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HYGROTHERMAL EFFECTS IN CONTINUOUS FIBRE REINFORCED COMPOSITES

PART IV: MECHANICAL PROPERTIES 2 FATIGUE AND TIME-DEPENDENT PROPERTIES

by

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PART IV: MECHANICAL PROPERTIES 2
FATIGUE AND TIME-DEPENDENT PROPERTIES

EFFETS HYGROTHERMIQUES DANS LES COMPOSITES À RENFORT DE FIBRE CONTINU

PARTIE IV: PROPRIÉTÉS MÉCANIQUES 2
FATIGUE ET PROPRIÉTÉS DÉPENDANT DU TEMPS

by/par

J.P. Komorowski

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SUMMARY

This is part IV 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 fatigue, creep or stress relaxation tests with variations in temperature and humidity accounted for in the results.

The other parts of the review are:

Part I:	Moisture and Thermal Diffusion
Part II:	Physical Properties
Part III:	Mechanical Properties 1
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 IV d'une série de documents traitant des effets hygrothermiques sur les matériaux composites à matrice polymérique. Elle comprend une étude des données recueillies sur leurs propriétés mécaniques lors d'essais de fatigue, de fluage et de relâchement des contraintes dont les résultats tiennent compte des variations de température et d'humdité.

Les autres parties de cette série sont les suivantes:

Partie I:	Diffusion de l'humidité et de la chaleur
Partie II:	Propriétés physiques
Partie III:	Propriétés mécaniques 1
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 IV: MECHANICAL PROPERTIES 2 – FATIGUE AND TIME-DEPENDENT PROPERTIES

1.0 INTRODUCTION

In this part of the review (Part IV) results of fatigue and creep and stress-relaxation tests are reported.

With the move towards primary load carrying structures made with composite materials, the number of publications related to the fatigue properties of these materials has rapidly increased. Most of these papers are concerned with graphite/epoxies, and with other materials receiving less attention either because of cost or inferior properties. In the future, if the trend towards higher strain allowables for structural composite materials is realized, fatigue will certainly become a major factor in the design of primary composite structures.

There is a strong indication that fatigue in composites is closely related to creep as time at load, rather then number of cycles, has had the greater effect on life in some experiments. Mechanical relaxation problems, however, have significance of their own for some applications like pressure vessels, bolted joints (relaxation of clamp up force) or structures which have to demonstrate dimensional stability (relaxation of residual stresses).

Fatigue tests of composite structures and joints will be reviewed in Part V.

2.0 FATIGUE

2.1 Constant Amplitude Loading Studies

Constant amplitude loading studies that produce S-N curves have limited value for composite designers. They are, however, a very convenient method of demonstrating behavior of materials under cyclic loading and have been used in environmental studies of composite materials. In this chapter investigations will be reviewed in which materials have been exposed to various conditions of temperature and humidity prior to, during or after constant amplitude cycling.

Hofer, Bennett and Stander[131] studied the effect of humidity preconditioning followed by stress cycling, on residual mechanical properties. Earlier in the study S-N curves were generated at room temperature. The stress ranges at 2×10^6 cycles were taken from these curves for all laminates under consideration. The average stress range was calculated and a 10% smaller value was used as stress range for stress cycling. Humidity preconditioning consisted of steady state 500 hour or 1000 hour soaking at 48.9°C – 98 ± 2% RH. Materials used were T300 (graphite) and S-glass in 5208 epoxy resin. Hybrid laminates of an interweaving type were made with varying proportions of all glass and all graphite plies. Lay-up was either unidirectional or quasi-isotropic. All materials were stressed in a tension-tension load cycle (R = 0.1) at 30 Hz up to 10^7 cycles but not all specimens survived this rather mild stress cycling regime. Those that did showed a decrease in residual strength but no loss of modulus and slight increase in Poisson ratio for the 0° lay-ups.

Rotem and Nelson[245] and Rotem[246] studied specimens of graphite/epoxy (T300/5208) in tension-tension (R = 0.1 and R = 10 at 30 Hz) at 22°C, 74°C and 114°C. They used unidirectional and angle-ply laminates as well as $[0°/\pm\theta/0°]_S$. These latter laminates were used to verify predictions of fatigue durability and failure mode based on results for unidirectional and angle-ply laminates. The fatigue behavior of a single lamina was characterized by its static strength and its "fatique function" which expresses the degradation in static strength due to cyclic loading. This function measured at some reference temperature together with temperature shifting factors can be used to calculate the fatigue function in a broad range of temperatures. Results generally correlated well with predictions but for laminates where angle-ply laminae contributed to the load to a greater extent ($\theta < 45°$), the viscoelastic character of the matrix had to be taken into account. Generally for matrix dominated laminates, fatigue failure was affected by cycling and temperature, and a shifting of the fatigue function was observed. However, the slope of the S-N curve was not affected by temperature. Fiber dominated laminates were not sensitive to temperature change (Figs. 1, 2, 3 and 4).

Haskins, Kerr and Stein[127] presented results for HT-S/710 graphite/polyimide in $[0^\circ/\pm45^\circ]_S$ lay-up. Fatigue tests were carried out at RT and 232°C. For the two loading ratios (R = −1, R = 0.1), there was little effect of temperature on fatigue of this fiber dominated laminate which supports the results of Rotem and Nelson.

Kan and Ratwani[161] presented results for matrix dominated laminates $(\pm45/90_2/\pm45/90_2)_S$ made from graphite/epoxy (AS/3501-6). Specimens were moisture preconditioned up to 1% weight gain with the uniform moisture distribution calculated using Fick's model as defined by Springer and Shen[212]. Fatigue tests were run under tension-compression fully reversed loading (R = −1). Test frequency of 5 Hz was chosen as the final loading frequency. Figure 5 shows the influence of moisture content on the fatigue behavior of the laminate. It was tentatively concluded that for this matrix dominated laminate, at room temperature, moisture had no influence on the compression fatigue life.

It would appear from all of the above reviewed results, that temperature is more detrimental than moisture, in reducing fatigue strength.

Ryder and Walker[251] have done an extensive study of the effect of compressive loading on the fatigue of graphite/epoxy (T300/934) laminates. The aim was to observe the effect of absorbed moisture under compressive loading in unnotched and notched (circular hole) specimens. Two lay-ups were used (1) 25% of 0° — $(0/45/90/{-45}_2/90/45/0)_S$, (2) 67% of 0° — $(0/45/0_2/{-45}/0_2/45/0_2/{-45}/0)_S$. The baseline dry condition was 22°C, 40 ± 10% RH while preconditioning took place at 82°C, 90% RH up to saturation for the wet tests. There are four ways of defining the loading variables in fatigue: load ratio (R), maximum stress (σ_{max}), stress range ($\Delta\sigma$) and minimum stress (σ_{min}). As $R = \sigma_{min}/\sigma_{max}$ and $\Delta\sigma = \sigma_{max} - \sigma_{min}$, during a fatigue test, any one of these variables may be held constant while the effect of one of the other three is being studied. Laminate (1) and (2) act as minimum columns. The maximum compressive stresses without lateral deflection greater than 0.0254 mm were therefore limited to −110 MPa and −207 MPa respectively. Constant load ratio was rendered impractical as it limited the maximum stresses. In these studies σ_{min} was held constant at either −110 MPa (−207 Ma) or 0(0). Failure was defined for tension-tension as breakage of coupon and for tension-compression as either breakage or an inability to sustain load due to severe delamination. Failure modes observed for elevated temperature, wet (ETW) conditions were similar to those obtained at RT. The only difference was that the type of damage which led to failure appeared much earlier in life for coupons tested at the same stress. For matrix dominated laminate (1) elevated temperature, wet conditions decreased life of unnotched specimens by a factor of 3, and for notched specimens by a factor of 10. For notched specimens, the tension-tension S-N curve changed from flat at RT to declining strength with number of cycles under ETW conditions. For the fiber dominated laminte (2) the results are not easy to discuss. A larger scatter was evident for ETW conditions. During tension-compression tests at RT, some specimens survived 10^6 cycles of +759 MPa stress, while under ETW, all specimens with stress above +550 MPa failed before 10^6 cycles were reached. The fatigue tests were followed by residual strength test and it was observed that the fatigue induced damage does not appear to have a direct effect on residual tensile strength. For laminate (2) cycled to 10^6 cycles under tension-tension ETW conditions, residual tensile strength was reduced by 20% while residual compressive strength was reduced by 40%. This indicates that ETW cycling has a significantly larger degrading effect than RT cycling which resulted in respective compressive strength reductions of 0% and 15%.

The data obtained was analyzed using Weibul distributions as well as other methods. Ryder and Walker concluded that significant statistical analysis efforts, combined with an extensive experimental investigation, is needed before any extrapolative procedures can be used with confidence to predict fatigue performance in an environmentally degraded condition.

The studies of the effect of moisture and temperature on composite material strength have indicated that compressive strength is particularly sensitive to these factors[187, 115]. Similarly compressive loads in fatigue have strong degrading effects on fatigue properties. However no evidence of a synergistic effect of compression fatigue and environment was found in the reviewed literature.

Grimes[115] carried out an investigation in which graphite/epoxy samples were loaded in compression-compression at R = 10. The material used was AS/3501-6 with the following lay-ups: $[0]_{nt}$, $[90]_{nt}$, $[\pm 45]_{nS}$ and $[(\pm 45)_5/0_{16}/90_4]$. Some samples were pre-soaked up to a 1.1% weight gain of moisture. Testing was carried out in a specially designed fixture which was used for both fatigue and residual strength tests. Fatigue testing was carried out at room temperature and the residual strength test was at an elevated (103.3°C) temperature. Only for $[90]_{24T}$ specimens were significant differences in fatigue properties found (Fig. 6). For dry samples, runouts were observed at stress levels of 126.9 Mpa (or 49% static dry strength) while for wet conditions, the runout stress level was 90 MPa (or 45% of static wet). However, these differences could be expected since for these samples, static dry strength is higher than static wet strength −258.6/−199.3 [MPa/MPa]. 'Wear out'* occurred in all specimens but was greater in matrix dominated laminates. Higher wearouts were attributed by Grimes to degradation of the interface.

Adams[3] reported results of an SEM study carried out on failed samples from Grimes' investigation. The influence of moisture or elevated temperature was not observed. No obvious differences were noted in the corresponding fracture surfaces. The author also tried to apply a micromechanics analysis which was developed earlier and successfully applied to calculate residual and environmental stresses. There is a similarity between static and fatigue failure in compression, however, the application of micromechanics analysis to fatigue is still far from being satisfactory.

Sumsion and Williams[277] and later Sumsion[278] studied the effects of temperature and water on flexural and torsional fatigue of AS/3501 graphite/epoxy laminates. The lay-ups used were 0°, ±45°, ±30° and woven (24 plies). Specimen shapes were as shown in Figure 7. Torsional fatigue tests were carried at 1 Hz under controlled strain (constant deflection) conditions and stopped if either the torque dropped to a preset level or if the required number of cycles was reached. Testing was carried out in air or water at both room and elevated (74°C) temperatures. After fatigue testing, the specimens were subjected to four point bending at room temperature to measure strength, and bending moment versus deflection curves were used to calculate failure energy. All the specimens exhibited fatigue damage. The effect of exposure to a water environment during torsion testing at 74°C and, to a lesser extent at 24°C was to decrease the 'incubation period'** and to increase the rate of accumulation of damage. At 74°C water also appeared to decrease (lower) the limiting torsional stiffness (Fig. 8). It should be noted that the cross plied specimens appeared more prone to fatigue damage in torsion than the unidirectional specimens. In contrast, in flexural fatigue, water had greater effect on unidirectional specimens.

The graphite/epoxy specimens showed significant flexural fatigue damage on both air and water when subjected to fully reversed plane bending at 30 Hz (Fig. 9).

Phillips, Scott and Buckley[233] also studied torsional fatigue of composites. The materials used were high modulus carbon, glass and Kevlar 49 in a Ciba-Geigy MY750 epoxy matrix cured with metyl nadic anhydride and benzyldimethylamine. A unidirectional lay-up was used and rods were machined to a 6 mm diameter. Test were run at room temperature and humidity. Samples were tested in either an as received state, or after seven days at 100°C water immersion. Some conditioned specimens were dried at 60°C for seven days prior to testing. Fatigue testing was carried out at 0.17 Hz, under either constant torque (±T) or constant twist ($\pm\theta$) amplitude. Significantly, the strength recovery upon drying observed during the static tests was not observed in torsional fatigue. Fatigue life for carbon and glass composites was permanently degraded by 100°C water immersion (Fig. 10). However, the boiling water test is very severe and is not similar to any situation encountered in service.

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* Observable damage.

** Time required to observe first damage of the material.

The rate of change of compliance has been measured in terms of ΔTorque/log N. For Kevlar composite, it is affected by various treatments as shown in Figure 11. In glass composites, this rate demonstrated partial recovery upon drying while in carbon composites, it was insensitive to moisture level.

Gauchel, Steg and Cowling[106] used Naval Ordinance Laboratory (NOL) ring samples filament wound using S-glass with various epoxy resins. Prior to fatigue testing in diametrical compression, rings were immersed at RT in water for 400 days. Testing was also carried out in water and concluded when the observed load at a given deflection dropped by 20%.

The percent retention of fatigue life after soak compared to dry specimens varied greatly (from 100% to 37.2%). The best results were achieved with a system containing 10 parts of N,N-diglycidyl tribromoaniline (DGTBA) for one part of meta-phenylenediamine (MPDS). Later tests showed that systems containing over 50% of DGTBA perform much better in fatigue under moisture influence than the other systems under consideration. These latter systems were mostly based on diglycidyl ether of bisphenol-A (DGEBA). The choice of resin may be detrimental to fatigue performance of a composite structure exposed to moisture.

