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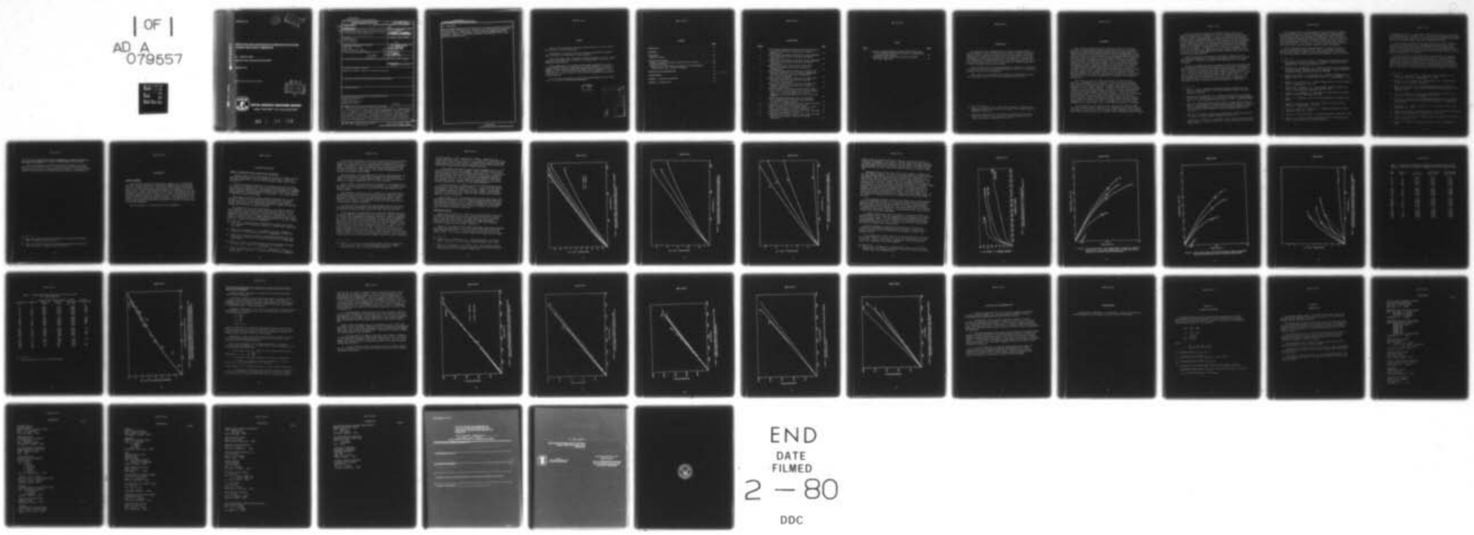

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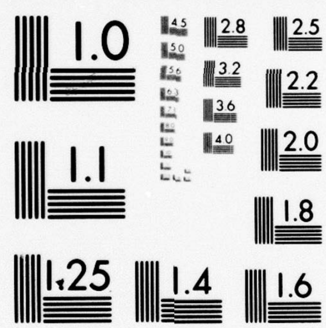

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LEVEL II

PREDICTION AND VERIFICATION OF MOISTURE EFFECTS ON CARBON FIBER-EPOXY COMPOSITES

BY JOSEPH M. AUGL

RESEARCH AND TECHNOLOGY DEPARTMENT

30 MARCH 1979

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Dahlgren, Virginia 22448 • Silver Spring, Maryland 20910

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Moisture has an effect on the elastic as well as on the strength properties of composites. In this work we have experimentally verified the prediction of changes in the longitudinal shear modules (G_{12}) as a function of moisture uptake and temperature. The properties of the bulk resin (measured independently as a function of temperature and moisture concentration) were used for predicting the composite response by using the simple Halpin-Tsai equation of micromechanics. The excellent agreement between the predicted and

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observed values demonstrates the feasibility of predicting changes in "reversible properties" of composite in a natural service environment as suggested in a previous report. This predictive scheme considered simultaneously the concepts of diffusion, environmental modeling, micromechanics, and a finite difference laminate theory.

SUMMARY

Hercules 3501-6/AS carbon fiber epoxy composites will be used in advance Navy aircraft as structural materials.

The purpose of this work is part of an effort to determine the environmental effects on the properties of these composites.

This report shows that a reasonable estimate of composite property changes, caused by the combined effect of moisture and temperature, can be made from known bulk resin behavior.

Since composites can be tailored from the same constituent materials in a great number of ways it is important to be able to predict the resulting changes for some projected service environment without an excessive number of expensive tests. It has been shown that such predictive methods are quite promising and, therefore, may be of great practical importance for design and systems engineers.

This work was sponsored by the Naval Air Systems Command during FY 1978, under the Task number A3200000/004A/9R02200000.

J. R. Dixon

J. R. Dixon
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INTRODUCTION

The purpose of this investigation was to verify a scheme proposed in previous reports[1,2] which would allow one to predict the reversible property changes in fiber reinforced structural materials under outdoor and service environments. Although the work was carried out specifically on a Hercules 3501-6/AS carbon fiber epoxy composite, it is expected that it applies for other organic matrix composites if the necessary constituent material data of the fibers and resins are available, and as long as a reasonably good matrix to fiber interfacial bonding can be assumed.

Since the Navy plans to use Hercules 3501-6/AS composites as structural materials for the future F-18 and the AV8B Harrier aircraft, this material was a natural choice for the investigation.

This is part of a continuing effort designed to understand and predict the environmental effects on fiber reinforced composite materials.

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1. Augl, J.M. and Berger, A.E., "The Effect of Moisture on Carbon Fiber Reinforced Composites IV Prediction of Changes in the Elastic Behavior," NSWC/WOL TR 77-61, 1977.

2. Augl, J.M. and Berger, A.E., "The Effect of Moisture on Carbon Fiber Reinforced Composites III Prediction of Moisture Sorption in a Real Outdoor Environment," NSWC/WOL TR 77-13, 1977.

BACKGROUND

Fiber reinforced composites will be used more and more as structural materials in advanced military weapons systems such as aircraft and missiles. During the last two decades there have been a number of advances made that allows one to predict the mechanical behaviors of these nonhomogeneous, anisotropic materials. The elastic behavior of a undirectional lamina and even its strength, its elasto-plastic, and its viscoelastic behavior can be reasonably well predicted by micromechanical theories from its constituent properties (resin and fiber). Laminate theory allows us, also, to predict the elastic behavior of multilayer angle-ply laminates. The strength of multilayer angle-ply composites is more a question of definition since there can be various irreversible property degradations without total fracture (e.g., partial cracking of the 90° plies in a 0/90° crossplied laminate when loaded in the 0° direction, such, that the ultimate strain of the 90° plies are exceeded). For practical purposes various design criteria, as those described in the Air Force Design Guide, are such that the stresses or strains do not exceed values that would lead to intolerable degradation of stiffnesses and to permanent deformations.

Most failure criteria for lamina and laminates are more intuitive, empirical or phenomenological than based on a solid theoretical foundation. Only recently, attempts have been made to predict failure modes and mechanisms under various loading conditions in unidirectional composites by finite difference or finite element methods. It appears that these numerical methods, though not without their shortcomings, will lead to a better understanding of the actual failure mechanisms in composite materials. Notably the finite element method (FEM) is particularly useful because it can be used for modeling various effects (such as multiaxial loading conditions, less than ideal resin-fiber bond strength and debonding, residual thermal stresses, stresses due to thermal excursions, creep under multiaxial loading, and elasto-plastic deformations) on a microscopic scale, of the magnitude of less than a fiber diameter, and on a macroscopic scale, on the order of several ply thicknesses.

Yet the properties of an organic matrix composite will undergo changes partially caused by loading conditions of its intended mission (static, dynamic and thermal) and partially by the environment itself (temperature and humidity). Each of those conditions may lead to reversible and irreversible property changes.

Aside from excessive, mechanical or thermal loading conditions, it appears that moisture will perhaps lead to the most significant property changes in organic matrix composites at least for the matrix dominated lamina properties such as intralamina shear, transverse tensile and both transverse and longitudinal compressive properties. Since moisture will have to penetrate into the composite to affect its properties and since the rate of penetration depends on the temperature and on the moisture concentration these property changes will be gradual, strongly dependent on the thickness of the composite and on the environment it is exposed to. The question is, therefore: will one be able to predict or at least estimate these property changes with time or must one rely on testing combinations of many variables which would quickly discourage the design engineer?

Several investigators have presented experimental evidence that the matrix dominated properties of advanced organic matrix composites are degraded by moisture.[3-7] This effect increases as the temperature of the composite is raised.[6]

We have also demonstrated that the property deterioration levels out after the absorbed moisture has reached equilibrium with the surrounding humidity[6] and is therefore, a function of the average relative humidity. This deterioration is reversible for matrixes that are not chemically changed by moisture (as is the case for most amine cured epoxy composites where moisture is thought only to plasticize the resin and thus make it more flexible).[5,6] It is not only the glass transition temperature of the resin that is lowered but the whole modulus curve is affected far below glass transition temperature and this effect increases as the glass transition temperature is approached.

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3. Hertz, J., et al., "Advanced Composite Application for Spacecraft and Missiles," AFML-TR-71-186, Vol. II (General Dynamics-Convair, San Diego, California), Mar 1972.

4. Browning, C.E., and Whitney, J.E., "The Effect of Moisture on the Properties of High Performance Structural Resins and Composites," American Chem. Soc., Div. of Org. Coatings and Plastic Chem., 33, No. 2, 1973, pp. 137-148.

5. Augl, J.M., "Environmental Degradation Studies on Carbon Fiber Reinforced Epoxies," Paper presented at the TTCP-Panel P3 Meeting, Melbourne, Australia, 1975, and paper presented at the Air Force Workshop on Durability Characteristics of Resin Matrix Composites, Battelle-Columbus Laboratories, Ohio, 1975.

6. Augl, J.M., "The Effect of Moisture on Carbon Fiber Reinforced Composites. II Mechanical Property Changes," NSWC/WOL TR 76-149, 1977.

7. Browning, C.E., Husman, G.E., and Whitney, J.M., "Moisture Effects in Epoxy Matrix Composites," Composite Materials: Testing and Design, ASTM STP 617, 1976, p. 481.

This is important for the understanding of the deterioration of properties in composites. Under reversible property changes it is meant that if the moisture is removed (in vacuum or by heating) the original properties will be regained within the experimental uncertainties. We do not intend to review here all the experimental evidences of moisture effects on fiber reinforced composites but refer only to a number of additional papers dealing with this subject[8-13]. The question is: can we quantitatively describe and thus predict these composite property changes in a real environment?

Various investigators have proposed schemes of solving the problem to predict the macroscopic lamina properties from its constituent properties (of matrix and fiber) by analytic or numerical methods.[14-18] Finite difference and finite element methods proved to be quite powerful tools in this area

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8. Hofer, K.E., Rao, N., and Larson, D., "Development of Engineering Data on the Mechanical and Physical Properties of Advanced Composite Materials," AFML-TR-72-205, IIT Research Institute, 1974.

