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Low-temperature and freeze–thaw durability of thick composites

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Low temperature produces internal stresses in composites of polymeric materials. The polymeric matrix phase becomes stiffer, and may suffer from damage-inducing stresses resulting from thermal coefficient mismatch of fibers and resins. These influences have been studied by subjecting two types of FRP composites to flexural tests. A commercially procured fiber reinforced plastic (FRP) composite indeed produced cracks on prolonged thermal cycling between 50°C (122°F) and −60°C (−76°F) temperature. But a specially manufactured woven glass reinforced FRP did not produce any visual cracks for two and half times more thermal cycling in the same temperature range. It is suspected that the resin type and the curing process control the thermal cycle response and ultimate durability of such FRP composites in extreme temperature environments.

(Keywords: composites; polymer-composites; fiber-reinforced-plastics; low-temperature; freeze–thaw; thermal-cycling; durability; cold-regions)

INTRODUCTION

Structures built with thick and mass-produced composites of fiber reinforced plastics (FRP) are increasingly being considered in regions of the world where the temperatures during winter plummet well below the freezing temperature. Lightweight structures are specially attractive in cold regions, where construction cost penalties increase tremendously with remoteness of the locations and heaviness of the construction materials and equipment. Transportation systems, including cargo containers, boat hulls, waterfront and offshore structures, and many infrastructure components of cold regions, in general, are poised to derive great benefits from this new, lightweight and durable class of engineered material, the FRP composites. In military applications the use of composites is becoming relatively widespread in a variety of ordnance, armor, lightweight bridging, naval, and aerospace systems. However, important questions are still being asked about the influence of severe environmental exposure on the performance and durability of these materials[1–6]. In the treatment of mechanics of materials, composites of polymers are a fairly complex group of materials. Appropriate models to describe their behavior and performance under multiaxial loading and environmental exposure are yet to be developed. External loading induces multiaxial stresses into the individual plies of multi-directional ply laminates, even when the overall loading condition is unidirectional. Superimposed on these stresses would be thermal effects in terms of change in stiffness of the composite's components and the induced thermal stresses resulting from their mismatch of thermal expansion coefficients. Additional complexities involve absorption of water and subsequent influence of freeze–thaw cycling on the changes of the material behavior through microstructural modifications. Thus, the associated fundamental response behaviors of these composite structures would be functions of external loading conditions, thermal and hygrothermal environment, structural geometry, and/or material characteristics. The parameters that effect the response of composites include fiber/matrix interfacial properties, volume ratios, fiber orientations, their moduli and Poisson's ratios, associated load transfer mechanisms, and the fabrication and processing history. Thus,

for both the designers and the analysts of composite structures it is necessary to establish how the geometric and the material variables would influence the composite's structural behavior under the various thermal regimes.

This study discusses the test results of one E-glass reinforced commercially available thick FRP composite and another S2-glass reinforced thick FRP composite specially manufactured for the US Army. Both of these composites were subjected to flexural tests at low temperatures and to low temperature thermal cycling. The commercial composite is available as bars of rectangular cross-section. From the manufacturer's literature it was clear that the material was produced by a pultrusion process using E-glass reinforcement in isophthalic polyester matrix. These kinds of pultruded composite products are commonly being aimed at the building and construction industries, and include uses in walkways, platforms, gratings, EMC (electromagnetic compatible) framing structures, and railroad shipping modules[7]. As stated before, the effects of very low ambient temperatures, and long-term exposure under cyclic extreme temperatures (thermo-oxidative and freeze–thaw exposure), on composite properties and durability are of vital concern to the designers of composite structures. In this study results of the flexural tests in terms of stiffness parameters such as Young's modulus (E) and shear modulus (G) were obtained at 27, −5, −40 and −60°C (81, 23, −40 and −76°F), with the temperature tolerance being maintained within ±1.5°C. The tested materials were then subjected to low temperature thermal cycles between 50°C (122°F) and −60°C (−76°F) temperature excursions with two hours duration at a time at each temperature. Following thermal cycling these materials were again due to be tested for determining the Young's modulus (E) and shear modulus (G) values to see if the stiffness is truly degraded. However, after 100 thermal cycles the commercial FRP materials developed cracks in the central part of the cross-section. This phenomenon will be discussed in detail later.