In all the above papers the test conditions were steady state temperature and humidity, Lundemo[200, 201] studied the influence of environmental cycling on the static and fatigue properties of T300/5208, $(\pm 45)_{4S}$. The environmental cycles used prior to mechanical testing were aimed at simulating fighter aircraft service, including thermal spikes and low temperature excursions, with the humidities set to result in a moisture content of approximately 1% weight gain. Tension-tension (R = 0.1) fatigue tests were performed at $\sigma_{max} = \frac{1}{2}\,\sigma_{ultimate}$ and a frequency of 28 Hz. No failures occurred after 10^6 cycles for these specimens which were not exposed to the environmental treatment. After four weeks of treatment, five out of eight specimens survived 10^6 cycles with no survivals observed after six weeks of treatment (the longest life recorded for these specimens was 8.7×10^4 cycles. For specimens coated with polyurethane, less degradation was observed. Despite the low number of environmental cycles imposed (30 in six weeks) the effect was significant.

2.2 Random Loading

Years of experience has led to the conclusion that fatigue and damage tolerance testing must be conducted under conditions representative of service environments. Constant amplitude testing may be used only for those parts which will have a constant amplitude service environment. For the majority of aircraft parts this is not the case and simulation must be representative of the random nature of the service load history, usually with both amplitude and frequency variation. The flight-by-flight conditions including reverse loading must be represented[317]. This is true for both metal and composite structures. For composites, there is no accepted cumulative damage theory that allows the extrapolation of simple constant amplitude test data to the evaluation of the effects of random loading. Representative spectrum tests must be used and generally must start at the coupon level. This is expensive and time consuming, and by its nature, must be directed towards a particular application.

In a series of articles Haskins, Kerr, Stein et al[166, 126, 127] presented a program and some results on long term evaluation of Advanced Composites for Supersonic Cruise Aircraft. The materials used were graphite and boron/epoxy and polyimide (notably AS/3501, HT-S/710). Flight simulation was carried out using random loading with temperature cycling representative of supersonic flight (Fig. 12). Baseline tests and short term tests (accelerated load frequency and maximum temperature) were used to set reasonable load limits for long term tests. After 100 hours of testing, a preliminary wearout model was set and later refined after 200 test hours (Fig. 13). For the long term test, loads were set so that approximately 80% of the specimens would survive 25,000 simulated flights of two hours duration (one lifetime). During each flight one compressive load was applied at the highest temperature point of the flight. After 8000 hours, more specimen failures were observed than predicted from short term tests. It was concluded that the wearout model used is not sufficient to accurately predict complex real time exposure effects. Delamination was extensive as the failures were due to compression — which indicates that ultimate tensile strength is not a good measure of damage in composite.

Sendeckyj et al[258] studied the effect of temperature on fatigue response of surface-notched $[(0/\pm45/0)_S]_3$ T300/5208 graphite/epoxy. Flight-by-flight spectrum loading was used in two versions, one with and the other without compressive loads present. One lifetime consisted of 1280 flights, each 44 seconds in duration. Load cycling was carried out at different temperatures from 27°C to 210°C. All survivors of two lifetimes were inspected using both C-scan and TBE radiography and subjected to a room temperature residual tensile strength test. Elimination of compressive stresses did not lead to a significant change of residual strength. However, the fatigue test temperature did influence the residual strength. The maximum residual strength was observed for specimens tested between 156°C to 182°C. No specimen survived two lifetimes at 210°C but all had survived below that temperature. At increased temperatures, the size of the damaged zone increases (delamination) however, the more damage present the worse the conditions are for load transfer by the matrix. This, in turn, reduces the local stress intensity due to the presence of a crack (notch).

Results from the US Air Force Materials Laboratory sponsored Advanced Composites Serviceability Program have appeared in several papers[230, 12, 176, 177]. The main aim of this study was to develop experimental information on the growth of flaws and to quantify their influence on residual strength. A real time matrix of load, temperature and moisture was reduced and compressed. This permitted one lifetime to be simulated by 24 hours test. Loading was representative of a vertical tail spectrum (B1 bomber) including fully reversed load ($R = -1.0$). The maximum test load was equal to the design limit load (2/3 of ultimate allowable or 80% of average ultimate stress). Figure 14 contains a truncated spectrum for metal vertical tail with all cycles below a load factor (L.F.) of 0.089 removed (only the positive side of the $R = -1.0$ spectrum is shown). The final load and temperature spectrum can be seen in Figure 15. The number of load cycles was reduced from 500,000 to 127,500 and the number of temperature cycles was reduced from 4000 to 6 to enable one test lifetime to be carried out in 24 hours. The temperature sequence in a mission was rearranged into a monotonically increasing sequence from low to high. Preconditioning with moisture was carried out at 74°C, 98% RH up to a 1.2 to 1.3% weight gain to represent the worst type of USAF basing conditions. In order to maintain the moisture content, steam was injected into the system during the 49°C and 82°C cycles. Compromises made are summed up in Figure 16.

Specimens used in these studies were made of AS3501-5A and T300/5208. The flaws were classified into categories which describe the stress gradients caused by a flaw embodied in a laminate undergoing a far field (away from the flaw) uniform stress. The likelihood of occurrence was used to estimate the flaw criticality[177] and fatigue tests were carried out. The flaw size was regarded as critical if it led to specimen failure after two lifetimes of spectrum loading.

It was found that the residual compressive strength of graphite/epoxy, after load cycling degrades with temperature, moisture, proof loading and size of imperfection or damage, Figures 17, 18 and 19[176]. As delamination was the dominant damage type, residual tensile strength was not significantlly affected[177].

Daniel and Schramm et al[79, 253] conducted nondestructive inspections aimed at monitoring damage growth as part of the above described studies. They found that flaw growth was much more pronounced for those specimens exposed to environmental fluctuations in addition to the load spectrum. The worst type flaws appeared to be[79]:

1. Circular hole.
2. Embedded film patch.
3. Internal ply gap.
4. Surface scratches.

Gerharz and Schutz[108] presented a "quasi real time" program and proposed several accelerated schedules for testing the composite upper surface wing root area of a fighter plane. The load spectrum used was FALSTAFF*. The objective of the accelerated test program was to achieve the same damage growth and residual strength as found in a "real time" loading with a shorter testing time. To date no results of these studies have been published.

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* For more information see "Introduction to a Fighter Aircraft Loading Standard for Fatigue Evaluation "FALSTAFF" by G.M. vanDijk and J.B. deJonge.

When developing accelerated testing schedules the effect of creep must be considered. Sun and Chim[279] found that fatigue life increases with time at load. For notched samples of T300/5208 in a $[\pm 45]_{2S}$ lay-up, fatigue life was significantly longer when cycling frequency was first low and later higher. The reverse order of cycling frequency resulted in shorter fatigue life. They concluded that a reduction of stress intensity at a crack tip due to creep was responsible for increasing the fatigue life during the "slow-fast" tests. As creep is clearly related to temperature it is obvious that time at temperature in the environmental cycle will also influence fatigue life.

Other spectrum loading tests will be reviewed in Part V of this review which deals with composite joints and structures.

2.3 Fatigue Testing in Simulated Environmental Conditions

Testing in hot-wet conditions was considered in Part III of the review series. There are some additional points specific to fatigue testing under such conditions, to be made.

Several authors reported problems with grip tab failures. Rotem[246] used graphite cloth T300/5208 tabs after tabs manufactured from other materials (glass epoxy and aluminium) failed. Ryder and Walker[251], after some research, chose American Cyanimide FM400 as the best tab adhesive for testing at 82°C, 95% RH. For tension-compression tests, a temperature rise in the tab area of 39°C was recorded and resulted in tab failure. This problem was alleviated by cooling the tabs with RT air. In the gauge length, the temperature rise was 3°-8°C which was accounted for in the analysis of the results. Kan and Ratwani[161] in an earlier mentioned tests found that initial test frequency of 10 Hz resulted in a temperature rise of 2.8°C in the gauge area. Therefore, 5 Hz was chosen as the final loading frequency.

Some authors reported on spectrum loading tests with the environmental conditions simulated,[108, 230, 267] and include a brief description of the equipment which they have used to produce required temperatures and humidities around the test specimen. The thermal spike test in[108] for example, required equipment capable of achieving slew rates of 60°C/min. The cost of such equipment is very high and lower slew rates (~20°C/min) are more typical of such testing equipment. These high rates are necessary to conduct accelerated tests.

2.4 Conclusions

1. The large scatter apparent in composite tests is usually increased by varying test temperature and moisture conditions which makes interpretation of results difficult.

2. Non-organic fiber dominated laminates generally show little sensitivity to environmental factors under fatigue.

3. For matrix dominated laminates, fatigue characteristics are affected by temperature, while moisture, at room temperature, seems to have no effect. (This may vary greatly depending on the matrix used.)

4. Environmental effects on the viscoelastic properties of a matrix may have to be taken into account in composite fatigue analysis.

5. The observation of sensitivity or insensitivity to environmental factors is very closely tied to the definition of failure in composite materials.

6. Elevated temperature wet conditions and particularly environmental cycling reduce the fatigue resistance of composites. The slopes of S-N curves for graphite composites are relatively flat and even slight shifts of the curve will result in significant reductions in fatigue life. Strength reduction, especially in tension, may not be as significant.

7. Fatigue damage in composites usually is not directly related to residual strength. This is frequently ignored in fatigue studies of composites.

8. A method has been proposed for calculating fatigue properties of laminates, at various temperatures, from simple unidirectional and crossply studies at some reference temperature. This method is, however, in the very early stages of development.

9. The following deficiencies exist with respect to composite fatigue: a) A general theory for predicting laminate fatigue properties analogous to lamination theory for static properties; b) A general cumulative damage theory like Miner's rule for metals; c) A theory accounting for degradation of properties due to environmental factors. As a result, verification of existing designs has to be through testing under representative loads and environments.

10. Simple environmental simulation in accelerated tests should be adequate for fiber dominated materials while realistic environmental simulation is required for matrix dominated materials.

3.0 TIME DEPENDENT PROPERTIES

3.1 Mechanical Relaxation

Mechanical relaxation phenomena are observed when material behavior is nonelastic and stress and strain are not only functions of one another but also of time. The most commonly studied transient effects are creep and stress relaxation. In simple creep, either the applied stress or load is held constant while an increase in strain with time is recorded. Stress relaxation is observed when the stress required to hold a specimen at constant deformation is gradually decreasing with time. Results of creep and stress relaxation tests are strongly affected not only by the stress levels used, but by the test temperature, and for the case of organic solids (matrix materials), the moisture content of the specimen.

3.1.1 Linear viscoelasticity — superposition principle

The constitutive equation for general time-dependent material behavior can be written as follows:

$$\epsilon_z = f(\sigma_z, t, T, M, \sigma_H, T_H, M_H) \tag{1}$$

where:

ϵ_z — strain
σ_z — stress
t — time
T — temperature
M — moisture content
H — history of (temperature, moisture, stress)

This equation is so complex that it has never been used and instead, material behavior is approximated by combinations of elastic and viscous models.

The most general form of linear viscoelastic stress-strain relation in contracted engineering notation is given by[74]:

$$\sigma_i(t) = \int_{0^+}^{t} Q_{ij}(t-\tau) \frac{d\epsilon_j(\tau)}{d\tau} \, d\tau \tag{2}$$

Q_{ij} are the viscoelastic relaxation moduli. If conditions of temperature and moisture are varying, then the relaxation moduli become functions of these as well as time. In this case, strain in Equation (2) should be represented as sum of strains due to load, thermal and moisture expansion:

$$\epsilon_j(\tau) = \epsilon_j^*(\tau) - \alpha_j \Delta T(\tau) - \beta_j \Delta M(\tau) \tag{3}$$

α_j and β_j are coefficients of thermal and moisture expansion ΔT and ΔM are variations in temperature and moisture.

An alternate form of viscoelastic stress-strain relation which is more useful for creep type experiments is[33]:

$$\epsilon_i(t) = \int_{o^+}^{t} S_{ij}(t-\tau) \frac{d\sigma_j(\tau)}{d\tau} d\tau \tag{4}$$

where S_{ij} are the creep compliances.

The concept of superposition of time-temperature or time-temperature/moisture permits the use of master creep compliance (or relaxation modulus) curve representation of data:

$$S_{ij}(T, M, t) = S_{ij}(T_o, M_o, \zeta) \tag{5}$$

where T_o, M_o are reference temperature and moisture conditions and ζ is reduced time:

$$\zeta = \int_{o}^{t} \frac{d\eta}{a_{TM}(T, M)} \tag{6}$$

a_{TM} are horizontal shifting factors representing the amount of shifting necessary to bring the $S_{ij}(T, M, t)$ ($Q_{ij}(T, M, t)$) data into coincidence with the master curve. The relaxation tests (creep and stress relaxation) are usually conducted at constant conditions so Equation (6) becomes:

$$\zeta = \frac{t}{a_{TM}} \tag{7}$$

or taking logarithm of both sides of Equation (7)

$$\log_{10}\zeta = \log_{10} t - \log_{10} a_{TM} \tag{8}$$

Plots of isothermal/moisture moduli, or compliances, can be shifted horizontally with the magnitude of the shift equal to $\log_{10} a_{TM}$.

Materials which lend themselves to this type of operation are called thermo-rheologically simple (TSM). Vertical shifting is required when horizontal shifting did not result in a smooth, well-defined master curve. The material is then termed thermo-rheologically complex (TCM).

For rigid plastics with and without reinforcement, the creep compliance relationship frequently used for approximation is:

$$S(t) = S_o + S_1 t^n \tag{9}$$

S_o, S_1, n — constant with time.

For TSM, the creep compliance in a series of isothermal tests is given by:

$$S = S_o + S_1 \left(\frac{t}{a_T}\right)^n \tag{10}$$

For TCM S_o is temperature dependent:

$$S = S_o(T) + S_1 \left(\frac{t}{a_T}\right)^n \tag{11}$$

The linear viscoelastic model of time-dependent material behavior is used because of its simplicity. The superposition principle, when applicable, dramatically reduces the amount of time and number of data points required to fully characterize the viscoelastic properties of the material. However, composite materials generally do not follow this approximation so it can be used only for limited conditions (usually low stress and temperature levels) on an individual basis[33, 328].

