

**Evaluation of high-modulus pitch/cyanate material systems
for dimensionally stable structures**

R. A. Brand

E. A. Derby

Composite Optics, Incorporated
San Diego, California

ABSTRACT

Dimensional stability in composite structures has mainly focused on near-zero CTE, high-modulus fiber/epoxy resin systems. However, hygrothermal stability has been demonstrated to be a serious concern for structures moisturized on earth and dried in orbit. Composite sealing techniques have been developed to prevent this moisture absorption and desorption with the concomitant dimensional changes. New resin systems are being developed which absorb significantly less moisture and show promise as optional systems in dimensionally stable structures. These resin systems have not been evaluated for their physical and mechanical properties.

This paper describes the testing of various high-modulus, cyanate ester resin systems for evaluation in dimensionally stable composite applications. Physical testing included moisture absorption testing, coefficient of thermal expansion, and hygrothermal strain change. Mechanical testing included tensile, compression, Iosipescu shear, and bonded joint allowables.

A comparison of the thermomechanical properties for the cyanate ester resin systems is made with a P75S/ERL1962 baseline epoxy resin system.

1. INTRODUCTION

Dimensionally stable composites have been employed for over 16 years in advanced space structures. Until recently, epoxy resin systems were exclusively employed as the matrix material. Epoxies have had some shortcomings, most notably in the moisture expansion area. Specific sealing approaches¹ were developed to minimize these effects. The development of new hydrophobic resin systems such as cyanate esters has provided alternative resins with unique properties. This paper addresses the physical and mechanical properties of several fiber/resin systems based on cyanate ester density.

Cyanate resin systems first appeared in the literature during the 1960s², but concerted investigations into this novel class of thermosetting materials did not begin until the early 1980s^{3,4,5,6}. Cyanates are formed by the reaction of a cyanogen halide with a phenol. In this manner, multifunctional resins can be synthesized. Cyanates react by the trimerization of the cyanate group into a cyanurate ring. This polymerization is usually accomplished with the addition of a transition metal salt⁷.

Unlike epoxy resins, cyanates do not generate hydroxyl (-OH) groups on polymerization. The hydroxyl group is thought to be an association site for diffusing moisture. As a result, their low moisture absorbing characteristics have led to the development of cyanates as matrix resin for composites. Cyanates were originally developed as bismaleimide substitutes (350°F use temperature, wet) in aircraft composites. The application of cyanates in space structures proved to be fortuitous. Since composites are generally sensitive to environmental influences (e.g., water), a low-moisture absorbing composite held potential for inherently dimensionally stable materials.

A thorough evaluation of the physical and mechanical properties of the cyanate composite is essential before the cyanate is proven to be a viable material for composite design. Cyanate composite materials have only recently become commercially available; therefore, mechanical property values are rather sparse.

The purpose of this paper is to present a summary of the physical and mechanical properties generated on selected high modulus pitch/cyanate quasi-isotropic material systems.

2. MATERIAL SELECTION CRITERIA

The materials employed to obtain dimensionally stable composites must have certain physical and mechanical properties which together yield a material with:

- A "near-zero" CTE (<0.05 ppm/°F)
- Minimal hygroscopic response (low β coefficient)
- Minimal moisture absorption
- An absence of microcracking on thermal cycling

In addition to these properties, maximization of the "normal" engineering properties (e.g., tensile and compression strength/modulus, shear strength, and interlaminar strength) is also desirable. Since most space structures are weight-critical, mechanical fasteners are often avoided and bonded joints are substituted; thus interlaminar strengths (shear and tension) become a very important consideration in bonded structures. The evaluation of a new material for use in dimensionally stable structures must encompass and evaluate all of these key properties.

As shown in Table 1, a total of five different material configurations were chosen for evaluation. Of the five material systems tested, three different fiber and three different resin systems were utilized. Table 2 gives the properties of the pitch-based fibers used in the current study. With the exception of the FT500 fiber system (70 msi), the nominal modulus of the fiber systems was 75 msi. The 75 msi fiber system was chosen as the baseline configuration due to the fact that the corresponding quasi-isotropic laminate would result in a near-zero CTE laminate.

Table 1

Material Systems Evaluated

Material	Cure Ply Thickness (mil)	Vendor
XN50/RS3	2.5	YLA
P75S/RS3	2.5	YLA
FT500/954-3	2.5	ICI/Fiberite
P75S/954-3	2.5	ICI/Fiberite
P75S/ERL1999	2.5	Amoco
P75S/ERL1962 (Epoxy) (Baseline)	2.5	Amoco

Table 2

Graphite Fiber Properties

Property	Units	P75S	XN50	FT500
Tensile Modulus	msi	75	75	70
Tensile Strength	ksi	270	470	> 430
Density	PCI	0.072	0.077	0.077
Ultimate Strain	%	0.4	0.66	0.6
Coefficient of Thermal Expansion	PPM/°F	-0.7	-0.72	-0.55

A summary of the overall testing conducted for each material system is presented in Tables 3A and 3B.

