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THE EFFECTS OF MOISTURE ON
THE PROPERTIES OF HIGH PERFORMANCE
STRUCTURAL RESINS AND COMPOSITES

CHARLES E. BROWNING

TECHNICAL REPORT AFML-TR-72-94

SEPTEMBER 1972

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THE EFFECTS OF MOISTURE ON THE PROPERTIES OF HIGH PERFORMANCE STRUCTURAL RESINS AND COMPOSITES

CHARLES E. BROWNING

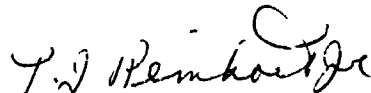
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FOREWORD

This report was prepared by Charles E. Browning, Plastics and Composites Branch, Nonmetallic Materials Division, and was initiated under Project Number 7340, "Nonmetallic and Composite Materials," Task Number 734003, "Structural Plastics and Composites." The work was administered by the Air Force Materials Laboratory, Air Force Systems Command, Wright-Patterson AFB, Ohio.

This report covers work conducted during the period of August 1970 through June 1971. The report was released by the author in July 1971.

This report has been reviewed and is approved.


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ABSTRACT

Graphite - and boron-fiber-reinforced composites, as well as castings of current resin systems, were evaluated to determine the effects of moisture and/or high humidity on their physical properties and their room and elevated temperature mechanical properties. Several of the resin systems investigated were not 350°F capability resins, even though they have been suggested for use in 350°F capability composites. All of the neat resin castings were found to absorb moisture and swell. Associated with moisture absorption is a loss in elevated temperature tensile strength, as demonstrated by the ERLA-4617 system which undergoes a precipitous loss of 350°F tensile strength as a result of ambient aging. All of the composite systems showed weight gains and thickness increases when subjected to a high humidity environment. However, the effect of absorbed moisture on the elevated temperature mechanical properties of composites is determined principally by the lay-up of the laminate and/or the test being applied, i.e., the method by which load is introduced into the laminate. This means that for a particular system, unidirectional composites may show a significant reduction of 350°F flexural strength due to absorbed moisture; however, for the same system, a multidirectional lay-up may show only a minor loss of 350°F tensile strength after equivalent moisture absorption (i.e., fiber-controlled-composite properties are relatively unaffected by absorbed moisture, whereas matrix-controlled properties are adversely affected). For both castings and composites the effects of moisture were found to be reversible.

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SECTION I

INTRODUCTION

High performance structural composites have progressed through several stages of development to where they are being readied for use in actual Air Force hardware. At this stage of their development, their performance during exposure to simulated aircraft environments must be evaluated. Environmental factors of major importance would include moisture (high humidity) and extremes in temperature. A major concern is retention of high-temperature composite properties after exposure to high humidity.

A program was undertaken to determine the effects of high humidity on the properties (both mechanical and physical) of high-performance composites. Graphite and boron-fiber-reinforced composites as well as cast resin systems were evaluated. Flexural, shear, tensile, and compressive properties were measured as a function of moisture, temperature, time of exposure to moisture, and number of exposure cycles. Quasi-isotropic laminates were studied because the critical design properties are the in-plane properties of multidirectional composites. Unidirectional properties were also obtained because certain ones (e.g., flex) are very sensitive to matrix properties. Physical parameters of weight gain and dimensional changes were also recorded.

The data are presented to show the effects of the environment on the properties of several current state-of-the-art composite and resin systems and one experimental composite system. No attempt is made to pinpoint the exact causes of moisture weight gains, thickness increases, or losses in strength. Such attempts will be the subject for future work.

SECTION II

EXPERIMENTAL PROCEDURES

1. MATERIALS SYSTEMS

The materials investigated in this program consisted of cast resins, boron-reinforced composites, and graphite-fiber reinforced composites. The cast resins investigated were the same types as those used as laminating resins in the composites.

Cast resins evaluated consisted of:

- a. NARMCO 2387 - The resin in NARMCO 5505 boron/epoxy prepreg.
- b. Union Carbide Corporation's (U.C.C.'s) ERL-2256 Epoxy Resin - Cured with U.C.C.'s ZZL-0820.
- c. U.C.C.'s ERLA-4617 Epoxy Resin - Cured with m-phenylenediamine (m-PDA).
- d. Shell's EPON 828 - Cured with m-PDA.
- e. U.C.C.'s X-2546 - A new high temperature epoxy containing U.C.C.'s ERLB-4617 Resin, ZXZL-5152 Hardener, and BF_3 .MEA Catalyst.

Boron reinforced composites were fabricated from NARMCO 5505 prepreg - prepreg tape consisting of boron fiber, NARMCO 2387 resin, and 104 glass fabric backing. Both postcured and nonpostcured laminates were considered.

All of the graphite-fiber reinforced composites contained Hercules' Type HT-S graphite fiber. The following graphite laminates were evaluated:

- a. HT-S Graphite and ERLA-4617
- b. HT-S Graphite and HYSOL's ADX-516 Adhesive
- c. HT-S and Geigy's P13N Polyimide
- d. HT-S and X-2546

e. HT-S and Fiberite Corporation's epoxy-phenolic (resin system X-911) - panel supplied by Fiberite.

f. HT-S and Fiberite Corporation's HT-Epoxy (a new experimental high-temperature epoxy system) - panel supplied by Fiberite.

ERLA-4617, ERL-2256, and EPON 828 were evaluated because they are used extensively as laminating epoxy resin systems, particularly with graphite fibers. X-2546 is a newly developed, modified epoxy resin with a high heat distortion temperature (485°F) and is therefore useful in composites at 350°F. This system has recently become commercially available in graphite prepregs. NARMCO 2387 was evaluated because it is the resin system used in the NARMCO 5505 boron/epoxy prepreg tape. The 5505 system is widely used in advanced composite developmental programs. Hercules Type HT-S fiber was utilized in the graphite composites because it is a widely used graphite fiber that is representative of the high-tensile-strength graphite fibers available today.

2. CYCLIC EXPOSURES

Cast resin and composite test specimens were subjected to the following stepwise environmental exposure cycle:

- Step 1. Relative humidity = 95-100%
Temperature = 120°F
Time = 22-1/2 hours
- Step 2. Specimens are removed from chamber and placed under normal room conditions for 15 minutes.
- Step 3. Temperature = -65°F
Time = 1 hour
- Step 4. Step 2 repeated.
- Step 5. Temperature = 250°F
Time = 30 minutes
- Step 6. Step 2 repeated

Data were obtained at room temperature, at 350°F after a one-hour soak, and at 350°F (one-hour soak) after 2, 10, 15, and 30 exposure cycles.

Cast resin specimens were tested in tension. Composite test specimens for tension and compression had a quasi-isotropic lay-up consisting of eight plies oriented 0°, +45°, -45°, 90°, 90°, -45°, +45°, 0°. Specimens were tested with the outer 0° ply parallel to the test direction. Compression data were obtained at room temperature, 350°F after a one-hour soak, and at 350°F (one-hour soak) after 30 cycles. Short-beam shear strengths were measured on unidirectionally reinforced specimens using the same test data points as in the compression testing. In addition to being tested at 350°F, the HT-S/P13N polyimide system was tested at 500°F after cycling. Tension, compression, and shear data were obtained at 500°F.

Tables I-VII show all of the cyclic exposure data obtained, including specimen weight gains and thickness changes as a function of the cyclic exposure. Figures 2, 6, 7, 8, 13, 14, and 15 show the percentage retention of room-temperature strength values as a function of the number of exposure cycles.

3. WATER BOIL EXPOSURES

Boron and graphite composite test specimens were also subjected to water boil exposures of varying duration. Specimens were boiled until their weight pick-up reached either their weight gain recorded during cycling - "equivalent water boil" - or a constant value - "equilibrium water boil." Flexural strengths and moduli and short-beam shear strengths were determined on quasi-isotropic and unidirectional lay-ups at room temperature, 350°F, and 350°F after water-boil exposure. Weight gain and thickness changes were also measured as a function of water boil exposure time. Resultant data are shown in Tables VIII and IX and depicted via bar graphs in Figures 16, 17, and 19.

A secondary factor of interest here was whether or not water-boil exposure could be used as a quick and effective screening test prior to cyclic exposure. Two groups of specimens were tested to determine if they had the same mechanical properties when one group had been cycled to the particular weight gain and the other group water-boiled to the same

weight gain. Table X shows the short-beam shear strengths at room temperature and 350°F for these specimens. At 350°F after 30 cycles (weight gain = 0.82%) and after 11 hours of water boil (weight gain = 0.81%), the room-temperature shear strength is approximately the same for the two groups.

4. TEST METHODS

Flexural testing was performed on specimens 4.0 in. by 0.5 in. wide utilizing the three-point loading method with a span-to-depth ratio of 32 to 1. Short-beam shear strengths were measured by the three-point loading method, employing a span-to-depth ratio of 4 to 1. Test specimens were 1.00 in. long by 0.25 in. wide. Tensile test coupons were 4.00 in. long by 0.375 in. wide. The 0.375 in. width is as large as the high-temperature strain-gage extensometer (0.5 in. gage length) can accommodate. Compression testing was performed in accordance with Federal Test Method Standard Number 406, Method 1021.

SECTION III

DISCUSSION OF RESULTS

1. CAST RESIN SYSTEMS

The cast resin systems were evaluated solely in tension after cyclic exposure. The data obtained are shown in Table I. Weight gain for the cast resin systems as a function of the number of exposure cycles is plotted in Figure 1. The extremely low strength values for ERL-2256/ZZL-0820 and EPON 828/m-PDA indicate they would not be useful systems at 350°F, even in dry conditions. This was expected, based on their low heat distortion temperatures (approximately 300°F for both systems). Both systems, however, were evaluated at room temperature after cyclic exposure with negligible mechanical property changes occurring after 30 exposure cycles. Each system did show a 30-cycle weight gain greater than 1.0% (Table I and Figure 1) and a 30-cycle thickness increase greater than 1.3%. The ERL-2256/ZZL-0820 system was evaluated at a more appropriate temperature (250°F) after cycling. The results are given in Table I and illustrated in Figure 2. The graph shows that the system experiences a considerable loss in strength due to the temperature alone. This initial loss is followed by a gradual decrease in the 250°F tensile strength after continued cyclic exposures and associated water absorption. Table I shows an increase in the 250°F total strain with continued cycling. Weight gains for this system averaged 1.40% after 30 cycles and approach an equilibrium value of 1.50-1.75%.

The ERLA-4617/m-PDA system was evaluated at both room temperature and 350°F after cyclic exposure. The room-temperature mechanical properties of the system changed only slightly after 30 exposure cycles, but the water weight gain was almost 3.0% and the thickness increased more than 1.5%. The curve in Figure 1 shows that equilibrium has not been reached after 30 cycles; in subsequent tests, equilibrium was reached after 42 cycles, with a weight gain of approximately 4%. At 350°F (Table I and Figure 2), the expected initial loss in strength and modulus occurred, as well as a concurrent increase in total strain (23%) due to the temperature

TABLE I
EFFECT OF EXPOSURE CYCLES ON THE TENSILE PROPERTIES OF CAST EPOXY RESINS

Test Temp (°F)	Number of Cycles	Tensile Strength (10 ³ psi)	Initial Tensile Modulus (10 ⁶ psi)	Total Strain (%)	Weight Gain (%)	Thickness Increase (%)
ERLA-4617 (Cured with 4,4'-methylene dianiline)						
R.T.	0	15.4	0.79	2.38	-	-
R.T.	2	16.3	0.83	3.29	0.54	0.46
R.T.	10	17.9	0.70	4.39	1.76	0.48
R.T.	15	15.9	0.76	3.40	2.02	0.82
R.T.	30	12.9	0.91	1.96	2.99	1.65
R.T.	0	15.40	0.79	2.38	-	-
350	0	5.69	0.52	23.15	-	-
350	2	1.62	0.038	21.29	0.51	0.46
350	10	1.04	0.023	16.10	1.90	0.49
350	15	0.86	0.014	14.00	2.56	0.80
350	30*	-	-	-	3.02	1.62
*Specimens were too "soft" to test						
ERL-2256 (Cured with 4,4'-methylene dianiline)						
R.T.	0	16.4	0.60	5.53	-	-
R.T.	2	15.1	0.54	7.66	0.26	0.40
R.T.	10	14.8	0.50	6.94	0.78	0.55
R.T.	15	14.5	0.55	6.33	0.95	1.10
R.T.	30	15.0	0.63	7.47	1.39	1.63
R.T.	0	16.4	0.60	5.53	-	-
250	0	8.72	0.44	10.1	-	-
250	2	6.78	0.40	14.6	0.31	0.46
250	10	6.24	0.38	13.8	0.81	0.60
250	15	5.25	0.36	14.7	1.35	1.16
250	30	5.97	0.34	14.4	1.51	1.83
350	0	1.60	-	-	-	-
EPON 828 (Cured with 4,4'-methylene dianiline)						
R.T.	0	13.8	0.50	6.10	-	-
R.T.	2	13.5	0.45	6.41	0.19	0.40
R.T.	10	13.2	0.44	5.87	0.53	0.58
R.T.	15	12.6	0.47	5.72	0.66	0.82
R.T.	30	13.4	0.52	5.12	1.05	1.37
350	0	0.80	-	-	-	-
U.C.C. 546						
R.T.	0	14.2	0.85	2.44	-	-
350	0	5.8	0.41	1.92	-	-
350	15	2.9	0.14	3.60	4.32	1.79
350	30	1.0	0.15	0.73	6.53	2.80
NARMCO 2387 (50% Composite Resin)						
R.T.	0	8.5	0.51	1.94	-	-
R.T.	2	8.3	0.48	2.09	0.35	-
R.T.	10	5.8	0.50	3.36	1.63	-
R.T.	15	8.8	0.49	2.42	2.14	0.94
R.T.	30	9.5	0.53	2.61	3.47	1.90
R.T.	0	8.1	0.50	1.88	-	-
350	0	3.5	0.13	5.94	-	-
350	15	1.9	0.14	5.0	1.92	0.90
350	30	1.2	0.04	5.9	3.07	1.64

