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MOISTURE EFFECTS IN EPOXY MATRIX COMPOSITES

Mechanics and Surface Interactions Branch  
Nonmetallic Materials Division

11  
May 77

12 36p.

TECHNICAL REPORT AFML-TR-77-17

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Interim Report, 1 Dec 1975 - 1 Jul 1976.

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This technical report has been reviewed and is approved for publication.

  
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20. ABSTRACT (Cont'd)

→ temperature is discussed. Particular attention is also given to the details of environmental testing.

Data indicates that absorbed moisture and temperature effects on the neat resin translate directly to matrix dominated properties of the composite and can lead to a change in failure mode, while filament dominated properties show very little environmental sensitivity. Results also indicate that resin glass transition temperature is a function of processing as well as absorbed moisture.

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FOREWORD

In this report, the effect of absorbed moisture and temperature on the mechanical properties of a neat epoxy resin and derived graphite/epoxy composites is investigated.

The work reported here was performed in the Nonmetallic Materials Division, Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio. Charles E. Browning and George E. Husman of the Composite and Fibrous Materials Branch and James M. Whitney of the Mechanics and Surface Interactions Branch were principal investigators. The authors wish to thank George Archibald of the Air Force Materials Laboratory and C. Lovett, T. Hartness, W. Ragland, and C. Fowler of the University of Dayton Research Institute for the fabrication, preparation, and testing of experimental specimens. This report was released by the authors in January 1977, and covers the time period of November 1975 to May 1976.

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## SECTION I

### INTRODUCTION

It is well recognized that current epoxy resins utilized in high performance structural composites absorb moisture from high humidity environments, for example, see References 1 through 3. This moisture absorption can largely be attributed to the moisture affinity of specific functional groups of a highly polar nature in the cured resin. The absorption of moisture causes plasticization of the resin to occur with concurrent swelling and lowering of the resin's glass transition temperature ( $T_g$ ). This type of behavior is classical in polymer science and is described in Reference 4 under the subject of polymer-diluent solutions. Similar effects are observed in composites. Thus, absorbed moisture can reduce the temperature range over which matrix dominated composite properties remain stable. Data is available in the literature (References 5 through 8) which indicates that moisture absorption does reduce the temperature at which degradation of composite mechanical properties occur.

In the present paper the effect of absorbed moisture and temperature on the mechanical properties of Hercules' 3501-5 neat resin and derived AS/3501-5 composites is investigated. In particular, the relationship between  $T_g$  and absorbed moisture is studied along with the corresponding effect of  $T_g$  on the mechanical properties. In addition, emphasis is given to the details of environmental testing, as the methodology is neither straightforward nor standardized.

SECTION II  
EXPERIMENTAL PROCEDURES

The 3501-5 neat epoxy resin specimens used in these studies were 0.125-inches thick and were prepared by casting the material between pyrex glass plates using 0.125-inch spaces. A seal around the periphery of the plates was made using a Teflon spaghetti tubing. The glass plates were sprayed with a silicone release agent which was baked on the surface. Maximum cure temperature for the castings was 350°F for 30 minutes with no postcure. Tensile testing was performed on dog-boned tensile specimens which were examined with polarized lenses prior to humid aging or testing to ensure that no residual stresses or cracking were present. Tests were performed in a conventional Instron test machine using a cross-head speed of 0.5 inches-per-minute. Strain measurements were obtained using a one-inch gage length, elevated temperature extensometer.

All composite data reported in this report was generated on 8-ply specimens machined from large autoclave cured panels. Panels were fabricated using AS/3501-5, 12-inch wide prepreg (Hercules Inc.) and standard lay-up and vacuum bagging procedures. Maximum cure temperature for the composites was 350°F for one hour with a three-hour postcure at 375°F. Specimens were machined from the panels using a diamond cut-off wheel which produces smooth specimen edges with no sanding required.

All specimens were preconditioned in a vacuum oven at 200°F and full vacuum for 24 hours to provide initially dry specimens. Dry control specimens were kept dessicated until tested. Specimens for humid aging were weighed and placed in two humidity cabinets set at 75% and 95% relative humidities and 160°F for accelerating moisture pick-up. All humid aging specimens were taken to near equilibrium moisture conditions and maintained at those conditions until tested.

Longitudinal and quasi-isotropic tension specimens required end tabbing; the loads required for the transverse and  $\pm 45^\circ$  tensile tests were sufficiently low to preclude the need for tabs. All elevated

temperature tensile tests were performed using a clam-shell oven heating only the gage-section of the specimen and thus precluding the need for a high temperature tabbing adhesive. A room temperature curing, anaerobic adhesive (e.g. Eastman 910 or M-Bond 200) was used for all tabs which were applied just prior to testing. The majority of the tests were performed using an extensometer to measure strain, however, strain gages were necessary for Poisson's ratio and in-plane shear tests. The same adhesive used for tabbing was used for strain gaging room temperature specimens. For strain gaging elevated temperature specimens a high temperature anaerobic adhesive (Loctite 306/NF Primer) was used. This adhesive will set-up at room temperature and complete its cure during the heat up of the specimen for test. This strain gaging procedure is fully described in Reference 9.

A matrix of the tests performed and reported in this report is shown in Table 1. Standard straight side coupons (IITRI specimen) 9 inch x 1 inch were used for all tensile specimens except longitudinal tension where specimen width was 3/4-inch to reduce load levels. Tabs, when used, were 1 1/2 inches long with a 30° taper for uniform load introduction. Flexure specimens were 4 inch x 1/2 inch and were loaded in a four-point fixture with a span-to-depth ratio of 32 to 1. In-plane unidirectional shear properties were determined from  $\pm 45^\circ$  tensile coupons, as illustrated in Figure 1, using the procedure described in Reference 10. All tests were performed at a constant cross-head speed of 0.5 in/min.

An extremely important aspect of this test program was the elevated temperature tests of humid aged specimens. It is imperative that the times to perform such tests are minimized. Humid aged specimens dry out very rapidly in hot, dry environments. The elevated temperature tests performed in this program were heated to test temperature at a rate of approximately 40-50°F/min, soaked at test temperature for three minutes, and then tested. The total time to heat and test all specimens was generally less than ten minutes. Even at these conditions the measured moisture loss was substantial, e.g., a specimen containing 1.73% moisture before test had only 1.31% after at 300°F. It is not only important to

minimize the test time, but it is also important to determine moisture content during the actual test so that data from various sources can be properly pooled and compared.

Several procedures were evaluated for determining the glass transition temperature of a composite; these included thermal mechanical analysis (TMA), relaxation modulus, and heat distortion temperature (HDT). The procedure that gave the most consistent results was a HDT test. In this test a matrix dominated specimen ( $90^\circ$  or  $\pm 45^\circ$ )  $4\frac{1}{2}$  inch x  $\frac{1}{2}$  inch was placed on edge in an American Instruments Company HDT test apparatus, and deflection vs. temperature was plotted. Typical results, test geometry, and applied load are shown in Figure 2. The choice of actual  $T_g$  is somewhat arbitrary; however, the first increase in deflection agrees well with predicted values.

## SECTION III

EFFECT OF ABSORBED MOISTURE ON  
GLASS TRANSITION TEMPERATURE

The glass transition temperature,  $T_g$ , of a polymer is defined as the temperature above which the polymer is soft and below which it is hard. The hard polymer is a glasslike material, while the soft polymer varies from a rubbery material for very high molecular weights to an oil for very low molecular weights. Thus, for epoxy resins the  $T_g$  is the temperature at which the polymer goes from a glassy solid to a rubbery solid.

