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# HYGROTHERMAL EFFECTS IN CONTINUOUS FIBRE REINFORCED COMPOSITES

## PART III: MECHANICAL PROPERTIES 1 STATIC TESTS

by

J. P. Komorowski

National Aeronautical Establishment

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**HYGROTHERMAL EFFECTS IN CONTINUOUS FIBRE  
REINFORCED COMPOSITES**

**PART III: MECHANICAL PROPERTIES 1 —  
STATIC TESTS**

**EFFETS HYGROTHERMIQUES DANS LES COMPOSITES  
À RENFORT DE FIBRE CONTINU**

**PARTIE III: PROPRIÉTÉS MÉCANIQUES 1 —  
ESSAIS STATIQUES**

by/par

J.P. Komorowski

National Aeronautical Establishment

OTTAWA  
SEPTEMBER 1983

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## SUMMARY

This is Part III of a series of literature reviews on hygrothermal effects on polymer matrix composite materials. It contains a review of papers on mechanical properties as measured in static tests and includes the effect of impact damage interaction with environmental conditions.

The other parts of the review are:

- Part I: Moisture and Thermal Diffusion
- Part II: Physical Properties
- Part IV: Mechanical Properties 2
- Part V: Composite Structures and Joints
- Part VI: Numerical and Analytical Solutions
- Part VII: Summary of Conclusions and Recommendations

A complete list of references is included in the Appendix and the numbers in the brackets appearing in the text refer to this list.

## RÉSUMÉ

Voici la partie III d'une série d'études documentaires traitant des effets hygrothermiques sur les matériaux composites à matrice de polymère. Elle contient une analyse des documents portant sur les propriétés mécaniques telles que mesurées lors d'essais statiques; les effets des conditions environnementales sur les dommages par chocs y sont également étudiés.

Les autres parties de cette série sont les suivantes:

- Partie I: Diffusion de l'humidité et de la chaleur
- Partie II: Propriétés physiques
- Partie IV: Propriétés mécaniques 2
- Partie V: Structures et joints composites
- Partie VI: Solution numériques et analytiques
- Partie VII: Résumé des conclusions et recommandations

Une liste complète des références est incluse en annexe et les nombres entre parenthèses dans le texte se rapportent à cette liste.

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## HYGROTHERMAL EFFECTS IN CONTINUOUS FIBRE REINFORCED COMPOSITES

### PART III: MECHANICAL PROPERTIES 1 - STATIC TESTS

#### 1.0 INTRODUCTION

During the past ten years numerous papers and reports have been published on the effects of moisture and temperature on polymeric matrix composite materials. The importance of environmental testing is recognized to the point where design allowables for existing materials or any new material cannot be developed without it<sup>[99, 125, 257]</sup>.

In this part of the review (Part III) results of mechanical tests on composite materials are reported. The tests of interest were tension, compression, torsion, in-plane shear, beam tests for interlaminar shear and flexure tests. Numerous types of specimen design were used in these tests and the properties reported are usually strength, modulus and notch or impact damage sensitivity. In order to assess the amount of degradation of mechanical properties which might be expected to occur due to temperature and humidity history, samples were often exposed to various conditions prior to and during the test.

Environmental effects on composite materials properties as measured in fatigue and creep tests will be reported in Part IV of the review.

Bolt bearing capabilities of composites in a varying environment will be reviewed in Part V.

#### 2.0 MATERIALS UNDER CONSIDERATION

The effects of moisture and temperature on a multitude of composite materials have been reported. Some of the materials studied are very exotic and have never reached commercial use, while others have been commercially available for several years. The results presented below should be treated as a rough guide to the effects on various types of fiber reinforced resins. The performance of composite materials may vary substantially depending on the molecular structure and processing history of the particular resin used, and this is illustrated in Figure 1 with reference to different composites fabricated from Thorneil Fabric 133. Terms like 'epoxy' or 'polyimide' resin are very loose descriptions of the matrix and cover a wide variety of chemical structures.

Clements and Leel<sup>[66]</sup> used unidirectional T300/5208 samples to study the effects of quality control variables such as batch variation, postcure and specimen quality under extreme moisture conditions. They found that in the fiber direction, differences between batches produced greater differences in strength and modulus than did the high level of absorbed moisture. However Chen and Hunter (Boeing)<sup>[59]</sup> pointed out that mechanical property tests are not adequate for quality assurance purposes. Slight differences in chemical content and cure quality may result in materials with equivalent short term mechanical properties, but having different rates of degradation due to the environment. For this reason Boeing is committed to very accurate material characterization methods supported by mechanical property tests.

The need for precise characterization of the resin has been recognized and a good example is the work of Lamothe, Halpin and Neall<sup>[185]</sup>, who were engaged in developing design allowables for glass/epoxy (S2-449/SP-250) for use in MILHDBK 174. Allowables include HPLC (High Pressure Liquid Chromatography) for checking component weight percent of SP-250 and FTIR (Fourier Transform Infrared Spectroscopy) for cure quality control. However, specimens used in most references under review have not been prepared under strict controls and therefore results should be treated as indications of the environmental stability of a given material.

### 3.0 TEST METHODS AND SPECIMEN DESIGN

Tension and beam tests are most frequently used. Tensile specimens are relatively easy to produce and the loading method is simple. Various specimen shapes have been used, and these are usually based on ASTM standards (i.e. ASTM 03039). These tests are primarily used to determine unidirectional tensile strength but off axis tensile specimens ( $10^\circ$  or  $\pm 45^\circ$ ) are also employed to measure changes in in-plane shear strength and modulus. Less common are notched shear specimens (see Fig. 2) also loaded in tension. Chapman<sup>[58]</sup> has described tension testing at elevated and cryogenic temperatures for unidirectional and  $\pm 45^\circ$  specimens of graphite/polyimide. Special attention was paid to the application of strain gauges.

The beam tests are of two types: flexure and short beam shear, they can both be either three or four point bend. The differences are in the ratio of span to depth. These are the easiest tests to prepare and perform under varying conditions, as tabs are not needed and the fixtures developed for room temperature can be used.

Torsion tests for shear were used by Adsit<sup>[5]</sup> with torsion tubes, while Philips, Scott, Buckley<sup>[233]</sup> and Hancox<sup>[122]</sup> used solid rod specimens.

Garcia and McWhitney<sup>[104]</sup> did both experiment and finite element analysis of rail shear test specimens made for graphite/polyimide and concluded that it is a good method for measuring the in-plane shear modulus but not strength.

For compression testing, several methods have been used in conjunction with environmental conditioning and this seems to be the most demanding of all tests. Good references for these types of test are Adsit<sup>[5]</sup>, Grimes<sup>[115]</sup>, Camarda<sup>[47]</sup> and Shuart<sup>[266]</sup>.

All of these tests provide information on mechanical properties which can be grouped into two categories -- fiber or matrix dominated properties. Whether the test provides the information on either of these property categories depends on both specimen loading and geometry (lay-up) i.e. a unidirectional specimen loaded in transverse direction to the fibers provides information on the matrix dominated properties. Interpretation is not straight forward as the specimen may fail in a mode where fiber splitting is dominant<sup>[11]</sup>. Generally, it can be said that  $0^\circ$  tension and flexure tests provide information on fiber dominated properties while other tests are matrix (or interface) dominated. This is important as most fibers demonstrate good resistance to temperature and moisture changes (Kevlar and other organic fibers are exceptions to this rule<sup>[11]</sup>) and it is usually the matrix or interface which shows degradation.

One of the great advantages of composite materials is the capability of changing laminate properties through various lay-up arrangements. The number of possible combinations, especially when hybridization is included, is almost infinite and it is not feasible to test all of them fully. Most frequently unidirectional and crossplied samples are tested and results from these tests are used with lamination theory to compute the effects of different lay ups. As residual and swelling strains significantly alter the overall performance of a laminate and are not easily accountable for in calculations, a limited number of tests must be conducted on the most frequently used lay-ups. The results of these tests are compared to theoretical results (lamination theory). Residual and swelling strains will be discussed in Part II of the series.

### 4.0 NOTCHED AND IMPACTED SPECIMENS

For practical applications the notch sensitivity of a material is an important factor. It also gives an indication of the damage and defect tolerance of the material. Low energy impact resulting in damage on the threshold of detectability has limited the design allowable strains to about  $4 \cdot 10^{-3}$   $\mu\text{mm/mm}$  in present day graphite/epoxies<sup>[99]</sup>. It has been shown that such damage will not propagate if post-impact strain values are kept below this limit.

Interactions between the notch, internal damage and the environment are other important factors requiring consideration. Wilkins<sup>316</sup>, Porter<sup>235</sup>, Baillie et al<sup>27</sup>, Lauraitis and Sandorff<sup>186</sup> all studied these type of interactions. Their results were similar despite the use of different materials, lay-ups and tests. Wilkins studied T300/5208 samples containing holes. Specimens with different lay-ups were saturated with water (1.5% weight gain) at 82°C and 98% RH. Tensile tests were carried out at room temperature and at 93°C on dry and wet specimens. Porter used T300/934 samples with various holes, notches and defects and three lay-up configurations. Conditioning was similar to that used by Wilkins except that instead of exposure at 98% RH immersion in water was utilized. Tensile testing was carried out at room temperature and 149°C with thermal spikes applied to reach testing temperature in 60 seconds and loading in 180 seconds after beginning of heat up. Baillie et al studied the influence of holes on cloth and tape laminates (HMF 330C/34 and AS/3501-6). Specimens were loaded in compression and their strength was determined at temperatures up to 157°C. Lauraitis and Sandorff also loaded their specimens in compression. They studied the interaction of moisture, low temperature and low level impact in AS/3501-6 material conditioned up to 1.29% and 1.75% weight gain of moisture.

The results of these tests led to the conclusion that notched or impacted material strength is only slightly dependent on lay-up and that there is no clear difference in notch or defect sensitivity as testing temperature or moisture content changes. This is probably because at higher temperatures notch stress concentration is reduced as well as the strength of the material. These results are significant and were best summed up by Porter<sup>235</sup> who concluded that environmental conditions generally have the greatest effect in the noncritical design condition of no defect. It has to be borne in mind, however, that only static loads were used in these tests. The same may not be true in fatigue tests.

## 5.0 TEST PROCEDURES -- TEMPERATURE AND HUMIDITY PROBLEMS

The test conditions which have been employed are: 1) constant temperature (high or low), 2) temperature cycling including thermal spikes, 3) constant humidity including immersion, 4) combinations of varying temperatures and humidities and 5) natural weathering on racks. Very few specimens have been made from components that have been in actual service for a number of years. This is partly due to the fact that data is often not available on the initial mechanical properties of many of the earlier composite materials employed.

Since advanced composite materials are exposed to a wide range of temperatures during their service (see Chapter 2 of Part I on environmental conditions), testing of conditioned specimens has to be carried out under similar ranges of temperature, and should include both the dry and wet states. Very often, in connection with determining mechanical properties, the question of reversability of property changes due to moisture is raised. Upon drying the property is usually restored or at least partially so. This is important in understanding the mechanisms of degradation, but it is irrelevant if one is only interested in developing design allowables, since, these should take into account the worst possible case.

Testing of composites at room temperature is difficult and standards have not been widely accepted for all types of tests. This applied specifically to shear and compression tests. The problems are even greater for tests conducted under hot/wet conditions. There is a paucity of data that might shed light on these problems. Some researchers have reported on the problems of conducting mechanical property tests in hot/wet conditions. Shen and Springer<sup>261</sup> studied the effects of temperature and moisture on the tensile strength of composites. They have used a computer program to estimate the thickness of the layer affected by three minutes of drying at different temperatures and the moisture loss of specimen in these conditions. Some of the results of these computations for T300/1034 system are presented in Figure 3. While loss of some moisture and consequently changes in moisture distribution during testing has to be expected, losses should be minimized. Frequently the time at temperature prior to testing is not reported and "soak times at temperature" of up to 30 minutes prior to testing are not uncommon.

Whitney and Husman<sup>[314]</sup> described the use of a flexure test for determining the environmental behavior of composites. Notably the authors recognized the need to control the weight loss. As 90 seconds were needed to bring their 8-ply T300/5208 and AS13501-5 specimens to temperature equilibrium, they calculated corresponding weight loss at the highest test temperature of interest. The authors assumed that the coefficient of diffusion is constant with temperature and calculated soak times at lower temperatures required to obtain similar weight losses.

## 6.0 FAILURE MODES

The ultimate goal of materials testing is development of data sets for designers (design allowables). For CM it became obvious that this data is closely related to the definition of failure. As failure mode is affected by the testing method and conditions in which it is carried out it is very important how the failure is defined. Tests carried out on CM structures have to be related to the design allowable data and this can only be done if the same failure has been induced in the structure as in the earlier coupon and element test.

Whitney and Husman<sup>[314]</sup> demonstrated that flexure testing provides a simple means for determining environmental conditions which induce significant changes in mechanical behavior related to flexure-stress conditions. Their results showed that moisture and temperature induce a change in failure mode from filament dominated to matrix dominated.