9. Browning, C.E., and Hartness, J.T., "Effect of Moisture on the Properties of High Performance Structural Resins and Composites," Composite Materials: Testing and Design, ASTM STP 546, 1974, pp. 284-302.

10. Kaelble, D.H., Dynes, P.J., and Crane, L.W., "Interfacial Mechanisms of Moisture Degradation in Graphite-Epoxy Composites," J. of Adhesion, 7, 25, 1977.

11. Kaelble, D.H., and Dynes, P.J., "Surface Energy Analysis of Treated Graphite Fibers," J. Adhesion, 6, 239, 1974.

12. Ashbee, K.H.G., and Wyatt, R.C., "Water Damage in Glass Fiber/Resin Composites," Proc. Royal Soc., Series A, 312, 553, 1969.

13. Halpin, J.C., and Pagano, N.J., "Consequences of Environmentally Induced Dilation in Solids," AFML-TR-68-395, 1969.

14. Ekvall, J.C., ASME Paper 61 AV-56, Aviation Conference, Los Angeles, Mar 1961.

15. Haskin, Z., and Rosen, B.W., "The Elastic Moduli of Fiber Reinforced Materials," J. Appl. Mech., 223, 1969.

16. Rosen, B.W., Don, N.F., and Haskin, Z., "Mechanical Properties of Fiber Composites," NASA CR-31, 1969.

17. Tsai, S.W., "Structural Behavior of Composite Materials," NASA CR-71, 1964.

18. Ekvall, J.C., AIAA 6th Structural and Material Conf., Palm Springs, Apr 1964, "Failure Mechanisms in Composite Systems."

of micromechanics.[19-23] The Halpin-Tsai equations (see Appendix A) that relate the constituent elastic constants (E_m, ν_m, G_m, E_{12f} E_{22f} ν_{12f} G_{12f}) to the lamina elastic constants (E_{11}, E_{22}, G_{12}, $_{12}$) for any given fiber volume fraction (V_f) are useful for design estimates and rapid computational purposes.

The next step in predicting the elastic behavior of multilayer angle-ply laminates from lamina properties is the subject of laminate theory. Since there are several excellent texts available[24-27] we shall not discuss this subject here but simply state that it is a procedure of summing up the directional elastic properties of all the plies in the composite, to give the overall macroscopic behavior of the laminate.

The question of how to use these theories when one expects to have non-uniform and continuously changing matrix properties due to variable moisture concentration we have discussed at length in References 1 and 2, where we described the concept of using finite Δ- layers for obtaining the composite elastic behavior and the internal moisture distribution as a function of time when at least average climatic data such as average temperatures and humidities are available. The approach described there was essentially a combination

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19. Adams, D.F., and Donner, D.R., "Transverse. Normal Loading of a Unidirectional Composite," J. Comp. Mat., 1, 152, 1967.

20. Karlak, R.F., and Crossman, F.W., LMSC-D457462, 1975. Lockheed Missile and Space Corp., Palo Alto, California.

21. Baker, J.D., and Foye, R.L., "Advanced Design Concepts for Advanced Composite Airframes," North American Rockwell Corp. AFML Contract F33616-68-C-1199, 1969.

22. Lin, T.H., Salina, D., and Ito, Y.M., "Elasto-Plastic Analysis of Unidirectional Composites," J. Comp. Mat., 6, 48, 1972.

23. Adams, F.D., and Miller, A.K., "Hygrothermal Microstresses in a Unidirectional Composite Exhibiting Inelastic Material Behavior," J. Comp. Mat., 11, 285, 1977.

24. Lekhnitskii, S.G., "Theory of Elasticity of an Anistropic Elastic Body," Holden-Day, Inc., 1963.

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26. Vinson, J.R., and Chou, T.W., "Composite Materials and Their Use in Structures," John Wiley, 1975.

27. Tsai, S.W., "Mechanics of Composite Materials. Part II," AFML-TR-149, 1966.

of the concepts of moisture diffusion, micromechanics, laminate theory and of modeling the environment such that the diffusion equation can be solved.

Thus it is necessary to determine the moisture diffusion and sorption behavior of the constituent resin. The data for the 3501-6 have been determined and will be given in a separate Technical Report.[28] The necessary resin property data have likewise been measured and are reported in Reference 29.

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28. Augl, J.M., "Moisture Sorption and Diffusion in Hercules 3501-6 Epoxy Resin," NSWC/WOL TR 79-39 (in print).

29. Augl, J.M., "Moisture Effects on the Mechanical Properties of Hercules 3501-6 Epoxy Resin," NSWC/WOL TR 79-41 (in print).

EXPERIMENTAL

COMPOSITE SAMPLES

The 8 ply $(\underline{+}45°)_{2S}$ 3501-6/AS carbon fiber composite with a fiber volume fraction of .62 was fabricated by the McDonnell Douglas Aircraft Corporation under the conditions that would be used for the aircraft structural materials. The 16 x 16 x 0.05 inches^3 panels were machined into specimens of 6.5 x 0.75 square inches. Biaxial strain gages were bonded to the specimens and then the specimens were dried at 105°C for 160 hours in vacuum. The specimens were then separated in groups which were exposed to 33, 55, 80, and 100 percent RH, and one set was kept at zero percent relative humidity. The humidity exposure was 50 days at 71°C, whereupon the samples were stored at room temperature in containers, above mixtures of various salts and water, to maintain the same respective relative humidities.

The cure schedule of the laminates is given in Appendix B.

DISCUSSION AND RESULTS

Change in Longitudinal Shear Properties Due to Moisture

The main purpose of these measurements was primarily to compare theoretically predicted changes in the G_{12} longitudinal shear moduli of Hercules 3501-6/AS composites as a result of moisture exposure rather than to obtain design data.

As we have proposed in Reference 2, if the matrix properties are known as a function of temperature and of moisture concentration, and if the average internal moisture concentration in the composite can be determined or estimated (from sorption and diffusion experiments together with climatic data such as average humidities and temperatures), then the changes in the elastic behavior of composites should be predictable.

These changes affected by moisture should be observable for all resin dominated properties such as longitudinal shear modules (G_{12}) and shear strength (S_{s12}), the corresponding transverse properties, E_{22} and S_{22}, in tension and compression, the longitudinal compressive strength, the inter-ply strength and the flexural strength and modules.

The longitudinal (also called interlaminar, or intralaminar) shear properties appeared to be the most suitable properties for a test of the proposed predictive scheme of Reference 2 for the following reasons: 1) the longitudinal shear properties are quite sensitive to moisture effects; 2) the mechanical analysis of the $\pm 45°$ laminate specimen is sufficiently well established on a theoretical and on an experimental basis[30-34]; 3) the $\pm 45°$ laminate shear test is simple and relatively inexpensive.

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30. Petit, P.H., "A Simple Method of Determining the Inplane Shear Stress-Strain Response of Unidirectional Composites," ASTM STP 460, Philadelphia, Pa., (1969).

31. Pipes, R.B., and Pagano, N.J., "Interlaminar Stresses in Composite Laminates Under Uniform Axial Extension," J. Comp. Mat., 4, 538 (1970).

32. Rosen, W.B., "A Simple Procedure for Experimental Determination of the Longitudinal Shear Modulus of Unidirectional Composites," J. Comp. Mat., 6, 552 (1972).

33. Hahn, H.T., "A Note on Determination of the Shear Stress-Strain Response of Unidirectional Composites," J. Comp. Mat., 7, 383 (1973).

34. Chiao, C.C., Moore, R.L., and Chiao, T.T., "Measurement of Shear Properties of Fiber Composites. I. Evaluation of Test Methods," Composites, 8, 161 (1977).

We have shown in Reference 2 that for the prediction of the initial shear modulus it is not essential that the internal moisture distribution in the composite be known as long as the average moisture concentration can be determined. The difference in G_{12} of composites with a uniform and a nonuniform moisture concentration, though with the same average concentration is, for all practical purposes, small enough to fall within the experimental error spread of the mechanical measurements.

Thus we decided to prepare samples with different, uniform moisture concentrations (obtained by equilibrating the samples at different moisture levels: 0, 33, 55, 80, and 100% relative humidity) and test these groups of samples at different temperatures (21°, 60°, 100°, and 150°C).

Then, in order to calculate the elastic constants of the composite as a function of moisture concentration and of temperature, the corresponding resin and fiber elastic constants have to be known for the same temperature and humidity range.

Micromechanical analysis has led to a simple set of equations known as Halpin-Tsai equations (see Appendix A) which relate the constituent elastic constants (of resin and fiber) to the unidirectional ply elastic constants. The essential validity of these equations has been confirmed by a large number of finite element calculations.[20] We have used these equations here to calculate the composite properties.

The necessary resin properties have been determined on the neat resin which has also been equilibrated at the same moisture levels (0, 33, 55, 80 and 100% RH) and which has been tested at 21°, 60°, 100°, and 150°C.[29]

All micromechanical approaches have the obvious shortcomings of having to use some simplified, mathematically tractable models. Some of these models use regular fiber arrangements (tetragonal-, hexagonal-, diamond- arrangement, etc.), they assume ideal interfacial bonding, no voids, perfect alignment of the fibers, no residual stress concentration, the matrix has everywhere the same properties as the bulk resin, the fibers have the same diameter and are flawless and both resin and fiber behave linearly elastic. However, it should be noted that the finite element technique can take into account: voids, partial debonding between resin and fiber, elastoplastic deformations, viscoelastic phenomena, residual stresses due to cooling from cure temperature, thermal stresses due to heating or cooling, stresses generated due to moisture sorption, effect of multiaxial and multidirectional stresses. Excellent papers on these subjects have been published by Adams[19], Karlak[20], Branca[35], Herakovich,

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35. Branca, T.R., "Creep of a Uniaxial Metal Matrix Composite Subjected to Axial and Normal Lateral Loads," NAVAIRSYSCOM Contract No. N00019-71-C-0323 (1971), Univ. of Illinois, T.E.A.M. Report 341.

et al[36], and Ebert, et al[37], among others. However, imperfections are usually taken into account, if at all, one at a time, and in dimensions within a unit cell (= fiber to fiber distance). Micromechanics appears to give some understanding of the various failure mechanisms, the cause and even the location of the initiation under specific loading conditions.

A real composite has neither a regular fiber arrangement nor is it void-free, nor has it perfect fiber alignment. There are stress concentrations, residual stresses, partial fiber debonding, microcracks, etc. It is therefore not surprising that the macroscopic quantitative results differ frequently more than one would like. Yet, we think that a micromechanics approach is still a valid concept as to the reversible moisture effects if the results are normalized to real, uniaxial lamina data (standardized to some uniform moisture concentration, preferably to "dry lamina properties."

