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ABSTRACT

The effects of moisture and temperature on unidirectional and multi-ply laminates of T300/934 and AS/3501 graphite-epoxy systems were investigated. Properties studied were static flexure strength, and flexure and torsion fatigue strengths at room temperature and at 74° C. Specimens with increased moisture content showed a reduced static flexure strength; water as the test environment had only a negligible influence. In flexure fatigue and torsion fatigue, the water environment caused somewhat reduced fatigue strengths at room temperature and significantly greater degradation in 74° C water. The failure mode in all cases was interlaminar delamination.

INTRODUCTION

Graphite-epoxy composites are receiving ever increasing attention for aerospace vehicle and automotive applications. This increasing interest has brought heightened concern regarding possible critical effects of environment, particularly that of moisture combined with high temperatures, which could significantly degrade the attractive mechanical properties of these materials. If the materials are to achieve their high potential for such applications, it must be demonstrated that the mechanical properties of the materials are reasonably predictable and that the properties are maintained over long periods under expected conditions of use. The influence of moisture and temperature on static mechanical properties of epoxy-matrix composites has been the subject of several investigations over the past few years. Results of these studies — summarized by Ramani and Nelson (1)[1] — have established that matrix-controlled properties, including flexure, are degraded by absorbed moisture, primarily as the result of plasticization of the resin matrix. In contrast, information on the effect of environment on fatigue properties is limited. Our previous reported work has shown that graphite-epoxy composites are susceptible to increasing flexure and torsion fatigue damage in a water environment relative to an ambient air environment (2,3).

The objective of this investigation was to determine the effects of temperature and of moisture (both in the environment and in the laminate) on static and cyclic flexure and torsion properties of representative widely used graphite-epoxy composites. Flexure strength and torsion and flexure fatigue properties were studied using T300/934 and AS/3501 graphite-epoxy composite systems of two fiber orientations — $(0°)_{16}$ and $(0°/\pm45°/0°)_4$. The effects of temperature and of moisture on the laminates were evaluated by comparing baseline ambient mechanical properties with corresponding properties of specimens tested at two temperatures and two initial moisture conditions in air and in water environments.

EXPERIMENTAL PROCEDURES

The two composite materials used in this investigation were Union Carbide T300 fiber in Fiberite 934 resin containing 61 ±2% fiber volume, and Hercules

[1]Numbers in parentheses designate references listed at the end of the paper.

AS fiber in 3501 resin containing 63 ±2% fiber volume. Fiber orientations studied were unidirectional, $(0°)_{16}$, and multi-ply, $(0°/\pm45°/0°)_4$. Prepreg properties for T300/934, as given by the producer, were: UTS = 2.68 GPa, modulus = 228.3 GPa, and density = 1.603 g/cc. For AS/3501, the properties were: UTS = 3.15 GPa, modulus = 222.1 GPa, and density = 1.803 g/cc. Void content was negligible (<0.5%) for both materials. The composite laminates were prepared by Lockheed Missiles and Space Co., Sunnyvale, Calif., in sheets that were 2 mm thick (nominal). A diamond saw was used to cut the sheets into specimens 100-mm long and 12.5-mm wide (both bend and torsion specimens).

The static bend tests and the flexural fatigue tests were run using an electrohydraulic, servo-controlled test sytsem having a linear actuator. A four-point test fixture, which kept the specimen in a fixed position by clamping the ends to the span supports of the fixture, was used. Clamped specimen ends were required for the fully reverse bending fatigue tests and were used on all tests so that data from both static and fatigue tests could be more reasonably compared. An analysis by Johnson (4) has shown that bend tests of end-constrained specimens give higher values than unconstrained specimens for transversely isotropic materials. This is consistent with the observations of the present study in which flexure strength values for clamped specimens were about 10% greater than those for unclamped specimens.

Fatigue tests were performed in fully reverse bending with the ratio of minimum-to-maximum stress (R) equal to -1. Fatigue tests were conducted at a frequency of 20 Hz, the alternating load being kept constant. Fatigue damage accumulation was determined by an increase in specimen deflection. Load and deflection were both recorded periodically on a high-speed, strip-chart recorder, or recorded with an electronic data acquisition system by which the load-deflection or flexural compliance relationship could be continuously monitored, stored, and plotted. Deflection limit detectors were set to terminate the test at fracture, or when the specimen deflection exceeded the initial value by any preselected amount. A 20% increase in deflection was the criterion for failure and the end point of most tests.

Torsion tests were run under conditions of controlled deflection (angle of twist) using an electrohydraulic, servo-controlled test system having a rotary actuator. A torsional force was applied at one end of the specimen, and the torque was measured by the output of a strain-gaged torque cell at the other end of the specimen. The output of both the torque cell and the potentiometer measuring the angle of twist was fed to an X-Y recorder. The gauge length between the grips was 53.1 mm for all tests. The shear modulus was determined from the angle of twist and resulting torque using the equations for a linearly elastic, orthotropic, rectangular parallel piped (5,6).

