Hygrothermal influence on the interlaminar shear strength of Kevlar-graphite/epoxy hybrid composites

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The hygrothermal influence on the interlaminar shear strength of Kevlar 49-graphite/epoxy hybrid composite was investigated in the temperature range -50 to 150 \degree C. Moisture was introduced into the specimen by immersion in distilled water until a specified weight gain occurred. Two material lay-up were used in this investigation. In Kev 49/T-300/Kev 49, Kevlar 49 and graphite prepreg tapes were used as outer and centre layer, respectively and in T-3OO/Kev 49/T-300, it was vice versa. In both cases the tapes were alternated until the total thickness was achieved. The results show that interlaminar shear strength change with the ply sequence of hybrid laminates. The interlaminar shear strength of T-3OO/Kev 49/T-300 is relatively higher than that of Kev 49/T-300/Kev 49. The interlaminar shear strength of both T-3OO/Kev 49/T-300 and Kev 49/T-300/Kev 49 decrease with the increase of temperature in the range -50 to 150° C. The addition of moisture further degrade the interlaminar shear strength in the same temperature range. Close physical observation showed clear evidence of interlaminar shear failure for most of the specimens.

1. Introduction

Failure of laminated composites is frequently due to three-dimensional stress state. Interlaminar stresses related to three-dimensional problems are not considered in classical lamination theory which is most widely used for composite laminate analysis. Current interest on thick section composite for submarine structure require extensive study of interlaminar stresses in such applications. Moreover, superior stiffness and strength of composites are not always dependent on parameters related to the thickness direction of the laminate [1].

The interlaminar shear stress is determined to be higher at the free edge of the laminate (sides or holes) and possibly the cause of debonding which appears in such regions [2, 3]. Similar effect is also studied by a finite-element model with nonlinear shear response [4]. Environmental factors such as moisture and temperature degrade the interlaminar shear strength of graphite/epoxy and Kevlar/epoxy [5, 6]. Among the possible reasons of such degradation, the damage of the resin by moisture because of swelling and the temperature effect on thermal expansion coefficient of both fibre and resin are important. Decomposition of the resin at elevated temperature is also responsible for loss in stiffness and strength of the composites. Due to moisture absorption the glass transition temperature (T_{g}) of the resin becomes low which in turn degrade the high temperature property of the resin. Detail degradation mechanism for environmental exposure is studied by Tsai [7].

Although the hygrothermal influence on the mech-

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anical properties of hybrid composite is related to the same influence on the individual fibre and matrix, it is required to generate similar data on hybrid composites. There exist a mismatch of thermal expansion coefficients and swelling coefficients of individual single fibre lamina used in hybrid. The prediction of hygrothermal influence for hybrids from the similar data of single fibre composites would not be accurate because resistance to the moisture and temperature mostly depends on the outer skin of the laminate. As a result of this understanding an attempt has been made to determine the hygrothermal influence on interlaminar shear strength with two different outer ply of the same hybrid system. No experimental data are available in the literature on the hygrothermal influence on interlaminar shear strength of Kevlar 49-graphite/epoxy hybrid composites. Data generated in this investigation could be used as a guideline for design of this material under hygrothermal influence.

2. Experimental work

2.1. Materials fabrication

Unidirectional prepreg materials of Kevlar 49/epoxy and T-300/epoxy in the form of tape were used for fabrication of interply hybrid laminates. The material was purchased from American Cyanamid under the brand name Cycom 985. Alternating plies of unidirectional prepreg tape were manually laid up on an aluminium template to make 0.3 cm square hybrid laminates. Plies were arranged symmetrically in the laminate to avoid warpage because of temperature change during curing. Total fibre content was

maintained at 60% by volume. The laminate was cured in a hot press made by Tetrahedron. The curing cycle was as follows: under vacuum of at least 26 inches of mercury, the laminate was heated to 66° C and then held for 1 h at 66° C. The vacuum was then released and pressure increased to 100 p.s.i. The temperature was then increased to 177° C at a rate of about -15.8° C min⁻¹ and held for 2h at a pressure of 100 p.s.i. Upon completion of cure, the heaters were turned off and cooling was continued under pressure until the laminate temperature fell below 66°C. Two thermocouples were inserted inside the laminate to record the temperature during the curing process. The complete curing cycle was programmed in five steps. After the curing was completed the laminate was debagged and used for making specimens.

2.2. Specimen design

Four different types of specimens were used for short beam shear test (ASTM designated D2344-84). Complete specimens configurations are as given in Table I.

2.3. Conditioning

Specimens with moisture content and those completely dry were used in this investigation. All specimens were pre-conditioned in a vacuum oven at 100° C until a near-equilibrium weight was obtained. This condition of the specimen is referred as dry.

