce the component weight of aircraft structures, which is fundamental to the aerospace industry. Among the high temperature polymers for composite materials, cyanate ester resins became attractive because of their low moisture absorption, good fracture toughness, favorable dielectric properties and excellent processing characteristics [1-5]. Actual performance of these composite materials is limited by their long-term durability under a variety of service conditions involving high temperature exposure, thermal cycling, load cycling and exposure to fuel and hydraulic fluid. Thermal cycling has been demonstrated to cause the thermal and oxidative degradation of polymer composites and thus result in a rapid decrease in physical properties including stiffness, strength, density, flexural stiffness and damage tolerance [6]. Therefore, it is essential to estimate the stability of the composites under thermal cycling as well as other environmental conditions.

Mahieux et al. [7] conducted the research on the effects of industrial VG46 oil immersion on five carbon fiber reinforced polymer based composites including unidirectional AS4/PEEK, AS4/PPS, AS4/PEI, woven AS4/PEI and short random AS4/PFA. The composite samples were immersed in VG 46 oil at four different temperatures ranging from 20 to 80°C. None of these materials showed significant weight gain due to oil immersions and only AS4/PFA showed the reduction of glass transition temperature by 30°C. Siddaramaiah et al. [8] investigated the effect of engine lubricating oil DERD-2472 at 100°C on the mechanical properties of glass/epoxy and glass/polyester composites. Their results revealed that after exposure for 7 days, there were up to 20% reductions in tensile and flexural strength and modulus for both composites. The oil conditioning did not affect the ILSS value of glass/epoxy but slightly reduced that of glass/polyester composite. Etxeberria et al. [9] found that the immersion of standard sheet molding compounds (SMC) APG 10-R glass/unsaturated polyester laminate in engine oil at 60°C for 30 days did not produce any important modification on the flexural properties. Their results indicated that the engine oil did not result in any plasticization in matrix and/or the fiber/matrix interface.

Several studies [10-12] have been reported on the thermal stability of cyanate ester composites under isothermal conditions. Hillermier and Seferis [10] investigated the effects of temperature and moisture on thermal and mechanical properties of unidirectional 16-ply glass fiber/cyanate ester laminate by exposing the material at 200°C in air for

1000 hours. They found that gaseous by-product caused delamination and therefore reduced the fracture toughness and strength of the composite. The composite also lost 0.5% weight and 20% of flexural strength. Chung and Seferis [11] reported a decrease in glass transition temperature (T_g) of carbon/Arocy B-30 unidirectional composite from 275°C to 190°C after 30 hours of aging at 275°C. Parvatareddy and Wang et al. [12] studied the chemical aging at 150°C on IM8 carbon/Fiberite 954-2 toughened cyanate ester unidirectional composite. Their results showed that aging for 2 months decreased T_g by 30°C. Then the trend reversed. T_g went up by 20°C after 9 months of aging. Furthermore, the bending strength and the ultimate strain decreased by 29% and 48%, respectively, after 6 months aging.

Only limited studies have reported the thermal cycling effect on cyanate ester composite. Lee and Holl [13] assessed the retention of in-plane shear strength of IM7 graphite/ICI 954-2 cyanate ester $(+45/-45)_s$ laminate after thermal cycling between room temperature (RT) and 150°C and between RT and 240°C in air for 5 days at a rate of 10 min/cycle with a 5 min hold at elevated temperature per cycle. They performed the thermal cycling by shuttling the specimen between the preheated oven and the open air cooled by a fan using a hydraulic test machine to lower and raise the specimen from the oven fitted to the test frame. The in-plane shear strength decreased by 20% after 5 days of cycling. They proposed a cumulative thermal oxidation model to account for degradations. Matsumoto, Mohri and Nanjo [14] evaluated several properties of carbon fiber reinforced toughened polycyanate ester resin system RS-3. They demonstrated that the RS-3 composite has better tolerance to thermal cycling than Fiberite 934 epoxy composite due to lower residual stress in RS-3 composite than in the Fiberite 934 composite. All the composites considered heretofore were laminates. No research has been reported on the thermal cycling effect on carbon fiber/cyanate ester pultruded composites.

The objective of this paper is to evaluate the effect of environmental conditioning including lubricant oil soaking and thermal cycling on mechanical properties of T650 carbon/Lonza Primaset PT30 cyanate ester composite rods manufactured by pultrusion process. The environment simulated is typical of a gas turbine engine where the material is subjected to high cyclic temperatures in the presence of lubricating oil. The specimen is a composite rod of diameter 0.5 mm ± 0.01 mm (0.019 in ± 0.0005 in). Two types of lubricants are chosen: Mil-L-7808 and Mil-L-23699. The thermal cycle has 1-hour period with 28 minutes hold at 315°C (600°F) and 24 minutes at RT. The five types of environmental conditioning chosen were: (1) soaking the rods in Mil-L-7808 oil for 720 hours; (2) soaking the rods in Mil-L-23699 oil for 720 hours; (3) thermal cycling the rods for 600 cycles; (4) soaking the rods in Mil-L-7808 oil for 720 hours and then thermal cycling the rods for 600 cycles; and (5) soaking the rods in Mil-L-23699 oil for 720 hours and then thermal cycling the rods for 600 cycles. Tensile and three-point bend flexure tests are conducted before and after environmental conditioning to explore the environmental conditioning effect on mechanical properties. Optical and SEM microscopy analysis is performed to investigate the microstructural changes in pultruded rods.