From a practical standpoint it is more appropriate to discuss a glass transition temperature region rather than a single glass transition temperature, as the change from a hard polymeric material to a soft material takes place over a temperature range. The concept of a  $T_g$  is for convenience and refers to the temperature at which there is a very rapid change in physical properties. As a result, there is no precise  $T_g$  or method of measurement, but only an indication.

It is an accepted theory (Reference 4) that at and below the glass transition temperature 1/40 of the total volume of the material is free volume. If this is true, then the  $T_g$  can be altered by changing its free volume at a given temperature. If a polymer were mixed with a miscible liquid that contains more free volume than the pure polymer, then the  $T_g$  will be lowered. In particular, if it is further assumed that the free volumes are additive, then the diluent-polymer solution will contain more free volume at any given temperature than would the polymer alone. As a result, the plasticized polymer must be cooled to a lower temperature in order to reduce its free volume to 1/40 of the total volume of the diluent-polymer combination. This is the process which occurs when moisture is absorbed into an epoxy resin.

Based on the above assumptions Bueche and Kelley (Reference 11) derived the following expression for the  $T_g$  of a plasticized system

$$T_g = \frac{\alpha_p V_p T_{gp} + \alpha_d (1-V_p) T_{gd}}{\alpha_p V_p + \alpha_d (1-V_p)} \quad (1)$$

where  $T_{gp}$  is the glass transition temperature of the polymer,  $T_{gd}$  is the glass transition temperature of the diluent,  $\alpha_p$  is the expansion coefficient of the polymer,  $\alpha_d$  is the expansion coefficient of the diluent (Reference 3), and  $V_p$  is the volume fraction of the polymer. It should be noted that  $\alpha$  in this case is not the usual coefficient of thermal expansion, but is  $\alpha_l - \alpha_g$  where  $\alpha_l$  is the linear thermal expansion coefficient above the  $T_g$  and  $\alpha_g$  corresponds to the same below the  $T_g$ . In terms of the percent weight gain in the polymer,  $M$ ,

$$V_p = \frac{1}{1 + \rho_p/\rho_d [(0.01)M]} \quad (2)$$

where  $\rho_p$  and  $\rho_d$  are the densities of the polymer and diluent, respectively.

Equation 1 for the 3501-5 epoxy system is shown in Figure 3 as a function of absorbed moisture. These calculations are based on the following properties

$$\alpha_p = 3.78 \times 10^{-4}/^{\circ}\text{C},$$

$$\alpha_d = 4 \times 10^{-3}/^{\circ}\text{C}, \quad T_{gd} = 4^{\circ}\text{C},$$

$$\rho_p = 1.28 \text{ g/cm}^3, \quad \rho_d = 1 \text{ g/cm}^3$$

It should be noted that the dry  $T_g$  is higher for the composite than that for the resin. This is due to the fact that the resin was not post-cured, while the composite was post-cured at 375°F. Thus, any processing which improves the initial  $T_g$  will also improve the wet  $T_g$ . Although the data is limited, results in Figure 3 indicate that Equation 1 yields the proper trend and is a reasonable method for estimating the plasticized  $T_g$ . The maximum weight gain in Figure 3 is six percent, as it is estimated

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that this is the maximum which can be obtained by full plasticization of the epoxy resin.

SECTION IV  
PREDICTION OF MOISTURE DIFFUSION

It has been previously shown (Reference 2) that moisture diffusion in laminated composites can be predicted by Fick's Second Law. For one-dimensional diffusion through-the-thickness of an infinite plate, the diffusion process is described by the relationship

$$\frac{\partial c}{\partial t} = D_z \frac{\partial^2 c}{\partial z^2} \quad (3)$$

where  $c$  is the moisture concentration,  $D_z$  is the diffusivity through-the-thickness,  $t$  denotes time, and  $z$  is the thickness coordinate. Consider the following boundary and initial conditions for a plate of thickness,  $h$

$$c(x,0) = c_i$$

$$c(0,t) = c(h,t) = c_a \quad (4)$$

where  $c_i$  is the initial moisture concentration in the material and  $c_a$  is the ambient moisture concentration. Classical separation of variables in conjunction with Equations 3 and 4 yields the following results (Reference 12)

$$\frac{c - c_i}{c_a - c_i} = 1 - \frac{4}{\pi} \sum_{i=0}^{\infty} \frac{1}{(2i+1)} \sin \frac{(2i+1)\pi z}{h} \exp \left[ \frac{-(2i+1)^2 \pi^2 D_z t}{h^2} \right] \quad (5)$$

The total weight of the moisture in the material is given by

$$m = \int_0^h c \, dz \quad (6)$$



Integration of Equation 5 yields

$$G = \frac{m - m_i}{m_a - m_i} = 1 - \frac{8}{\pi^2} \sum_{i=1}^{\infty} \frac{\exp \left[ -(2i + 1)^2 \pi^2 \left( \frac{D_z t}{h^2} \right) \right]}{(2i + 1)^2} \quad (7)$$

It should be noted that Equations 5 and 7 are based on starting with a uniform moisture distribution through-the-thickness. In terms of percent weight gain in the material,  $M$ , Equation 7 can be written in the form (Reference 2)

$$M = G(M_e - M_i) + M_i \quad (8)$$

where  $M_e$  denotes percent weight gain when equilibrium with respect to the ambient humidity is reached and  $M_i$  is the initial equilibrium weight gain in percent. Convergence of Equations 5 and 7 will depend on the value of the parameter  $t^*$  where

$$t^* = \frac{D_z t}{h^2} \quad (9)$$

for values of  $t^* \geq 0.1$ , accuracy to seven significant figures can be obtained with five terms. For most problems of interest four figures can be obtained with a maximum of ten terms.

Absorption data on AS/3501-5 composites compared to predictions based on Equations 7 and 8 are shown in Figure 4. Each data point is an average of 18 to 60 specimens. Dimensions of specimens were 1" x 9" x 0.044" for tensile coupons and 1/2" x 4" x 0.044" for flex specimens. No edge corrections were used. Good agreement is obtained between the moisture absorption data and the theory. Diffusivity was determined from the initial portion of the moisture absorption curve, which is linear, in the same manner as discussed in Reference 2.

As previously discussed, as the specimens dry out rapidly, the test time for wet specimens at elevated temperatures is critical. Moisture profiles are shown in Figure 5 for specimens tested at 300°F with an initial uniform moisture profile of 1.05% weight gain. These profiles are based on Equation 5, and the diffusivity was determined from experimentally measured drying rates. Obviously less drying out will occur at lower temperatures. These profiles also illustrate the necessity of performing the wet temperature experiments in as brief a time period as feasible.

SECTION V  
DISCUSSION OF MECHANICAL PROPERTIES

Neat resin properties are shown in Table 2. While temperature has a measurable effect on the dry resin modulus, absorbed moisture causes temperature to have an even more severe effect; that is, moisture causes changes in mechanical properties to occur at lower temperatures. As previously discussed, this is primarily due to the shift in  $T_g$  which accompanies the plasticization process. However, it should be noted that temperature actually causes an increase in dry resin strength for the elevated temperatures investigated due to increased ductility. This can be readily observed in Figure 6 where increasing nonlinear behavior occurs for the dry resin at 250°F and at 300°F. It is anticipated that higher temperatures close to the dry  $T_g$  would produce a loss in resin strength. In the case of the wet specimens, nonlinearity is observed at 200°F and above with the 300°F stress-strain curve being almost elastic-plastic. This behavior again reflects a shift in the  $T_g$ . Data in Table 2 and the stress-strain curves in Figure 6 also reveal a room temperature effect on both neat resin strength and modulus which cannot be associated with a shift in  $T_g$ . Examination of wet resin specimens with polarized lenses revealed surface crazing and cracking due to the swelling which is associated with the plasticization process. It is believed that this crazing phenomenon is responsible for the reduction in room temperature wet properties.