After pre-conditioning, the specimens which were labelled dry were stored in a dessicator and those labelled as wet were placed in a container of distilled water inside an oven at a temperature of 23° C for moisture absorption. It should be noted that a temperature of 23° C is much lower than the wet glass transition temperature of the resin. Higher temperature may induce permanent damage in the test specimen in the form of cracks. Specimens were removed from the container at various time intervals for weight measurements. The effect of this removal on weight gain determination has been shown to be negligible. Specimens were allowed to cool for a short period of time before being weighed. In case of specimens with moisture, the surfaces were carefully wiped before weighing. The weight gain process was continued until the specified weight gain occurred. Tests were performed at -50 , 23, 100 and 150 \degree C for both dry and wet specimens in a thermally stabilized test chamber. It required 4 min for setting and loading the specimen to its maximum load. Moisture desorption for 4 min at all temperatures was calculated by placing a specimen within the chamber. The amount of moisture desorbed during the short span of setting and loading time was found to be negligible.

2.4. Test **method**

Short beam shear tests were performed for the determination of interlaminar shear strength. The tests were performed in a 22 Kips MTS machine attached with an environmental chamber. Span to thickness ratio of 4/1 was selected for the test. Rate of crosshead motion was maintained at 0.05 inch min⁻¹. The load was applied to the specimen and load-deflection data plotted in a recorder.

3. Results and discussion

The absorption of moisture as a function of time is presented in Fig. 1 for the two types of hybrid specimens used in this study. The weight gained as a result of water absorption was computed and plotted as a function of the square root of time. It should be mentioned that size of the short beam shear specimens are relatively smaller and determination of moisture content increase is unpredictable from a single specimen. The increase of moisture content to a total of 20 specimens were determined and the average of this weight gain was considered in determining the increase in per cent fraction of the specimen weight. The two curves are straight over the initial period which shows that they satisfy Fick's law. The slope of the linear region is directly proportional to diffusivity of the material. It appears from the plot that the diffusivity of Key 49/T-300/Kev 49 specimen is higher than that of T-300/Kev 49/T-300 specimen. Key 49/T-300/Kev 49 and T-300/Kev 49/T-300 specimens attain a maximum moisture content of 2.3 and 1.8%, respectively, in 24 days. Five specimens of each type were tested at

Figure i Moisture absorption in Kevlar 49-graphite/epoxy hybrid composite. (*-*) T-300/Kev 49/T-300. (\triangle - \triangle) Kev 49/T-300/Kev 49.

Figure 2 Load versus deflection curve for short beam test as function of temperature and moisture, Kev 49/T-300/Kev 49. (1) Dry, 23° C. (2) 2.3% M, 23° C. (3) Dry, 100° C. (4) 2.3% M, 100° C. (5) Dry, 150° C. (6) 2.3% M, 150° C.

this stage and remaining specimens were kept immersed in distilled water for a period of 90 days. No significant weight increase was observed between 24 and 90 days exposure. Sometimes a decreased weight was marked during that period. Specimens were tested again after 90 days exposure to determine the change in interlaminar shear strength with exposure times.

Load against deflection response of Kev 49-graphite/ epoxy as a function of temperature and moisture content are shown in Figs 2 and 3. It should be noted that the load-deflection plots are continued slightly beyond the maximum load. All the curves display an amount of nonlinearity. It appears from Figs 2 and 3 that the brittleness of both Kev 49/T-300/ Kev 49 and T-300/Kev 49/T-300 decrease with the increase of temperature. In both the load-deflection curves maximum loads decrease with the increase of temperature. The addition of moisture further reduces the maximum load.

Data for interlaminar shear strength of all the four types of materials are presented in Table II. It is evident that interlaminar shear strength of T-300/Kev 49/T-300 specimens are comparatively higher than those of Key 49/T-300/Kev 49, Kevlar 49/epoxy has the lowest interlaminar shear strength in comparison to hybrid and graphite/epoxy.

Figs 4 and 5 present effects of moisture content and temperature on the interlaminar shear strength of all four types of materials used in this study. The data for Kev 49/T-300/Kev 49 shows a reduction of 28% in the interlaminar strength at 150° C than the corresponding values at 23° C. The 90 days moisture exposure of the

Figure 3 Load versus deflection curve for short beam test as function of temperature and moisture, T-300/Kev 49/T-300. (1) Dry, 23° C, (2) 1.8% M, 23° C, (3) Dry, 100° C, (4) 1.8% M, 100° C, (5) Dry, 150° C. (6) 1.8% M, 150° C.

material further reduces the strength to 57%. Similarly, interlaminar shear strength for T-300/Kev 49/T-300 shows 33% reduction at 150° C in dry conditions than the corresponding values at room temperature. The strength reduction rate increase to 51% after 90 days moisture exposure for the same material. The results show that the interlaminar shear strength decrease negligibly with moisture contents at -50° C for both T-300/Kev 49/T-300 and Key 49/T-300/Kev 49. In case of Kevlar 49/epoxy, the interlaminar shear strength appear to be higher with moisture content at -50° C.