Table 3A

Cyanate Composite Test Matrix

Test Condition	Laminate Orientation	Laminate Thickness	Test Direction	Number of Specimens	Specimen Preconditioning
Tension Modulus (msi) Strength (ksi) ASTM D3039	(0,45,90,135) _s	0.04	0 & 90	12	N/A
	(0,45,90,135) _s	0.04	0 & 90	12	N/A
Compression Modulus (msi) Strength (ksi) ASTM D3410	(0,45,90,135) _s	0.08	0 & 90	12	N/A
	(0,45,90,135) _s	0.08	0 & 90	12	N/A
In-Plane Shear Modulus (msi) Strength (ksi) Iosipescu Test	(0,45,90,135) _s	0.08	0 & 90	12	N/A
	(0,45,90,135) _s	0.08	0 & 90	12	N/A
CTE (200°F to -200°F) (PPM/°F)	(0,60,-60) _s	0.04, 0.06, 0.08	0 & 90	2	Cycled 10 times 200°F to -200°F
CME (PPM/%M)	(0,60,-60) _s	0.04	0	2	55% R.H.

Table 3B

Cyanate Composite Test Matrix

Test Condition	Laminate Orientation	Laminate Thickness	Test Direction	Number of Specimens	Specimen Preconditioning
Bonded Joints Lap Shear Gr-to-Ti Gr-to-Gr Butt Joints Tension Shear	(0,45,90,135) _s	0.04	0 & 90	12	Thermal Cycled 10 times 150°F to -100°F
		0.04	0 & 90	12	
	(2)*	0.04, 0.06, 0.08	0	12	Thermal Cycled 10 times 150°F to -100°F
		0.04, 0.06, 0.08	0	12	

(2)* Both laminate configurations (i.e., [0,45,90,135]_s and [0,60,-60]_s)

Preimpregnated unidirectional graphite tape (0.0025 in./ply cure thickness) was obtained for evaluation so as to minimize the possibility of microcracking when cycled down to cryogenic temperatures.

The P75S/ERL1962 epoxy material system was included to serve as a baseline for comparison purposes.

3. LAMINATE FABRICATION

Based on the test requirements outlined in Table 1, a total of three different quasi-isotropic panels (i.e., $t = 0.04, 0.06,$ and 0.08 in.) were laid up and cured for each material system (for a total of 21 panels) at 350°F for 2 hours per COI's processing specification (COPS-0001-6). Panel sizes of 4 ft by 8 ft for the 0.04-in.-thick laminates and 4 ft by 4 ft for the 0.06- and 0.08-in.-thick laminates were fabricated to ensure that all the specimens would come from the same panel cure.

3.1 Specimen preparation

Final specimen preparation consisted of rough-cutting the specimens via water jet cutting techniques, then final machining to size using a high-speed router with a diamond bit. All specimen edges were then final dressed with 400 grit or finer abrasive paper to remove any flaws from which failure may be initiated during subsequent testing.

All bond joint specimens were prepared by grit blasting the faying surface, followed by an isopropyl alcohol wipe prior to bonding. In the case of titanium, all bond surfaces were etched and primed with BR127 prior to bonding. Hysol's EA9394 adhesive system was used in all bonding operations.

4. TEST METHODS

4.1 Mechanical testing

All mechanical testing was performed on an Instron Model 4505 test machine at a cross-head rate of 0.050 in./minute.

4.1.1 Tensile tests

Tensile tests were conducted according to the procedures outlined in ASTM D3039 using a 1.0-in.-wide by 9-in.-long test specimen. Fiberglass doublers 0.03 in. thick by 2.0 in. long with a 5° taper were bonded to the specimen using 3M's 2216 adhesive system. A nominal specimen thickness of 0.04 in. was utilized in all testing.

The tensile stress-strain response of each specimen was measured using an Instron (1.0 in. gage) longitudinal extensometer. In order to measure Poisson's ratio, three specimens from each configuration were instrumented with a (0/90) strain gage.

4.1.2 Compression tests

Compression testing was conducted according to the procedures outlined in ASTM D3410 using a specimen 0.5 in. wide by 4.6 in. long with a gage section of 0.5 in. Fiberglass doublers 0.03 in. thick by 2.0 in. long were bonded to the specimens using 3M's 2216 adhesive system. Uniformity of bondline thickness was controlled by the use of 0.005 in. glass microbeads. A nominal specimen thickness of 0.08 in. was utilized in all tests.

In order to detect possible eccentricity effects or Euler buckling, three specimens from each orientation were instrumented with back-to-back longitudinal strain gages. The remaining specimens were instrumented with a single longitudinal gage. Specimens were tested using a Wyoming modified Celanese compression test fixture.

4.1.3 In-plane shear tests

In-plane shear modulus and strengths of the quasi-isotropic laminates were determined using the Iosipescu test method as defined in COI specification SP-T-017.

