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SELECTION OF FIBERGLASS MATRIX RESINS FOR INCREASED TOUGHNESS AND ENVIRONMENTAL RESISTANCE IN WIND TURBINE BLADES

by

John F. Mandell, Daniel D. Samborsky, Mei Li and Ricardo Orozco
Department of Chemical Engineering
and
Douglas S. Cairns,
Department of Mechanical Engineering
Montana State University
Bozeman, MT 59717

ABSTRACT

The DOE/MSU Fatigue Database has been expanded to include a number of matrix resins of potential interest in wind turbine blades. The main considerations in resin selection have been to increase the structural integrity (such as delamination resistance) in blades while maintaining or improving other mechanical properties, particularly under hot, wet conditions. The resins included in the study are also appropriate for the wind turbine blade application in terms of cost and processing characteristics (all materials were prepared by resin transfer molding). Resins included unsaturated polyesters, vinyl esters, epoxies, and a urethane. Mechanical properties have been obtained for wet and dry specimens tested at temperatures from -20 to 70°C. Fatigue, delamination resistance (Mode I and II crack growth), and performance in stiffened substructure sections have been evaluated for selected cases. Significantly improved performance relative to baseline polyester is shown for several resins.

INTRODUCTION

Wind turbine blades should perform under a variety of loads and environmental conditions for a twenty to thirty year service life. Fiberglass blade materials derive much of their strength and stiffness from the fiber reinforcement. However, several key properties are dominated by the matrix resin, including resistance to delamination between plies and compressive strength. Delamination is a dominant failure mode in composite material structures, leading to the breakdown of structural integrity in areas such as the trailing edge, spars, and root connections. Experience in aerospace composites [1] indicates that the toughness of the matrix resin, as well as the design of details controls interlaminar fracture resistance and structural performance, as well as facewise impact resistance [2]. The low cost matrix resins (general purpose polyesters, vinyl esters, and epoxies) used in most turbine blades are relatively brittle, and so the delamination resistance of most blade materials is relatively low. Tougher versions of these and other resins are investigated in this study. A second type of resin, thermoplastics, also have high toughness, but their high viscosity limits their use in conventional blade manufacturing techniques. Tougher resins which bond well to the fiberglass also tend to give higher strengths in off-axis directions relative to the fiber reinforcement.

A second concern with matrix resins is that if their elastic moduli are not high enough, they do not support the fibers adequately against compressive buckling. Thus, a softer matrix will produce a lower compression strength for loads along the fiber axis, usually the lengthwise direction of the blade. Compression strength and fatigue resistance are design drivers of primary importance. Typical matrix resins used in blades, such as ortho polyesters, generally have adequate elastic modulus at moderate temperatures to provide good compressive strength. However, at elevated temperatures and with high moisture contents, these resins may not retain sufficient modulus (a neat resin modulus of around 3.0 GPa is usually adequate). Resins such as polyesters and epoxies will generally absorb several weight percent

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moisture, which swells and softens the polymer network (reversibly) and reduces the elastic modulus and glass transition temperature (T_g). Toughened resins can have reduced modulus relative to the base resin if toughness is achieved through the addition of low modulus materials like elastomers.

This study evaluated a number of base and toughened resin systems which are suitable for common blade manufacturing processes (including resin transfer molding (RTM), which requires a low resin viscosity). Resin cost was limited to about $3.00/lb to be competitive in blade applications, which eliminated many of the toughened aerospace resins. The main objective was to evaluate resins with improved toughness and temperature and moisture resistance as compared with common blade resins.

EXPERIMENTAL METHODS

All materials were resin transfer molded in closed molds, including neat resin samples (without reinforcement), which were molded into their final dog-bone shape without machining. Types and sources of resins and reinforcement are listed in Table 1. Test methods for static and fatigue tension and compression followed standard procedures described in detail in Reference 3.

Delamination resistance in Modes I and II used unidirectional 0° double cantilever beam (DCB) and end notched flexure test specimens [4,5]. These specimens used a teflon crack-starter strip embedded during fabrication as an initial crack. The Mode I fracture toughness, G_{IC}, was determined after a short increment of crack growth beyond the starter strip; this value is termed the initial G_{IC} to distinguish it from higher values, which result from fiber bridging as the crack grows longer. The Mode II value, G_{IIC}, was determined using the Mode I specimens after the crack was grown for several cm, with the specimen then loaded in three-point bending for Mode II. These methods are described in References 1 and 6. The structural integrity was evaluated with the T-section pull-off test shown in Figure 1 and described in detail in Reference 5. The typical load-displacement curve in Figure 2 was used to determine the initial damage force, the maximum force, and the displacement at maximum force.

RESULTS AND DISCUSSION

Matrix Resin Properties

Table 1 lists the resins studied, all of which are thermoset polymers. Further details of the resins and their processing can be found in Reference 6. Figure 3 gives prices quoted (spring 1999) for each resin in 55 gallon drums for a total of 40,000 lb. lots. Prices can vary significantly. More costly resins with improved properties are also available, but are not studied here.

Figure 4 compares tensile stress-strain curves for several of the neat resins, and Table 2 lists their properties. Due to difficulty in preparing neat resin specimens, such as the urethane matrix, some resin properties are not included in Table 2. The target modulus of 3.0 GPa is not achieved by the 980 vinyl ester, SC-14 epoxy, or the urethane. The stress-strain curves for the more brittle resins such as unmodified polyesters and epoxies can vary significantly depending on sample molding and machining procedures. The yield strength is taken as the 0.2% offset yield strength where this could be determined. Table 2 gives heat deflection temperatures measured for each resin. This may be taken as an upper use limit.