Unidirectional composite properties are shown in Table 3 and quasi-isotropic laminate data in Table 4. As anticipated, the matrix dominated unidirectional properties are affected by moisture. This can be seen more clearly in Figures 7 and 8 where the transverse and in-plane shear stress-strain curves are shown as a function of moisture content and temperature. Comparison of the transverse and in-plane shear stress-strain curves in Figures 7 and 8 with the resin stress-strain curves in Figure 6 shows that these composite properties reflect the same trends due to environmental effects as the matrix. In particular, modulus

reduction and strength reduction due to moisture in the composite are very similar to that of the matrix. Also, it should be noted in Table 3 and in Figure 7 that the transverse composite properties also reflect the same room temperature wet strength and modulus effect as found in the matrix, with the strength loss being more severe for the composite. It is believed that residual stresses due to swelling and possible interface degradation due to moisture are possible mechanisms which induce this strength loss. Although the shear modulus and strength are not reduced at room temperature wet, a cursory examination of Figure 8 reveals a room temperature effect on the details of the stress-strain curve, with moisture inducing more nonlinearity. The same interface and swelling mechanisms postulated for transverse tension should be present in the case of shear. However, flaw sensitivity for brittle materials in shear, is much less than in tension. Although swelling and interface degradation occurs at all temperatures, it is believed that the effects are most prevalent at room temperature, or below, where the resin is most brittle.

The 0-degree unidirectional properties in Table 3 and the quasi-isotropic properties in Table 4 show very little effect of moisture and temperature on strength and modulus, as these properties are filament dominated even under temperature and moisture conditions. It should be noted, however, that the details of the tensile stress-strain curve in the case of the quasi-isotropic laminate are influenced by temperature and moisture. In particular, after 0-degree ply failure, the stress-strain response is governed almost entirely by the response of the  $\pm 45$  degree plies which are highly influenced by temperature and moisture. This is illustrated in Figure 9 where typical stress-strain response corresponding to different wet and dry conditions at room temperature and 250°F are shown. The linear portion of these curves corresponds to filament dominated response. Departure from linear response occurs at a strain level which corresponds approximately to 0-degree failure strain as shown in Table 3. The upper portion of the wet quasi-isotropic stress-strain curves in Figure 9 produces large strains similar to the shear curves in Figure 8.

Reduction in matrix properties due to temperature and moisture can lead to a change in failure mode from filament dominated to matrix dominated. Such is the case for 0-degree flex strength which is illustrated in Figure 10. Unidirectional flex properties are usually filament dominated; however, reduction in in-plane shear strength in conjunction with highly nonlinear stress-strain response in shear, as illustrated in Figure 8, leads to a flex strength which is highly dominated by interlaminar yielding. This conclusion can be supported by examining failed specimens and noting that the 300°F wet composites did not display fiber breakage as the mode of failure, but were permanently deformed near the load noses where the shear stress is maximum. For conditions under which brittle failure was induced, fiber breakage occurred between the loading pins where the interlaminar shear stress vanishes.

Experimental difficulties with strain gage data was encountered for the 95% R.H. specimens at elevated temperatures. In particular, unreasonably low strains were measured; for example, a longitudinal modulus of  $27 \times 10^6$  psi was measured at 300°F for a unidirectional wet specimen exposed at 95% R.H. to an equilibrium weight gain of 1.6%. Extensometer data for these same conditions yielded a modulus of approximately  $17 \times 10^6$  psi. It is not clear at this time whether these difficulties were associated with a breakdown of the anaerobic adhesive or with the extreme matrix softening under these conditions preventing proper strain transfer to the gage. Even the extensometer data was less consistent under these conditions. Due to the difficulties encountered with the strain gage method, some data is omitted from Table 3 for the 95% R.H. wet condition.

## SECTION VI

## CONCLUSIONS

At least two mechanisms influence the moisture degradation of epoxy matrix composites. A plasticization process occurs in which the  $T_g$  of the resin is lowered, producing a degradation of matrix dominated composite properties at temperatures lower than occurs in dry laminates. The shift in the  $T_g$  appears to be predictable by the Bueche-Kelley theory. There is, however, another mechanism or mechanisms, which appear to induce a room temperature change in matrix dominated composite properties. Such effects cannot be associated with a shift in resin  $T_g$ . Residual stresses due to matrix swelling and fiber-matrix interface degradation are possible sources of the room temperature effect. Further work is necessary in these areas to more firmly establish the mechanisms of wet room temperature property changes.

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TABLE 1  
TEST MATRIX, AS/3501-5 GRAPHITE/EPOXY

Orientation	Flexure	Tension	Inplane Shear	T <sub>g</sub>
0°	36	52	---	---
90°	---	52	---	6
±45°	---	---	52	---
0/±45/90	---	52	---	---

- 2 Moisture Conditions: Near Equilibrium at 75% R.H. & 95% R.H.
- 3 Replicates for Dry Controls
- 4 Test Temperatures: R.T., 200°F, 250°F, 300°F
- 5 Replicates for Wet Tests

TABLE 2  
3501-5 NEAT RESIN PROPERTIES

	DRY				WET (5.6% WT.GN., (95% R.H.))			
	RT	200°F	250°F	300°F	RT	200°F	250°F	300°F
$\sigma_m$ (KSI)	6.8	5.0	8.2	9.2	5.4	5.6	2.8	1.4
$\epsilon_m$ (%)	1.4	1.3	2.6	3.3	1.4	2.7	2.0	24.0*
$E_m$ (10 <sup>6</sup> PSI)	0.46	0.43	0.37	0.33	0.40	0.30	0.25	0.20

\*Two piece failure did not occur. Test terminated due to extreme yielding.



TABLE 3  
0° PLY PROPERTIES AS/3501-5

	DRY												WET (1.05% WT. GN., 75% R.H.)						WET (1.6% WT. GN., 95% R.H.)										
	RT			200°F			250°F			300°F			RT		200°F		250°F		300°F		RT		200°F		250°F		300°F		
$\sigma_L$ (KSI)	186	177	187	199	199	191	184	192	180	194	213	205	196																
$\epsilon_L$ (%)	0.97	0.91	0.89	0.99	0.99	1.09	0.96	1.03	0.82	1.13	---	1.10	---																
$E_L$ ( $10^6$ PSI)	17.8	18.6.	18.9	18.8	18.8	18.1	19.3	18.5	19.5	16.6	17.1	16.7	17.1																
$\nu_{LT}$	0.36	0.35	0.34	0.38	0.38	0.40	0.39	0.40	0.46	0.30	---	---	---																
$\sigma_T$ (KSI)	7.9	7.4	6.5	5.4	5.4	4.4	3.4	2.8	1.4	2.9	2.2	1.9	0.89																
$\epsilon_T$ (%)	0.58	0.67	0.64	0.64	0.64	0.37	0.46	0.63	0.62	0.23	0.32	0.41	0.67																
$E_T$ ( $10^6$ PSI)	1.4	1.1	1.0	1.0	1.0	1.2	0.83	0.46	0.28	1.29	0.82	0.51	0.24																
$\tau_{LT}$ (KSI)	13.4	13.0	12.7	10.5	10.5	15.3	11.3	9.2	8.1	15.5	10.5	9.5	7.6																
$G_{LT}$ ( $10^6$ PSI)	0.87	0.77	0.64	0.61	0.61	0.86	0.64	0.39	0.16	0.86	---	---	---																