RESIDUAL STRESSES FROM CURING TEMPERATURE

Manufacturing of FRP composites involves the use of strong inorganic (glass or carbon) or organic (Kevlar) fibers as the reinforcing phase and a thermosetting polymeric material (polyester) as the matrix phase. The polymeric matrix, in the presence of a catalyst, heat and pressure, solidifies through an irreversible exothermic chemical reaction (cure). Before curing, the polymer phase is a viscous fluid. It flows under pressure. As a result of curing, the polymer forms a covalently bonded three-dimensional molecular network with increasing viscosity and gel formation. The flow ceases at this stage, but reactions continue to form a tightly cross-linked structure with characteristics of a glassy solid[8]. In thin section composites the heat distribution is approximately uniform from the interior of the section to the surface. However, in relatively thick section composites, as in many pultruded FRP composites, the heat distribution may not be uniform at the time of curing. Bogetti *et al.*[9] have discussed the problem in a recent article on the manufacturing problem of thick composites for the US Army's composite infantry fighting vehicle. The problem arises from the difficulties in controlling the reaction exotherm. During the curing stage, as the chemical reactions proceed, residual stresses are developed with progressive changes in modulus and thermal expansion coefficients, and volume shrinkage of the resin. If the processing temperature is not well-controlled in the heat curing environment, because of the exotherm, the exterior regions can cure first, while the interior regions are still un-cured and relatively more viscous. Thus, the continuing curing in a high heat environment can induce significant residual stresses: transverse tensile stresses in the interior, and transverse compressive stresses in the exterior (*Figure 1*). In very low temperature environment, continuing shrinkage of the exterior past the ambient room temperature to lower temperatures will therefore cause more severe stresses.

At the microstructural level, the influence of low temperature on the induced stresses at the matrix/fiber interfaces, within the matrix and in the interlaminar layers, has been analyzed and experimentally investigated by many investigators[4,10–12]. We can use a mechanics-of-materials approach to look at the microstructural elastic response of the unidirectional composite due to thermal expansion (shrinking) and stresses. Assuming uniform strain in the longitudinal direction and uniform stress in the transverse direction, Hahn[13] has shown that the matrix longitudinal stresses are given by:

$$\sigma_{mL} = (V_f E_f E_m)(\alpha_f - \alpha_m)(T - T_0)/(V_f E_f + V_m E_m) \quad (1)$$

where E is the elastic modulus, V is the volume ratio, α is the coefficient of thermal expansion, T is the temperature, σ_L is the longitudinal stress, and subscripts m and f refer to matrix and fiber, respectively. The quantity T_0 is

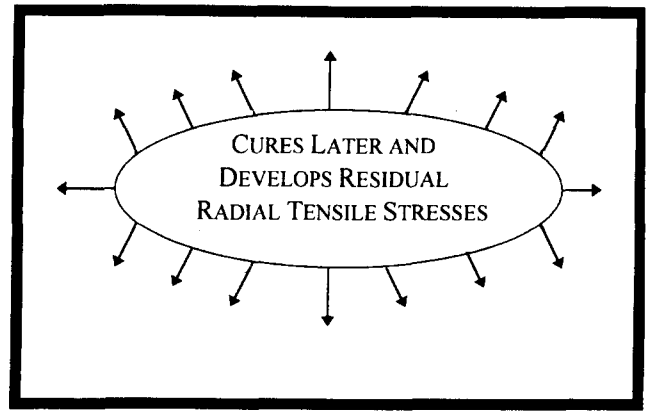

Figure 1 Development of internal residual stresses from non-uniform cooling and curing of FRP composites

the 'stress-free' temperature, usually taken as the cure temperature of the composite. For a quick estimate of the average residual tensile stress, consider the volume fraction of the order of 0.5, stiffness ratio 10 : 1, and the fiber's coefficient of thermal expansion almost negligible in comparison to that of the matrix resin. Equation (1) then reduces to:

$$\sigma_{mL} = -E_m \alpha_m \Delta T \qquad (2)$$

For our experimental FRP test specimens we can reasonably estimate the values of $E_m = 3.45\,\text{GPa}$ (500×10^3 psi), $\alpha_m = 7.2\,\text{cm/cm/°C}$ (4×10^{-5} in/in/°F), and $\Delta T = -184°\text{C}$ (−300°F). These values predict that at room temperature an axial tensile residual stress of 41 MPa (6000 psi) would be expected to develop if the cure temperature is about 190°C (370°F). This value is equal to about 50% of the matrix tensile strength. In our tests the specimens were cooled down to −60°C (−76°F), and the expected matrix tensile stress would be of the order of 62 MPa (8920 psi). As we will see later on, this amount of stress could have very well initiated the internal microcracks in the commercial FRP. *Figure 2(a)* and *(b)*, reproduced from Dutta[12], show simple analytical results from the Tsai and Hahn method of computing residual stresses in unidirectional composites in the longitudinal direction. Thus, it is obvious that the large residual stresses induced at lower temperatures become potentially damaging for polymer matrix composites with curing temperature environment. The damage may begin with the formation of microscopic cracks in the matrix or at the fiber/matrix interface. When these cracks develop to a certain density and size, they will tend to coalesce to form macroscopic matrix cracks[14]. Transverse matrix cracking in composites affects stiffness, strength, dimensional stability, and fatigue resistance. Of particular interest in this paper is the case of prolonged low temperature thermal cycling where material damage can grow and accumulate to result in composite material degradation.

