INFLUENCE OF ENVIRONMENTAL CYCLING ON THE MECHANICAL
PROPERTIES OF CARBON FIBRE REINFORCED
PLASTIC MATERIALS

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Abstract

Static and fatigue tests were carried out on Carbon Fibre Reinforced Plastic specimens exposed to environmental cycling with the purpose of simulating the temperature/humidity conditions an aircraft may meet during actual use. The specimens were built up from carbon-fibre prepreg THORNELL 300/NARMCO 5208.

The investigation has comprised $[\pm 45^{\circ}]_{S8}$ - and $[0^{\circ}/\pm 45^{\circ}/90^{\circ}]_{S8}$ - reinforced test specimens and beams. The fatigue tests of $[\pm 45^{\circ}]$-specimens were loaded in pure tension with the stress ratio $R = 0.1$. The fatigue tests of $[0^{\circ}/\pm 45^{\circ}/90^{\circ}]$-specimens were loaded in both tension $(R = 0.1)$ and in tension/compression with $R = -1$.

The results show clear detoriations in both static and fatigue properties of the matrix controlled $[\pm 45^{\circ}]$ specimens when exposed to hard environmental conditions. The material becomes more ductile under influence of moisture, and after prelonged exposure one can notice a debonding effect between the fibres and the matrix. The fibre controlled $[0^{\circ}/\pm 45^{\circ}/90^{\circ}]$-specimens were exposed to a less hard environmental cycle and showed no degradation in mechanical properties.

I. Introduction

Fibre reinforced plastics are being developed for use in many weight critical systems, and their performance during real environmental exposure should be evaluated. Environmental factors of major importance include a combination of high huminity and high temperature. Several investigations have been made during the last years in order to study environmental effects on composite materials. Studies made with both epoxy resins alone and with graphite-epoxi composites have demonstrated that absorbed moisture has a plasticizing effect which causes softening of the resin, specially at high temperatures, and a corresponding decrease in mechanical properties. The degree of mechanical property degradation are not so marked for all types of composites but seems to be a function of:
1. The fibre orientation
2. The specific resin matrix
3. The temperature and humidity during exposure
4. The time of exposure
5. The ratio of specimen surface to specimen volyme

The data obtained from Browning [1] show that fibre-dominated failures are relatively unaffected by absence or presence of moisture, whereas matrix-dominated properties are adversely affected, especially at elevated temperatures and also to a substantial degree at room temperature.

Different resins have different absorption rates, different equilibrium levels of absorbed moisture and different sensitivities to absorbed water with respect to physical property degradation. When laminates are exposed to constant-temperature/constant humidity condition, moisture is absorbed at a rate determined by the time of exposure. An initial, rapid moisture absorption occurs at the surface and the continuing absorption is much slower which means that the time to reach equilibrium is also dependent on the ratio of specimen surface area to specimen volyme. The presense of void content is on additional variable of interest in the study of moisture degradation effects.

The influence of pre-loading the specimens before the exposure was also a factor of interest. By pre-loading the testspecimens it was expected that micro-cracks would occour in the laminate causing faster moisture absorption.

The present investigation has been devided into two parts where the first part [4] was supposed to show that environmental exposure can be a serious problem for reinforced plastic laminates. Therefore a matrixcontrolled $[\pm 45^{\circ}]$-reinforced laminate was used which was known from the litterature to be sensitive to humidity. Also a rather hard environmental cycle was used. (See fig 1)
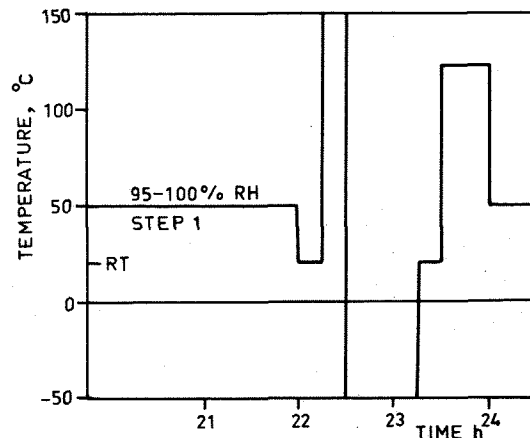