2 Materials

The materials considered are Cytec T650 carbon fiber (3k tow) and Primaset PT-30 cyanate ester supplied by Lonza Corporation. Fig. 1 shows the molecular structure of PT-30 cyanate ester. It is a thermoset resin that has 65% char yield (same as phenolics) and less than 0.5% volatiles and generates no gaseous by-products during cure. PT-30 has a low viscosity at Resin Transfer Molding temperature (80 c.p.s. at 121°C) and is post-curable to achieve T_g in excess of 371°C (700°F). It has excellent electrical and dielectrical properties and thermo-mechanical stabilities.

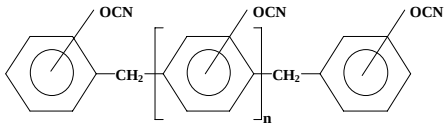


Fig. 1. Molecular structure of Primaset PT-30 cyanate ester resin

3 Pultrusion Process

Pultrusion is a highly automated process for the manufacture of constant volume shaped profiles made of composite materials. The composite rods of 0.5 mm ± 0.01 mm (0.019 in ± 0.0005 in) diameter were made by Aztex, Inc. by the pultrusion process. Each composite rod was composed of Cytec T650 Carbon Fiber (3k Tow) and Lonza Primaset PT-30 Cyanate Ester Resin. The pultrusion process consists of guiding the T650 fiber tow from a creel through a set of guides, then pulling through a PT-30 resin

bath that is maintained at proper temperature so that the tow is adequately impregnated with the resin. The impregnated fiber tow is then pulled through a heated die, where the tow is shaped into a circular shape. Finally the fiber tow is pulled through a thermal curing chamber to complete the cure. Curing is done in two stages, first at 260°C (500 °F) for 2 minutes and then at 315°C (600 °F) for two minutes. The fully cured fiber rod is collected by the take up spool. Fig. 2 illustrates the schematic of the pultrusion process.

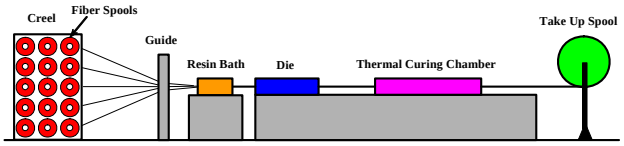


Fig. 2. Schematic illustration of the pultrusion process for T650/PT-30 composite rods

4 Environmental Conditioning

The T650/PT-30 pultruded composite rods were supplied in lengths of 300 mm (12 in). The environmental conditioning involves soaking in lubricant oils for 720 hours and thermal cycling. The thermal cycle profile is shown in Fig. 5. Specimens were held at 315°C (600°F) for 28 minutes with 12-minute pre and post hold at RT. Groups of 10 composite rods were exposed to the following environmental conditions and then were mechanically tested.

a. Base line: As received samples from the pultruder (Test Case #1).
b. Soaking in lubricant oils: The composite rods were soaked in Mil-L-7808 and Mil-L-23699 lubricant oils for a period of 720 hours (Test Case #4 and #6).
c. Thermal cycling without lubricant oil soaking (Test case #2): The rods were thermal cycled using the profile shown in Fig. 5 for 600 cycles.
d. Thermal cycling with lubricant oil soaking: After soaked in Mil-L-7808 (Test Case #3) and in Mil-L-23699 (Test Case #5) for 720 hours, the rod specimens were taken out and then thermal cycled for 600 cycles using the profile in Fig. 5.

Note that the cases b and d each consist of two groups since there are two types of lubricants. Microscopy was conducted on specimens from each of the environmental conditions to determine any manufacturing defects and visible damage caused by the conditioning. After exposure to the environmental conditioning of each case, the composite rods were tested for tensile modulus, strength and fracture strain, and flexural stiffness and fracture load. The flexural test is a qualitative test to assess the matrix damage. The test matrix with the number of specimens used in each of the mechanical tests is listed in Table 1.

Table 1. Test matrix

Test Case#	# of Tension Specimens	# of Flexure Specimens	Lubricant Soak 720 hrs @ RT	Thermal Cycles (600 cycles)	Tensile and Flexural Properties
1	10	9	---	---	√
2	10	9	---	√	√
3	10	9	7808 oil	√	√
4	5	9	7808 oil	---	√
5	10	9	23699 oil	√	√
6	5	9	23699 oil	---	√

4.1 Conditioning in Lubricant Oils

The composite rods (a minimum of 20 per grade of oil) were fully immersed in two separate sealed stainless steel containers, one containing Mil-L-7808 and the other containing Mil-L-23699 turbine oil (Fig. 3). The rods were then left to soak for 720 hours (30 days) in the containers. After the soaking period, the composite rods were drained and wiped free of oil using acetone before proceeding to further conditioning and/or testing.