The moisture absorption characteristics of several resins are shown in Figure 5 as weight gain in distilled water at 50°C vs. square root of time in hours, following typical Fickian diffusion representation. As expected, the vinyl esters and the iso-polyester absorb much less moisture than the ortho-polyester and the epoxy. The composites (Figure 6) absorb less moisture, since the entire composite is not resin, but the ordering of the materials according to weight gain is consistent with the neat resin data.

Interlaminar Fracture Toughness

Figures 7 and 8 summarize the Mode I and Mode II interlaminar fracture toughness, respectively, for selected resin systems. Additional data are given in Ref. 6. The baseline ortho- polyester has a very low $G_{IC,}$ typical of the lowest cost polyesters, vinyl esters and epoxies. The other matrices have significantly higher Mode I toughness. All systems have increased Mode I toughness at 50°C wet conditions due to increased fiber debonding and fiber bridging, as found in other composites [7]. The Mode II toughness in Figure 9 tends to correlate more closely with the T-stiffener test, described below. The toughened vinyl ester and epoxy SC14 show the highest

G_{IIC} values at room temperature, dry, but the epoxy loses Mode II toughness at elevated temperature, particularly when conditioned and tested wet. The iso-polyester has higher G_{IIC} than the ortho-polyester, particularly at elevated temperatures. The two vinyl esters show very good toughness under all conditions. While the vinyl ester and epoxy toughness values are slightly lower at -20 °C than at room temperature, the differences do not indicate any ductile-brittle transitions in this temperature range.

T-Stiffener Pull-off

Figure 9 shows typical T-stiffener pull-off specimens after testing; see Figure 1 for the test configuration. These show the usual delamination-dominated fracture mode, simulating separation of the skin-spar interface area of blades. The damage has been modeled in detail and associated with the basic G_{IC} and G_{IIC} results in Reference 5. Figure 10 compares several load-displacement curves from the pull-off tests, and Table 3 lists results for seven resin systems. The tougher resin systems produce increased stiffener pull-off resistance, as expected. Since slight thickness differences can affect this test significantly [5], the results should be viewed in terms of both the force levels and the displacement, with higher values of both indicating greater structural integrity. The room temperature G_{IC} and G_{IIC} values for the untoughened System 41 epoxy were 231 J/m^2 and 3776 J/m^2, respectively [6]. Thus, there is a good correlation between G_{IIC} (Figure 8) and T-stiffener pull-off resistance, including the System 41 matrix, which had a surprisingly high G_{IIC} for an unmodified epoxy (this system was not tested at elevated temperature, but has a high heat distortion temperature in Table 3).

Composite Strength and Modulus vs. Temperature and Moisture Condition

Figures 11-17 give basic composite mechanical properties for composites fabricated with five of the more interesting resins as a function of temperature, both for dry (ambient) conditioned specimens and for specimens conditioned for approximately 45 days in 50°C distilled water. The laminates were either $[0/\pm45/0]_s$ tested at 0° or 90° or $[(\pm45)_3]$ tested at 0° as indicated.

Figure 11 gives the most critical matrix sensitive property: compression strength in the 0° direction. The compression strength decreases moderately for dry specimens up to 70°C, with the greatest decrease shown in the ortho polyester. The wet conditioned and tested specimens show even greater decreases, particularly the ortho-polyester and the epoxy (which also absorbs the most moisture, Figure 5). The iso-polyester and both vinyl esters are much less sensitive to moisture. The sensitivity of the ortho polyester composite to moisture at elevated temperature for longer times is even more significant, as shown in Table 4, with reductions of 26% and 30% under hot-wet conditions for composites based on D155 and A130 0° fabrics, respectively. These are very serious decreases, particularly for the A130 fabric, whose woven architecture gives a low baseline compressive strength.

Tension properties in the 0° direction are fiber dominated, and are not much affected by temperature and moisture (Figures 12 and 13). The same laminate tested in tension in the 90° direction is more matrix sensitive, showing decreases in modulus which parallel the compressive strength (Figure 14); 90° tensile strength (Figure 15) is surprisingly insensitive. The ±45° laminates tested in tension in the 0° direction are also matrix dominated, giving significant temperature and moisture sensitivity (Figures 16 and 17).

Fatigue Resistance

The fatigue sensitivity has been found to be matrix insensitive in earlier results [3]. Figure 18 compares the baseline ortho-polyester with the two Derakane vinyl esters under tensile, compressive, and reversed loading, R = 0.1, 10, and –1, where R is the ratio of minimum to maximum stress in each cycle. Again, there is no significant improvement in fatigue resistance, even for the toughened vinyl ester 8084. Fatigue crack resistance for the systems is currently being tested. Future work will also include fatigue at hot, wet conditions, but experience with carbon/epoxy material suggests little change in the fatigue S-N curves when normalized by the static strength [8].

CONCLUSIONS

More ductile resin systems produce improved structural integrity at moderate cost. The hot/wet properties are much better for the iso-polyester and vinyl ester systems than for the ortho-polyester or the epoxy SC14, again for moderate cost increases over the ortho-polyester. Thus, while the iso-polyester provides improved environmental resistance over the ortho polyester for a small increase in cost, the 411 and 8084 vinyl esters additionally provide much greater toughness and structural integrity for a slightly greater cost increase. The fatigue resistance was not affected by the matrix, following earlier results.