Torsional fatigue tests were conducted at a frequency of about 1 Hz at constant angle of twist. The cyclic deflection was set at the beginning of the test ($\pm$ angle θ determined by the initial stress τ_0), and was maintained constant thereafter. The initially applied torque stress was about 40% of the static failure strength of the laminate. The fatigue tests were run until the torque had decreased to the predetermined level of 80% of the initial torque.

Specimens were tested at two moisture contents: as-received and after immersion in room temperature (23° C) water for at least 180 days ("presoaked"). This minimum presoaking time was determined by the moisture absorption curves for T300/934 $(0°)_{16}$ in 23° C and 74° C water (Fig. 1) which show maximum water absorption (saturation) at both temperatures at approximately 160 days. Also, data reported by McKague, Reynolds, and Halkias (7) show maximum absorption of water by similar laminates after approximately 180 days in an environment of 98% relative humidity (RH). Moisture content of specimens was determined by desorbing the moisture in a vacuum desiccator at 100° C until a constant weight was obtained. The moisture contents of the as-received composite sheets were 0.6% for both the T300/934 and the AS/3501 systems in both laminate configurations. Immersion in room temperature water for 180 days increased the moisture content to 1.4% for both laminates of both systems. Figure 2 shows the typical vacuum desorption curve for as-received and pre-soaked $(0°/\pm45°/0°)_4$ T300/934 specimens. The initial rate at which water was absorbed by the multi-ply layups was greater than for the uniaxial layups; however, this had no discernible effect on the total amount of water absorbed.

Both flexure and torsion fatigue tests were performed in the following environments: room-temperature air (23° C at 40–50% RH), room-temperature water, 74° C air, and 74° C water. (The test temperature of 74° C was chosen because it is considered to be the maximum temperature attained by structural components of commercial aircraft.) A Statham temperature-controlled oven was used to provide necessary temperatures. Water was contained in a vessel surrounding the immersed specimen. As previously stated, it was established that the equilibrium moisture absorption limit at 74° C was essentially identical to that of 23° C. Therefore, moisture preconditioned specimens at 23° C would not be expected to gain additional water while immersed for testing at 74° C.

RESULTS AND DISCUSSION

Static Flexure Tests

The effect of moisture on the flexure strength of T300/934 and AS/3501 graphite/epoxy laminates having fiber orientations of $(0°)_{16}$ and $(0°/\pm45°/0°)_4$ are shown in Table 1. As shown in the table, presoaked specimens (1.4% moisture) exhibit lower flexure strengths than the as-received specimens (0.6% moisture) in all three of the environments investigated. Specifically, for T300/934 the flexure strength of the $(0°)_{16}$ laminate was reduced about 12% as a result of the pre-soak treatment and the $(0°/\pm45°/0°)_4$ was reduced about 7%; similar effects for AS/3501 are shown. There appeared to be little or no influence of the test environment when the tests were conducted at room temperature; the as-received specimens and the presoaked specimens exhibited similar flexure strengths in both room-temperature air and room-temperature water. Flexure strength of T300/934 in 74° C water was reduced approximately 18%, however, for the $(0°)_{16}$ laminate and 9% for the $(0°/\pm45°/0°)_4$ laminate compared with room temperature strength for both the as-received and presoaked specimens, with similar effects noted for AS/3501. Augl and Berger (8) showed that all composites tested in their investigation (uniaxial) lost flexure strength after moisture exposure, but no significant changes in strength values were observed after thermal cycling between room temperature and 150° C. Browning (9) showed that flexure strength of composite test specimens decreased with increased moisture content on the basis of equivalent water weight gains, whether from water-boil exposure or exposure to a high-humidity environment. Additionally, he reported that mechanical properties of the composites tested were essentially unaffected by temperatures to 120° C. From the above, it appears that the increased degradation effect of water at 74° C compared to 23° C is the result not of increased moisture content but of increased temperature, concomitant greater plasticizing of the epoxy matrix at the higher temperature, and closer approach to the glass transition temperature of the laminate.

Flexure Fatigue Tests

The results of flexure fatigue tests conducted on T300/934 graphite-epoxy laminates in fully reverse bending ($R = -1$) are summarized in Figs. 3 and 4 for the $(0°)_{16}$ laminate and the $(0°/\pm45°/0°)_4$ laminate, respectively. All specimens were in the as-received condition. As can be seen from these figures, the fatigue strengths of both laminates were significantly lower when they were tested in water at room temperature than they were when the laminates were tested in air. This, of course, differs from what was observed in the static flexure tests in which room-temperature water was found to have little influence on the flexure strength (Table 1). Additionally, 74° C water was found to further reduce the fatigue strength of both laminates (Figs. 3 and 4). One series of tests was conducted on pre-soaked specimens of the $(0°)_{16}$ laminate tested in room-temperature water. The results of those tests are summarized in Fig. 5 and are compared to the results on as-received specimens tested in the same environment. As can be seen from Fig. 5, presoaking had little or no influence on the moisture-induced degradation of fatigue strength. Again, this differs from what was observed in the static flexure tests in which presoaking was found to significantly reduce the flexure strength compared with as-received specimens (Table 1). These results are in agreement with our previously reported work which showed greater degradation in fatigue properties than in static properties as a result of exposure to a moisture environment. Results of axial fatigue testing of GE composites by Lundemo and Thor (10) show similarly increased degradation if specimens are preloaded prior to exposure.