LOW TEMPERATURE FLEXURE TEST

The ASTM D790 three-point bending test is one of the simplest methods for evaluation of strength and Young's modulus of composites. However, simple considerations of mechanics of three-point bending show that the thick-section-composites exhibit significantly high transverse shear and transverse normal deformations—the type of three-dimensional stress contributions that are negligibly small in thin laminates ($L/d \geq 100$). Therefore, for thick composites evaluation alternative methods of testing have always remained attractive and the ASTM Short Beam Method (D 2344) has become most widely used. Fischer *et al.*[15] proposed a method of simultaneous determination of transverse shear and bending moduli of composites by measuring P/δ for two different values of L/d obtained from two different sizes of test specimen. This method is based on the bending and shear strain

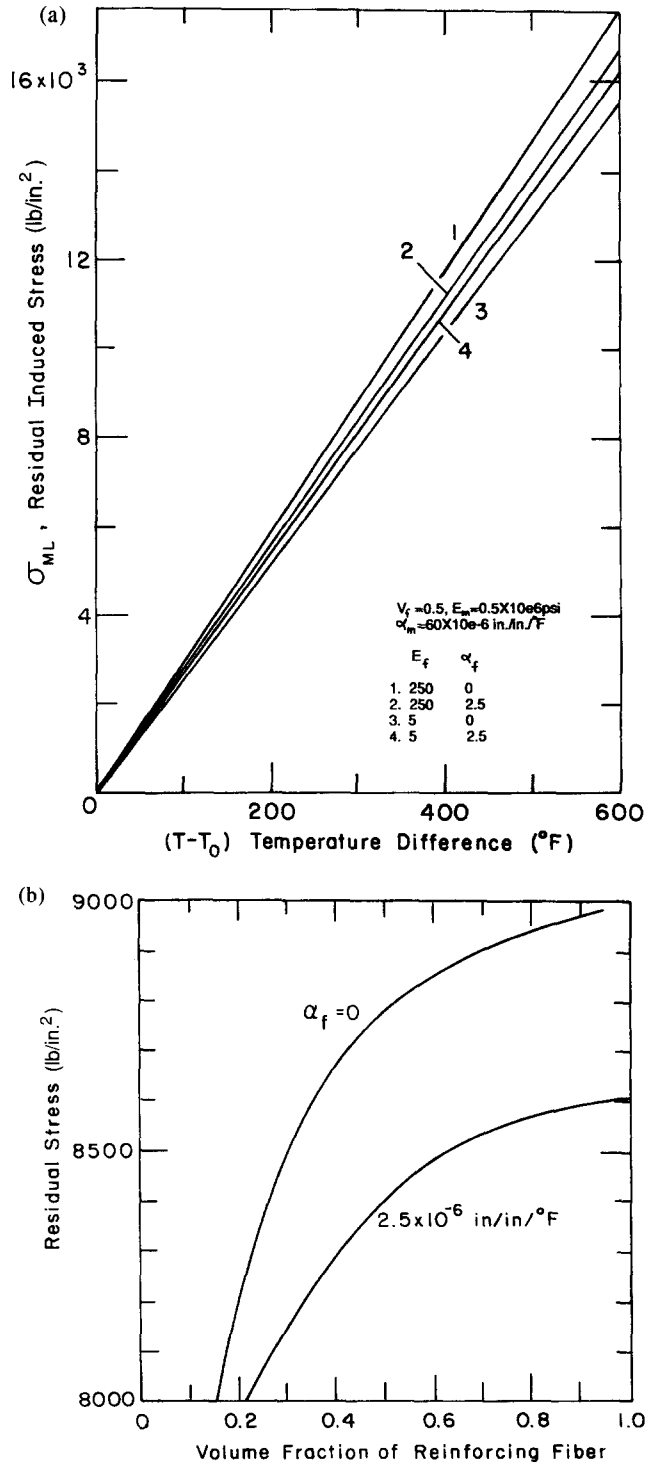

Figure 2 (a) Effect of temperature difference on longitudinal tensile stress in the matrix of unidirectional FRP composite. (b) Residual stresses as a function of the fiber volume fraction in unidirectional FRP composites

energy analysis given by Ugural and Fenster[16]. Since we have also followed this analytical approach for developing our experimental method this approach will be discussed in more detail in Appendix A.