REFERENCES

1. Hunston, D.L., Moulton, R.J., Johnston, and Bascom, W.D., in Toughened Composites, ASTM STP 937, N.J. Johnston, Ed., American Society for Testing and Materials, Phil., 1987, pp. 74-94.

2. Hunston, D.L., Composites Technology Review, vol. 6, 1984.

3. Mandell, J.F. and Samborsky, D.D, DOE/MSU Composite Material Fatigue Database: Test Methods, Materials, and Analysis, Report SAND97 – 3002, Sandia National Laboratories, Albuquerque, NM (1997).

4. Russell, A.J., and Street, K.N., in Delamination and Debonding, ASTM STP 876, W.S. Johnston, Ed., American Society for Testing and Materials, Phil., 1986, pp. 349-370.

5. Cairns, D.C, Haugen, D.J, Mandell, J.F., and Samborsky, D.D., Proc. 1998 ASME Wind Energy Symposium, ASME International, New York, pp. 334-343.
6. Orozco, R. “Effects of Toughened Matrix Resins on Composite Materials for Wind Turbine Blades,” M.S. Thesis, Dept. Chemical Engr., Montana State University, Bozeman, MT. 1999.

7. Mandell, J.F, and Tsai, J.Y., “Effects of Porosity on Delamination of Resin Matrix Composites,” Report WRTC-TR-89-3032, Flight Dynamics Laboratory, Wright Patterson Air Force Base, Ohio (1990).

8. Rotem, A. and Nelson, H.G., in Fatigue of Fibrous Composite Materials, ASTM STP 723, American Society Testing and Materials, Phil., 1981, p. 152

Table 1. Materials Investigated

MATRIX MATERIALS			
Resin	Description	Product Description	Supplier
Ortho-polyester	orthophthalic	63-AX-051	Interplastics Corporation
Iso-polyester	isophthalic	75-AQ-010	
PET P460 polyester	PET modified orthophthalic	PET P460	Alpha Owens Corning
Vinyl ester 980	rubber toughened	Swancorp 980	TECTRA Incorporated
Vinyl ester 411C-50	unmodified	Derakane 411C-50	Dow Chemical
Vinyl ester 8084	rubber toughened	Derakane 8084	
Epoxy system 41	unmodified	System 41	System Three
Epoxy SC-12	acrylate modified	SC-12	Applied Poleramic Inc.
Epoxy SC-14	acrylate modified	SC-14	
Urethane	unmodified	Poly 15-D65	Polyteck Development Co.
FIBER REINFORCING FABRICS			
Fabric	Type		Supplier
0° Unidirectional fabrics			
D155	Stitched unidirectional		Owens Corning Fabrics
A130	Woven unidirectional		
±45° Fabrics			
DB120	Bias, stitched		Owens Corning Fabrics

Table 2. Tensile and Thermal Properties of Neat Resins

Resin	UTS, MPa	0.2% Offset Yield Strength, MPa	Modulus, GPa	Failure Strain, %	Heat Deflection Temperature, °C
Ortho-polyester	54.1 (4.6)[1]	45.2 (2.5)	3.18 (0.12)	2.0 (0.3)	55 (0.9)
Iso-polyester	34.6 (2.8)	----	3.32 (0.14)	1.2 (0.2)	69 (1.2)
Vinyl ester 980	25.7 (0.3)	20.6 (0.5)	1.63 (0.02)	30 (15)	60 (1.7)
Vinyl ester 411C-50	57.7 (0.8)	50.4 (2.5)	3.21 (0.04)	2.1 (0.1)	78 (3.7)
Vinyl ester 8084	72.6 (2.7)	55.2 (2.4)	3.25 (0.15)	3.0 (0.3)	75 (1.4)
Epoxy System 41	52.6 (1.1)	52.6 (1.1)	3.56 (0.06)	1.6 (0.1)	56 (3.6)
Epoxy SC-12	44.3 (3.1)	----	3.48 (0.04)	1.4 (0.1)	95 (1.2)
Epoxy SC-14	68.3 (2.7)	48.5 (1.3)	2.80 (0.03)	3.3 (0.3)	83 (1.9)
[1] Numbers in parentheses indicate the sample standard deviation.					

Table 3. Effects of Matrix on T-Stiffener Pull-off Resistance (average values).

Resin	Initial Damage Load, N/cm[1]	Maximum Load, N/cm	Displacement at Maximum Load, mm	Specimens Tested
Ortho-polyester	87 (6)[2]	135 (6)	6.8 (0.6)	3
PET P460 polyester	120	164	8.4	1
vinyl ester 980	119 (9)	182 (6)	13.5 (1.8)	4
vinyl ester 8084	144	194	9.0	2
epoxy System 41	168	209	6.7	2
epoxy SC-14	132	192	19.1	2
urethane	141	262	11.6	1
[1]N per cm of T specimen width, [2]Numbers in parentheses indicate the sample standard deviation.				

