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# VISCOELASTIC BEHAVIOR OF FIBER-REINFORCED COMPOSITE MATERIALS

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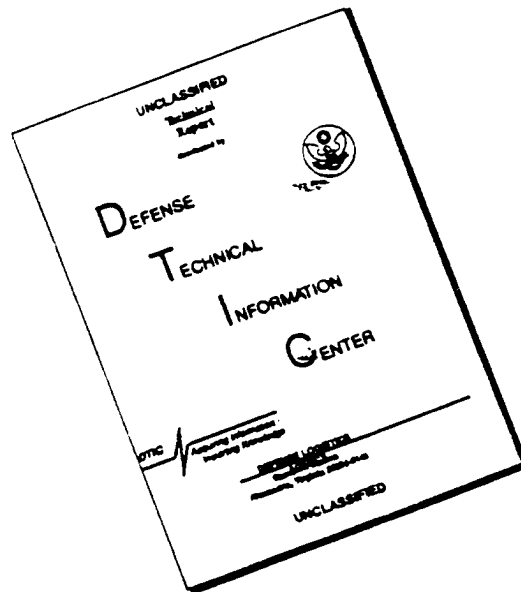
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COMPOSITE MATERIALS

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## FOREWORD

This report was prepared by Purdue Research Foundation, Lafayette, Indiana under USAF Contract F33615-67-C-1412. The contract was initiated under Project No. 7342, "Fundamental Research on Macromolecular Materials and Lubrication Phenomena," Task 734202, "Studies on the Structure-Properties Relationships of Polymeric Materials." The work was administrated by the Nonmetallic Materials Division, Air Force Materials Laboratory, with Mr. J. C. Halpin, MANE, as project scientist. This report prepared under Purdue Research Foundation, Project No. 4958, covers the period from 1 February 1967 to 31 December 1967, and was released by the authors May 1968 for publication as an AFML Technical report.

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W. P. JOHNSON, Acting Chief  
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## ABSTRACT

Tensile creep and recovery response of unidirectional, glass fiber-epoxy specimens in investigated. In the series of tests reported herein, all loading is normal to the fiber axis. The glass transition temperature,  $T_g$ , for the epoxy is determined from thermal expansion measurements normal to the fibers, and the temperature at which the material starts to appreciably soften is found by measuring creep in the presence of a slowly increasing temperature. Linearity of creep and recovery behavior is then investigated at room temperature and near  $T_g$ . The material at room temperature is found to be approximately linearly viscoelastic out to fracture, but considerable nonlinearity in the form of nonrecoverable deformation appears at temperatures in the neighborhood of  $T_g$ . In a concluding analytical study the influence of initial transients in creep and relaxation tests is estimated. This information provides a useful guideline for reducing creep and relaxation test data.

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## Section I

### INTRODUCTION

This report covers experimental work on creep and recovery behavior of unidirectional, glass fiber-reinforced epoxy. It represents the initial phase of a study directed toward the development of viscoelastic constitutive equations for hard fiber-reinforced materials.

Very little work in this specific area of composite research has appeared in the literature. However, as can be seen in the article by Halpin [1], existing data indicate that considerable nonlinearity may appear at stresses significantly below ultimate values, with the extent of nonlinearity dependent on the angle between fibers and loading axis.

Motivated by these observations, we are attempting to combine experimental and theoretical efforts in order to determine the limits of linear viscoelastic theory, and then develop constitutive equations which characterize filamentary composites under moderate and high stresses, relative to ultimate values.

In this report we discuss creep and recovery behavior when loading is normal to the fiber axis. This orientation was chosen for the initial work since the influence of the viscoelastic epoxy matrix is much greater than with loading along the elastic fibers. Experimental effort in the next reporting period will include studies on specimens with fibers oriented at intermediate angles.

In the following Section we first describe the specimen and experimental equipment. Then, in order to establish some guidelines for testing

at elevated temperatures, we report on measurements of the epoxy's glass transition and softening temperatures; these temperatures are found to be 130° F and 180° F, respectively. The fact that the softening temperature is so far above the epoxy's glass-transition value may be due to the reinforcement offered by the fibers.

The need for controlling relative humidity is brought out by showing the effect of water vapor loss on specimen strain.

These preliminary results are followed by data on creep and recovery along with predicted recovery based on linear theory. At room temperature the specimen is found to be approximately linear with respect to stress practically up to the ultimate value. However, significant nonlinearity is observed near the glass-transition temperature.

As mentioned above, we plan to test specimens having acute angles between fibers and loading axis in the next reporting period. On the basis of the data in [1], greater nonlinearity than shown here is expected.

The influence of initial transients in creep and relaxation tests is established analytically in the final Section of this report. It is shown, by using linear theory, and neglecting inertia, that initial transients are practically negligible at five times the loading time.

The Appendix contains an abstract of some theoretical work which was completed during the period covered by this report. The full paper is not included here since it was published in the July 1967 issue of Journal of Composite Materials.

## Section II

### EXPERIMENTAL WORK

#### 1. DESCRIPTION OF SPECIMEN

Unidirectional glass fiber-epoxy specimens, as shown in Figure 1, made of commercially available material called Scotchply were provided by the Contracting Agency. They were cut from larger plates to the 6" x  $\frac{1}{2}$ " size illustrated.

