Carbon-Fiber-Reinforced Phenolics

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Synopsis

Carbon-fiber-reinforced (CFR) phenolic composites with equivalent mechanical properties and much superior thermal stability to laboratory-prepared and present commercial epoxy analogs have been achieved by reinforcing 35–50 wt % of an amine-catalyzed resole with either the Hercules' Magnamite carbon fabric or the Great Lakes' Varinit carbon fiber/glass cloth hybrid. Improvement of the interlaminar shear strength (ILSS) of the hybrid-reinforced phenolic laminates was observed with increasing molding pressure and alternate carbon/glass/carbon/glass geometry. Sizings on the carbon fiber do not have any significant effect on the mechanical properties.

INTRODUCTION

The most widely used thermosets for carbon-fiber-reinforced (CFR) composites are epoxy resins because of their ease of processability, good interfacial adhesion, and attractive mechanical properties. However, compared with resin matrices, like phenolics, epoxies generally have lower thermal stability and flammability drawbacks.

In view of the thermal and flame resistance of phenolic resins, this work was initiated to demonstrate, on a laboratory scale, a CFR phenolic molding materials with mechanical properties equivalent to present commercial CFR epoxy and with better thermal stability at elevated temperature.

A general survey of the literature shows an inadequate comparison between carbon-fiber-reinforced epoxy and phenolic composites. Goan and Prosen,[1] in their interfacial bonding studies, have compared composites made with phenolics and amine-cured epoxy systems, and reported that the CFR phenolic composites were generally inferior. Terwiesch et al.[2] in their comparison studies show, however, similar tensile strength, modulus, and interlaminar shear strength (ILSS) between the two CFR resin systems, and believe that a phenolic matrix would have better properties and thermal stability at elevated temperatures. In the work of Artis and Joiner,[3] surface-treated fiber tow was used for matrices which included epoxy, phenolic, polyesters, and polyimide resins. Their data show that phenolic is equivalent to epoxy in ILSS, though inferior in transverse flexural and tensile strengths. In spite of the controversies, it is generally recognized that, because of the volatiles evolved during the curing process of phenolic, void formation, which is detrimental to the composite properties,[4–7] has to be avoided. The main objectives of this work are (i) to identify some of the factors which influence the mechanical properties of CFR phenolic and (ii) to demonstrate on a laboratory scale that CFR phenolic composites have mechanical properties equivalent and thermal stability superior to CFR epoxy analogs.

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EXPERIMENTAL

Materials Used

Carbon Fabric and Fiber

1. Polyacrylonitrile-based carbon fabrics (Magnamite A193P and A370-8H) were purchased from the System Group, Hercules, Inc. Magnamite A193P is a high-performance graphite fabric, woven in a balanced plain weave construction, while Magnamite A370-8H is a high-performance graphite fabric woven in a balanced eight-harness, satin weave construction. Both fabrics have been surface-treated and epoxy-sized.

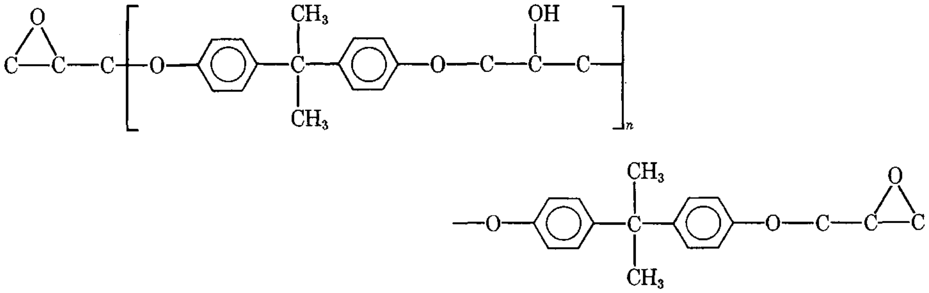
2. Carbon fiber/glass cloth hybrid (Fortafil-3 Varinit) was purchased from the Great Lakes Carbon Corp. The hybrid material consists of parallel untwisted continuous tow of (40,000 filaments) Fortafil-3 carbon fibers interlaid and adhered on a thin glass cloth tape (26.8 wt%) for ease of fabrication. The carbon fibers were surface-treated and supplied with no sizing, epoxy sizing, and phenolic sizing.