If moisture penetrates a composite, most of the above mentioned deviations from the ideal model remain unchanged (i.e., there are no new fabrication parameters, fiber misalignments, or voids). Though in certain cases there may be a weakening of the interfacial bond strength. This might be the case for certain glass fiber composites which require special coupling agents for fiber resin wetting.

Thus we expect to see a considerably better internal consistency in the predicted and experimental results by simply changing the matrix properties with moisture and temperature than if different polymer matrixes would have been used with equivalent elastic constants (corresponding to these temperatures and moisture loadings) because chemically different resins usually require different fabrication procedures.

Experimental Results

Since it was not possible to obtain the total stress strain curves with the bonded strain gages, only the initial strains were measured (in the 0° and 90° direction) with biaxial strain gages while the rest of the stress-strain curves were measured with a uniaxial strain gage extensometer. Unfortunately none of the strain gages exposed to 100% relative humidity conditions survived the harsh exposure conditions.

Figures 1-3 show typical initial shear stress-shear strain curves obtained from strain gage measurements. The initial shear modulus was derived from the straight line portion of the stress strain curves between strains from .0008 to .0018. In all cases an apparent higher initial modulus at very small

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36. Reniere, G.D., and Herakovich, C.T., "Nonlinear-Analysis of Laminated Fibrous Composites," Doctoral Thesis, Virginia Polytech. Inst., 1976.

37. Ebert, L.J., Griesbach, T.J., and Flynn, P.L., "Finite Element Analysis System for the Mechanical Behavior of Oriented Fiber Composite Materials Under Combined Stresses," AFOSR-TR-75-0042, 1974.

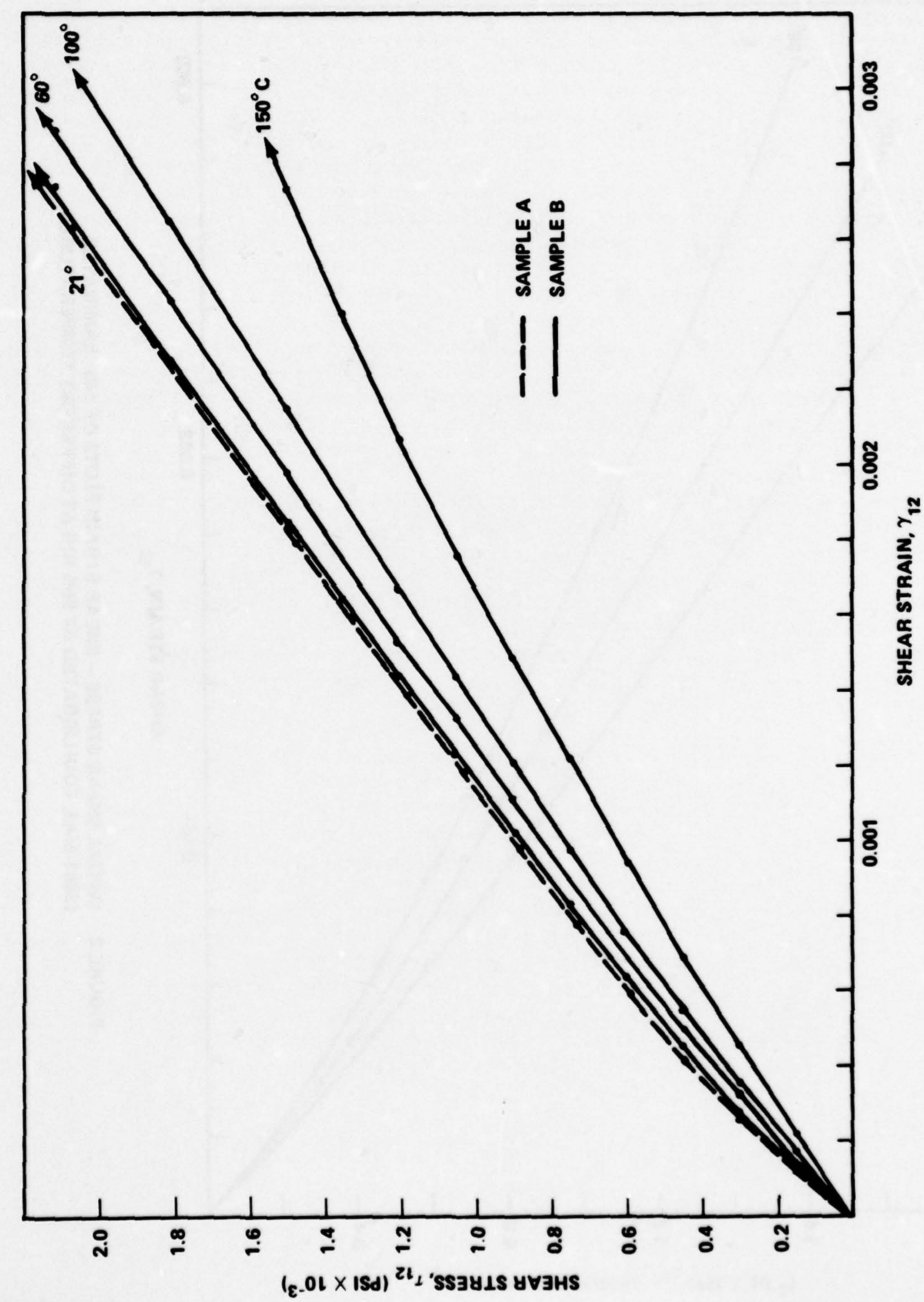

FIGURE 1 INITIAL SHEAR STRESS – SHEAR STRAIN CURVES OF ±45° TENSILE SPECIMENS (3501-6/AS, DRY LAMINATE) AT DIFFERENT TEMPERATURES

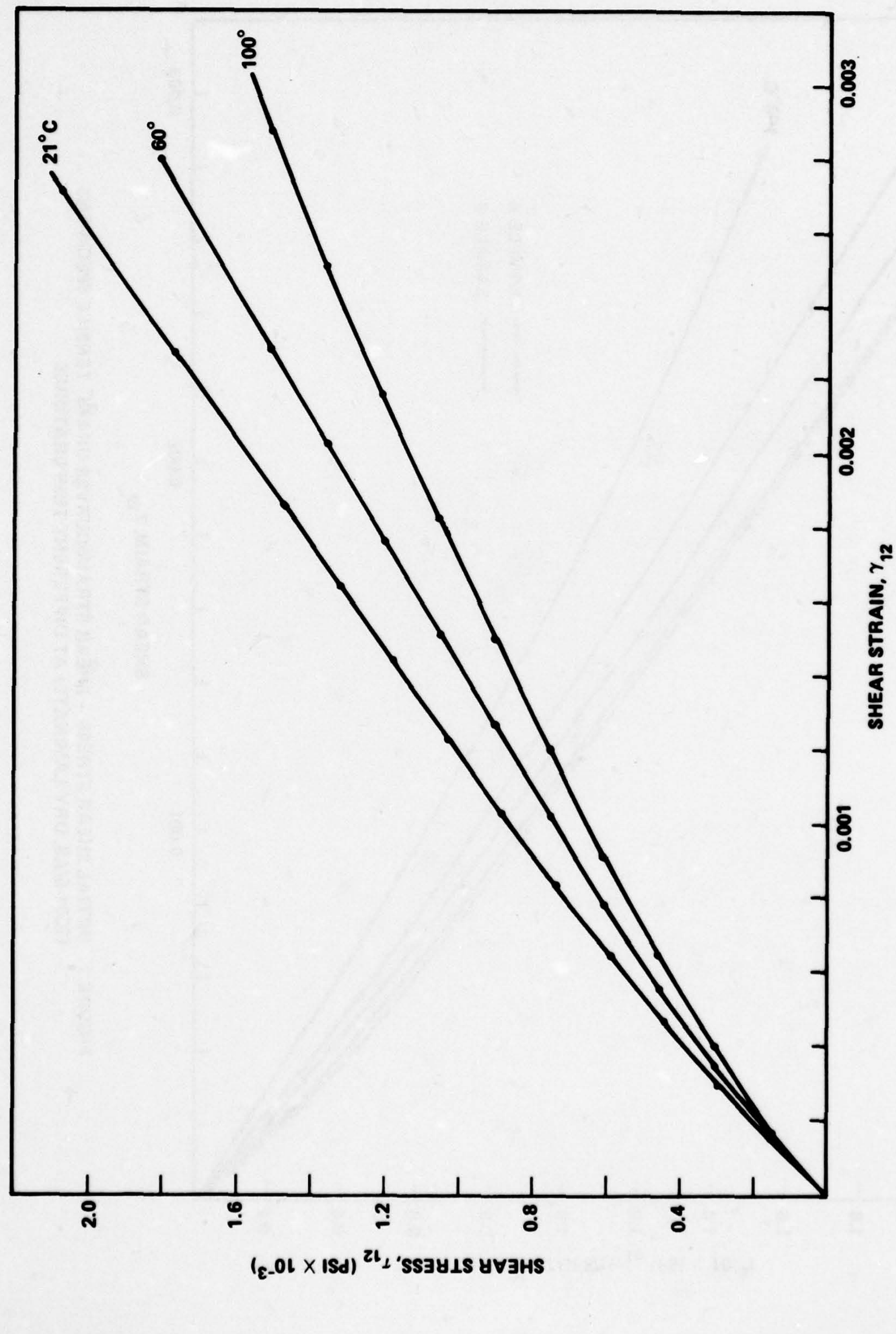

FIGURE 2 INITIAL SHEAR STRESS – SHEAR STRAIN PLOTS OF ±45° LAMINATES (3501-6/AS, EQUILIBRATED AT 55% RH) AT DIFFERENT TEMPERATURES