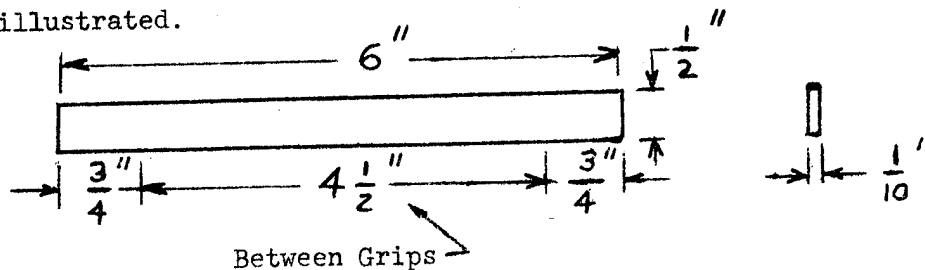


Figure 1. Tensile Specimen Configuration with  
Nominal Dimensions Shown

The specimens were fabricated by contacting eight plies for five minutes and then applying a pressure of 25 psi for 35 minutes. A temperature of 330° F was maintained throughout the pressure cycle.

The composite has a density of 1.67 g/cc, 62.4 weight percent fiber, and 47.6 volume percent fiber.

We are reporting here on tests made on five different specimens. For later reference, these specimens are numbered and listed in the following Table I, which shows information on the strain transducer used.

Table I

List of Specimens

Specimen No.	Strain Transducer Information
1	Strain gage with bonding cement having an allowable maximum temperature of 400°F and a bonding temperature at 225°F with bonding pressure of 15 psi.
2	Same as specimen no. 1
3	Strain gage with bonding cement having a maximum allowable temperature of 150°F and a bonding temperature at room temperature with bonding pressure of 10 psi.
4	Tuckerman optical strain gage for room temperature tests.
5	Same as specimen no. 4

## 2. DESCRIPTION OF EQUIPMENT

Figure 2 shows the equipment used for room-temperature testing. The specimen is loaded by means of weights attached to the end of a 6:1 lever arm.

Strain is measured using a Tuckerman gage [2] which consists of a three-mirror system; it is attached to the specimen by a rubber band. The illustrated autocollimator is used to read strain.

Another set of equipment is used for high temperature and it is shown in Figure 3. The control panel for an MTS\* hydraulic loading system is at the left side while on the right is a Bristol temperature and humidity controller. A Develco temperature chamber is shown inside the MTS loading frame. An adequate strain recorder was not available at the time these tests were made; therefore, a BLH\*\* strain indicator was used instead, and it is shown in front of the temperature chamber.

With existing equipment we were unable to apply a well-controlled load below the ultimate stress of the material, so the dead-weight system shown in Figure 4 was used.

An Instron grip at the top is attached to the MTS loading frame by a Formica connector (which is a low heat conductivity composite material) and the hook-type loading plate shown is used to hold weights up to 15 pounds.

The weight is supported by the ram of the MTS machine during the heating process. Then the weight is applied to the specimen by moving the

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\* MTS Systems Corporation, Minneapolis, Minnesota