Phenolic Resins

The experimental phenolic (Durez A) resin was obtained from the Durez Division of Hooker Chemical and Plastics Corp. It is an amine-catalyzed thermosetting phenol–formaldehyde of the one-step type (resole) in ethanol solution containing 62.5 wt% solid. The viscosity of this resin solution is 300 cps at 25°C, $N_D = 1.5255$ with 0.4% free formaldehyde.

Epoxy Resins

1. The Epon 828 epoxy resin and the hardener DTA were purchased from Miller-Stephenson Chemical Co., Inc.; Epon 828 is a bisphenol A epichlorohydrin having the following repeating unit.



with 75% of the molecules having $n = 0$ and 25% of $n = 1$. The hardener DTA is diethylene triamine.

2. The Araldite 720 epxoy resin was purchased from Ciba-Geigy Corp. It is a tetrafunctional liquid resin which offers exceptionally good heat resistance. The hardener used is HY906 which is methyl 1,5-endomethylene tetrahydrophthalic anhydride.

Commercial CFR Epoxy Composite

A commercial carbon fabric reinforced-epoxy composite; A370-8H/3501-5A, based on Hercules' Magnamite A370-8H fabric and an undisclosed amine-cured general purpose epoxy (3501-5A), was obtained through the courtesies of the System Group, Hercules, Inc.

Fabrication Method

The fabricated specimens in this work consist of two types: (1) CFR bidirectional laminates based on woven carbon fabrics and (2) unidirectional CFR laminates based on the carbon fiber/glass fabric hybrid material. A wet hand layup technique was used to fabricate the laminates. About 10 plies of the carbon fiber material were precut to the desired (6 in. × 6 in. or 8 in. × 8 in.) dimension and weighed. A calculated amount of resin/hardener (in order to yield the desirable weight percent of the resin) was then weighed and mixed thoroughly. oughly.

A single ply of the carbon fabric was then placed on a polypropylene film. About 10% of the resin or resin/hardener was then transferred and spread evenly by a tongue depressor onto the fabric surface. The procedure was repeated with the subsequent nine remaining layers.

In the case where the carbon/glass hybrid material was used for the fabrication of the unidirectional laminates, the hybrid material was divided into two stacks of eight plies, each treated with the resin on the faceup, all parallel carbon fiber surface. The two stacks were subsequently combined with more resin. The amount of the resin was adjusted to yield a laminate of a desirable resin content. The CFR resin systems were then cured under the following conditions:

Resin	Precuring cycle	Curing cycle
Durez A	30 min at 125°C in oven	40 min at 165°C in Carver press; vented frequently for first 5 min (releasing vapors), gradually increased pressure to 285 psi (low pressure curing) or 785 psi (high pressure curing)
Epon 828/ DTA	40 min at 129°C in Carver press.	Cured at room temperature in Carver press without pressure for 2 h; pressure gradually increased to 785 psi as resin cured; overnight cured composite then post-cured at 110°C for 48 h
Araldite 720/ HY906	20 min at 129°C in Carver press	2.5 h at 180°C in Carver press at 785 psi pressure

Testing Methods

Tensile Strength and Modulus

The test was performed on samples of the dimensions in accordance to the ASTM D638 specifications. An Instron machine with a crosshead speed of 0.4 in./min was used.

Flexural Strength

The flexural properties were measured by the ASTM D790 Method I (three-point loading) on rectangular samples of 3-in. length, 1-in. width, and about ⅛-in. thickness. The selected span-to-depth ratio was 16:1. Crosshead speed on the Instron was 0.006 in./min.

Compressive Strength

The compressive strength measurement was carried out in accordance with ASTM D695 (less than 3.2 mm thickness) specification. Crosshead speed of 0.005 in./min was employed on the Instron machine using the compression mode.

Interlaminar Shear Strength

In this test, a three-point bend configuration was used, and the span length is sufficiently short to preclude tensile or compressive failures. A span to depth ratio of 5:1 was used for all specimens. The loading nose and supports have a diameter of 0.25 in. An Instron machine with a crosshead speed of 0.04 in./min and a load cell of 5620 lb was used.

Thermogravimetric Analysis (TGA)

The TGA measurements were carried out in air, at a heating rate of 10°C/min on a Mettler Thermoanalyzer-2.

Scanning Electron Microscopy

Scanning electron microscopy studies were carried out with a JEOL-JSM50A scanning electron microscope.