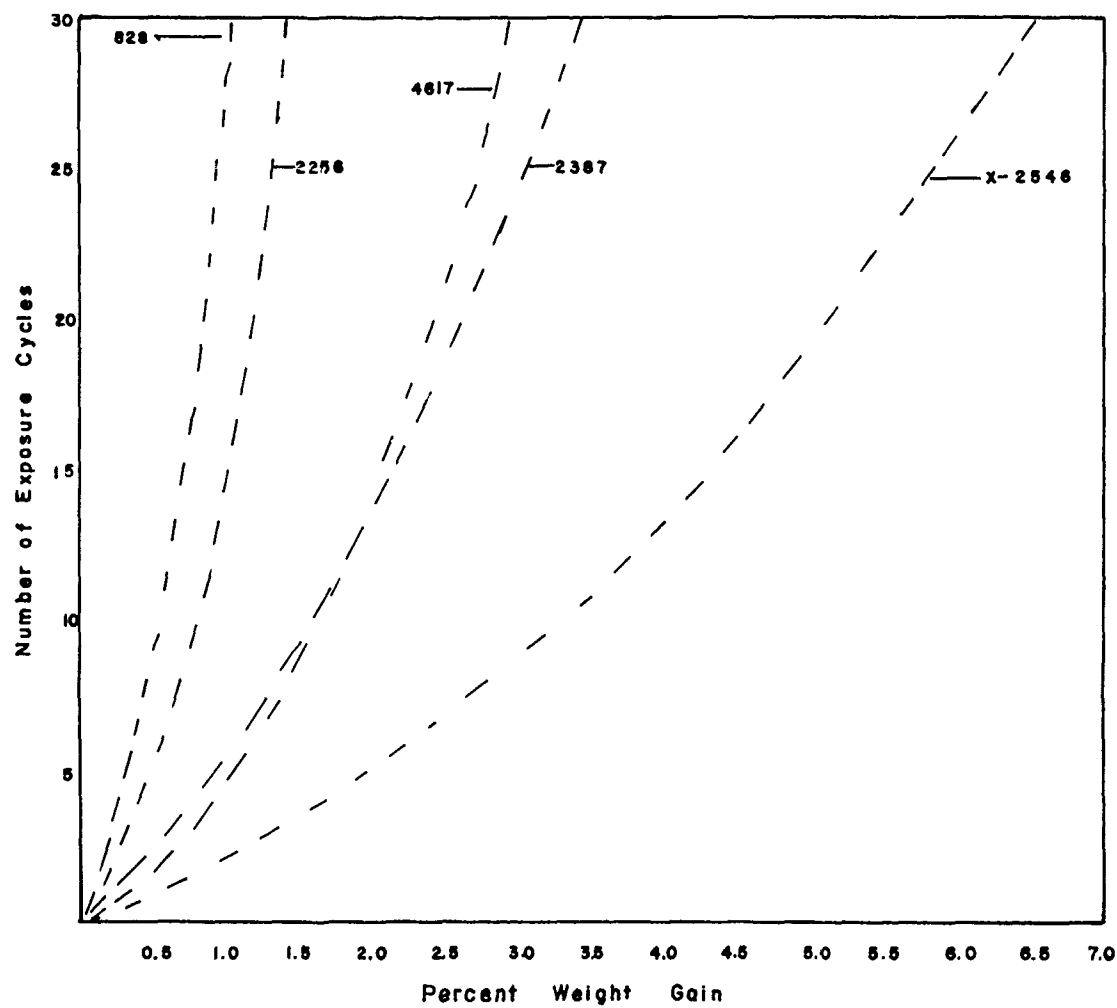


Figure 1. Effect of Exposure Cycles on the Weight Gain of Cast Resin Systems

ERL-2256

ERLA-4617

NARMCO 2387

X-2546

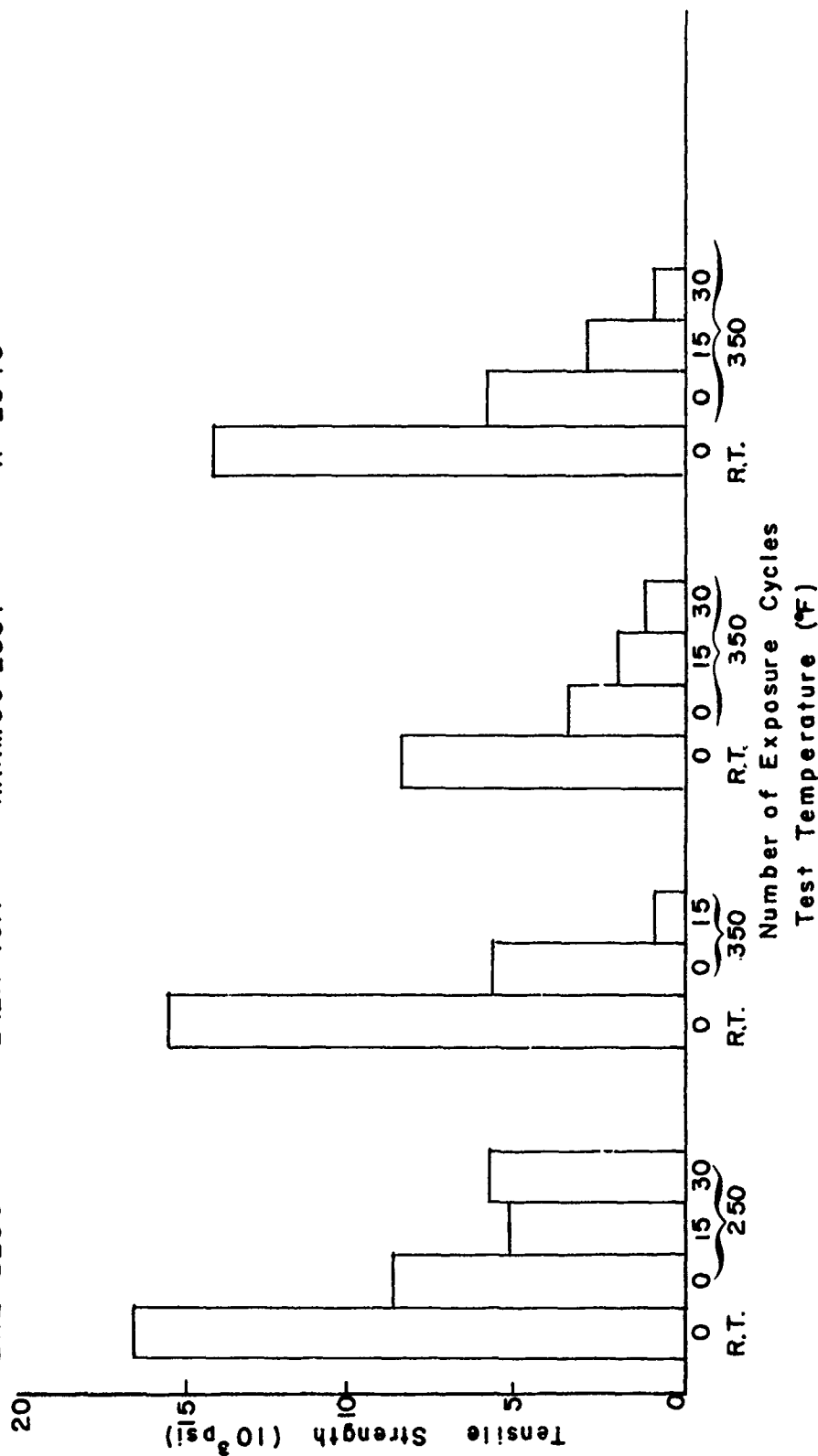


Figure 2. Effect of Exposure Cycles on the Tensile Strength of Cast Epoxy Resins

alone; this was anticipated because of the system's 347°F heat distortion temperature. Continued cyclic exposure resulted in a drastic loss of 350°F tensile strength, as shown in Table I and Figure 2. After 15 exposure cycles the 350°F tensile strength was only 15% of that of the unexposed 350°F strength with a water weight gain of 2.56%. The combined effects of water absorption (3.02%) and 350°F temperature caused such a large degree of plasticity or "softness" that the values could not be measured at the 30-cycle point. The effects of the environment are also reflected in the drastic loss in modulus. The stress-strain curves for the ERLA-4617 system as a function of exposure cycle and test temperature are shown in Figure 3. The drastic effects of water absorption on the 350°F mechanical properties of this system can readily be seen.

Specimens of ERLA-4617/m-PDA were aged under normal room temperature conditions for six months to see the effect of long-term room temperature aging on the 350°F tensile properties. Data obtained show that the 350°F tensile strength after six months aging (1330 psi) is 77% lower than the 350°F strength value prior to aging (5700 psi). The six months old specimens were dried at 250°F in vacuo until maximum weight loss occurred in order to determine the amount of water absorption; a value of 0.65% was obtained. After drying, the specimens recovered their original strength.

NARMCO's 2387 resin system was also evaluated at room temperature and 350°F after cyclic exposure. This system is important because of its use in NARMCO 5505 boron/epoxy prepreg. The data at room temperature (Table I) show that no significant change in mechanical properties occurs after 30 exposure cycles. However, a significant amount of moisture was absorbed, as evidenced by a 3.47% weight gain (Figure 1) and a thickness increase of 1.90%. The data obtained at 350°F (Table I and Figure 2) show that the temperature alone causes a 57% reduction in the tensile strength; however, this strength reduction was not accompanied by an extremely large increase in strain, as was the case with the 4617 system. Normal tensile failures were observed, but continued cyclic exposure resulted in a considerable loss of 350°F tensile strength, as shown by

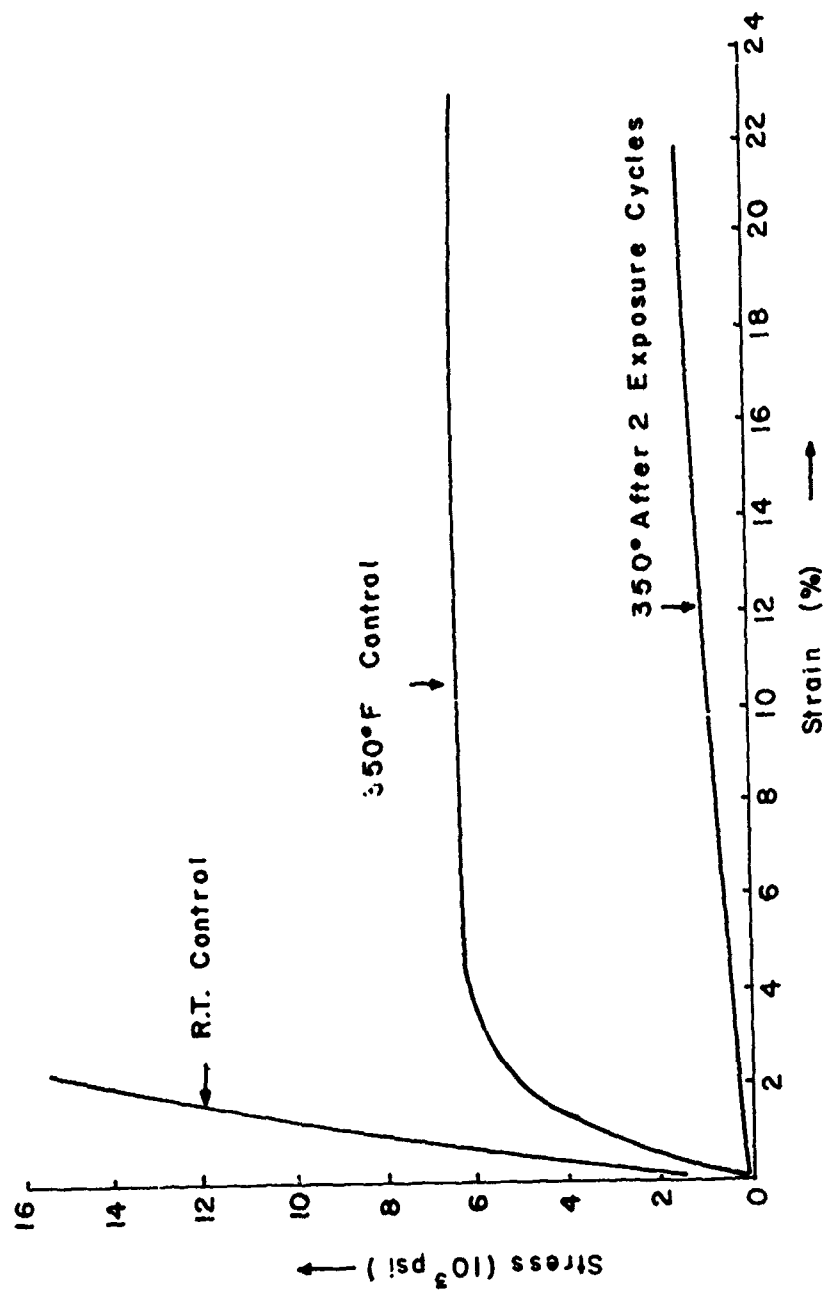


Figure 3. Effect of Exposure Cycles on the Stress-Strain Behavior of ERLA-4617 Cast Epoxy Specimens

Table I and Figure 2. The 350°F/30-cycle value was 35% of the 350°F/0-cycle value. A moisture weight pick-up of 3.07% and a thickness increase of 1.64% were found. The system still behaved normally with respect to the type of failure observed. All of the specimens failed at 350°F after cycling in a more "brittle" fashion instead of the high elongation type failures of the 4617 system. The 350°F/30 cycle specimens did show a significant loss in modulus as compared to the uncycled 350°F specimens. The modulus value of 40,000 psi is only 30% of the 350°F control modulus and only 8% of the initial room temperature control. A heat distortion temperature of 360°F was found for this system.