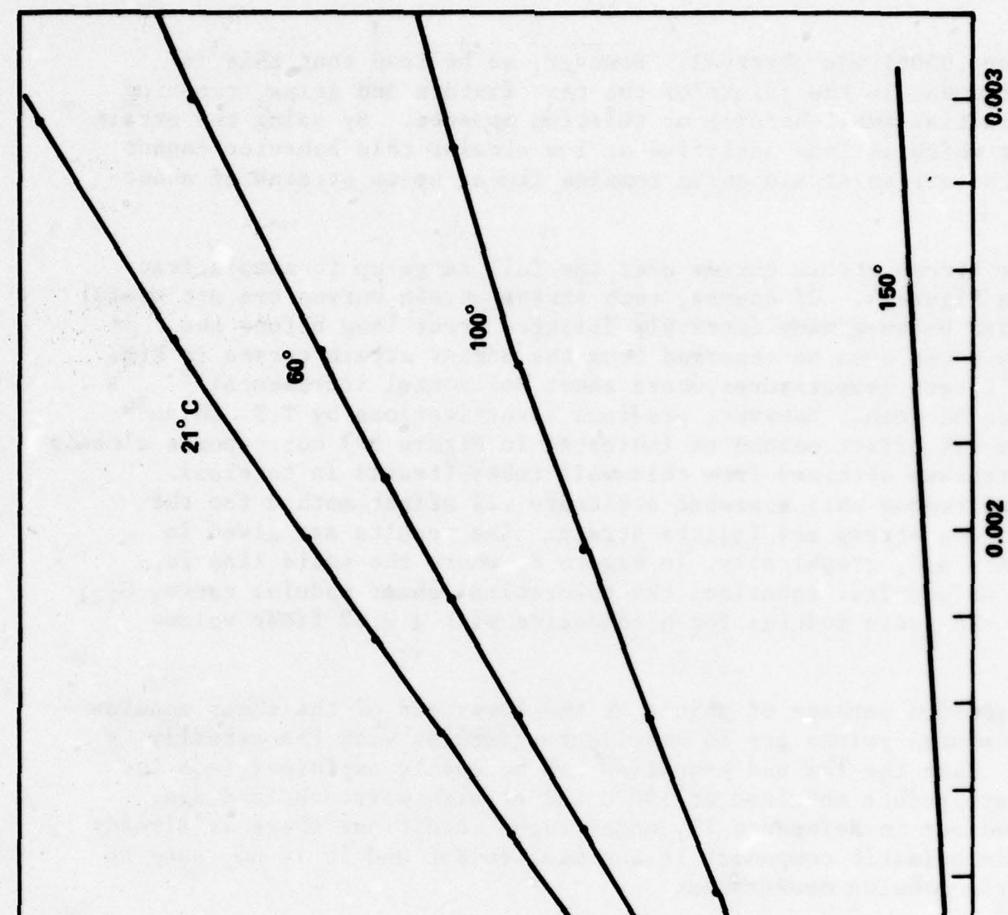

FIGURE 3 INITIAL SHEAR STRESS – SHEAR STRAIN PLOTS OF ±45° LAMINATES (3501-6/AS, EQUILIBRATED AT 80% RH) AT DIFFERENT TEMPERATURES

strains (less than .0006) was observed. However, we believe that this is caused by the alignment in the joints of the test fixture and grips, removing or causing some initial small bending or twisting moments. By using the strain gage extensometer which is less sensitive at low strains this behavior cannot be observed and the stress strain curve remains linear up to strains of about .004.

Extensometer stress strain curves over the full range up to sample fracture are shown in Figure 4. Of course, such stress strain curves are not useful for design purposes because many intra-ply failures occur long before the sample fails. This can even be observed from the stress strain curves in Fig. 4 at 150° and 60°C test temperatures where short horizontal incremental fracture lines can be seen. However, previous investigations by T.T. Chiao[34] have shown that a .2% offset method as indicated in Figure 5-7 corresponds closely to the failure stresses obtained from thin wall tubes (tested in torsion). Therefore, we also choose this somewhat arbitrary .2% offset method for the definition of failure stress and failure strain. The results are given in Table 1 and Table 2 and, graphically, in Figure 8, where the solid line is, according to the Halpin-Tsai equation, the theoretical shear modulus curve, G_{12}, as a function of the resin modulus for a composite with a 0.62 fiber volume fraction.

With the exception perhaps of points at the lower end of the shear modulus curve, the experimental points are in excellent agreement with the actually measured values. Even the low end exception can be easily explained from the fact that these are points obtained at 150°C and at high moisture loadings. As we have pointed out in Reference 29, under these conditions there is already a considerable viscoelastic component in the measurement and it is not easy to obtain a good resin modulus measurement.

It should be noted that it was not necessary to postulate any additional moisture effects such as resin-fiber debonding or resin degradation to account for the property changes. The only parameters that were employed were neat resin properties. With this resin it was not even necessary to use the above mentioned normalization of resin to composite since the value calculated for G_{12} of the dry composite was actually found.

This good agreement of predicted and observed values increases our confidence in the proposed prediction scheme of Reference 2, thus we feel it is well justified to pursue an extension of this approach to predict also composite failure.

It is recognized, of course, that this is much more difficult since there are no simple Halpin-Tsai equations that relate pure matrix failure to composite failure. Though a finite element approach for calculating single ply failure has already been considered by Adams.[38]

38. Miller, A.K., and Adams, D.F., "Micromechanical Aspects of the Environmental Behavior of Composite Materials," University of Wyoming, Department of Mech. Eng., Report UWME-DR-7011111.

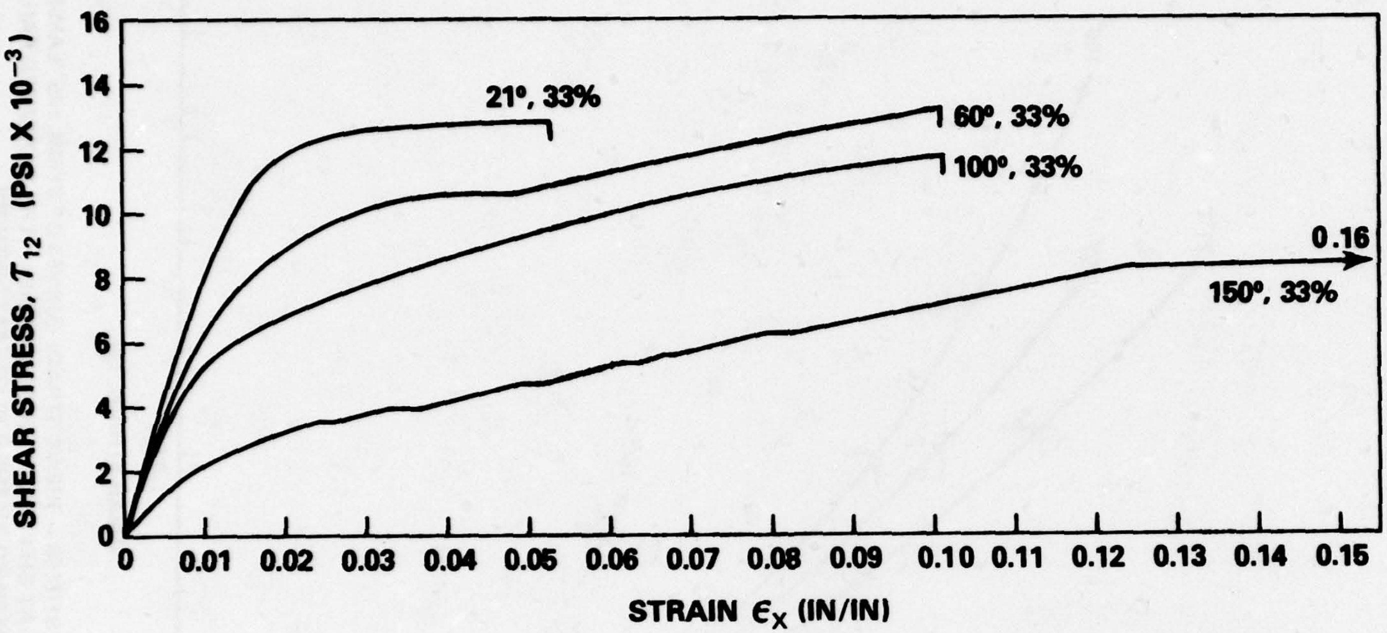

FIGURE 4 TYPICAL STRESS–STRAIN CURVES OF $\pm 45^{\circ}$ CARBON FIBER COMPOSITE SPECIMENS (3501-6/AS) AS A FUNCTION OF TEMPERATURE

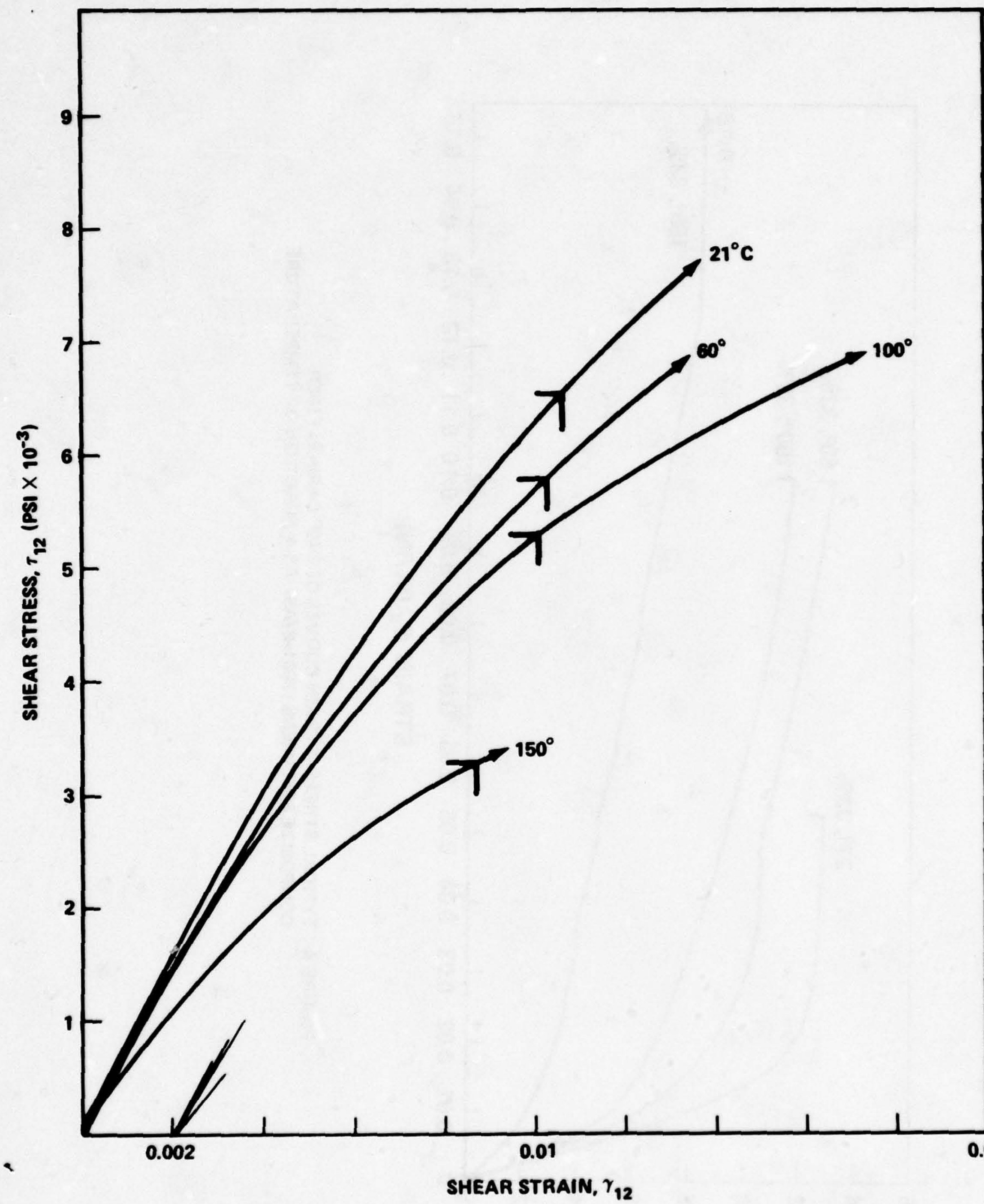

FIGURE 5 TYPICAL SHEAR STRESS – SHEAR STRAIN CURVES OF SYMM. ±45° LAMINATES (3501-6/AS, DRY) AT DIFFERENT TEMPERATURES. (THE INDICATED FAILURE STRESSES ARE DEFINED BY THE 0.2% OFF-SET METHOD)