3.1.2 Time-temperature superposition

Yeow, Morris and Brinson[328] studied time-temperature behavior of unidirectional graphite/epoxy (T300/934). The stress levels used in this study were such that the applied axial stress did not exceed 10% UTS. Specimens were subjected to various loads (mechanically conditioned) to determine whether load history affected material response. Since the stress-strain curves did not change, the number of specimens required for testing was reduced (specimens could be used several times). The material was tested for linearity — creep and recovery for [90°] specimen must have equal instantaneous strain (ϵ_o in Fig. 20) and the stress-strain curve after 15 minutes of creep must be linear (Fig. 21). Isothermal creep tests were conducted for temperatures of 20°C to 210°C and results for the reciprocal of reduced compliance ($1/S_{22}$) are shown in Figure 22. Reduced compliance is defined as:

$$S_{22} = \frac{\epsilon_2(t)}{\sigma_o} \frac{T}{T_o} \tag{12}$$

where

σ_o	—	applied stress
T_o	—	reference temperature
T	—	temperature of the test
$\epsilon_2(t)$	—	strain (transverse to fiber direction)

The master curve obtained from these data is shown in Figure 23 while shift factors a_T are plotted in Figure 24. It was found that shift factors for all fiber orientations as well as for the reduced shear modulus S_{66}, are equal. The deviation of the shift factor for reduced Poisson coefficient ν_{21} was attributed to data scatter.

Fiber dominated properties (S_{11} and ν_{12}) were found to be time and temperature insensitive.

Beckwith[33] reported on a study of Shell 58-68R epoxy and its composite with S-901 glass fiber. The temperature range used in the study was $-7°C$ to $60°C$. Tests were single and multiple (3) cycle creep and recovery. Both the resin and composite were found to be TCM materials as initial compliance (S_o see Eq. (11)) was temperature dependent (Figs. 25 and 26). The master curve for net creep compliance was obtained for the epoxy after initial compliance for each temperature was subtracted from the total compliance measured (Fig. 27). The glass/epoxy tested can be regarded as linearly viscoelastic. The range of linearity depends on temperature and fiber angle. At 24°C (±45°) glass/epoxy recovers completely for 35 MPa but not for higher loads. However at 60°C it does not recover even for 20 MPa (Fig. 28). Beckwith observed a change in initial and net creep compliance in composites due to multiple loading. The nonlinear effects were assumed to be predominantly microcracking and the crack growth seemed to cause a disproportionate amount of damage during the first few loading cycles.

For those cases where the superposition principle can be applied, the key to obtaining the master curve are the time-temperature shift factors. Kibler and Carter[167] examined the possibility of using electrical conductivity measurements for obtaining these factors.

Direct-current resistance of neat epoxies is very high. It can be therefore assumed that charge transport proceeds by the short range migration of heavy ions. In that case, the direct-current electrical conductivity is itself inversely proportional to viscosity. Samples of 5208 epoxy resin were used to extract the time-temperature shift factors from thermomechanical and electrical conductivity measurements:

$$a_T = \frac{\eta(T)}{\eta(T_R)} = \frac{\sigma(T_R)}{\sigma(T)} \tag{13}$$

where η - viscosity σ - electrical conductivity. Only linear effects were studied. The a_T factors extracted from both methods are compared in Figure 29. Carter and Kibler concluded that conductivity data may be useful in obtaining quick estimates of the effects of temperature on time-dependent mechanical response.

3.1.3 Time-temperature-moisture superposition

The considerable 'plasticizing' effect of moisture on matrix type resins relaxation modulus can be seen in Figure 30 taken from Browning[42, 44]. The two master curves shown correspond to dry and wet (equilibrium wt. gain at 71°C, 100% RH) samples. A shift factor from wet to dry was found to be 10. This implies that the same response will be exhibited by a wet sample 10 times faster then by the dry sample.

The effect of moisture on time-dependent response in composites has been studied only recently and by relatively few authors. In one of the earlier works Wang and Liu[304] studied creep in graphite/epoxy (Modmor II/1004) unidirectional composite. Specimens were placed in various humidity conditions at 21°C and 65°C. A significant increase in creep with humidity and temperature was observed (Figs. 31 and 32). Within the limits of linear response, the time-temperature-humidity shifting was shown to be effective (Fig. 33).

A much broader study was undertaken by Crossman et al[70]. The materials used were GY70/CE339, T300/5209 (both unidirectional) and HMF 330C/934 (T300 satin weave). All these materials are graphite/epoxies. Specimens were exposed to humidity conditions until saturation prior to stress relaxation testing. Results are shown in Figures 34, 35 and 36. For HMF 330C/934, the relaxation modulus at 149°C dry corresponds to the 71°C wet values. Part of this study was directed at demonstrating the effect of relaxation effects on residual stresses (see Part II of this review series).

Kibler[168] presented creep test results for T300/5208 and AS-3502 graphite/epoxies. Only the linear range of behavior was studied (stress levels at 25% — 35% UTS). The creep compliance master curve (Fig. 37) is essentially the same for both materials. Longer-term tests agree well with short-term tests. For fiber-dominated properties no time dependence was found (Fig. 38). From the above data $G_R(t)$ was calculated and is shown in Figure 39. It can be seen that when both moisture and elevated temperature are present, the shifts are considerable.

3.1.4 Nonlinear responses and time-temperature-stress superposition

Composite materials generally demonstrate nonlinear time-dependent behavior. Linear viscoelasticity is applied only for low stresses. Wang and Liu[304] encountered nonlinear response at 65°C, 95% RH at a stress level of 29% UTS (Fig. 32). This stress level is rather low from a design point of view. Wang and Wang[301] measured creep response at glass/epoxy (Scotchply 1002) at various load levels with controlled temperature and moisture content. For (90°) laminates the degradation of material is found to be quite serious when the temperature and moisture content increase (Figs. 40 and 41). Similar behavior was found for (±45°) specimens. Even the (0°) laminate, at high temperature and moisture exhibited a definite increase in creep strain (Fig. 42).

Griffith, Morris and Brinson[114] demonstrated that in some cases the time-temperature-stress superposition principle (TTSSP) can be used to produce unified master curves reduced to particular stress-temperature conditions. The procedure is explained schematically in Figure 43. The method is regarded to be valid if smooth curves can be produced. For the T300/934 material tested good correlation was obtained between the master curve from short-term tests and a long-term test (Fig. 44).

3.1.5 Predictions

a) Micromechanics — Halpin-Tsai

Halpin-Tsai micromechanics equation was used[168, 73] to predict viscoelastic modulus from bulk properties with good correlations (Fig. 45).

b) Macromechanics

Various authors have adopted elastic lamination theory to account for time-dependent behavior[168, 328, 114, 301, 74].

As a first step, it is necessary to calculate the transformed reduced compliances for a lamina arbitrarily oriented with respect to a laminate or global axis system. Yeow et al[328] used a visco-elastic analog of elastic orthotropic equation and compared it with the master curve from short-term data and long-term data (Fig. 46). Correlation was good, however, in a more recent publication, Griffith et al[114] concluded that all axis predictions must account for the stress-dependent nature of the master curves. The uniaxial stress must be transformed into stress components in the principal material directions and master curves for stresses associated with these directions should then be used with the transformation equation. For this approach, prediction was within 10% of measured responses.

A procedure for calculating creep response of a laminate from measured creep responses of laminae was shown in[301] and the results of comparison of predictions with measurements are in Figure 47. This procedure allows for the nonlinear nature of creep responses. For linear viscoelastic approximations, computer programs based on an elastic response model and data from master curves were used with satisfactory results[168].

3.2 Stress-Rupture

Creep tests can be carried out to failure (stress-rupture). For this case, the life of a composite does not seem to correlate to the initial static strength or to the residual static strength taken at any point before rupture. More experimental data is needed to determine times to rupture characteristics. As several years may be needed before rupture occurs accelerated testing methods are required. Chiao et al[60] and later Hahn and Chiao[120] compared long-term test results to results from a time-temperature reduction based on an Arrhenius type of equation. The materials tested were Kevlar 49 and S-glass strands impregnated with epoxy resins (several resins were used). In the earlier work[60] a discrepancy was found between the predicted and measured rupture times for stresses over 85% UTS. In[120] lifetime data spanning over eight years have been analyzed by a two-parameter Weibull distribution and it was found that above 80% UTS the failure process changes from a wear-out type to initial defect controlled. At high temperatures the failure process was also wear-out type and this explained the difference between the predicted and experimental data for higher stresses. The stress rupture of S-glass epoxy composite was found to be a random failure process (shape parameter of Weibull distribution $\alpha \approx 1$) regardless of stress level. The logarithmic characteristic lifetime was linearly related to the applied stresses.

Aveston et al[26] tested fiber bundles unimpregnated and impregnated with resins in wet environments. For carbon fibers no effect of time was observed. For E-glass considerable degradation was observed with worst effects of immersion in water of epoxy and polyester impregnated strands. Cemfil — glass fiber (alkali resistant) demonstrated much better performance while Kevlar 49 performance was somewhere between E-glass and Cemfil.

Allen[8] suggested the use of flexural creep and rupture tests to screen reinforced epoxy resins for environmental performance. Creep rates and rupture incidence are increased by immersion of specimens in water.

3.3 Conclusions

1. The linear viscoelastic model is a useful approximation for predicting CM performance at low stress levels and moderate conditions of temperature and moisture.

2. Fiber dominated properties (compliance S_{11} and Poisson ratio) are time and temperature/moisture insensitive (graphite) or very slightly sensitive (glass and Kevlar 49).

3. For pressure vessels and similar applications where material remains loaded for extensive periods, stress rupture in the fiber direction is a possibility. Arrhenius type relations may be used to predict time to rupture from short-term data.

4. Most composites are thermo-rheologically complex materials as initial compliance depends on testing conditions.

5. Moisture has a considerable "plasticizing" effect and the same creep response may be expected from a wet sample 10 times sooner then from a dry sample.

6. The Halpin-Tsai equation predicts the viscoelastic modulus from bulk properties with good results.

7. Lamination theory may be adopted to successfully predict the laminate viscoelastic response, both linear and nonlinear.

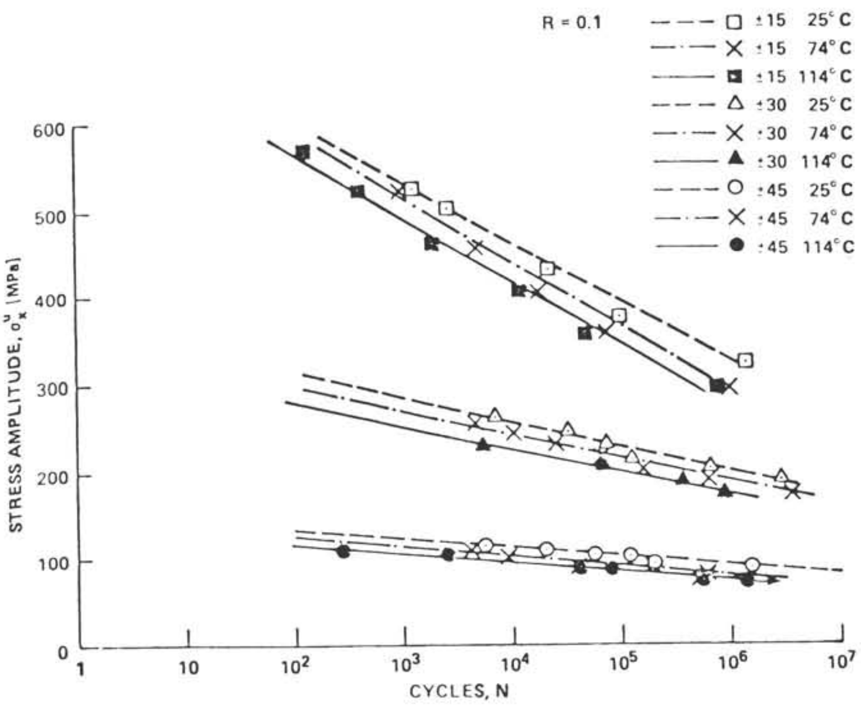


FIG. 1: S-N CURVES OF SOME ANGLE-PLY LAMINATES[245]

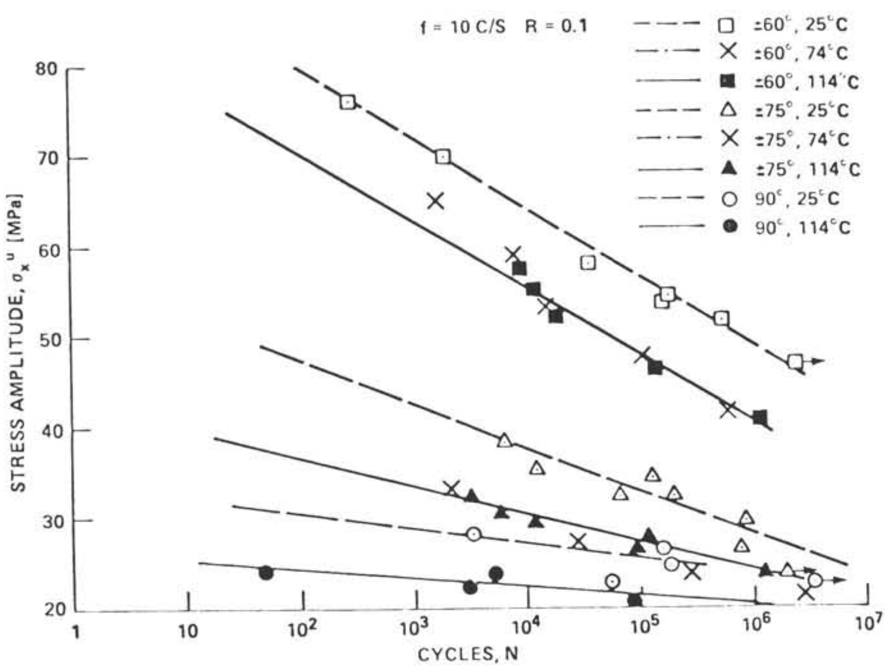


FIG. 2: S-N CURVES OF SOME ANGLE-PLY LAMINATES[245]