If the results obtained in hot/wet conditions are to be meaningful and provide a basis for quantitative assessment of the property loss relative to dry room temperature conditions, tests should be designed in such a way as to induce the same failure mode for all conditions. Crossman and Mauri<sup>[69]</sup> addressed this problem in connection with a interfacial shear strength hygrothermal degradation evaluation. Depending on the span to depth ratio used and on environmental conditions a bend test provides information on either the flexural strength or on the interlaminar shear strength. Crossman and Mauri demonstrated that the four point bend test gives wider than three point bend test range of span to depth ratios for which an interlaminar shear failure can be induced. Once the proper ratio is chosen for dry room temperature conditions failure should always be through interlaminar shear. This may not be true for flexure where a transition to shear failure may occur if span to depth ratios are not sufficiently high.

Changes in the failure mode with higher moisture content were also reported<sup>[267]</sup> for compression tests. In spite of great care in specimen preparation and test method design, tab failures in tension and compression tests are reported, i.e.<sup>[115]</sup>. Adsit<sup>[5]</sup> has discussed test methods and procedures that have been used at elevated temperatures for graphite/epoxy and polyimide composites. Tests include flexure, tension, compression and shear and test temperatures of up to 370°C are reported. This work should be of interest to anyone involved in environmental testing of CM.

## 7.0 FRACTURE SURFACE ANALYSIS

Miller and Wingert<sup>[217]</sup> demonstrated that despite the fact that fracture process is complicated, fracture surface analysis does yield information showing fundamental differences between resin systems and influences of environment. For this purpose scanning electron micrographs (SEM's) of gold plated specimens are most suitable, while optical microscopy on polished surfaces may provide additional information.

## 8.0 FRACTURE MECHANICS AND ENVIRONMENTAL EFFECTS ON PROPERTIES

Success in the application of fracture mechanics to composites has been limited since the models developed for metals cannot be directly applied. However, Beaumont and Harris<sup>[31]</sup> measured the work of fracture required to fracture unidirectional carbon fiber reinforced epoxies. They tried to establish the nature of the major energy absorbing fracture processes and to isolate mechanisms which influence crack motion. Some samples were exposed to water at 23°C and steam (100°C) up to two weeks prior to testing.

A similar approach was adopted by Kaelble et al in series of articles<sup>[157, 158, 159, 156]</sup>. They concentrated on developing theory and analysis of fracture energy and have studied the influence of degradation of interfacial bond strength upon fracture energy. Permanent degradation of bond strength was reported with a simultaneous increase in fracture energy.

Mandell<sup>[205]</sup> studied the effects of moisture on crack propagation in fiberglass laminates. While water immersion caused reduced static and fatigue tensile strength and consequently increased crack extension rates under cycling loading, it greatly reduced crack extension under static loading. It was shown by the author that this was due to moisture increasing the intraply delamination region in the damage zone thus reducing the local fiber stresses. For cyclic loading, this region is large even in dry conditions and reduction of material strength plays a dominant role.

## 9.0 EFFECT OF HUMIDITY AND TEMPERATURE ON VARIOUS MATERIALS

### 9.1 Graphite and Boron Composites for Service Up to 200°C

Degradation due to exposure at constant temperature for extended periods of time has been studied by Haskins, Kerr and Stein<sup>[166, 127]</sup>. After 10,000 hours at 121°C samples of AS/3501 in  $[0]_6$  and  $[0/\pm 45]_{56}$  lay-up which were tested at 177°C showed no degradation of tensile strength. Similar samples aged at 177°C after 1000 hours began to degrade, and after 10,000 hours demonstrated 20% and 57% decrease in strength at 177°C when compared to unaged samples. The greater amount of degradation was observed for a  $[0/\pm 45]_{56}$  lay-up.

Kong et al<sup>[174]</sup> observed the effect of quenching from above the glass transition temperature ( $T_g$ ) and subsequent  $T_g$  quenching for up to  $10^5$  min. on T300/5208 ( $\pm 45$ )<sub>4S</sub> laminates. Decrease of ultimate tensile strength (32%), strain to failure (93%), and toughness (68%) after  $10^5$  min. were interpreted as physical aging effects and were explained in terms of decreases in free volume and the tendency to move towards thermodynamic equilibrium in the glassy state.

Thermal cycling effects were reported by Camahort, Rennhack and Coons<sup>[46]</sup>. Samples were cycled 25 times between -196°C (liquid nitrogen) and 100°C (boiling water). Materials used were HMF 330C/934 fabric and HMS/934, HMS/3501 and HMS/759 unidirectional tape graphite/epoxies. All these materials were specified as 177°C cure but in order to reduce residual stresses were cured at 135°C. Some samples were made from HMS/339 which is a 121°C cure material. Microcracking was severe in all but the 121°C cured materials. However, there was no loss in RT tensile properties with some improvement in transverse tensile strength. These results are not surprising as TTT diagrams for thermosets indicate that the materials were not fully cured.

Mazzio and Mehan<sup>[209]</sup> thermally cycled HTS surface treated fibers in Epon 828 (manufactured by Shell) cured with hexahydrophthalic anhydride (HHPA) and benzyldimethylamine (BOHA) (100:78:1). The cycle temperatures were between -53°C and 149°C of which 250 and 500 were slow cycles and 500 were fast cycles (see Fig. 4). Results are presented in Figure 5. Mazzio and Mehan prepared some samples with a lower volume fraction of fibers and studied residual stress effects optically. Fibers after cure were in compression for low  $v_f$ , and occasionally fiber buckling was observed. During cycling some of these stresses may relieve themselves by breaking molecular bonds and as a result mechanical properties will change. A decrease in compression strength seemed to confirm this but the authors appear to have contradicted themselves by saying that interlaminar shear strength increased through improved interfacial action. No effect of cycling rate was observed.

Loos and Springer<sup>[198]</sup> studied the effect of thermal spiking on Graphite-Epoxy T300/1034 composites. The material properties studied included moisture absorption, tensile strength and buckling modulus. No effect was observed in the properties that were studied. Loos and Springer compiled results of other researchers (see Table 1) and concluded that the effect of spiking depends on the particular material system being studied. In most cases, the effect is small with fiber dominated properties especially insensitive. See also<sup>[200, 201]</sup>.

In<sup>[267]</sup> Shyprykevich and Wolter determined compressive strength as a function of moisture content. AS/3501-5A was used in two different lay-ups and testing was performed at room and elevated (127°C) temperatures. At higher humidities (>1.2% wt gain) thermal spiking clearly degraded the material, and this was related to glass transition temperature (T<sub>g</sub>) exceedences. Thermal spiking caused higher moisture absorption which lowered the compressive strength and also changed the failure mode.

Browning, Husman and Whitney<sup>[41]</sup> tested AS/3501-5 in various lay-ups after near equilibrium moisture gain at 71°C (75% and 95% RH). Test temperatures were RT, 93°C, 121°C and 149°C. Typical results are presented in Figure 6. Browning et al. suggested three possible mechanisms of degradation: 1) matrix plasticization associated with T<sub>g</sub>, 2) degradation due to residual and swelling stresses and 3) possible degradation of the interface. Tests on neat 3501-5 led Browning et al to conclude that room temperature properties are degraded due to matrix crazing.

Shen and Springer<sup>[260, 261]</sup> studied the effect of moisture and temperature on tensile strength and buckling moduli of T300/1034 in various lay-ups. Different conditioning temperatures and humidities were employed. The moisture distributions and losses due to testing in high temperature were calculated. Typical results are presented in Figure 7 and were compared to previously reported data (see Table 2 and 3). Results are consistent and indicate that:

- a) Temperature and moisture had little effect on fiber dominated longitudinal strength below 100°C and slightly decreased strength at 180°C. Elastic moduli was not affected regardless of moisture and temperature up to 180°C.
- b) Transverse properties were severely degraded with increase in temperature and moisture and both the modulus and strength maybe degraded by as much as 90% of their original values.
- c) Distribution of moisture in 0° and quasi-isotropic laminates did not seem to affect the results. For 90° specimens the moisture distribution may have influenced the absolute value of ultimate strength and modulus but it was unlikely to have affected the trend in the data.

Bohlmann and Derby<sup>[38]</sup> used sandwich beam specimens to test T300/339 fabric (HMF-330B) in the warp direction. Specimens were conditioned at 82°C and 70% RH and tested at both room temperature and 177°C.

Grimes<sup>[115]</sup> made extensive studies of AS/3501-6 material in compression. As different lay-ups were tested, various loading fixtures were utilized. Testing was conducted at room (22.8°C) and elevated (103°C) temperatures, with specimens in dry (0 - 0.4% wt gain) and wet (1.1 ± 0.2% wt gain) states. Typical results are shown in Figure 8.

Laumitis and Sandorff<sup>[187]</sup> studied the effect of environment on the compressive strength of T300/5208 and AS/3501-5A in various lay-ups. Specimens were soaked up to equilibrium wt gain at 82°C - 90% RH and tested in -54°C, 22°C, 93°C and 135°C (above "wet" T<sub>g</sub>). Behavior of graphite/epoxy laminates was divided into three regimes:

- 1) Long column range — elastic stability.
- 2) Very short column — specimens fully supported failure by crushing, delamination (compression ultimate).
- 3) Intermediate short column — inelastic failures resulting in rupture.

It was found that in regime 1) the behavior of the laminate was independent of temperature and humidity while in 2) and 3) the environment affected results depending upon material and lay-up.

Ekvall and Griffin<sup>[99]</sup> performed over 2000 coupon tests to develop design allowables for T300/5208 tape and fabric laminates with a 3/16-inch hole, at -54°C, room temperature dry and 82°C wet (1% weight gain condition). Data were analyzed statistically to determine the best fit probability distribution and the results at the lower tail of the test data were compared to the minimum values obtained from different regression equations. An overall best fit equation (in this case normal distribution) provided good agreement near the lower tail of the distribution. Final design allowables for worst environmental conditions (in this project), 3/16-inch diameter notch and nonvisible impact damage were calculated (Table 4) and included a statistical reduction factor (B allowable factor). This methodology can be used for other composite materials.

Several authors published data from shear tests conducted on pre-conditioned samples. Philips, Scott and Buckley<sup>[233]</sup> used rod specimens prepared from graphite, glass and Kevlar 49 fibers in HY750 epoxy resin. Results are shown in Table 5 and Figure 9. Crossman, Mauri and Warren<sup>[69]</sup> used four point beam tests to study interlaminar shear properties of T300/5208 and T300/5209 specimens. Conditioning was 55% RH or 85% RH at 70°C with some samples dried. Testing was conducted at 20°C, 70°C and 93°C. Several specimens were subjected to temperature cycles varying from -56°C to 70°C or 93°C. The results led the authors to conclude that neither testing temperature nor conditioning significantly degraded T300/5208 dried samples. However, T300/5209 showed significant loss of interlaminar shear strength (ILSS) after humid hot conditioning and this seemed to be related to the exceedence of T<sub>g</sub> (5209 is a 121°C cure material). For low moisture gain T300/5209 does not seem to be affected.

Halloff<sup>[121]</sup> tested short beam shear (SBS) strength of T300/5208, HT-S/3501 and Fibredux 914C. Samples were subjected to different and sometimes severe temperature and humidity treatments. However, Halloff concluded that the chemical composition of the polymer system most significantly influenced the effects of heat treatments and moisture absorptions of composite materials.

Lifshitz<sup>[193]</sup> measured longitudinal, transverse and axial shear properties of T300/5208 laminates. The test program included three strain rates, three temperatures and three levels of moisture. The matrix properties were influenced by all those parameters with axial shear exhibiting the most variability (Fig. 10). Poisson's ratio  $\nu_{12}$  did not change in these tests, however, the value was slightly less than usual.

Augl<sup>[123]</sup> presented the effects of moisture and test temperature on T300/5208 as demonstrated in beam tests. Figure 11 demonstrates the usefulness of three dimensional (3D) graphs in reporting degradation of strength due to moisture and temperature.

All above reported tests were done on samples subjected to arbitrarily chosen conditions. It is not known whether materials in real service, subjected to atmospheric conditions, would be degraded to the same extent. To gain knowledge on the natural weathering of composites, several materials have been exposed on racks in loaded and unloaded state and some tests were carried out on specimens recovered from composite structures after extended period of service.

Trabocco and Stander<sup>[291]</sup> used two exposure sites (Panama Canal and Warminster, PA) to represent tropical and temperate climates. Specimens were either painted or unpainted and loaded in flexure with strain at 4000  $\mu\text{mm/mm}$ . Tests were carried out at room temperature and at 177°C (Fig. 12). The materials that were used in this work have been replaced by those currently used in aerospace industry (AS/3501-6, T300/5208 and AS/3004). The results for these new materials were reported by Vadala and Trabocco<sup>[294]</sup>. Two aircraft carriers operating in the Pacific were included as exposure sites. Tests were carried out at room temperature, 82.2°C and 121.1°C with materials originally intended for 177°C service limited to 121.1°C. Tension, flexure, compression (IITRI compression fixture) and SBS tests were carried out. Weight gains were monitored but specimens were kept in sealed bags prior to testing. However, sealed bags do not prevent changes in moisture distribution. Typical test results are shown in Table 6 and Figure 13. In general Panama exposure was slightly more severe than at Warminster. The rate of decrease of strength varies for each material system with AS/3004 (graphite/polysulfone) showing better resistance to environment than epoxies.

In<sup>[57]</sup> Chapman, Hoffman and Hodges and in<sup>[88]</sup> Dexter and Chapman presented some results obtained by NASA for commercial aircraft composite laminates. Composites were exposed at ground exposure sites around the world and in actual service. No significant deterioration was observed owing mainly to mild testing conditions. Information on specimen lay-up and of failure modes was not given. See also<sup>[29, 199, 30]</sup>.