The test specimens were 0.75 in. wide by 3.0 in. long with a nominal thickness of 0.08 in. A 90° notch was cut into each of the upper and lower edges of the specimen at mid-length to a depth of 0.169 in. with a notch tip radius of 0.035 in. Fiberglass doublers 0.03 in. thick by approximately 1.25 in. long were bonded on each side of the specimen using 3M's 2216 adhesive system. The purpose of the fiberglass doublers was to increase the bearing area under the load points. A two-element ($\pm 45^\circ$) strain gage rosette was bonded to each specimen in the central region between the notches. The strain readings from each of the tensile and compression gages were monitored independently as opposed to connecting them in a half-bridge as is commonly done. The basic concept behind this approach was that the minimal acceptance of an in-plane shear result would require that equal (in magnitude) readings between the tension and compression gages be maintained throughout the test.

The specimens were tested in an Iosipescu shear test fixture manufactured by Wyoming Test Fixtures, Inc.

4.1.4 Coefficient of thermal expansion (CTE) testing

Coefficient of thermal expansion (CTE) measurements were conducted using an in-house-developed laser optical comparator technique⁹ capable of measuring to a resolution of 5×10^{-8} in/in. A coupon size of 1.75 by 6.0 in. was utilized. The specimens were cycled between +200°F and -200°F a total of ten times at a maximum heatup/cooldown rate of 10°F/minute. The CTE coupons were then vacuum dried at 220°F for a minimum of 72 hours or until constant weight was obtained prior to testing.

The CTE coupons were then affixed to ULE standards and stabilized under vacuum/heat (+150°F) until no strain changes were detected using the laser optical comparator. The coupons were then cycled from -200°F to +200°F and strain readings taken approximately every 45°F.

4.1.5 Coefficient of moisture expansion (CME) testing

The coefficient of moisture expansion (CME) measurements were conducted using an HP5528A laser interferometer. A standard 1.75 by 6.0 in. specimen was used for the CME testing.

Additionally, the CME coupons had slotted ceramic beads bonded to the mid-point of the specimen ends using EA9394 epoxy adhesive. The coupons were dried to constant weight at 200°F under vacuum (28 in. mercury, minimum). Individual specimens were then placed in a constant humidity container (55, relative humidity, room temperature). Daily weight gains were noted to 0.1 mg and the concurrent strain change was measured in a HP5528A laser interferometer using an Invar standard maintained at constant temperature.

The resulting CME values were determined by the ratio of $(\Delta L/L)/(\Delta W/W)$ at saturation.

4.1.7 Tensile lap shear testing

Single lap shear testing of graphite-to-graphite and graphite-to-titanium joint configurations were conducted in accordance with the procedures outlined in ASTM D1002. The specimens were 10.25 in. long by 1.0 in. wide with an overlap length of 0.72 in. Adherend thicknesses of 0.04 in. were utilized throughout. A nominal bondline thickness of 0.007 in. was maintained for all specimens via microbeads. The specimen configuration was modified so that the line of action of the load would be through the center of the adhesive bondline. Prior to testing, each specimen was cycled ten times from +150°F to -100°F at a maximum rate of 10°F/minute.

4.1.8 Bonded joint tests

Bonded joint strengths were generated for both tensile and shear loading conditions using the specimens shown in Figure 1. Special bond fixtures and test fixtures were designed and built to control specimen alignment during bonding as well as load alignment during subsequent testing.

5. TEST RESULTS AND DISCUSSION

5.1 Mechanical properties

The mechanical properties and strengths of the various quasi-isotropic material configurations are presented in Table 4. Fiber-dominated properties, such as tensile and compression strengths and modulus, have been normalized to 60% fiber volume. A discussion of these results is presented below.

Table 4
Room Temperature Mechanical Properties and Strengths of
Quasi-Isotropic Graphite/Cyanate Ester Laminates

	XN50/ RS3	P75S/ RS3	FT500/ 954-3	P75S/ 954-3	P75S/ ERL1999	P75S/ ERL1962
TENSILE						
Modulus (msi)						
0-Degree Direction	14.5	14.31	12.37	14.39	16.3	14.43
90-Degree Direction	14.67	14.25	11.86	14.78	16.06	14.69
STRENGTH (ksi)						
0-Degree Direction						
Mean	73.6	46.2	75.4	42.3	50.4	59.2
90-Degree Direction						
Mean	97.2	53.7	84.6	47.0	56.8	61.8
Poisson's Ratio						
NUXY	0.343	0.339	0.359	0.336	0.342	0.331
NUYX	0.372	0.334	0.394	0.359	0.349	0.333
COMPRESSION						
Modulus (msi)						
0-Degree Direction	9.3	10.51	8.8	9.83	12.98	10.88
90-Degree Direction	10.09	10.54	8.6	10.79	12.43	11
Strength (ksi)						
0-Degree Direction						
Mean	33.6	29.05	32.5	27.5	28.3	31.7
90-Degree Direction						
Mean	31.5	26.8	27.9	27.7	30.3	33.4
IOSIPESCU SHEAR						
0-Degree Direction						
Modulus (msi)	3.99	4.74	4	4.31	4.77	4.78
Strength (ksi)						
Mean	26.6	19.95	21.3	21.2	20.2	22.5
90-Degree Direction						
Modulus (msi)	3.68	4.24	3.69	4.68	4.87	4.47
Strength (ksi)						
Mean	26.5	19.95	20.7	19.4	20.5	25.0