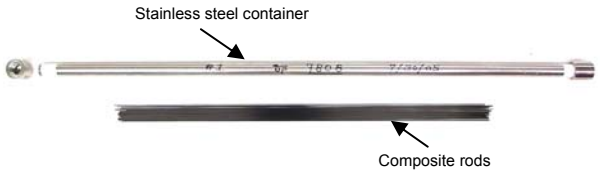


Fig. 3. Rod samples and oil-soak containers before insertion

4.2 Thermal Cycling

The composite rods were thermally cycled 600 times in a compressed air heating/cooling apparatus specially designed and built for this test (see Fig. 4). Fig. 5 shows the proposed and measured temperature profiles for each cycle. Dashed lines represent the proposed thermal cycle while the solid lines represent the actual one. The actual cycle almost duplicated the proposed cycle. The only difference is that the heating and cooling ramps were 4 minutes instead of 2 minutes. Fig. 6 shows three segments of the temperature profiles acquired during one of the tests. The three segments are 1-5, 300-304, and 596-600 cycles. Thermal cycling was performed on non-conditioned and oil-soaked samples.

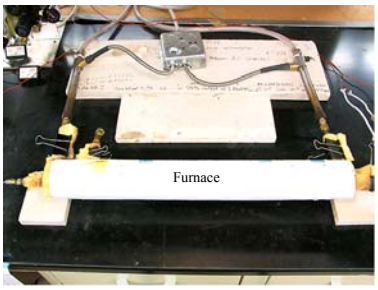


Fig. 4. Thermal cycling apparatus

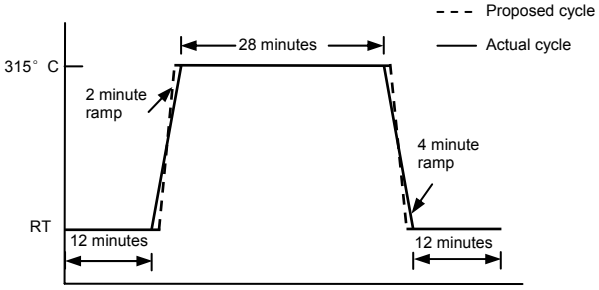
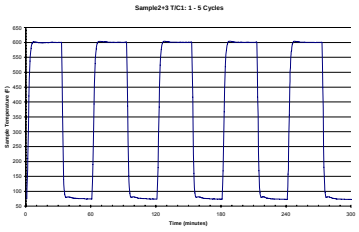
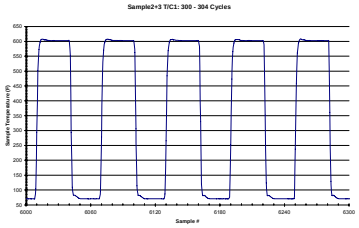


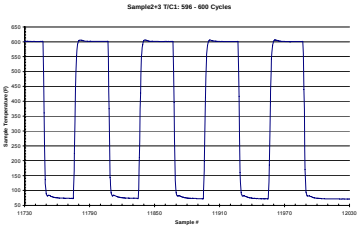
Fig. 5. Proposed and actual thermal cycles



(a) First 5 cycles



(b) Cycles 300 - 304



(c) Last 5 cycles

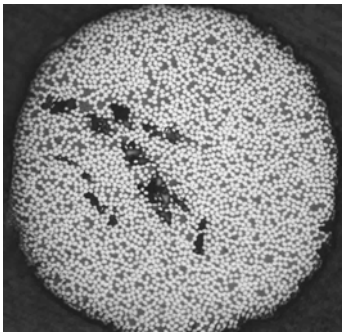
Fig. 6. Plot segments from a thermal cycling run

5 Microscopy of Environmentally Exposed Pultruded Composites

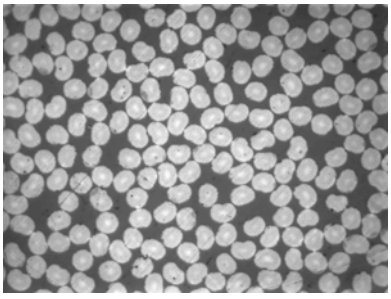
Microscopic analysis was conducted on samples from baseline (as received), lubricant oil soaked, thermal cycled, and lubricant oil soaked + thermal cycled rod samples. These six cases are listed in Table 1. Optical microscopic and Scanning Electronic Microscopic (SEM) images of rod cross-sections were captured and analyzed to investigate the environmental effects on the morphologies of pultruded composite rods.

5.1 Baseline (As-Received) Composite Rods

Fig. 7 shows the optical microscopic image of a baseline composite rod cross-section. Fig. 7(a) displays the whole rod cross-section and 7(b) shows the details of fiber distribution at the magnification of x1k. Fibers are uniformly and densely distributed except at some dark regions indicating the voids or resin deficiency. This is verified by the SEM images as in Fig. 8. Fig. 8(b) is the SEM image under higher magnification (x1k), which shows the details of the resin deficiency. Other than that fiber-matrix bonding is very good.