Gibbins and Hoffman in<sup>[110]</sup> report on a long term program by Boeing aimed at developing accelerated aging methods for composites used in commercial aircraft. Various graphite fiber and one Kevlar fiber in epoxy materials were tested after exposure to actual flight conditions, on ground and in accelerated laboratory chambers conditions. In<sup>[110]</sup> results obtained up to now are presented (2 years of the projected 10 years of total program duration).

Coggeshall in<sup>[67]</sup> reported on the Boeing 737 graphite composite spoiler flight service evaluation. This is the seventh annual report on a program involving 114 spoilers installed on 27 aircraft operating around the world. Various types of materials were used in the spoilers and samples are also exposed on the ground. Periodically some spoilers and ground exposed samples are mechanically tested. No significant deterioration of properties has been reported up to now.

## 9.2 Glass Composites for Service Up to 200°C

Glass composites have been available since the 1950's and studies on environmental degradation of these materials were initiated in the 1960's. The degrading effect of moisture and temperature on matrix dominated properties is similar to the graphite reinforced composites which basically depends on type of resin used.

It has been found that glass fibers are degraded by water due to a reaction between alkali ions in the glass and water which in turn affects the siloxan bond (the structural backbone of glass), forming an alkaline medium on the surface of the glass. If uncoated glass is used in a resin matrix the rapid migration of water along the fiber interface is observed. This is paralleled by degradation of the interface and tensile strength of the fiber. Present day glass fibers are used in strongly-bonded polymeric coatings and a considerable amount of research has concentrated on the reinforcement mechanism of glass fiber reinforced plastics. Ishida and Koenig<sup>[142]</sup> reviewed literature on this subject with an emphasis on the microscopic aspects (molecular structures of glass/matrix interfaces). The glass/matrix interface was studied as a three-phase system and includes a) glass/coupling agent interface, b) coupling agent and c) coupling agent/matrix resin interface. Each of these phases was studied individually using modern spectrometers.

Antoon and Koenig<sup>[141]</sup> used Fourier-transform infrared (FTIR) spectroscopy to identify irreversible chemical effects of moisture on highly cross-linked anhydride-cured epoxy resin. The effects of high tensile stresses and presence of filler were also investigated. It was concluded that hydrolytic attack of water on the ester linkages was accelerated in the alkaline media, enhanced by the presence of inorganic filler (glass fiber) and was a mechanically activated process (external or residual stresses). Further studies of the interface were reported by Koenig in<sup>[172]</sup>. Spectroscopic studies should be correlated with mechanical property changes and are indispensable in understanding degradation mechanisms. A good example of such a study was work by Ishai<sup>[141]</sup> where samples of glass fabric/epoxy were subjected to several absorption-desorption cycles and their wrap and fill strength was tested in tension. Infrared spectroscopy was used to analyze the water during immersion and distinct traces of silica compounds were found. Ishai pointed out that caution is called for in using elevated temperatures for accelerated testing, since correlation between short-term effects and their long-term counterparts at low temperatures is not clear.

Scola<sup>[255]</sup> studied shear and flexure properties of three types of S-glass/epoxy composites and their resins after various temperature and humidity conditions. The typical results are presented in Figure 14. One interesting observation was that the principal cause of composite shear strength loss due to water is weakening or debonding of the resin-fiber interface, however, the strength and modulus changes of the resin appear to be insignificant in the degradation process. Some degrading influence of the resin was found by Scola in a further study<sup>[254]</sup>.

Nicholas and Ashbee<sup>[226]</sup> studied the effect of freezing or boiling of phase-separated water, and concluded that: 1) non-spherical water-filled cavities, specifically disc shaped cavities present at water soluble inclusions propagate as cracks during the volume expansion associated with the water-to-ice transition, 2) generation of the osmotic pressure is also responsible for the observed failure of phase-separated water to boil during high temperature excursions.

Docks and Buck<sup>[92]</sup> subjected several glass fiber/resin composites to thermal cycling and grouped the resins according to their susceptibility to degradation in these conditions. Some vinyl or modified vinyl resins seemed to perform best.

Rao et al<sup>[238]</sup> measured the effect of moisture and glass contents on the Poisson's ratio of glass fiber reinforced plates. Laser interferometry was used to measure Poisson's ratio and the results are in Figure 15.

Lamothe, Halpin and Neal<sup>[185]</sup> developed design allowables for glass/epoxy (S2-449/SP-250). After the test specimens were subjected to different preconditioning cycles, tension, compression and SBS tests were carried out, Table 7.

Lubin and Donohue<sup>[199]</sup> presented unique and encouraging data from tests carried out on samples of glass fiber composites cut from aircraft structures which were in service for up to 19 years and the results were compared with data obtained at the time the structures were manufactured. Some of the results are in Tables 8 and 9. Most of specimens showed excellent strength retention, but, only a few of these parts were exposed to temperatures over 82°C which may be the reason for the low degradation.

### 9.3 Aramid (Kevlar) Fiber Reinforced Composites

Despite the growing amount of Kevlar composites being used in aerospace and other industries relatively little work has been published on hydrothermal effects on these composites. Kevlar fibers are organic and, in contrast to carbon or glass fibers, they absorb moisture.

Wu<sup>[322]</sup> studied strength degradation in filament wound Kevlar 49/XD7818, Jeffamine T403 epoxy. Samples tested in longitudinal and transverse tension, and in longitudinal compression were the flat coupon type. Transverse compression and shear was measured on tube specimens while biaxial tests were performed on pressurized tube specimens. All specimens were dried in desiccant and then subjected to one of the following conditions: 52% RH at 23°C, water at 23°C or water at 100°C until moisture equilibrium was reached. The typical effect of moisture on ultimate strength is shown in Figure 16, which indicates that substantial degradation occurs for room temperature water immersion. (However, in service this is not likely to occur as much lower wt gains are observed.)

Wu concluded that due to large differences between tensile and compressive strengths and different degrees of degradation in the fiber-controlled vs matrix controlled strengths which caused the failure surface to shift and deform, a polynomial third order tensor strength criterion would be more suitable. However, this required four additional biaxial experiments to determine coefficients for the polynomial.

Allred<sup>[10]</sup> studied the effects of temperature and moisture content on flexural response of two Kevlar 49 fabric (181)/epoxy laminates (5208 and Ferro CE-9000 both 190°C cure) in two lay-ups [0/90] and [ $\pm 45$ , 0/90]. The testing temperature range was from -55°C to 150°C. Both materials exhibited fairly similar properties. For both lay-ups the results indicated that Kevlar 49/epoxies have temperature dependent mechanical properties over the range investigated, and that moisture increased temperature sensitivity. Load vs deflection for [0/90] CE-9000 is shown in Figure 17.

In flexure tests at 150°C and near the moisture saturation content (5% wt), a loss in strength of 60-70% and effective stiffness loss of 40% was recorded. At 21°C the saturated specimens were 35-40% weaker than dry material. Specimens having a [ $\pm 45$ , 0/90] lay-up seemed to be slightly less sensitive to moisture. Failure modes which were distinctively different than for other composites were reported, i.e. compression buckling of filaments of [0/90] or tensile delamination for the [ $\pm 45$ , 0/90] lay-up.

For the [ $\pm 45$ , 0/90] lay-up the effects of voids, long term moisture exposure, freeze-thaw cycling and reversibility of moisture exposure on drying was also investigated. All these conditions were found to degrade laminate strength and degradation due to moisture exposure was found to be irreversible (dried specimens had the same strength as specimens saturated with moisture at room temperature).

Deteresa et al<sup>[86]</sup> used single filament techniques to establish the critical length of Kevlar 49 fiber in poly(amide (Nylon 6) film. Tests were performed at room temperature. It is surprising that soaking in water seemed to shorten the critical length apparently strengthening the bond between the fiber and matrix, but further studies are required to clarify this point.

Humphrey et al<sup>[136]</sup> subjected Kevlar 49, filament wound composites with various resins to 71°C and 95% RH. After 21 days of exposure specimens tested in short beam shear demonstrated reduction in strength from 29% to 46% depending on type of resin.

Kevlar 49/Fiberite 934 epoxy composite, in transverse direction was tested by Allred and Roylance<sup>[111]</sup>. At 25°C moisture saturation (> 5% wt gain) caused a 35% decrease in ultimate strength and 25% decrease in elongation. Stiffness was found to be less sensitive with a 14% decrease from the dry room temperature value. Microscopy of the fracture surfaces revealed that property reductions were accompanied by a change in failure mode from an interface dominated one in dry condition to a filament splitting mode in the moisture saturation condition.

In 1973, Lockheed under a contract from NASA manufactured and installed Kevlar 49 fairing panels in three L1011 wide body transports. Concurrently, NASA runs ground-based exposure tests on Kevlar 49 coupons. Reference<sup>[276]</sup> is the eighth annual flight service evaluation report which includes the ground-based coupon test results. The Kevlar 49 fairings continue to perform satisfactorily and no major damage or defects have been observed after eight years of service. However, these components are lightly loaded and so far no tests have been performed to assess the amount of degradation of properties or to measure the moisture content. Tests were carried out only on the ground exposed samples. It was found that moisture contents stabilized after five years at slightly over 2% wt gain. No degradation in flexural strength was found while shear and compressive strengths after five and seven years decreased in the 15% to 20% range.

There are other on-going service evaluation programs, i.e.<sup>[30]</sup> which should provide more information on degradation of Kevlar composites.

#### 9.4 Composites for Service Above 200°C

An excellent review on synthetic resin matrices for use up to 300°C can be found in a recent book by Delmonte<sup>[84]</sup>. Recent papers have been reviewed which discuss moisture effects in these materials.

One of earliest works of the effects of water on the properties of a glass/polyimide laminate was reported by DeJasi<sup>[83]</sup>. After 1200 hours of exposure to 100°C water or 100% RH specimens made of 7781 glass fabric with 1100S finish and Monsanto's Skybond 709 polyimide resin lost 90% of their flexural strength when tested in dessicated state. The calculated value of the activation energy of the degradation process indicates selective hydrolysis of the matrix material followed by a breakdown of the polymer-fiber interface.

Lisagor<sup>[194]</sup> studied the effect of moisture on short beam shear and compression strengths in HTS2/PMR15 and Celion 6000/PMR15. Specimens were conditioned to saturation at 100% RH and 82°C and tested at -96°C, 21°C and 316°C. Vacuum drying of as processed samples produced improved mechanical properties. Results for "wet" samples should be compared with dessicated samples, i.e. for compression strength compare Figures 18 and 19. Moisture conditioning of graphite/polyimide composites produced moderate to severe reduction in compressive and interlaminar shear properties at 316°C. This degradation appeared to be associated with the lowering of the Tg of the matrix.

Rummier and Clark<sup>[250]</sup> presented results of thermal aging of HTS/710 for times up to 25,000 hours. Specimens were tested in tension at the aging temperature. The results indicate that HTS/710 material shows degradation after 1000 hours at 288°C and after 10,000 hours it was severely degraded. For lower aging temperature (232°C) no degradation was observed after 25,000 hours. Thus the maximum service temperature for HTS/700 should be reduced to 232°C for long time application in aircraft such as supersonic transports.

Scola<sup>[256]</sup> investigated the effect of thermal aging and moisture on several composite systems consisting of addition type polyimides PMR-11, PMR-15, P13N in combination with the fiber reinforcements S-glass, Thormel 300, HMS and HTS graphite fibers. PMR-11 and P13N systems proved to be fairly resistant to degradation due to moisture and PMR-11 systems demonstrated good flexural and shear strength retention after 2500 hours in air at 288°C.

Pater<sup>[22]</sup> showed that PMR resins modified with N-phenylmaleimide had superior properties to standard PMR-15. Tests included isothermal exposure at 315°C for up to 1500 hours in air and hygrothermal exposure for 360 hours in 95% RH at 82°C, followed by flexural and shear strength testing at 315°C.

For graphite/polyimides strength degradation is directly associated with fiber thermo-oxidative resistance while stiffness retention seems to be controlled by the thermal stability of the matrix in flexural and shear tests<sup>[181]</sup>.

Serafini and Hanson<sup>[259]</sup> investigated the effects of thermo-oxidative and hydrothermal exposure on T300/PMR-15 and HTS2/PMR-15 composites. It was very difficult to carry out tests in "wet" condition at 200°-300°C since desorption at these temperatures was very rapid. This cast doubt on the validity of other elevated temperature "wet" results. The case of loaded and soaked material being subjected to temperature spike is not unlikely in actual service and greater strength reductions for this case can be expected.

## 10.0 CONCLUSIONS AND RECOMMENDATIONS

Many conclusions can be reached from the preceeding sections and only the most important ones are listed below along with recommendations for further work proposed by the reviewer.

- (i) There is a growing need for standards for the testing of composites. The areas that have to be addressed are:
  - a) Standard methods are required for material characterization, including chemical content and cure quality.
  - b) Specimen and fixture designs are required which take into account the hot/wet conditions used in testing.
  - c) Standard methods are required for predicting the realistic moisture contents which can be expected during service.
  - d) Standard methods are required for preconditioning the sample before testing in wet conditions.
  - e) Standard technique of testing under hot/wet conditions (temperature gradients, humidities, loading rates, etc.).
  - f) Greater emphasis should be placed on the statistical analysis of data.