5.1.1 Tensile properties

It can be seen from Table 4 that with the exception of Fiberite's FT500/954-3 material system, the normalized tensile modulus fall in the range of 14.25-16.3 msi. In order to compare the materials on an equivalent basis, the tensile modulus was taken as the slope of the stress-strain curve between 800-1600 microstrain. Good correlation between moduli in the 0° and 90° directions were obtained. With the exception of the P75S/ERL1999 material system, the tensile moduli of the remaining 75 msi material systems were tightly bounded. A similar trend is also reported for compression modulus and CTE values. Variations of these magnitudes would be representative of a longitudinal fiber modulus variance of 75 ± 5 msi, which is not unreasonable for this type of fiber. However, this would indicate that the effective fiber modulus for the P75S/ERL1999 system is in the 75-77 msi range, while the remaining P75S material systems are in the range of 70-71 msi.

Testing of unidirectional laminates fabricated from each type of material is currently underway in order to confirm or dispute these suspicions. While the tensile moduli for the various materials may be nearly equivalent, the same is not true for strengths. YLA's XN50/RS3 system results in a tensile strength nearly 47% higher than the corresponding P75S material systems. A greater difference in strengths between the 0° and 90° directions has also been noted.

5.1.2 Compression properties

Compressive properties (modulus and strengths) reported in Table 4 indicate a substantial reduction in both modulus and strength when compared to the corresponding tensile properties. A discussion of these results is presented below.

As previously mentioned, the longitudinal compressive stress-strain curves were generated from back-to-back strain gages. Generally, the curves from the two strain gages were similar. In some cases, however, strain divergence was seen at the higher loads indicating possible instability (buckling) effects.

The average stress-strain curves for each specimen were determined by averaging the strain readings from the back-to-back gages. The compression modulus for each specimen was determined by averaging the slopes of the stress-strain curves between 500-1000 microstrain.

Compression moduli of 10-12 msi have been reported for the various pitch 75 msi fiber systems. These types of values are typical of the values reported for the baseline P75S/ERL1962 material system.

Reductions in modulus between the tensile and compressive loading conditions can be traced back to the fiber itself. Typical unidirectional tensile and compressive moduli of 45 msi and 36 msi, respectively, have been reported in the literature for 60% fiber volumes.

The resulting compressive strengths are nearly equivalent for all material configurations.

5.1.3 In-plane shear properties

In-plane shear modulus and strengths reported in Table 4 were obtained via the Iosipescu test method. A discussion of these results is presented below. As previously mentioned, the in-plane shear stress-strain curves for each of the two gages (+45 and -45) were monitored separately. In materials where the tensile and compressive moduli are identical, a state of pure shear would result in both strain gages having the same magnitude.

For the current material systems in which the tensile and compressive moduli are different, it is more important that a biaxial state of stress be produced in which the stresses monitored by each gage are equivalent in magnitude, but opposite in sign. Thus a state of pure shear is produced on a plane oriented at 45° to the gages (i.e., center of the test specimen). In such a case, the resulting strain gages would differ by a factor of the ratio of the tensile to compressive modulus of the material.

Results indicate that the differences between the tensile and compressive strain gages were nearly identical to the ratio of the tensile and compressive moduli. Thus, a state of pure shear in the test area was produced. The resulting shear modulus was calculated based on the average of the tensile and compressive strain readings.

The ratio of the tensile to compressive strains became larger as the stress levels increased. Thus the resulting shear modulus was calculated from the initial portion of the shear stress-strain curve assuming an average of the tensile and compressive strain gages. A typical plot of stress versus strain for the two gages is presented in Figure 2.

Average ultimate shear strengths for each material system were within the range of 20-28 ksi. A detailed review of the failed specimens indicated that bearing failure under one of the loading pins closest to the center notch occurred in nearly 85% of the total specimens. Unfortunately, the doublers that were added to increase the bearing area were terminated just prior to this location.

Additionally, delaminations emanating from this location as well as in the notch region were observed in a large number of the specimens. The failure mode observed for most specimens was very complex. Previous investigations¹⁰⁻¹⁴ have shown the state of stress within a quasi-isotropic laminate is three-dimensional near the free edges. Thus the stress distribution in an Iosipescu shear specimen at the notch root for a quasi-isotropic laminate could be dominated by interlaminar stresses. This may account for the delaminations which were observed in many of the specimens. The ultimate stresses reported in Table 4 are therefore considered conservative.

5.2 Coefficient of thermal expansion (CTE) tests

The coefficient of thermal expansion for the quasi-isotropic laminates summarized in Table 5 was measured over the range of +200°F to -280°F. The values reported in Table 5 represent a best fit of a straight line through the data. A typical CTE plot is presented in Figure 3.