RESULTS AND DISCUSSIONS

Bidirectional CFR Laminates Based on Hercules' Magnamite Carbon Fabric: Comparison of Laboratory Prepared CFR Phenolic vs. Epoxy Analogs (50 wt % Carbon Fabric)

Mechanical Properties

Table I compares the mechanical properties of the laboratory-prepared CFR Durez A phenolic laminate vs. a general purpose fast-cured Epon 828/DTA and a high-temperature, slow-cured Araldite 720/HY906 analogs. It can be observed that comparable tensile flexural and interlaminer shear strengths are observed for both the phenolic and epoxy resin matrices, indicative of the suitablity of this phenolic as a resin matrix for CFR composites.

Thermal Stability

Table II compares the thermal stability of the above laminates by monitoring the percentage weight loss using thermogravitmetric analysis (TGA). It is in-

TABLE I
Comparison of Mechanical Properties between Laboratory-Prepared CFR Phenolic and Epoxy Laminates

Properties/sample	Unit	A	B	C
Type of fabric		Hercules Magnamite A 193 P		
Type of resin	—	Durez A phenolic	Epon 828/DTA epoxy	Araldite 720/HY906 epoxy
Wt % of resin		~50	~50	~50
Tensile strength	psi	59,000 ± 4000	55,000 ± 3000	60,000 ± 3000
Flexural strength	psi	75,000 ± 2000	70,000 ± 4000	74,000 ± 7000
Flexural modulus	psi × 10^6	5.10 ± 0.9	3.80 ± 0.2	5.4 ± 0.1
Interlaminar shear strength	psi	5500 ± 500	6200 ± 200	5100 ± 200

teresting to note that, although at low temperature (200–300°C) the CFR phenolics show some initial weight loss of up to 4%, due most probably, to low molecular weight volatiles or free monomers, the high-temperature stability of the CFR phenolics is very evident. At 500°C almost quantitative weight loss was observed for the general purpose Epon 828 epoxy and over 75% weight loss of the high-temperature Araldite epoxy. However, less than 25% weight loss was indicated by the Durez A phenolic resin system indicative of its higher thermal stability.

Unidirectional CFR Laminates Based on Great Lakes' Varinit–3 Carbon Fiber/Glass Cloth Hybrid

In order to identify the different factors that affect the mechanical properties of CFR phenolic laminates, the Great Lakes' Varinit carbon fiber/glass cloth hybrid, as a substitute for the woven carbon fabric was used in view of the following advantages:

1. Its lower cost as compared to that of the woven carbon fabric.
2. The availability of different sizings in the carbon fiber.
3. The different possible ways of stacking up the carbon fiber and glass cloth layers.

TABLE II
Comparison of Thermal Stability between Laboratory-Prepared CFR Phenolic and Epoxy Laminates

Sample	% Weight loss at								
	100°C	150°C	200°C	250°C	300°C	350°C	400°C	450°C	500°C
Laminate-A[a] (amine-catalyzed resole) Durez A	0	0.4	1.3	2.7	3.4	4.2	10.3	16.8	23.4
Laminate-B[a] (Epon 828/DTA) general purpose epoxy	0	0	0	0	1.6	22.6	78.4	94.1	96.6
Laminate-C[a] (Araldite 720/HY906) high-temperature epoxy	0	0	3.2	13.9	26.8	48.3	63.3	70.8	75.3

[a] With ~50 wt % of the resin (laminates on Table I).

TABLE III
Effect of Sizing on the Mechanical Properties of Unidirectional Phenolic Laminates Based on Fortafil-3 Varinit Hybrid

Properties	Unit	Formulations		
		D	E	F
Type of resin		Amine-catalyzed resole Durez A		
Sizing on carbon fiber	—	Unsized	Phenolic	Epoxy
Resin content	wt %	50	50	50
Tensile strength	psi	71,000 ± 8000	66,000 ± 5000	74,000 ± 5000
Flexural strength	psi	92,000 ± 11000	100,000 ± 3000	100,000 ± 8000
Flexural modulus	psi × 10^6	6.56 ± 0.29	6.86 ± 0.58	6.63 ± 0.56
Interlaminar shear strength	psi	5400 ± 3000	5800 ± 600	5000 ± 1000

4. The possibility of achieving unidirectional CFR laminates by stacking layers of the hybrid's carbon fibers parallel to one another.