- (ii) Environmental conditions generally have the greatest effect on the noncritical design condition of no defect. There is no clear difference in notch or defect sensitivity as testing temperature and moisture content changes.
- (iii) Slight differences in chemical content and cure quality may result in materials with equivalent short term mechanical properties but different environmental sensitivities.
- (iv) Materials used in most references have not been accurately characterized and therefore the material properties reported may only be treated as indications. This is especially true of environmental sensitivity of composite materials.
- (v) Maximum service temperature for some epoxies, formerly advertised as 177°C (350°F) should be lowered generally by 50°C due to hot/wet properties being greatly reduced.
- (vi) Fiber dominated properties seem to be little affected by moisture and temperature.
- (vii) Results of tests on samples exposed either on the ground or taken from structures that were in actual service for several years, show little degradation due to environmental exposure. However, most of these samples were only lightly loaded.

**TABLE 1**  
**SUMMARY OF EXPERIMENTAL DATA ON THE EFFECTS OF THERMAL SPIKES**  
**ON GRAPHITE/EPOXY COMPOSITES<sup>[198]</sup>**

| Material        | Reference              | Absorption<br>Behavior | Tensile<br>Strength | Compres-<br>sion<br>Strength | Shear<br>Strength | Flexural<br>Strength | Buckling<br>Modulus | Tensile<br>Modulus | Fatigue |
|-----------------|------------------------|------------------------|---------------------|------------------------------|-------------------|----------------------|---------------------|--------------------|---------|
| T300/1034       | Present work           | N                      | N                   |                              |                   |                      | N                   |                    |         |
| T300/934        | Bohlmann-Dergy [1]     | N                      |                     |                              |                   |                      |                     |                    |         |
|                 | Reinhart [2]           | N                      |                     |                              |                   |                      |                     |                    |         |
| T300/5208       | McKague et al [3]      | L                      |                     |                              |                   |                      |                     |                    |         |
|                 | Kibler [4]             | L                      |                     |                              |                   |                      |                     |                    |         |
|                 | Augl [5]               |                        |                     |                              |                   | N                    |                     |                    |         |
|                 | Lundemo-Thor [6]       |                        | S                   |                              |                   |                      |                     |                    | L       |
| T300/5209       | Stoecklin* [7]         |                        | S                   | N                            | N                 | S                    |                     |                    |         |
| T300/2544       | Stoecklin* [7]         |                        | S                   | S                            | S                 | S                    |                     |                    |         |
| T400/2544       | Trabocco-Stander* [8]  |                        | N-L                 | S                            | N                 |                      |                     |                    |         |
| AS/3501         | Stoecklin* [7]         |                        | S                   | S                            | N                 |                      |                     |                    |         |
|                 | Trabocco-Stander* [8]  |                        |                     | N                            |                   |                      |                     |                    |         |
| AS/3501-5       | Delsai-Whiteside [9]   | N                      |                     |                              |                   |                      |                     |                    |         |
| AS/X-2546       | Browning-Hartness [10] |                        |                     |                              |                   | L                    |                     |                    |         |
| HMS/339         | Camahort et al [11]    | N                      |                     |                              |                   |                      | N                   |                    |         |
| HMS/934         | Camahort et al [11]    | S                      |                     |                              |                   |                      | N                   |                    |         |
| HMS6759         | Camahort et al [11]    | S                      |                     |                              |                   |                      |                     | N                  |         |
| HMS/3501        | Camahort et al [11]    | N                      |                     |                              |                   |                      |                     | N                  |         |
| HMS/X-2546      | Browning-Hartness [10] |                        |                     |                              |                   | L                    |                     |                    |         |
| HTS/3002        | Trabocco-Stander* [8]  |                        |                     |                              | N                 |                      |                     |                    |         |
| HTS/4617        | Browning-Hartness [10] |                        | N                   | N                            | S                 |                      |                     |                    |         |
| HTS/ADX 516     | Browning-Hartness [10] |                        | N                   | N                            | S                 |                      |                     |                    |         |
| HTS/P 13 N      | Browning-Hartness [10] |                        | N                   | N                            | S                 |                      |                     |                    |         |
| HTS/X-2546      | Browning-Hartness [10] |                        | N                   | L                            |                   | L                    |                     |                    |         |
| Modmor II/5206  | Trabocco-Stander* [8]  |                        |                     |                              | N                 |                      |                     |                    |         |
| Narmco 2387(nr) | Browning-Hartness [10] |                        | L                   |                              |                   |                      |                     |                    |         |
| ERL 2256(nr)    | Browning-Hartness [10] |                        | S                   |                              |                   |                      |                     |                    |         |
| ERLA 4617(nr)   | Browning-Hartness [10] |                        | L                   |                              |                   |                      |                     |                    |         |
| X2546(nr)       | Browning-Hartness [10] |                        | L                   |                              |                   |                      |                     |                    |         |

N - negligible effect; S - small effect; L - large effect; (nr) neat resin; \* - weathering test

NOTE: Numbers in square brackets refer to literature in [198]

**TABLE 2**  
**SUMMARY OF EXPERIMENTAL DATA ON THE EFFECTS OF MOISTURE AND**  
**TEMPERATURE ON THE ULTIMATE TENSILE STRENGTH OF COMPOSITES<sup>[361]</sup>**

| Composite                                | Reference                | Laminate Lay-Up Orientation |      |       |      |       |      | Remarks                                                                             |
|------------------------------------------|--------------------------|-----------------------------|------|-------|------|-------|------|-------------------------------------------------------------------------------------|
|                                          |                          | 0°                          |      | 45°   |      | 90°   |      |                                                                                     |
|                                          |                          | Moist                       | Temp | Moist | Temp | Moist | Temp |                                                                                     |
| Thornel 300/Fiberite 1034                | Shen & Springer, 1976    | L                           | N    | L     | N    | S     | S    | Limited data<br>(2-3 points)<br>Two data points<br>for 90° laminates                |
| Hercules AS-5/3501                       | Browning et al, 1976 [3] | N                           | N    | N     | N    | S     | S    |                                                                                     |
|                                          | Verette, 1975 [4]        | N                           | N    | N     | -    | S     | S    |                                                                                     |
|                                          | Kerr, et al, 1975 [5]    | -                           | N    | -     | N    | -     | S    |                                                                                     |
| Thornel 300/Narmco 5208                  | Kim & Whitney, 1976 [6]  | -                           | -    | N     | N    | -     | -    | Very scattered data<br>for 90° laminates<br>Only two data points<br>for temperature |
|                                          | Hofer et al, 1975 [7]    | L                           | L    | N     | L    | S     | S    |                                                                                     |
|                                          | Husman, 1976 [8]         | -                           | -    | -     | -    | S     | L    |                                                                                     |
| Modmor II/Narmco 5206                    | Hofer et al, 1974 [9]    | N                           | L    | N     | L    | S     | S    |                                                                                     |
| Courtaulds HMS/Hercules 3002M            | Hofer et al, 1974 [9]    | N                           | N    | N     | N    | S     | S    |                                                                                     |
| HT-S/ERLA-4617                           | Browning, 1972 [10]      | -                           | -    | L     | S    | -     | -    |                                                                                     |
| HT-S/Fiberite X-911                      | Browning, 1972 [10]      | -                           | -    | N     | N    | -     | -    |                                                                                     |
| HT-S/U.C.C.X-2546                        | Browning, 1972 [10]      | -                           | -    | L     | N    | -     | -    |                                                                                     |
| PRD 49/ERLB-4617                         | Hanson, 1972 [11]        | -                           | L    | -     | -    | -     | -    |                                                                                     |
| HT-S/(8183/137-NDA-BF <sub>3</sub> :MEA) | Hertz, 1973 [12]         | -                           | -    | -     | -    | S     | S    |                                                                                     |
| HT-S/Hysol ADX-516                       | Browning, 1972 [10]      | -                           | -    | N     | S    | -     | -    |                                                                                     |
| Hercules HT-S/710 Polyimide              | Kerr, et al, 1975 [5]    | -                           | N    | -     | N    | -     | N    |                                                                                     |
| HT-S/P13N Polyimide                      | Browning, 1972 [10]      | -                           | -    | -     | L    | -     | -    | Only two data points<br>for 90° laminates                                           |
| Boron/AVCO 5505                          | Hofer, et al, 1974 [9]   | L                           | N    | L     | L    | S     | S    |                                                                                     |
| Boron/Narmco 5505                        | Kaminski, 1973 [13]      | -                           | L    | -     | -    | -     | S    |                                                                                     |
|                                          | Browning, 1972 [10]      | -                           | -    | N     | N    | -     | -    |                                                                                     |

(a) N = Negligible effect      (b) L = Little effect (< 30%)      (c) S = Strong effect (> 30%)

(a) N = Negligible effect    (b) L = Little effect (< 30%)    (c) S = Strong effect (> 30%)

NOTE: Numbers in square brackets refer to literature 'n' [198]

**TABLE 3**  
**SUMMARY OF EXPERIMENTAL DATA ON THE EFFECTS OF MOISTURE AND**  
**TEMPERATURE ON THE ELASTIC MODULUS OF COMPOSITE MATERIALS [200]**

| Composite                                                                              | Reference                | Laminate Lay-Up Orientation |      |       |      |       |      |
|----------------------------------------------------------------------------------------|--------------------------|-----------------------------|------|-------|------|-------|------|
|                                                                                        |                          | 0°                          |      | 45°   |      | 90°   |      |
|                                                                                        |                          | Moist                       | Temp | Moist | Temp | Moist | Temp |
| BUCKLING TEST                                                                          |                          |                             |      |       |      |       |      |
| Thornel 300/Fiberte 1034                                                               | Shen & Springer 1977     | N                           | N    | N     | N    | S     | S    |
| TENSILE TEST                                                                           |                          |                             |      |       |      |       |      |
| Hercules AS-5/3501                                                                     | Browning, et al 1976 [5] | L                           | N    | L     | N    | S     | S    |
|                                                                                        | Verette 1975 [6]         | N                           | N    | N     | -    | S     | S    |
|                                                                                        | Kerr et al 1975 [7]      | -                           | N    | -     | N    | -     | -    |
| Thornel 300/Harmco S208                                                                | Hofer et al 1975 [8]     | N                           | N    | N     | N    | N     | N    |
|                                                                                        | Husman 1976 [9]          | -                           | -    | -     | -    | S     | S    |
| Modmor 11/Harmco S206                                                                  | Hofer et al 1974 [10]    | N                           | N    | N     | N    | S     | S    |
| Courtaulds HMS/Hercules 3002N                                                          | Hofer et al 1974 [10]    | N                           | N    | N     | N    | N     | S    |
| HT-S/ERLA-4617                                                                         | Browning 1972 [11]       | -                           | -    | N     | S    | -     | -    |
| HT-S/Fibersce X-911                                                                    | Browning 1972 [11]       | -                           | -    | N     | N    | -     | -    |
| HT-S/UCC X-2546                                                                        | Browning 1972 [11]       | -                           | -    | N     | L    | -     | -    |
| PRD-49/ERLA-4617                                                                       | Hansom 1972 [12]         | -                           | S    | -     | -    | -     | -    |
| HT-S/(8185/137-NDA-BF3: MEA)                                                           | Mertz 1973 [13]          | -                           | -    | -     | -    | N     | S    |
| TENSILE TEST                                                                           |                          |                             |      |       |      |       |      |
| HT-S/HS-01 ADX-516                                                                     | Browning 1972 [11]       | -                           | -    | N     | S    | -     | -    |
| HT-S/710 Polyimide                                                                     | Kerr et al 1975 [7]      | -                           | N    | -     | N    | -     | -    |
| HT-S/PIV Polyimide                                                                     | Browning 1972 [11]       | -                           | -    | -     | L    | -     | -    |
| Boron/WCO S505                                                                         | Hofer et al 1974 [10]    | N                           | N    | N     | N    | S     | S    |
| Boron/Harmco S505                                                                      | Browning 1972 [11]       | -                           | -    | N     | N    | -     | -    |
| COMPRESSIVE TEST                                                                       |                          |                             |      |       |      |       |      |
| Hercules AS-5/3501                                                                     | Verette 1975 [6]         | N                           | N    | -     | -    | L     | S    |
| Thornel 300/Harmco S208                                                                | Hofer et al 1975 [8]     | L                           | N    | N     | N    | L     | N    |
| Modmor 11/Harmco S206                                                                  | Hofer et al 1974 [10]    | N                           | N    | N     | N    | S     | S    |
| Courtaulds HMS/Hercules 3002N                                                          | Hofer et al 1974 [10]    | N                           | N    | N     | N    | S     | S    |
| Boron/WCO S505                                                                         | Hofer et al 1974 [10]    | N                           | N    | N     | N    | S     | S    |
| a) N = Negligible effect<br>b) L = Little effect (<50%)<br>c) S = Strong effect (>50%) |                          |                             |      |       |      |       |      |

**NOTE:** Numbers in square brackets refer to literature in [200]

**TABLE 4**  
**GRAPHITE/EPOXY TAPE AND FABRIC LAMINA DESIGN**  
**ALLOWABLES\* [181]**

| LOAD<br>DIRECTION | 0° PROPERTY                     | MATERIAL |        |
|-------------------|---------------------------------|----------|--------|
|                   |                                 | TAPE     | FABRIC |
| TENSION           | MODULUS · 10 <sup>8</sup> PSI   | 20.8     | 8.8    |
|                   | STRENGTH · 10 <sup>3</sup> PSI  | 97.4     | 38.2   |
|                   | STRAIN · 10 <sup>-6</sup> IN/IN | 4750     | 3900   |
| COMPRESSION       | MODULUS · 10 <sup>8</sup> PSI   | 18.8     | 8.7    |
|                   | STRENGTH · 10 <sup>3</sup> PSI  | 74.0     | 34.8   |
|                   | STRAIN · 10 <sup>-6</sup> IN/IN | 4000     | 4000   |