Normalization of the CTE data presented in Table 5 was conducted through a combination of micromechanic techniques and laminate test data. Based on a combination of vendor data, and test data generated under current and past programs at COI for various laminate configurations, a consistent set of fiber properties for the P75S fiber and the cyanate resin systems has been generated.

Micromechanic techniques¹⁵ were then used in combination with standard lamination theory to predict the thermoelastic properties of a quasi-isotropic laminate as a function of fiber volume fraction. A plot of predicted CTE versus fiber volume fraction for a range of fiber moduli is presented in Figure 4. Given the fiber volume fractions of the individual panels, the CTE data presented in Table 5 may be normalized to 60% FVF using the trends presented in Figure 4. Results of this normalization process are presented in Table 6. By comparing the results presented in Tables 5 and 6, may be seen that some of the differences in CTE can be attributed to variations in laminate/panel FVFs. However, a significant difference still exists between the P75S/ERL1999 material system and the others.

It is believed that the remaining difference may be attributed to variations in the effective fiber modulus of the various materials. Fiber certifications obtained from the vendors tend to confirm these suspicions. However, a more detailed evaluation is currently underway.

It can be seen from Figure 4 that variations in fiber modulus on the order of 75 ± 5 msi can more than account for the variations seen in Table 6.

Table 5

Coefficient of Thermal Expansions for
Quasi-Isotropic Graphite/Cyanate Ester Laminates

	XN50/ RS3	P75S/ RS3	FT500/ 954-3	P75S/ 954-3	P75S/ ERL1999	P75S/ ERL1962
CTE (PPM/°F) +200°F TO -280°F						
PANEL: .040						
0-Degree Direction	-0.04	-0.142	0.181	-0.094	-0.314	-.12
90-Degree Direction	-0.068	-0.13	0.106	-0.13	-0.285	
PANEL: .060 (0,90)						
0-Degree Direction	-0.028	----	0.164		-0.225	----
90-Degree Direction	0		0.071	----	-0.25	
PANEL: .080 (0,90)						
0-Degree Direction	-0.035	-0.127	0.146	-0.107	-0.13	----
90-Degree Direction	-0.016	-0.12	0.098	-0.145	-0.237	

Table 6

Normalized Quasi-Isotropic CTEs

Panel	XN50/RS3	P75S/RS3	FT500/954-3	P75S/954-3	P75S/ERL1999
0.040	-0.085/-0.11	-0.142/-0.13	0.15/0.075	-0.094/-0.13	-0.329/-0.30
0.060	-0.0861/-0.053	-0.127/-0.12	0.164/0.071	--	-0.231/-0.256
0.080	-0.09/-0.075	--	0.170/0.059	-0.131/-0.169	-0.192/-0.299

5.2.1 Onset of microcracking

Prior to the initiation of the CTE tests, it was important to determine the temperature at which microcracking initiates for each of the materials being evaluated. This is important due to the fact that if this temperature is exceeded during thermal cycling, microcracking of the resin system is initiated, microcracking occurs, and the resulting CTE is unstable until which time a saturated crack density is obtained. Once stabilized the material is considered to be stable from a CTE standpoint as long as this new temperature range is not exceeded.

The approach normally taken in establishing the temperature at which microcracking occurs is via CTE testing. The specimen is continually cycled between a specified upper temperature bound and a continually colder and colder temperature extreme.

Any shift in the slope of the CTE curve usually indicates the formation of microcracks. Microsections of the specimens are normally taken in order to confirm that microcracking has occurred.

The approach taken under the current program, in order to save time, was to conduct a worst case scenario in which the specimens were wrapped in aluminum foil and placed in a Dewar containing liquid nitrogen (LN₂) for a total of 5 minutes. The specimens were then removed from the LN₂, unwrapped, and allowed to warm to room temperature. The specimens were then microsectioned and observed under a microscope (50X) in order to see if the material had microcracked.

A total of two 2 in. by 2 in. test specimens were cut from each cured panel and segregated into two control groups for subsequent testing. The first group was kept in a vacuum oven at 200°F for approximately 28 days prior to tests while Group 2 specimens were stored at room temperature (70°F) and 50% R.H. for approximately 28 days prior to testing.

Results of these studies indicate that no microcracks were observed in any of the material systems investigated. The microcracking temperature may therefore be considered to be somewhere below liquid nitrogen temperature (i.e., less than -310°F).

5.3 CME testing

Saturation values for weight gain and strain changes presented in Table 7 were extrapolated from an approximately 80% saturation condition assuming linear behavior for the strain changes and by employing the moisture diffusion model of reference 8 for the weight gain.