Table 4. Effect of Moisture Exposure and Elevated temperature Testing on Compressive Strength of [0/±45/0] Laminates. Distilled Water Conditioning at 40 °C for the First 5000 Hours, Followed by 20°C Conditioning. (Ortho-polyester, D155 and A130 0° Fabrics, $V_F = 0.36$)

Exposure Time, hours	Test Temperature, °C	Average Weight Gain (S.D.), %		D155 Ave. strength (S.D.), MPa	% Change	A130 Ave. strength (S.D.), MPa	% Change
		D155	A130				
0	20	0	0	517 (39)	--	265 (39)	--
0	50	0	0	472 (57)	-9.5	250 (17)	-5.7
24	20	0.20 (0.01)	0.29 (0.03)	516 (19)	-0.3	262 (55)	-0.8
144	20	0.47 (0.01)	0.54 (0.02)	481 (30)	-6.9	287 (27)	8.4
1,315	20	0.61 (0.06)	0.73 (0.04)	471 (35)	-9.0	219 (26)	-17
4,650	20	0.62 (0.11)	0.64 (0.08)	421 (31)	-19	240 (17)	-9.3
4,650	50	0.62	0.64	403 (30)	-15	174 (32)	-30
15,355	20	0.94 (0.25)	1.02 (0.05)	404 (31)	-22	203 (28)	-23
15,355	50	0.99 (0.22)	0.99 (0.04)	348 (34)	-26	175 (40)	-30

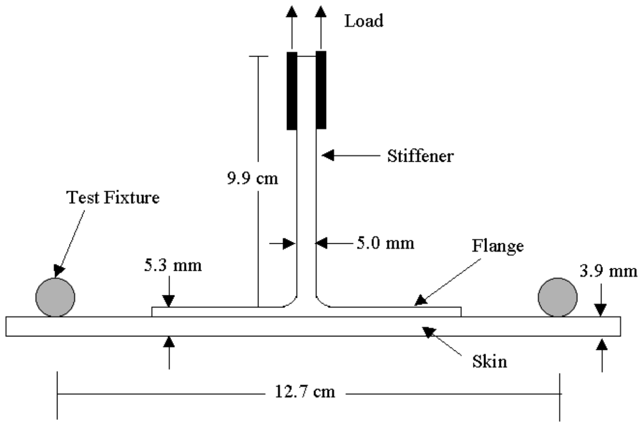

Figure 1. Loading and approximate dimensions for skin-stiffener T-specimens.

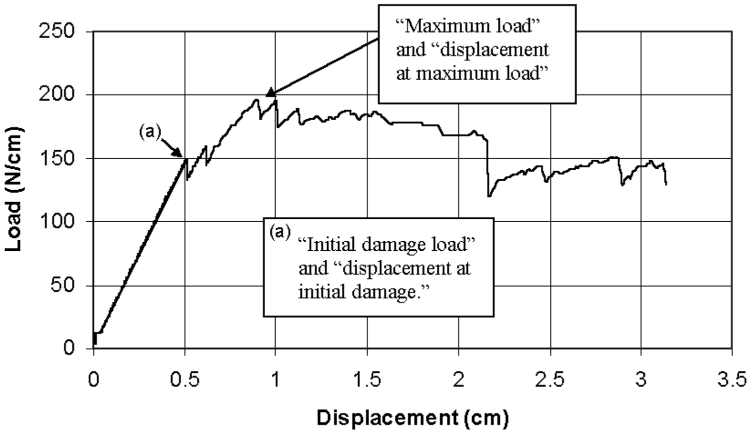

Figure 2. Typical load-displacement curve for a skin-stiffener specimen

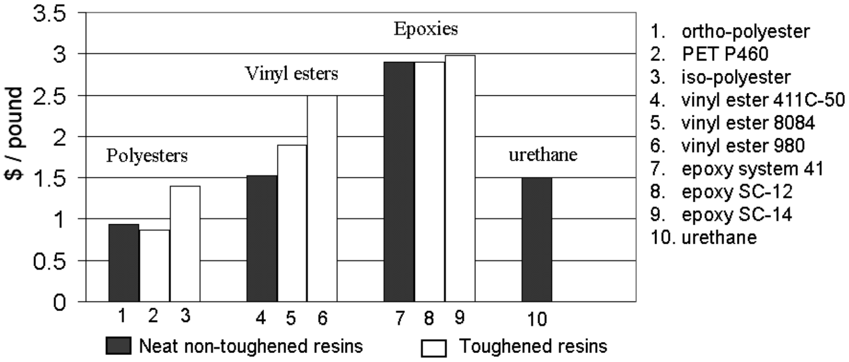

Figure 3. Price comparison for different resins (40,000 pound base estimation)

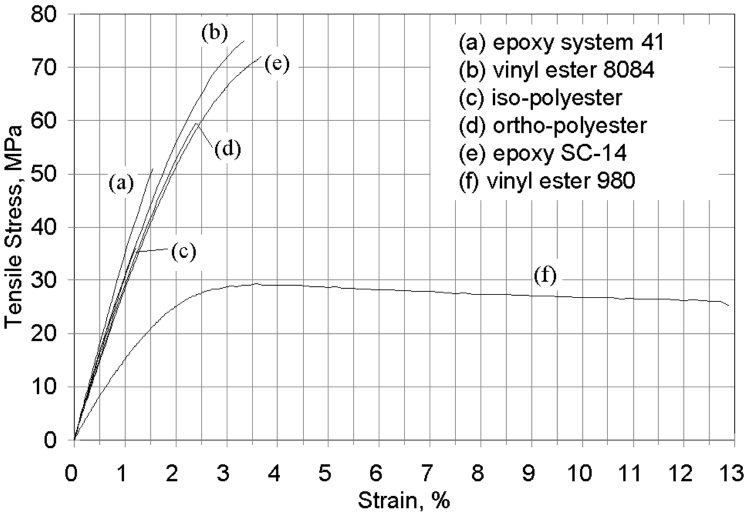

Figure 4. Stress-strain curves for neat resins.

