Fracture Mechanics Evaluation of Filament Wound Case Materials Subjected to Operational Environments

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The fracture characteristics of several filament wound composite materials were evaluated over operational conditions typical of solid rocket motor composite cases. The fracture toughness (K_Q) for Mode I crack growth was determined for three composite systems, a Kevlar-49/AS4 graphite laminate and two AS4 graphite laminates of different orientation layup. Double-edge notch tensile coupons were used to generate K_Q data at several crack sizes at room temperature to determine that value of K_Q which was independent of crack length. The fracture toughness and net stress were found to depend on crack length over a fairly large range. These results were in good agreement with other work reported on flat plate, autoclaved laminate systems. The effects of elevated temperatures (150° to 200°F) were compared to the ambient (77°F) results for single cycle loading and found to be within the scatter of the tests. Since potential applications of the composite systems involved multiple cycle loading, high humidity exposure, and salt water submersion, several tests were conducted to evaluate these effects, on K_Q. All samples were subjected to three loading cycles to 75% of ultimate failure and then placed in other environments. High humidity (75% RH) and salt water exposure effects were evaluated in two sample sets. These sets, plus one control set, were then tested at 200°F after removal from the environment. The data show that the fracture toughness, K_Q, was unaffected by the operational environmental exposure and multiple load cycles.

Nomenclature

a = crack length of edge notch
b = half-width of tensile specimen
c_v = coefficient of variation
D = diameter
$F(a/b)$ = geometry correction factor for finite dimension specimens
K_I = Mode I stress intensity factor
K_{Ic} = critical Mode I stress intensity factor or fracture toughness
K_Q = quasifracture toughness for Mode I loading on composite materials
L = length
W = sample width
σ = far-field tensile stress
σ_c = critical far-field tensile stress
σ_{net} = net tensile stress in crack region
θ = helical winding angle

Introduction

THE use of fibrous composites in structural assemblies is widespread. One typical application is in composite pressure vessels for solid rocket motors. There are several types of fibrous composite processing, but the two primary ones are autoclave (pressure-temperature cure) and filament winding. Autoclaved composites usually have no interweaving of the fibers, very few gaps within a given layer, and, generally, because of the pressure cure, have a very low void content. Filament wound composites have considerable interweaving due to the helical ($\pm\theta$) windings in the axial direction. Gaps are generally more prevalent in these structures, and the void content is typically higher. With good compaction and processing, the void content is generally less than 2 to 4%.

Recent interest in fiber-reinforced composite pressure vessels has been with larger diameter cases (74 to 146 in.) for solid rocket motors. Typical operational environments for a solid rocket case have generally been within the 65° to 95°F range. Considering propellant cure conditions and aeroheat environments, the upper end of this range can easily be expanded to encompass thermal soak conditions up to approximately 200°F. A feasibility study on the Space Shuttle Program has considered the use of filament wound composite cases (FWC) to replace the D6AC steel solid rocket motor (SRM) cases.[1] The filament-wound Space Shuttle cases would undergo a rather different environment in some respects. The primary differences are multiple use for several flights, high humidity exposure prior to launch (around 70 to 80% RH nominal), and salt water immersion during recovery operations.

Fracture mechanics is one method to assess the ability of potential composite materials to withstand multiple loading and severe environments. Considerable work has been conducted on flat plate, autoclaved composites to demonstrate that fracture mechanics concepts can provide good engineering design and analysis tools.

The program which was conducted was designed to provide some comparisons between filament-wound and autoclaved composites. The study was also interested in evaluating Kevlar-49/AS4 graphite hybrid composites relative to pure AS4 graphite laminates. A side study on the effects of two lamination configurations with the all-graphite systems was also of interest. Because of the operational environments found on the Space Shuttle, the effects of multiple load cycling, high humidity, salt water exposure, and higher soak temperatures on the fracture toughness characteristics were of interest. As a result, the work discussed in the following sections was aimed at determining these effects using established fracture mechanics techniques.