Figure 1. Environmental cycle used for testing the $[\pm 45^{\circ}]_{S8}$ reinforced specemens

In the second part of the investigation the purpose was to investigate the environmental effects on a more commonly used fibre controlled laminate with the lay-up angles $[0^{\circ}/\pm45^{\circ}/90^{\circ}]$. The temperature range of the environmental cycle was redused from $(-50^{\circ}C$ to $+150^{\circ}C)$ to $(-40^{\circ}C$ to $75^{\circ}C)$ in order simulate a more suitable environmental cycle. (See fig 2)
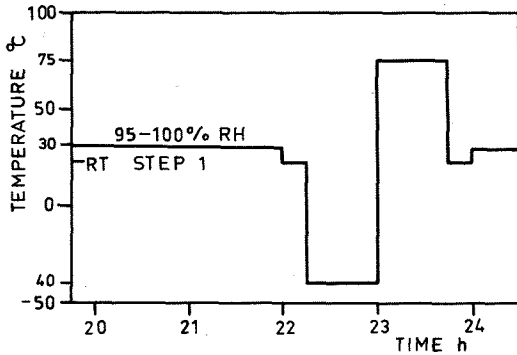

Figure 2. Environmental cycle used for testing the $[0^{\circ}/\pm45^{\circ}/90^{\circ}]_{S8}$ reinforced speciemens

II Conclusions

Static and dynamic properties of $[\pm45]_{S8}$ graphite/epoxy Thornel 300/Narmco 5208^{S8} are presented in Table 1 for different values of the following parameters: exposure time, surface treatment and pre-loding. The resulting conclusions are as follows:

1. The static tensile strength decreases with longer time of exposure.

2. Specimens that have not been pre-loaded show a higher tensile strength compared with the pre-loaded group when exposed to environmental cycling for the same length of time. The polyurethane-covered specimens also show better mechanical properties.

3. The fatigue properties are reduced significantly, after six weeks of exposure none of the specimens survived 10^6 load cycles.

4. The influence of moisture and thermal cycling plasticizes the composite, as seen both from the ultimate strain and from the studies of the fracture surface in sweep electron microscope. From the fracture surface, Figures 3 and 4 it is possible to see a debonding between fibers and matrix in specimens that have been cycled for six weeks. This effect has not been noticed in the specimens aged for less than six weeks, nor in the surface treated specimens.

TYPE OF ENVIRONMENTAL AND MECHANICAL INFLUENCE	TIME OF EXPOSURE (WEEKS)	NUMBER OF SPECIMENS		STATIC TESTS		FATIGUE TESTS		
		Static	Dynamic	(N/mm^2)	$s\,(\%)$	N_{avg}	Retaining strength (N/mm^2)	$s\,(\%)$
Uninfluenced	0	10	8	$\bar{\sigma}_B = 156.7$	4.65	10^6	$\bar{\sigma}_B = 154.9$	7.00
				$\bar{\epsilon}_B = 0.011$	10.01		$\bar{\epsilon}_B = 0.017$	10.52
				$\bar{E} = 22644$	2.4		$\bar{E} = 20111$	5.17
Mechanical and environmental influence	2	10	8	$\bar{\sigma}_B = 156.4$	2.44	.9047 * 10^6	$\bar{\sigma}_B = 136.1$	3.68
				$\bar{\epsilon}_B = 0.017$	8.52		$\bar{\epsilon}_B = 0.009$	1.88
				$\bar{E} = 19801$	3.85		$\bar{E} = 21644$	2.72
Mechanical and environmental influence	4	10	8	$\bar{\sigma}_B = 145.1$	2.19	.9191 * 10^6	$\bar{\sigma}_B = 125.8$	5.12
				$\bar{\epsilon}_B = 0.017$	7.71		$\bar{\epsilon}_B = 0.010$	5.70
				$\bar{E} = 18510$	3.09		$\bar{E} = 20250$	5.97
Mechanical and environmental influence	6	10	8	$\bar{\sigma}_B = 128.2$	3.68	.0780 * 10^6		
				$\bar{\epsilon}_B = 0.010$	10.67			
				$\bar{E} = 20950$	3.32			
Surface treatment + + mechanical and environmental influence	6	10	8	$\bar{\sigma}_B = 148.4$	2.15	.7759 * 10^6		
				$\bar{\epsilon}_B = 0.017$	13.86			
				$\bar{E} = 20995$	4.41			
Environmental influence	6	10	8	$\bar{\sigma}_B = 136.1$	1.38	.3331 * 10^6		
				$\bar{\epsilon}_B = 0.012$	7.97			
				$\bar{E} = 20847$	2.30			