Materials

As mentioned before, the S2-glass fabric and polyester resin composite was specially fabricated for the Army

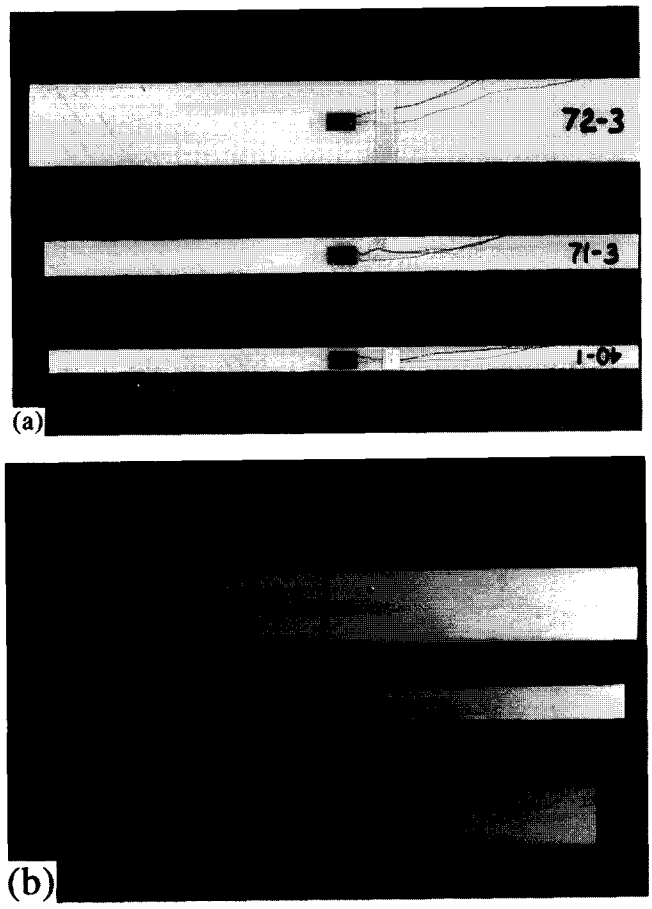

Figure 3 (a) Specially manufactured S2-glass woven fabric reinforced composites. (b) Commercially available E-glass reinforced polyester resin FRP composites

with great quality control, and the materials were obtained from the US Army Materials Technology Laboratory, Watertown, Massachusetts[17]. The specimens are shown in *Figure 3(a)*. Details of processing these thick-section composites involved innovative silicone rubber vacuum bag technique, and are described by Thomas[18]. In all, 27 specimens were prepared in three batches of three cross-sectional sizes, as shown in *Table 1*.

The second material [*Figure 3(b)*] is a commercial pultruded composite product which uses E-glass and isophthalic polyester resin. Pultrusion is a continuous manufacturing process which involves pulling (thus the name 'pultrusion') the flexible reinforcing fibers through a bath of other liquid raw materials (base resin, various additives, catalysts, etc.) and next through a heated die where the final product is shaped and cured. Liskey[7] has described in detail the general processes involved in manufacturing the pultruded composite products. In all, 18 specimens were prepared from the pultruded bars. Details of both materials are given in *Table 1*.

Tests

The flexural tests were performed in three-point bending conditions, using the test apparatus shown in *Figure 4*. The apparatus is housed in an environment

Figure 4 Three-point flexural test set-up for thick FRP composites

chamber. The load was applied using a 267 kN (60 kips) servo-hydraulic universal testing machine. The span between the supports was 254 mm (10 in). The supports were provided by the loading pins of diameter 25.4 mm (1 in). The load was applied at a cross-head speed of 2.54 mm/s (0.1 in/s) for the 44.5 mm (1.75 in) and 38.1 mm (1.50 in) samples, and at 5.08 mm/s (0.2 in/s) for thinner, i.e. 12.7 mm (0.50 in) and 19.1 mm (0.75 in), samples. During the tests the load, displacement, and deflections were recorded at 5 ms intervals by a Nicolet 4094A Digital Oscilloscope. Some specimens were instrumented with strain gauges. The strain was recorded on the fourth channel of the Nicolet Oscilloscope. Load and displacements were recorded directly as voltage outputs from the testing machine, but deflection was read through an auxiliary cantilevered extensometer. This consists of a thin steel strip, 228 mm (9 in) long, with a small rounded probe at the free end which contacts the

Table 1 Test materials and specifications

S2-glass FRP composite	E-glass FRP composite
Process: Vacuum bag	Process: Pultrusion
Fiber: S2-glass®	Fiber: E-glass
Matrix: Polyester	Matrix: Polyester
Density: 1.890 (g/cm^3) (0.068 lb/in^3)	Density: 1.971 (g/cm^3) (0.071 lb/in^3)
Volume fraction of fiber: 0.532	Volume fraction of fiber: 0.593
Number of specimens: 27	Number of specimens: 18
Specimens per batch: 9	Specimens per batch: 9
Dimensions:	Dimensions:
Length: 330.2 mm (13.0 in)	Length: 330.2 mm (13.0 in)
Section: Square	Section: Square
Width and thickness:	Width and thickness:
Batch A1: 12.7 mm (0.50 in)	Batch A2: 12.7 mm (0.50 in)
Batch B1: 19.1 mm (0.75 in)	Batch B1: None
Batch C1: 44.5 mm (1.75 in)	Batch C2: 38.1 mm (1.50 in)
Conditions of test:	Conditions of test:
Span: 254 mm (10 in)	Span: 254 mm (10 in)
Test temperatures (nominal)	Test temperatures (nominal):
27°C (81°F)	27°C (81°F)
−5°C (23°F)	−5°C (23°F)
−40°C (−40°F)	−40°C (−40°F)
−60°C (−76°F)	−60°C (−76°F)
Thermally cycled 250 times between +50°C (122°F) and −60°C (−76°F) temperatures	Thermally cycled 100 times between +50°C (122°F) and −60°C (−76°F) temperatures