\*\* Baldwin-Lima-Hamilton Corporation

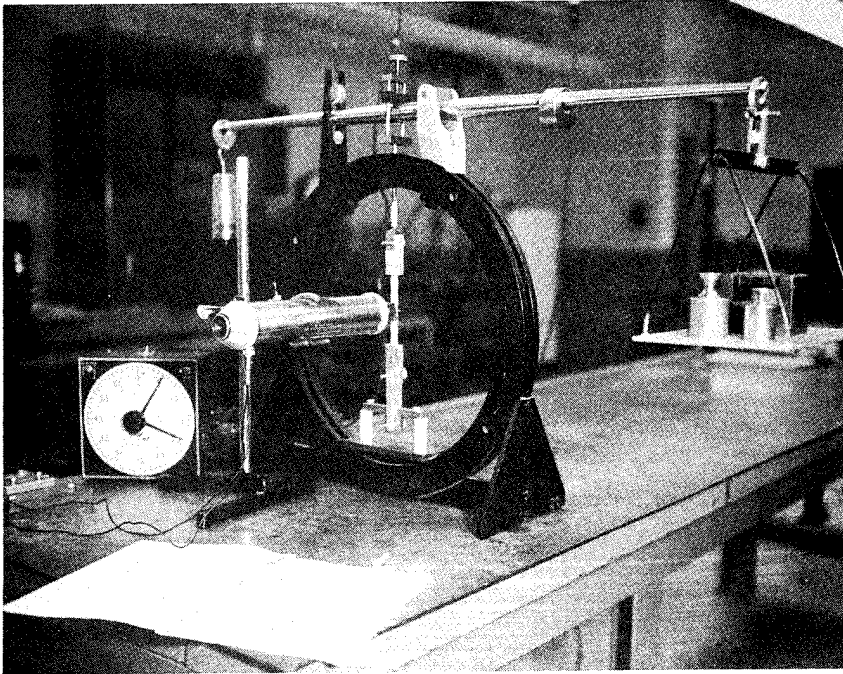


Figure 2 . Room-Temperature Testing Equipment

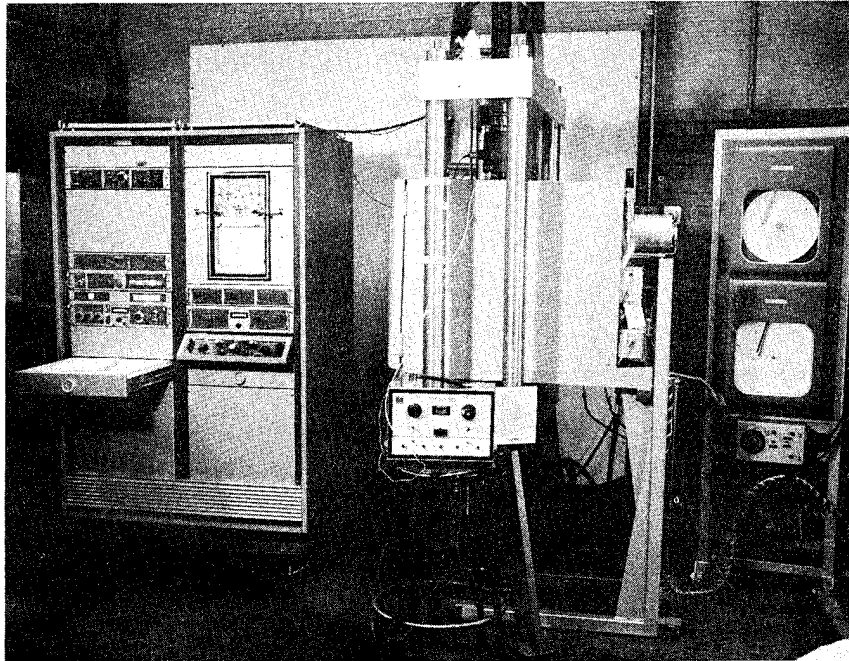


Figure 3. High-Temperature Testing Equipment

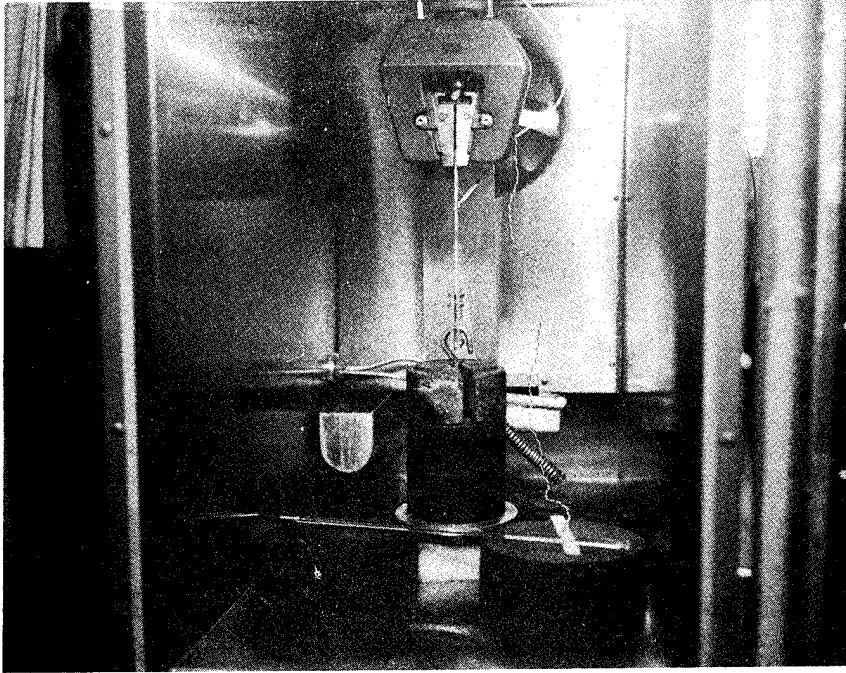


Figure 4. Loading System for High-Temperature Tests

ram down. A piece of silicon foam rubber is used under the top plate of the ram, and two pieces of transite plates are used on the top of the chamber to reduce heat leakage.

In order to measure strain at elevated temperatures, we used a BLH strain gage (FAER-25R-35 S 13) with a high resistance of 350 ohms.

Two different bonding agents were tried. First, we used EPY 600, which has a maximum allowable temperature of 400° F, and the curing is one hour at 225° F and 15 psi pressure. We later found that 225° F is higher than the softening temperature of the composite, so that the bonding pressure may have damaged the sample. We then used EPY 150, which has a maximum allowable temperature of 150° F, and cures at room temperature for 72 hours under a 10 psi pressure.

A check was made on the accuracy of the strain gage. At room temperature (74° F), records of strain under different load levels were taken simultaneously on sample No. 1 using both the Tuckerman gage and the strain gage. Figure 5 shows that they are in good agreement.

### 3. DETERMINATION OF GLASS TRANSITION AND SOFTENING TEMPERATURES

#### a. Glass Transition Temperature.

The thermal expansion coefficient of resin has an approximate discontinuity at a particular temperature, called the glass transition temperature. At this point the physical mechanism of deformation changes due to a significant change in free volume [1]. Typically, as the epoxy resin is heated through the transition temperature, its expansion coefficient can be expected to increase by a factor of two or three.