Table 1. Static and dynamic properties of $[\pm 45]_{S8}$ graphite/epoxy Thornell 300/Narmco 5208 obtained from tests
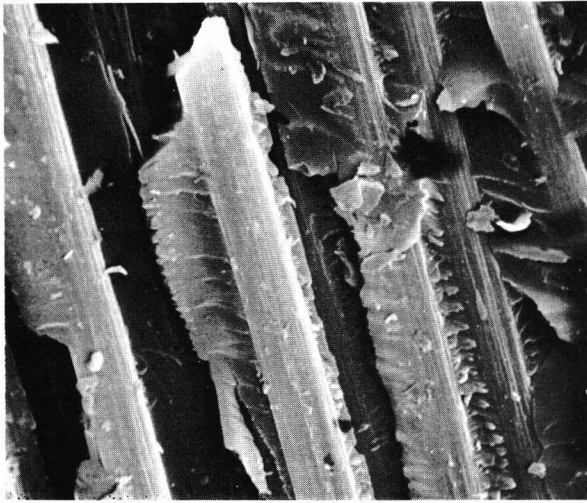
Figure 3. Electron microscope enlargement (750 times) of fracture surface taken from virgin specimens.
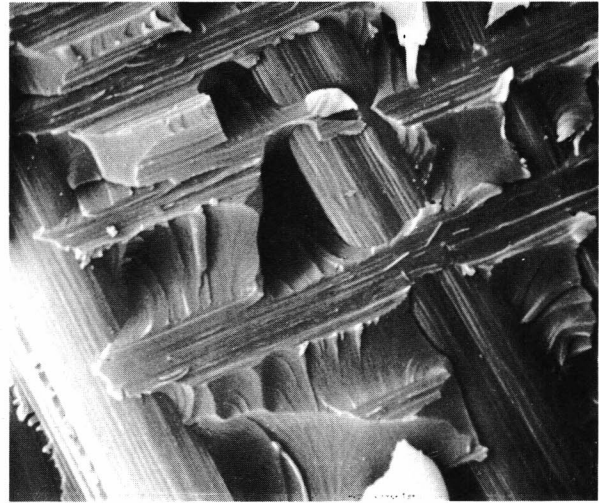
Figure 4. Electron microscope enlargement (1500 times) of fracture surface taken from specimen exposed for six weeks of environmental cycling. Note debondning between matrix and fibres.

Static and dynamic properties of $[0°/\pm45°/90°]$ Graphite/epoxy Thornell 300/Narmco 5208 are presented in table 2.