Figure 5 Commercially available FRPs cracked after one hundred thermal cycles

bottom center of the specimen, and two strain gauges at the fixed end. The strain gauges were calibrated to read for deflection of the probe tip.

For low temperature testing, the interior of the environmental chamber was cooled by evaporated liquid nitrogen gas. The chamber has several round ports for in-flow and out-flow of cooling gases and instrumentation, as well as a hinged door. Instrumentation is wired through the side of the box, in addition to a light

Table 2 Effect of temperature on moduli

Thermal condition [°C (F°)]	Young's modulus (E_{11}) [GPa (psi × 10^6)]	Shear modulus (G_{12}) [GPa (psi × 10^6)]
S2-glass composite		
27 (81)	27.72 (4.02)	1.10 (0.16)
−40 (−40)	28.28 (4.10)	1.17 (0.17)
−60 (−76)	28.63 (4.15)	1.31 (0.19)
Th.*	26.00 (3.77)	1.03 (0.15)
E-glass composite		
27 (81)	51.66 (7.49)	1.45 (0.21)
−5 (23)	53.51 (7.76)	2.07 (0.30)
−40 (−40)	53.66 (7.78)	2.27 (0.33)
−60 (−76)	57.93 (8.40)	2.55 (0.37)

*Thermally cycled 250 times between +50°C (122°F) and −60°C (−76°F) and then tested at 27°C (81°F).

and a fan to uniformly distribute the cooling nitrogen gases. The cooled nitrogen gas flow was varied by a thermostatically controlled solenoid valve, which in turn was controlled by a thermocouple located near the sample inside the chamber.

Thermal cycling

After the flexure tests, both types of specimen were subjected to low temperature thermal cycling in a