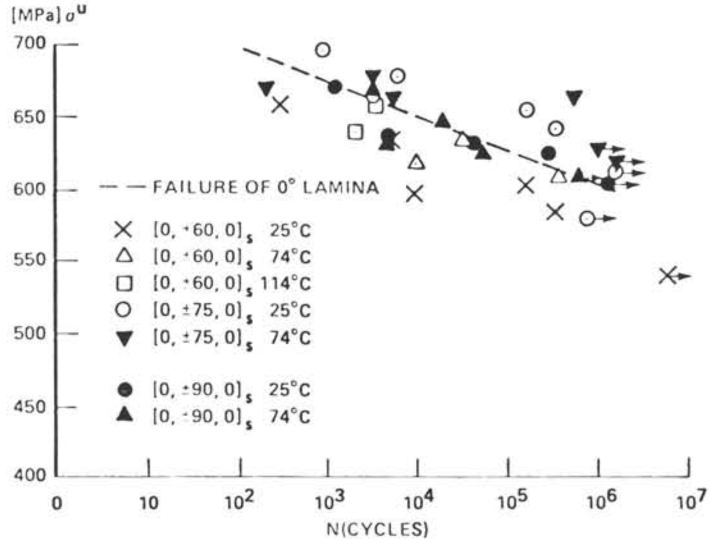


FIG. 3: S-N CURVES OF SOME SYMMETRICALLY BALANCED LAMINATES THAT FAIL BY FIBER FRACTURE[245]

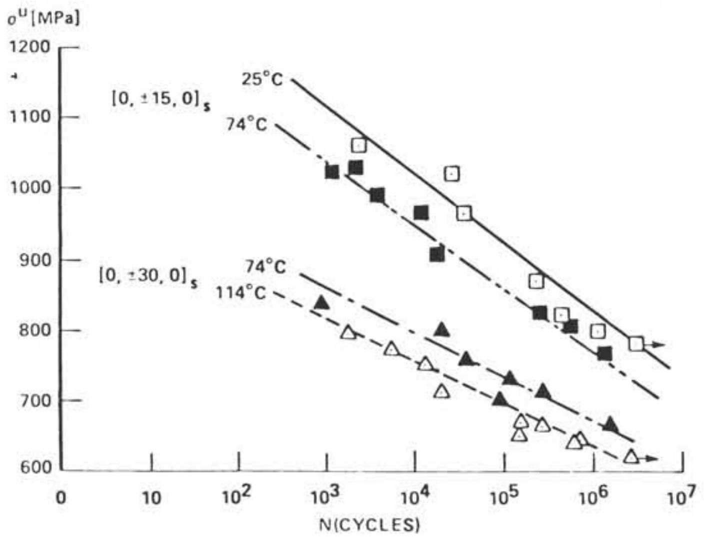


FIG. 4: S-N CURVES OF SOME SYMMETRICALLY BALANCED LAMINATES, SHIFTED WITH TEMPERATURE[245]

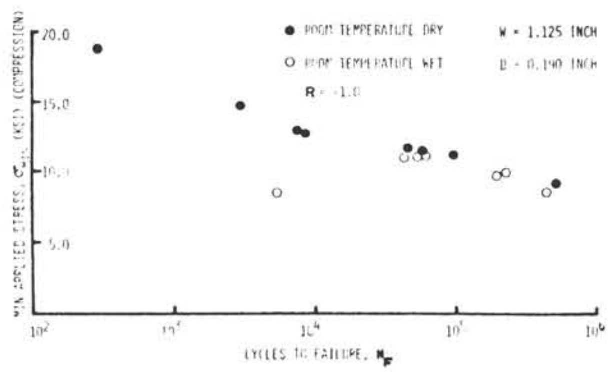


FIG. 5: S-N CURVE FOR LAMINATE 1 SPECIMENS [RTD AND RTW][161]

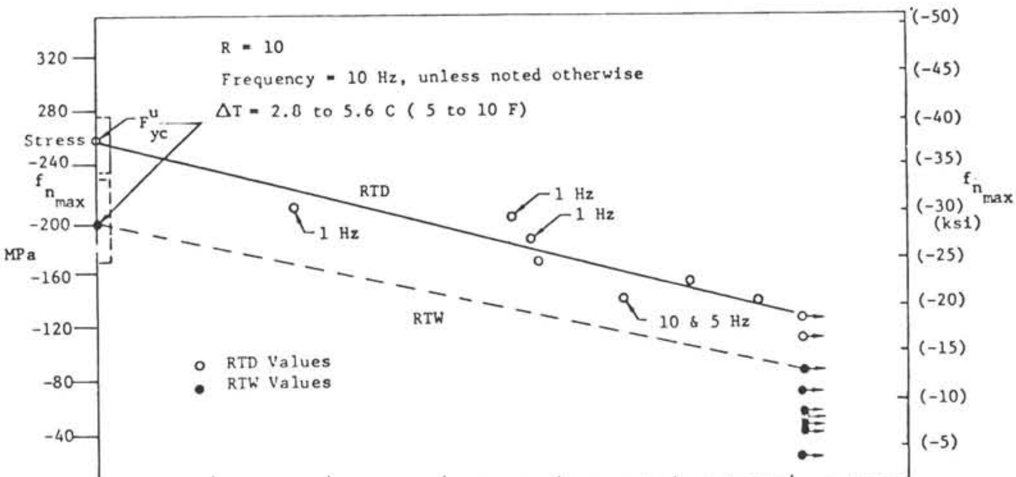


FIG. 6: 90-DEG COMPRESSION FATIGUE OF LAMINATE B, $[0]_{24T}$ – RTD AND RTW[115]

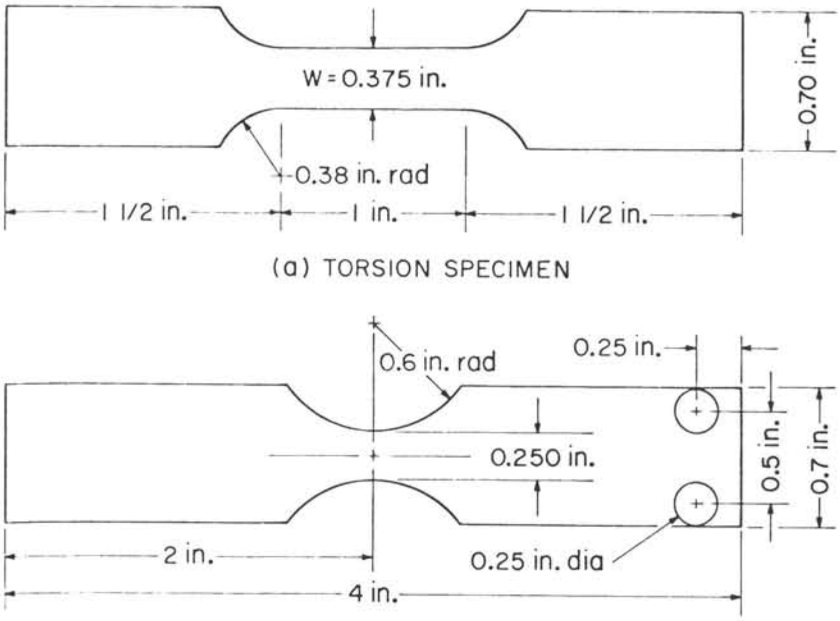


FIG. 7: TEST SPECIMENS[277]

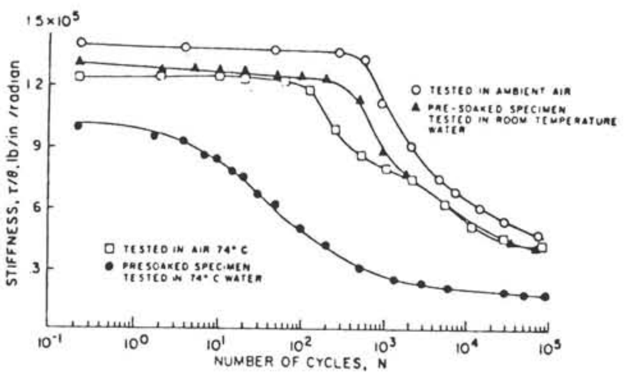


FIG. 8: TORSION FATIGUE OF ±45° FIBER-ORIENTED GRAPHITE-EPOXY COMPOSITE. DECREASE IN STIFFNESS $[\tau/\theta]$ WITH NUMBER OF CYCLES (LOG N) AS A FUNCTION OF ENVIRONMENT. ±45° FIBER ORIENTATION. $\Delta\tau_o$ = ±11,200. TEST FREQUENCY 1 Hz[278]

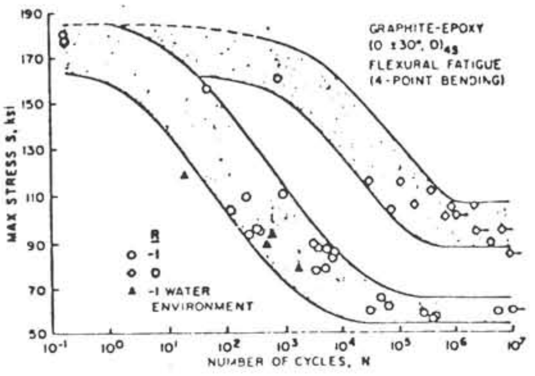


FIG. 9: FLEXURE FATIGUE TESTS OF ±30° FIBER-ORIENTED SPECIMENS IN REVERSE (R = −1) AND REPEATED (R = 0) CYCLING[278]

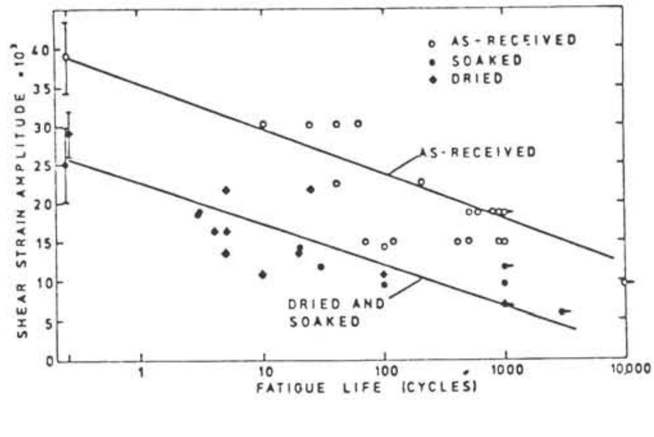


FIG. 10: FATIGUE LIVES OF CFRP UNDER CONSTANT SHEAR STRAIN AMPLITUDE CYCLING AFTER VARIOUS TREATMENTS[233]

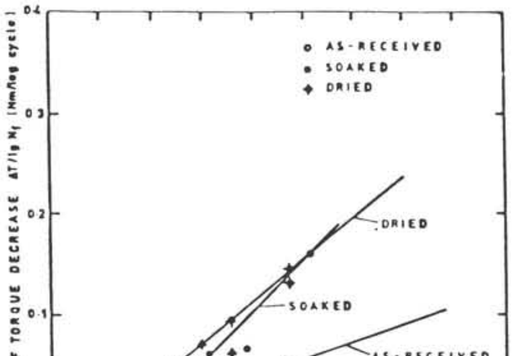


FIG. 11: ΔT/LOG N DURING CONSTANT STRAIN AMPLITUDE CYCLING OF KFRP[233]

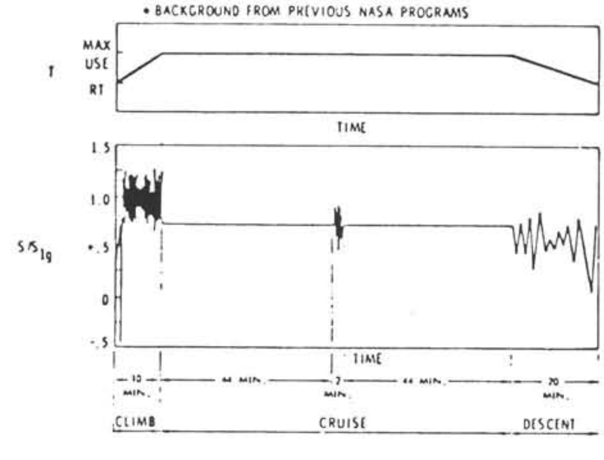


FIG. 12: LOAD/THERMAL HISTORY IS REALISTIC[126]

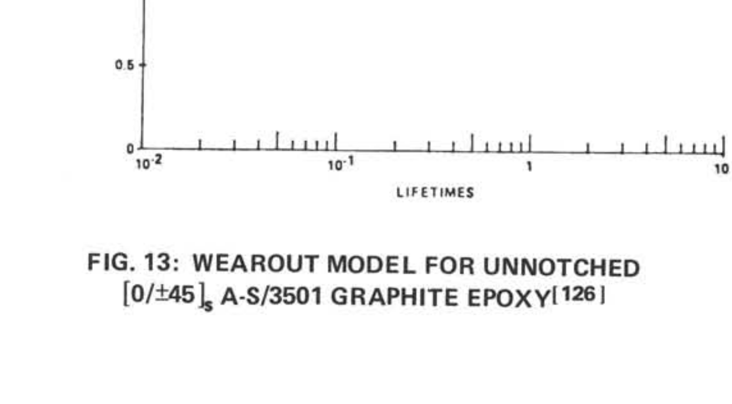


FIG. 13: WEAROUT MODEL FOR UNNOTCHED $[0/\pm45]_s$ A-S/3501 GRAPHITE EPOXY[126]