Figs 6 and 7 present interlaminar shear strength against moisture exposure time. It can be seen from the graphs that interlaminar shear strength for Kev 49/T-300/Kev 49 and T-300/Kev 49/T-300 continue to decrease with exposure time beyond 24 days at all temperature ranging from 23 to 150° C. It should be noted that increase in moisture absorption after 24 days was unnoticeable.

Microscopic failure analysis was not done in this study. Interlaminar shear failure was clearly evident in most of the specimens after close physical observation.

4. Conclusion

From the results of the investigation of the hygrothermal influence on the interlaminar shear strength of Kevlar 49-graphite/epoxy hybrid composites the following conclusion can be drawn:

1. The interlaminar shear strength decreases with the increase of temperature in the range -50 to 150~ for both Kev 49/T-300/Kev 49 and T-300/Kev

TABLE II Comparison of hygrothermal influence on interlaminar shear strength: Kevlar 49/epoxy, graphite/epoxy and hybrid laminates

Conditioning		Short beam shear strength			
Temperature $(^{\circ}C)$	Dry D Saturated S	Kev 49/T300/Kev 49 (k.s.i.)	T300/Kev 49/T300 (k.s.i.)	T300/Ep (k.s.i.)	Kev 49/Ep (k.s.i.)
-50	D	12.15	13.12	18.39	6.49
		11.63	12.82	16.69	8.99
23	D	11.14	12.57	15.23	9.65
		9.57	10.18	12.25	6.68
100	D	9.70	10.79	10.83	7.72
		7.73	8.81	10.19	6.22
150	D	7.98	8.43	6.61	5.15
	S	4.68	6.05	5.46	3.50

Figure 4 Moisture and temperature effect on interlaminar shear strength of Kevlar 49-Graphite/epoxy. \bullet Dry, T-300/Kev 49/T-300. Saturated, T-300/Kev 49/T-300. \longrightarrow Dry, Kev 49/T-300/Kev 49. *--* Saturated, Kev 49/T-300/Kev 49.

Figure 5 Moisture and temperature effect on interlaminar shear strength of Kevlar 49/epoxy and graphite/epoxy. \bullet Dry, Kev 49/Epoxy. \blacksquare Saturated, Kev 49/Epoxy. \blacktriangle Dry, T-300/Epoxy. \ast - \ast Saturated, T-300/Epoxy.

49/T-300. The addition of moisture further degrades the interlaminar shear strength in the same temperature range for the same materials.

2. The interlaminar shear strength vary with the ply sequence of the hybrid laminate. The interlaminar shear strength of T-300/Kev 49/T-300 is relatively higher than that of Kev 49/T-300/Kev 49.

Figure 6 Interlaminar shear strength of T-300/Kev 49/T-300 as a function of moisture exposure days and temperature. -50° C. \blacksquare \blacksquare 23° C. \blacktriangle \blacktriangle \blacktriangle 100° C. \blacktriangleright \blacksquare \blacklozenge 150° C.

3. Interlaminar shear strength changes with exposure time even after attaining maximum moisture contents.

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Figure 7 Interlaminar shear strength of Kev 49/T-300/Kev 49 as a function of moisture exposure days and temperature. *--*- -50° C. \blacksquare \blacksquare 23° C. \blacktriangle \blacksquare -4 100° C. \blacklozenge -4 \blacksquare 150° C.

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References

- 1. S. W. TSAI, (ed.) "Composite Design", 4th edn (Think Composite Publisher, 1988) Ch. 22, p. 11.
- 2. R.B. PIPES and N. J. PIGANO, *J. Comp. Mater.* 4(4) (1970) 538.
- 3. R. B. PIPES and I. M. DANIEL, *ibid.* 5(2) (1971) 255.
- 4. G. ISAKSON and A. LEVY, *ibid.* 5(2) (1971) 273.
- 5. G. S. SPRINGER, (ed.) "Environmental Effects on Composite Materials", Vol. 1 (Technomic, 1981).
- 6. G. S. SPRINGER, (ed.) "Environmental Effects on Composite Materials", Vol. 2 (Technomic, 1984).
- 7. S. W. TSAI, "Environmental Factors in the Design of Composite Materials" 5th Symposium Naval Structural Mechanics, 1967 (Pergamon, New York, 1967).

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