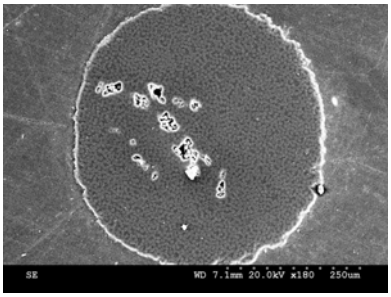


(a) Whole rod cross-section (x200)

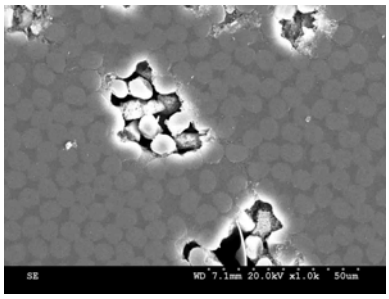


(b) Magnification x1k

Fig. 7. Optical images of baseline rod cross-section



(a) Whole rod cross-section (x180)



(b) Details of defective areas (x1k)

Fig. 8. SEM image of baseline rod cross-section

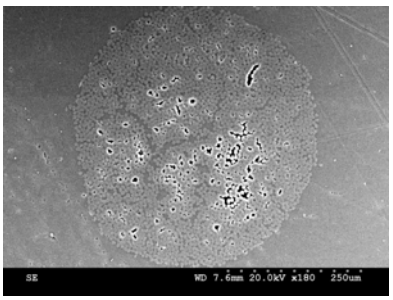
The microscopy study was repeated for five cross-sections of each rod and for five rods. All the images show the same trend. This study demonstrated that carbon fibers are uniformly distributed in the matrix with some small areas of voids and/or resin deficiency. The calculated fiber volume fraction based on the optical images is about 65%, which was expected for pultrusion process.

5.2 Lubricant Oil Soaked Composite Rods

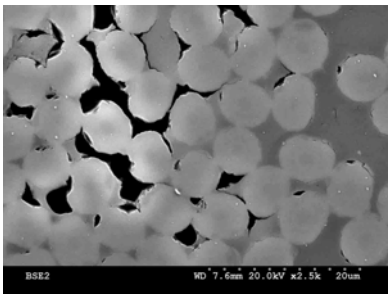
Optical microscopic images of composite rods soaked in Mil-L-7808 and in Mil-L-23699 were analyzed. The images showed no noticeable morphological differences compared with images of as-received rod except for that the matrix changed to a darker color. This indicates that oil soaking did not affect the morphology of pultruded rod.

5.3 Thermal Cycled Composite Rods

Pultruded composite rods were exposed to a 60min/cycle thermal cycle shown in Fig. 5 for 600 cycles, after which the microscopy study was conducted. Fig. 9 shows the SEM images of a thermal cycled rod cross-section. A large amount of fiber-matrix separation (debonding) and micro-cracks in the matrix due to matrix shrinkage are seen in these images. The reasons could include fiber sizing failure/oxidation, matrix shrinkage and/or oxidation, cyclic thermal stress fatigue etc. This damage may lead to the degradation of tensile and flexural properties.



(a) Whole rod cross-section (x180)



(b) Magnification x5k

Fig. 9. SEM images of thermal cycled rod cross-section

5.4 Lubricant Oil Soaked + Thermal Cycled Composite Rods

Fig. 10 shows the SEM image of a Mil-L-7808 soaked + thermal cycled rod cross-section. All the lubricant oil soaked + thermal cycled images demonstrate similar morphologies to those of thermal cycled only samples. Therefore, fiber-matrix interfacial separation and matrix cracks are due to thermal cycling effects while lubricant oil soaking has no effect. The thermal cycling caused matrix shrinkage, which sets up the high transverse tensile stress in the weak matrix direction followed by the fatigue degradation of the material. Furthermore, high temperature 315°C (600°F) in air could have oxidized the active interface region. The combined effects of these phenomena are seen in the microscopic images.

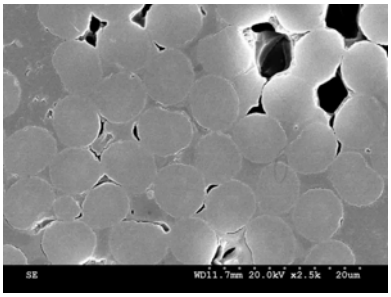


Fig. 10. SEM image of Mil-L-7808 soaked + thermal cycled rod cross-section

6 Mechanical Properties of Pultruded Composite Rods

Two types of mechanical tests were conducted to assess the impact of lubricant oil soaking and thermal cycling. One is tension test to measure the ultimate strength, fracture strain, and modulus. The other is three point bend flexure test. This measures the flexural stiffness (not the modulus) and the fracture load. This is a qualitative test that reflects

the degradation of the matrix and the fiber-matrix interface.