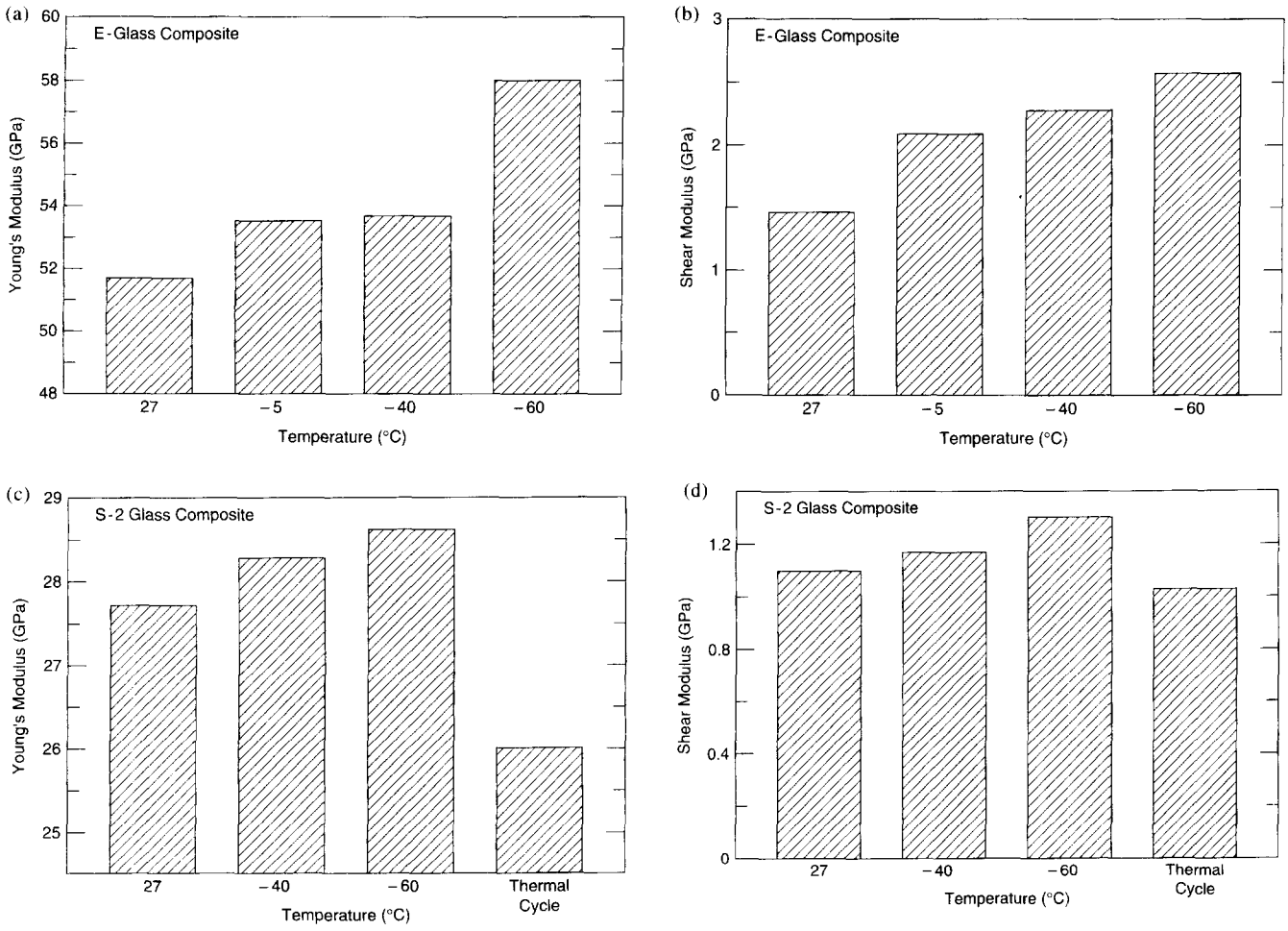

Figure 6 Effects of low temperature on (a) the elastic modulus E of the E-glass composite; (b) the shear modulus G of the E-glass composite; (c) the elastic modulus E of the S2-glass composite; and (d) the shear modulus G of the S2-glass composite

computer controlled thermal cycling chamber. The chamber was connected to a liquid nitrogen supply line. The chamber was programmed by the computer for alternatively heating and cooling between +50°C (122°F) and −60°C (−76°F) temperatures. A period of two hours was allowed to elapse before switching on the chamber from one temperature to the other. A number of thermocouples mounted on the specimens were used to monitor the actual specimen temperatures. It took about 15–20 minutes for the temperature to rise and fall and then stabilize to within 1.5°C. The S2 FRP composites were thermally cycled 250 times. No visible signs of degradation were noted on these samples. So the flexure tests were continued on these specimens. On the other hand, all the commercially available E-glass FRP specimens developed cracks in the interior (*Figure 5*) after only 100 thermal cycles. Since these samples degraded so badly, no further flexural tests were performed with these specimens.

RESULTS AND DISCUSSION

The results of the three-point bending test for both S2-glass composite and E-glass composite have been summarized in *Table 2*. One will note that both the E values and G values increased with reduction of temperature. This is clear in *Figure 6(a)* and *(b)* for E-glass composites, and in *Figure 6(c)* and *(d)* for S2-glass composites. For S2-glass composites, the E value increased approximately at a rate of 10.5 MPa/°C (846 psi/°F), and G at 2.41 MPa/°C (194 psi/°F). The increase of E for E-glass composite is more dramatic, 72.1 MPa/°C (5806 psi/°F). The shear modulus G increased at 12.64 MPa/°C (1019 psi/°F). *Figure 7(a)* and *(b)* graphically represent those data. Using experimental observations, Dutta[19] has shown earlier that for unidirectional composites under flexural loading the major area of the beam will be in compression, therefore it is expected that the increase of E values in both composites primarily is as a result of increase of the E value of the matrix at low temperatures. Quoting Hartwig[20], it has been shown by Dutta[21] that for most resins the Young's modulus increase is of the order of 20 MPa/°C (1611 psi/°F), thus while the E-glass composite's E value increase at the rate of 72.1 MPa/°C (5806 psi/°F) appears to be too high, the S2-glass® composite's value at 10.5 MPa/°C (846 psi/°F) appears to be more reasonable. Referring to *Figure 6(a)* it will be obvious that the E value for the E-glass composite increased between −40°C (−40°F) and −60°C (−76°F) more significantly than between 27°C (81°F) and −40°C (−40°F), and the former increase has possibly dramatically influenced the result. It is possible that the isophthalic polyester resin used in the E-glass composite stiffens up at a higher rate at lower temperatures than the polyester used for the S2-glass FRP. But if so, this must be reflected in the shear modulus data too. In fact, the increase of shear modulus for E-glass