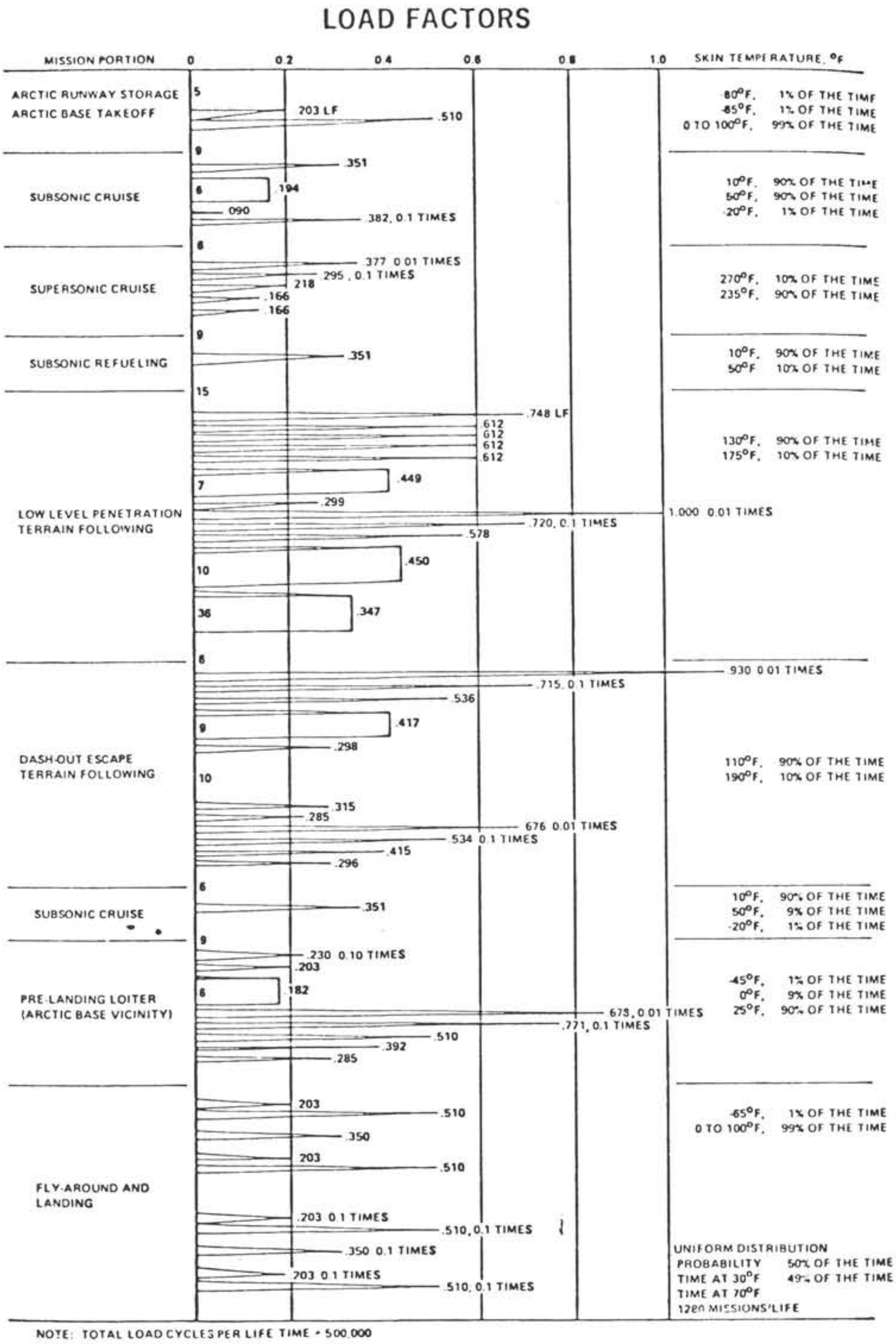


FIG. 14: SKIN TENSILE STRESS HISTORY[230]

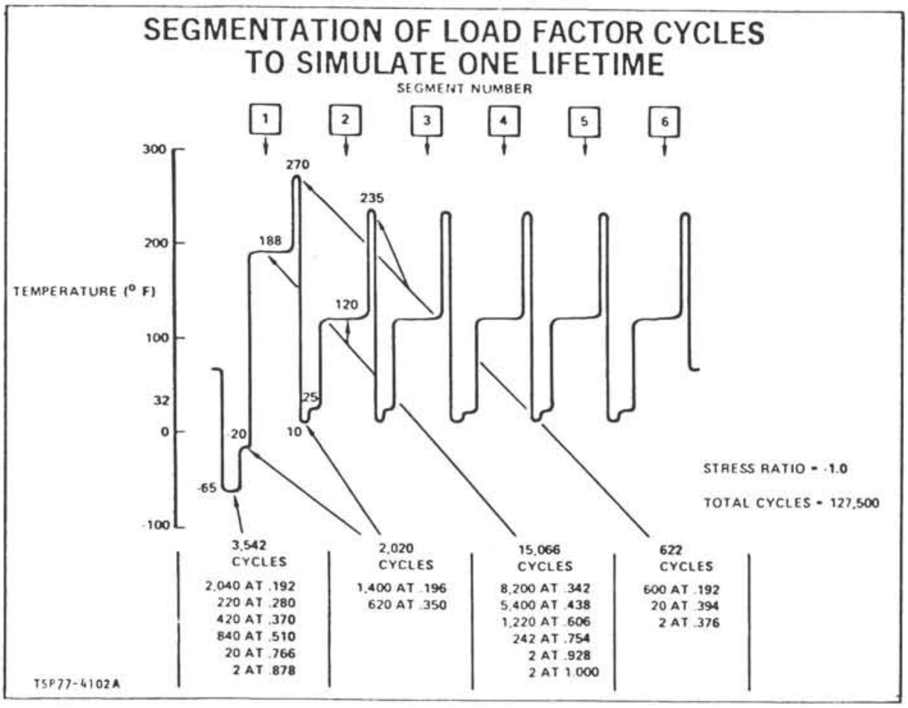


FIG. 15: SEGMENTATION OF LOAD/TEMPERATURE CYCLES[230]

COMPROMISES

- 500, 000 CYCLES ⟶ 127, 500 CYCLES

  EFFECT: CLIPPING OF LOWER STRESS EXCURSIONS

- MONOTONICALLY ARRANGE TEMPERATURE EXCURSION

  EFFECTS: REDUCES FREEZE-THAW OR LIQUID-STEAM CYCLES; REORDERS STRESS/TEMPERATURE EXCURSIONS

- REDUCE SEGMENTS FROM 100 TO 6

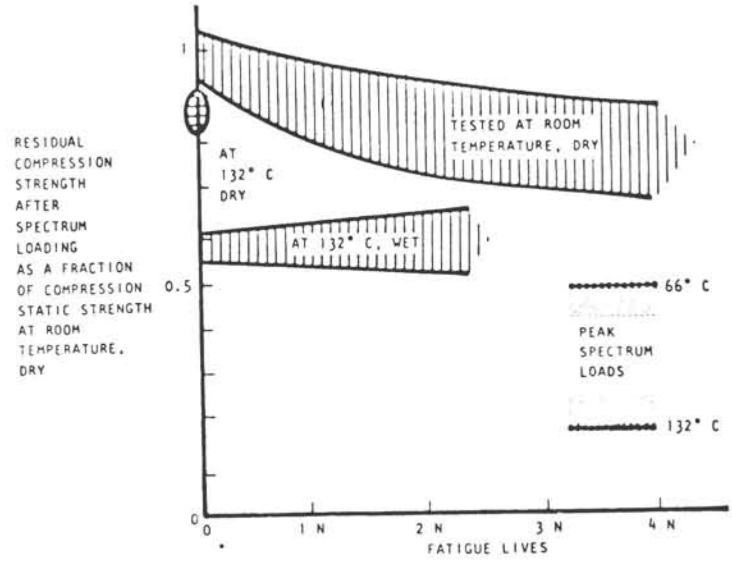
  EFFECT: REDUCES FREEZE-THAW OR LIQUID-STEAM CYCLES

- REDUCED DWELL TIME AT TEMPERATURE

  EFFECT: MAY BE SIGNIFICANT (CREEP/RELAXATION)

NOTE: FREEZE-THAW CYCLES REDUCED FROM 10, 000 TO 6
LIQUID-STEAM CYCLES REDUCED FROM 1, 300 TO 6

FIG. 16: COMPROMISES[230]



NOTE: 1 ON THE ORDINATE DESIGNATES 87,000 PSI)0.6 GP$_c$)

FIG. 17: RESIDUAL STRENGTH OF FATIGUE AS/3501-5-DEGRADES WITH TEMPERATURE, HYDRATION, PROOF LOADING, AND SIZE OF IMPERFECTION OR DAMAGE[176]

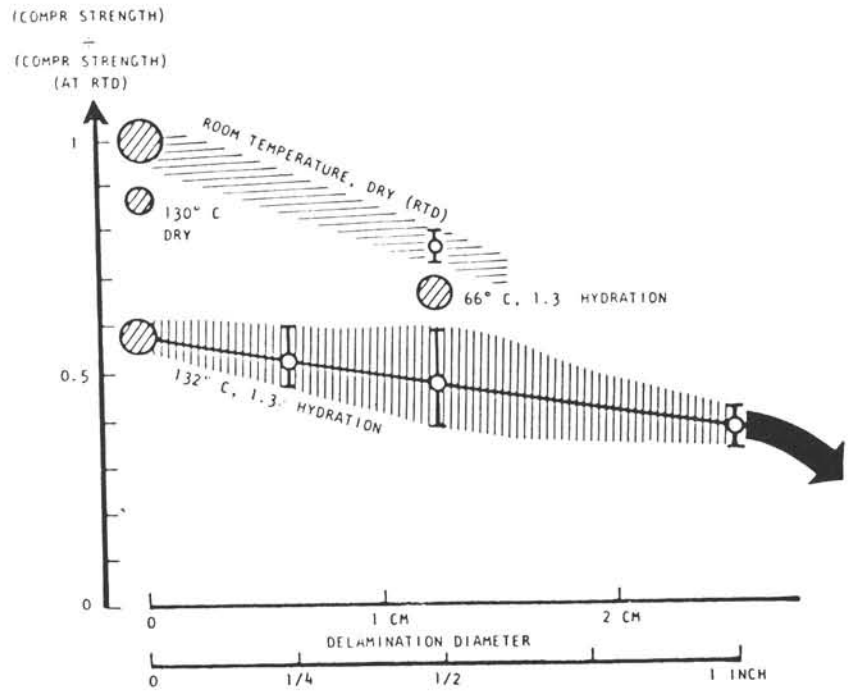


FIG. 18: THE COMPRESSIVE STRENGTH OF AS/3501-5-DEGRADES WITH TEMPERATURE, HYDRATION, PROOF LOADING, AND SIZE OF IMPERFECTION OR DAMAGE[176]

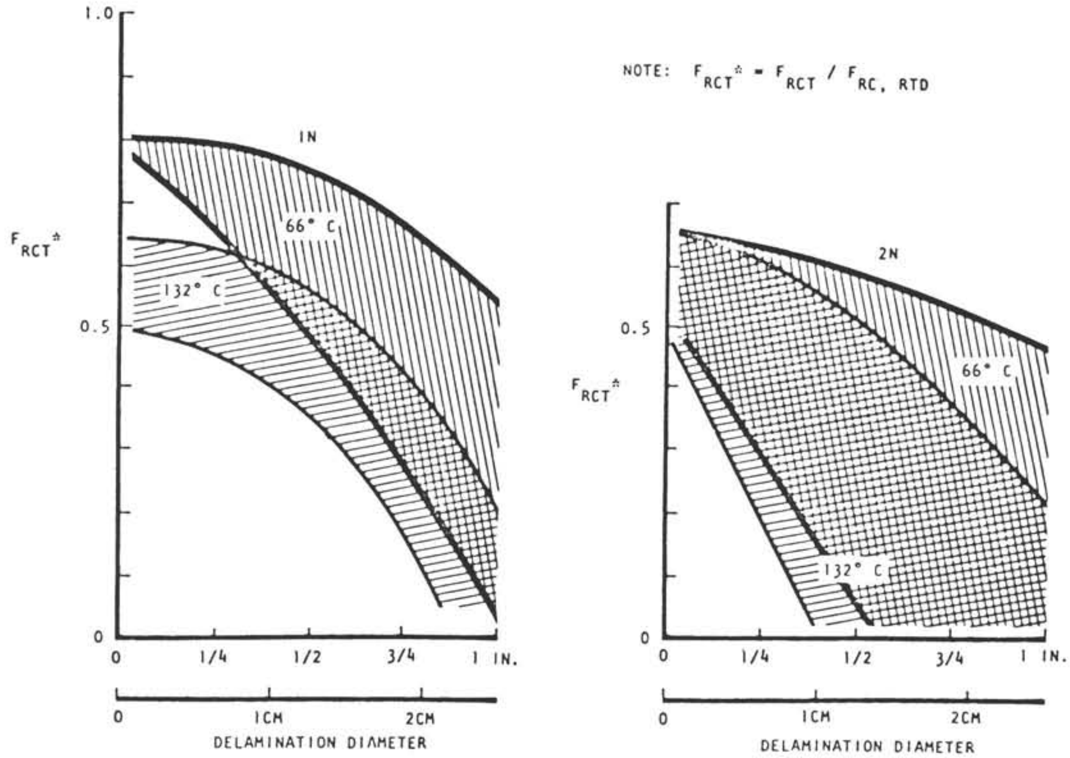


FIG. 19: RESIDUAL STRENGTH AT 66 AND 132°C (150 AND 270°F) OF FATIGUE GRAPHITE/EPOXY WITH DELAMINATIONS OF VARIOUS EXTENT[176]