L - Direction parallel to the fibers

T - Direction transverse to the fibers

TABLE 4  
TENSILE PROPERTIES AS/3501-5, (0, ±45, 90)<sub>S</sub>

	DRY											
	WET (1.05% WT.GN., 75% R.H.)				WET (1.6% WT. GN., 95% R.H.)							
	RT	200°F	250°F	300°F	RT	200°F	250°F	300°F	RT	200°F	250°F	300°F
$\sigma$ (KSI)	72.6	66.5	76.6	76.0	75.2	71.7	71.4	71.1	74.8	73.7	68.9	65.3
$\epsilon$ (%)	1.1	1.1	1.2	1.4	1.8	1.6	2.6	2.8	1.4	2.3	2.4	1.9
E (10 <sup>6</sup> PSI)	6.4	7.1	6.9	6.1	6.7	5.7	5.3	5.3	7.2	7.3	6.1	6.5

## INPLANE SHEAR TEST

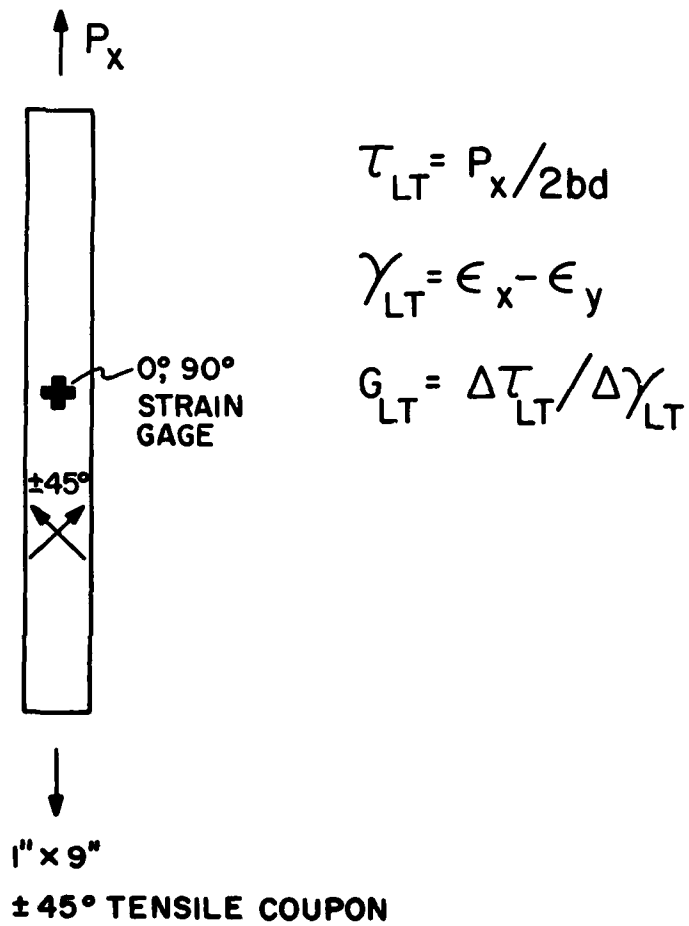


Figure 1. In-Plane Shear Specimen

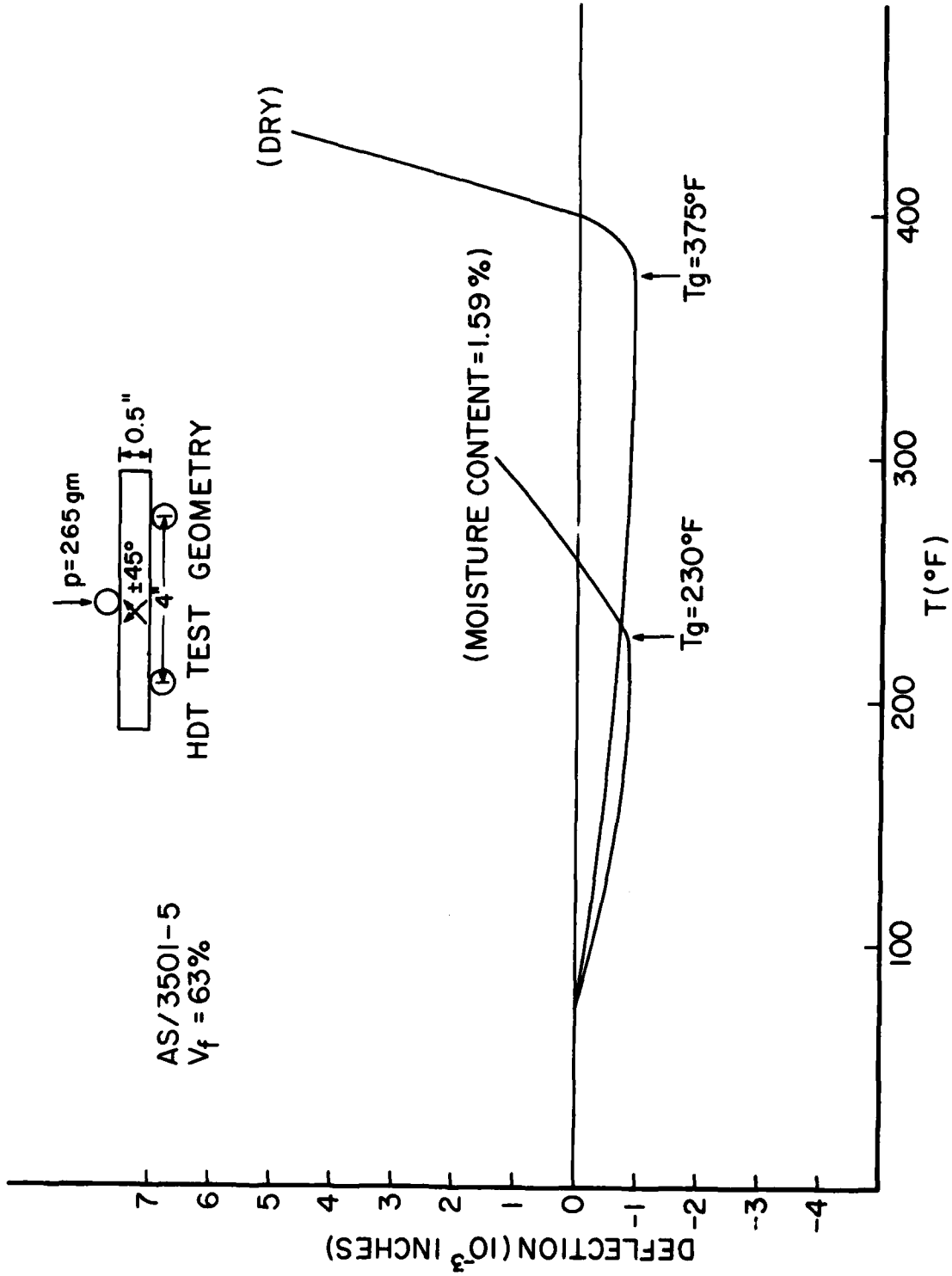


Figure 2. Typical HDT Test Results for AS/3501-5 Composites

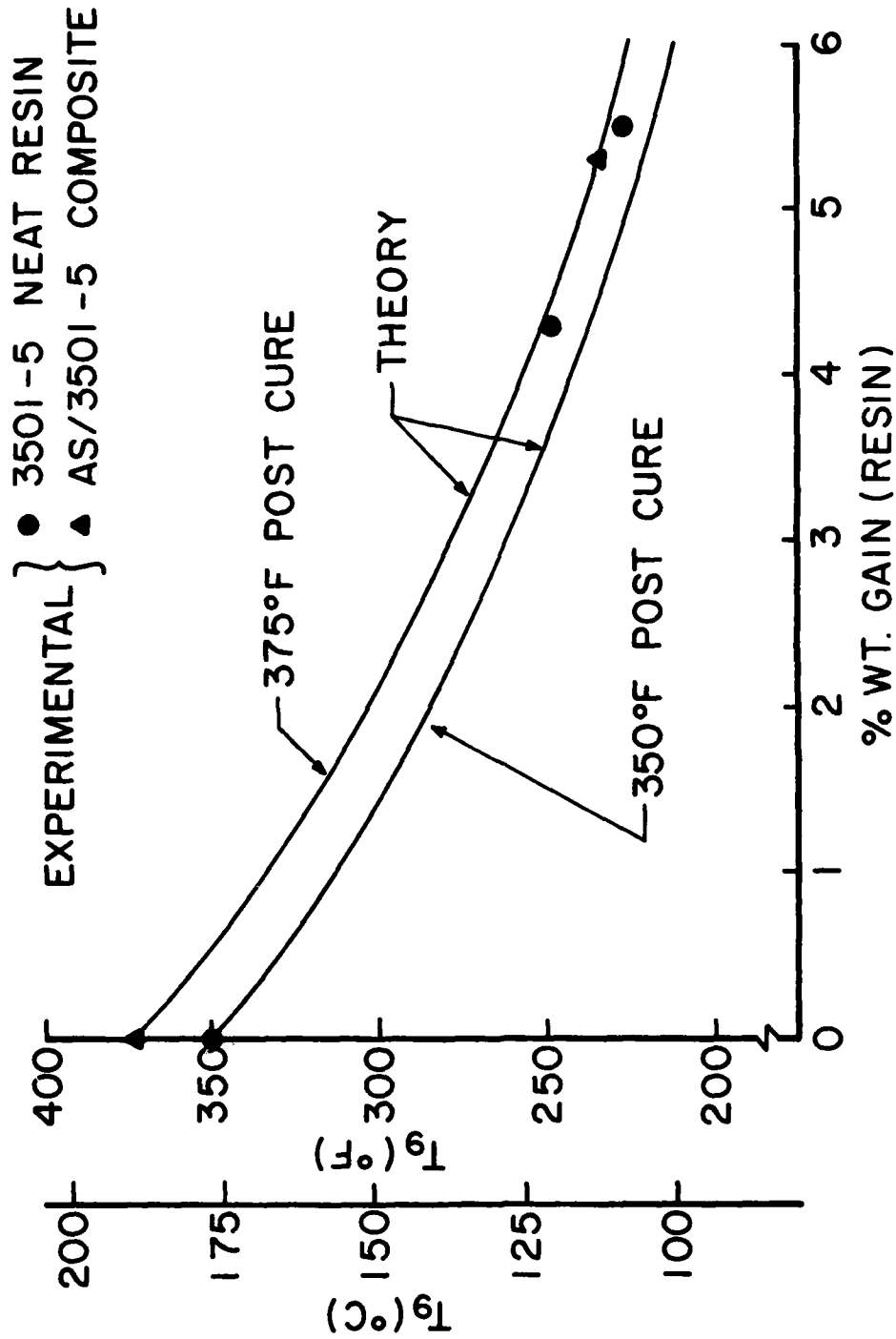


Figure 3. Glass Transition Temperature as a Function of Absorbed Moisture in Resin