After approximately 80% saturation at $55 \pm 5\%$ R.H., the CME results (Table 7) indicated that the XN50/RS3 and the FT500/954-3 have about the same β coefficient (165 average) with the same quantity of moisture absorbed. The Amoco P75S/ERL1999 CME results indicated a low strain response to moisture; however, the quasi-isotropic modulus of this specimen was 15% higher than the XN50/RS3. The quantity of moisture absorbed at saturation is almost the same as the XN50/RS3 system. The Fiberite P75S/954-3 results indicate a similar lower response to moisture. These coupons, however endured several moisturization and dry-out cycles. In each subsequent moisturization cycle, the strain response to a given quantity of moisture was reduced, perhaps indicating a post-curing effect.

The interpretation of CME is not a straightforward proposition. The modulus of the laminate and the hygrothermal history of the panel appear to have an effect on the β coefficient. Clearly, more investigation into the hygroscopic response of the high-modulus cyanate ester laminates is necessary. Generally, the strain change of a cyanate is about one-third to one-fourth that of an epoxy laminate of equivalent modulus.

5.4 Tensile lap shear tests

Single lap shear test results for graphite-to-graphite and graphite-to-titanium joint configurations with an overlap length of 0.72 in. are presented in Table 8.

Failure was primarily within the first ply of the graphite laminate nearest to the adhesive bondline. Peel failure was also evident due to a slight eccentricity in the load path.

Lap shear results for the graphite/cyanate material system are equivalent to those of the baseline epoxy system.

5.5 Butt joint tests

Testing is currently underway. Preliminary results indicate that the cyanate material systems are nearly equivalent to the corresponding epoxy material systems.

Table 7

Coefficient of Moisture Expansion for Quasi-Isotropic Graphite/Cyanate Ester Laminates

	XN50/RS3	FT500/954-3	P75S/954-3	P75S/ERL1999	P75S/ERL1962
CME (PPM/%M) 55% R.H.	173	157	105	112	162
$M_{MAX} = 55\% \text{ R.H.}$ %M @ 55% R.H SATURATION	0.14	0.15	0.18	0.13	0.42

Table 8

Tensile Lap Shear Tests

Vendor Fiber Matrix	XN50/RS3	FT500/954-3	P75S/ERL1999	P75S/ERL1962
Number of Specimens	12	15	13	17
GR/GR (Ftu) (psi)	1173.9	872.1	1018	1027
Number of Specimens	15	15	13	16
GR/TI (Ftu) (psi)	1226	1229	1103	1081

NOTE: Specimens cycled 10 times between +150°F and -100°F prior to testing at R.T.

6. CONCLUSIONS

- The mechanical properties for the various graphite/cyanate quasi-isotropic laminate configurations are nearly equivalent to those of the baseline epoxy system.
- Variations in mechanical properties between P75S/ERL1999 and the remaining cyanate ester systems is thought to be attributed to variations in fiber modulus.
- The resulting quasi-isotropic CTE for the graphite/cyanate material system is slightly more negative than the corresponding epoxy system due to the lower modulus of the cyanate ester resin system (e.g., 0.4-0.5 msi).
- The cyanate ester resin system moisture absorption values were significantly lower than the baseline epoxy system.
- The CME of the cyanate ester material systems are generally equivalent to the baseline epoxy system. However, the associated strain is significantly lower for the cyanate ester resin systems.

7. REFERENCES

1. Krumweide, G.C., Derby, E. A., and Chamberlin, D. N., "The Performance of Effective Moisture Barriers for Graphite/Epoxy Instrument Structures," SAMPE, Atlantic City, NJ, 1989.
2. Grigat, E. and Putter, R., *Ange W/. Chem Int. Ed.*, pp. 6, 206, 1967.
3. Delano, C. B., McLeod, A. H., and Kiskiras, NASA Contract Final Report FR-80-42, Contract NAS3-22025, 1980.
4. Brand, R. A., and Harrison, E. S., NASA Contract Report 3615, Contract NAS1-15859, September 1982.
5. Shimp, D. A., *Proceedings of the 32nd International SAMPE Symposium*, pp. 1063-1072, 1987.
6. Shimp, D. A., Huddock, F. A., and Ising, S. J., *Proceedings of the 33rd International SAMPE Symposium*, PP. 754-766, 1988.
7. Shimp, D. A., *ACS PMSE, Prepr* 84, 107, April 1986.
8. Brand, R. A., "A Probabilistic Model for Moisture Diffusion in Graphite/Resin Laminates," 13th International SAMPE Conference, Hamburg, Germany, 1992.
9. Stumm, J. E., Pynchon, G. E., "Thermal/Hygro Response Measurement and Behavior of Selected Composite Systems," Testing, Evaluation and Quality Control Conference (TEQC83), 1983.
10. Spiegel, B. S., "An Experimental and Analytical Investigation of the Iosipescu Shear Test for Composite Materials," M.S. Thesis, Old Dominion University, Norfolk, Virginia (August 1984).
11. Walrath, D. E. and Adams, D. F., "Iosipescu Shear Properties of Graphite Fabric/Epoxy Composite Laminates," Report No. UWME-DR-501-103-1, Department of Mechanical Engineering, University of Wyoming, Laramie, Wyoming, (NASA Grant No. NAG-1-272) (June 1985).
12. Adams, D. F., and Walrath, D. E., "In-Plane and Interlaminar Iosipescu Shear Properties of Various Graphite Fabric/Epoxy Laminates," *Journal of Composites Technology and Research* (1987).
13. Spiegel, B. S., Sawyer, J. W., and Prabhakaran, R., "An Investigation of the Iosipescu and Asymmetrical Four-Point Bend Tests for Composite Materials," *Proceedings of the 1985 SEM Spring Conference on Experimental Mechanics*, Society for Experimental Mechanics, pp., 35-44 (June 1985).
14. Adams, D. F., Walrath, D. E., "Current Status of the Iosipescu Shear Test Method," *Journal of Composite Materials*, Volume 21, June 1987.
15. Kibler, J.J., Ph.D., "Integration of Theoretical and Practical Micromechanics Procedures into the Design and Analysis of Composite Materials," *Proceedings of the Eighth International Conference on Composite Materials*, SAMPE, 1-R, pp. 1-12.