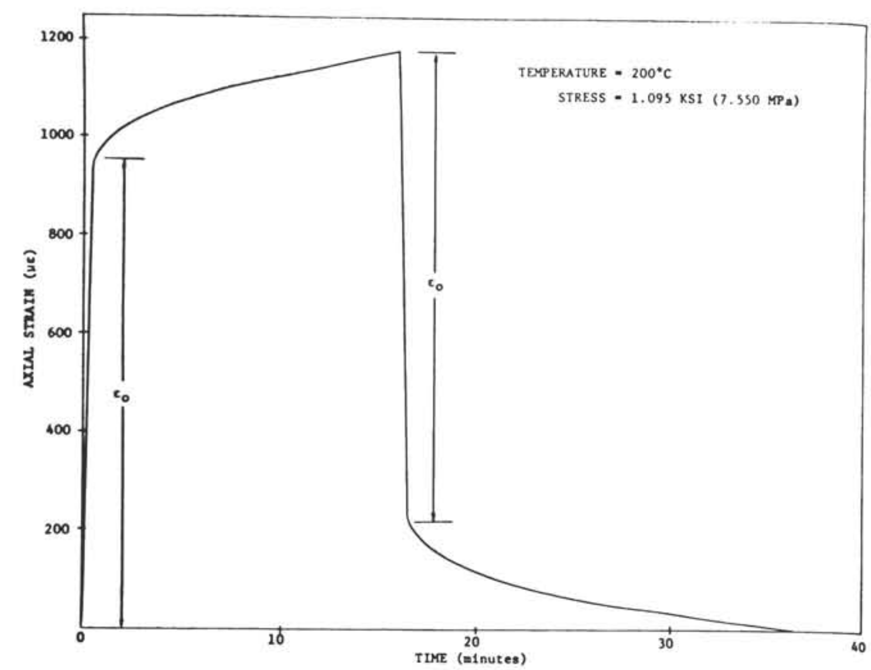


FIG. 20: CREEP AND CREEP RECOVERY OF $[90^\circ]_{8s}$ LAMINATE[328]

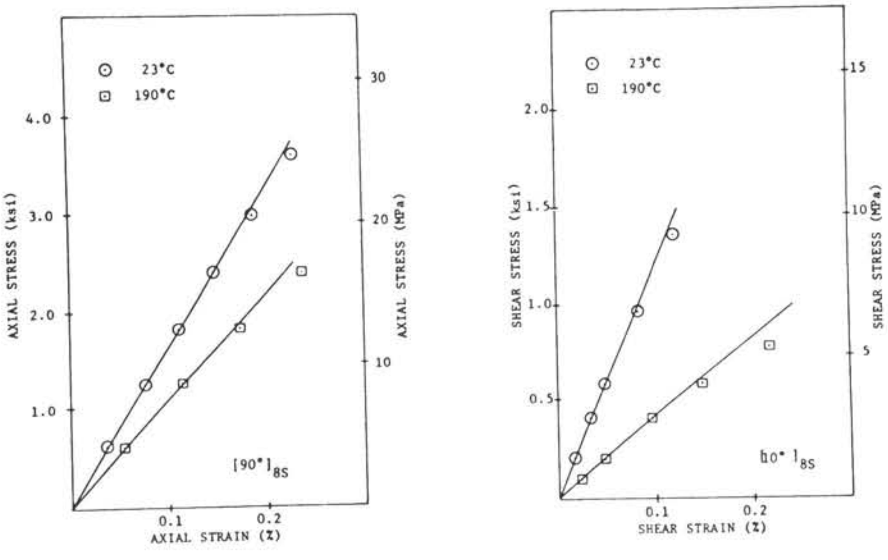


FIG. 21: LINEARITY CHECK (STRESS-STRAIN CURVES AFTER 15-MIN CREEP)[328]

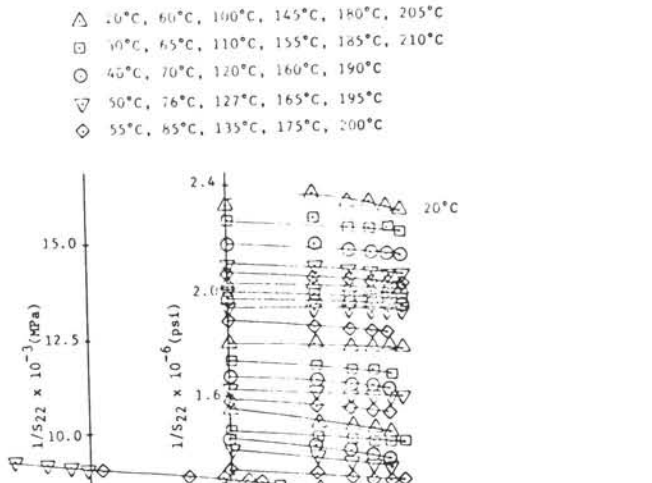


FIG. 22: REDUCED RECIPROCAL OF COMPLIANCE, $1/S_{22}$, AND PORTION OF 180°C MASTER CURVE FOR $[90°]_{8s}$ T300/934 GRAPHITE/EPOXY LAMINATE[328]

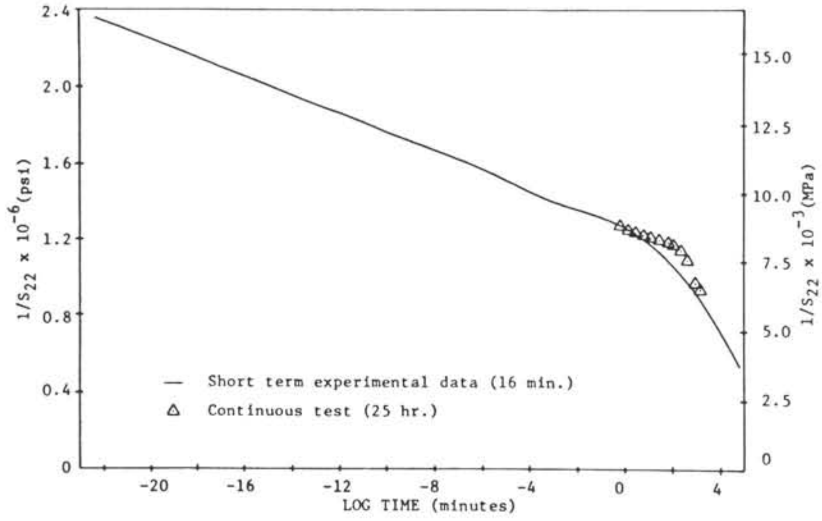


FIG. 23: MASTER CURVE OF THE RECIPROCAL OF REDUCED COMPLIANCE, $1/S_{22}$, OF $[90°]_{8s}$ LAMINATE AT 180°C[328]

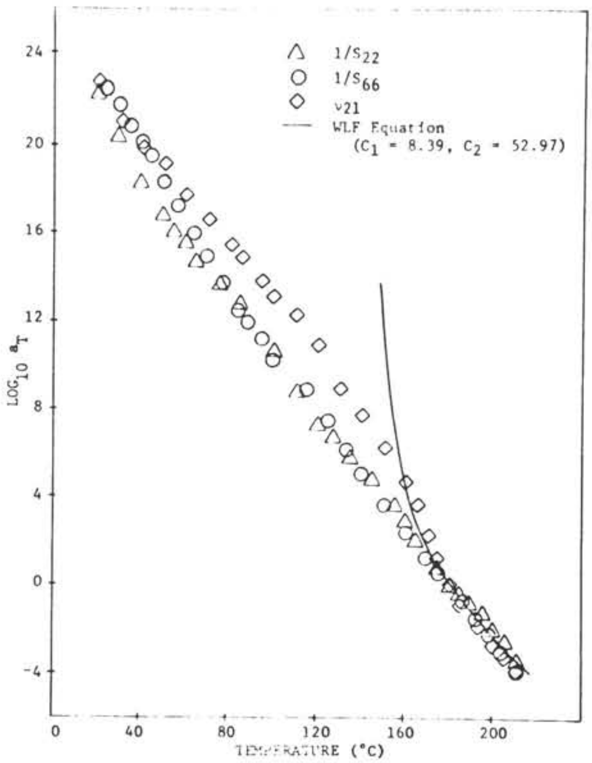


FIG. 24: LOG a_T VERSUS TEMPERATURE[328]

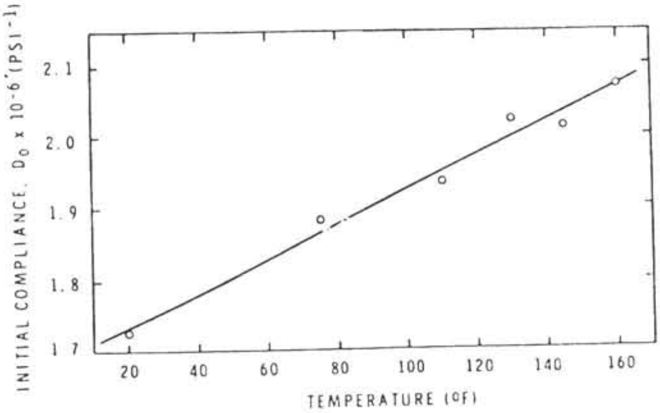


FIG. 25: TEMPERATURE DEPENDENCE OF INITIAL COMPLIANCE, D_o FOR SHELL 58-68R EPOXY[33]

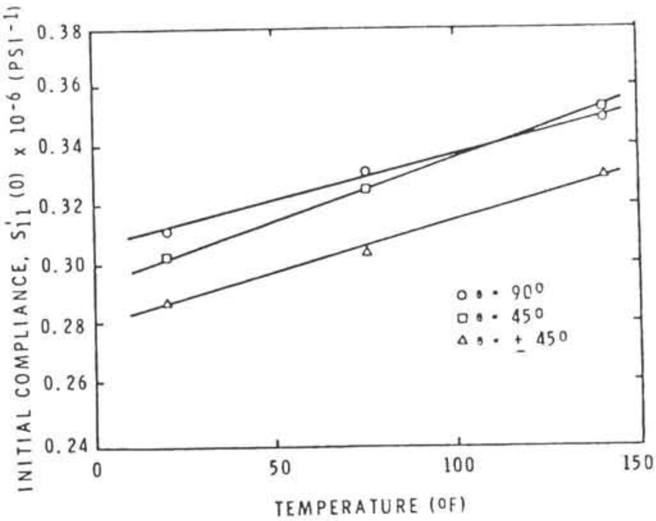


FIG. 26: TEMPERATURE DEPENDENCE OF INITITAL CREEP COMPLIANCES FOR THE GLASS/EPOXY COMPOSITE[33]

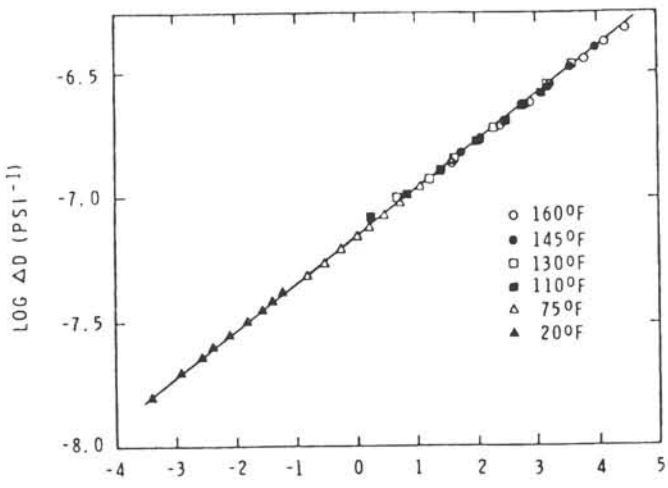


FIG. 27: MASTER CURVE FOR NET CREEP COMPLIANCE, ΔD, FOR SHELL 58-68R EPOXY[33]

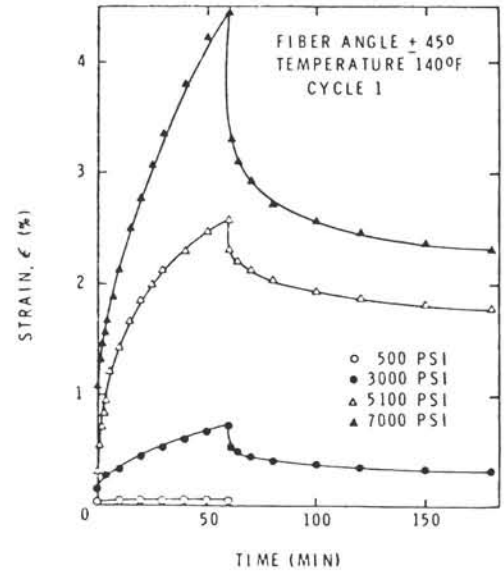


FIG. 28: CREEP AND RECOVERY ±45° GLASS/EPOXY AT 140°F (60°C)[33]

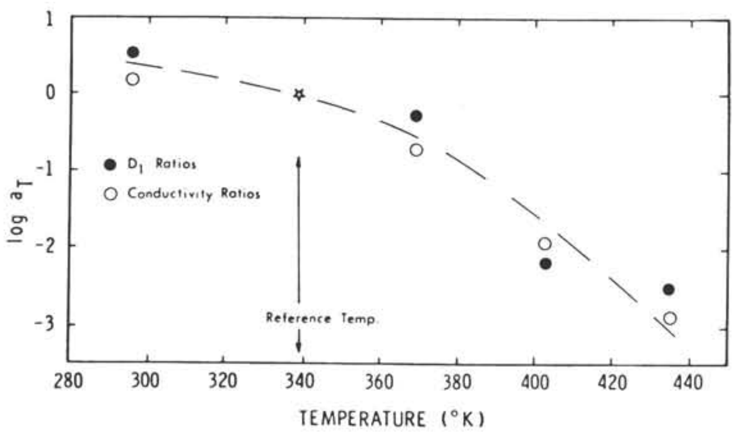


FIG. 29: TEMPERATURE DEPENDENCE OF THE SHIFT FACTOR, a_T, DETERMINED FROM ELECTRICAL CONDUCTIVITY MEASUREMENTS AND ISOTHERMAL CREEP COMPLIANCES[167]