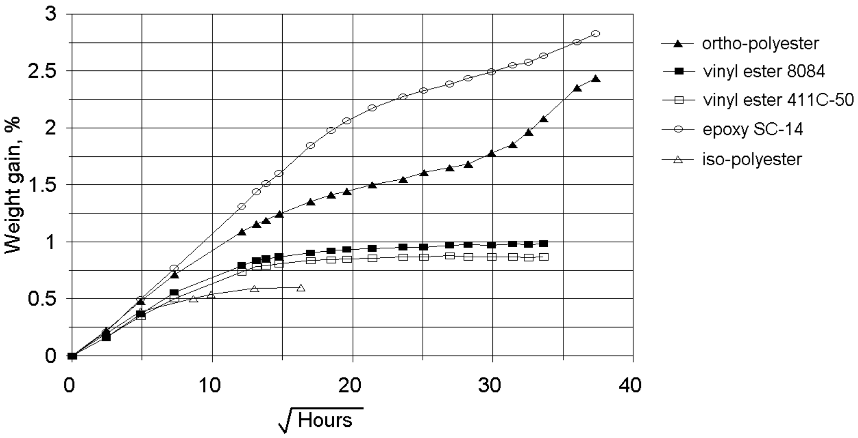

Figure 5. Water absorption for neat resin in distilled water at 50°C.

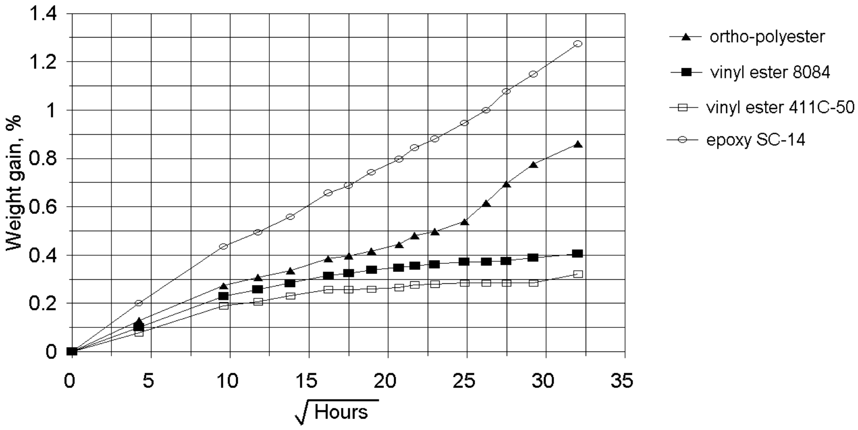

Figure 6. Water absorption at 50°C in distilled water for composites $[0/\pm45/0]_S$.

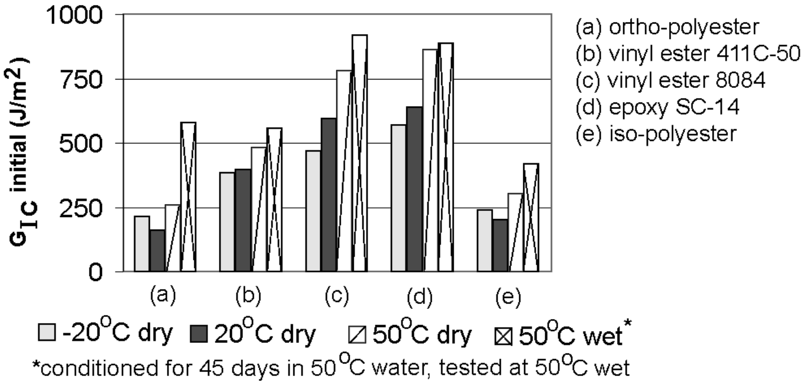

Figure 7. Effect of matrix on the initial mode one interlaminar fracture toughness (0 degree D155 fabric, $V_F = 0.36$)

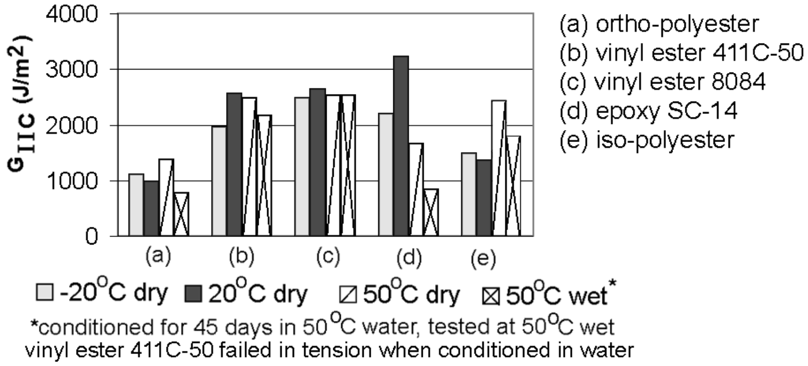

Figure 8. Effect of matrix on the mode two interlaminar fracture toughness (0 degree D155 fabric, $V_F = 0.36$)