6.1 Tension Test

The tension test specimen configuration is shown in Fig. 11. The total length of the specimen was 300 mm (12 in) and the tab length was 25.4 mm (1 in). The composite rod diameter was 0.5 mm (0.019) inch with ± 0.01 mm (0.0005 in) variation. The test-length L, which was approximately 254 mm (10 in), was measured to the nearest 0.025 mm (0.001 in) for each specimen. Because of the small size of the rod, a special tabbing system was devised and tests were conducted with special instrumentation and procedures.

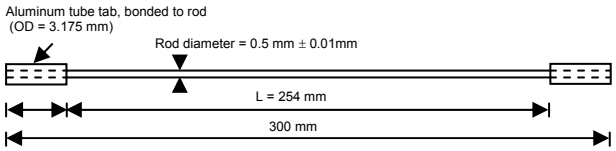
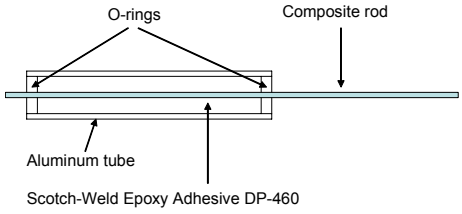


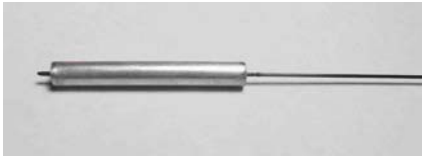
Fig. 11. Tension specimen configuration

6.1.1 Test Specimen Preparation

The ends of the composite rods were immersed into acetone and washed for no less than 5 minutes for good adhesive bonding. Scotch-Weld Epoxy Adhesive DP-460 was used for bonding. For the collet type gripping technique, 3.175 mm (1/8 in) OD aluminum tube segments, cut to 25 mm (1 in) length, were filled with the adhesive using a syringe. Small rubber O-rings were fitted into the ends with the rod placed through the tube and left overnight to cure at room temperature (Fig. 12). Fig. 12(a) shows details of the aluminum tube, adhesive, O-rings, and composite rod assembly at the tabbing location. A photograph of the tabbing assembly at one end of a test sample is shown in Fig. 12(b).



(a). Schematic of the tabbing



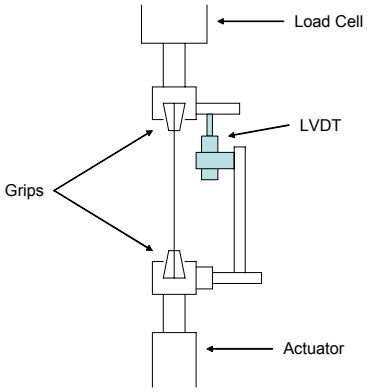
(b). Photograph of tab

Fig. 12. Tabbing and test specimens

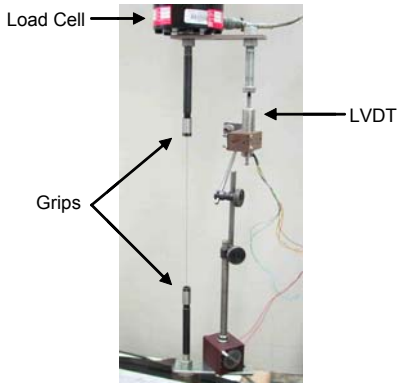
6.1.2 Testing

Each specimen was gripped by double-angle collet grips over the aluminum tube tabs on each end of the sample. The collets were tightened with a wrench while load was kept at zero. The grip extensions had been threaded to adapt to the load cell and actuator of MTS 810 testing machine and the load-train re-aligned with the new fixturing installed.

After preliminary testing on dummy specimens, a load cell with a full-scale range of 890N (200 lbs) was chosen for the tests. Load was recorded to the nearest 0.04 N (0.01 lb). An LVDT was used to directly measure the change in displacement between the 2 grips, thus eliminating the load-train deformation if the actuator displacement had been used. Fig. 13 shows the schematic (a) and photograph (b) of the instrumentation and test setup of the tension test. The tests were run at a constant displacement rate of 0.5 mm/min (0.02 in/min).



(a) Schematic



(b) Photo

Fig. 13. Tension test setup

The LVDT displacement reading and the load were recorded continuously (every ½ second) during

the test. The strain was calculated by dividing the displacement by the initial test-length of the specimen. Tensile stress was calculated by dividing the load by the cross-sectional area of the circular rod. The test was continued until the specimen fractured. The load and displacement at fracture were recorded and then the tensile strength and fracture strain were calculated.

6.1.3 Tension Test Results

For each test specimen, the complete stress-strain response from zero load until failure was acquired. This information gave us the strength, modulus, and fracture strain of the test specimen. The tensile modulus is defined as the initial slope of the stress vs. strain plot, $E=\Delta\sigma/\Delta\varepsilon$. An actual stress vs. strain plot for a baseline (unconditioned) specimen is shown in Fig. 14.