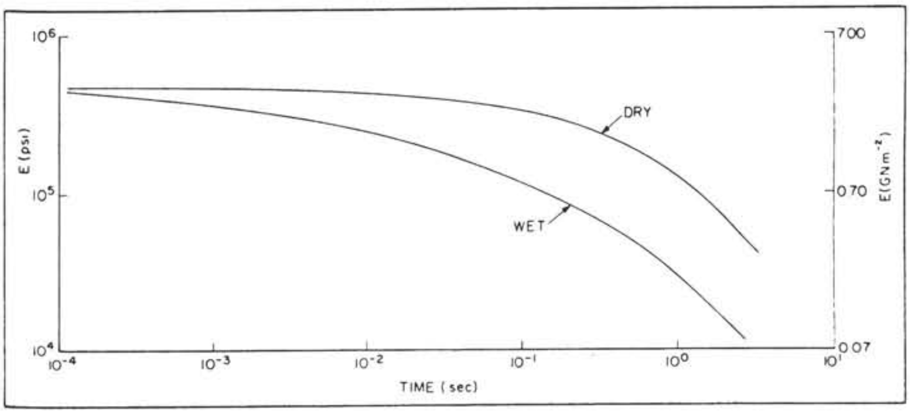


FIG. 30: RELAXATION MODULUS MASTER CURVES, REFERENCE TEMPERATURE = 300°F (149°C). EPOXY RESIN – TETRAGLYCIDYLMETHYLENE DIANILINE DIAMINE CURING AGENT[42]

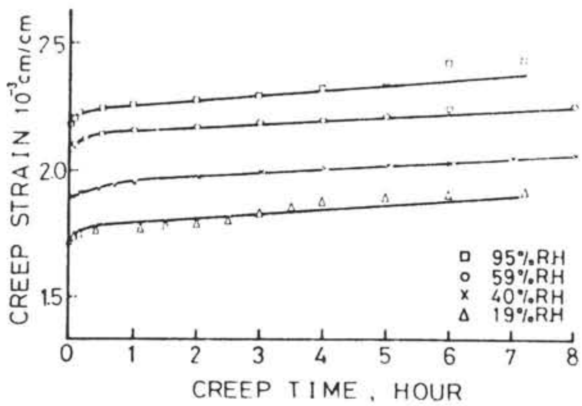


FIG. 31: CREEP STRAIN HISTORY – 21°C[304]

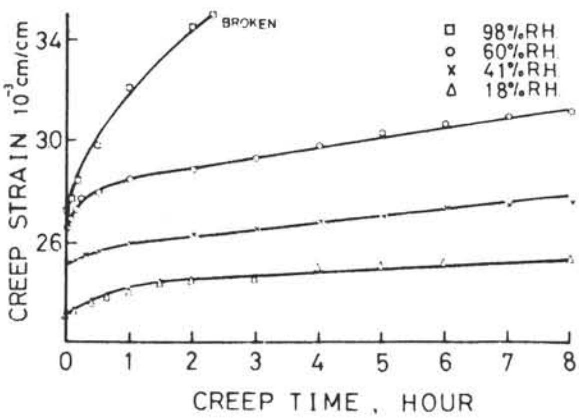


FIG. 32: CREEP STRAIN HISTORY – 65°C[304]

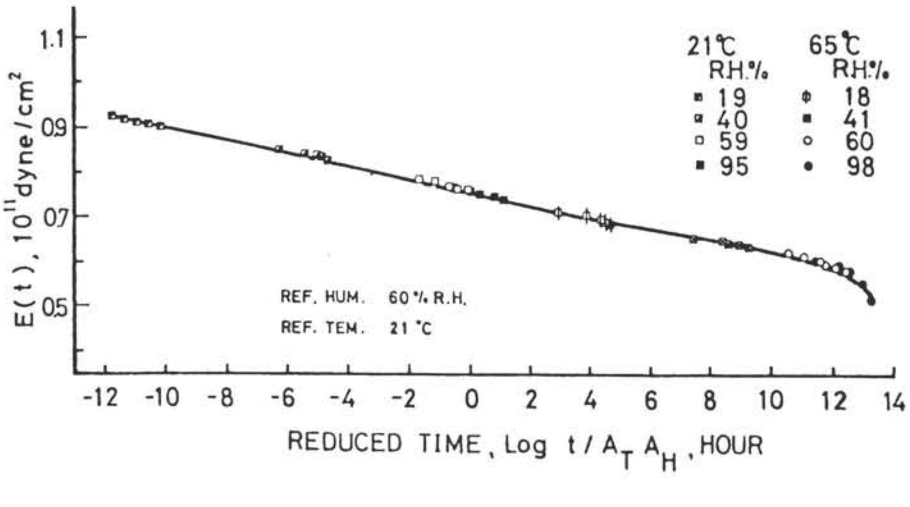


FIG. 33: COMPOSITE (MASTER) RELAXATION MODULUS UNDER TEMPERATURE-HUMIDITY EFFECTS[304]

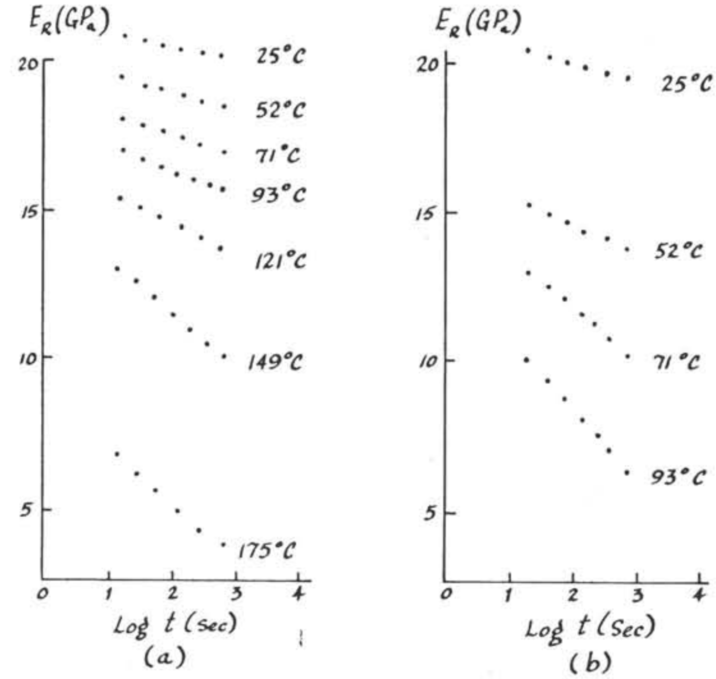


FIG. 34: RELAXATION MODULUS OF $(\pm45)_{2s}$ T300/934 LAMINATES CONTAINING (a) 0.14 PERCENT MOISTURE AND (b) 1.40 PERCENT MOISTURE[70]

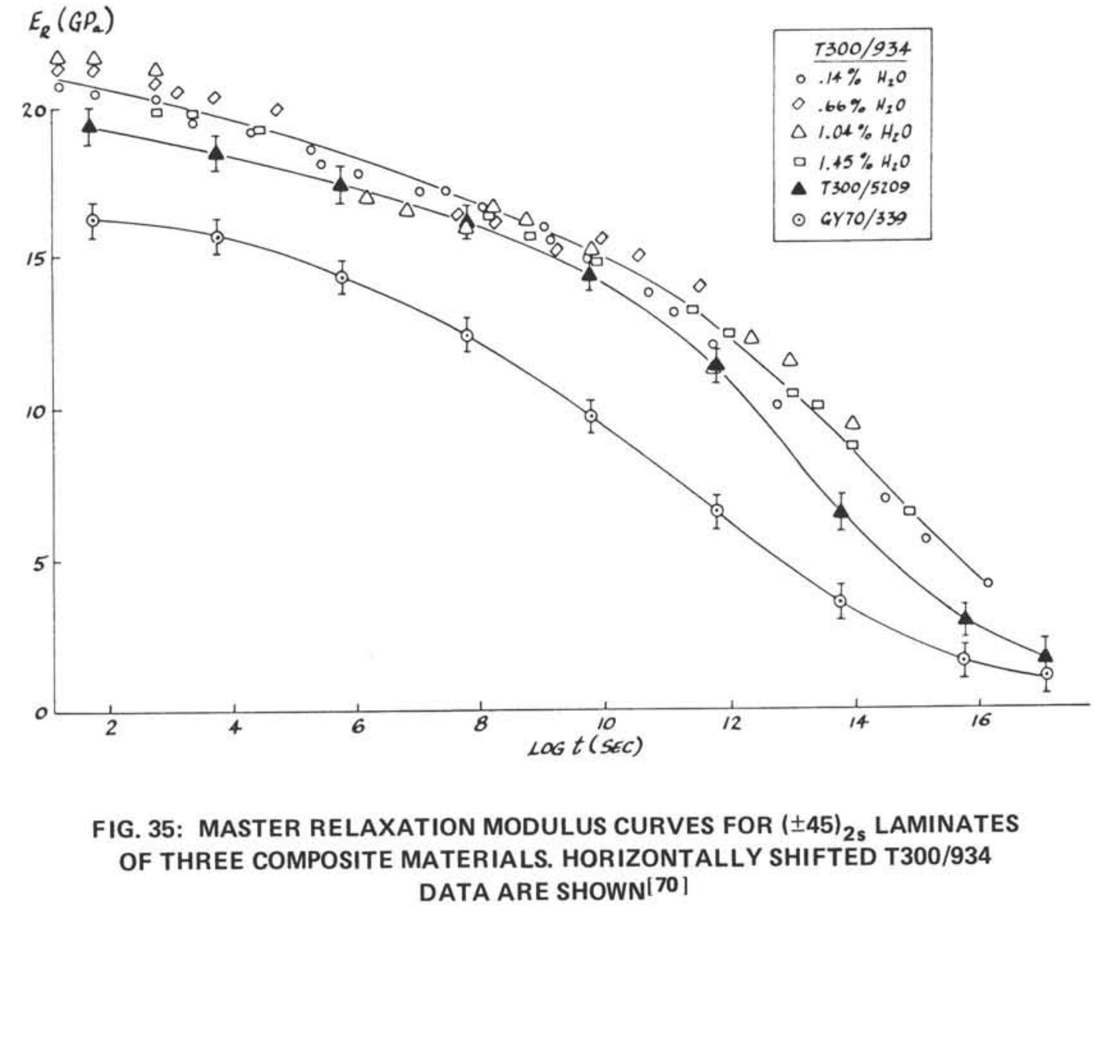


FIG. 35: MASTER RELAXATION MODULUS CURVES FOR $(\pm 45)_{2s}$ LAMINATES OF THREE COMPOSITE MATERIALS. HORIZONTALLY SHIFTED T300/934 DATA ARE SHOWN[70]

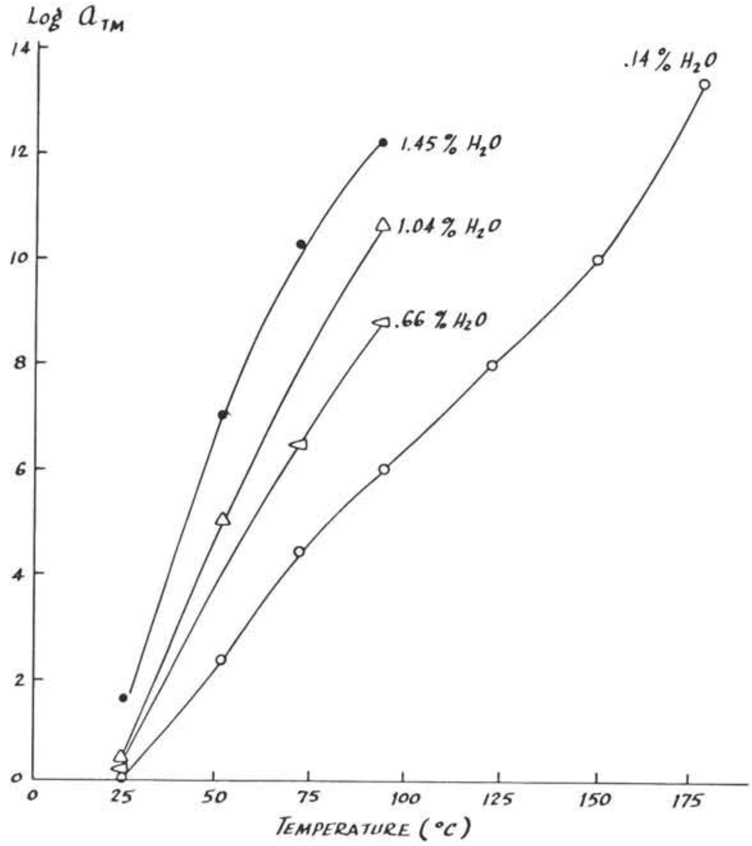


FIG. 36: SHIFT FACTOR a_{TM} FOR T300/934 VERSUS TEMPERATURE AND MOISTURE[70]

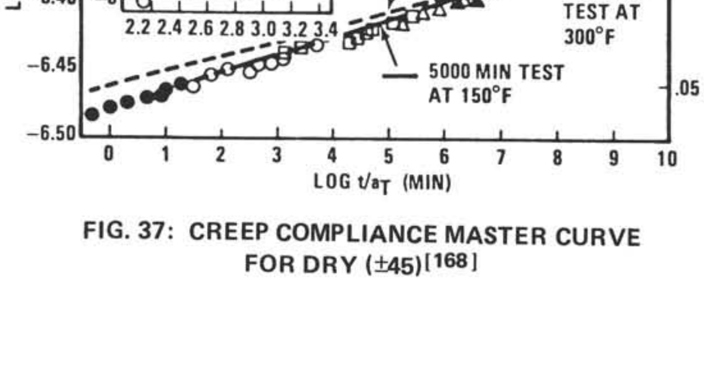


FIG. 37: CREEP COMPLIANCE MASTER CURVE FOR DRY (±45)[168]