A comparison of the flexure fatigue results and the static flexure results suggests that moisture absorption is enhanced during cyclic loading. Sufficient moisture is absorbed during the flexure fatigue test of an as-received specimen (0.6% initial moisture content) to cause degradation in fatigue strength equivalent to a fully saturated specimen at room temperature (1.4% moisture). Again, the additional degradation observed in 74° C water is attributed not to increased moisture content in the specimen but to increased plasticization of the matrix resulting from this higher test temperature, and the closer approach to the glass transition temperature of the laminate.

Torsional Fatigue Tests

For a comparison of the observed influences of moisture on the flexure fatigue strength of the T300/934 and AS/3501 graphite-epoxy systems, torsional fatigue tests were conducted on similar unidirectional and angle-ply laminates of the AS/3501 graphite-epoxy system. Here, the tests were run at a constant angle of cyclic deflection, and failure was taken as the number of cycles at which the torsional shear load had decreased to 80% of its initial value. The results of the fully reverse (R = -1) torsion fatigue tests are summarized in Figs. 6 and 7 for the $(0°)_{16}$ and $(0°/\pm45°/0°)_4$ laminates, respectively. In these tests, the environments were room-temperature air and water and 74° C water as well as 74° C air using only as-received specimens (0.6% moisture). Tests were also conducted on presoaked specimens but the results are not shown in these figures because presoaked specimens were found to behave similarly to the as-received specimens — comparable to the results from the flexure fatigue tests. As can be seen from these figures, the environmental influences on torsional fatigue strengths were, in general, similar to but somewhat greater than those observed for the flexure fatigue on the T300/934 system (Figs. 3 and 4). The torsion fatigue strengths of specimens tested in 74° C air were lower than those tested in room-temperature air. Torsion fatigue strengths of laminates tested in 74° C water were lower than those tested in room-temperature water or 74° C air. These results support the explanation previously given that at the higher temperature increased plasticization of the matrix and closer approach to the glass transition temperature cause greater degradation of the laminate fatigue strength.

Mode of Failure

Both flexure and torsion fatigue specimens were analyzed to establish the primary mode of cracking. Interlaminar delamination was found to be the primary mode of failure for both types of tests in both unidirectional and angle-ply laminates of both the T300/934 and AS/3501 graphite-epoxy systems. Because failure was defined as a degradation of strength or load-carrying ability of the specimens and not complete separation, transverse cracks were not observed. The surface appearance of the specimens, typical of all specimens, is shown in Fig. 8, which is a scanning electron micrograph showing numerous delaminations.

A few specimens were cross sectioned in order to establish the form of cracking in the interior of the specimens. Figure 9 is a scanning electron micrograph of a cross section of an AS/3501, unidirectional, torsion fatigue specimen which had been tested in a 74° C water environment under an initial torque equal to 60% of its fracture stress. Failure in this specimen occurred by delamination (as with other specimens) and no transverse cracking was visible at the edges. In the cross-section view, however, fiber-matrix "interfacial" cracking is seen to have occurred — not at the interface itself, but in the fiber periphery close to the interface indicating excellent interfacial bonding between fiber and matrix.

SUMMARY OF RESULTS AND CONCLUSIONS

The influences of temperature and of moisture, both in the environment and in the laminate, were investigated in unidirection and multi-ply laminates of T300/934 and AS/3501 graphite-epoxy systems. Properties studied were static flexure strength, and flexure and torsion fatigue strengths at room temperature and at 74° C. Specimens were tested in two conditions: as-received, containing 0.6% moisture, and presoaked, containing 1.4% moisture. Major results and conclusions are:

1. Static flexure strength of the laminates was reduced by higher test temperature and increased moisture content (presoaking of specimens); water at room temperature had negligible effect.

2. Flexure and torsion fatigue strengths were significantly reduced by increased test temperature and by the water test environment. Higher temperature of the water test environment resulted in further degradation of the fatigue strength.

3. Increased degradation caused by water at higher temperature is attributed to increased plasticizing of the epoxy matrix by water at the higher temperature, and by the closer approach to the glass transition temperature.

4. Cyclic stress appears to accelerate water absorption.

5. In evaluating composite laminates for aerospace or automotive structural applications, moisture content of the laminate and the effects of temperature and moisture in the environment of use are critical factors.

6. The effect of cyclic stress on moisture absorption by graphite-epoxy laminates may be a critical factor in their application. We are continuing investigation of this effect and correlation of increased moisture content with degradation of mechanical properties.

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soaked laminate specimens in room temperature air, room temperature water, and 74° C water.