The last resin system to be evaluated in cast form was U.C.C.'s X-2546, a new high temperature epoxy containing U.C.C.'s ERLB-4617 resin, ZXZL-5152 hardener, and BF_3 .MEA catalyst. The heat distortion temperature of this system is approximately 485°F. Data obtained at room temperature and 350°F after cyclic exposure are shown in Table I and Figure 2. The 350°F value shows that the temperature factor alone results in a 59% reduction of the tensile strength. After 30 exposure cycles the 350°F tensile strength was only 17% of the unexposed 350°F tensile strength. This system exhibited the largest amount of water pick-up (6.53%), shown in Figure 1, and thickness increase (1.90%), shown in Table I, after 30 exposure cycles of any cast resin system evaluated. Large cracks had formed in the tensile coupons after 30 cycles (Figures 4 and 5). Tensile failures were similar to those of the 2387 resin system in that "brittle" failures were observed at 350°F instead of the high elongation type failure of the 4617 system.

2. GRAPHITE COMPOSITES UNDER CYCLIC EXPOSURE

Graphite composites utilizing several different resin systems were subjected to the environmental exposure cycle outlined in Section II. Tension and compression data were obtained on quasi-isotropic (0°, +45°, -45°, 90°, 90°, -45°, +45°, 0°) lay-ups while short-beam shear strengths were measured on unidirectional lay-ups.



Figure 4. Cast Resin Specimen of X-2546 After 30 Environmental Exposure Cycles, Face-View



Figure 5. Cast Resin Specimen of X-2546 After 30 Environmental Exposure Cycles, Side View

Tension data obtained on HT-S graphite/ERLA-4617 (m-PDA) composites are shown in Table II. The data are further illustrated in Figure 6, which indicates the effects of environmental cycling on the retention of unexposed, room-temperature control tensile strength. The graph reveals that the 350°F unexposed control retains only 57% of the tensile strength of the room-temperature unexposed control. After this initial loss in strength due to the temperature, continued exposure cycles produced no significant additional reduction in the 350°F tensile strength. Similar behavior was observed in the compression and short-beam shear testing (Tables III and IV and Figures 7 and 8). The 350°F temperature, alone, resulted in a 78% loss of compressive strength (Figure 7) and a 77% loss in short-beam shear strength (Figure 8). The 350°F/30 exposure-cycle values are not significantly changed from the 350°F control values in both compression and shear.

A new adhesive system (Hysol's ADX-516 epoxy/polysulfone adhesive) which has promising properties at +300°F was evaluated in composite form with the HT-S fiber. Composites of HT-S and ADX-516 had fairly good room temperature properties (Table II). The tensile modulus was slightly low, but the tensile, compressive, and shear strengths compare favorably to those of HT-S composites having a typical epoxy system. Under cyclic exposure conditions, HT-S/ADX-516 composite behavior was similar to that of the HT-S/4617 composites (see Figures 6, 7, and 8 and Tables II, III, and IV). The 350°F temperature alone resulted in a 57% reduction of the tensile strength (Figure 6), a 92% reduction of the compressive strength (Figure 7), and a 66% reduction of the short-beam shear strength (Figure 8). The 350°F strength values (tension, compression, and shear) were essentially unaffected after 30 exposure cycles (Figures 6, 7, and 8).

Polyimide/graphite composites were also evaluated in this program. The resin chosen was Geigy's P13N, a typical "state-of-the-art" polyimide. Testing was performed at 350°F and 500°F after cycling. Tensile moduli were not measured at 500°F. Weight gains for this system (Tables II and III) are affected considerably by the 250°F/30-minute step of the exposure cycle. Weight gains of over 2% were found before the 250°F step

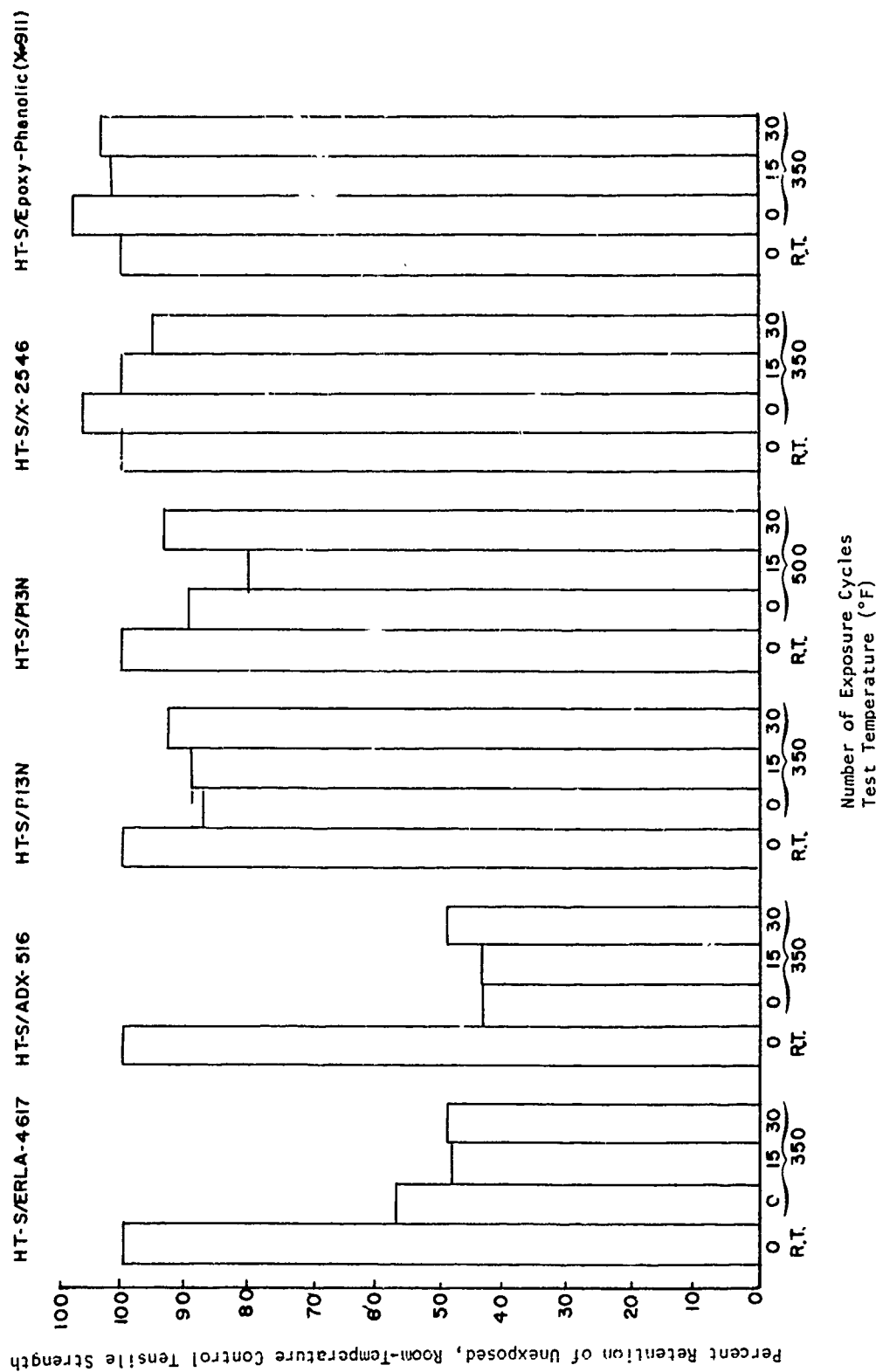


Figure 6. Effect of Exposure Cycles on the Tensile Strength of Quasi-Isotropic Graphite/Epoxy Composites

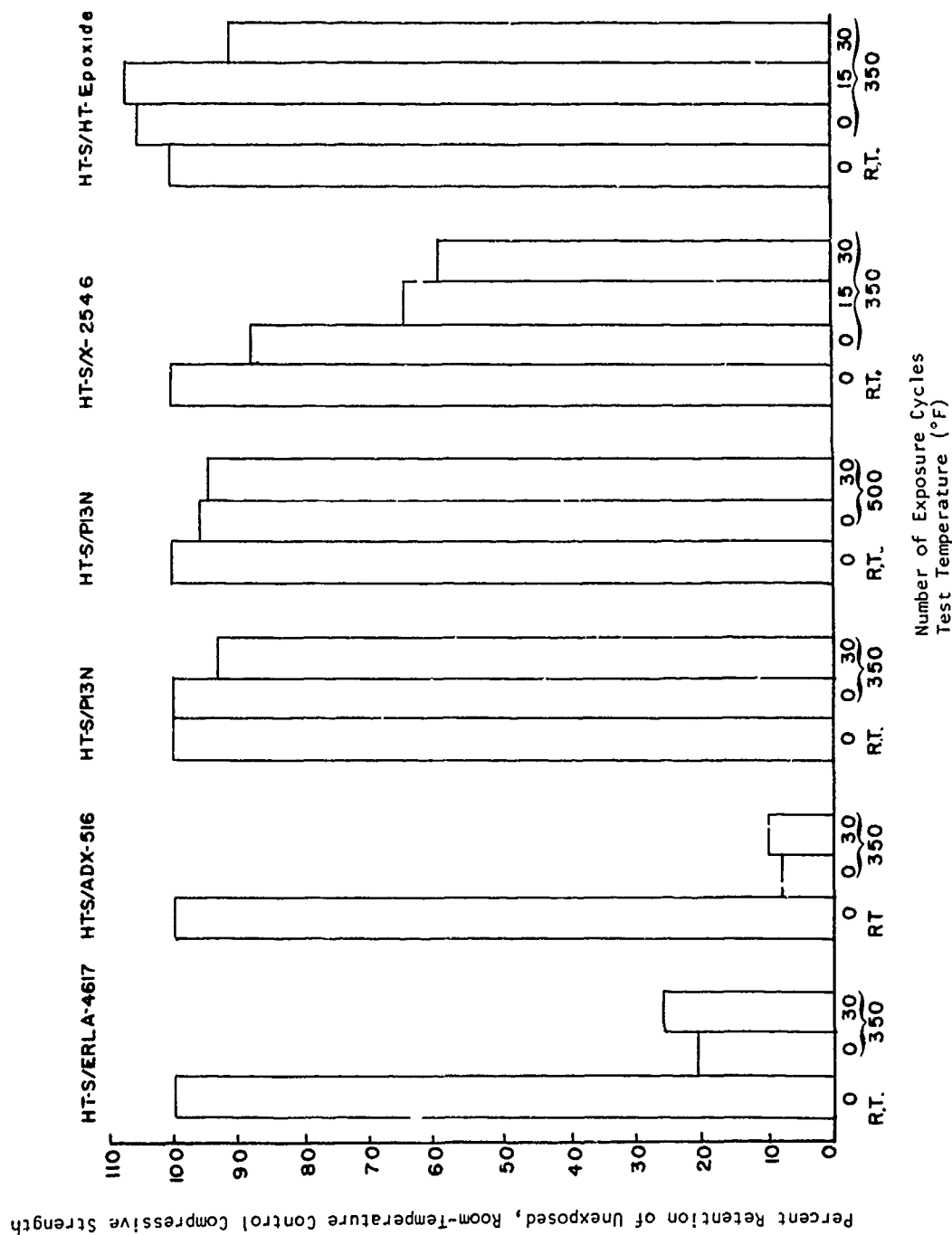


Figure 7. Effect of Exposure Cycles on the Compressive Strength of Quasi-Isotropic Graphite/Epoxy Composites