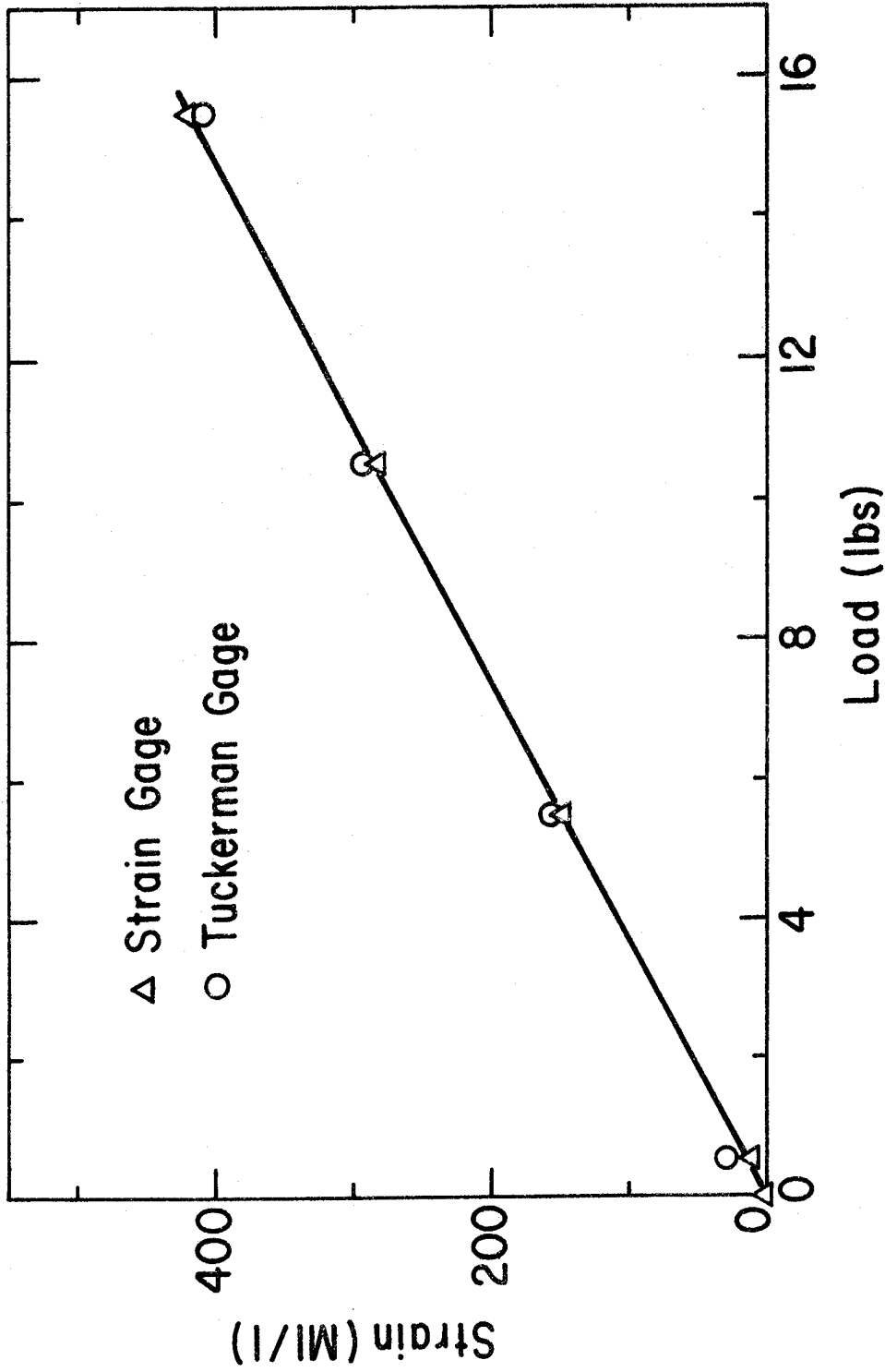


Figure 5. Comparison of Strain Gage and Tuckerman Gage Data

For the composite this increase will be smaller because of the constraint offered by the stiff fibers. Nevertheless, there should be an observable change in expansion coefficient, especially if expansion normal to the fibers is measured.

A thermal expansion test was made to find this transition temperature. We placed sample No. 1 in the temperature chamber, along with a free compensating gage. The presence of the free gage inside the temperature chamber compensates for the change of temperature coefficient of resistivity and the change in cross section area of the gage.

The strain recorded due to the temperature increases is  $\Delta\epsilon = \epsilon_s - \epsilon_g$  where  $\epsilon_s$  = strain due to the elongation of the sample and  $\epsilon_g$  = strain due to the elongation of the gage. The strain gage has a coefficient of thermal expansion of  $13.0 \text{ MI/I}^*/\text{F}$ , which is added to the gage reading to obtain total strain.

Figure 6 shows the data of three runs for the strain measured normal to the fiber axis. Coefficient  $\alpha_1$  is the initial thermal coefficient of the composite.

All three tests show that the glass temperature is around  $T_g = 130^\circ \text{ F}$ , and they all increase by a factor of 1.4 when passing through  $T_g$ . One possible reason different values of  $\alpha_1$  were found for each run is that the material was taken through its softening temperature ( $180^\circ \text{ F}$ ) each time; when the epoxy softens, it releases some residual strain and the fibers change position. Also, the gage apparently does not follow the specimen's strain above  $180^\circ \text{ F}$  because of the relatively high gage modulus, as implied by the data above  $180^\circ \text{ F}$  in Figure 6.