Test environment	Maximum stress, S_{max}, MPa			
	Uniaxial, $(0°)_{16}$		Multi-ply $(0°/\pm45°/0°)_4$	
	As-received[a]	Presoaked[a]	As-received[a]	Presoaked[a]
T300/934				
Room temp. air	2150	1890 (-12%)	1380	1280 (-7%)
Room temp. water	2140	1910 (-11%)	1390	1280 (-7%)
74° C water	1760 (-18%)	1590 (-26%)	1260 (-9%)	1170 (-16%)
AS/3501				
Room temp. air	2280	2020 (-11%)	1100	1030 (-6%)
Room temp. water	2300	2010 (-12%)	1090	1040 (-6%)
74° C water	2020 (-11%)	1930 (-15%)	1050 (-5)	1010 (-8%)

[a]Figures in parentheses give percent degradation below room temperature air baseline values.

Figures

Fig. 1 Moisture absorption by T300/934 $(0°)_{16}$ composite; full immersion in 74° C and in room-temperature water.

Fig. 2 Moisture content of T300/934 $(0°/\pm45°/0°)_4$ composite as-received and after 90-day immersion in room-temperature water; vacuum desorbed at 100° C.

Fig. 3 Flexure fatigue (R = -1) of T300/934 $(0°)_{16}$ composite as-received specimens tested in room-temperature air, room-temperature water, and 74° C water environments.

Fig. 4 Flexure fatigue (R = -1) of T300/934 $(0°/\pm45°/0°)_4$ composite as-received specimens tested in room-temperature air, room-temperature water, and 74° C water environments.

Fig. 5 Flexure fatigue (R = -1) of T300/934 $(0°)_{16}$ composite as-received specimens tested in room-temperature air, room-temperature water, and 74° C water; presoaked specimens tested in room temperature water.

Fig. 6 Torsion fatigue (R = -1) of $(0°)_{16}$ specimens as-received, tested in room-temperature air, room-temperature water, 74° C air, and 74° C water.

Fig. 7 Torsion fatigue (R = -1) of $(0°/\pm45°/0°)_4$ specimens as-received, tested in room-temperature air, room-temperature water, 74° C air, and 74° C water environments.

Fig. 8 Scanning electron micrograph of delaminations in AS/3501 $(0°/\pm45°/0°)_4$ composite specimen (unpolished) torsion fatigue tested in 74° C water.

Fig. 9 Scanning electron micrograph of cross section of AS/3501 $(0°)_{16}$ composite specimen torsion fatigue tested in 74° C water.

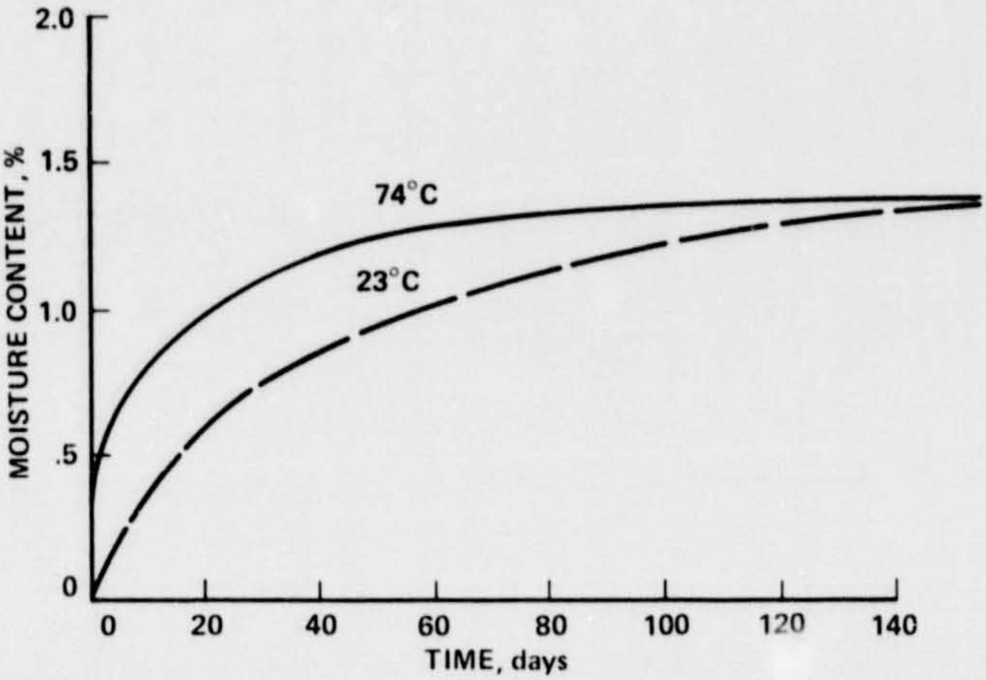
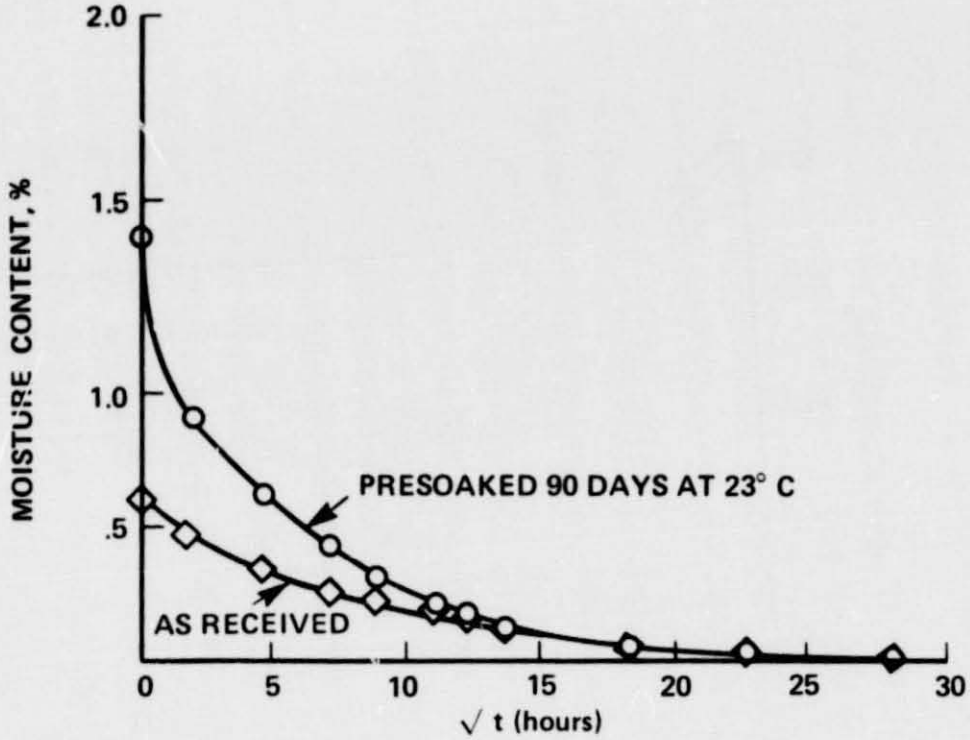