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Background

The purpose of this section is to provide the reader with an overview of related work in the field and to develop the essential analytical relationships necessary for evaluation of the materials studied under the experimental program.

Theoretical Development

The concept of fracture mechanics has been in existence for some time and is discussed in detail in the work of Tada, et al.[2] Generally speaking, most work is done within the framework of linear elastic fracture mechanics (LEFM), although it is well recognized that plasticity and anisotropy play important roles in the overall analysis of structures with inherent or induced flaws. Since the program involved primarily an evaluation of potential material candidates and their response to certain environmental extremes, the stress intensity factor, or fracture toughness, approach was selected since most of the published work on autoclaved composites used this particular concept.

In all cases, the loading to be induced in the tensile samples was to be an opening mode, Mode I, or normal tension load to the crack.[2] The stress intensity factor for Mode I loading is given by

$$K_I = \sigma\sqrt{\pi a} \tag{1}$$

The critical stress intensity factor, K_{Ic}, relates to the far-field stress which would cause the crack to grow (σ_c), and, therefore, is a measure of the material's resistance to crack growth. The critical stress intensity factor is often referred to as the fracture toughness of the material.

Because the concepts of K_I and K_{Ic} are based on linear elastic fracture mechanics, the fact that many materials inherently fall outside this behavior has prompted us to use the broader designation K_Q. In this concept, the value K_Q is used to symbolize the fact that we are measuring a quasi-fracture toughness of the materials due to their departure from linear elastic assumptions. This approach has been typically used by others working with fiber-reinforced composite materials.[3-6]

Several sample geometries may be used to generate appropriate fracture toughness values of K_Q depending on the manner of loading. The experimental program was designed to use a double-edge notch tensile coupon for which K_Q takes the following form

$$K_Q = \sigma_c\sqrt{\pi a}\,F(a/b) \tag{2}$$

The geometry correction factor $F(a/b)$ is developed by finite element analysis or closed form solution for various sample geometries. The form selected to reduce the data was[2]

$$F(a/b) = 1.122\text{-}0.561(a/b) - 0.205(a/b)^2 + 0.471(a/b)^3 - 0.190(a/b)^4/\sqrt{1-a/b}) \tag{3}$$

This relationship has a numerical accuracy of 0.5% over the entire a/b range. Other solutions, such as those by Irwin,[7] are also available, but do not significantly change the results.

Related Composite Fracture Mechanics Work

Most of the fracture mechanics work on fiber-reinforced composites has been conducted to support either aircraft industry[3-6, 8-12] or automotive applications[13] simply by the nature of the composite processing itself.

Nuismer and Whitney[3,5] performed work on Scotchply 1002 (glass/epoxy) and Thornel 300/Narmco 5208 (graphite/epoxy), using thin laminates of several layup configurations. Their work involved circular holes and center cracks in tensile coupons where the crack length and sample width were varied. The a/b ratio covered a range from 0.2 to 0.67. They were one of the first to show that K_Q increased at low a/b ratios and gave the highest values for the largest sample width.

Similar studies were performed on Thornel 300/Narmco 5208 by Konish and Cruse,[4] using edge-notched bending specimens and centernotched tensile specimens with a thickness range that varied from 0.07 to 0.35 in. They also noted the increase in K_Q with a/b ratio. The effect of various laminate construction was studied by Cruse.[6] Further studies were conducted by Konish[8] on Mode I fracture using center-cracked and double edge-notched tensile coupons in angle-ply composite laminates.

It should be noted at this point that all the work discussed above showed a rather strong dependence of fracture toughness on crack length ratio (a/b). However, recent work by Bathias, et al.[9] and Beaumont and Server[10] showed almost no change in K_{Ic} or K_Q values with this ratio. The results on both centercracked and edge-notched tensile coupons showed the value of fracture toughness to be independent of crack length but increasing with sample thickness. Charpy impact specimens were used by Beaumont and Server,[10] having a 0.°/90° balanced crossply configuration. Their range of variables included impact velocity (0.33 to 5.25 ft/s), temperature (77° to 300°F), and specimen thickness. None of these variables had a significant effect on the fracture toughness.