5. The predicted reinforced transverse strength of the unidirectional CFR laminates due to the interlay glass cloth.

Factors Affecting the Mechanical Properties of CFR Phenolics

A. Effect of Sizing: Since the Varinit hybrid materials were supplied with no sizing, epoxy sizing, or phenolic sizing, a study of the effect of sizing on the mechanical properties of the corresponding unidirectional CFR phenolic laminates was carried out. Table III compares the mechanical properties of such laminates having 50 wt % of the Durez A resin. It can be observed that, in this phenolic system, the effect of sizing does not seem to be important since the mechanical properties do not deteriorate without sizing on the fiber and do not show any significant improvement when the fibers/resin are compatibly sized (i.e., phenolic with phenolic sizing). The very comparable interlaminar shear strength obtained with phenolic/epoxy or no sizing strongly suggest that sizing of the pre-surface-treated fiber is not important in improving the bonding efficiency or the ILSS in the present phenolic resin system.

B. Effect of Resin Content: A study was made to determine the effect of resin content on the mechanical properties of the CFR phenolic laminates. Table IV compares the mechanical properties of such laminates based on different weight percents of the Durez A resin. It is interesting to note that going from 50 to 30 wt % resin, the tensile strength and flexural modulus increase with decrease in the resin content to 40 wt %, after which the reinforcing effect seems to level off. The flexural strength appears to show a maximum at about 37.5–40 wt % resin content, although the statistical error is also high in the low resin content laminates. The high fluctuation in properties of samples E(5) and E(6) is due presumably to the uneven wetting between the resin and fiber (as reflected by the low ILSS) and is attributable to the nonuniform spreading of the low amount of resin. In view of the better wetting and consistent best properties shown by the laminates with 35–40 wt % resin, this range of resin content is chosen and maintained constant for the subsequent ILSS enhancement on these phenolic laminates.

TABLE IV
Effect of Resin Content on the Mechanical Properties of Fortafil-3-Varinit-Reinforced Durez A Laminates

Properties	Unit	E(1)	E(2)	E(3)	E(4)	E(5)	E(6)
Sizing of fiber	—	Phenolic	Phenolic	Phenolic	Phenolic	Phenolic	Phenolic
Orientation of fiber	—	Unidirectional					
Tensile strength	psi	66,000 ± 5000	73,000 ± 3000	88,000 ± 8000	91,000 ± 7000	94,000 ± 14,000	98,000 ± 14,000
Flexural strength	psi	100,000 ± 13,000	100,000 ± 16,000	130,000 ± 16,000	130,000 ± 17,000	91,000 ± 41,000	95,000 ± 66,000
Flexural modulus	psi × 10^6	6.86 ± 0.58	7.68 ± 0.78	9.25 ± 1.44	9.72 ± 0.27	9.98 ± 1.09	10.8 ± 1.6
Interlaminar shear strength	psi	5800 ± 600	3700 ± 300	4000 ± 1100	4700 ± 1000	3700 ± 500	3400 ± 1300
Wt % of resin	%	50	43.6	40.2	37.5	33.1	30

TABLE V
Effect of Molding Pressure on the Mechanical Properties of Fortafil-3-Varinit-Reinforced Durez A Laminates

Properties	Unit	P(1)	P(2)
Tensile strength	psi	60,000 ± 10,000	70,000 ± 9000
Flexural strength	psi	95,000 ± 900	86,000 ± 7000
Flexural modulus	psi (10^6)	6.81 ± 0.41	6.20 ± 0.16
Interlaminar shear strength	psi	3700 ± 300	5100 ± 700
Wt % of resin	%	≃50	
Pressure of molding	psi	295	780

The ILSS Problem: One of the common and effective ways of enhancing the bonding efficiency between the resin and carbon fiber involves the surface treatment of the fiber. Methods of whiskering,[8–10] heat cleaning,[11,12] air oxidation,[13–15] nitric acid oxidation,[16] treatment in sodium hypochlorite,[17] irradiation,[18] and coatings of various kinds[19] have been reported. Since the carbon fiber and fabrics used in this work are all commerical products with prior surface treatment by the supplier, the problem of ILSS is pursued only through optimization of the following processing variables.