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\*  $\text{MI/I} \equiv$  microinches per inch

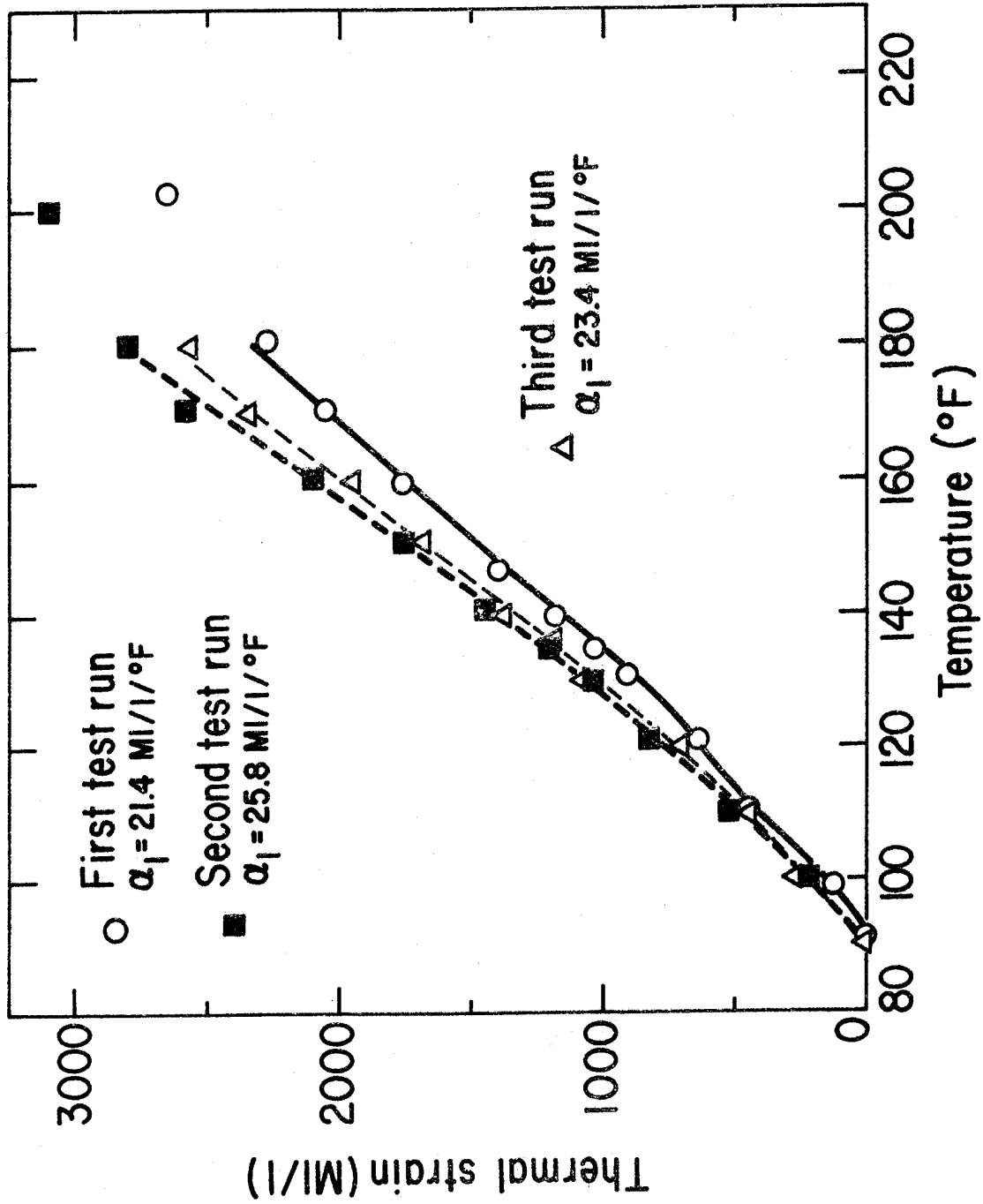


Figure 6. Effect of Increasing Temperature on Thermal Expansion

b. Softening Temperature.

Here, we define the softening temperature as the approximate value at which a rapid increase in creep rate begins as the specimen is slowly heated. This temperature is above the glass transition value, with the difference depending not only on the epoxy behavior, but also on the extent of reinforcement offered by the glass fibers.

Data on specimens Nos. 2 and 3 are shown in Figure 7. The indicated heating rates were sufficiently low that the specimen temperature was approximately uniform.

While the amount of creep is somewhat different for each run, all three indicate that the softening temperature is around 180° F. The difference between the creep above 180° F for the first and second runs probably is due to the difference in heating rates.

4. ROOM TEMPERATURE BEHAVIOR

a. The Stress-Strain Relation.