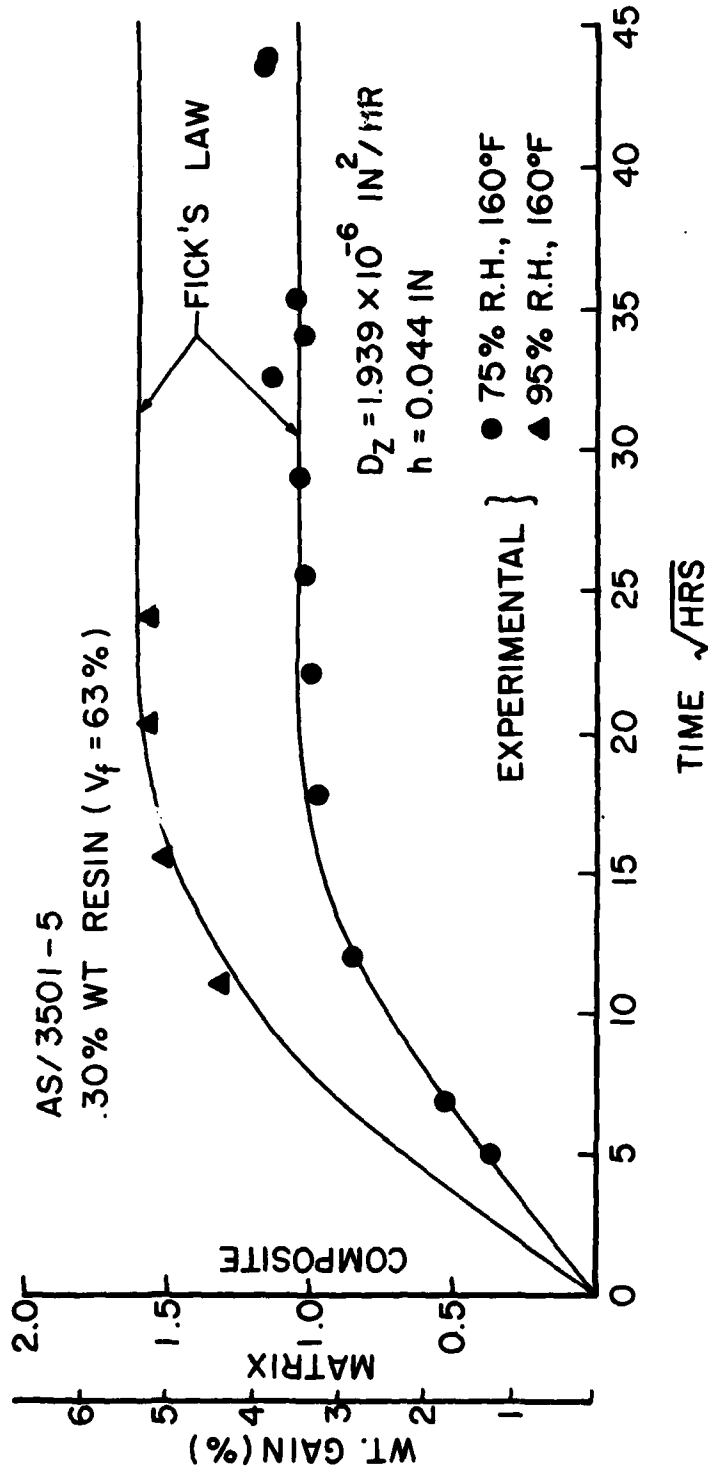


Figure 4. Moisture Gain as a Function of Time for AS/3501-5 Composites

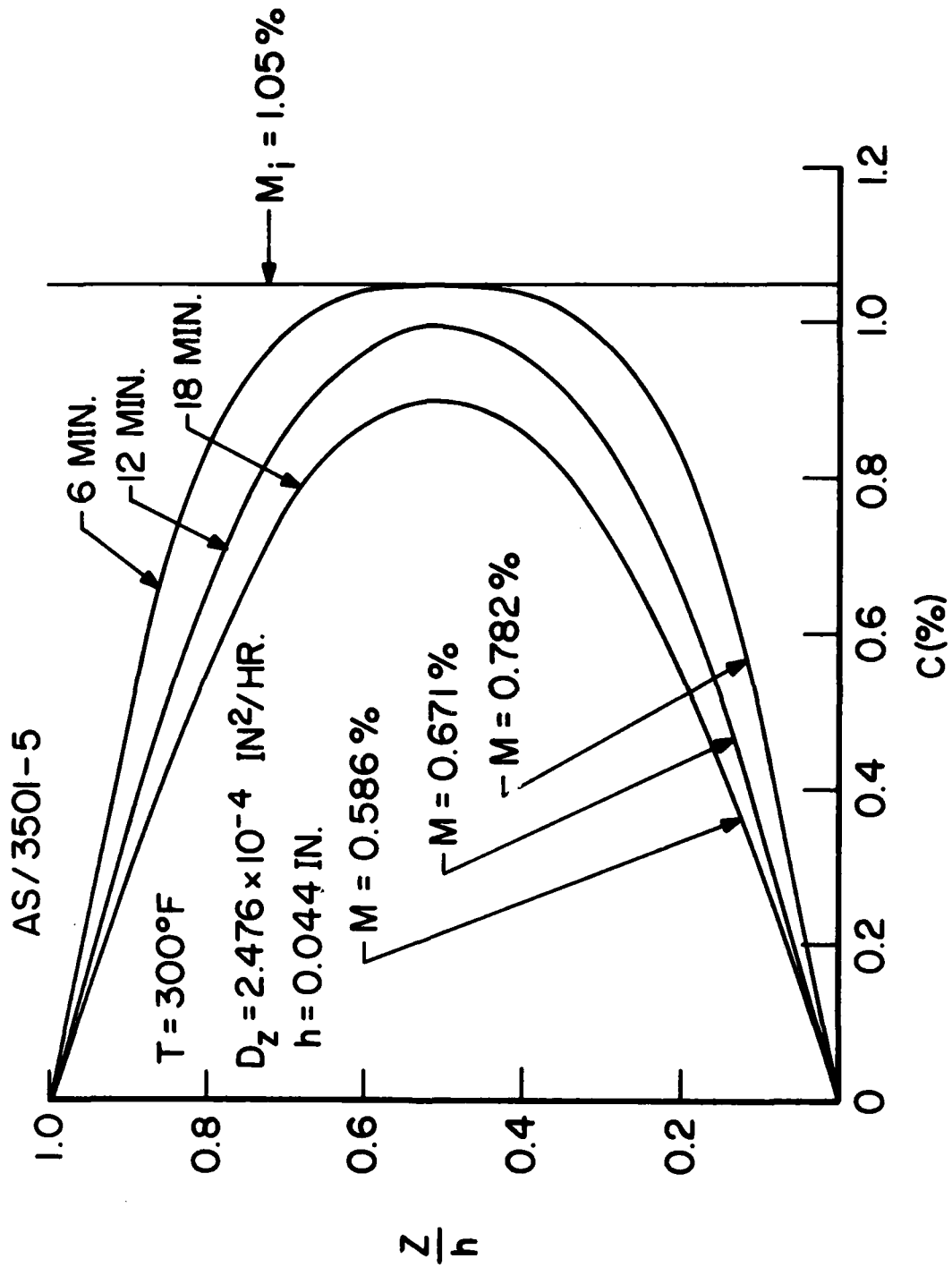


Figure 5. Moisture Profile Through-the-Thickness