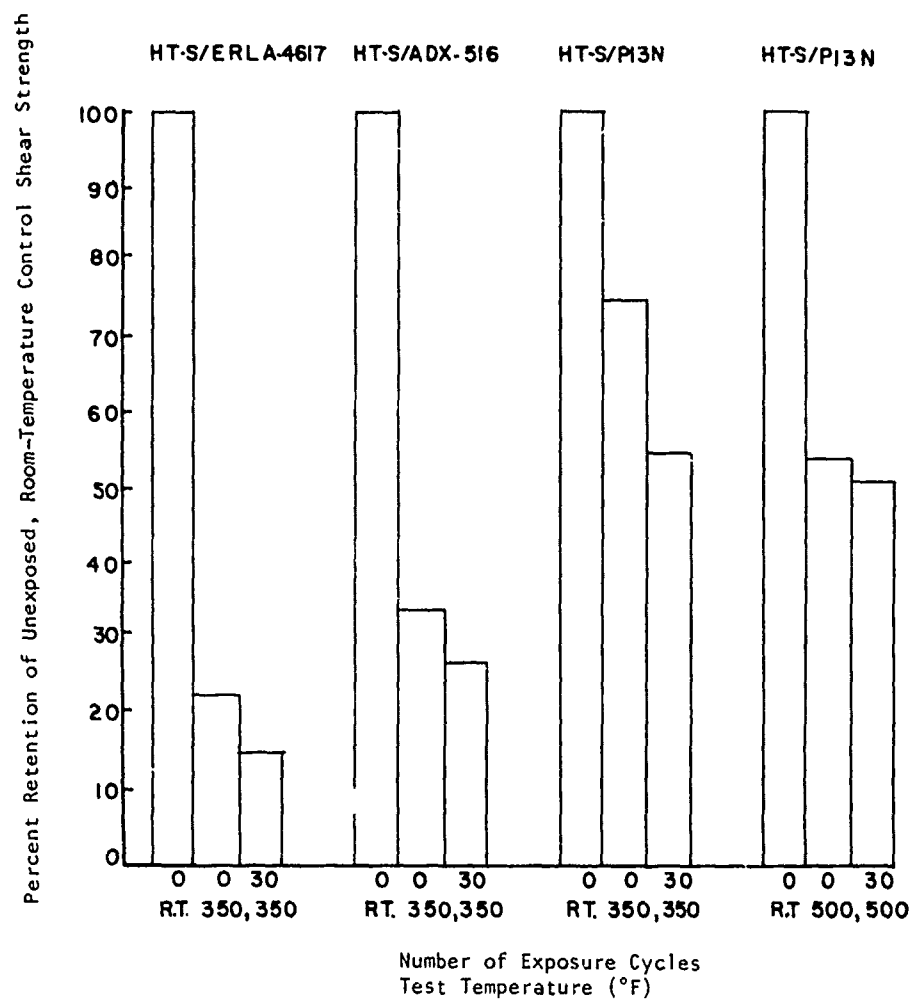


Figure 8. Effect of Exposure Cycles on the Short-Beam Shear Strength of Unidirectional Graphite/Epoxy Composites

TABLE II
EFFECT OF EXPOSURE CYCLES ON THE TENSILE PROPERTIES OF
QUASI-ISOTROPIC GRAPHITE/EPOXY COMPOSITES

Test Temp (°F)	Number Of Cycles	Tensile Strength (10 ³ psi)	Tensile Modulus (10 ⁶ psi)	Total Strain (%)	Weight Gain (%)	Thickness Increase (%)
HT-S Graphite/ERLA-4617 Epoxy						
R.T.	0	64.4	7.46	1.32	-	-
350	0	36.6	5.20	1.16	-	-
350	2	36.8	4.70	0.63	0.71	0.34
350	10	35.8	5.69	0.70	0.86	0.70
350	15	31.2	4.91	0.66	1.20	1.04
350	30	31.9	4.96	1.67	1.05	1.75
HT-S Graphite/Hysol ADX-516 Adhesive (Epoxy-Polysulfone)						
R.T.	0	50.2	5.7	0.90	-	-
350	0	21.5	3.8	0.53	-	-
350	2	22.0	3.8	0.57	0.28	-
350	10	24.3	3.6	0.66	0.74	1.19
350	15	21.9	3.3	0.64	0.90	-
350	30	24.7	3.6	0.66	1.30	1.70
HT-S Graphite/P13N Polyimide						
R.T.	0	41.2	5.90	0.88	0	-
350	0	35.9	5.17	0.96	0	-
350	2	37.6	4.92	0.93	0	-
350	10	35.4	5.30	0.91	0	-
350	15	36.7	4.82	0.93	0	-
350	30	38.2	5.10	0.84	0.06	0.74
HT-S Graphite/P13N Polyimide						
R.T.	0	41.2	-	-	0	-
500	0	36.7	-	-	0	-
500	2	33.9	-	-	0	-
500	10	36.9	-	-	0	-
500	15	32.8	-	-	-	-
500	30	38.6	-	-	0.10	0.85
HT-S Graphite/Fiberite Epoxy-Phenolic (X-911)						
R.T.	0	58.2	9.6	0.73	-	-
350	0	62.6	10.4	0.65	-	-
350	15	59.1	11.2	0.55	0.30	0.56
350	30	60.1	10.4	0.68	0.36	0.77
HT-S Graphite/U.C.C.'s X-2546 Epoxy						
R.T.	0	49.7	7.5	0.72	-	-
350	0	53.1	6.5	1.05	-	-
350	15	49.5	7.3	0.76	1.42	0.70
350	30	47.0	6.2	0.81	2.15	1.90

TABLE III
EFFECT OF EXPOSURE CYCLES ON THE COMPRESSION PROPERTIES OF
QUASI-ISOTROPIC GRAPHITE/EPOXY COMPOSITES

Test Temp (°F)	Number of of Cycles	Compressive Strength (103 psi)	Weight Gain (%)	Thickness Increase (%)
HT-S GRAPHITE/ERLA-4617 EPOXY				
R.T.	0	46.0	-	-
350	0	10.0	-	-
350	30	11.9	1.12	0.70
HT-S GRAPHITE/HYSOL ADX-516 ADHESIVE				
R.T.	0	38.3	-	-
350	0	3.1	-	-
350	30	4.0	1.23	1.65
HT-S GRAPHITE/P13N POLYIMIDE				
R.T.	0	31.3	-	-
350	0	32.4	-	-
350	30	29.1	-0.21	0.30
HT-S GRAPHITE/P13N POLYIMIDE				
R.T.	0	31.3	-	-
500	0	30.0	-	-
500	30	29.5	-0.18	0.28
HT-S GRAPHITE/FIBERITE HT-EPOXIDE				
R.T.	0	31.5	-	-
350	0	33.0	-	-
350	15	33.7	0.30	0.56
350	30	28.6	0.36	0.77
HT-S GRAPHITE/U.C.C.'s X-2546 EPOXY				
R.T.	0	38.4	-	-
350	0	33.8	-	-
350	15	25.0	1.34	1.16
350	30	22.9	2.05	1.95

TABLE IV

EFFECT OF EXPOSURE CYCLES ON THE SHORT-BEAM SHEAR
STRENGTHS OF UNIDIRECTIONAL GRAPHITE/EPOXY COMPOSITES

Test Temp (°F)	Number Of Cycles	Shear Strength (10 ³ psi)	Weight Gain (%)	Thickness Increase (%)
HT-S GRAPHITE/ERLA-4617 EPOXY				
R.T.	0	12.8	-	-
350	0	2.9	-	-
350	30	1.9	0.66	0.35
HT-S GRAPHITE/HYSOL ADX-516 ADHESIVE (EPOXY-POLYSULFONE)				
R.T.	0	12.1	-	-
350	0	4.1	-	-
350	30	3.3	0.67	0.90
HT-S GRAPHITE/P13N POLYIMIDE				
R.T.	0	13.2	-	-
350	0	9.9	-	-
500	0	7.1	-	-
350	30	7.2	0.90	0.49
500	30	6.7	1.05	0.50

of the 30th cycle, whereas values as low as 0.06% were found after this step. All composites and castings were affected in this manner, but none were affected to such an extent as were the polyimides. The higher void content of the polyimides (4-5%) as opposed to the epoxies (less than 2%) would contribute to this unusual behavior. Another factor would be that the more polar epoxies would hold moisture more tenaciously than the relatively less polar polyimide. The 350°F and 500°F tension and compression data (Tables I and II and Figures 6 and 7) show that this system is generally unaffected by the temperature or the environment. There is a slight reduction in the shear strength due to the temperatures (Table IV and Figure 8); however, the cyclic exposures did not significantly affect the 350°F or 500°F short-beam shear values.

Tension and compression data obtained as a function of cyclic exposure on quasi-isotropic composites of HT-S and U.C.C.'s X-2546 epoxy resin are plotted in Figures 6 and 7. Tensile strengths are largely unaffected by the temperature or environmental cycling (Table II and Figure 6), although this system did exhibit a considerable weight gain (2.15%) and thickness increase (1.90%). Photomicrographs (Figures 9 - 12) of composite tensile test specimens before and after 30 exposure cycles show that the cracks that had formed in the cast resin specimens after 30 cycles are not present in the composite specimens after equivalent aging. In compression the 350°F temperature caused only a 12% loss of room-temperature strength (Figure 7). The 350°F compressive strengths are seen to be reduced in proportion to the exposure cycles. The 350°F compressive strength after 30 cycles is 30% lower than the uncycled 350°F control and 40% lower than the room-temperature control.

The last systems evaluated were HT-S graphite and Fiberite's epoxy-phenolic resin (X-911) and HT-S graphite with Fiberite's new high-temperature epoxy resin (HT-Epoxy). Tension (X-911 system) and compression (HT-Epoxy system) data for these composites are shown in Tables II and III and Figures 6 and 7. Figures 6 and 7 show that neither the temperature nor the cyclic exposure has any detrimental effect on the tensile or compressive strength of the systems. The tensile strength of the X-911 composites at 350°F after 30 cycles is equivalent to the room-temperature

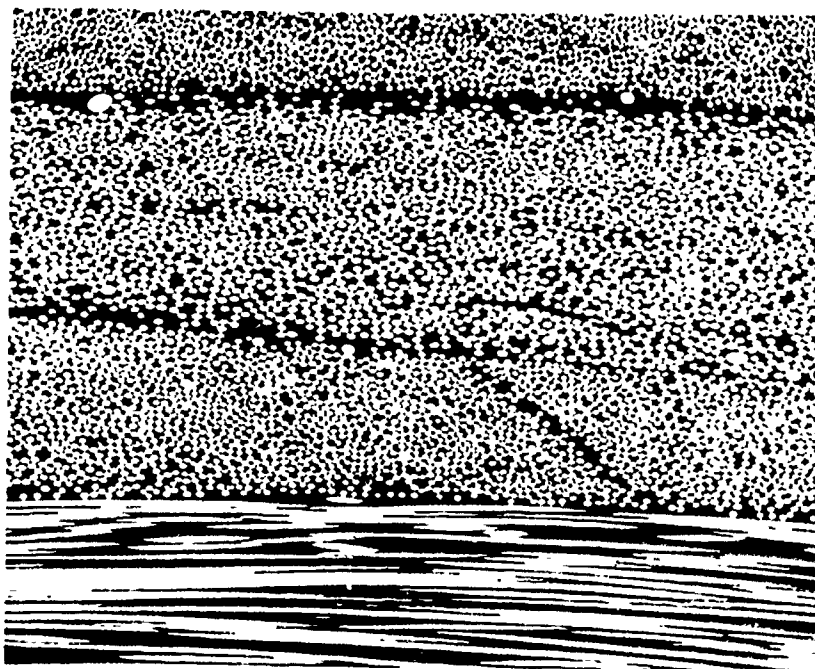


Figure 9. Photomicrograph of Unexposed Quasi-Isotropic Tension Specimen of HT-S/X-2546 (End View, 100 X)