TYPE OF ENVIRONMENTAL AND MECHANICAL INFLUENCE	TIME OF EXPOSURE (WEEKS)	NUMBER OF SPECIMENS		STATIC TESTS		FATIGUE TESTS			
		Static	Dynamic	(N/mm^2)	S(%)	R = 0.1			R = -1
						N_{avg}	(N/mm^2)	S(%)	N_{avg}
Uninfluenced	0	8	8	$\bar{\sigma}_B = 483.7$ $\bar{\epsilon}_B = 0.0095$ $\bar{E} = 56031$	3.7 6.6 2.6	10^6	$\bar{\sigma}_B = 455.6$ $\bar{\epsilon}_B = 0.0086$ $\bar{E} = 55367$	5.4 9.8 5.8	54.600
Mechanical and environmental influence	2	8	8	$\bar{\sigma}_B = 491.9$ $\bar{\epsilon}_B = 0.0094$ $\bar{E} = 55840$	5.3 7.0 1.7	10^6	$\bar{\sigma}_B = 438.0$ $\bar{\epsilon}_B = 0.0095$ $\bar{E} = 54950$	13.5 19.3 3.4	171.500
Mechanical and environmental influence	4	8	8	$\bar{\sigma}_B = 519.3$ $\bar{\epsilon}_B = 0.0101$ $\bar{E} = 55715$	6.6 6.2 3.8	10^6	$\bar{\sigma}_B = 445.9$ $\bar{\epsilon}_B = 0.0034$ $\bar{E} = 54082$	7.3 9.1 1.9	120.000
Mechanical and environmental influence	6	8	8	$\bar{\sigma}_B = 494.4$ $\bar{\epsilon}_B = 0.0095$ $\bar{E} = 55353$	5.6 5.6 2.4	10^6	$\bar{\sigma}_B = 476.6$ $\bar{\epsilon}_B = 0.0088$ $\bar{E} = 55559$	7.1 8.2 1.5	147.000
Surface treatment + + mechanical and environmental influence	6	8	8	$\bar{\sigma}_B = 524.2$ $\bar{\epsilon}_B = 0.0097$ $\bar{E} = 54741$	4.9 4.5 1.4	10^6	$\bar{\sigma}_B = 458.8$ $\bar{\epsilon}_B = 0.0085$ $\bar{E} = 54977$	9.4 10.5 3.3	176.000
Environmental influence	6	8	8	$\bar{\sigma}_B = 512.1$ $\bar{\epsilon}_B = 0.0095$ $\bar{E} = 56737$	5.5 4.6 1.4	10^6	$\bar{\sigma}_B = 501.8$ $\bar{\epsilon}_B = 0.0096$ $\bar{E} = 55494$	5.0 5.6 1.8	157.000

Table 2. Static and dynamic $[0°/\pm45°/90°]_{S8}$ Graphite/epoxy Thornell 300/5208 obtained from tests

The mechanical properties of the $[0^{\circ}/\pm 45^{\circ}/90^{\circ}]_{S8}$ laminate seem to be rather uninfluenced by the relatively moderate environmental cycle used in this part of the investigation. Small variations in static strength properties were measured but the standard deviations are too high for making any obvious conclusions. Fatigue properties seem to have been positively influenced by the environmental exposure. As found in the first part of the investigation, the matrix becomes more ductile and is therfore less crack sensitive. Another explanation for the fatigue results could be the theories discussed by Hahn [2] and Herkovich [3]. They say that residual stresses induced during the fabrication could be released during the environmental exposure.

III   Experimental procedure

The test procedures in the two parts of the investigation are very much alike. It is only the laminate and the environmental cycle that has been changed and that the second part is also including fatigue tests with R = -1.

All specimens were exposed to the environmental cycle 5 days a week, the other two days they were kept in the constant-temperature/constant-humidity conditions defined be step 1 in the environmental cycles. The weights of the specimens were registrated before and after step 1.

In part 1 where the harder environmental cycle (figure 1) was used the level of equilibrium was around 1% (See figure 5) for the uncoated specimens. The polyurethane covered specimens absorbed more moisture during the first weeks and almost all water was dried out during the temperature spike. Probably the water was only absorbed in the polyurethane cover and diffused later into the specimens.
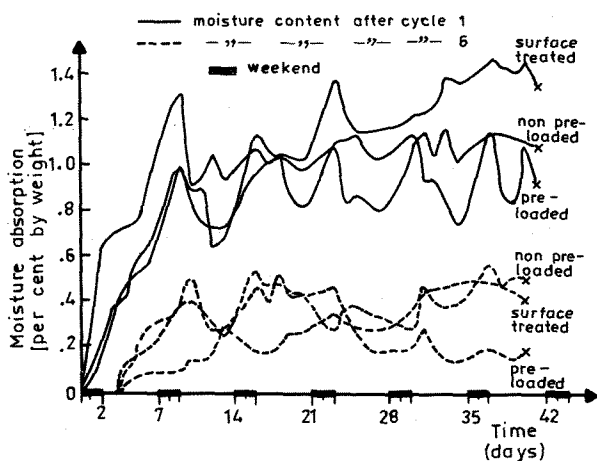

Figure 5. Moisture absorption in pre-loaded, not pre-loaded and surface treated specimens of $[\pm 45^{\circ}]_{S8}$ laminate