of AS/3501-5 Composites After Drying-Out for a Period of Time

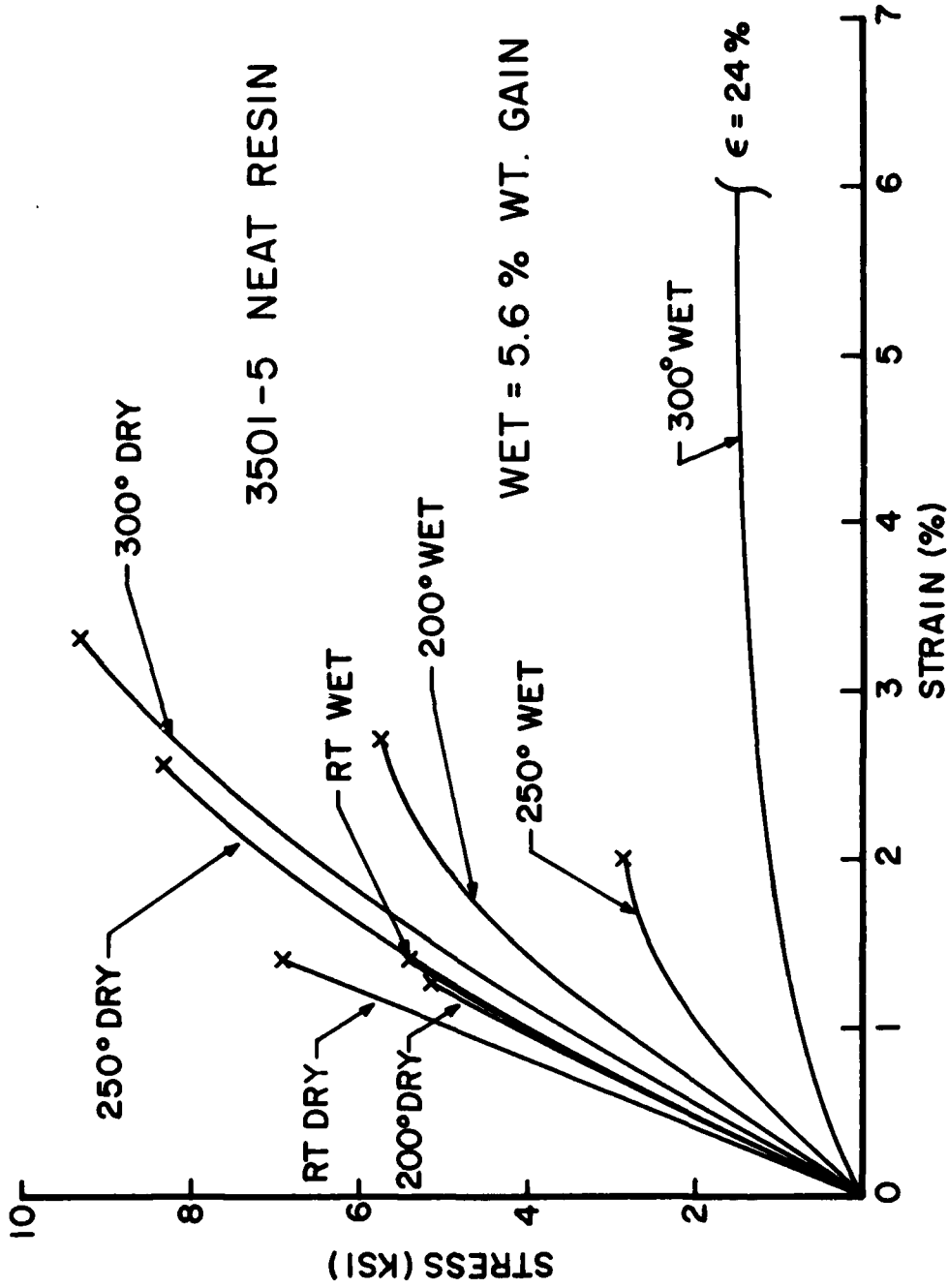


Figure 6. Average Stress-Strain Curves as a Function of Temperature for 3501-5 Neat Resin