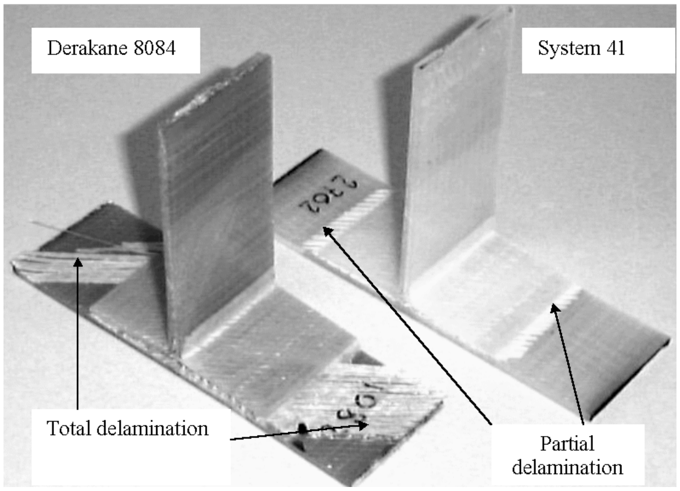

Figure 9. T-stiffener pull off specimens of vinyl ester 8084 and epoxy system 41, showing delamination damage.

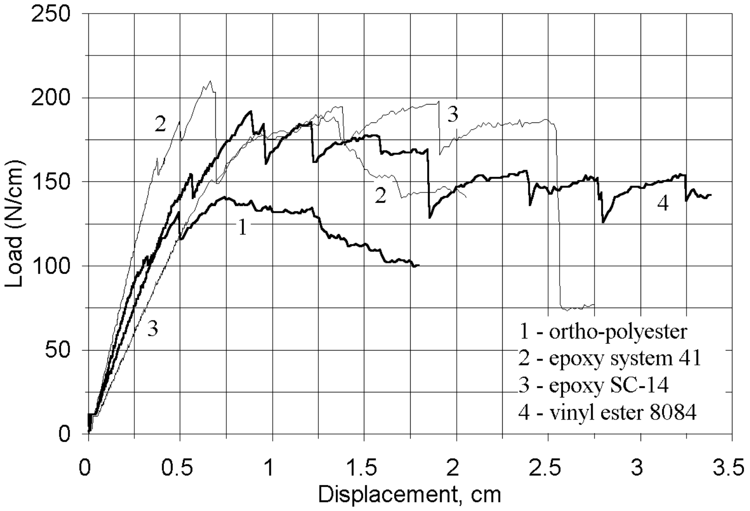

Figure 10. Typical Load-displacement curves for T-specimens

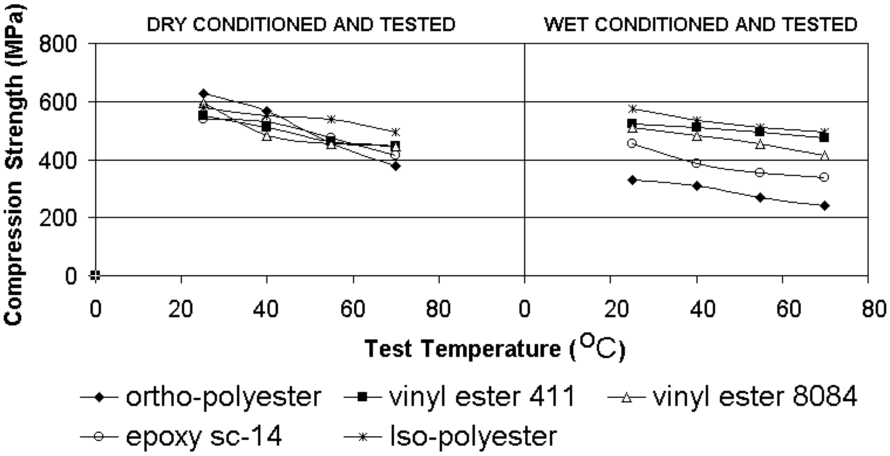

Figure 11. Compression strength in the 0° direction versus test temperature, dry and wet, $[0/\pm45/0]_S$ laminates.

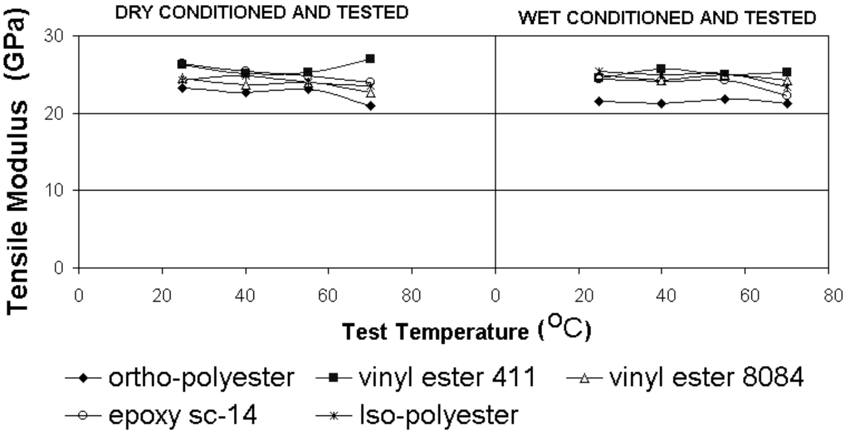

Figure 12. Tensile modulus in the 0° direction versus test temperature, dry and wet, $[0/\pm45/0]_S$ laminates.