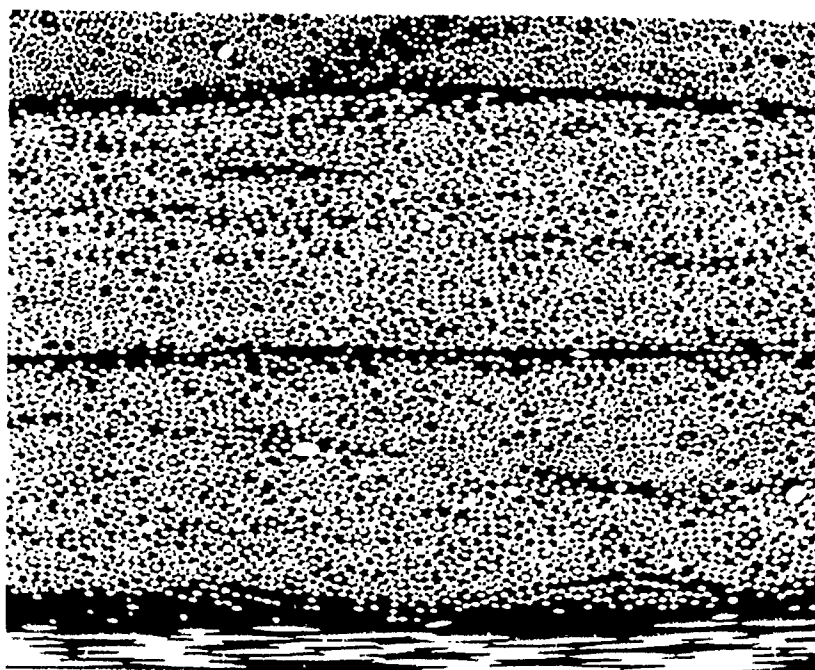


Figure 10. Photomicrograph of Quasi-Isotropic Tension Specimen of HT-S/X-2546 (End View, 100 X) After 30 Exposure Cycles

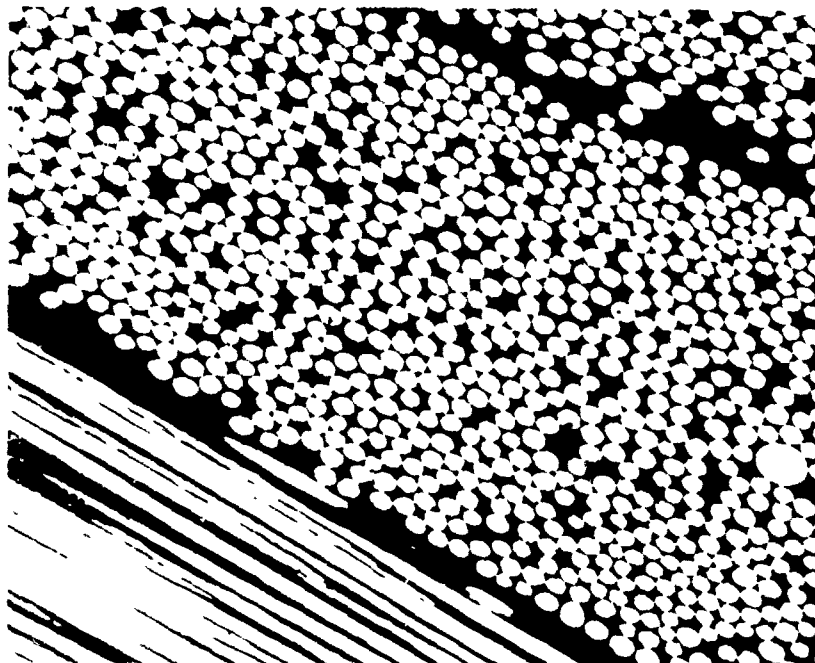


Figure 11. Photomicrograph of Unexposed Quasi-Isotropic Tension Specimen of HT-S/X-2546 (End View, 250 X)

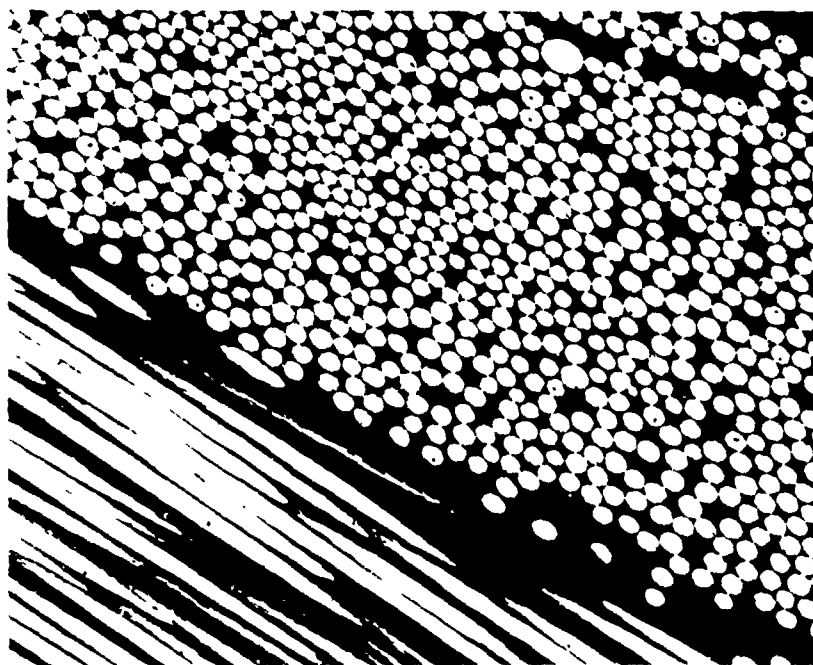


Figure 12. Photomicrograph of Quasi-Isotropic Tension Specimen of HT-S/X-2546 (End View, 250 X) After 30 Exposure Cycles

control strength, and the compressive strength of the HT-Epoxy composite at 350°F after 30 cycles is only 9% less than that of the room-temperature control. Weight gains and thickness increases were minimal (Tables II and III).

3. BORON COMPOSITES UNDER CYCLIC EXPOSURE

Boron composites were fabricated from NARMCO's 5505 boron/epoxy prepreg. Quasi-isotropic composites (same orientation as the graphite composites) were evaluated in tension and compression at room temperature and 350°F after environmental cycling. Short-beam shear strengths were measured on unidirectional composites under the same conditions as the tension and compression laminates. The data obtained are presented in Tables V - VII, and illustrated in Figures 13 - 15. Both postcured and nonpostcured panels were evaluated.

The nonpostcured panels tested at room temperature after 2, 10, 15, and 30 exposure cycles showed no significant loss in tensile strength. Both the nonpostcured and postcured panels tested at 350°F after cyclic exposure showed no loss in room-temperature tensile strength due to the temperature and no loss in 350°F tensile strength due to the environmental cycling (Figure 13).

Both postcured and nonpostcured panels behaved similarly in compression and shear (Figures 14 and 15). There was an initial loss in strength due to the temperature with the compressive strengths reduced 30-40% and the shear strength lowered about 50%. The 350°F strengths (both shear and compression) were not significantly affected after 30 exposure cycles. Low weight gains and thickness increases were found for both the nonpostcured and postcured panels.

TABLE V
EFFECT OF EXPOSURE CYCLES ON THE TENSILE PROPERTIES
OF QUASI-ISOTROPIC BORON/EPOXY COMPOSITES

Test Temp (°F)	No. of Cycles	TENSILE STRENGTH (10 ³ psi)	TENSILE MODULUS (10 ⁶ psi)	TOTAL STRAIN (%)	WEIGHT GAIN (%)	THICKNESS INCREASE (%)
NARMCO 5505 BORON/EPOXY - NOT POSTCURED						
R.T.	0	48.7	10.3	0.69	-	-
R.T.	2	41.7	9.8	0.55	0.06	-
R.T.	10	47.3	9.6	0.62	0.33	0.20
R.T.	15	43.7	8.4	0.56	0.20	0.15
R.T.	30	43.3	10.2	0.50	0.22	0.16
NARMCO 5505 BORON/EPOXY - NOT POSTCURED						
R.T.	0	47.6	11.4	0.57	-	-
350	0	43.5	10.1	0.50	-	-
350	2	49.0	10.4	0.61	0.19	-
350	10	44.6	9.0	0.61	0.76	0.58
350	15	40.1	9.7	0.54	0.64	0.50
350	30	45.3	9.0	0.65	0.55	0.43
NARMCO 5505 BORON/EPOXY - POSTCURED						
R.T.	0	43.0	10.9	0.56	-	-
350	0	44.0	10.1	0.51	-	-
350	2	44.3	10.1	0.60	0.35	-
350	15	44.0	9.4	0.60	0.52	0.45
350	30	43.0	9.2	0.69	0.63	0.52

TABLE VI

EFFECT OF EXPOSURE CYCLES ON THE COMPRESSION PROPERTIES
OF QUASI-ISOTROPIC BORON/EPOXY COMPOSITES

Test Temp (°F)	No. of Cycles	Compressive Strength (10 ³ psi)	Weight Gain (%)	Thickness Increase (%)
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NARMCO 5505 BORON/EPOXY NOT POSTCURED

R.T.	0	42.5	-	-
350	0	30.6	-	-
R.T.	30	41.4	0.38	0.25
350	30	29.9	0.42	0.29

NARMCO 5505 BORON/EPOXY - POSTCURED

R.T.	0	50.8	-	-
350	0	29.5	-	-
350	30	30.2	0.25	0.18

TABLE VII

EFFECT OF EXPOSURE CYCLES ON THE SHORT-BEAM SHEAR
STRENGTHS OF UNIDIRECTIONAL BORON/EPOXY COMPOSITES

Test Temp (°F)	No. of Cycles	SHEAR STRENGTH (10 ³ psi)	Weight Gain (%)	Thickness Increase (%)
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NARMCO 5505 BORON/EPOXY - NOT POSTCURED

R.T.	0	14.9	-	-
350	0	6.3	-	-
R.T.	30	12.0	0.55	0.41
350	30	5.5	0.58	0.42

NARMCO 5505 BORON/EPOXY - POSTCURED

R.T	0	13.3	-	-
350	0	6.0	-	-
350	30	5.6	0.82	0.62

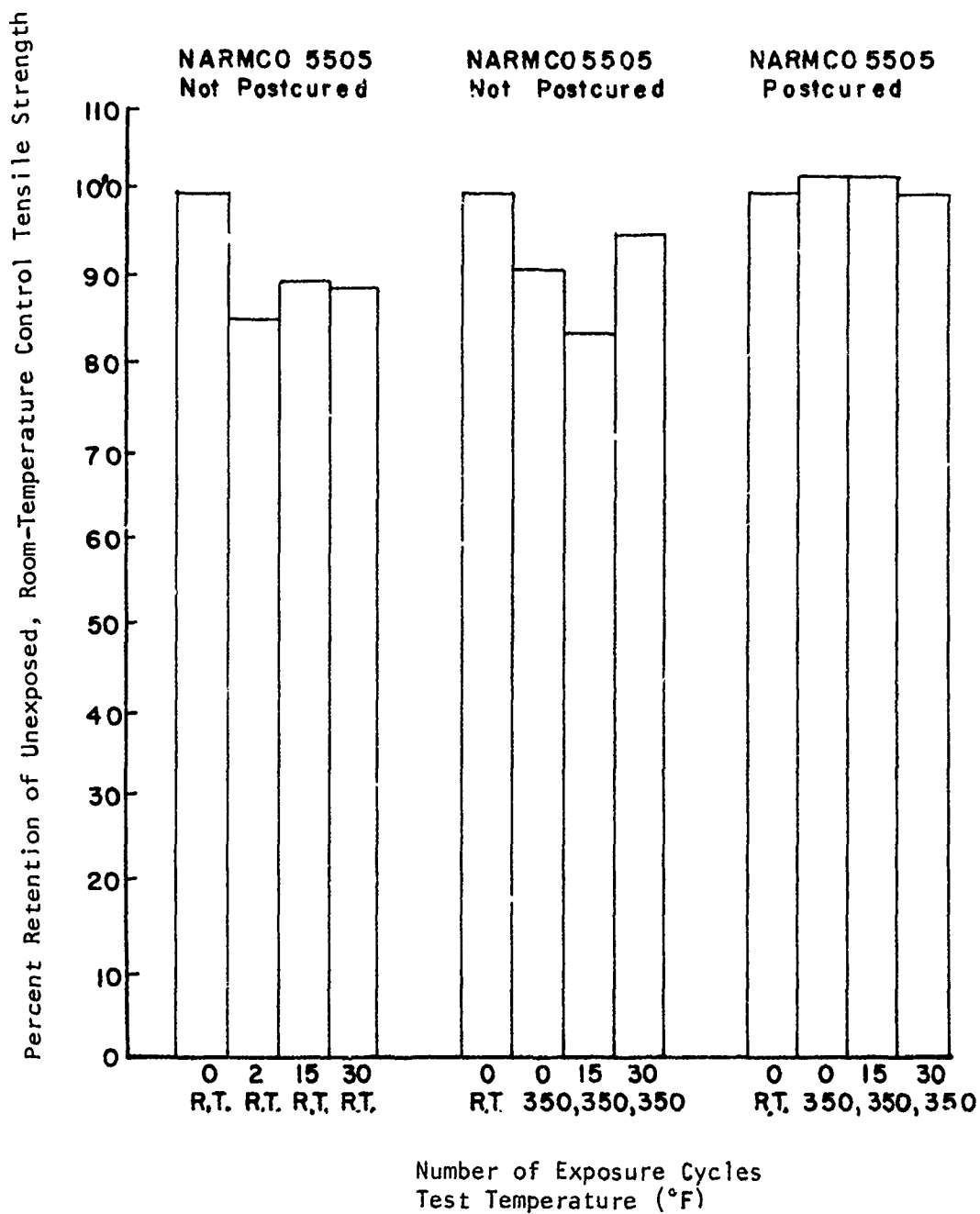


Figure 13. Effect of Exposure Cycles on the Tensile Strength of Quasi-Isotropic Boron/Epoxy Composites