In summary, much work has been done on flat plate laminates in Mode I tension, but none to date on filament-wound composite systems. and little on relatively thick-wall composites. Furthermore, some of the results are not in agreement concerning the effect of crack length on the fracture toughness, although several investigators are working in this area. Partially because of the lack of data on thick-wall, filament-wound composites, the experimental program discussed in the sections that follow was designed to look into some of these areas.

Experimental Program

As noted in the earlier discussions, a considerable amount of work has been conducted on glass/epoxy composites. However, to the authors' knowledge, no work has been reported on filament-wound composite materials. The existing data on composites were derived from flat plate laminates prepared using pressure-cure autoclave processing and, furthermore, were done on relatively thin (<0.10 in.) composite sheets. Since the present interest lies in the area of filament-wound pressure vessels which are relatively thick (>0.20 in.), the experimental program was designed to examine potential differences in filament-wound composite fracture toughness characteristics.

Two materials are in wide use for pressure vessel applications: Kevlar-49 organic fiber and AS4 graphite fiber. The Kevlar-49 fiber is manufactured by DuPont, and the AS4 graphite fiber is produced by the Hercules Aerospace Division. The experimental program was established to evaluate the fracture characteristics of all-graphite composite laminates relative to a hybrid Kevlar/graphite system. The effect of layup construction on the all-graphite composite was further evaluated by selecting two configurations.

Sample Source

All the samples were ultimately taken from a filament-wound cylinder with nominal dimensions of 12.7 in. $D \times 48$ in. L. The 12-in. cylinder fabrication details are outlined in Table 1. The hoop materials (windings) were either Kevlar-49/HBRF-55A or AS4 graphite/HBRF-55A, depending on whether the particular section was a Kevlar/graphite hybrid or all-graphite. The 5-6 lb/roving winding tension on the hoops, coupled with the use of 2-in.-wide Armalon tape, provided good compaction so that the void content was kept below 3% (by volume).

Table 1 12 in. cylinder fabrication details

	Materials
Hoop (90°)	AS4 graphite/HBRF-55A
	Kevlar-49/HBRF-55A
Helical (±10°)	AS4 graphite/HBRF-55A
Reinforcements	AS4 graphite/HBRF-3501
(0° and ±45°)	Prepreg broadgoods
	Winding tension (lb/roving)
Hoop	5-6
Helical	2-3
	Compaction method
Armalon tape (lb/in.)	20
	Cure conditions
Temperature (°F)	350
Time (m)	120

Table 2 12 in. cylinder laminate description

	Laminate percentages (%)		
	$[\pm 10°/90°]_3$	$[\pm 10°/\pm 45°/90°]_3$	$[\pm 10°/0°/\pm 45°/90°]_3$
Kevlar-49 hoops	50	—	—
AS4 graphite (±10°) helicals	50	41	41
AS4 graphite hoops	—	28	28
AS4 graphite (0°) broadgoods	—	—	12
AS4 graphite (±45°) broadgoods	—	31	19

The helical windings were all wet-wound with AS4/HBRF-55A resin at an angle of ±10° to the axis. Reversals of the helicals on the aluminum cylindrical mandrel were accomplished by pin rings installed at each end. A lower winding tension (2 to 3 lb/roving) was used so that fiber breakage was reduced to a miminum around the pin rings. The reinforcement areas were wound with 0° and ±45° fiber angles using AS4 graphite/HBRF-3501 prepeg broadgoods. The broadgoods were applied and cut to give the desired fiber angle. Hand layup was used to apply the broadgoods during cylinder fabrication. After completion of each broadgood buildup layer, the Armalon tape was wound over the cylinder at 20 lb/in. tension to help compact the broadgoods and minimize voids. The tape was removed prior to winding the helicals. Cure conditions were 350°F for 120 m.