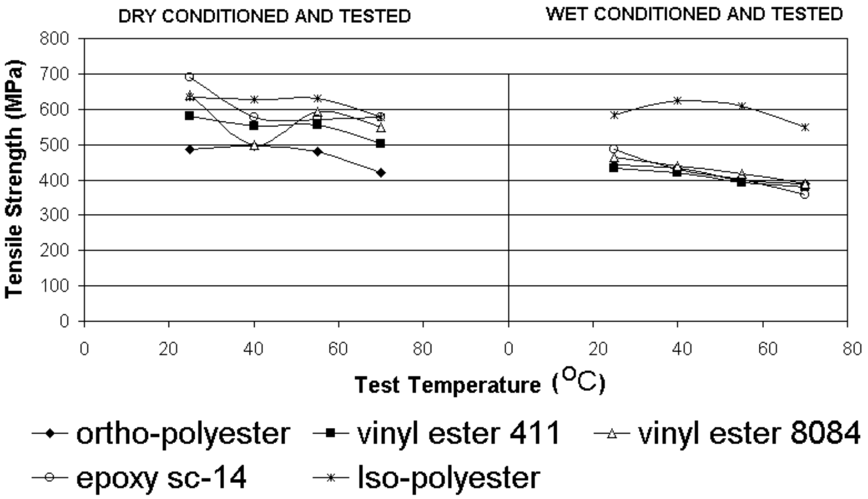

Figure 13. Tensile strength in the 0° direction versus test temperature, dry and wet, $[0/\pm45/0]_S$ laminates.

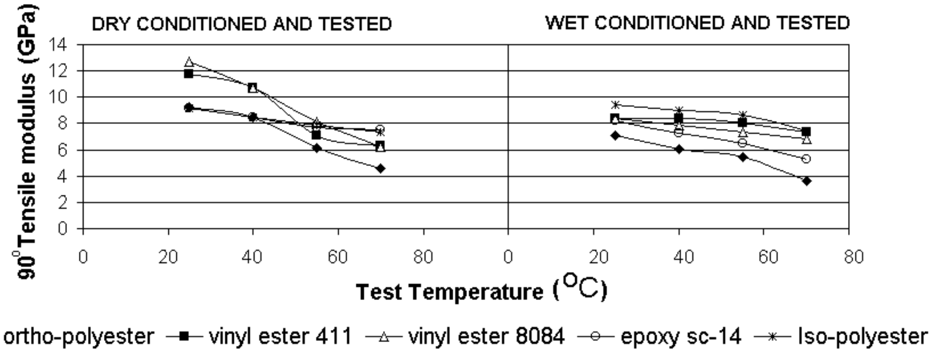

Figure 14. Tensile modulus in the 90° direction versus test temperature, dry and wet, $[0/\pm45/0]_S$ laminates.

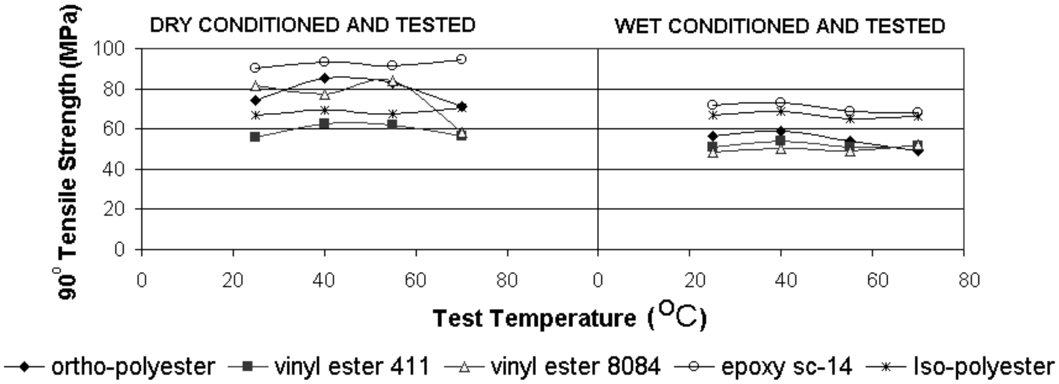

Figure 15. Tensile strength in the 90° direction versus test temperature, dry and wet, $[0/\pm45/0]_S$ laminates.

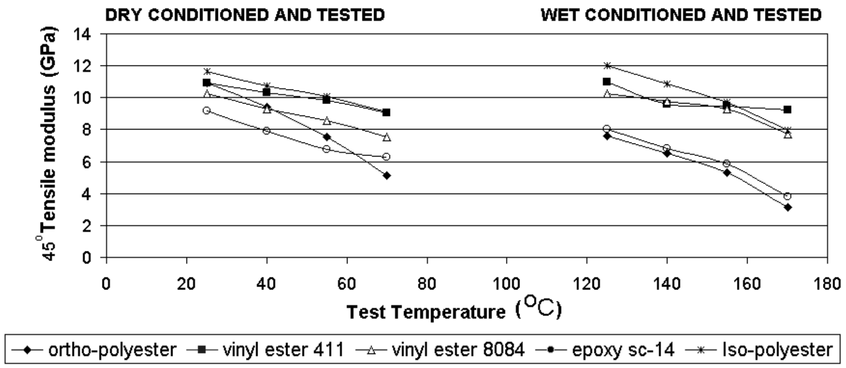

Figure 16. Tensile modulus in the 0° direction versus test temperature, dry and wet, $[(\pm 45°)_3]$ laminates.