2.0
1.5
1.0
.5
0
MOISTURE CONTENT, %
74°C
23°C
0
20
40
60
80
100
120
140
TIME, days

Fig. 1



2.0
1.5
1.0
.5
MOISTURE CONTENT, %
PRESOAKED 90 DAYS AT 23° C
AS RECEIVED
0
5
10
15
20
25
30
√ t (hours)

Fig. 2

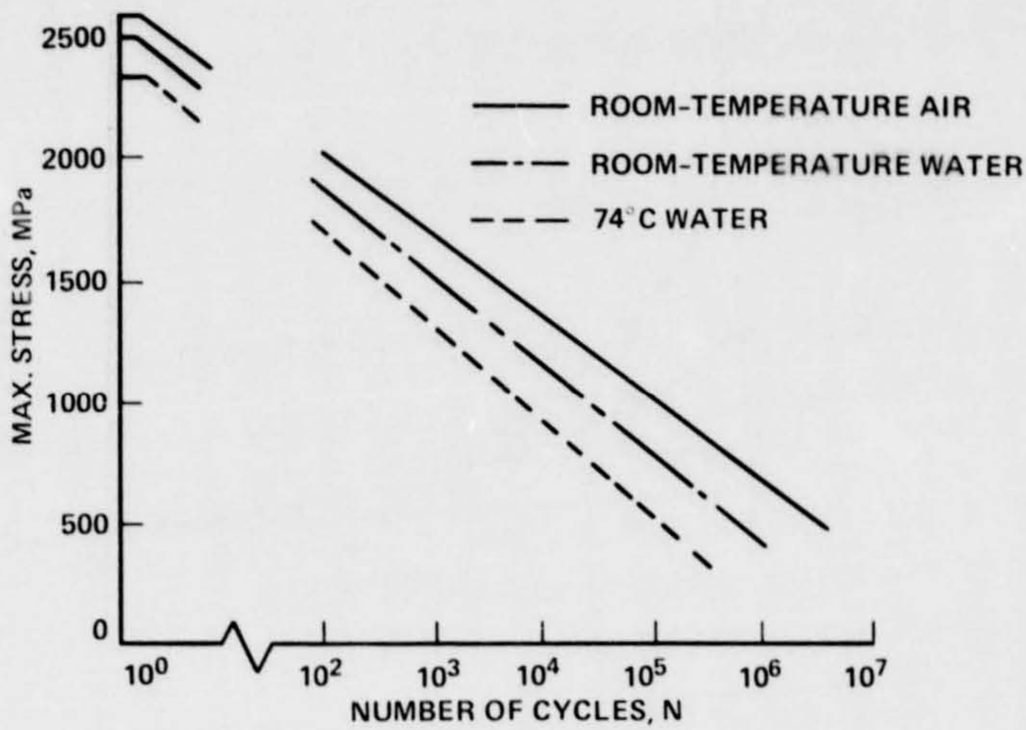

2500
2000
1500
1000
500
0
MAX. STRESS, MPa
ROOM-TEMPERATURE AIR
ROOM-TEMPERATURE WATER
74°C WATER
10^0
10^2
10^3
10^4
10^5
10^6
10^7
NUMBER OF CYCLES, N