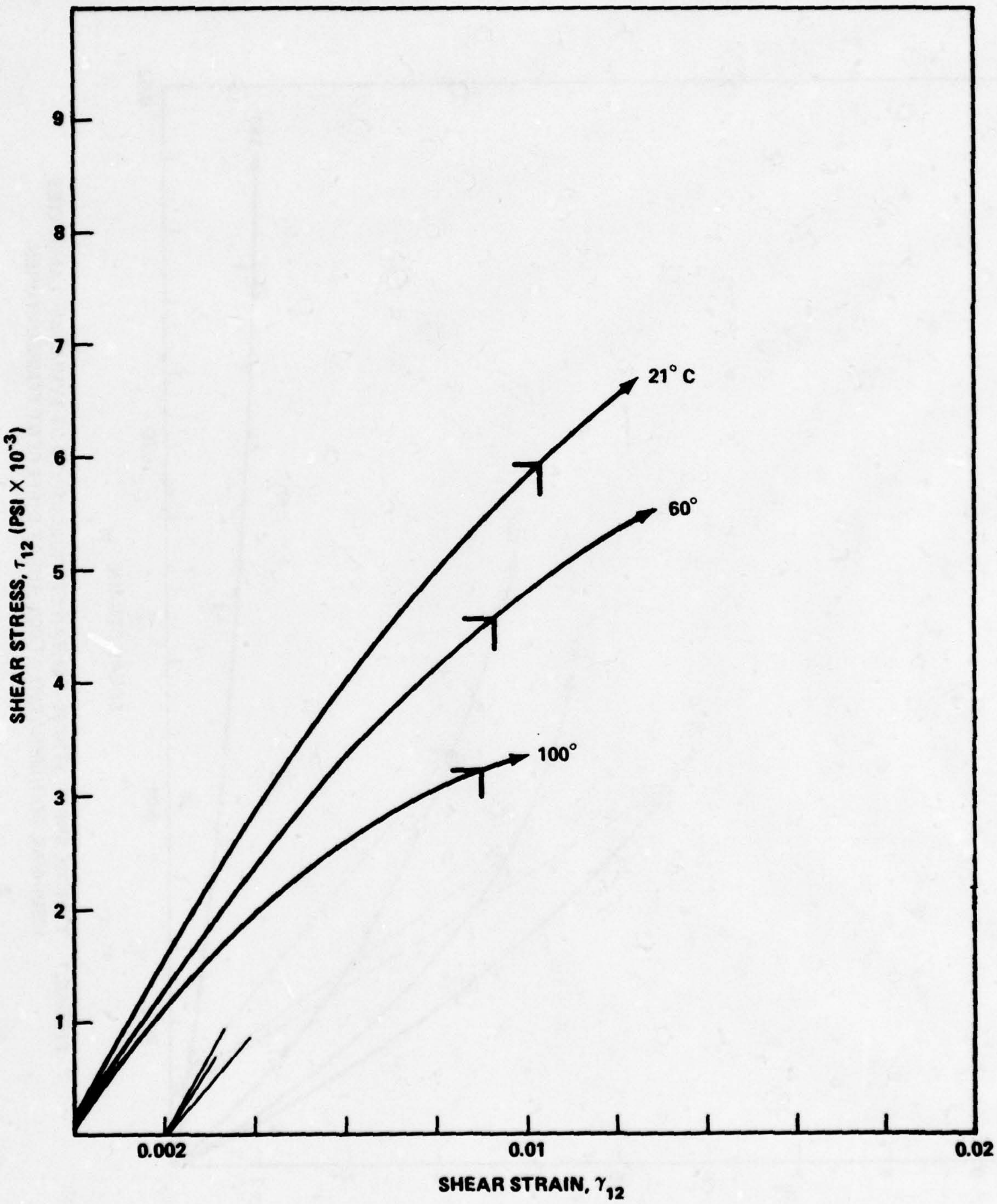

FIGURE 6 TYPICAL SHEAR STRESS –SHEAR STRAIN CURVES OF SYMM. ±45° LAMINATES (3501-6/AS, EQUILIBRATED AT 55% RH) AT DIFFERENT TEMPERATURES

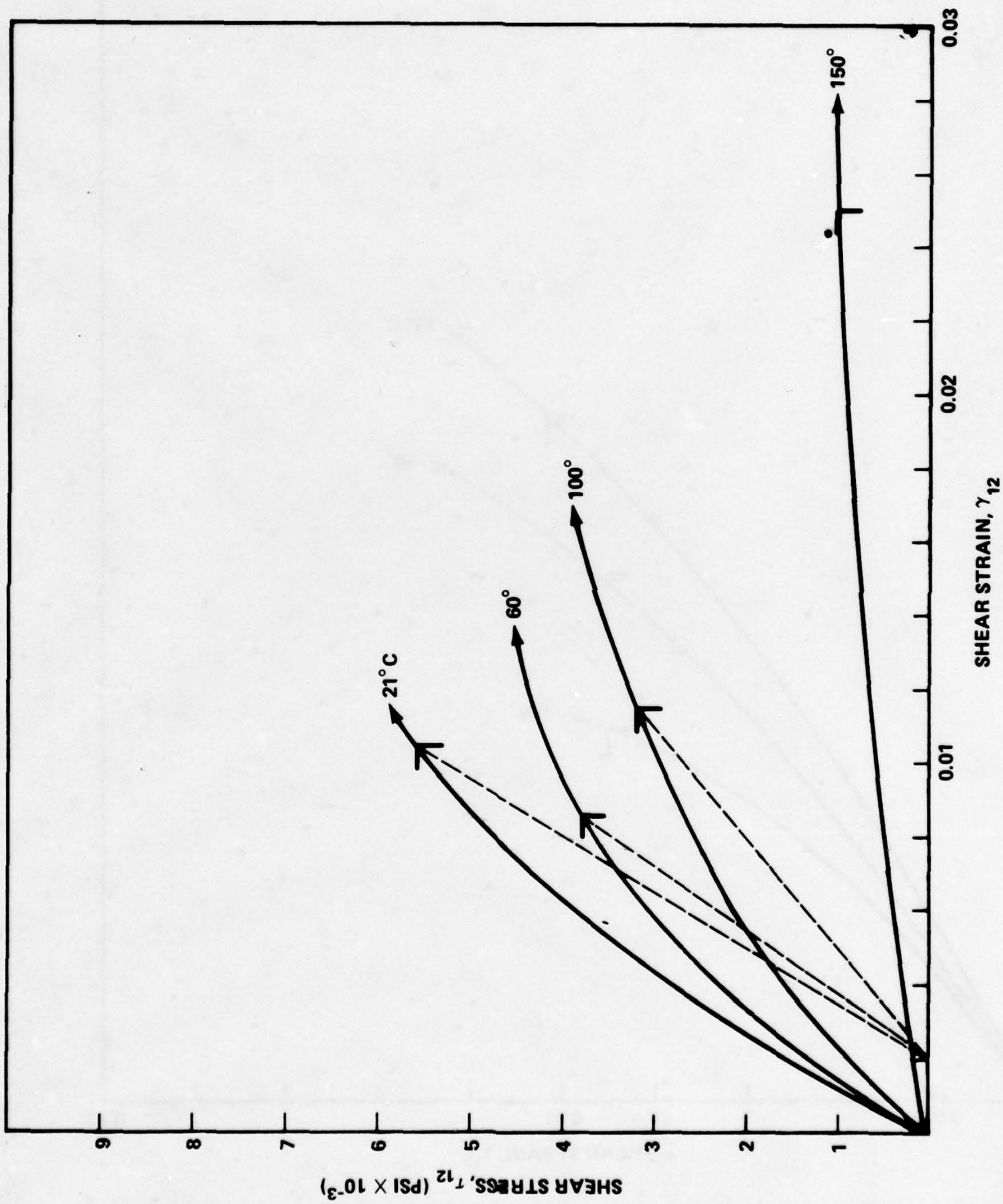

FIGURE 7 TYPICAL SHEAR STRESS – SHEAR STRAIN CURVES OF SYMM. ±45° LAMINATES (3501-6/AS, EQUILIBRATED AT 80% RH) AT DIFFERENT TEMPERATURES

Table 1. Predicted and Experimentally Observed Shear Moduli (G_{12}) in $\pm 45°$ Laminates (3501-6/AS) as Function of Temperature and Moisture Equilibration

Temp. °C	Equil. at RH	G_m (PSI x 10^{-6})	G_{12} (Predict) (PSI x 10^{-6})	G_{12} (Observed) (PSI x 10^{-6})
21	0	0.222	0.736	0.734
21	33	0.220	0.732	0.726
21	55	0.211	0.710	0.710
21	80	0.190	0.654	0.642
21	100	0.173	0.607	0.592
60	0	0.212	0.711	0.708
60	33	0.203	0.688	0.694
60	55	0.190	0.654	0.608
60	80	0.168	0.592	0.600
60	100	0.150	0.540	0.517
100	0	0.192	0.659	0.653
100	33	0.182	0.630	0.621
100	55	0.168	0.592	0.513
100	80	0.122	0.453	0.462
100	100	0.083	0.321	0.291
150	0	0.150	0.540	0.563
150	33	0.101	0.384	0.405
150	55	0.066	0.260	0.133
150	80	0.027	0.112	0.047
150	100	0.0065	0.028	0.023

Table 2. Longitudinal Shear Properties of Hercules 3501-6/AS $\pm 45°$ Epoxy Composites