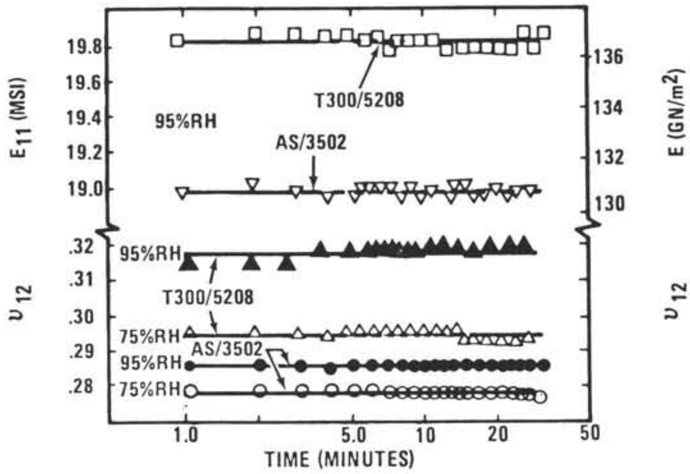


FIG. 38: TYPICAL E_{11} AND ν_{12} FOR RT WET $(0)_6$ COUPONS[168]

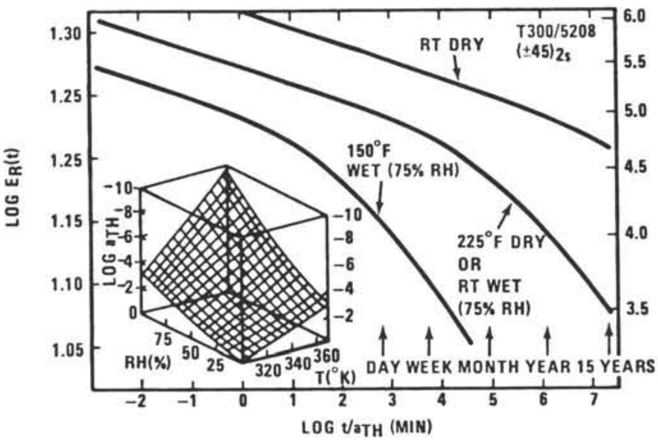


FIG. 39: RELAXATION MODULI FOR DRY (±45)[168]

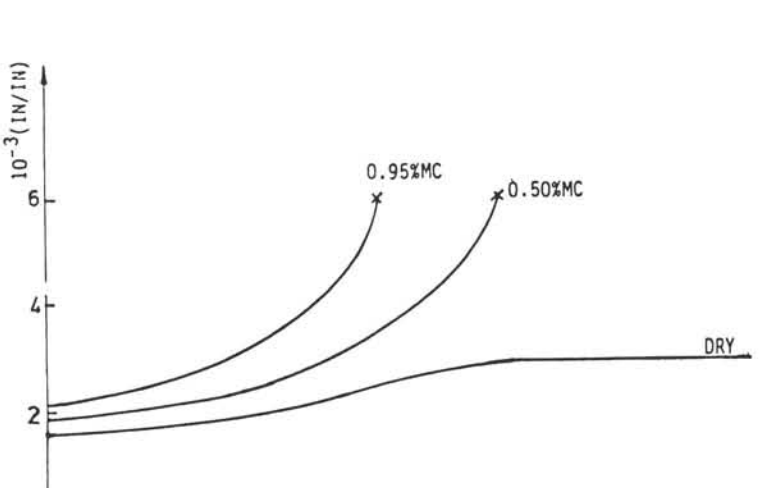


FIG. 40: CREEP OF (90°) LAMINATE AT 215°F (102°C) AND VARIOUS MOISTURE CONTENTS (900 psi = 6.2 MPa)[301]

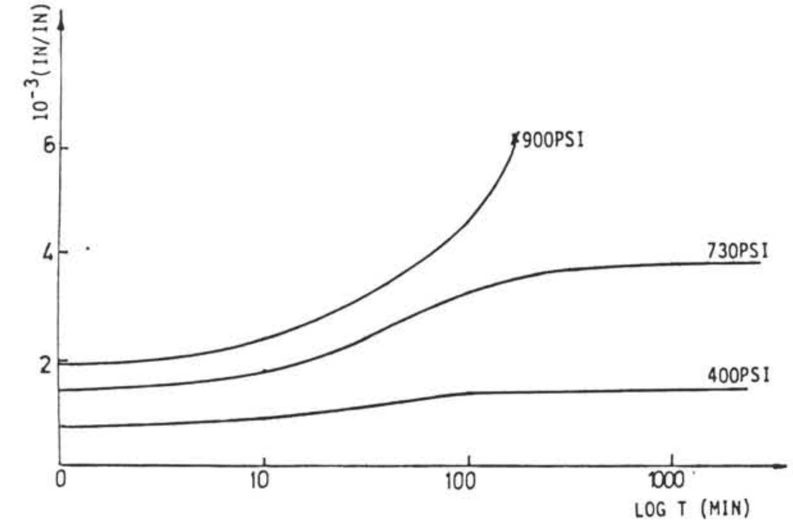


FIG. 41: THE EFFECT OF STRESS LEVEL ON CREEP STRAIN FOR (90°) LAMINATES AT 215°F (102°C) AND 0.5% MC[301]

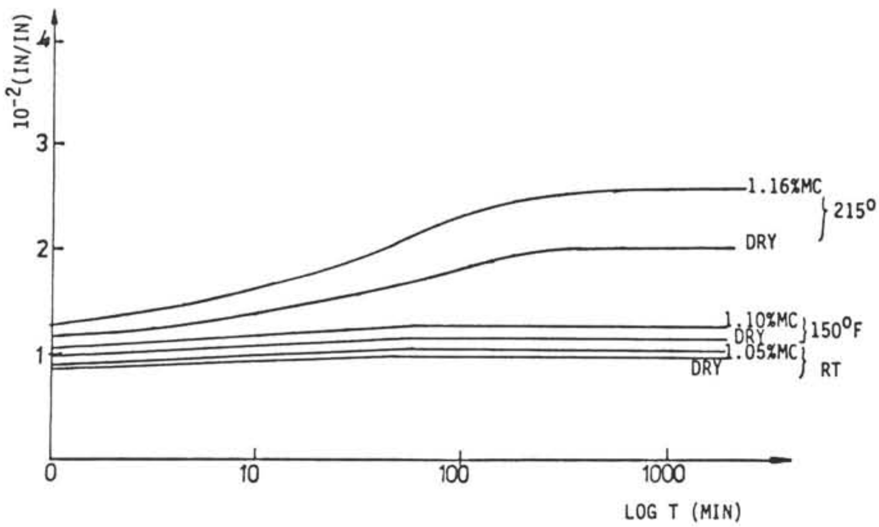


FIG. 42: CREEP OF (0°) LAMINATE UNDER VARIOUS ENVIRONMENTAL CONDITION (46600 psi = 321 MPa)[301]

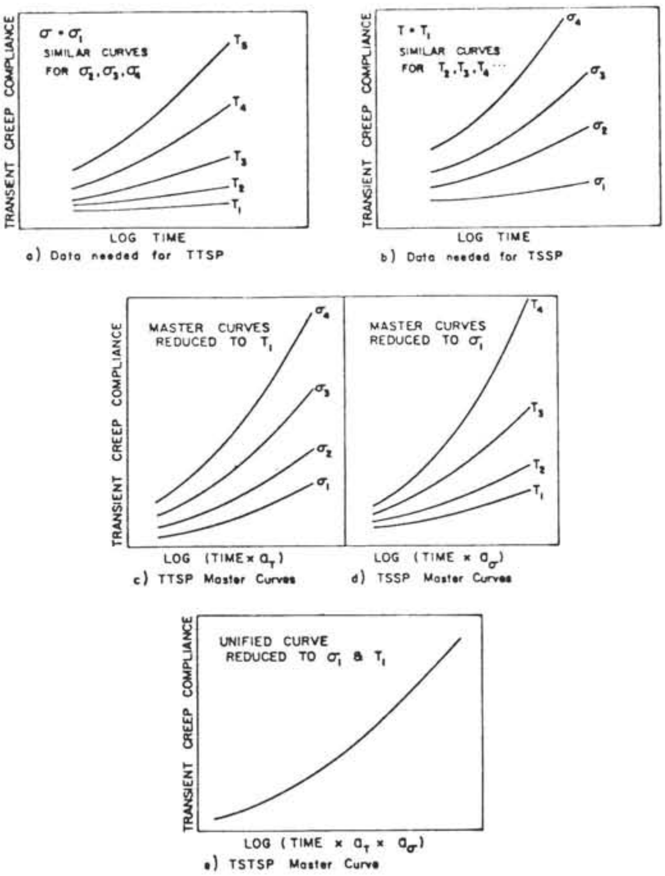


FIG. 43: SCHEMATIC DIAGRAM TO ILLUSTRATE THE TIME-STRESS-TEMPERATURE SUPERPOSITION PRINCIPLE[114]

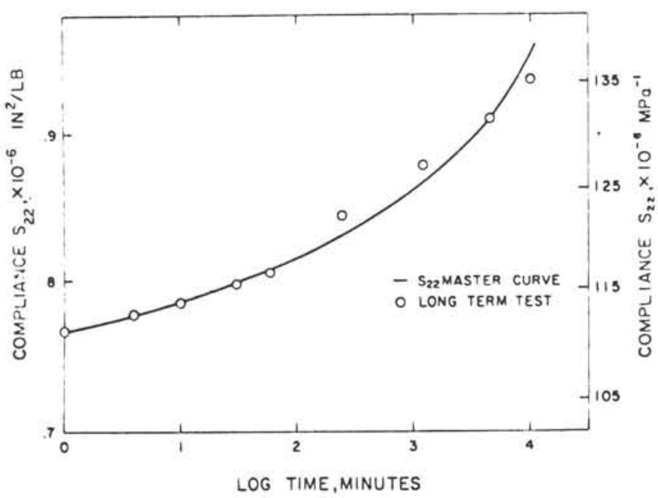


FIG. 44: COMPARISON OF S_{22} MASTER CURVE WITH A LONG TERM TEST AT 320°F (160°C) AND 2,750 psi (19 MPa)[114]

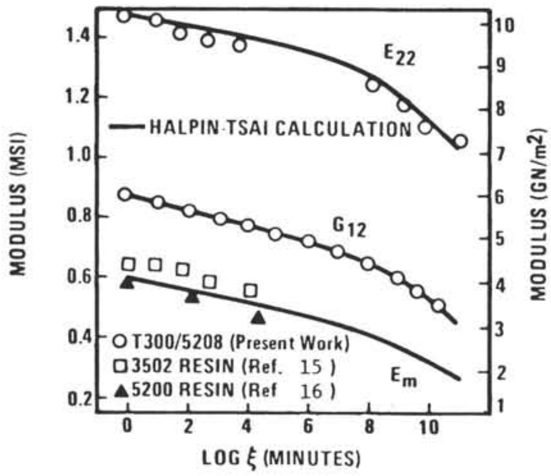


FIG. 45: MEASURED AND PREDICTED LAMINATE PROPERTIES[168]

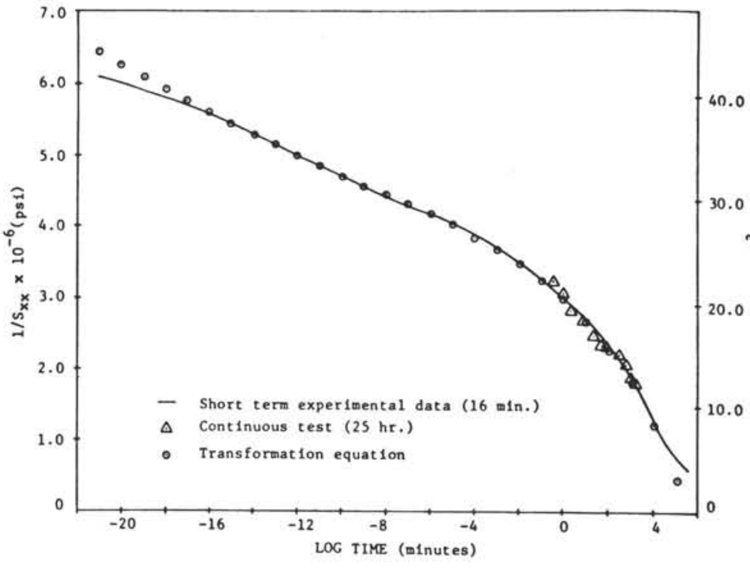


FIG. 46: MASTER CURVE OF THE RECIPROCAL OF REDUCED COMPLIANCE, $1/S_{xx}$, OF $[30^\circ]_{8s}$ LAMINATE AT 180°C[328]

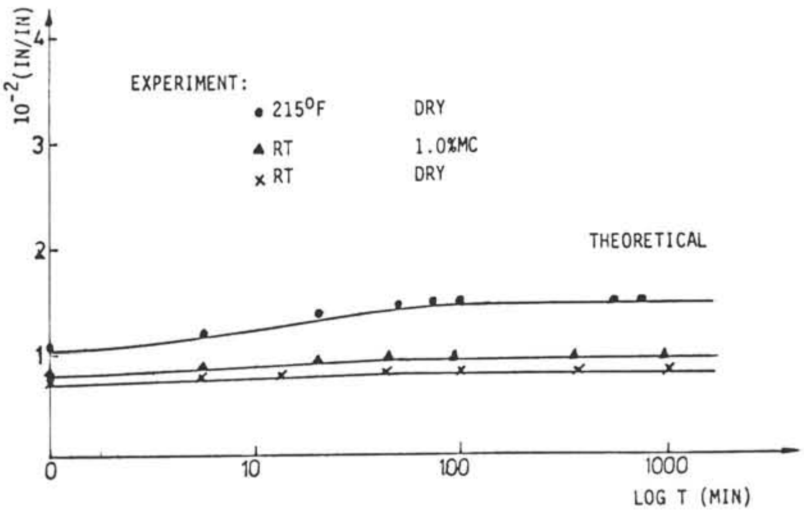


FIG. 47: COMPARISON OF THEORETICAL AND EXPERIMENTAL CREEP FOR QUASI-ISOTROPIC LAMINATE (14000 psi = 97 MPa)[301]

APPENDIX A — BIBLIOGRAPHY

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