Fig. 3

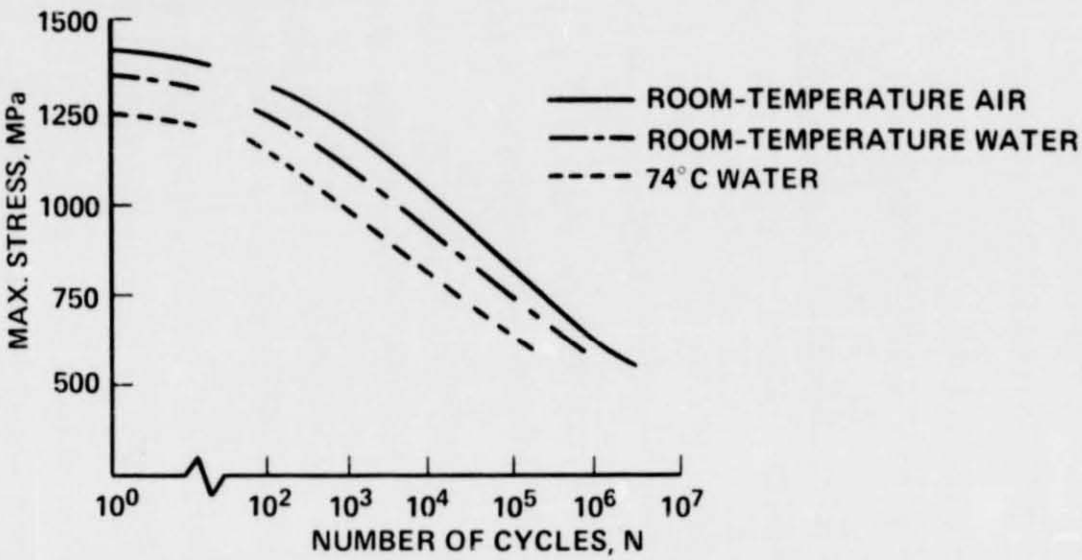

1500
1250
1000
750
500
MAX. STRESS, MPa
10^0
10^2
10^3
10^4
10^5
10^6
10^7
NUMBER OF CYCLES, N