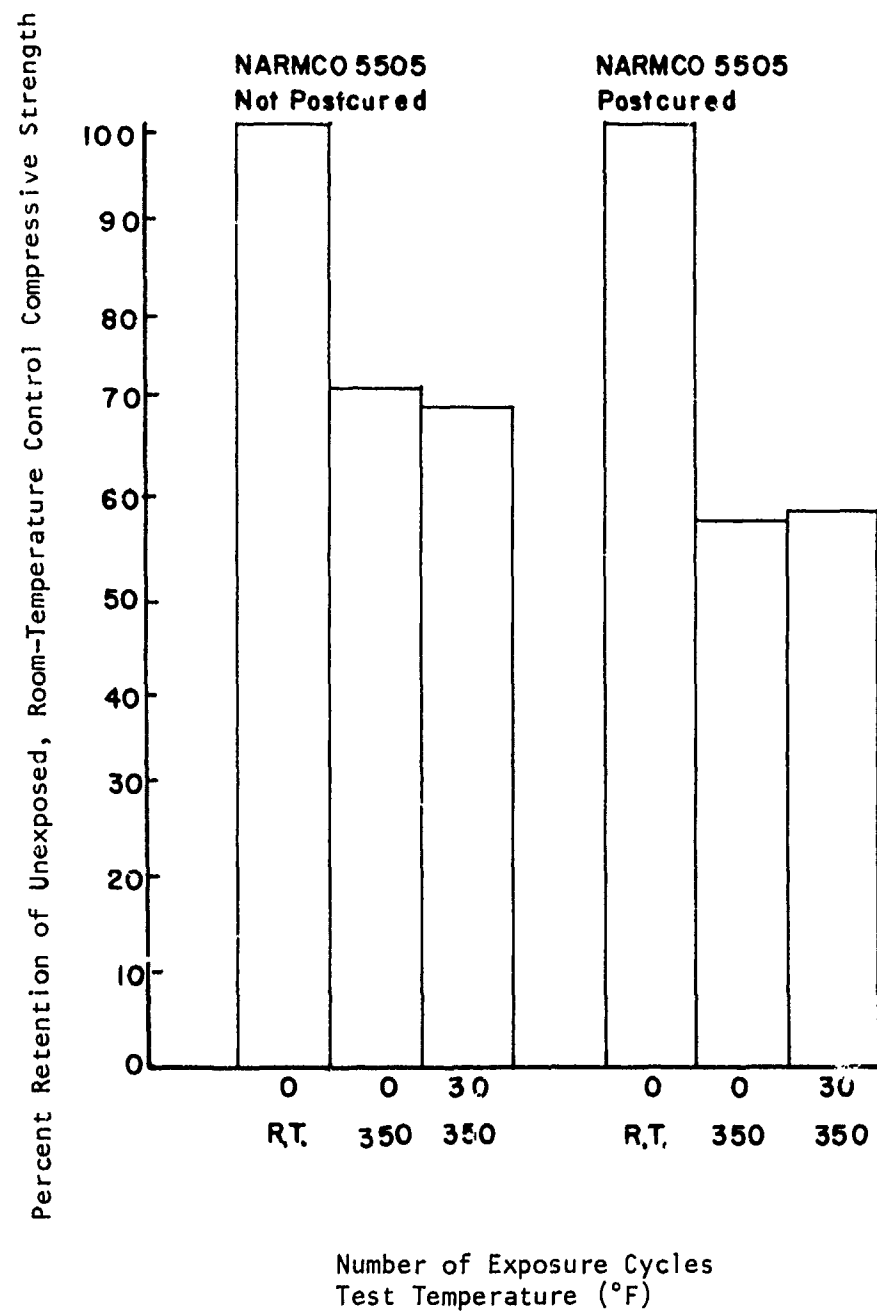


Figure 14. Effect of Exposure Cycles on the Compressive Strength of Quasi-Isotropic Boron/Epoxy Composites

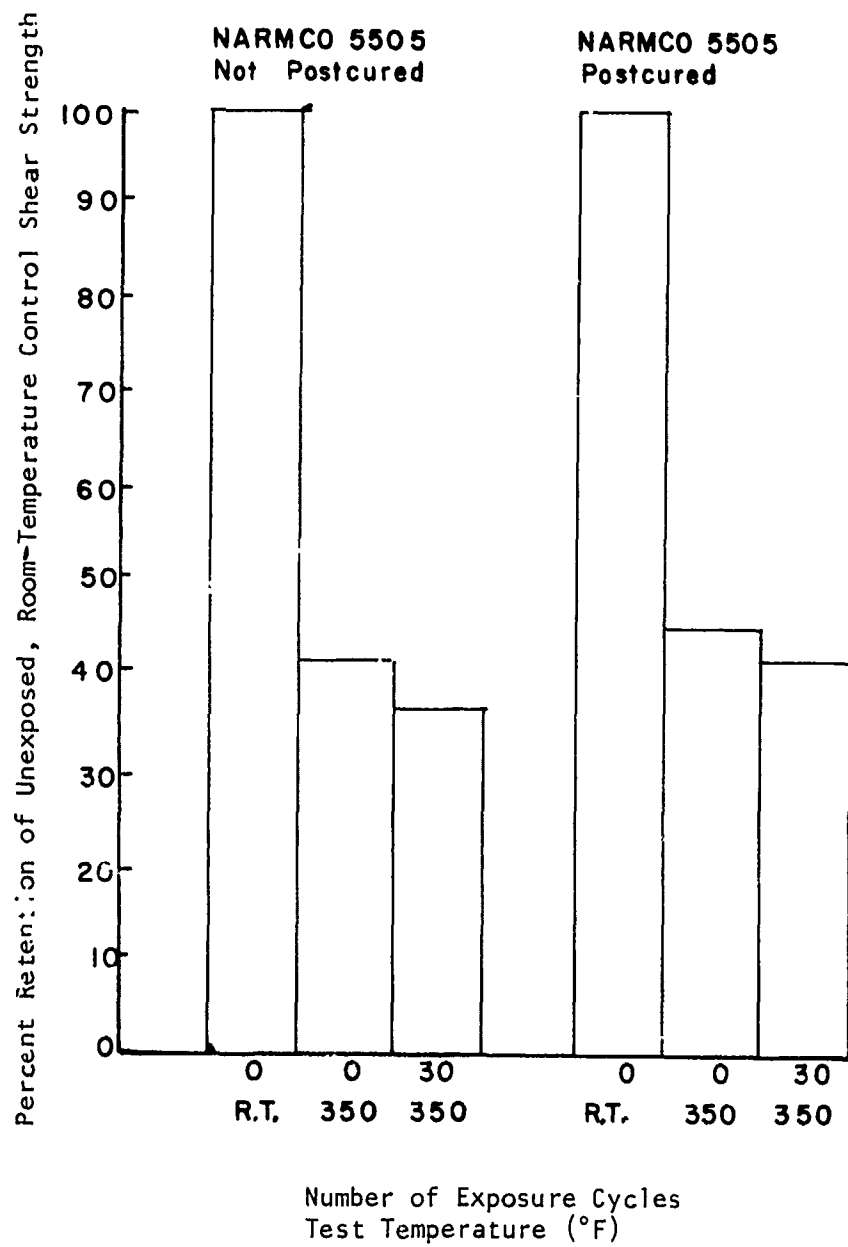


Figure 15. Effect of Exposure Cycles on the Short-Beam Shear Strength of Unidirectional Boron/Epoxy Composites

4. GRAPHITE COMPOSITES UNDER WATER BOIL EXPOSURE

Graphite composites utilizing several different resin systems were subjected to water boil exposure, as discussed in Section II. Flexural strength and modulus and short-beam shear strength were determined at room temperature and 350°F (before and after water-boil exposure) on quasi-isotropic and unidirectional lay-ups. Weight gain and thickness change were also measured as a function of water-boil exposure time.

It is recognized that in the flexural and shear testing of quasi-isotropic specimens, one can compare loads only. The normal calculations for stress cannot be performed since the actual stresses are not known. In our work, the strengths and moduli were calculated as in the normal flexural or shear testing, and therefore, one should use these numbers for comparisons only, not as absolute true values.

Water-boil exposure was of particular interest because of the possibility of utilizing this as a quick and effective screening test for cyclic exposure. It was found that two groups of specimens having the same weight gain, but with one group having been cycled to the particular weight gain and the other having been water boiled to the same weight gain (Table X) exhibited similar mechanical properties.

All of the data obtained on the graphite composite systems are shown in Table VIII. Figures 16 and 17 show the effects of test temperature (350°F) and water-boil-exposure time on the percent retention of the unexposed-room-temperature strength value.

The first system evaluated was HT-S and ERLA-4617. Water-boil exposure for a half-hour resulted in specimen weight gains equivalent to those for the same system after 30 environmental exposure cycles. Test results are shown in Table VIII and Figures 16 and 17. The quasi-isotropic specimens undergo a 75% loss of load due to the 350°F temperature alone. After a half-hour water boil, the 350°F value has declined only slightly. The unidirectional specimens exhibited a 39% loss in flexural strength due to the temperature factor; after a half-hour water boil, the 350°F

TABLE VIII
EFFECT OF WATER BOIL ON THE FLEXURAL AND SHEAR PROPERTIES
OF GRAPHITE/EPOXY COMPOSITES

Test Temp (°F)	Water Boil Time (Hrs)	Flexural Strength (10 ³ psi)	Flexural Modulus (10 ⁶ psi)	Shear Strength (10 ³ psi)	Weight Gain (%)	Thickness Increase (%)
HT-S GRAPHITE/U.C.C. X-2546 EPOXY UNIDIRECTIONAL LAY-UP						
R.T.	-	200.0	18.4	15.1	-	-
350	-	193.7	18.7	9.0	-	-
350	2	178.8	17.1	9.0	0.86	0.83
350	16*	136.5	15.1	-	2.80	1.70
350	26	112.7	13.4	5.7	4.77	2.58
HT-S/X-2546 QUASI-ISOTROPIC LAY-UP						
R.T.	-	135.4	12.9	5.5	-	-
350	-	107.9	12.6	4.2	-	-
350	2	89.2	11.4	4.3	0.98	0.59
350	16*	84.8	11.3	3.5	2.17	1.34
HT-S GRAPHITE/FIBERITE EPOXY-PHENOLIC (X-911) QUASI-ISOTROPIC LAY-UP						
R.T.	-	142.4	18.0	6.9	-	-
350	-	119.3	18.2	5.0	-	-
350	22**	81.3	14.6	3.5	0.95	0.52
HT-S GRAPHITE/ERLA-4617 EPOXY UNIDIRECTIONAL LAY-UP						
R.T.	-	260.4	16.8	-	-	-
350	-	158.7	17.7	-	-	-
350	0.5*	134.7	16.0	-	0.80	0.45
HT-S/ERLA - 4617 QUASI-ISOTROPIC LAY-UP						
R.T.	-	136.2	11.6	7.2	-	-
350	-	20.9	5.7	1.8	-	-
350	0.5*	14.9	5.0	1.5	1.09	0.71