\*WORST ENVIRONMENTAL CONDITION. 3/16 IN DIA  
NOTCH OR IMPACT DAMAGE

**TABLE 5**  
**STATIC SHEAR PROPERTIES OF THE COMPOSITE MATERIALS AFTER**  
**CONDITIONING [233]**

| Material | Treatment   | Shear Strength<br>MPa <sup>-2</sup> | Shear Modulus<br>GPa <sup>-2</sup> | Shear Strain<br>(x 10 <sup>-3</sup> ) |
|----------|-------------|-------------------------------------|------------------------------------|---------------------------------------|
| CFRP     | As-received | 76 ± 9 (8)                          | 3.5 ± 0.6 (8)                      | 39 ± 11 (8)                           |
|          | Soaked      | 61 ± 6 (4)                          | 3.4 ± 0.2 (4)                      | 25 ± 10 (4)                           |
|          | Dried       | 72 ± 9 (4)                          | 3.5 ± 0.2 (4)                      | 29 ± 4 (4)                            |
|          | Annealed    | 81 ± 13 (4)                         | 3.7 ± 0.6 (4)                      | 37 ± 11 (4)                           |
| GRP      | As-received | 79 ± 3 (5)                          | 3.8 ± 0.2 (3)                      | 67 ± 4 (5)                            |
|          | Soaked      | 55 ± 6 (4)                          | 4.3 ± 0.2 (4)                      | 40 ± 8 (4)                            |
|          | Dried       | 63 ± 7 (4)                          | 4.0 ± 0.4 (3)                      | 31 ± 4 (4)                            |
|          | Annealed    | 82 ± 2 (3)                          | 3.5 ± 0.3 (3)                      | 37 ± 4 (3)                            |
| KFRP     | As-received | 48 ± 4 (6)                          | 1.5 ± 0.1 (6)                      | 27 ± 3 (6)                            |
|          | Soaked      | 27 ± 2 (4)                          | 1.4 ± 0.3 (4)                      | 34 ± 1 (4)                            |
|          | Dried       | 33 ± 2 (3)                          | 1.3 ± 0.1 (4)                      | 31 ± 5 (4)                            |
|          | Annealed    | 34 ± 2 (4)                          | 1.4 ± 0.1 (4)                      | 27 ± 1 (3)                            |

Uncertainties are standard deviations

Figures in brackets are the number of specimens

**TABLE 6**  
**RESULTS OF EXPOSURE ABOARD US CONSTELLATION AND NIMITZ**  
**CARRIERS OF AS/3501-6 MATERIAL<sup>(204)</sup>**

|                          | <u>Unpainted<br/>Unexposed</u> | <u>US CONSTELLATION<br/>Exposure</u> | <u>NIMITZ<sup>*</sup><br/>Exposure</u> |
|--------------------------|--------------------------------|--------------------------------------|----------------------------------------|
| <b>Tensile Strength</b>  |                                |                                      |                                        |
| <b>P.S.I.</b>            |                                |                                      |                                        |
| R.T.                     | 55831                          | 62922                                | 58821                                  |
| 82.2°C                   | 57432                          | 61674                                | 63292                                  |
| 121.1°C                  | 61016                          | 60111                                | 57767                                  |
| <b>Shear Strength</b>    |                                |                                      |                                        |
| <b>P.S.I.</b>            |                                |                                      |                                        |
| R.T.                     | 6257                           | 6149                                 | 6015                                   |
| 82.2°C                   | 5964                           | 6157                                 | 6807                                   |
| 121.1°C                  | 6108                           | 5920                                 | 4276                                   |
| <b>Flexural Strength</b> |                                |                                      |                                        |
| <b>P.S.I.</b>            |                                |                                      |                                        |
| R.T.                     | 53232                          | 52178                                | 56489                                  |
| 82.2°C                   | 52389                          | 47253                                | 59933                                  |
| 121.1°C                  | 53544                          | 46164                                | 49191                                  |

\* Nuclear Carrier

**TABLE 7**  
**SUMMARY OF MECHANICAL PROPERTIES OF SP-350/S-2 FIBERGLASS EPOXY<sup>(188)</sup>**

|                                     |                              |       |                             |                      |                                    |                                 |                   |
|-------------------------------------|------------------------------|-------|-----------------------------|----------------------|------------------------------------|---------------------------------|-------------------|
| Fabrication                         | Layup:<br>0°, quasiisotropic |       | Pressure:<br>50 PSI         | Cure:<br>30 min/250F | Post Cured:<br>90 min/250F         | Plies:<br>8,12,16               |                   |
| Physical Properties                 | Weight % Resin:<br>50.1      |       | Density:<br>1.85            | Avg. % voids<br>0.53 |                                    | Avg. Thickness:<br>0.009 in/ply |                   |
| Test Methods                        | Tension:<br>ASTM D-3039      |       | Compression:<br>ASTM D-3410 |                      | Interlaminar Shear:<br>ASTM D-2344 |                                 |                   |
| Temperature                         | 75F                          |       |                             |                      |                                    | 160F                            |                   |
| Condition                           | 0% RH<br>1 Month             |       | 50% RH<br>3 Months          |                      | 95% RH<br>3 Months                 |                                 | 95% RH<br>1 Month |
|                                     | Avg                          | SD    | Avg                         | SD                   | Avg                                | SD                              | Avg SD            |
| Tension, QI*                        |                              |       |                             |                      |                                    |                                 |                   |
| ultimate stress, ksi                | 74.32                        | 4.94  | 75.56                       | 3.00                 | 62.87                              | 3.42                            | 48.26 3.09        |
| ultimate strain, %                  | 3.48                         | 0.44  | 3.61                        | 0.19                 | 2.75                               | 0.06                            | 2.07 0.13         |
| lower modulus, 10 <sup>6</sup> psi  | 3.50                         | 0.28  | 3.22                        | 0.13                 | 3.31                               | 0.18                            | 3.34 0.18         |
| upper modulus, 10 <sup>6</sup> psi  | 1.80                         | 0.15  | 1.77                        | 0.10                 | 1.89                               | 0.15                            | 1.93 0.10         |
| secant modulus, 10 <sup>6</sup> psi | 2.12                         | 0.14  | 2.15                        | 0.15                 | 2.26                               | 0.14                            | 2.32 0.10         |
| Tension, 0°                         |                              |       |                             |                      |                                    |                                 |                   |
| ultimate stress, ksi                | 228.62                       | 16.00 | 222.90                      | 15.43                | 174.25                             | 13.20                           | 141.53 14.16      |
| ultimate strain, %                  | 3.31                         | 0.43  | 3.55                        | 0.17                 | 2.78                               | 0.12                            | 2.25 0.07         |
| secant modulus, 10 <sup>6</sup> psi | 6.43                         | 0.56  | 6.37                        | 0.31                 | 6.41                               | 0.37                            | 6.49 0.25         |
| Compression, QI**                   |                              |       |                             |                      |                                    |                                 |                   |
| ultimate stress, ksi                | 73.11                        | 3.73  | 72.75                       | 3.47                 | 67.81                              | 3.56                            | 62.76 3.88        |
| ultimate strain, %                  | 2.96                         | 0.34  | 3.06                        | 0.13                 | 2.73                               | 0.23                            | 3.10 0.35         |
| secant modulus, 10 <sup>6</sup> psi | 2.55                         | 0.19  | 2.49                        | 0.30                 | 2.31                               | 0.18                            | 2.31 0.50         |
| Interlaminar Shear, 0°              |                              |       |                             |                      |                                    |                                 |                   |
| ultimate stress, ksi                | 9.72                         | 0.76  | 9.38                        | 0.64                 | 9.44                               | 0.67                            | 7.74 0.60         |

\* (0/+45/-45/90)<sub>8</sub>

\*\* (0/+45/-45/90)<sub>28</sub>

**TABLE 8**  
**FLEXURAL TESTS ON E-2A ROTODOME, SERIAL NO. 1 AT 25°C(199)**

| TEST AREA                                               | AGED/NORMALIZED (0.011" PLY) |      |      |      | ORIGINAL DATA (POLAR PLOTS) |     |      |      |
|---------------------------------------------------------|------------------------------|------|------|------|-----------------------------|-----|------|------|
|                                                         | STR.                         |      | MOD. |      | STR.                        |     | MOD. |      |
|                                                         | KSI                          | MPa  | MSI  | GPa  | KSI                         | MPa | MSI  | GPa  |
| UPPER SKIN -<br>PAINTED                                 | 53.8                         | 371  | -    | -    | 61.5                        | 424 | -    | -    |
|                                                         | 56.6                         | 390  | -    | -    | 63.5                        | 438 | -    | -    |
|                                                         | 57.0                         | 393  | -    | -    | 63.5                        | 438 | -    | -    |
|                                                         | AVG.                         | 55.8 | 384  | -    | 62.8                        | 433 | -    | -    |
| PERCENT RETENTION                                       | 49                           |      | -    |      | -                           |     | -    |      |
| BOTTOM SKIN -<br>PAINTED                                | 76.5                         | 526  | 2.34 | 16.1 | 64.0                        | 441 | 2.75 | 18.9 |
|                                                         | 65.9                         | 545  | 2.37 | 16.3 | 57.5                        | 396 | 2.30 | 15.8 |
|                                                         | 76.4                         | 526  | 2.37 | 16.3 | 62.0                        | 427 | 2.50 | 17.2 |
|                                                         | AVG.                         | 72.9 | 502  | 2.36 | 61.2                        | 422 | 2.52 | 17.4 |
| PERCENT RETENTION                                       | 100+                         |      | 93.7 |      | -                           |     | -    |      |
| UPPER SKIN -<br>NO PAINT                                | 57.2                         | 394  | 1.88 | 13.0 | 63.0                        | 434 | 1.9  | 13.1 |
|                                                         | 59.6                         | 411  | 1.88 | 13.0 | 66.0                        | 455 | 2.0  | 13.8 |
|                                                         | 59.9                         | 413  | 1.90 | 13.0 | 67.3                        | 464 | 2.1  | 14.5 |
|                                                         | AVG.                         | 58.9 | 406  | 1.89 | 65.9                        | 454 | 2.0  | 13.8 |
| PERCENT RETENTION                                       | 39.4                         |      | 94.5 |      | -                           |     | -    |      |
| INNER SKIN -<br>NO PAINT MOIS-<br>TURE CONTENT: 1.1%    | 53.5                         | 369  | 1.36 | 9.4  | 61.5                        | 424 | 1.8  | 12.4 |
|                                                         | 56.3                         | 391  | 1.45 | 10.0 | 59.0                        | 407 | 1.9  | 13.1 |
|                                                         | 57.3                         | 395  | 1.63 | 11.2 | 60.0                        | 413 | 1.7  | 11.7 |
|                                                         | AVG.                         | 55.9 | 385  | 1.48 | 60.2                        | 415 | 1.8  | 12.4 |
| PERCENT RETENTION                                       | 92.8                         |      | 82.2 |      | -                           |     | -    |      |
| CAP -<br>NO PAINT, ERODED<br>MOISTURE CONTENT:<br>0.84% | 43.9                         | 302  | -    | -    | 65.0                        | 488 | -    | -    |
| PERCENT RETENTION                                       | 67.5                         |      | -    |      | -                           |     | -    |      |

TABLE 9

TENSILE TESTS ON E-2A ROTODOME, SERIAL NO. 1, 25°C<sup>(199)</sup>

| TEST AREA             | TESTED & FRACTURED (0.0117/PLY) |     |         |      | ORIGINAL DATA (FOR OR FLOTS) |     |      |      |
|-----------------------|---------------------------------|-----|---------|------|------------------------------|-----|------|------|
|                       | Strength                        |     | Modulus |      | Str.                         |     | Mod. |      |
|                       | ksi                             | MPa | MSI     | GPa  | ksi                          |     | MSI  |      |
| Butt - Skin - Painted |                                 |     |         |      |                              |     |      |      |
| Moisture              | 37.2                            | 256 | 2.07    | 14.3 | 32.0                         | 220 | 2.1  | 14.4 |
| Content: 1.32%        | 25.3                            | 174 | 1.98    | 13.6 | 32.0                         | 220 | 1.7  | 11.7 |
|                       | 34.4                            | 237 | 1.94    | 13.4 | 31.5                         | 215 | 2.2  | 15.2 |
|                       | 35.1                            | 242 | 2.89    | 13.0 | 36.5                         | 251 | 2.3  | 15.8 |
|                       | 37.8                            | 260 | 1.85    | 13.0 | 36.5                         | 251 | 2.3  | 15.8 |
| AVG.                  | 34.0                            | 234 | 1.95    | 13.4 | 33.7                         | 232 | 2.1  | 14.4 |
| Percent Retention     | 100+                            |     | 92.9    |      |                              |     |      |      |
| Upper Skin Painted    |                                 |     |         |      |                              |     |      |      |
| Moisture              | 39.5                            | 272 | 1.83    | 12.6 | 36.3                         | 250 | 2.3  | 15.8 |
| Content: 1.46%        | 42.9                            | 296 | 2.09    | 14.4 | 39.4                         | 271 | 2.5  | 17.2 |
|                       | 43.5                            | 300 | 2.27    | 15.6 | 38.4                         | 265 | 2.6  | 17.9 |
| AVG.                  | 42.0                            | 289 | 2.06    | 14.2 | 38.0                         | 262 | 2.5  | 17.2 |
| Percent Retention     | 100+                            |     | 82.4    |      |                              |     |      |      |
| Upper Skin No Paint   |                                 |     |         |      |                              |     |      |      |
| Moisture              | 35.0                            | 241 | 1.77    | 12.2 | 30.0                         | 255 | 2.5  | 17.2 |
| Content: 1.00%        | 34.0                            | 234 | 1.88    | 13.0 | 40.0                         | 276 | 2.8  | 19.3 |
|                       | 38.0                            | 265 | 1.94    | 13.4 | 39.0                         | 269 | 2.5  | 17.2 |
|                       | 32.3                            | 223 | 1.74    | 12.0 | 40.0                         | 276 | 2.5  | 17.2 |
|                       | 38.5                            | 265 | 1.98    | 13.6 | 40.0                         | 276 | 2.7  | 18.6 |
| AVG.                  | 35.7                            | 246 | 1.86    | 12.8 | 39.0                         | 269 | 2.6  | 17.9 |
| Percent Retention     | 91.5                            |     | 71.5    |      |                              |     |      |      |