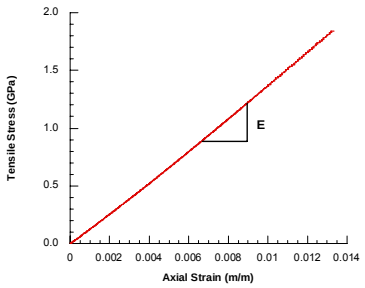


Fig. 14. Typical tension test data

The tension test results for baseline, lubricant oil soaked, thermal cycled and lubricant oil soaked + thermal cycled rods are summarized in Table 2. In Table 2, the average values of strength, modulus and fracture strain for each case, percent coefficient of variation and the property change in percentage compared with baseline samples are listed. The modulus of the composite rods was unaffected by soaking in either of the lubricant oils. The modulus is about 138 GPa (20Msi) and its variation is within the data scatter. The baseline tensile strength of the composite rods is 1.76 GPa (254.6 ksi). Soaking the specimens in the two lubricant oils reduced the strengths by 2% and 7%. Both are within the data scatter. The fracture strain of the composite rods had the same trend as the strength. Thermal cycling for 600 cycles did reduce the tensile strength by 28% and the fracture strain by 30%. The modulus remained almost unaffected by the thermal cycling for oil soaked samples but was reduced by 5% for base line samples.

For the test specimens that were lubricant oil soaked prior to thermal cycling, the properties closely matched those of the test specimens which were only thermal cycled. That is, the lubricant oil soaking had little effect on the tensile properties either before or after thermal cycling.

Table 2. Summary of tension test results

Test Samples	Tensile Strength		Tensile Modulus		Fracture Strain	
	Strength (GPa)	% change	Modulus (GPa)	% change	Strain (%)	% change
Baseline	1.76 (4.1)*	0	138 (0.9)	0	1.27 (3.8)	0
Mil-L-7808, soaked for 720 hrs	1.63 (7.7)	-7%	136 (1.8)	-1%	1.19 (5.9)	-6%
Mil-L-23699, soaked for 720 hrs	1.73 (6.3)	-2%	139 (1.2)	1%	1.24 (5.6)	-2%
Thermal Cycled	1.26 (10.6)	-28%	144 (0.8)	5%	0.89 (10.5)	-30%
Mil-L-7808 soaked + TC	1.28(6.3)	-27%	137 (4.1)	-1%	0.96 (4.9)	-24%
Mil-L-23699 soaked + TC	1.19 (6.7)	-32%	133 (3.0)	-4%	0.91 (7.8)	-28%

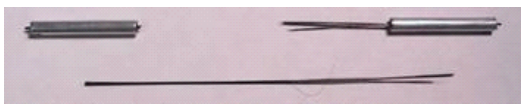
* Percent Coefficient of Variation

6.1.4 Tensile Failure Modes

Post-test photos in Figs 15, 16 and 17 illustrate typical tensile failures for baseline, lubricant oil soaked, thermal cycled, and lubricant oil soaked + thermal cycled test samples. Both baseline and oil-soaked test samples showed brittle fracture typical of unidirectional carbon composite specimens (Fig. 15(a)) with the exception of two oil-soaked test specimens (Fig. 15(b)). These two exceptions exhibited splintering, brooming, and finally balling due to high energy release at fracture. These two test samples also had higher strength compared to the oil-soaked samples exhibiting typical brittle failures.

All thermal cycled test specimens, without lubricant oil soaking or with lubricant oil soaking, showed splintering (shattering) of fibers due to degraded matrix properties. These failure modes are illustrated in Figs 16 and 17, respectively.

baseline



baseline

oil soaked

(a) Typical failures of baseline and oil-soaked rods

div-ten-OS1-4

div-ten-OS2-5

(b) Typical failures (exceptions) of two oil-soaked rods

Fig. 15. Typical tensile failure modes of baseline and oil-soaked composite rods

Fig. 16. Typical tensile failure modes of thermal cycled composite rods

Fig. 17. Typical tensile failure modes of oil-soaked + thermal cycled composite rods

6.2 Flexure Test

This is a qualitative test to evaluate the extent of matrix damage due to environmental exposure of the composite rods. The matrix damage due to micro-cracking and interfacial separation should effect the flexural modulus (stiffness) much more readily than the tensile modulus. Therefore a 3-point bend test, with a span to diameter ratio greater than 20, was chosen. The test setup and the specimen are shown in Fig. 18.

For each sample category, 3 of the 30 cm (12 in) rods were used for the flexure tests. From each of these 3 rods, the flexure samples were cut as three 25.4 mm (1 in) segments from one end of the rod. The samples were designated as the 1st, 2nd, and 3rd inch segments from the ends of the rods. The test specimen numbering system reflects this procedure: The first number is the rod # and the last number is the test specimen from that rod. For example, "div-flex-OS1TC-2-3" is the 3rd sample from rod #2, for the case of "Oil Soak #1 + Thermal Cycled."