The HBRF-55A winding resin and the HBRF-3501 prepeg resin systems have been used extensively in other programs with good success. Both have excellent properties over the range of temperature conditions of interest.

Three laminate sections were wound on the 12-in. cylinder as shown in Table 2. The sections consisted of a hybrid Kevlar/graphite layup with an orientation of $[\pm 10°/90°]_3$ and two all-graphite sections. The AS4 graphite sections had orientations of $[\pm 10°/\pm 45°/90°]_3$ and $[\pm 10°/0°/\pm 45°/90°]_3$ in order to evaluate the contribution of the 0° fiber angle. The overall thickness of the filament wound composite cylinder was approximately 0.28 in.

Sample Configuration

The 12-in. cylinder was sectioned axially, and the fracture toughness samples were then cut in the axial direction to minimize curvature effects. A schematic of the sample is shown in Fig. 1. The thickness was a nominal 0.28 in., but

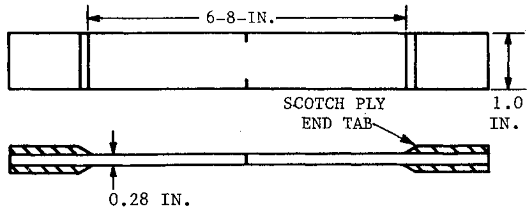

Fig. 1 Schematic of fracture toughness tensile specimen.

depended to some degree on the particular laminate construction. Sample width was chosen as 1 in. in order to obtain approximately 30 to 36 samples from each section. Scotchply (E-glass/epoxy) and tabs were bonded on using EC 2216 adhesive. The test section of each sample was approximately 6 to 8 in.

The double-edge notch fracture toughness test was chosen primarily for two reasons: the availability of previous Kevlar-49/HBRF-55A data on this configuration[14] and the ease with which the edge-notch crack could be cut into the samples.

Test Program

Prior to conducting the fracture program, several of the tensile specimens were prepared with surface strain gages before cutting the edge notch. These samples were conditioned at 77°, 150°, 200°, and 250°F and then loaded at 0.05 in./min to approximately 30,000 psi to obtain stiffness properties. After unloading, they were then used in the fracture study.

All edge notches were cut into the samples to various depths ranging from 0.10 to 0.35 in., using a narrow diamond saw. Care was taken to ensure crack alignment with the opposite edge and to ensure the crack was perpendicular to the loading direction.

The first portion of the program was aimed at establishing the optimum crack size to obtain a value of the fracture toughness K_Q which was independent of crack size. As a matter of definition, the crack length was defined as a and the sample width as $2b$. Other studies have defined the sample width as W such that the ratio a/W is comparable to $a/2b$ and not a/b. Crack sizes of $a=0.10$, 0.175, 0.25, 0.30, and 0.35 in. were selected to define the fracture characteristics over an a/b range from 0.2 to 0.7. All of these tests were conducted at 77°F using a crosshead rate 0.05 in./min on all three materials.

The next series of tests was conducted using a crack size of $a=0.25$ in. The value was selected on the basis of the initial studies and was found to be near the point at which the value of K_Q began to level off. Three samples of each material configuration were tested at a crosshead rate 0.05 in./min after temperature conditioning to 150° and 200°F. These conditions are felt to be within the range of normal pressure vessel operational limits for thermal soak conditions. Aeroheat conditions could drive the temperature to higher levels on the outer surface, but a "through-crack" would not experience the aeroheat temperature throughout the thickness. The aeroheat environment would be more applicable to "part-through crack" conditions, which were not part of this study.