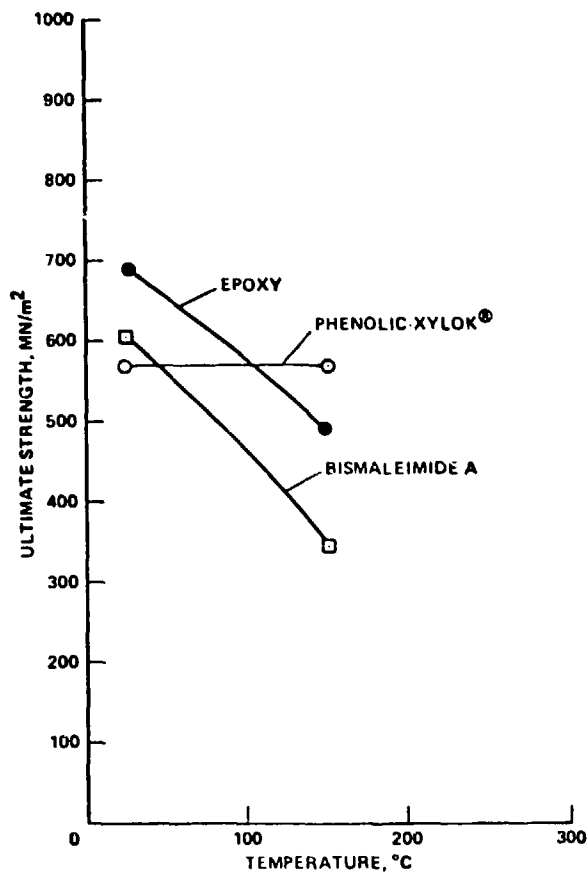


FIG. 1: EFFECT OF TEMPERATURE ON TENSILE STRENGTH OF GRAPHITE COMPOSITES<sup>[178]</sup>

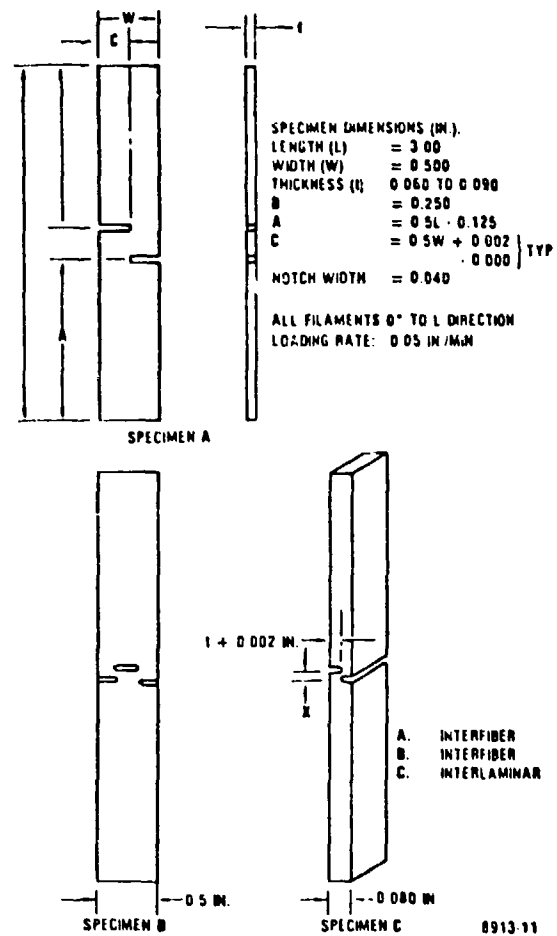


FIG. 2: SHEAR TEST SPECIMEN<sup>[5]</sup>

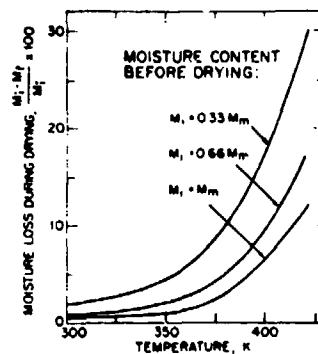


FIG. 3: MOISTURE LOSS OF SPECIMEN DURING THREE MINUTES OF DRYING AT DIFFERENT TEMPERATURES.  $M_i$  AND  $M_f$  ARE THE MOISTURE CONTENTS BEFORE AND AFTER DRYING.  $M_m$  DENOTES MOISTURE CONTENT AT FULL SATURATION[261]

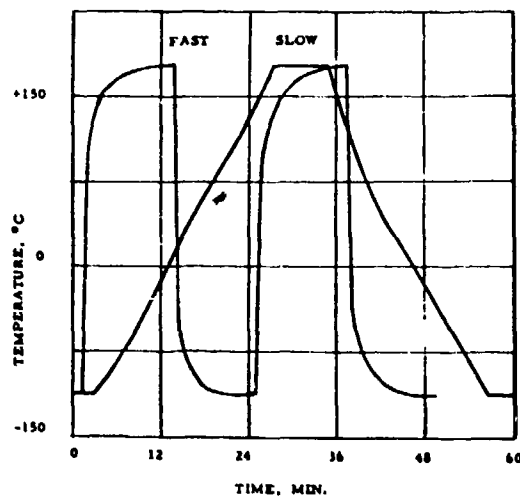


FIG. 4: SCHEMATIC OF THERMAL CYCLES FOR TEMPERATURE CYCLING SYSTEMS[268]

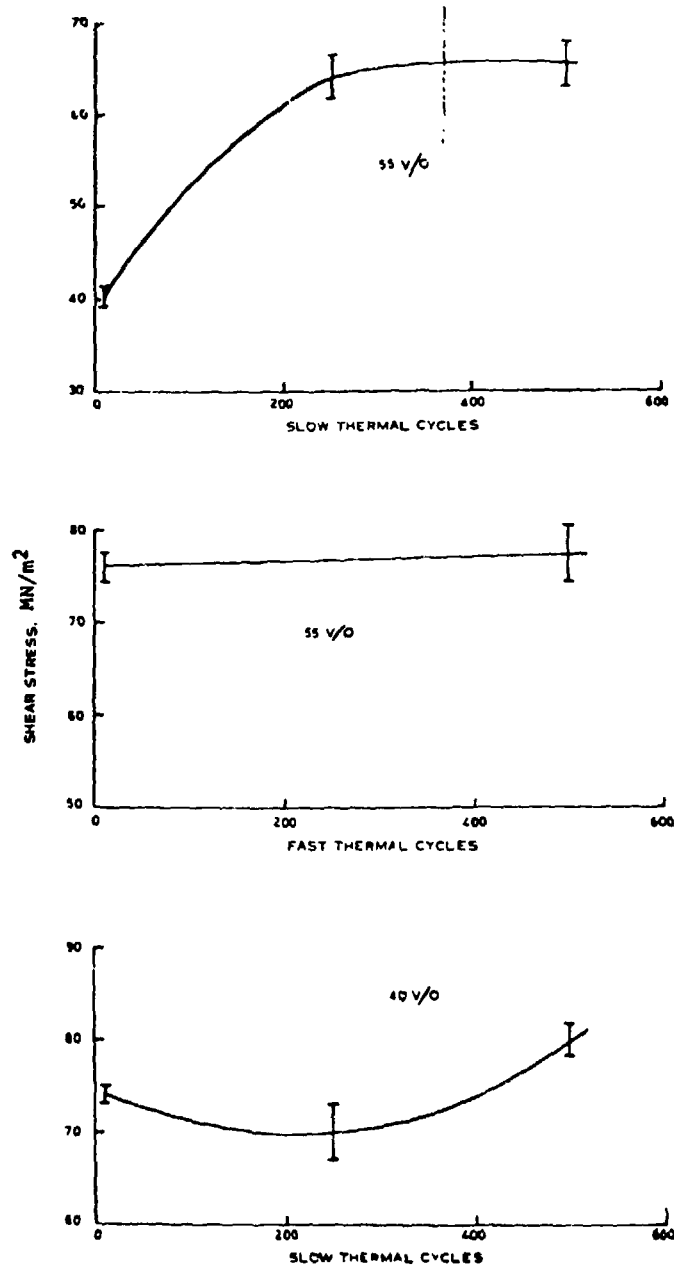


FIG. 5: SHEAR STRENGTH OF CARBON/EPOXY COMPOSITES AS A FUNCTION OF THERMAL CYCLING[209]

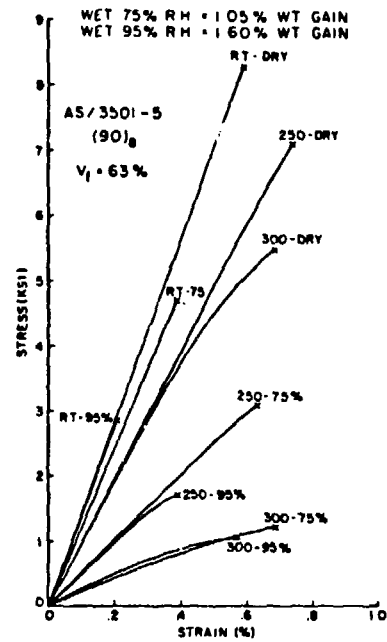


FIG. 6: TYPICAL TRANSVERSE STRESS-STRAIN CURVES AS A FUNCTION OF TEMPERATURE FOR AS/3501-5 UNIDIRECTIONAL COMPOSITES[41]

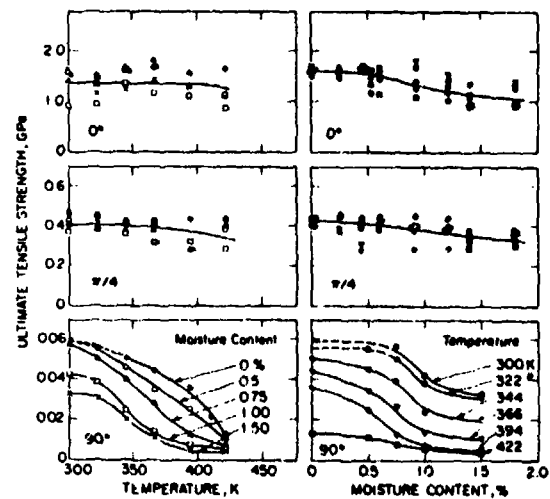


FIG. 7: ULTIMATE TENSILE STRENGTH OF THORNEL 300/FIBERITE 1034 AS A FUNCTION OF TEMPERATURE AND MOISTURE CONTENT. PRESENT DATA. FIBER VOLUME FRACTION  $\sim 0.68$ .  $1 \text{ GPa} = 1.450 \times 10^5 \text{ lbf/in}^2$ [261]