6.2.1 Flexure Test Setup

A small precision bending fixture (As per ASTM D2344) was used for the test (Fig. 18). The lower loading supports were 3.175 mm (0.125 in) diameter and the upper support was 6.35 mm (0.25 in) diameter. Load data was acquired with a resolution of 0.004 N ± 0.002 N (0.001 lbs ± 0.0005 lbs). The displacement rate was 0.5 mm/min (0.02 in/min).

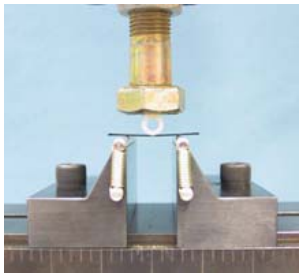


Fig. 18. Flexure test setup

6.2.2 Flexure Test Results

Each flexure test determined both the maximum load and the stiffness of the test specimen. The stiffness change from the specimens that are unconditioned to conditioned measured the effect of environmental exposure. The stiffness is defined as the slope of the load vs. displacement plot, $K_b=\Delta P/\Delta d$, as illustrated in the schematic in Fig. 19. Fig. 20 shows typical flexure results for baseline, lubricant oil soaked, thermal cycled and lubricant oil soaked + thermal cycled specimens. Note the different characteristics between the test results for the non thermal cycled and thermal cycled specimens. These differences are explained in Figs 20 and 21 for lubricant oil soaked and thermal cycled specimens.

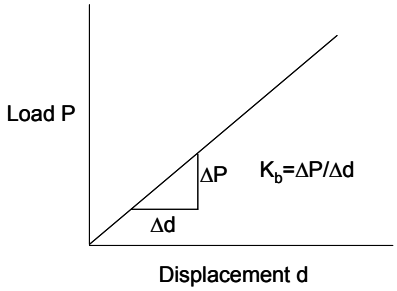
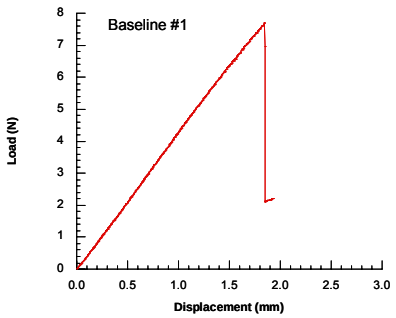
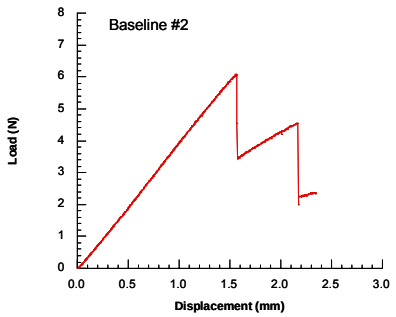
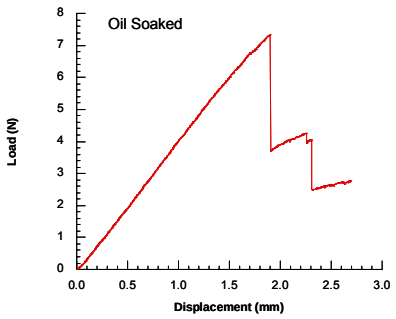
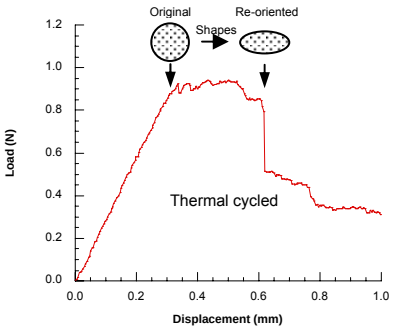


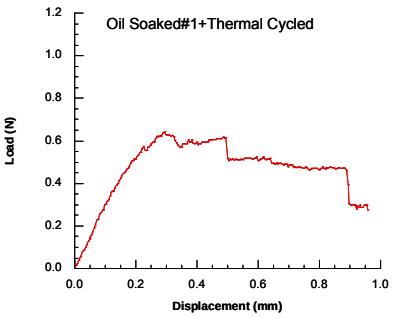
Fig. 19. Flexure stiffness determination











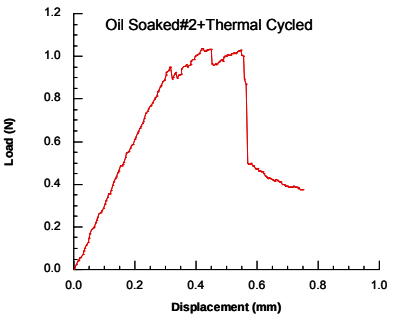


Fig. 20. Typical flexure test plots

Fig. 21 illustrates lubricant oil soaked test specimen # div-flex-OS1-1-1 going through the failure progression. Similar failure progression was seen in all baseline and lubricant oil soaked flexure specimens. Notice the fibers on the underside failing in tension in the last 2 images as the specimen is deflected at 0.5 mm/min (0.02 in/min). The load drops each time a group of fibers fails. After stopping the test and unloading the specimen, the specimen becomes straight due to the remaining unbroken fibers at the top.