ROOM-TEMPERATURE AIR
ROOM-TEMPERATURE WATER
74°C WATER

Fig. 4

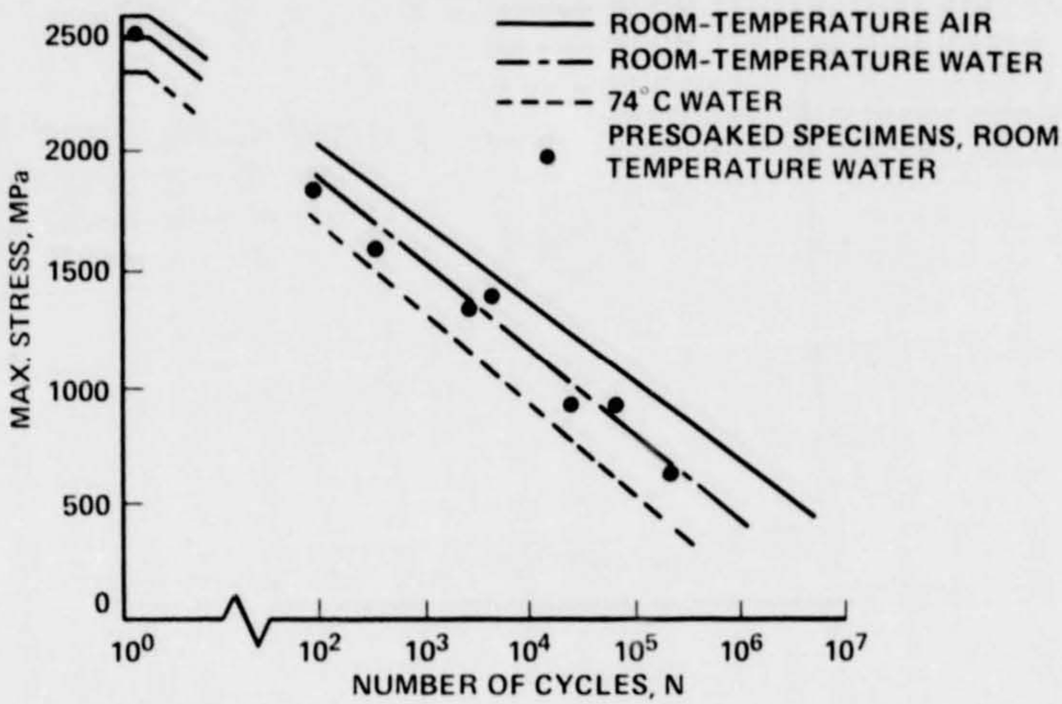
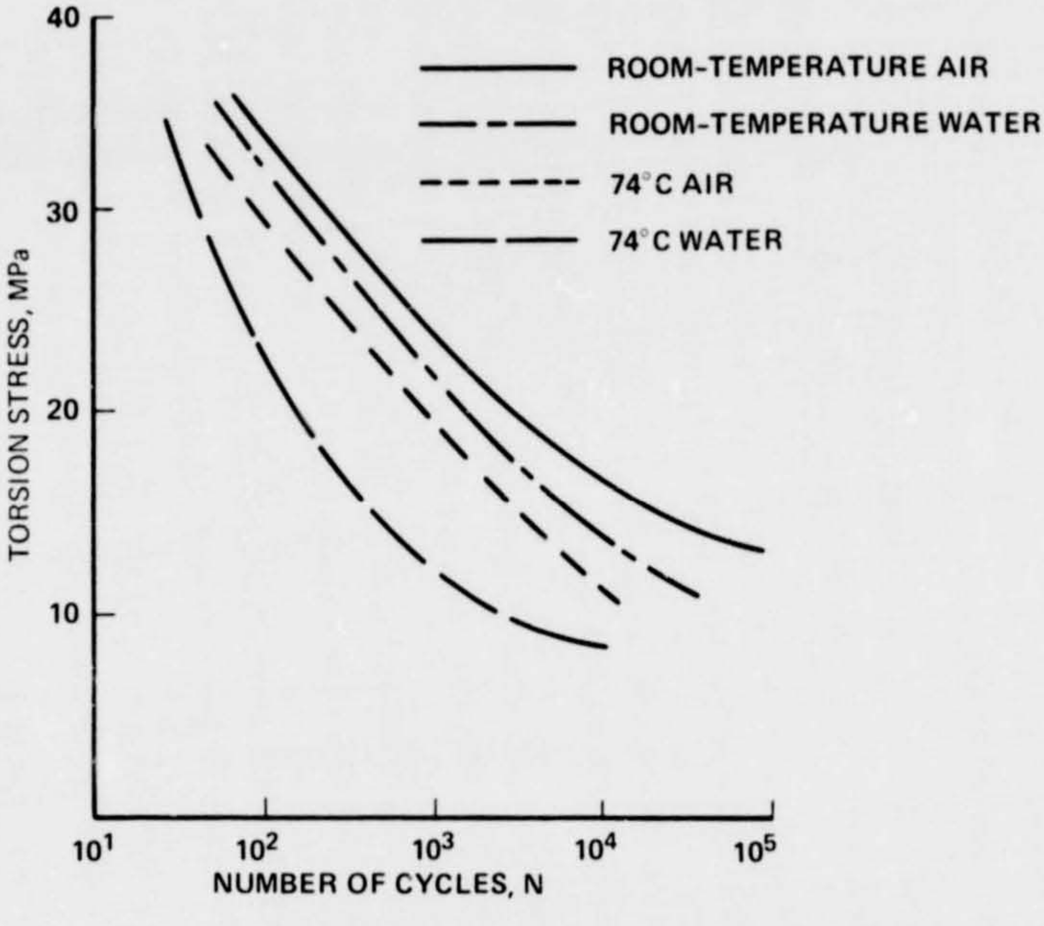