C. Effect of Molding Pressure: In an effort to understand the effect of molding pressure on the flow and wetting efficiency (hence ILSS) of the resin, laminates of similar composition were compression-molded on the Carver press at two different pressures. Table V compares the mechanical properties of a 50 wt % resin formulations molded under 285 and 780 psi pressure. It is evident that increase in molding pressure produced better and more uniform wetting and adhesion between the fiber and resin, which is reflected in the increase of ILSS [comparing Sample P(2) to P(1)], with no adverse effect on other mechanical properties.

D. Effect of the Stacking Sequence: Since the hybrid material consists of continuous strands of carbon fiber adhered onto a glass cloth tape, the interlaminar shear strength would be expected to be dependent upon the layup sequence of the laminate because of the different interfacial strengths among carbon–resin–carbon, carbon–resin–glass, and glass–resin–glass. Table VI shows the mechanical properties of CFR phenolic laminates stacked up in two different configurations. In both cases of either 33 or 50 wt % resin content, the alternate carbon/glass layer configuration shows significantly higher ILSS as compared to the carbon–carbon/glass–glass configuration. In an attempt to understand the governing mechanism for such a difference in the observed ILSS values, fractured surfaces of Samples #70A (ILSS = 3700 psi with carbon–carbon/glass–glass/carbon–carbon geometry) and #70B (ILSS = 5700 psi with the carbon/glass/carbon/glass geometry) were examined by scanning electron microscope. Figures 1(a)–(d) show their fracture morphology together with the Si (from glass) mapping, which indicated that delamination is the mode of failure, and this occurred preferentially at the glass–resin–glass interfaces in the #70A sample. It is inferred, therefore, that the glass–resin–glass interfacial shear strength is weaker than those of the carbon–resin–glass or carbon–resin–carbon interfaces. Such weaker glass–resin–glass interfacial shear strength contributed most probably to the observed lower ILSS of samples #70A.

TABLE VI
Effect of Stacking Sequence of the Mechanical Properties of Fortafil-3-Varinit-Reinforced Durez A Laminates

Properties	Unit	E(1)	P(1)	#70A	#70B
Tensile strength	psi	66,000 ± 5000	60,000 ± 10,000	94,000 ± 14,000	93,000 ± 6000
Flexural strength	psi	100,000 ± 13,000	95,000 ± 1000	91,000 ± 41,000	161,000 ± 11,000
Flexural modulus	psi (10^6)	6.86 ± 0.58	6.81 ± 0.41	9.98 ± 1.09	10.4 ± 0.61
Interlaminar shear strength	psi	5800 ± 600	3700 ± 300	3700 ± 500	5700 ± 500
Wt % of resin	%	50		33	
Pressure of molding	psi	285	285	780	780
Stacking geometry[a]		C/G/C—G/C	G/CC/GG—CC/G	G/CC/GG—CC/G	C/G/C—G/C

[a] C/G/C/G: carbon fiber and the glass fabric stacked up in alternate layers; CC/GG/CC: pair of carbon fibers and pair of glass fabric stacked up in alternate pairs.