Since the use of this composite would potentially involve other extreme conditions such as high humidity, salt water exposure, and multiple use (or load cycling) at elevated temperature, other tests were conducted to assess these effects. Again, a nominal crack size of $a=0.25$ in. was selected to provide a common data base. Multiple load cycling was a key environment. Consequently, nine samples of each material configuration were notched and then load-cycled at 77°F and a 0.05 in./min crosshead rate for three full cycles. Each cycle consisted of tension loading the specimen to 75% of the ultimate stress (load) established in the virgin crack size studies for the $a=0.25$ in. tests. After reaching the peak load,

Table 3 Tensile coupon properties[c]

Laminate orientation	Ultimate tensile stress (ksi)	Tensile modulus (10^6 psi)
$[\pm 10°/90°]_9$[a]	106.9	8.67
$[\pm 10°/\pm 45°/90°]_9$[b]	112.3	9.72
$[\pm 10°/0°/\pm 45°/90°]_9$[b]	124.5	11.91

[a]Kevlar-49 hoops (90°) and AS4 graphite helicals. [b]AS4 graphite hoops and helicals. [c]Obtained from a full-thickness (0.96-in.) filament wound cylinder.

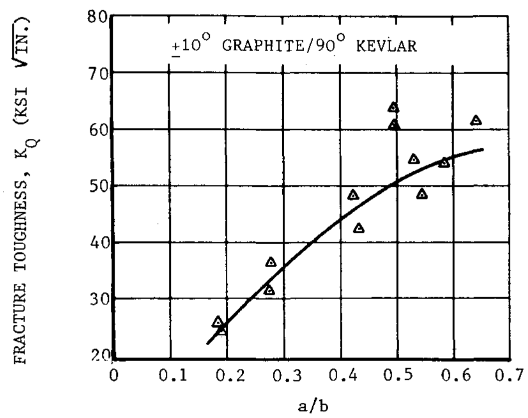

Fig. 2 Fracture toughness dependence on crack size (*a/b*) for $[\pm 10°/90°]_3$ composite.

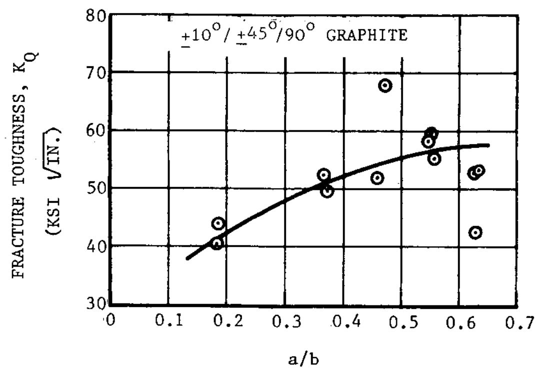

Fig. 3 Fracture toughness dependence on crack size (*a/b*) for $[\pm 10°/\pm 45°/90°]_3$ composite.

the sample was unloaded at the same crosshead rate until a 200-lb tension load was reached. The next cycle was repeated by starting at the 200-lb load level. At the end of the multiple load cyling, the samples were removed for the next phase of the environmental conditioning. It should be noted at this point that the 200-lb tension limit was arbitrarily selected so as not to accidentally induce compression buckling loads in the test sample.

Having conditioned the samples using multiple load cycles, the next phase evaluated the additional effects of high temperature, high relative humidity, and sea water exposure on the composite fracture characteristics. The first set of samples was conditioned to 200°F and then pulled to failure at 0.05 in./min to simulate a fourth loading cycle at elevated temperature. The second set of samples was conditioned over a glycerine-water solution mixed to give 75% RH at ambient temperature. The samples were conditioned for two weeks, removed and conditioned to 200°F, and pulled to failure at 0.05 in./min. The time interval between removal and the

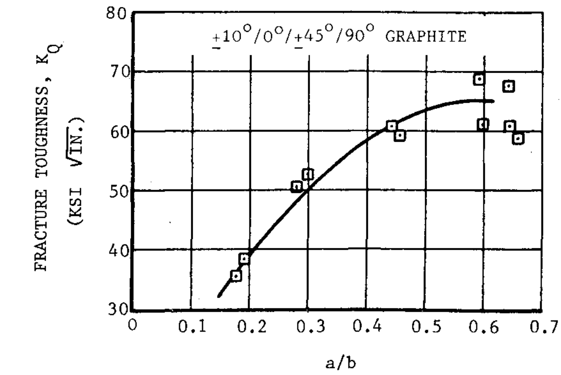

Fig. 4 Fracture toughness dependence on crack size (*a/b*) for $[\pm 10°/0°/\pm 45°/90°]_3$ composite.