Test Temp. °C	Rel. Humidity %	Failure Shear[a] Stress τ_{12} (PSI)	Failure Shear[a] Strain γ_{12} (PSI)	Fracture Shear Stress τ_{12u} (PSI)	Initial Poisson's Ratio ν_{xy}
21	0	6560	.0106	11800	.725
21	33	8000	.0175	13760	.726
21	55	5920	.0103	14350	.732
21	80	5600	.0105	14210	.720
21	100	5920	.0118	13680	-
60	0	5800	.0103	12200	.735
60	33	6100	.0170	13200	.750
60	55	4500	.0093	13278	.803
60	80	3840	.0086	12360	.800
60	100	4240	.0114	11369	-
100	0	5300	.0100	12024	.755
100	33	4480	.0137	13720	-
100	55	3240	.0090	11517	.803
100	80	3160	.0115	10590	.750
100	100	1800	.0980	8641	-
150	0	3800	.0087	9273	.750
150	33	2080	.0182	8455	-
150	55	-	$>.04$	7185	-
150	80	~ 1000	$\sim .025$	7269	1.00
150	100	-	$>.04$	7137	-

a. Data are based on the 0.2 percent offset method

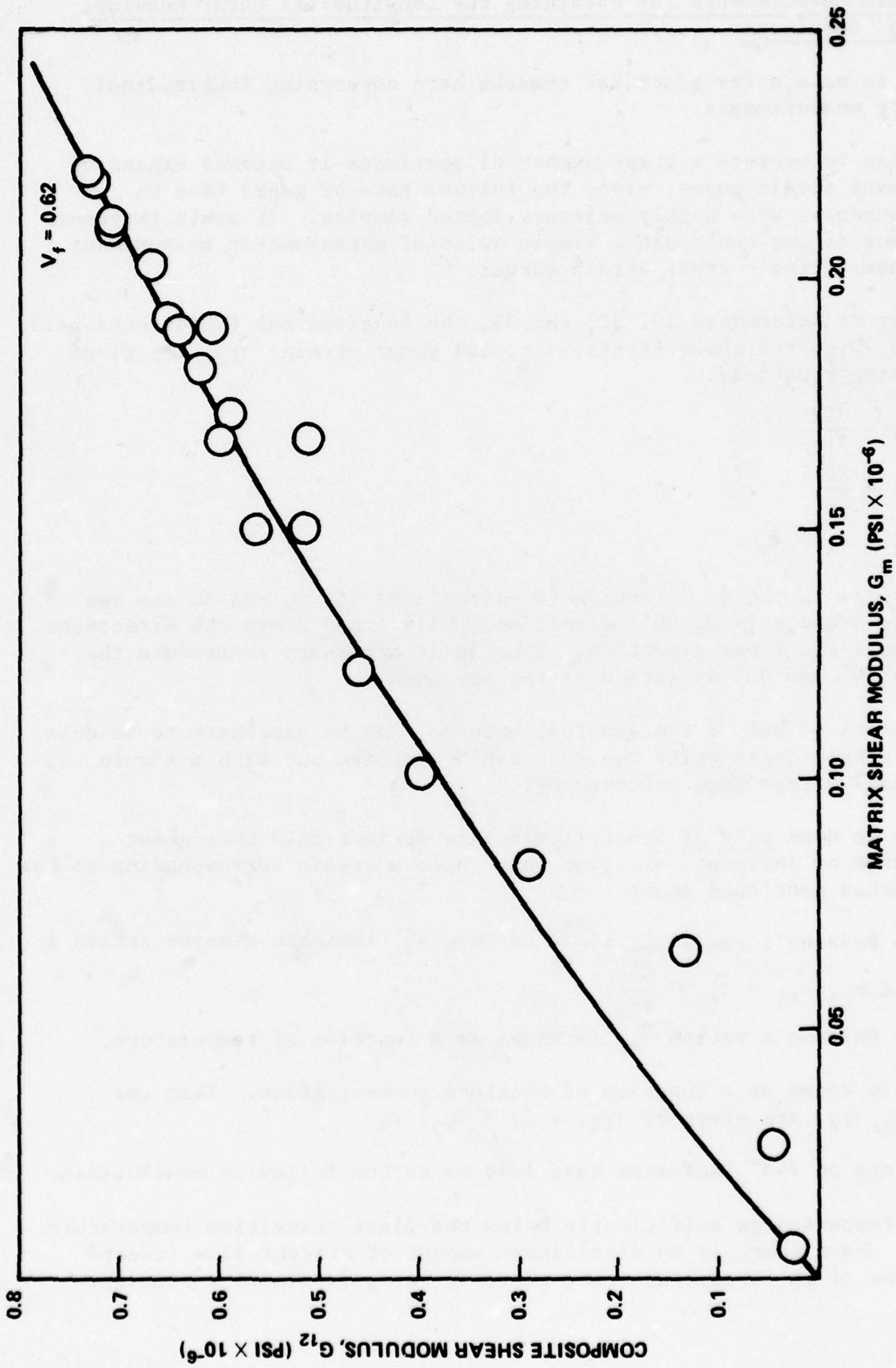

FIGURE 8 PREDICTED CHANGE IN THE COMPOSITE SHEAR MODULUS (SOLID LINE, FOR V_f = 0.62) THE DATA POINTS ARE THE OBSERVED VALUES FOR A 3501-6/AS COMPOSITE AT DIFFERENT TEMPERATURES AFTER EQUILIBRATION AT 0, 33, 55, 80 AND 100% RH

Uniaxial Strain Measurements for Obtaining the Longitudinal Shear Modulus, G_{12}, from ±45° Specimens

We want to make a few practical remarks here concerning longitudinal shear property measurements.

If one has to measure a great number of specimens it becomes expensive to mount so many strain gages, also, the failure rate of gages (due to debonding) increases with highly moisture loaded samples. It would therefore be advantageous if one could use a simple uniaxial extensometer measurement to obtain shear stress - shear strain curves.

According to References 30, 32, and 33, the longitudinal (unidirectional), shear modulus, G_{12}, the shear stress, τ_{12}, and shear strain, γ_{12}, are given by the following equations:

$$G_{12} = \frac{\tau_{12}}{\gamma_{12}}$$

$$\tau_{12} = \frac{\sigma_x}{2}$$

$$\gamma_{12} = \epsilon_x - \epsilon_y$$

where σ_x = stress in the x- direction (0°-direction) and ϵ_x and ϵ_y are the strains in the x and y (= 0, 90°) direction, while 1 and 2 are the directions along and across the fiber direction. Thus it is necessary to measure the strains in the 0° and 90° direction of the specimen.

Measurements of only a few samples, however, may be necessary to be done with biaxial strain gages while the rest can be carried out with a simple (unidirectional) strain gage extensometer.

This can be done <u>only if</u> the following assumptions hold throughout the strain range of interest, i.e., at least over a strain corresponding to the .2% offset method mentioned above:

1. The Poisson's ratio $\nu_{xy} = \frac{-\epsilon_y}{\epsilon_x}$ of the ±45° laminate remains strain independent, i.e., $\nu_{xy} = \overline{\nu}_{xy} \equiv -\frac{d\epsilon_y}{d\epsilon_x}$;

2. The Poisson's ration $\overline{\nu}_{xy}$ is known as a function of temperature;

3. It is known as a function of moisture concentration. Then the shear strains, γ_{12}, are given by $\gamma_{12}, = (1 + \nu_{xy})\ \epsilon_x$.

Our measurements on ±45° laminates have lead us to the following conclusions:

a. At temperatures sufficiently below the glass transition temperature of the resin, where there is no significant amount of viscous flow (creep) during the time of the measurement the curve ϵ_y vs ϵ_x is almost a straight line even into the region of nonlinear shear stress-shear strain as can be seen from the solid curve in Figure 9. While at higher temperatures, and increased moisture loadings (dashed line), the curve bends upward due to non-negligible creep deformation. The strain dependent Poisson's ratio ν_{xy} in this case should not be taken from the experimental incremental points $-\Delta\varepsilon_y/\Delta\varepsilon_x$ but rather from the smooth curve fit through these points. Even then it is questionable, whether the Poisson's ratio ν_{xy} is strain dependent. We rather believe this is a time dependent effect due to creep, i.e., dependent on the strain rate (see the note on viscoelastic behavior of the 3501-6 resin in Reference 29).

b. The measurement of the Poisson's ratio seems to be quite sensitive to the accuracy of mounting the strain gages and to the alignment of the test specimens with the pull axis. Thus the inaccuracy of the ν_{xy} value is probably more affected by misalignment than by temperature as can be seen in Figure 10 where the measured ν_{xy} on some samples seem lower at 100° and 150°C than at 21°C.

Using a single dry sample (Fig. 11) for measuring ν_{xy} up to 150°C shows that there is only a very small temperature effect (ν_{xy} slightly increasing with temperature) only at 150°C which approaches the resin glass transition temperature is the usual upward curvature observed. Figures 12 and 13 show the curves for samples exposed to 55% and 80% RH.

c. From the above it is apparent that from a small number of biaxially strain gaged samples, measured under several different conditions, one obtains ν_{xy} as a function of temperature and moisture concentration. The rest of the samples can be measured with a conventional uniaxial strain gage extensometer (saving time and labor).

d. ν_{xy} is sufficiently linear in the region for the 0.2 offset method. Thus it is possible to obtain shear stress-shear strain curves with a uniaxial extensometer measurement.