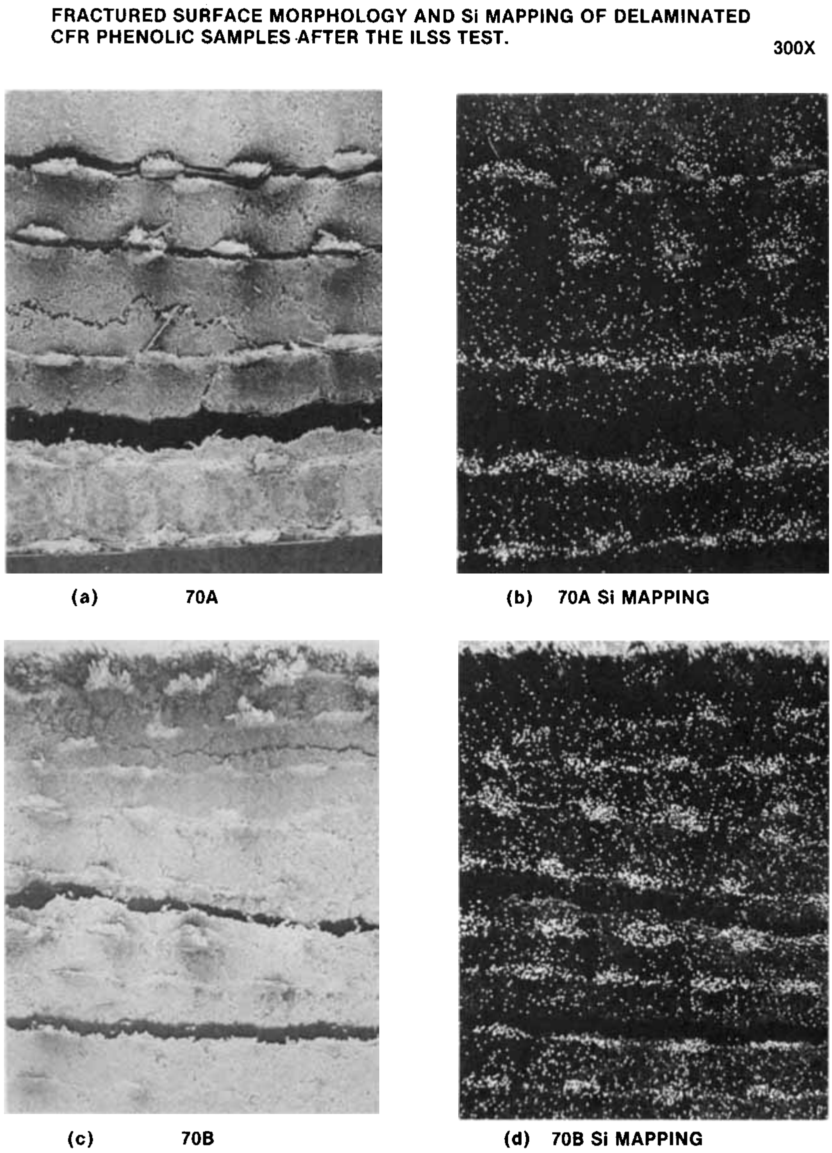


Fig. 1. Comparison of fracture morphology of CFR phenolic laminates with different stacking geometry.

Comparison of Mechanical Properties of Laboratory Optimized CPR–Phenolic Laminates vs. Laboratory and Commercial Analogs

Mechanical Properties

Based on the above studies on the effects of resin content, molding pressures, and stacking sequence on the mechanical properties, optimized samples were prepared by using (1) the Hercules' 370-8H carbon fabrics for the 0–90°C bidirectional laminates and (2) Fortafil-3 Varinit hybrid material for the unidirectional laminates. Table VII compares the mechanical properties of our opti-

TABLE VII

Comparison of Mechanical Properties of Bidirectional Carbon-Fabric-Reinforced Phenolic Laminates vs. Epoxy Analogs

Properties	Unit	Experimental CFR phenolic (1)	Experimental CFR epoxy (1)	Hercules' A3708H/3501-5A
Type of resin	—	Durez A amine-catalyzed resole	Epon 828/DTA	Amine-cured general purpose epxoy (undisclosed)
Resin content	wt %	36	30	30
Carbon fabric type	—		Hercules A370-8H	
Orientatioin of fabric	—	0–90 bidirectional	0–90 bidirectional	0–90 bidirectional
Tensile strength	psi	85,000 ± 7000	86,000 ± 4000	85000[a]
Elongation at break	%	0.9 ± 0.1	N.A.	0.75[a]
Tensile modulus	psi × 10^6	12.0 ± 0.6	N.A.	10.0[a]
Flexural strength	psi	90,000 ± 3000	103,000 ± 7000	108,000 ± 5000[b]
Flexural modulus	psi × 10^6	6.2 ± 0.4	7.62 ± 1.53	6.4 ± 0.26[b]
Interlaminar shear strength	psi	6500 ± 200	6600 ± 100	7600 ± 300[b]
Compressive strength	psi	51,000 ± 4000	39,000 ± 6000	55,000[b]

[a] Values quoted in Hercules' Product Data Bulletin No. 837-1, 1-78.

[b] Values tested in our laboratory.