ROOM-TEMPERATURE AIR
ROOM-TEMPERATURE WATER
74°C WATER
PRESOAKED SPECIMENS, ROOM TEMPERATURE WATER
MAX. STRESS, MPa
2500
2000
1500
1000
500
0
10^0
10^2
10^3
10^4
10^5
10^6
10^7
NUMBER OF CYCLES, N

Fig. 5



ROOM-TEMPERATURE AIR
ROOM-TEMPERATURE WATER
74°C AIR
74°C WATER
TORSION STRESS, MPa
40
30
20
10
10¹
10²
10³
10⁴
10⁵
NUMBER OF CYCLES, N

Fig. 6

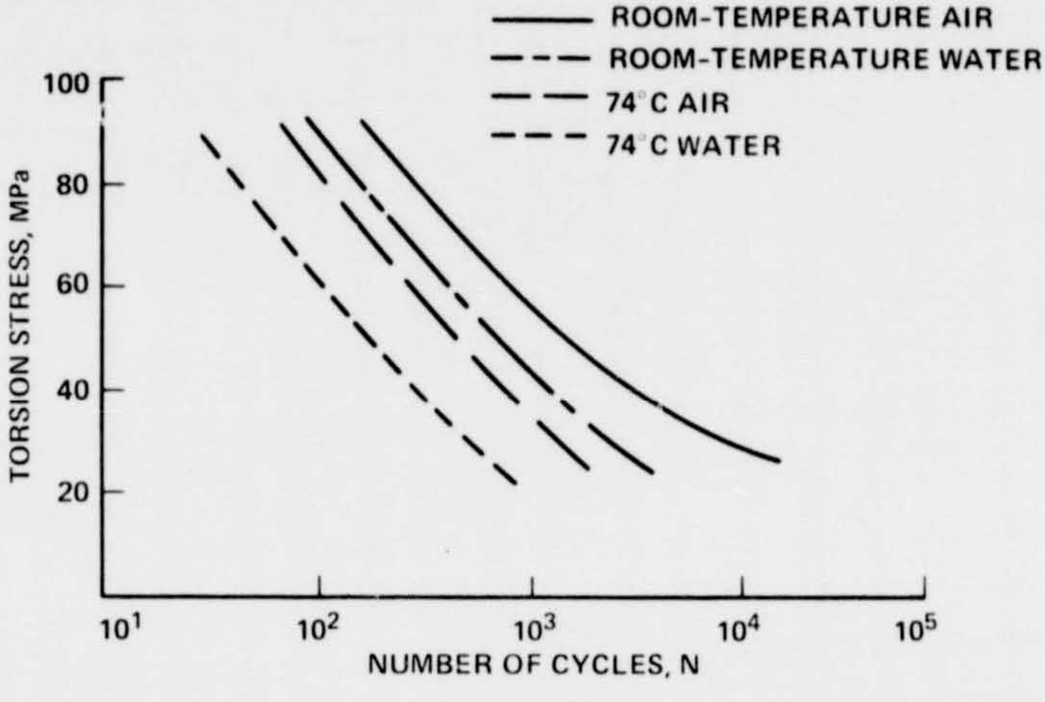

ROOM-TEMPERATURE AIR
ROOM-TEMPERATURE WATER
74°C AIR
74°C WATER
100
80
60
40
20
TORSION STRESS, MPa
10^1
10^2
10^3
10^4
10^5
NUMBER OF CYCLES, N

Fig. 7

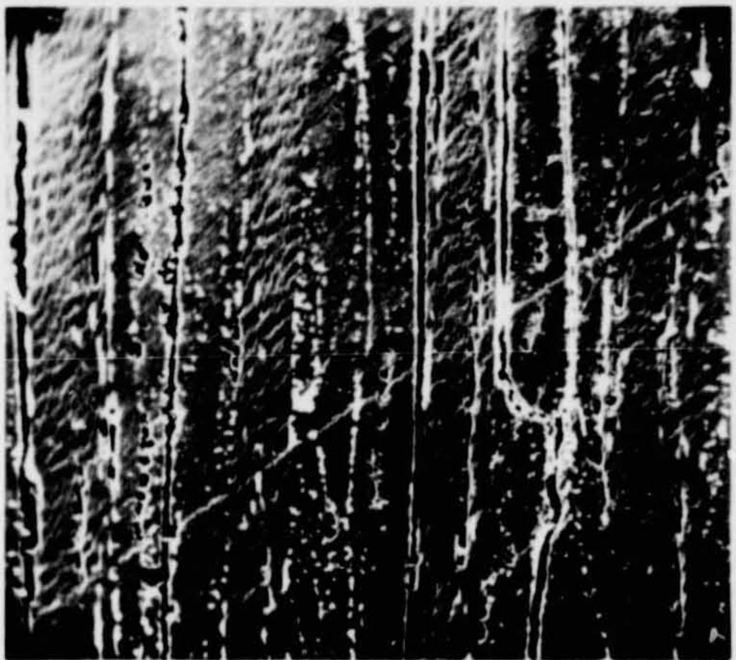


Fig. 8

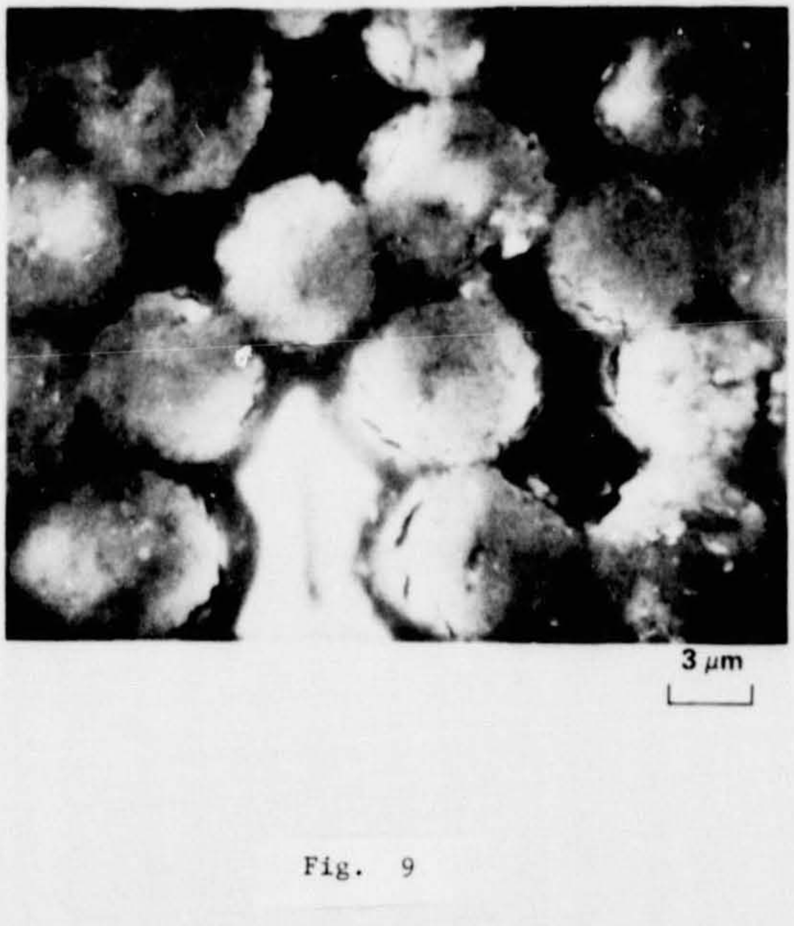


Fig. 9