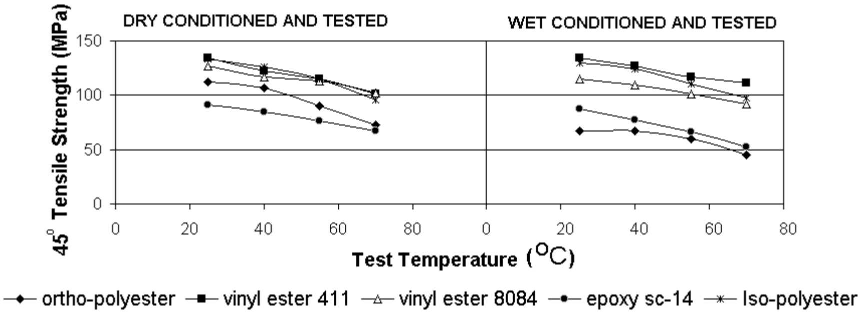

Figure 17. Tensile strength in the 0° direction versus test temperature, dry and wet, $[(\pm 45°)_3]$ laminates.

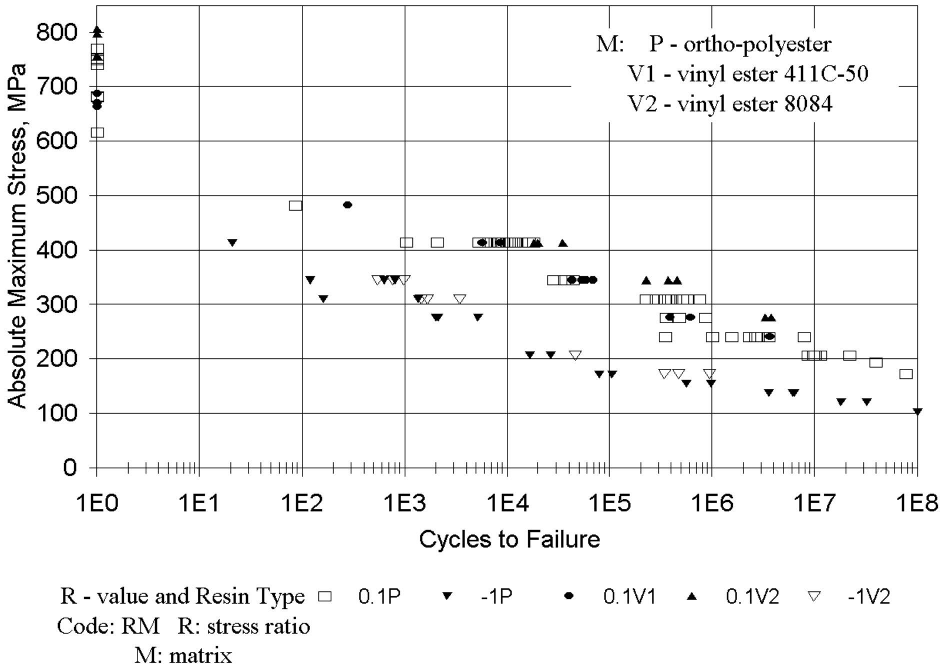

Figure 18a. Effect of matrix on fatigue resistance in the 0° direction under tensile (R=0.1) and reversed loading (R = -1); $[0/\pm 45/0]_S$ laminates, V_F = 0.34 - 0.36.

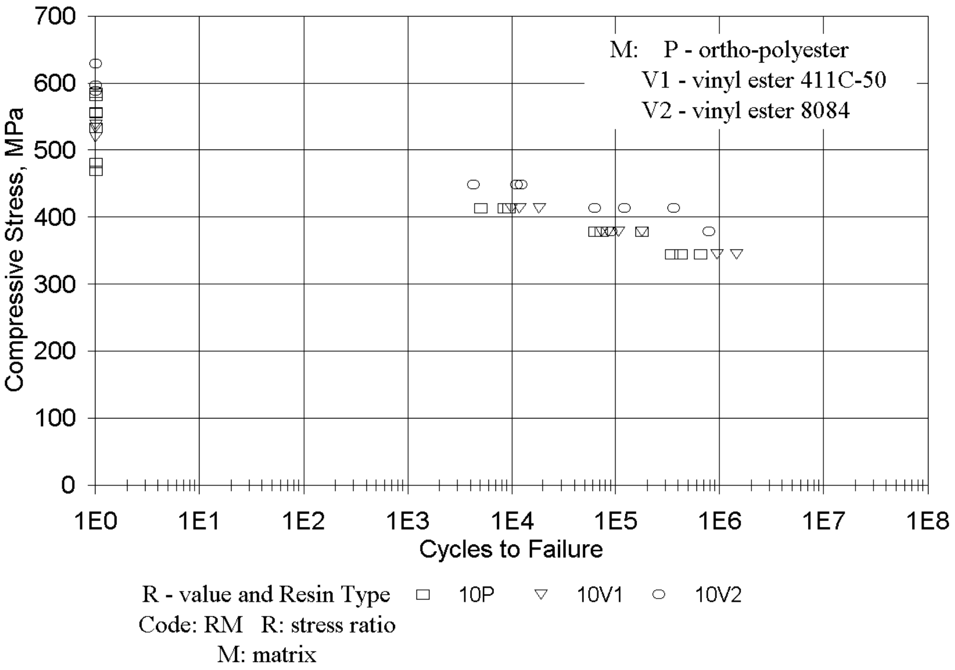

Figure 18b. Effect of matrix on fatigue resistance in the 0° direction under compression (R=10) $[0/\pm45/0]_S$ laminates, V_F = 0.34 - 0.36.