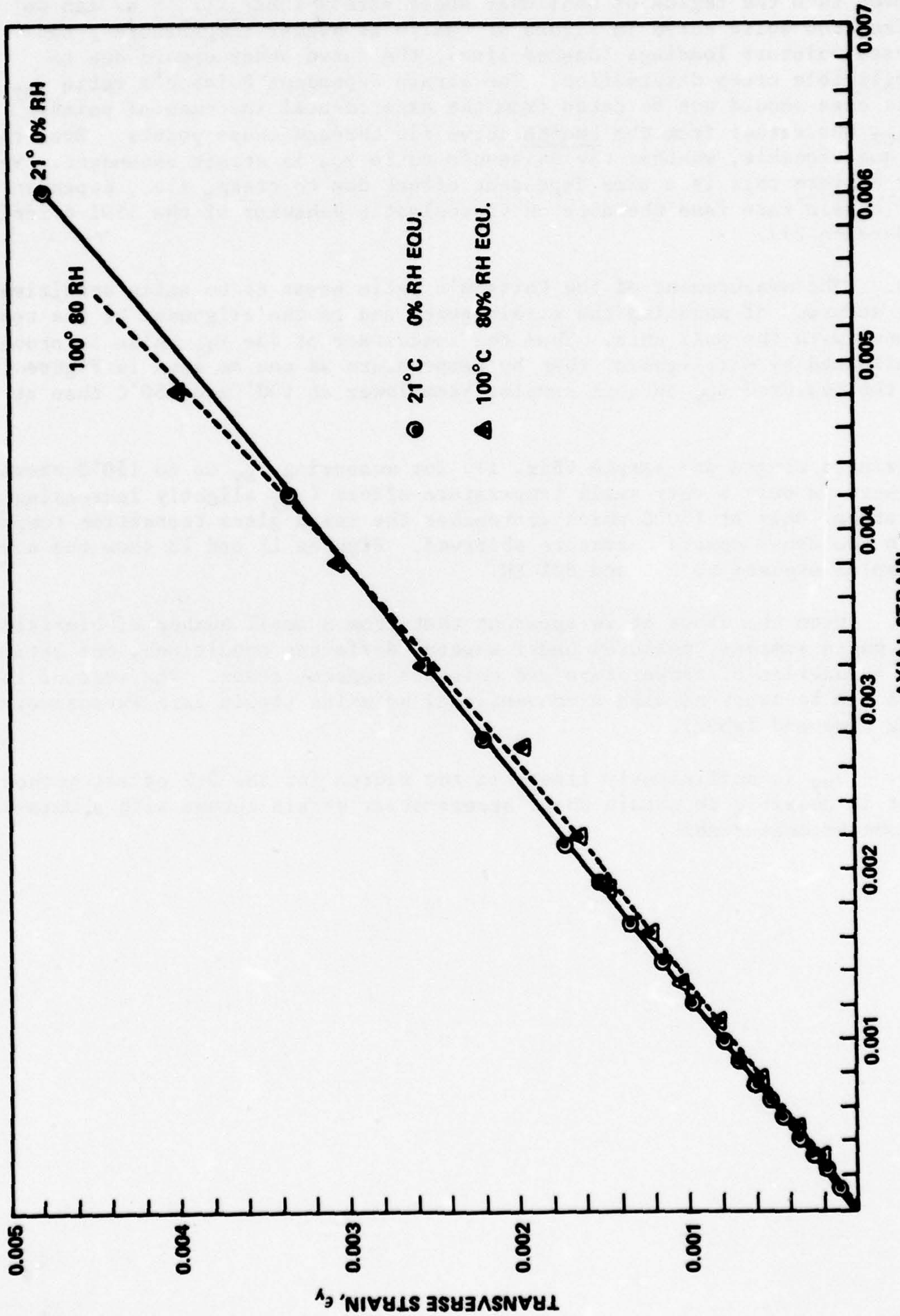

FIGURE 9 TYPICAL STRAINGAGE MEASUREMENT DATA OF LONGITUDINAL AND TRANSVERSE STRAINS WITH ±45° SYMM. LAMINATES OF 3501-6/AS CF EPOXY COMPOSITES