In the second part of the investigation the level of equilibrium was lower, about 0.5 % for the uncoated specimens (See figure 6). That was because of the less hard environmental cycle used (See figure 2). The surface treated specimens absorbed less moisture than the other groups and reached equilibrium at 0.25 % after a few days of exposure. The surface treatment for the $[0^{\circ}/\pm 45^{\circ}/90^{\circ}]$ specimens consisted of an epoxy sealing wax.
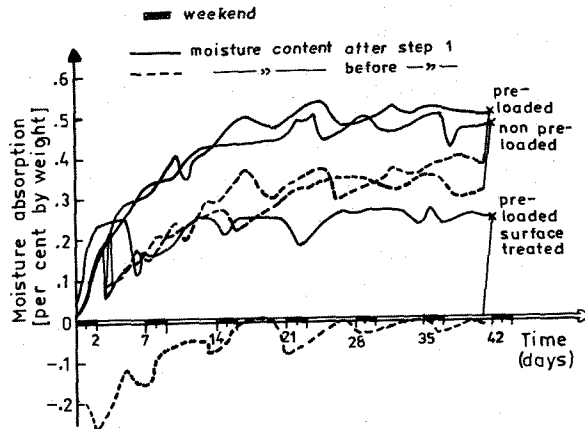

Figure 6. Moisture absorption in preloaded and non preloaded and surface treaded specimens of $[0^{\circ}/\pm 45^{\circ}/90^{\circ}]_{S8}$ laminate

No significant differens in moisture absorption between preloaded and non-preloaded laminates could be noticed in any of the two parts of the investigation.

After testing for environmental influence had been completed static strength, stiffness and fatigue strength were tested, these tests were also conducted on an uninfluenced group.

To measure strains in test specimens during static tests, foil strain gauges were applied with an isocyanate M-BOND 200 to the static test specimens. Load-strain curves registered in this test were used for determination of ultimate stress, ultimate strain and initial stiffness. The rate of loading was 0.015mm/mm min for all static tests.

Stiffness was also measured before the environmental exposure at all specimens, except for the group of specimens which were not supposed to be pre-loaded.

The fatigue tests with R = 0.1 were performed in a FFA pulsator machine. The specimens were fastened with special holders for composite materials. The freqency was 1700 load cycles per minute.

The fatigue tests with R = -1 were performed on test beams subjected to four point bending in a specially developed testing machine for constant amplitude fatigue (See figure 7). The amplitude in these tests was 7 mm, and the frequency was 500 load cycles per minute.

Maximum stress σ_{max} was for all fatigue tests chosen to $\sigma_{u/2}$ where σ_u (ultimate stress) was determined from the reference group of uninfluenced specimens.
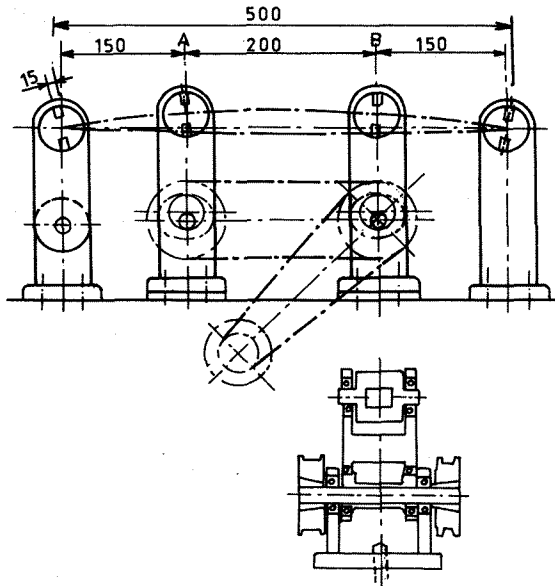
Figure 7
Principal drawing of the fatigue testing machine used for combined tension/compression (R = -1).

In the static tests and in the fatigue tests (R = 0.1) a 24 mm wide and 230 mm long specimen with straight sides was used. These specimens were provided with glass-fibre stiffeners at the ends, See figure 8. The glass fibre stiffeners were bonded with Araldite AW 106/HU-953 epoxy.
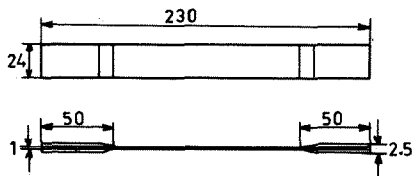
Figure 8
Test specimen for the static and fatigue tests (R = 0.1).