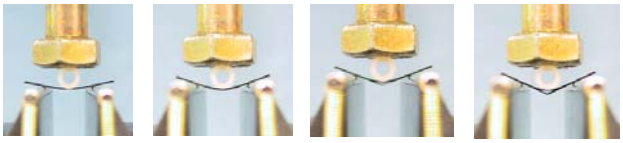


Fig. 21. Illustration of failure progression of baseline and lubricant oil soaked specimens

The flexure specimens of thermal cycled and oil soaked + thermal cycled did not fail as in Fig. 21; the load did not drop off suddenly after the initial linear portion of the load vs. displacement curve and no fibers were seen breaking at the tensile side of the specimen. Instead, the shape of the rod changed from circular to elliptical as shown in Fig. 20.

During failure, the individual fibers in these specimens re-oriented themselves into an elliptical shape and continued to lose stiffness. The

reorientation of fibers is caused by the fiber-matrix interfacial separation and matrix damage as shown previously through SEM images (Figs 9 and 10). Therefore at the middle of the specimen the circular cross-section becomes more elliptical as the failure occurs (see Fig. 20 "Thermal cycled" case).

The flexure test results for baseline, lubricant oil soaked, thermal cycled and lubricant oil soaked + thermal cycled rods are summarized in Table 3. In Table 3, the average values of maximum load and flexural stiffness for each case, percent coefficient of variation and the property change in percentage compared with the baseline specimens are listed. Neither the failure load nor the stiffness was affected by soaking the specimens in either lubricant. Thermal cycling for 600 cycles, however, did significantly reduce the failure load (by 82%) and the stiffness (by 27%). Specimens that were oil soaked in Mil-L-7808 prior to thermal cycling lost 30% of stiffness and 87% of failure load whereas the Mil-L-23699 soaked + thermal cycled specimens showed lower loss of stiffness (19%) and failure load (76%).

Table 3. Summary of flexure test results

Test Specimens	Maximum Load		Flexural Stiffness	
	Load (N)	% change	Stiffness (N/mm)	% change
Baseline	6.51 (9.7)*	0	4.06 (2.6)	0
Mil-L-7808 soaked	6.84 (4.1)	+5%	4.06 (1.7)	0%
Mil-L-23699 soaked	6.39 (5.3)	-2%	4.04 (2.7)	0%
Thermal Cycled	1.15 (25.4)	-82%	2.97 (9.1)	-27%
Mil-L-7808 soaked + TC	0.86 (30.3)	-87%	2.85 (6.6)	-30%
Mil-L-23699 soaked + TC	1.55 (16.9)	-76%	3.30 (5.8)	-19%

* Percent Coefficient of Variation

7. Concluding Remarks

This paper evaluated the effect of environmental conditioning on tensile and flexural properties of Cytec T650 carbon/Lonza Primaset PT-30 cyanate ester composite rods manufactured by the pultrusion process. The five types of environmental conditioning were: (1) soaking the rods in Mil-L-7808 lubricant oil for 720 hours; (2) soaking the rods in Mil-L-23699 lubricant oil for 720 hours; (3) thermal cycling the rods for 600 cycles; (4) soaking the rods in Mil-L-7808 oil for 720 hours and then thermal cycling the rods for 600 cycles; and (5) soaking the rods in Mil-L-23699 oil for 720 hours and then thermal cycling the rods for 600 cycles. A special high-rate heating/cooling furnace was built, verified, and then used for thermal cycling samples. The heating and cooling rate achieved was about 73 °C/minute (132 °F/minute). A special tabbing and tensile gripping method was developed and tested. Tensile strength, modulus, and fracture strain were determined and failure modes were recorded. Three-point bend flexure tests were conducted to measure stiffness, failure load, and failure modes. This is a qualitative test to measure the degree of fiber-matrix interfacial and matrix degradation due to lubricant oil soaking and thermal cycling.

The tensile strength of T650/PT30 pultruded rod was 1.76 GPa (254.6 ksi) with 4% coefficient of variation (CV) and was little affected by soaking in either of the lubricant oils for 720 hours. The same trend was noted for fracture strain. The modulus was not affected by the oil soaking procedures. The microscopy work showed that the oil soaking did not affect the fiber-matrix interface except for the color change.

Thermal cycling the samples at RT-600°F for 600 cycles did reduce the tensile strength by 28% and the strain by 30%. However, the tensile modulus remained unchanged (less than 3%). The optical and SEM microscopy showed that thermal cycling caused a widespread fiber-matrix interfacial separation and matrix shrinkage, in addition to symptoms of matrix oxidation. The interfacial separation did impact the tensile strength and fracture strain and flexural stiffness. For the samples that were oil-soaked prior to thermal cycling, the tensile and flexural properties closely matched those of the samples which were only thermal cycled. A large reduction in flexural stiffness was due to reorientation of fibers caused by interfacial separation because of fiber-matrix degradation under thermal cycling condition.

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