TABLE VIII
Comparison of Mechanical Properties of Unidirectional Carbon-Fiber-Reinforced Phenolic Laminates vs. Some Epoxy Analogs

Properties	Unit	Experimental CFR phenolic (2)	Experimental CFR epoxy (2)	Experimental CFR epoxy (3)
Type of resin	—	Durez A amine-catalyzed resole	Epon 828/DTA	Araldite 702/HY906
Resin content	wt %	35	35	35
Carbon fabric type		Fortafil-3 Varinit	Fortafil-3 Varinit	Fortafil-3 Varinit
Orientation of fabric	—	Unidirectional	Unidirectional	Undirectional
Tensile strength	psi	93,000 ± 5000	88,000 ± 4000	103,000 ± 4000
Elongation at break	%	0.7 ± 0.4	0.6 ± 0.2	0.9 ± 0.1
Tensile modulus	psi × 10^6	17.9 ± 0.7	13.0 ± 0.1	14.6 ± 0.2
Flexural strength	psi	120,000 ± 3000	130,000 ± 2000	140,000 ± 3000
Flexural modulus	psi × 10^6	8.5 ± 0.2	7.1 ± 0.9	9.9 ± 0.2
Interlaminar shear strength	psi	6400 ± 1700	7900 ± 800	7100 ± 500

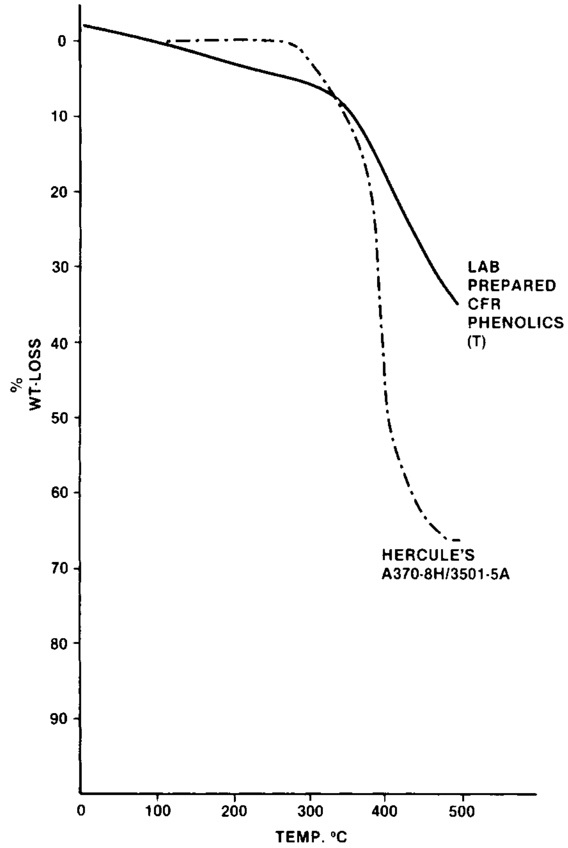


Fig. 2. Comparison of thermal stabilty between a laboratory-prepared CFR phenolic vs. a commercial epoxy analog.

mized, fabric-reinforced, amine-catalyzed resole (Durez A) laminate vs. a laboratory-prepared epoxy (Epon 828/DTA) analog and a commerical epoxy system. It is apparent that our laboratory-prepared phenolic laminates, in spite of the slightly higher resin content (36 vs. 30 wt %), which would be predicted to yield lower moduli according to the rule of mixtures, shows equivalent all-round mechanical properties with either our laboratory-prepared or the Hercule's epoxy analog. In the case of the unidirectional carbon fiber/glass cloth hybrid reinforced laminates (Table VIII), the phenolic laminate again compares favorably in overall mechanical properties with the two epoxy analogs.

Thermal Stability

Figure 2 compares the thermal stability of our experimentally optimized carbon-fabric-reinforced phenolic and epoxy vs. the Hercules A 370-8H/3501-5A sample by using thermogravimetric analysis (TGA). In confirmation with what was observed previously in Table II for the 50 wt % carbon-fabric-reinforced systems, the experimental CFR phenolic again show distinctively better thermal stability at higher temperatures (350–500°C) than the experimental and the commercial epoxy systems.

SUMMARY AND CONCLUSIONS

It has been demonstrated in this work that CFR phenolic laminates with equivalent mechanical properties and much superior thermal stability to laboratory-prepared and present commercial epoxy analogs can be obtained by reinforcing 35–50 wt % of an amine-catalyzed resole with either the Hercule's Magnamite carbon fabric or the Great Lakes' Varinit carbon fiber/glass cloth hybrid. In the different methods of improving the ILSS, it has been demonstrated that increase in molding pressure and alternate carbon/glass/carbon/glass stacking sequence improves significantly the ILSS.

The effect of sizing on the pre-surface-treated carbon fibers is found not important in improving the bonding efficiency or the ILSS of the present phenolic resin system.

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