In the fatigue tests with stress ratio R = -1 a 510 mm long and 30 mm wide test beam was used, figure 9. The core was aluminium honeycomb , the upper face was a CFRP-laminate and the lower was 3 mm glass-fibre.
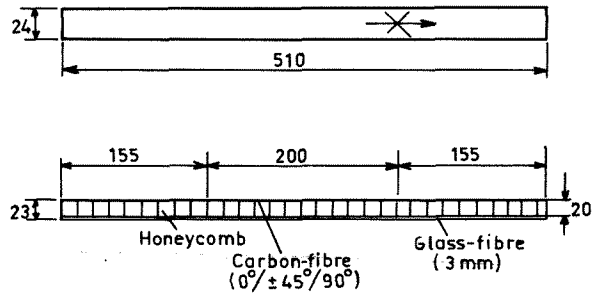
Figure 9.
Test beam used for fatigue tests (R = -1).

IV Test results

STATIC TEST $[\pm 45^\circ]_{S8}$ -laminate

The test results for the control group of specimens, which were not exposed to environmental conditions, are shown in Tabel 1. The ultimate stress was determined to σ_u = 157 N/mm^2, the ultimate strain ϵ_u = 0.011 and the initial stiffness E_{xo} = 22.640 N/mm^2.

After two weeks of exposure in the environmental cycle, the ultimate stress is unaffected by the exposure. The material is, however, more ductile, the ultimate strain has increased by 56 percent to ϵ_u = 0.017, the initial stiffness has decreased by 14 percent.

One more week of exposure decreases the ultimate stress by seven percent, while the ultimate strain remains constant, ϵ_u = 0.017. The stiffness reduction is 19 percent, which is five percent more than the "two weeks" group.

After six weeks of exposure in the environmental cycle, the ultimate stress has decreased by 19 percent compared with the unaffected specimens. The material is no longer as ductile as the "two- and four-week groups", ultimate strain ϵ_u = 0.011. The stiffness reduction is about 10 percent.

STATIC TESTS $[0^\circ/\pm 45^\circ/90^\circ]_{S8}$ -laminate

The results of the control group of specimens which are not exposed to environmental exposure are shown in table 1. The ultimate stress was determined σ_u = 484 N/mm^2, the ultimate strain ϵ_u = 0.0096 and the modulus E_{xo} = 56.031 N/mm^2.

After two, four and six weeks, the ultimate stress is still around 500 N/mm^2 at room temperature and the modulus is around 55 000 N/mm^2. The ultimate stress for the dried specimens is around 420 N/mm^2. Any significant difference in strength properties between the preloaded and the non preloaded specimens cannot be noticed.

The elastic modulus differs very little between different test groups, it is around 55 000 N/mm^2 for all the groups.

All changes in static strength properties are within the standard deviation, in the tests carried out at room temperature.

FATIGUE TESTS of $[\pm 45^{\circ}]_{S8}$-laminates

Test results from the fatigue of the specimens uninfluenced by mechanical and environmental effects are shown in Table 1. All specimens survived 10^6 load cycles and were used to determine residual strength. The static-strength properties were unaffected by the fatigue, only stiffness was affected negatively. The standard deviations are one or two percent higher for both σ_u and E, compared with the corresponding static tests.

After two weeks of exposure to environmental cycling, the fatigue properties seem to have been reduced; only three specimens survived 10^6 load cycles and they showed reduced properties in the static tests. The results from this test group are not significant since trouble with the fatigue machine caused the sudden death of two specimens.

Five of the eight specimens exposed to four weeks of environmental cycling survived 10^6 load cycles. The retaining tensile strength is 19 percent lower than for the corresponding uninfluenced group.

Two weeks further exposure reduced the fatigue in a very hard way. None of the specimens survived more than $8.7 \cdot 10^4$ load cycles.