The creep compliance for cross-linked polymeric materials can often be described quite adequately by the power law form [3],

$$\epsilon/\sigma_0 \equiv D(t) = D_g + \Delta D(t/\tau_0)^m \quad (1)$$

at short times. At long times it is necessary to account for the equilibrium compliance by using a modified form of the relation (1). The following definitions are used in Equation (1):

$$m = \text{constant (typically } 0 < m < 0.5)$$

$$\Delta D = \text{constant}$$

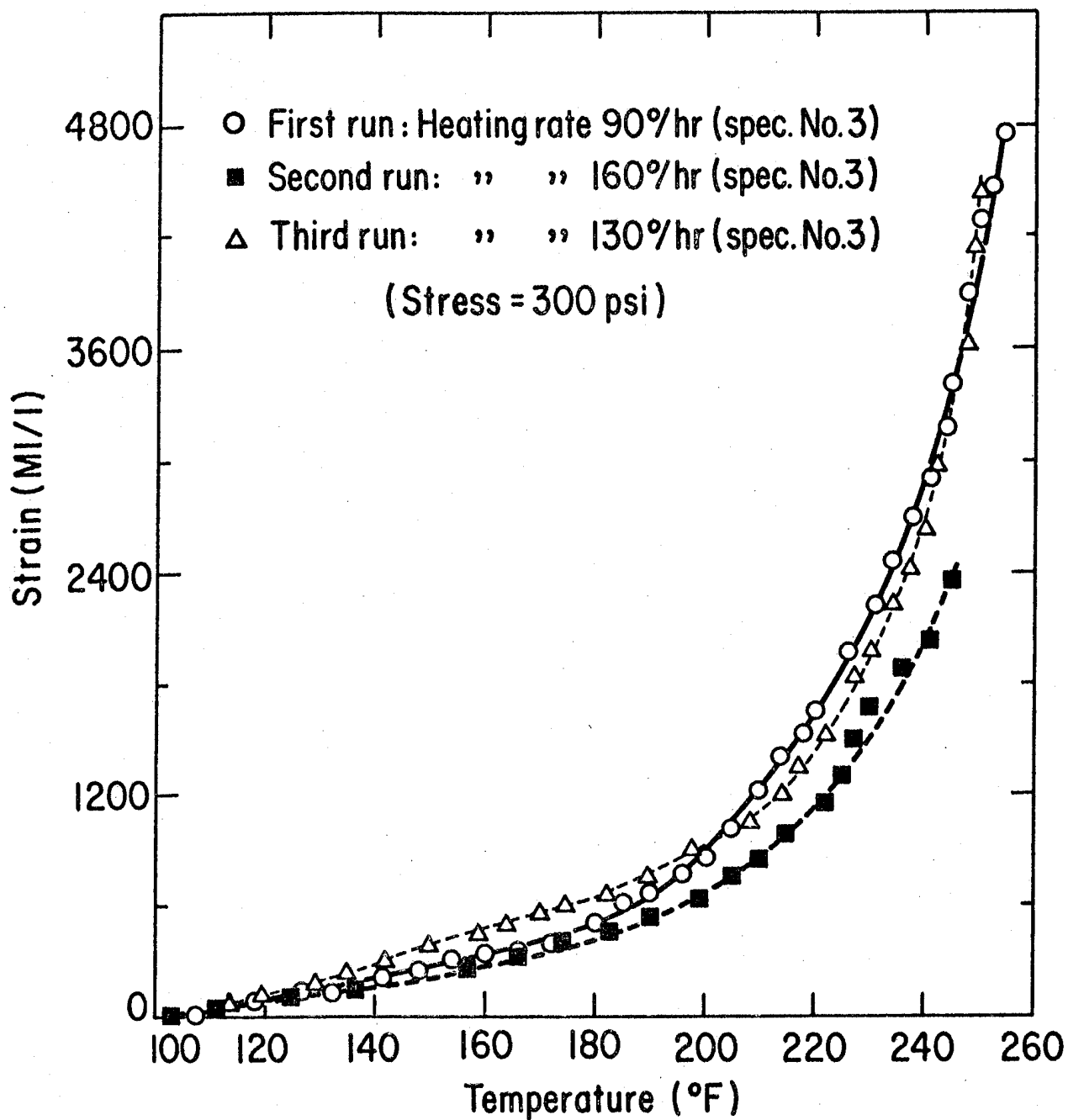


Figure 7. Effect of Increasing Temperature on Creep

$D_g \equiv D(o) =$  initial elastic compliance

$\tau_o =$  time constant

$\sigma_o =$  constant applied stress

It will be shown that this power law form fits our room temperature results on the composite very well. Namely, the creep strain is

$$\epsilon(t) = \epsilon_o + ct^m \quad (2)$$

where  $\epsilon_o = D_g \sigma_o$

$$c = \frac{\Delta D}{(\tau_o)^m} \sigma_o$$

Here  $D_g$  and  $\Delta D$  refer to the composite specimen's compliance for loading normal to the fiber axis.

$\epsilon_o$  is the constant strain produced at the moment the load is applied. Experimentally, it took us approximately 5 seconds to read the first data, and therefore we were unable to accurately determine the initial compliance.

Nevertheless, for convenience, we shall call our first reading the "initial strain."

Initial strain results on two different specimens are shown in Figure 8. Apart from the data on the post-cured specimen, the response is essentially linear with an initial modulus of approximately

$$E(o) \equiv \frac{1}{D_g} \sim 1.0 \times 10^6 \text{ psi} \quad (3)$$

It was found that the breaking stress is around 2000 psi, and that the samples broke abruptly without any noticeable cracking prior to complete fracture.