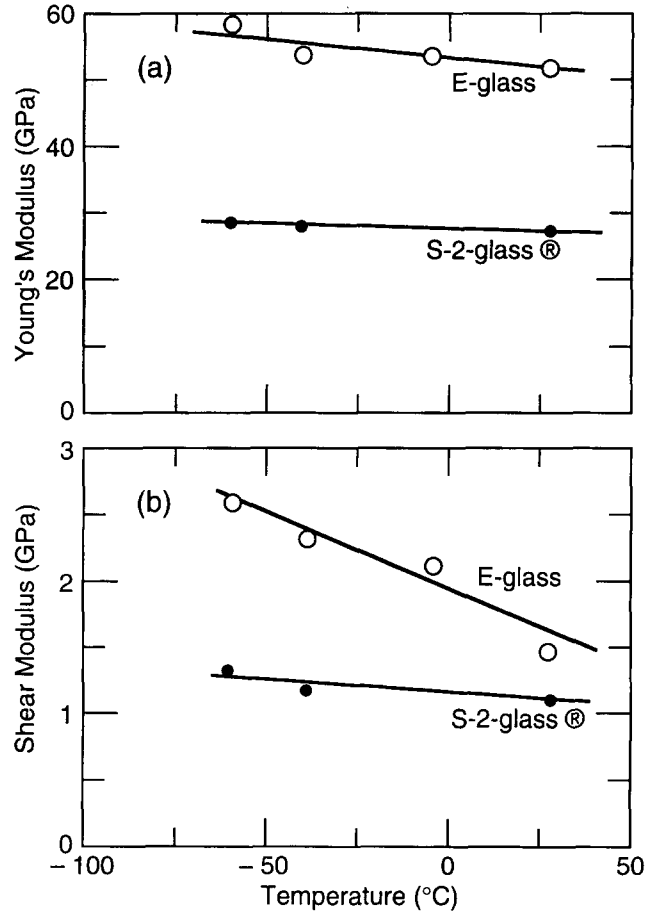

Figure 7 Comparison of effects of temperature on (a) the elastic modulus E for the two composites; and (b) the shear modulus G for the two composites

composites after cooling is also dramatic, almost 1.75 times. However, because the E-glass composite has all straight glass fibers, as opposed to woven glass fibers in the S2-glass composite, a higher shear modulus value and higher rate of increase of the shear modulus value are expected.

The low temperature thermal cycling has a significant influence on the degradation of both the E and G values of the composite. Only S2-glass® composite was thermally cycled 250 times between +50°C (122°F) and −60°C (−76°F) and then tested at 27°C (81°F). The Young's modulus degraded 6.2% and shear modulus degraded 6.3%. The nature of degradation is not obvious from the results. This composite being reinforced with plain-weave glass, it is expected that degradation is primarily the result of matrix degradation.

The thermal cycling results of the commercially available FRP are, in fact, greatly disappointing. As discussed before, under the residual stresses from curing, developing of significant internal stresses is suspected. The ultimate tensile strength of polymer resins is expected to be about 10,000–12,000 psi; and it has been shown that the excursion to −60°C (−76°F) could easily push the matrix to its tensile strength limit, producing microscopic cracks at the beginning. It is also possible that a non-uniform curing across the thickness has

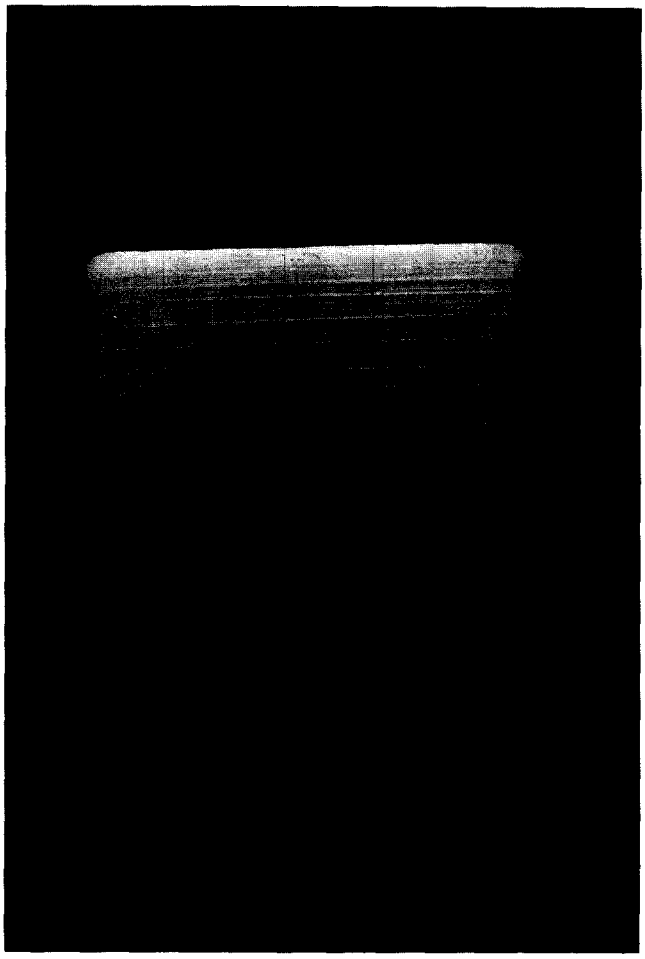

Figure 8 A close-up view of the crack in E-glass FRP composite from thermal cycling