Almost the same results were obtained for the corresponding "non pre-loaded group" The specimens lived for between $1-5 \cdot 10^4$ load cycles.

The group of specimens covered with polyurethane show some better fatigue properties compared with the other two groups exposed for six weeks, see Table 1.

Clearly, the fatigue properties are affected only after prolonged environmental cycling (more than two weeks). The degradation is very strong for long-term exposure. One can note the correspondence in time between debonding between fibre-matrix and degradation in fatigue properties, see figure 4.

The polyurethane-covered specimens exposed for six weeks show about the same mechanical properties as the group exposed for two weeks. The surface treatment seems to delay the influence of moisture on the mechanical properties of the composite.

The results of static tests on non pre-loaded specimens exposed to environmental cycling for six weeks show that the ultimate stress is about sex percent higher than for the pre-loaded specimens exposed for six weeks. The difference in ultimate strain is within the limits of the standard deviation. The stiffness shows about the same value as the pre-loaded group.

FATIGUE TESTS of $[0^{\circ}/\pm 45^{\circ}/90^{\circ}]_{S8}$-laminates

In the fatigue tests with the ratio of lower to higher stress level R = 0.1 and σ_{max} =242 N/mm^2 none of the specimens failed. All specimens survived 10^6 load cycles and were therefore tested in static tension after the fatigue test.

In the uninfluenced groups one could observe small cracks in the 90° layers after $2 \cdot 10^5$ load cycles. The cracks started at the free edges and grew towards the middle of the specimens. A delamination occurred at about a fourth of the specimen area. The effect on the residual strength was not yet so obvious, a five percent degradation was noted.

At the other test group which were exposed to the environmental cycle no cracks were visible. The residual strength was nevertheless reduced by 2-14 percent. There is no great difference in residual strength between the groups exposed to environmental cycling for different time.

The results from the fatigue tests with the stress ratio R = -1 showed that the crack appearing between the layers showed to be of a more serious nature because of the compressive loading. The cracks grew fast and within a few thousand cycles the layers outside the cracks buckled out, see figure 10.
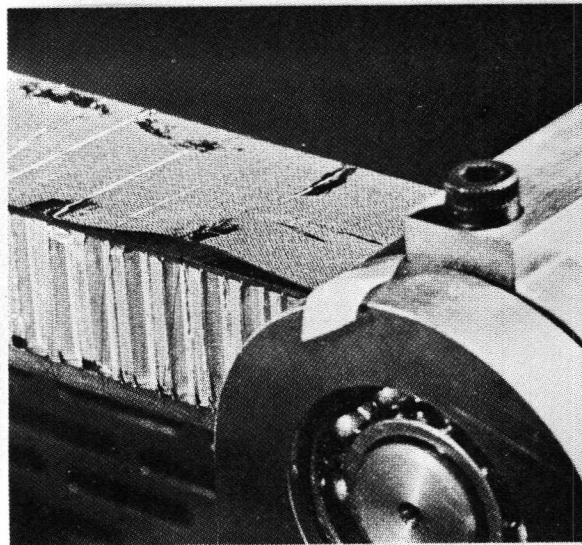

Figure 10 Fracture of sandwich beam after fatigue tests with the load ratio R = -1.

The test of the uninfluenced group showed the
the poorest fatigue properties. The layer
buckling occured after about $5 \cdot 10^4$ load cycles.
In the groups exposed to environmental cycling,
buckling occured after $1.2 - 1.8 \cdot 10^5$ load
cycles.

Interlaminar stresses in composites are in-
duced during fabrication and by environmental
exposure. Curing stresses in the transverse
direction can be higher than 50 % of the
transverse strenght [2]. Moisture absorption
under room temperature conditions can render
laminates free of residual stresses [2].

Tension loading on the laminate used in this
investigation causes interlaminar stresses
which tend to delaminate the laminate for
this stacking sequence (Poisson's mismatch).
The stresses due to thermal mismatch may be of
the same sign as those due to Poisson's mis-
match or they may be of opposite sign depending
upon material properties, stacking sequence
and direction of loading [3]. In the present
investigation these stresses are of the same
sign during tensile loading. This might
explain the difference in fatigue strength
between the untreated and the specimens ex-
posed to environmental cycling.

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