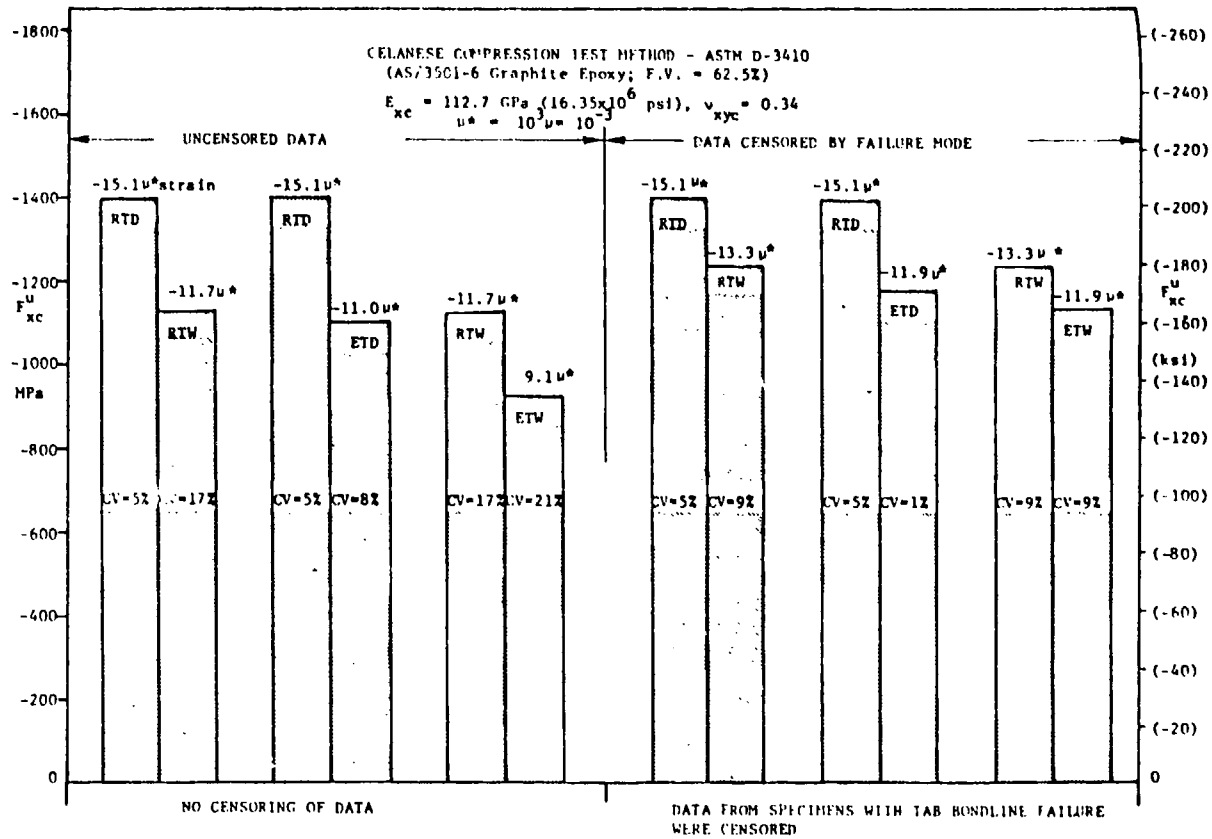


FIG. 8: 0-DEGREE STATIC COMPRESSION STRENGTH OF LAMINATE A,  $[0]_{16}T^{115}$

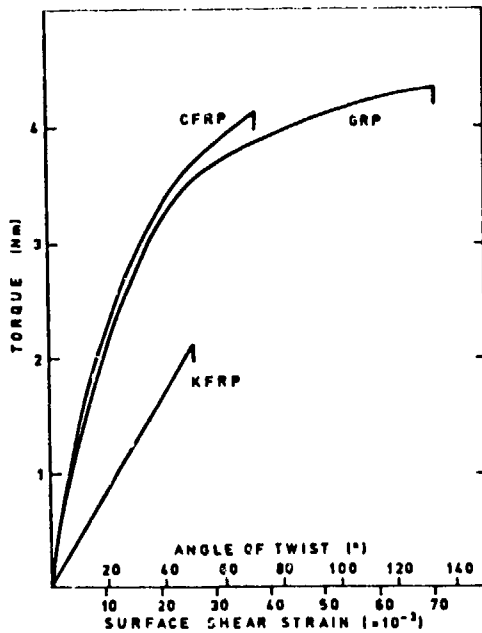


FIG. 9: TORQUE-TWIST BEHAVIOR OF THE AS-RECEIVED COMPOSITES[233]

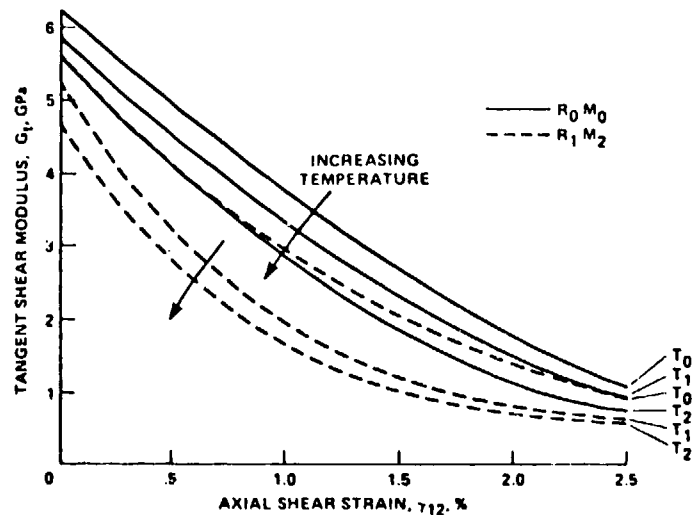


FIG. 10: TEMPERATURE EFFECTS ON TANGENT SHEAR MODULUS[193]

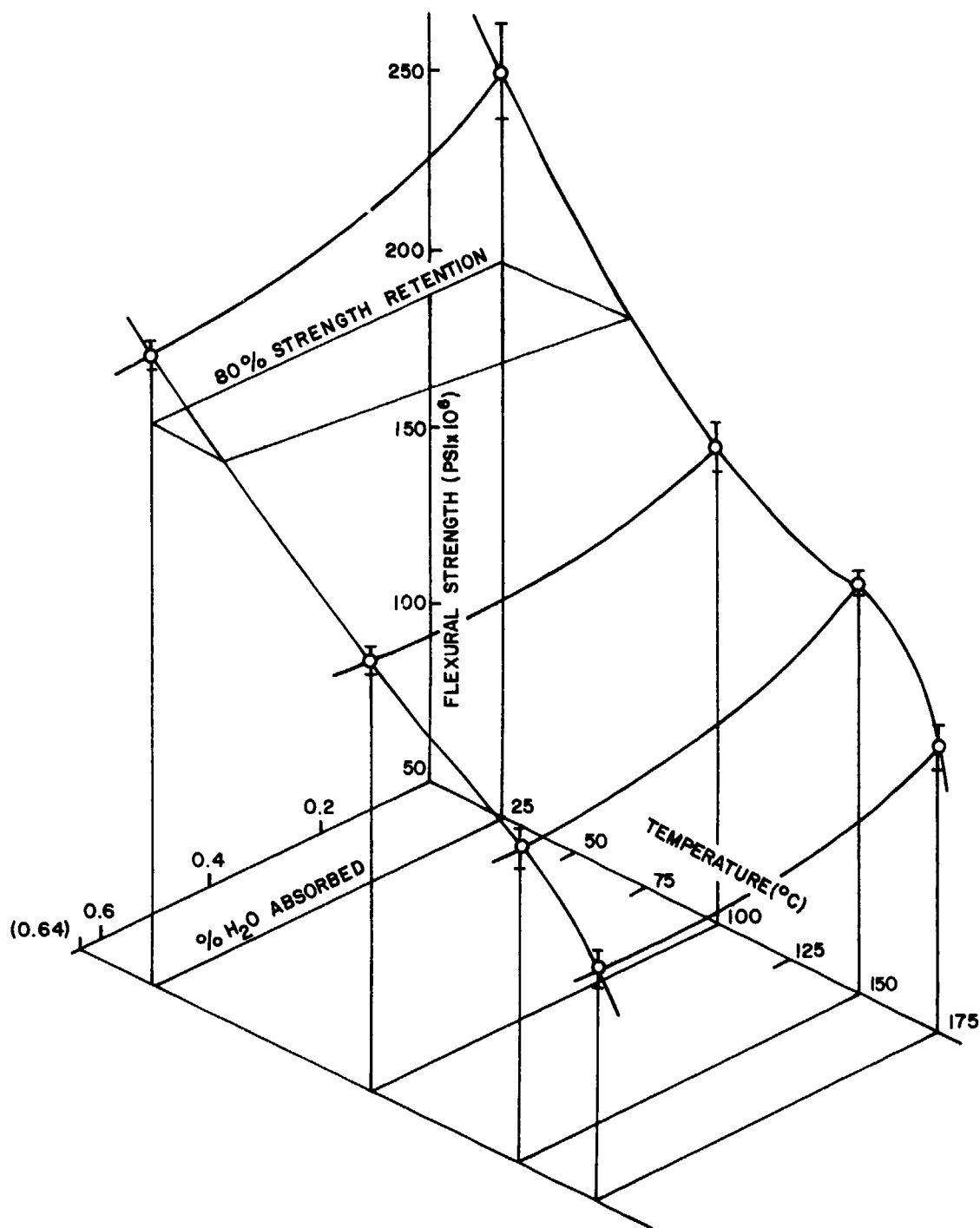


FIG. 11: FLEXURAL STRENGTH OF UNIDIRECTIONAL CARBON FIBER COMPOSITES (NARMCO 5208/T300) AS A FUNCTION OF TEMPERATURE AND PERCENT MOISTURE ABSORBED<sup>[23]</sup>

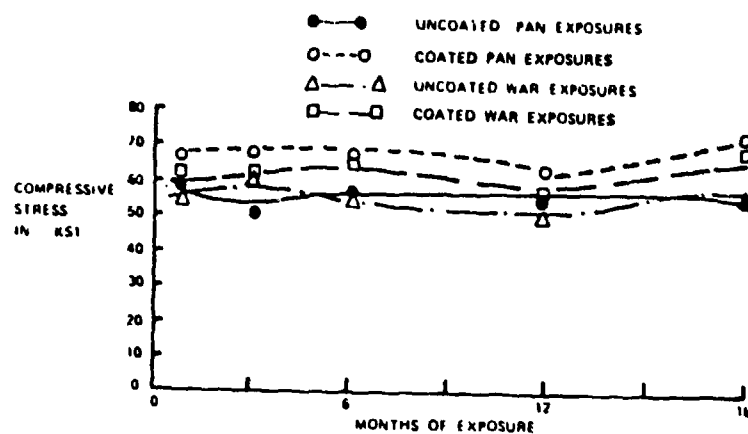


FIG. 12: THE EFFECT OF EXPOSURE TIME ON COMPRESSIVE STRENGTH OF 3002T GRAPHITE/EPOXY[291]

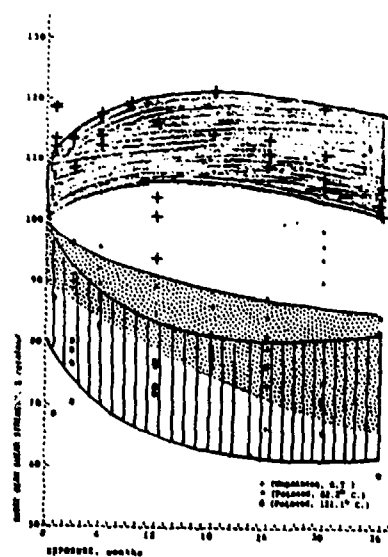


FIG. 13: 400 SERIES, 10 PLY (0/±45/90) WARMINSTER, % RETAINED SHORT BEAM SHEAR STRENGTH, AS/3501-6 GRAPHITE/EPOXY[294]

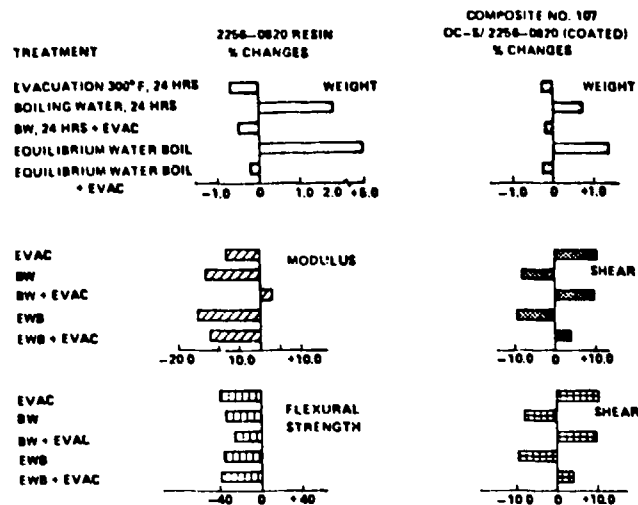


FIG. 14: WATER EFFECTS ON RESIN AND COMPOSITE[255]

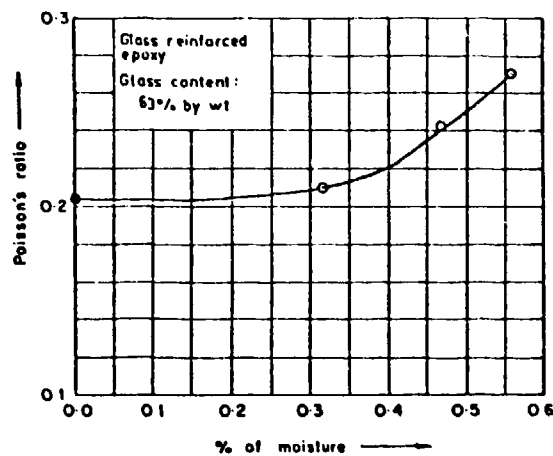


FIG. 15: EFFECT OF MOISTURE ON THE POISSON'S RATIO OF A GLASS CLOTH REINFORCED EPOXY COMPOSITE[238]