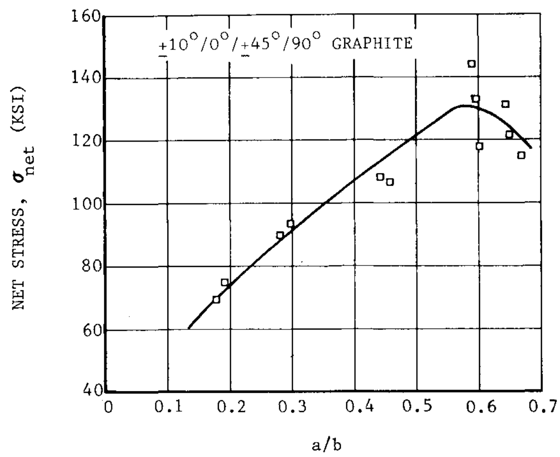

Fig. 5 Typical net stress dependence on crack size (*a/b*).

actual failure test was kept below 3 h to maintain a high humidity around the notch area. The third set of samples was immersed in a salt water solution for 48 h to simulate a typical ocean recovery period for a pressure vessel following launch and splashdown. These specimens were then removed and tested at 200°F in the same manner as the 75% RH samples.

Discussion of Results

The first set of tests was conducted to determine axial coupon stiffness over the 77° to 250°F temperature range. The axial modulus was found to be relatively insensitive to the temperature. Other data were also available on a thick-wall (0.96-in.) cylinder of the same laminate construction as shown in Table 3. The absence of the ±45° broadgoods in the Kevlar hybrid results in a lower axial stiffness of at least 10% as expected. However, incorporating 0° reinforcement in the AS4 graphite laminate increases the stiffness considerably (23%).

The results of the crack length studies are shown in Figs. 2 through 4. It should be kept in mind that these tests were all conducted at the same crosshead rate and temperature (77°F). All three composites shown a strong dependence of K_Q on the crack length. The data are not sufficient to define clearly the crack length at which K_Q levels out to the maximum value. However, K_Q tends to start reaching an asymptotic value in the range of $a/b = 0.5$ to 0.6. Both AS4 graphite laminates appear to have reached a maximum, whereas the Kevlar/graphite hybrid has not leveled out. The results agree with those of previous researchers, which show an increase in fracture toughness with crack length.[3-5] Some recent work on filament wound Kevlar/HBRF-55A tensile coupons with double-edge notch samples has shown the same effect.[14] It is

Table 4 Effect of temperature on fracture toughness, K_Q

Laminate orientation	Materials	Temperature, °F	Fracture toughness, K_Q (ksi $\sqrt{\text{in.}}$)
$[\pm 10°/90°]_3$	Kevlar-49 hoops	77	45.3
		150	46.8
		200	43.2
$[\pm 10°/\pm 45°/90°]_3$	All-graphite	77	59.6
		150	57.7
		200	53.9
$[\pm 10° 10°/0°/\pm 45°/90°]_3$	All-graphite	77	60.0
		150	64.1
		200	61.1

Crack length of approximately 0.25 in. with corresponding a/b ratio of 0.5.