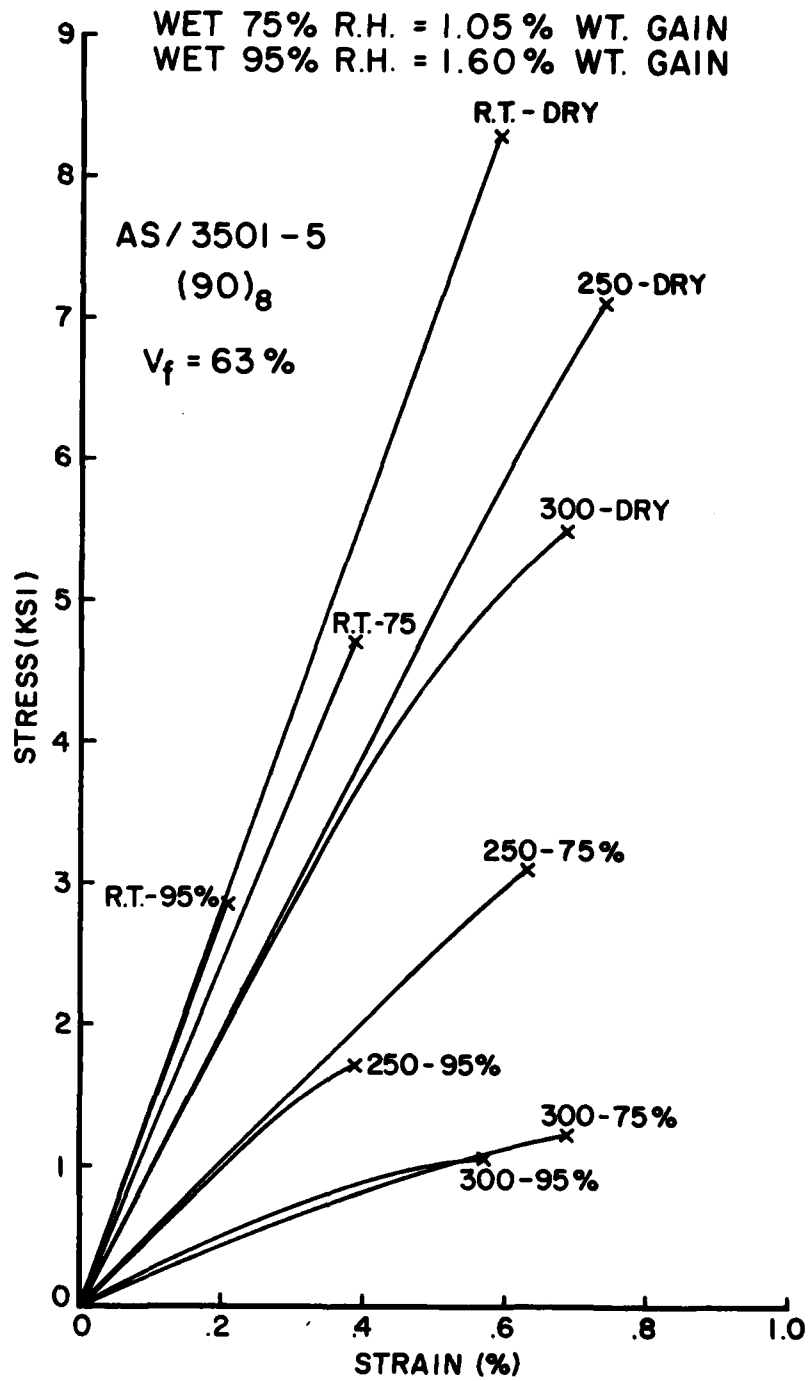


Figure 7. Typical Transverse Stress-Strain Curves as a Function of Temperature for AS/3501-5 Unidirectional Composites

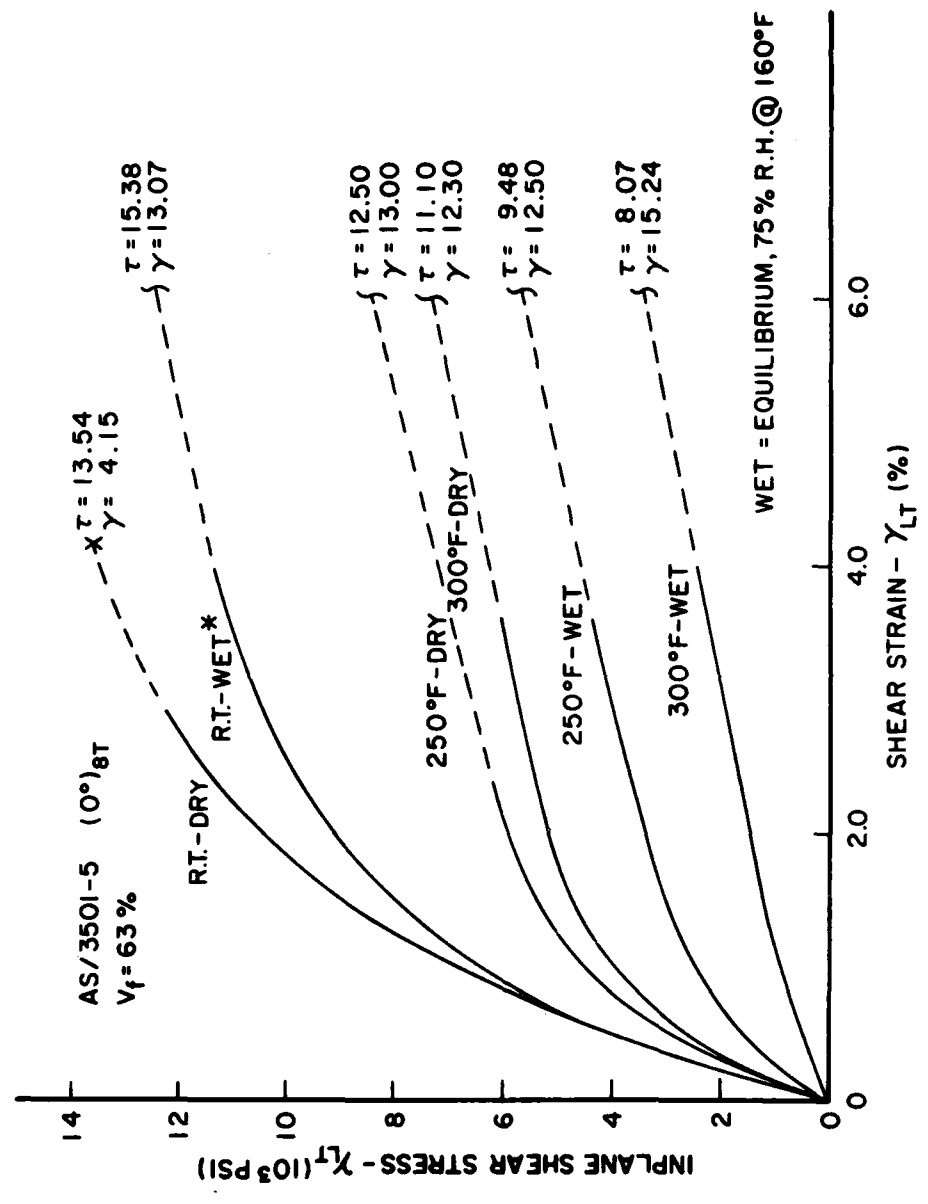


Figure 8. Typical In-Plane Shear Stress-Strain Curves for AS/3501-5 Unidirectional Composites