contributed additional stresses in the center regions of these composites. With repeated thermal cycling these cracks probably coalesced and produced larger cracks. A close-up view of the crack in the commercial grade FRP composite following the low temperature thermal cycling is shown in *Figure 8*. The cracks seem to have passed only through the matrix and are radial in direction, with maximum width of the crack occuring at the center of the cross-section. Although the limits of temperatures to which these materials were subjected to cycling were too extreme, and in real life such extreme variation of temperatures will be encountered only rarely, it is however important to resolve the technical issues surrounding the mechanisms of such crack development. An understanding of the process will elevate the confidence limit on the durability of commercial FRPs in extreme environmental situations.

CONCLUSIONS

In general, at low temperature the FRP composites will stiffen up. The performance of its matrix dominated behavior will improve. Flexural behavior of the composites is overwhelmingly a matrix dominated behavior, and the increase of E and G values at lower temperatures control the composite's flexural properties. The type of polymer matrix itself, and its mechanical properties at low temperature (stiffness or brittle behavior) would significantly influence the composite's behavior at sub-zero temperatures. It is obvious that not all polyester resins behave the same way. The polyester resin matrix of E-glass composites appears to have a higher rate of modulus increase than the polyester matrix composite used with S2-glass. Low temperature thermal cycling has shown degradation of both the Young's modulus and shear modulus of the plain-weave glass composites. This is expected, however, the data available is too meager to develop any predictive methodology of this degradation prediction based on low temperature thermal cycling. The results of thermal cycling for the E-glass composites are disappointing. The crack growth raises important questions about its applicability in severely cold climatic regions.

Remarks

Mention of any commercial or trade name in this article does not mean any endorsement of the products by the authors. The trade names have been used purely for identification purposes.

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APPENDIX A

Procedure for simultaneous determination of Young's modulus and shear modulus by flexural test:

NOMENCLATURE

A	cross sectional area
d	depth of specimen
E	Young's modulus of elasticity
f_s	form factor for shear
G	shear modulus of elasticity
I	moment of inertia
L	length of span
M	bending moment
P	applied load
U	strain energy in bending
V	shear force
w	width of specimen
δ	deflection

Subscripts

b	bending
s	shear

The mathematical model for this analysis assumes that the material of the beam under three-point bending is isotropic, homogeneous, linear elastic, of negligible weight, and local indentation effects at the supports are ignored. The total energy U_T at any point x from the support end in bending the beam consists of the strain energy in bending U_B and the strain energy in shear U_S.

From Ugural and Fenster[16]:

$$U_B = \int [(M^2\,dx)/(2EI)]\,dx \tag{A1}$$

and

$$U_S = \int [(f_s V^2)/(2GA)]\,dx \tag{A2}$$

The total energy $U_T = U_B + U_S$.

Therefore,

$$U_T = \int [(M^2\,dx)/(2EI)]\,dx + \int [(f_s V^2)/(2GA)]\,dx \tag{A3}$$

For applied load P at the mid-point of the span the shear force $V = (P/2)$ and moment $M = (Px/4)$, which after substituting in equation (3) and integrating give

$$U_T = [(1/48)(P^2L^3/(2EI)] + [(f_s/2)(P^2L)/(4GA)] \tag{A4}$$

Using Castigliano's theorem, which gives deflection, $\delta = \partial U/\partial V$, we have by differentiation of equation (4):

$$\delta = [(PL^3)/(48EI)] + [(f_s/4)(PL)/(GA)] \tag{A5}$$

Thus, in three-point bending test the slope of the load–deflection curve must be:

$$\delta/P = [(L^3)/(48EI)] + [(f_s/4)(L)/(GA)] \tag{A6}$$

If m = slope of the load–deflection curve $(P - \delta)$, so that

$m = P/\delta$, then equation (6) can be rewritten as

$$m = [(c/E) + (d/G)]^{-1} \tag{A7}$$

where $c = L^3/(48I)$, and $d = (f_s L/4A)$.

By measuring the slope, m, at two span-to-depth ratios, the moduli can be calculated using the above simultaneous equation (7). The resulting equations for the moduli obtained from two sizes (1 and 2) of the specimens are:

$$E = (m_1 m_2)(c_1 d_2 - c_2 d_1)(m_2 d_2 - m_1 d_1)^{-1} \tag{A8}$$

and

$$G = (m_1 m_2)(c_2 d_1 - c_1 d_2)(m_2 c_2 - m_1 c_1)^{-1} \tag{A9}$$

The experiment was designed to vary the span-to-depth ratio by varying the specimen thickness while maintaining constant span L. The ASTM D790 was used as a guide line for the test conditions[22] using the three-point loading configuration. The specimens were cut to have the same width as the thickness.