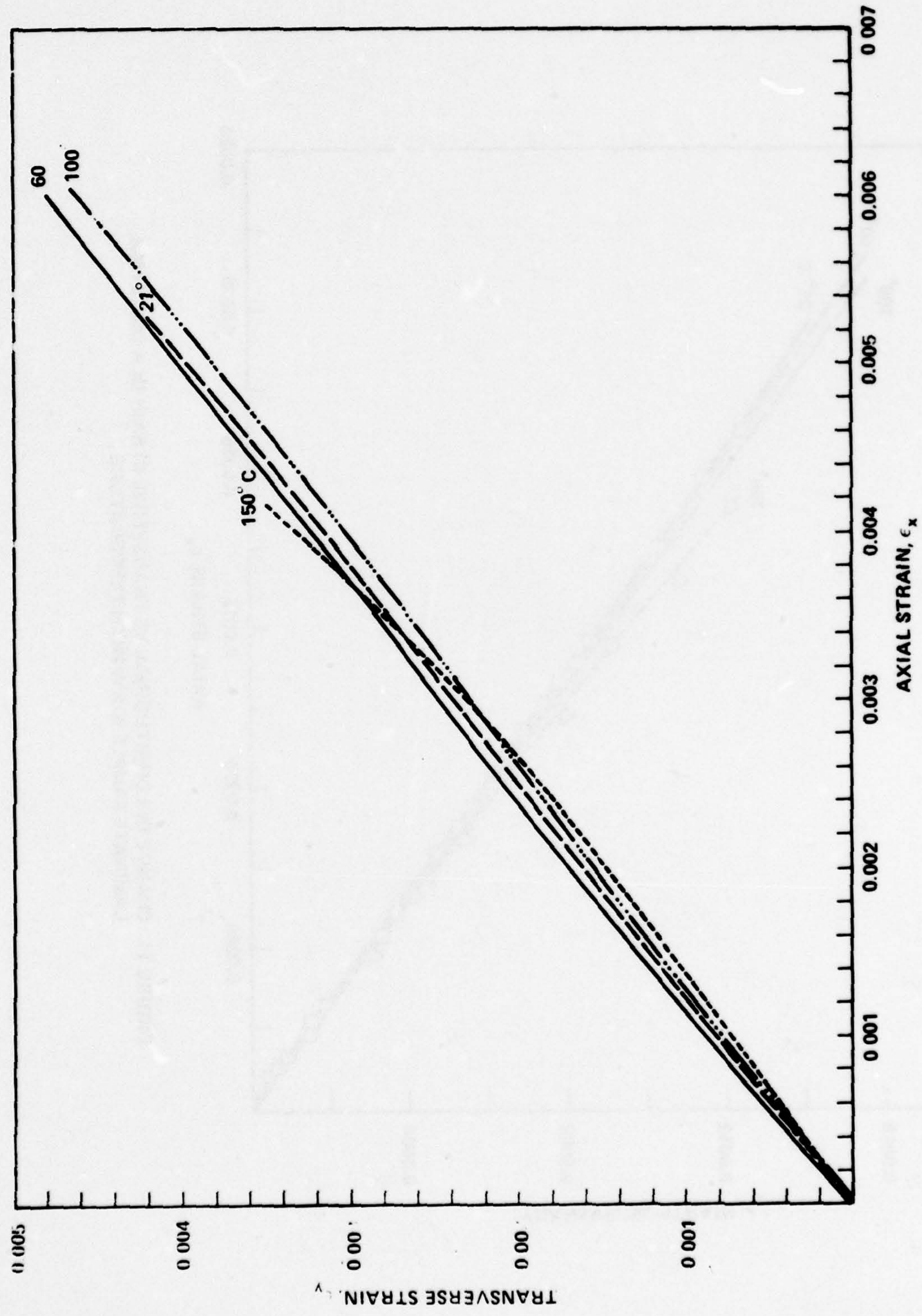

FIGURE 10 LONGITUDINAL VS TRANSVERSE STRAIN IN ±45° SYMM. 3501 6/AS LAMINATES (DRY) AT DIFFERENT TEMPERATURES

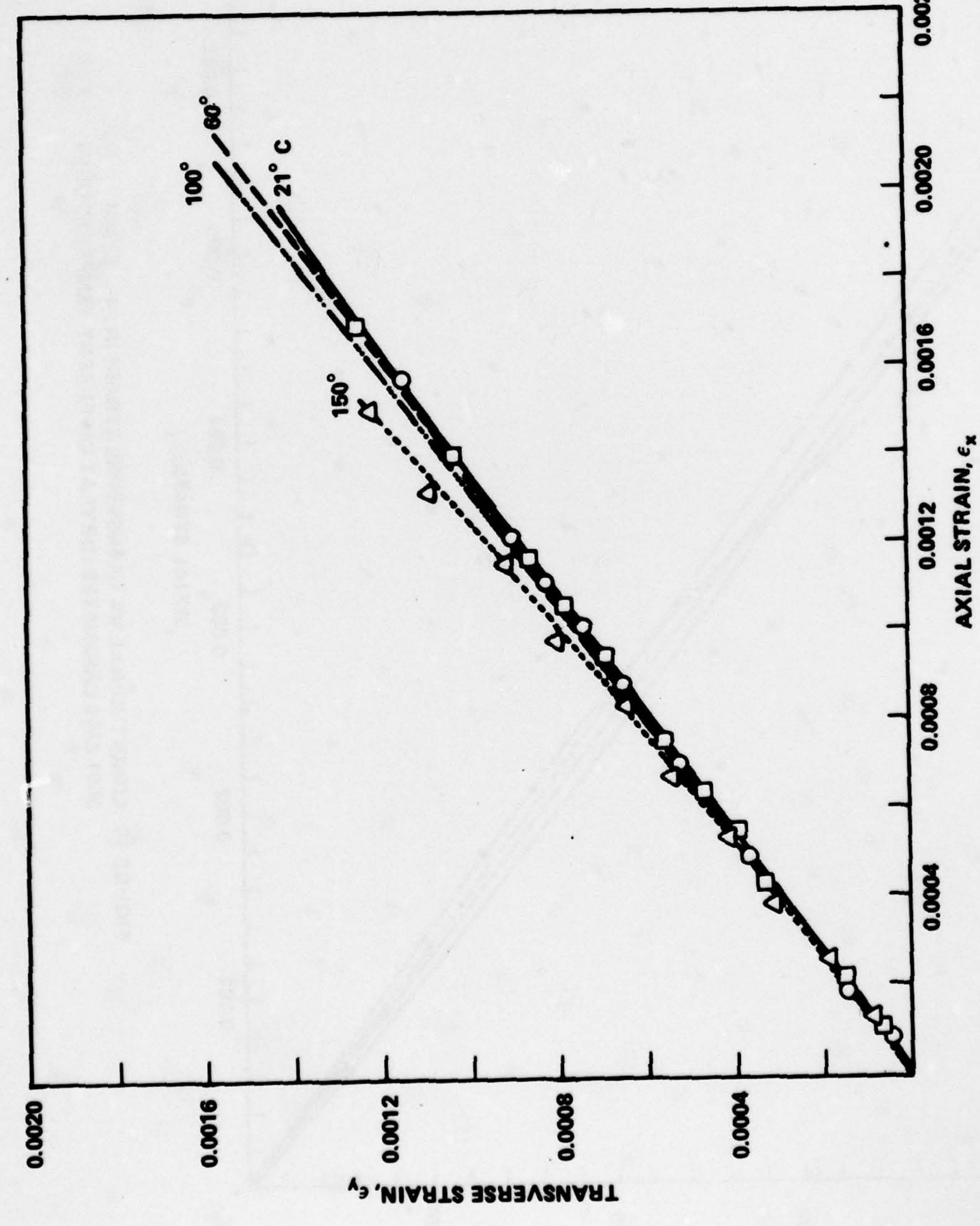

FIGURE 11 CHANGE IN LONGITUDINAL VS TRANSVERSE STRAIN IN A ±45° SYMM. LAMINATE SAMPLE (DRY) WITH TEMPERATURE

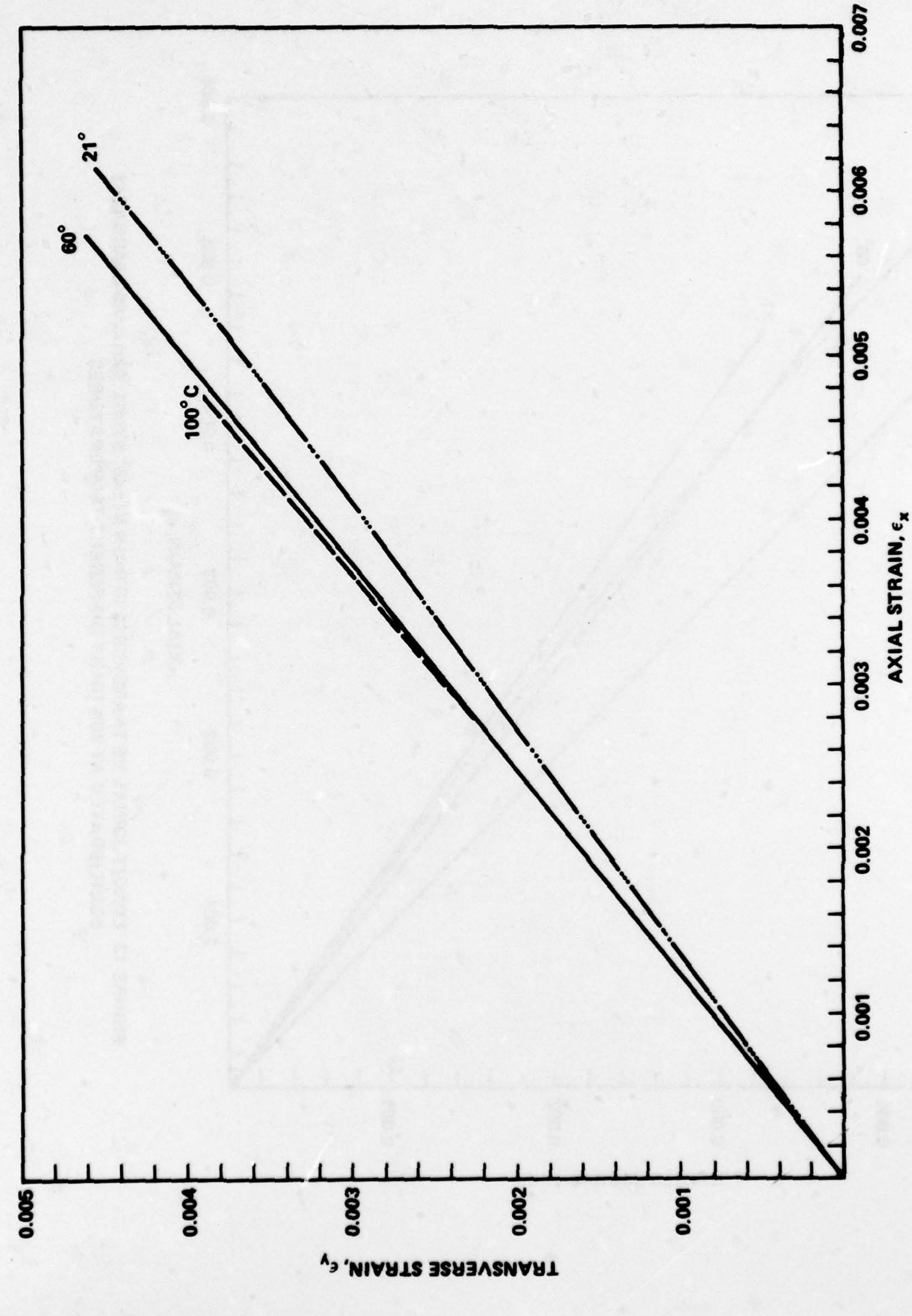

FIGURE 12 LONGITUDINAL VS TRANSVERSE STRAIN IN ±45° SYMM. 3501-6/AS LAMINATES EQUILIBRATED AT 55% RH

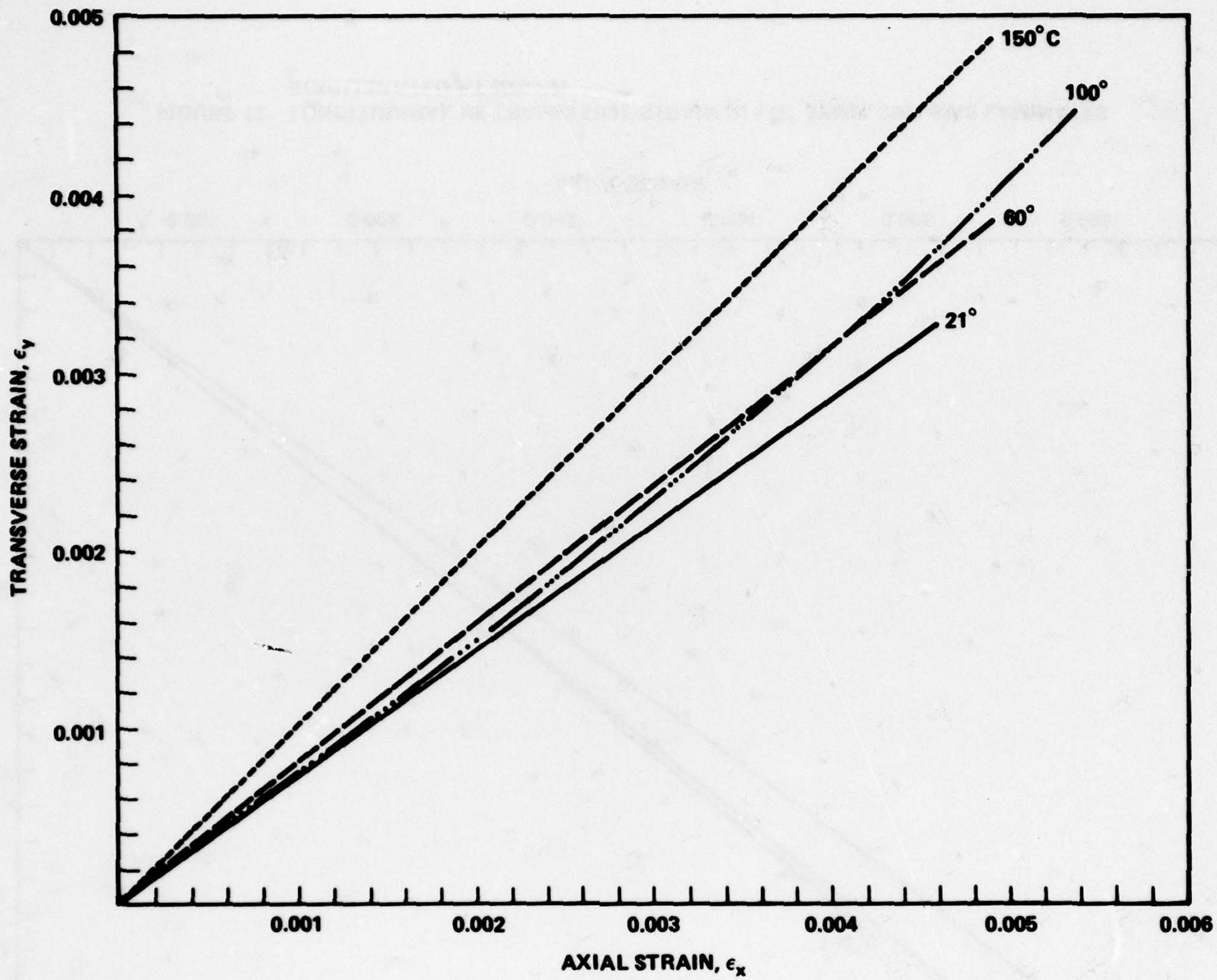

FIGURE 13 LONGITUDINAL VS TRANSVERSE STRAIN IN ±45° SYMM. 3501-6/AS LAMINATES EQUILIBRATED AT 80% RH AT DIFFERENT TEMPERATURES

CONCLUSIONS AND RECOMMENDATIONS

1. It has been shown that there is an excellent agreement between the reversible property changes (G_{12}), caused by moisture uptake, as predicted by micromechanical methods and those experimentally observed.

2. Micromechanics (which uses the constituent properties of matrix and fiber) is especially well suited for predictions of this sort, since there are no additional fabrication parameters or other causes of laminate imperfections to be taken into account, as one would, if different resins with different moduli would have been used to observe the same correlation between micromechanics and experiment.

3. This good agreement between prediction and experiment encourages further extension of predictive methods to nonlinear composite behavior for predicting changes in failure stresses and strains. The difficulties are greater because there are no Halpin-Tsai analog equations that relate resin failure to composite failure. Yet a systematic investigation of the nonlinear resin-composite response shows great promise. It is therefore recommended that further effort in this direction be carried out.

4. It is concluded that, by applying simultaneously the concepts of moisture diffusion, environmental modeling, micromechanics and a finite difference (or finite element) laminate theory (as outlined in References 1 and 2), one can predict at least the reversible property changes in a composite as a function of time and as a function of the environment.

ACKNOWLEDGEMENT

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Appendix A

HALPIN-TSAI EQUATIONS

For design purposes and rapid computational procedures Halpin and Tsai have proposed a set of equations which are simpler than most of the formulas derived from micromechanical analyses, however, are good enough for estimating with reasonable accuracy the ply properties:

$$E_{11} \simeq E_f V_f = E_m V_m$$

$$\nu_{12} \simeq \nu_f V_f + \nu_m V_m$$

$$\frac{\overline{P}}{P_m} = \frac{(1+\zeta\eta V_f)}{(1-\eta V_f)}$$

where

$$\eta = \left[(p_f/p_m)-1\right] / \left[(p_f/p_m)+\zeta\right]$$

$\overline{p}$ = composite moduli E_{22}, G_{12}, or G_{23};

p_f = corresponding fiber modulus E_{11f} G_{12f} ν_{12f} (or ν_{23f});

p_m = corresponding matrix modulus E_m, G_m, ν_m;

ζ = a measure of reinforcement which depends on the boundary conditions.

Approximate values are given by $\zeta_{E22} = 2(a/b)$ and by $\log \zeta G_{12} = 3 \log (a/b)$,

i.e., for round fibers $\zeta_{E12} = 2$ and $\zeta_{G12} = 1$

Appendix B

LAMINATE CURE

The laminate $(\pm 45°)_{2s}$ plates of 3501-6/AS carbon fiber composite was fabricated by the McDonnell Douglas Aircraft Corp., St. Louis. The cure schedule used was described as follows:

The laminate was pressurized in the autoclave to 85 ±5 psig while the interior bag pressure was held at 24-29 inches of mercury. The temperature was then raised to 240° ±10°F in 30 to 85 minutes, holding the autoclave pressure at 85 ±5 psig while the interior bag pressure was held at 24-29 inches of mercury.

The temperature was held at 240 ±10°F for 60-70 minutes and then the pressure was increased to 100 ±5 psig while the vacuum in the bag was released to atmospheric pressure. The temperature was now raised to 350 +10°F at a rate of 2 to 6°F per minute.

The bag pressure was not allowed to exceed 5 psig (it was vented to atmospheric pressure if this limit was reached).

The temperature was held at 350 ±10°F for 2 hours. Then the autoclave was allowed to cool to 200°F in not less than 30 minutes while maintaining at least 8 psig.

The parts were then transferred to an oven (not above 200°F) for post cure and reheated to 350 ±10°F in not less than 30 minutes and post cured for 8 ±1/2 hours.

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