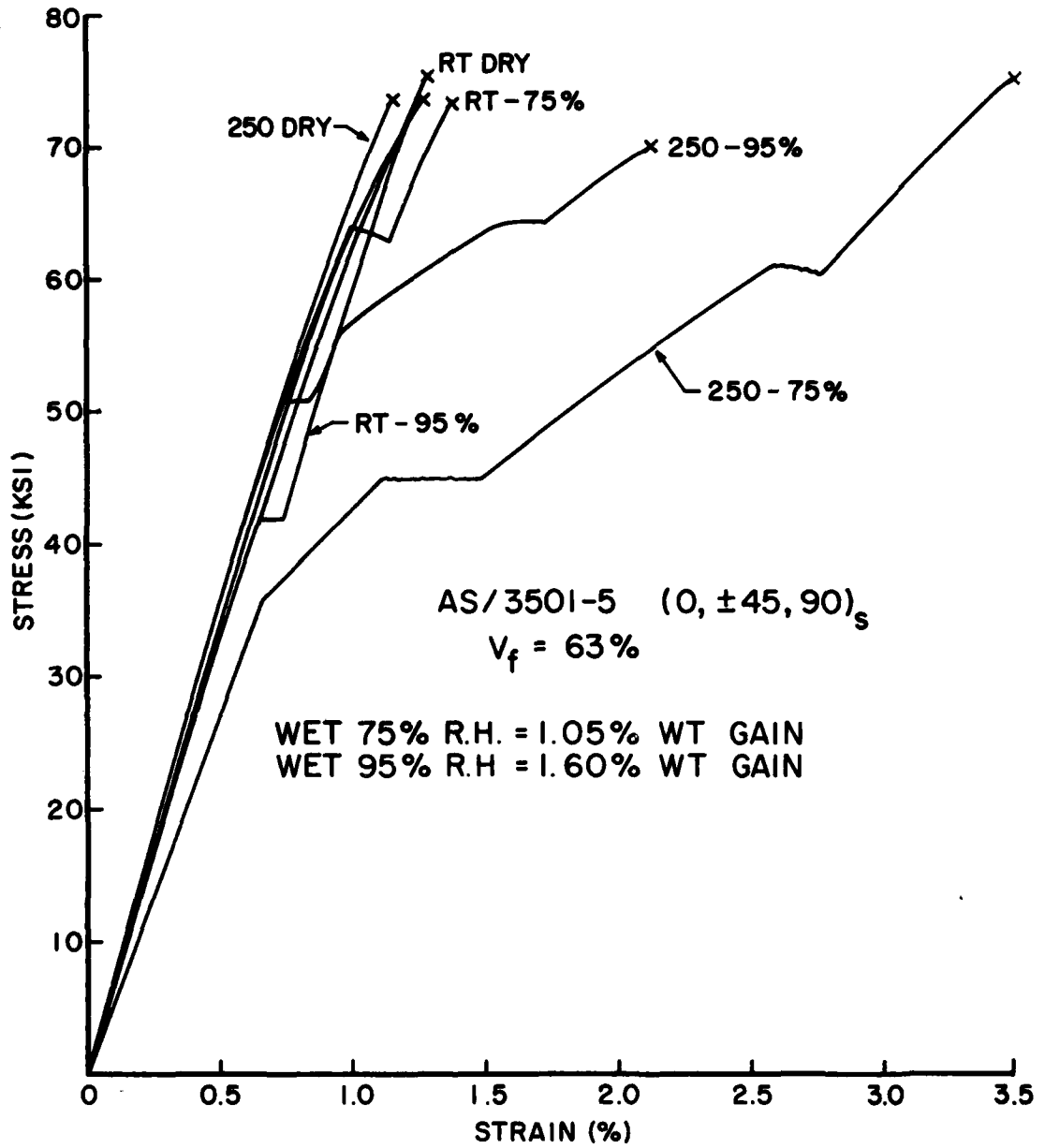


Figure 9. Typical Tensile Stress-Strain Curves for AS/3501-5 Quasi-Isotropic Laminates

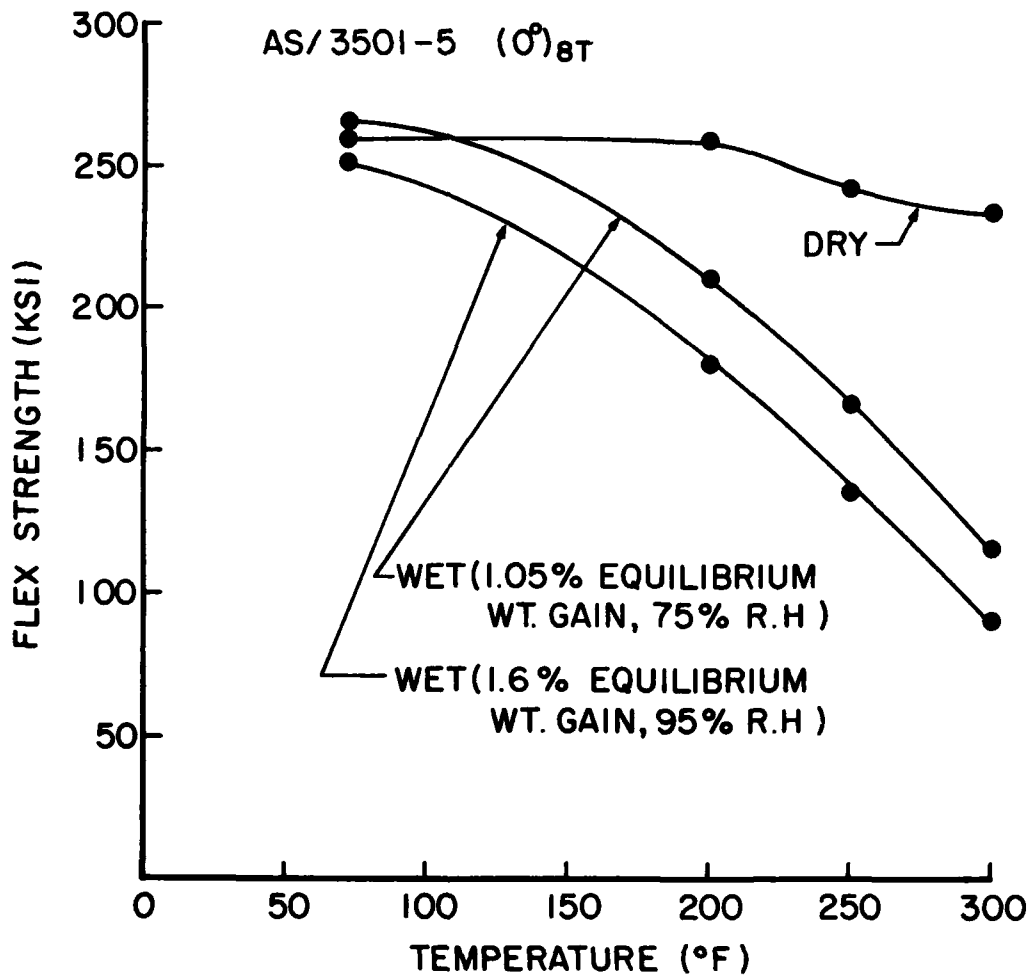


Figure 10. Flexure Strength as a Function of Temperature for AS/3501-5 Unidirectional Composites