Table 5 Summary of effects of various environmental exposures on fracture toughness (K_Q)

Laminate orientation	200°F (control)	3 Load cycles to 75% ultimate, 200°F	3 load cycles to 75% ultimate, 75% RH for 2 weeks, 200°F	3 load cycles to 75% ultimate, 48 hours in salt water, 200°F
	Fracture toughness, K_Q (ksi $\sqrt{\text{in.}}$)			
$[\pm 10°/90°]_3$	43.2	43.1	47.4	49.3
(Kevlar-49 hoops)	$C_v = 10.0\%$			
$[\pm 10°/\pm 45°/90°]_3$	53.9	52.6	55.1	56.2
(All-graphite)	$(C_v = 7.9\%)$			
$[\pm 10°/0°/\pm 45°/90°]_3$ (All-graphite)	61.1 $(C_v = 7.\%)$	53.0	52.7	56.0

Crack length of approximately 0.25 in. with corresponding a/b ratio of 0.5.

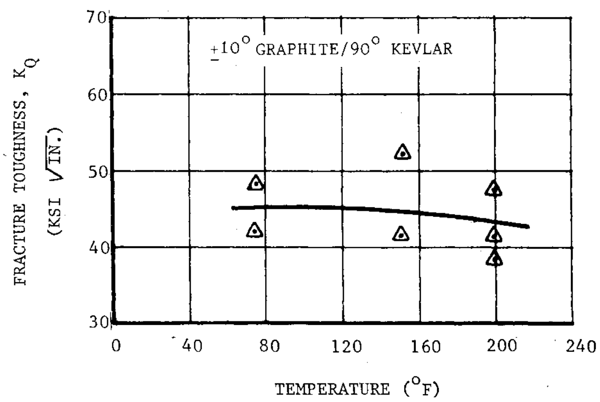

Fig. 6 Effect of temperature on fracture toughness of $[\pm 10°/90°]_3$ composite.

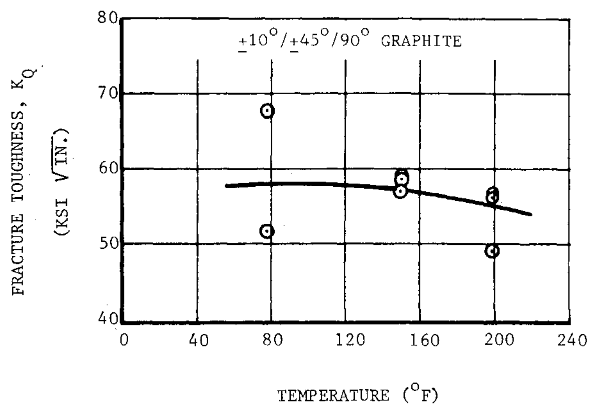

Fig. 7 Effect of temperature on fracture toughness of $[\pm 10°/\pm 45°/90°]_3$ composite.

also interesting that the net tensile stress shows the same effect as K_Q (Fig. 5).

Using Figs. 2 through 4, the value of effective fracture toughness, K_Q, can be estimated as

$[\pm 10°/90°]_3$	56 ksi $\sqrt{\text{in.}}$
$[\pm 10°/\pm 45°/90°]_3$	57 ksi $\sqrt{\text{in.}}$
$[\pm 10°/0°/\pm 45°/90°]_3$	65 ksi $\sqrt{\text{in.}}$

These should be considered as strictly estimates, but the data points appear to be reasonable. The first observation to be made is that the Kevlar hybrid exhibits comparable fracture toughness to its AS4 graphite counterpart (without the 0° reinforcement). As expected, the addition of 0° fibers creates a significant increase in fracture toughness, being about 10 to 15%. Nuismer and Whitney[3] report values ranging between 47 to 62 ksi $\sqrt{\text{in.}}$ for Thornel 300/Narmco 5208 flat plate laminates with similar, but not identical, laminate construction. These results are encouraging, since differences in Mode I fracture behavior between these filament wound thick-wall composites and the flat plate results shown to date do not appear to be significant.