* Equivalent Water Boil

** Equilibrium Water Boil

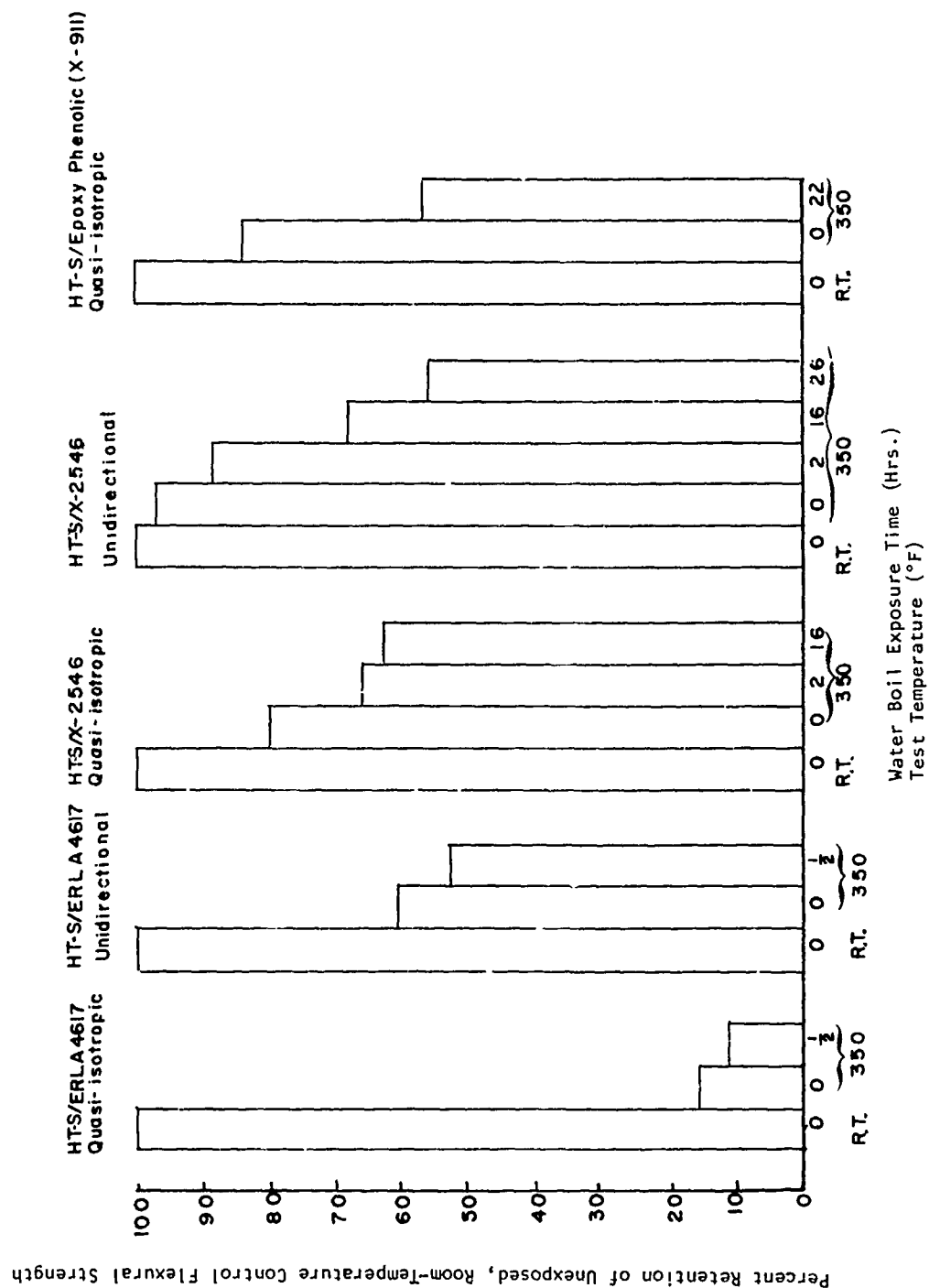


Figure 16. Effect of Water Boil on the Flexural Strength of Graphite/Epoxy Composites

Percent Retention of Unexposed, Room-Temperature Control Shear Strength

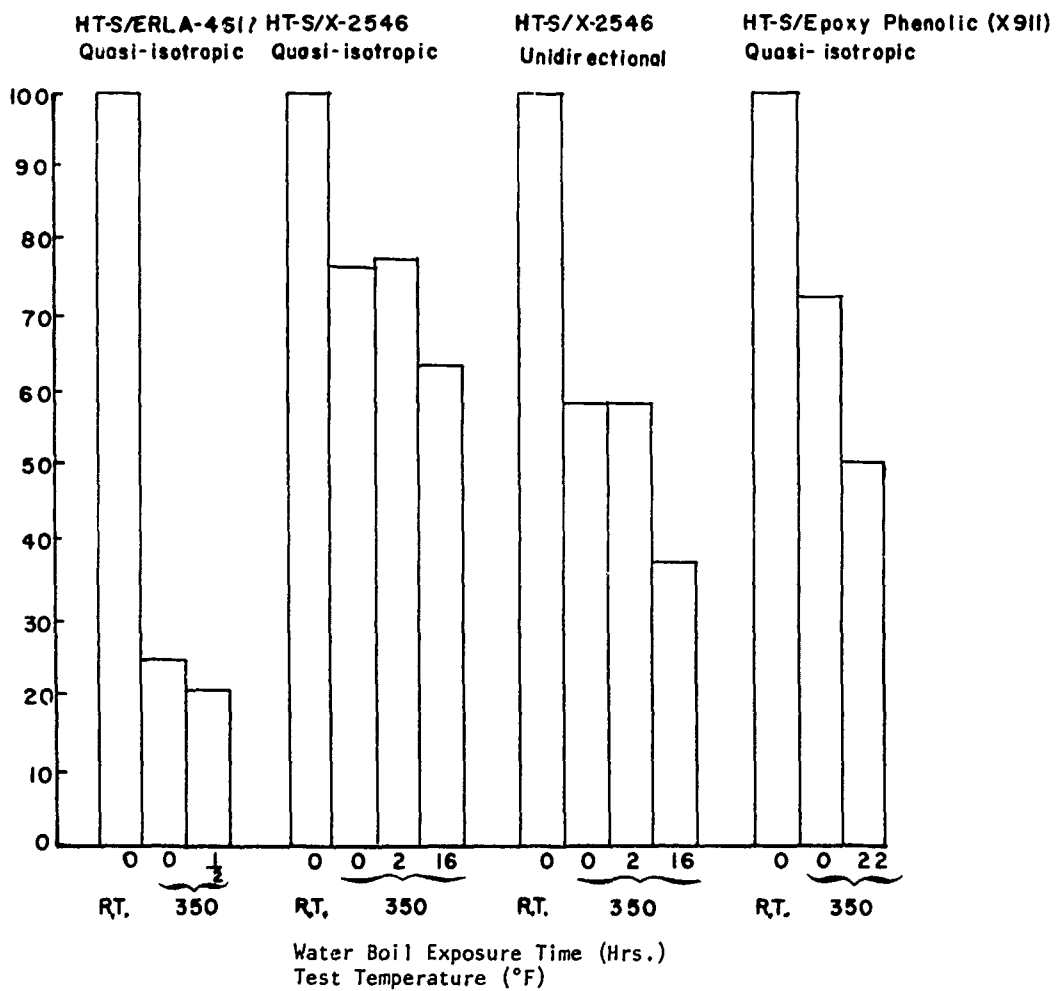


Figure 17. Effect of Water Boil on the Short-Beam Shear Strength of Graphite/Epoxy Composites

flexural strength is lowered only an additional 8%. The short-beam shear testing (Figure 17) on quasi-isotropic specimens produced results almost identical to those found for the flexural specimens. The weight gains for the unidirectional and quasi-isotropic specimens (0.80% and 1.09%, respectively) were equivalent to the weight gains found on the same systems after 30 environmental exposure cycles (Table II).

The second system evaluated was HT-S and U.C.C.'s X-2546 epoxy resin. Quasi-isotropic and unidirectional test specimens were water-boiled for up to 26 hours. The room-temperature data and 350°F data after water boil are presented in Table VIII and Figures 16 and 17. Comparisons show that a 16-hour water boil gives approximately the same weight gain as 30 exposure cycles (Table II). Flexure testing on quasi-isotropic laminates shows that the system loses 20% of the initial room-temperature strength when exposed to 350°F (Table VIII and Figure 16). The 350°F flex strength values decreased about 20% after 16 hours of water boil exposure. This exposure resulted in a weight gain of 2.7% and a thickness increase of 1.34%. Short-beam shear testing (Table VIII and Figure 17) of the quasi-isotropic specimens yielded results almost identical to those of the flex tests. Unidirectional specimens were water boiled for up to 26 hours, giving the property data shown in Table VIII and Figure 16 and 17. These specimens showed essentially no loss in flex strength when subjected to 350°F temperatures, but a 44% loss when exposed to a 26-hour water boil. Representative load-deflection curves from the flexural testing of unidirectional coupons are shown in Figure 18. The degree of specimen deflection does not change substantially as a function of temperature and/or exposure cycles. Short-beam shear specimens lost 41% of their strength due to exposure to 350°F temperature, then decreased approximately 20% more after 26 hours of water-boil exposure.

HT-S and Fiberite epoxy-phenolic was the last system evaluated. Quasi-isotropic lay-ups only were tested, and results are presented in Table VIII and Figures 16 and 17. This system was water boiled until maximum water absorption occurred (equilibrium water boil). An equilibrium

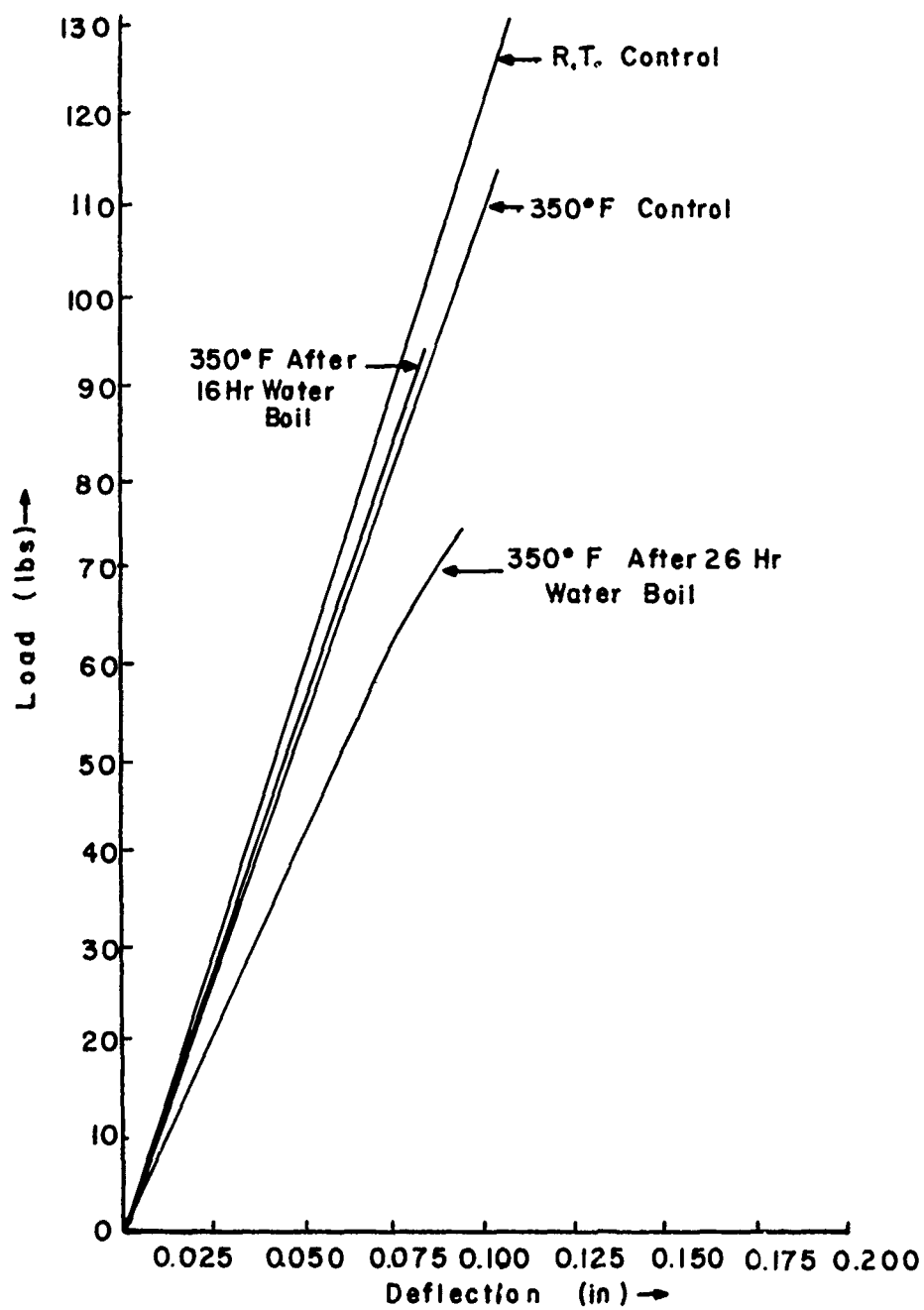


Figure 18. Load-Deflection Curves for Unidirectional HT-S/X-2546 Composite Test Specimens

weight gain of 0.95% occurred after 22 hours of water boil. The flexural loading of this system decreased only slightly (16%) upon exposure to 350°F. At the equilibrium point (22-hour water boil), the 350°F loadings had dropped 27%.

5. BORON COMPOSITES UNDER WATER-BOIL EXPOSURE

Boron composites utilized in this study were fabricated from NARMCO 5505 Boron/Epoxy prepreg. Unidirectional postcured composites were evaluated in flex and shear at room temperature, 350°F, and 350°F after exposure to a water boil. Resultant data are presented in Table IX and Figure 19.

These composites were exposed to an 11-hour water boil. Specimen weight gains were equivalent to the weight gains of unidirectional shear specimens after 30 environmental exposure cycles (Table VII). The flex strength (Figure 19) is lowered only slightly (12%) as a result of the 350°F temperature, but the 350°F flex strength is reduced 40% after exposure to the equivalent water boil. As a result of the moisture pick-up, the failure mode changed from tension to deformation by the loading pins; no fiber failure occurred. On cooling to room temperature, the specimens retained their deformed shape, as shown in Figure 20. The specimen tested at 350°F prior to water-boil exposure showed fiber failure in the bottom face of the composite (tensile failure), but those tested at 350°F after water-boil exposure showed no fiber failure, but were deformed into a curved shape associated with the loading pins. This change in failure mode is further illustrated by representative load-deflection curves from the flexural testing of unidirectional specimens given in Figure 21. The deflections for specimens at room-temperature and at 350°F with no water-boil exposure are approximately the same, while specimens tested at 350°F after water-boil exposure show a large increase in deflection at a comparatively lower load level. The short-beam shear specimens lost 52% of their initial room-temperature strength when subjected to the 350°F temperatures (Figure 19), but the value is lowered only 3% by the "equivalent" water-boil exposure of 11 hours, as shown in Table X.