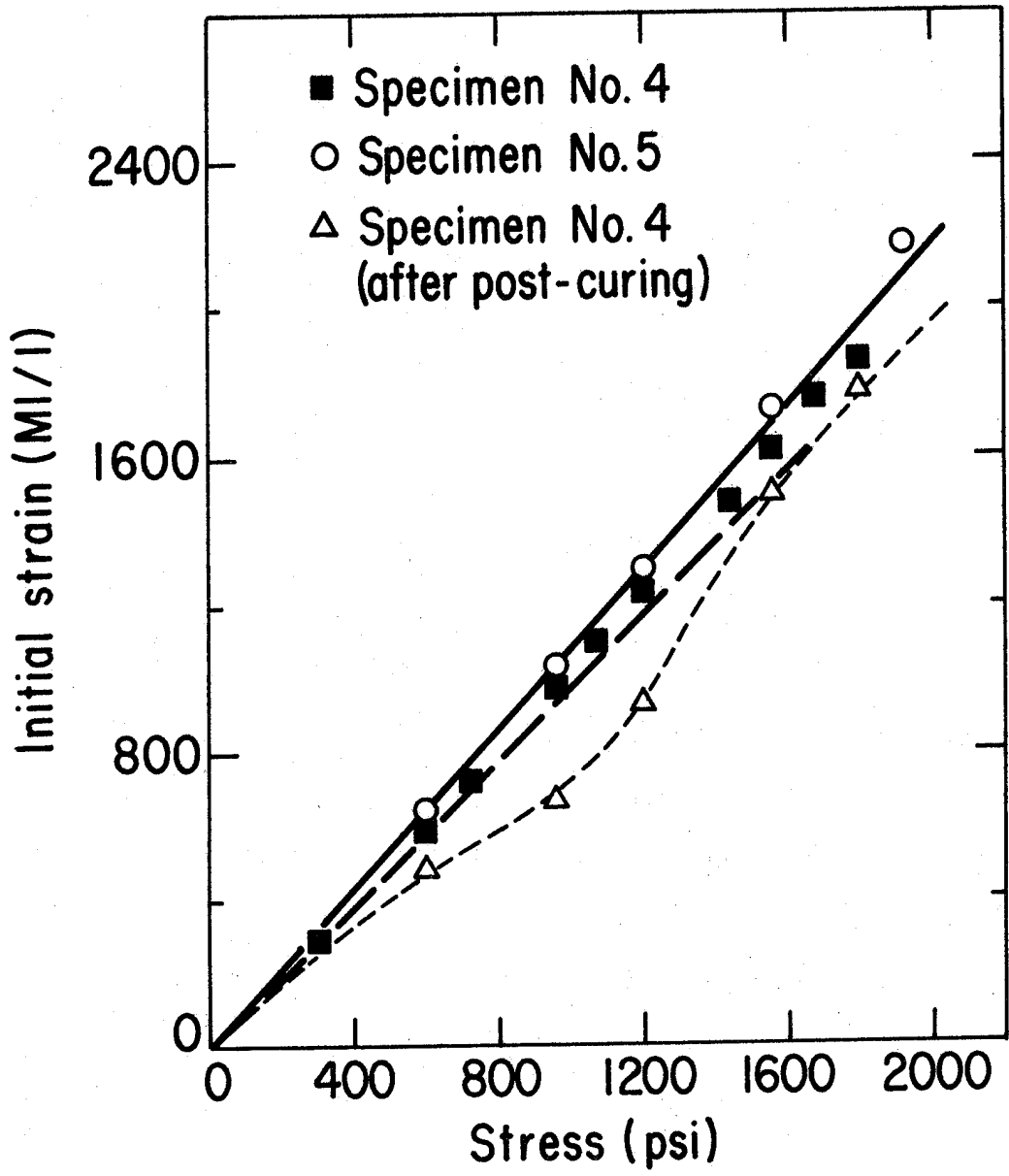


Figure 8. Initial Strain at Room Temperature

An attempt was also made to determine if the material was fully cured. Sample No. 4 was reheated to 346° F and held at this temperature for 12 hours. This treatment turned its color much darker and produced the non-linear behavior shown in Figure 8. However its breaking stress was still around 2000 psi, and average modulus at fracture about the same as observed prior to the post-curing. Therefore, the original specimens are probably well-cured, but in future work this point will be re-examined.

Finally, it is of interest to compare the measured transverse modulus, Equation (3), with a micromechanics prediction. Using typical elastic properties [4] for the resin

$$E_r = 0.6 \times 10^6 \text{ psi}, \nu_r = 0.34 \quad (4a)$$

and for the fiber

$$E_f = 11 \times 10^6 \text{ psi}, \nu_f = 0.22 \quad (4b)$$

we find from Hermans' model [5] that the transverse modulus is

$$E = 1.12 \times 10^6 \text{ psi} \quad (5)$$

Certainly within experimental error and the uncertainty in using values (4), the measured value (3) and predicted value (5) are in agreement. It is also noteworthy that when we use the lower bound prediction  $E_L$  (which is valid regardless of phase geometry [6]),

$$\frac{1}{E_L} = \frac{V_f}{E_f} + \frac{V_r}{E_r} \quad (6)$$

we find essentially the same result; viz  $E_L \cong 1.10 \times 10^6$  psi. Here,  $V_f$  and  $V_r$  are the volume fraction of fiber and resin, respectively.

b. Effect of Humidity.