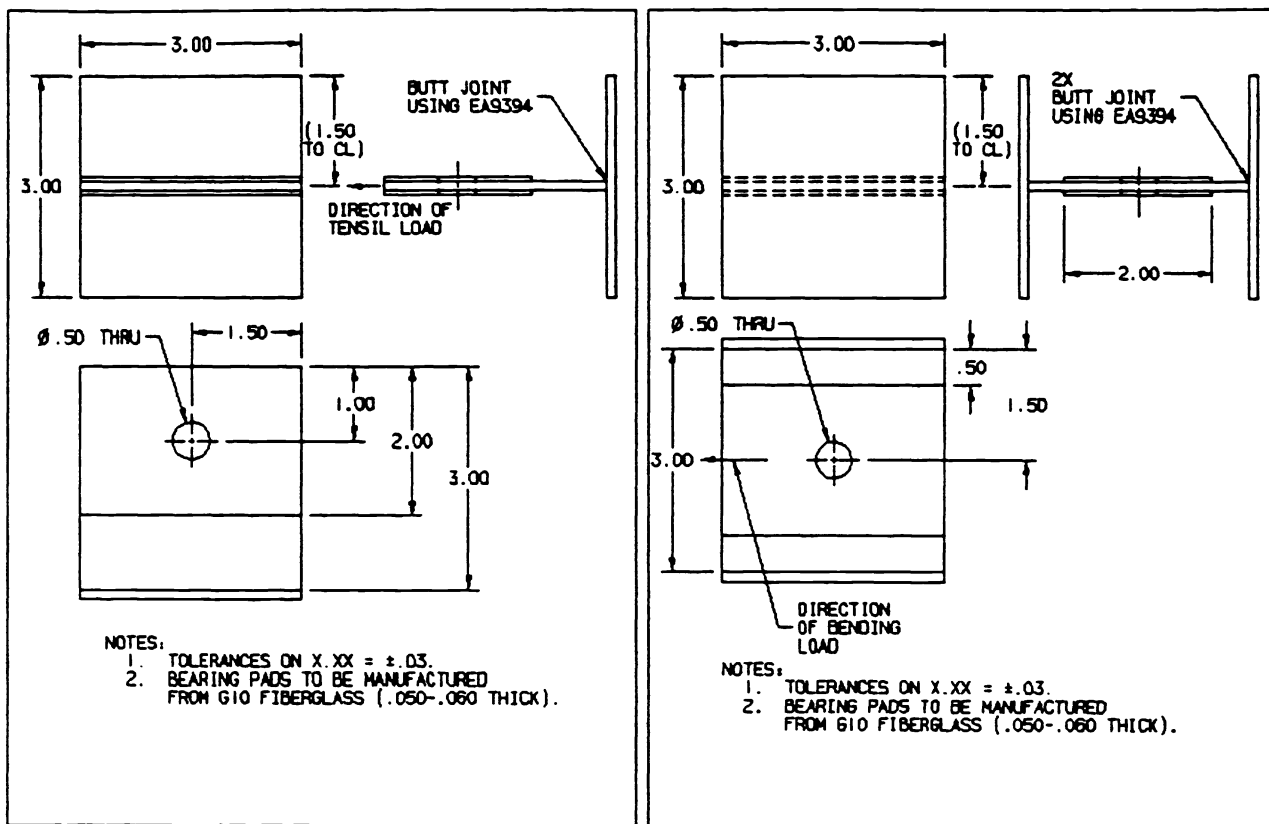


Fig. 1A Tensile Joint Specimen Configuration

Fig. 1B Shear Joint Specimen Configuration

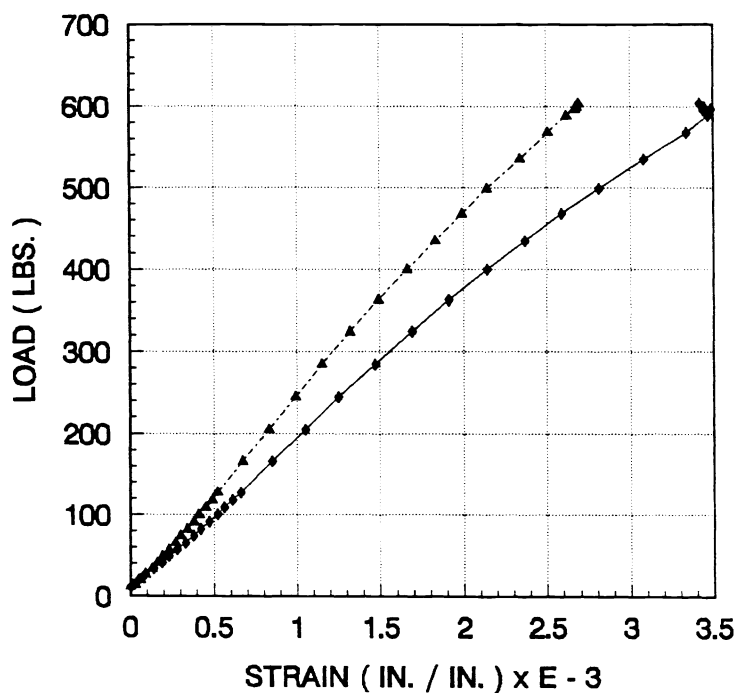


Fig. 2 Iosipescu Shear Test Response
 P75S/ERL1999 Specimen #4

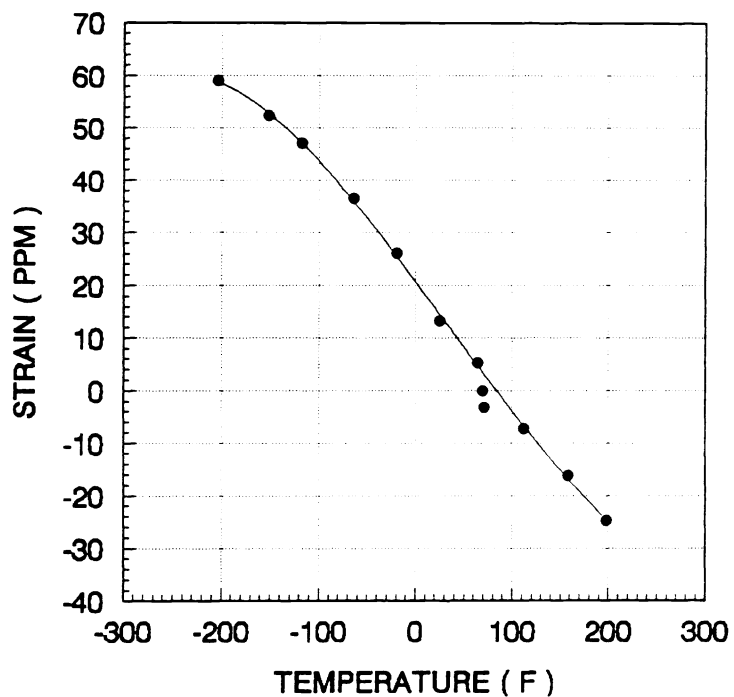