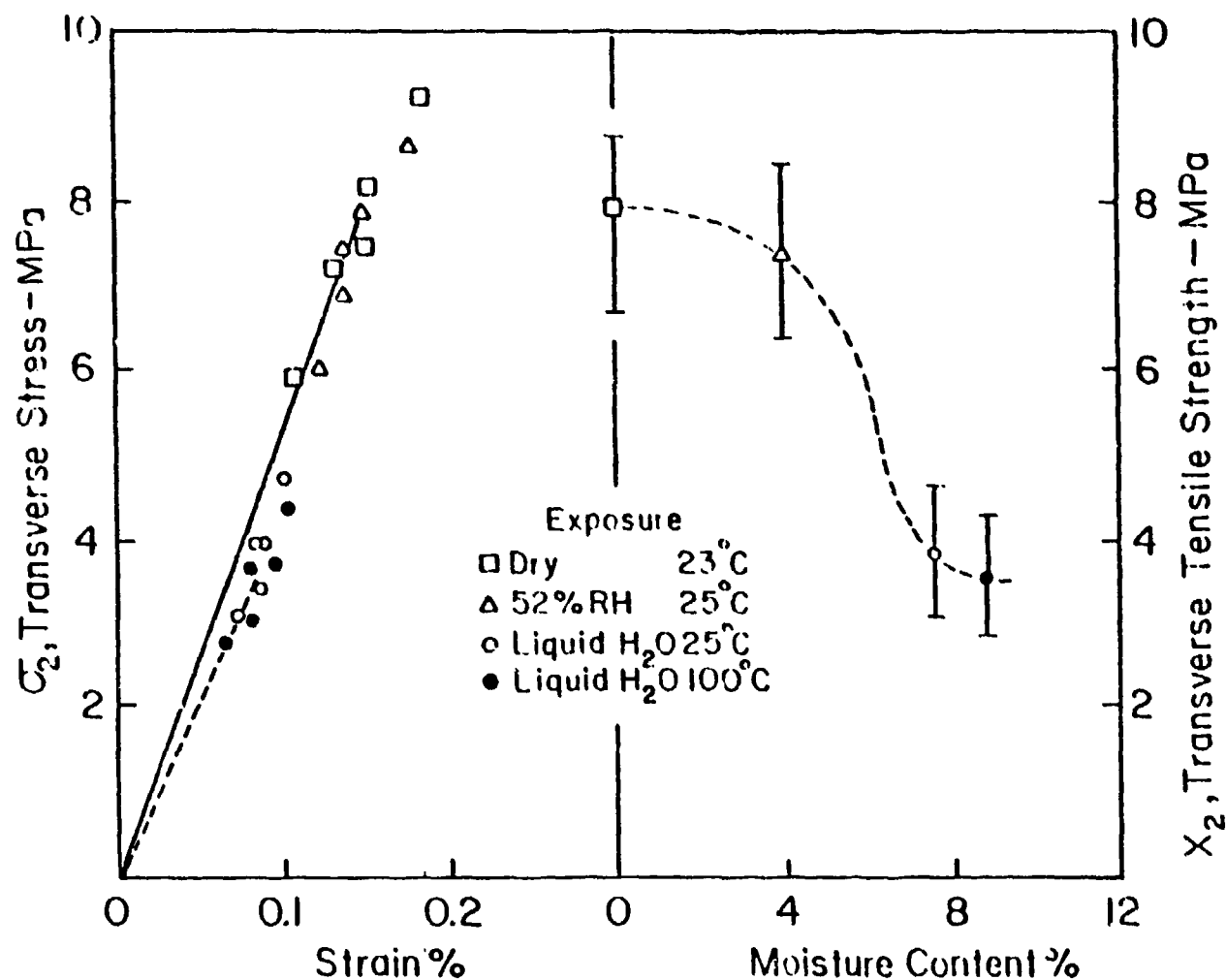


FIG. 16: TRANSVERSE TENSILE BEHAVIOR OF K-49 EPOXY AT DIFFERENT ENVIRONMENTAL CONDITIONS. LEFT, AVERAGE STRESS-STRAIN RESPONSES AND FAILURE POINTS; RIGHT, TRANSVERSE TENSILE STRENGTH AS A FUNCTION OF MOISTURE CONTENTS ( $\text{MPa} \times 0.145 = \text{Ksi}$ )<sup>[322]</sup>

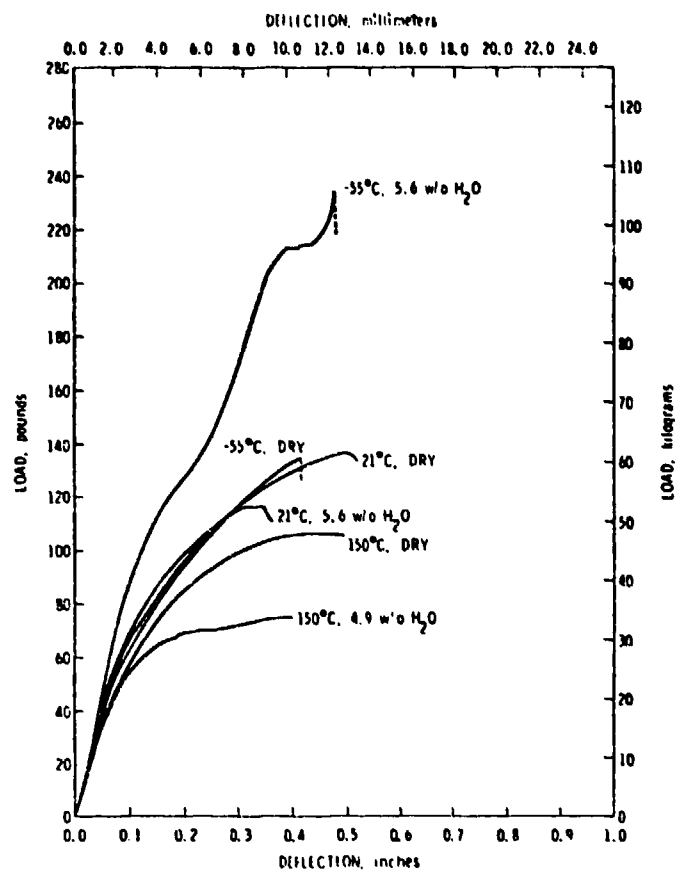


FIG. 17: LOAD VERSUS DEFLECTION BEHAVIOR FOR [0/90] KEVLAR 49 181 STYLE FABRIC/CE-9000 EPOXY LAMINATES AS A FUNCTION OF TEMPERATURE AND MOISTURE CONTENT<sup>(10)</sup>

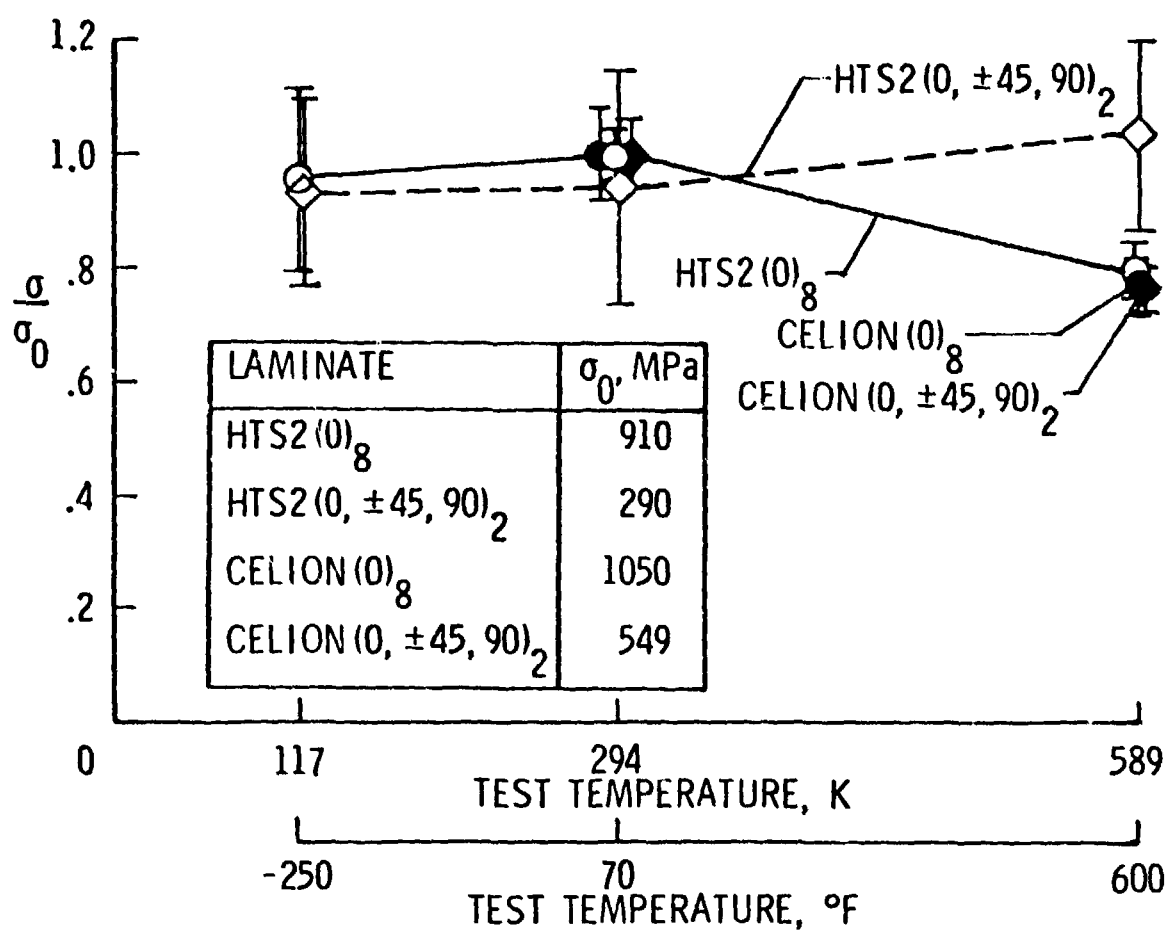


FIG. 18: FLEXURAL STRENGTH OF DESSICATED SPECIMENS<sup>[194]</sup>

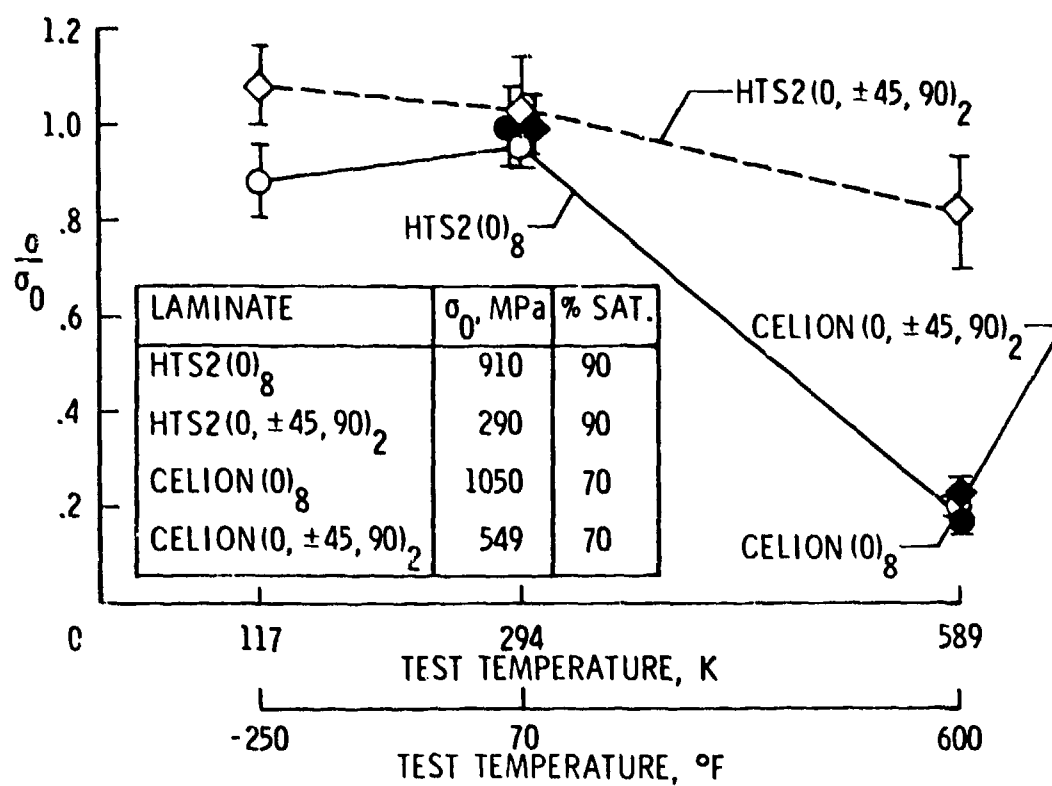


FIG. 19: FLEXURAL STRENGTH OF "WET" SPECIMENS<sup>[194]</sup>

## APPENDIX A - BIBLIOGRAPHY

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| NOTES<br><br>13                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  |                        |                                                  |                        |                                  |
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| SUMMARY/SOMMAIRE<br><br>This is Part III of a series of literature reviews on hygrothermal effects on polymer matrix composite materials. It contains a review of papers on mechanical properties as measured in static tests and includes the effect of impact damage interaction with environmental conditions.<br><br>The other parts of the review are:<br><br>Part I: Moisture and Thermal Diffusion<br>Part II: Physical Properties<br>Part IV: Mechanical Properties 2<br>Part V: Composite Structures and Joints<br>Part VI: Numerical and Analytical Solutions<br>Part VII: Summary of Conclusions and Recommendations<br><br>A complete list of references is included in the Appendix and the numbers in the brackets appearing in the text refer to this list.<br>15 |                        |                                                  |                        |                                  |