TABLE IX
EFFECT OF WATER BOIL ON THE FLEXURAL AND
SHEAR PROPERTIES OF BORON/EPOXY COMPOSITES

Test Temp (°F)	Water Boil Time (Hrs)	Flexural Strength (10 ³ psi)	Flexural Modulus (10 ⁶ psi)	Shear Strength (10 ³ psi)	Weight Gain (%)	Thickness Increase (%)
NARMCO 5505 BORON/EPOXY - POSTCURED UNIDIRECTIONAL LAY-UP						
R.T.	-	247.1	31.0	14.4	-	-
350	-	217.4	26.8	6.9	-	-
350	11	119.1	19.8	6.7	0.81*	0.60

* WEIGHT GAIN IS EQUIVALENT TO WEIGHT GAIN DURING CYCLIC EXPOSURES

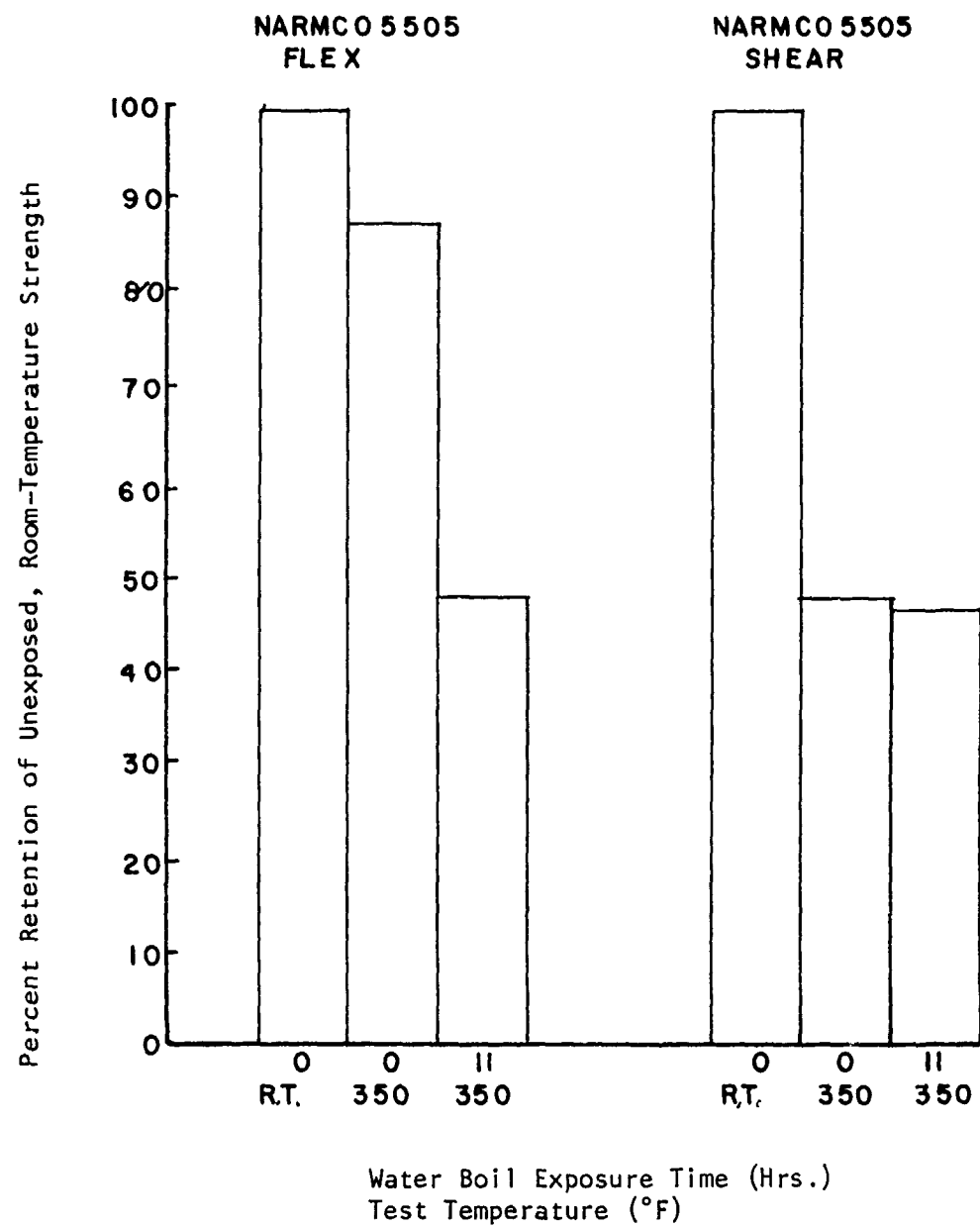


Figure 19. Effect of Water Boil on the Flexural and Short-Beam Shear Strength of Boron/Epoxy Composites



a. Tested at 350°F, No Water-Boil Exposure



b. Tested at 350°F After Equivalent Water-Boil Exposure



c. Tested at 350°F After Equivalent Water-Boil Exposure

Figure 20. Failed Boron/Epoxy Composite Test Specimens
Before and After Water-Boil Exposure

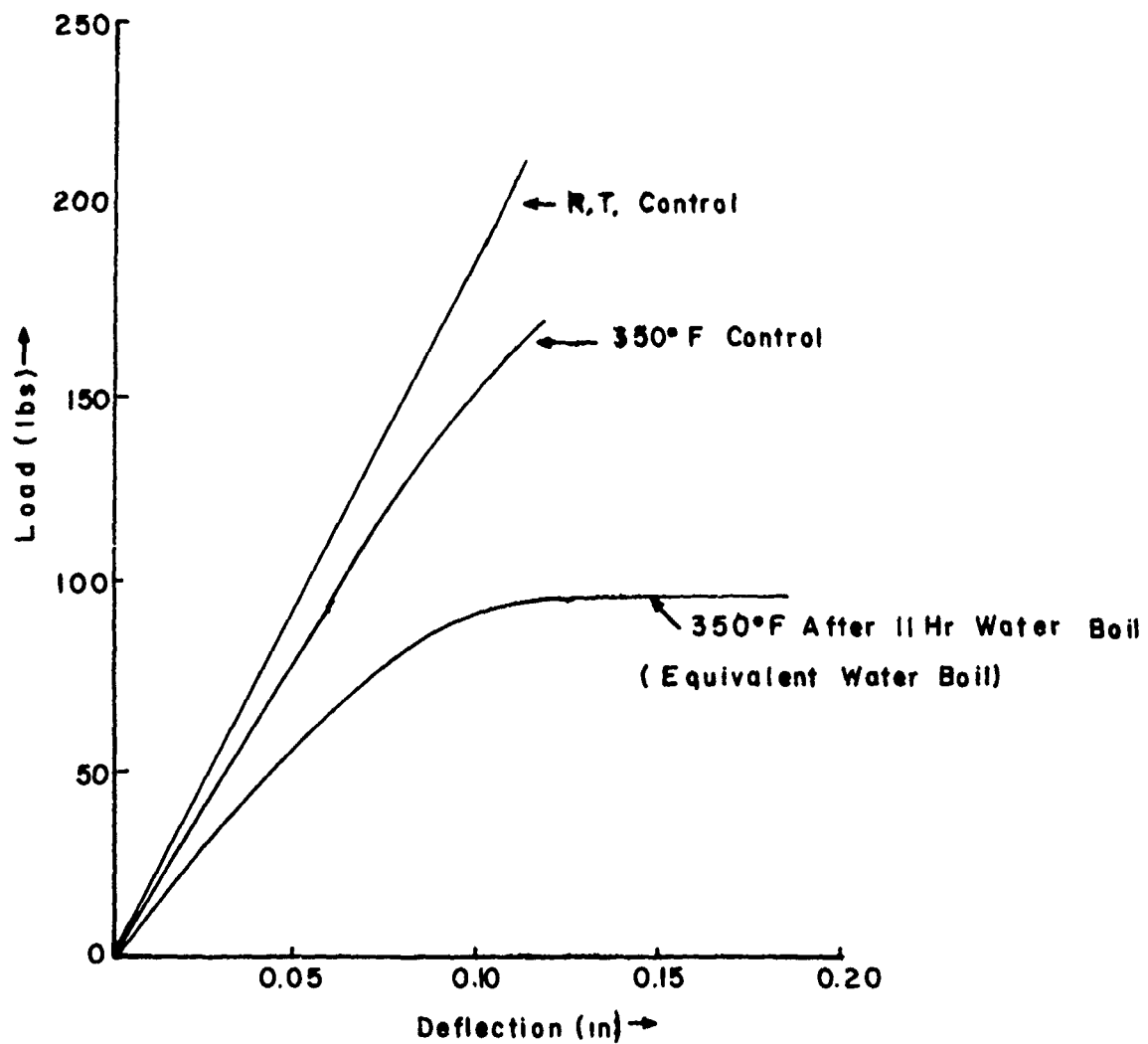


Figure 21. Load-Deflection Curves for Postcured Unidirectional Boron/Epoxy (NARMCO 5505) Composite Test Specimens

COMPARISON OF THE SHEAR STRENGTH OF WATER-BOILED AND CYCLED SPECIMENS OF NARMCO 5505 (BORON/EPOXY)

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SECTION IV

CONCLUSIONS

Several important conclusions may be garnered from this work.

1. The effect of absorbed moisture on the mechanical properties of composites at elevated temperature is determined principally by the lay-up of the laminate and/or the test being applied, i.e., the method by which load is introduced into the laminate. Thus, a given type of laminate undergoing a specific method of elevated-temperature mechanical testing may show no loss in the particular mechanical property (at temperature) even though it has absorbed a significant amount of moisture. On the other hand, this same system having a new lay-up, undergoing a different high-temperature mechanical test (different method of load introduction), and having absorbed an equivalent amount of moisture, may show a substantial loss in the particular mechanical property. This behavior is most aptly demonstrated by the boron/epoxy composite system. A quasi-isotropic laminate tested in tension at 350°F after 30 exposure cycles has essentially the same tensile properties as it did at 350°F prior to any exposure, even though it has absorbed a significant amount of moisture. This same boron/epoxy system, having a unidirectional lay-up and being tested in flexure, shows almost a 50% loss in its 350°F flexural strength after it has absorbed an amount of moisture equivalent to that picked up by the quasi-isotropic/tension laminate.
2. Water behaves in the manner of a plasticizing agent, apparently disrupting the strong hydrogen bonding present in the highly polar epoxy systems. Evidence for this are the reversibility of the water absorption effect and the change of failure mode from "dry" to "wet" specimens.
3. The test results after water-boil exposure can be correlated with those of high humidity exposure based on equivalent water weight gains.
4. The use temperatures of several resin systems are too close to their heat distortion temperatures.

5. The high heat-distortion-temperature resins are not as significantly affected by moisture at 350°F as are the lower heat-distortion-temperature resins.
6. The shear strengths, both dry and wet, of the boron/epoxy (NARMCO 5505) system are still within the specifications (room temperature and 350°F) set forth in the Structural Design Guide for Advanced Composite Applications (Reference 1).
7. The mechanical properties of both "wet" and "dry" composites (all systems) are essentially unaffected by temperatures up to 250°F.
8. The effect of moisture is a reversible one. Drying of "wet" test specimens restores the original dry strength.
9. The cast resin systems are not hydrolytically unstable, as evidenced by the reversibility of the moisture absorption process.
10. The ERLA-4617 cast resin system experiences a precipitous loss of 350°F tensile strength due to room-temperature aging.

REFERENCE

1. "Structural Design Guide for Advanced Composite Applications", November, 1968.

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13. ABSTRACT Graphite - and boron-fiber-reinforced composites, as well as castings of current resin systems, were evaluated to determine the effects of moisture and/or high humidity on their physical properties and their room and elevated temperature mechanical properties. Several of the resin systems investigated were not 350°F capability resins, even though they have been suggested for use in 350°F capability composites. All of the neat resin castings were found to absorb moisture and swell. Associated with moisture absorption is a loss in elevated temperature tensile strength, as demonstrated by the ERLA-4617 system which undergoes a precipitous loss of 350°F tensile strength as a result of ambient aging. All of the composite systems showed weight gains and thickness increases when subjected to a high humidity environment. However, the effect of absorbed moisture on the elevated temperature mechanical properties of composites is determined principally by the lay-up of the laminate and/or the test being applied, i.e., the method by which load is introduced into the laminate. This means that for a particular system, unidirectional composites may show a significant reduction of 350°F flexural strength due to absorbed moisture; however, for the same system, a multi-directional lay-up may show only a minor loss of 350°F tensile strength after equivalent moisture absorption (i.e., fiber-controlled-composite properties are relatively unaffected by absorbed moisture, whereas matrix-controlled properties are adversely affected). For both castings and composites the effects of moisture were found to be reversible.			

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