Fig. 3 Typical Thermal Expansion Curve
P75S/ERL1999 Quasi-Isotropic Laminate

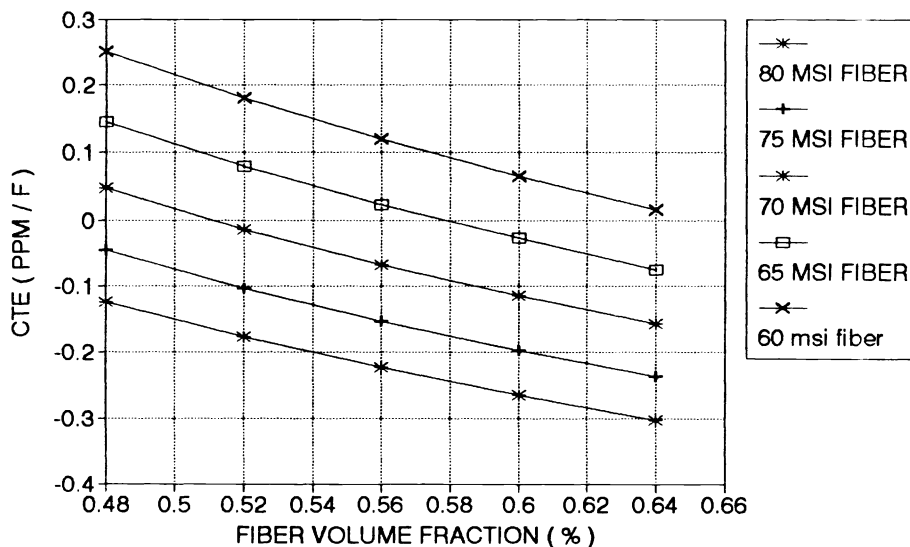


Fig. 4 CTE vs. Fiber Volume Fraction for a Quasi-Isotropic Graphite/Cyanate Material as a function of Longitudinal Fiber Modulus