The temperature study results are shown in Table 4 and Figs. 6 through 8. It should be remembered that the a/b ratio for these and the environmental tests was 0.5. The results show that K_Q is not sensitive to temperature for any of the three composites. The coefficients of variation material were between 5 to 10%. The only temperature data available, that of Beaumont and Server,[10] showed the fracture toughness to be independent of temperature. Although not stated in Ref. 6, the resin was believed to be the same as that used in the present broadgoods, HBRF-3501.

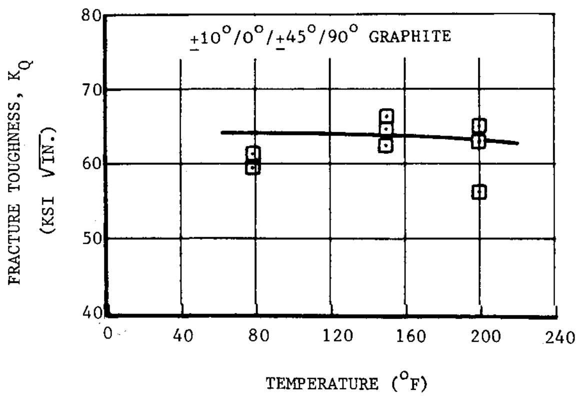

Fig. 8 Effect of temperature on fracture toughness of $[\pm 10°/0°/\pm 45°/90°]_3$ composite.

Finally, the results of the multiple load cycling and environmental exposure tests are given in Table 5. Neither the load cycling nor the extreme environments had an effect on the composite fracture toughness. The data indicate no difference between first cycle loading at 200°F or a subsequent fourth cycle at 200° after multiple loading. Although the samples contain cut edges and notches, the 75% RH and salt water environments would not be able to penetrate the entire width of the tensile coupon fully. However, the time durations are expected to be sufficient to influence the crack growth behavior around the notches if the environment were to be a strong effect. The results show that the effects are negligible, even at the 200°F test temperature.

Conclusions

The Mode I fracture toughness tests on the filament wound composite materials yields values of K_Q that were comparable to those reported for flat plate, autoclaved laminate systems. The initial series of tests on the unnotched specimens to determine axial stiffness were in agreement with analytical predictions (within 5%). Furthermore, it was found that the stiffness did not change appreciably over a temperature range of 77° to 250°F for short-term, thermal soak conditions.

The value of K_Q showed a strong dependence on crack size. These data were in agreement with the results shown by Beckwith,[14] Nuismer and Whitney,[3] Konish and Cruse,[4] and Whitney and Nuismer.[5] This dependence was observed over a wide range of crack length-to-half-width ratios (a/b) up to about $a/b = 0.5$ to 0.7. The Kevlar-49/AS4 graphite hybrid yielded a value of $K_Q = 56$ ksi $\sqrt{\text{in.}}$ whereas the AS4 graphite laminates were found to have a value of $K_Q = 57$ ksi $\sqrt{\text{in.}}$ and 65 ksi $\sqrt{\text{in.}}$ (with 0° reinforcement). It was noted that the replacement of some of the ±45° broadgoods with 0° reinforcement provides approximately a 10 to 15% increase in the fracture toughness.

The effect of temperature (77° to 200°F) was found not to be significant over the range studied. All of the fracture toughness (K_Q) values were within the scatter of the tests (roughly 10%). Tests conducted at 200°F after three multiple loading cycles to 75% of ultimate strength were comparable to the virgin tests on the three notched materials. When the three multiple load cycles were coupled with high humidity (75% RH) or salt water exposure followed by subsequent testing at 200°F, no degradation of the fracture toughness (K_Q) was observed.

In all cases, the values of the fracture toughness for the filament-wound composite laminates appear to be comparable to those reported on flat plate, autoclaved laminates. Furthermore, the effect of thickness (0.28 in.) does not seem to be significant. Most reported data were derived from relatively thin laminates, typically approximately 0.06 to 0.10 in. nominal plate thickness. The results of Konish and Cruse[4] over a thickness range of 0.07 to 0.35 in. suggest the same.

Acknowledgments

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