It is known that the absorption of water vapor by the specimen will affect the material in the same way as an increase in temperature [1]. Specifically, the specimen will dilate and its relaxation times will shorten due to water vapor. Our results show that even a small change of humidity can affect the sample appreciably at room temperature.

Sample No. 4 was stored in a refrigerator, which had a constant temperature of  $36.5^{\circ}$  F and relative humidity of 52.5%. However, the laboratory had a constant temperature of  $74^{\circ}$  F and humidity of 26%. As a result, after the specimen was removed from the refrigerator and allowed to reach room temperature, it was observed to shrink continuously. A record was taken over a range of eighteen hours, and the data are shown in Figure 9.

Three creep tests were performed on sample No. 4 under the stress input of 960 psi, to show how greatly the humidity influences the response. The results are shown in Figure 10. The first run was made on the same day the specimen was removed from the refrigerator, while the sample was still losing water vapor rapidly. It shows less creep than in the later tests because its volume was continuously shrinking. In fact, the recovery curve reached negative strain values with the help of the shrinkage due to the loss of moisture. No significant shrinkage occurred on the seventh day, and the creep and recovery for this period is also shown in Figure 10.

c. Creep Results.

A series of creep and recovery tests were made on specimen No. 4 when it had reached approximate equilibrium with respect to room temperature and humidity. First we shall report on the creep portion. A plot of

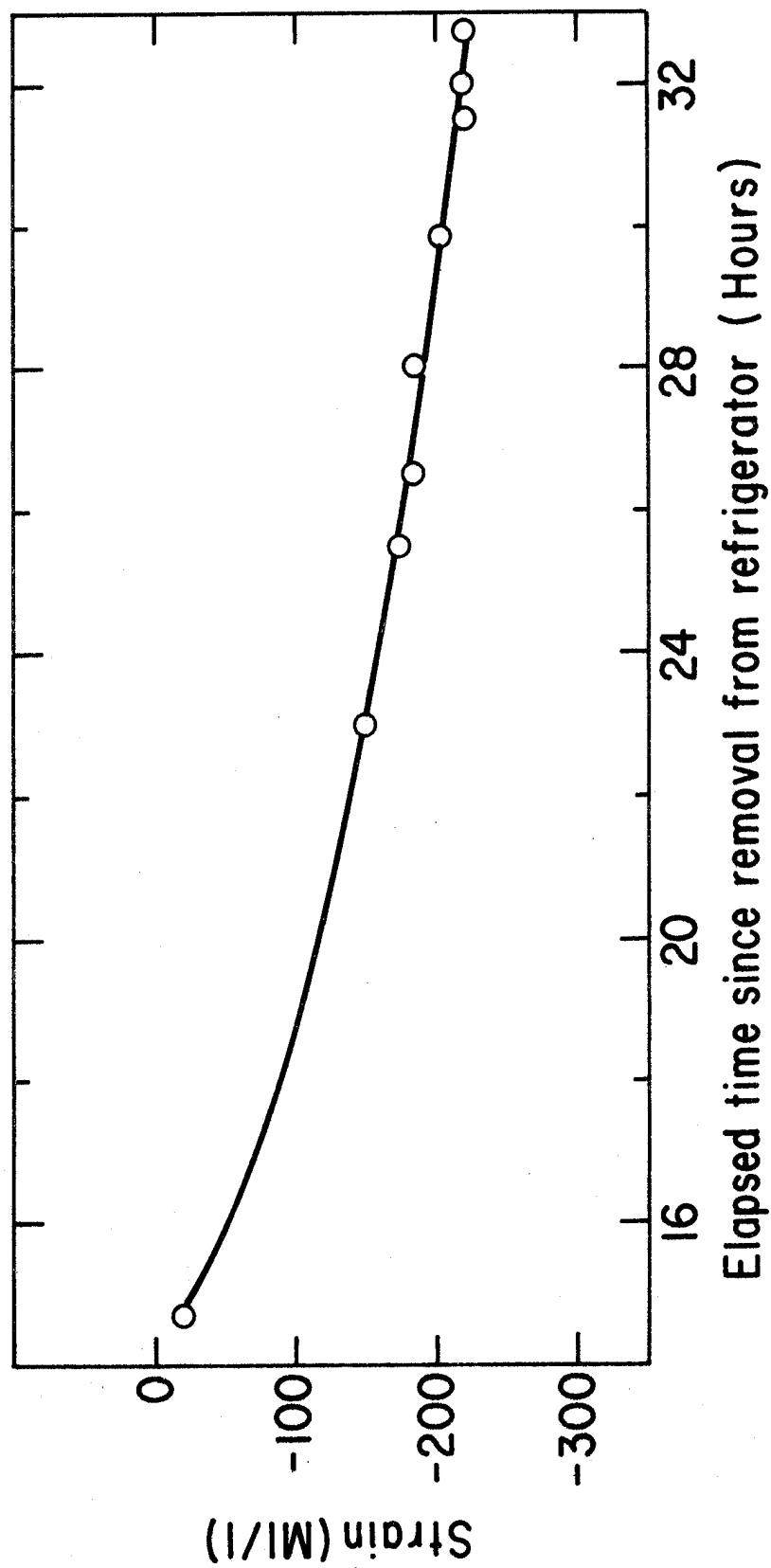


Figure 9. Shrinkage Effect Due to Loss of Water Vapor

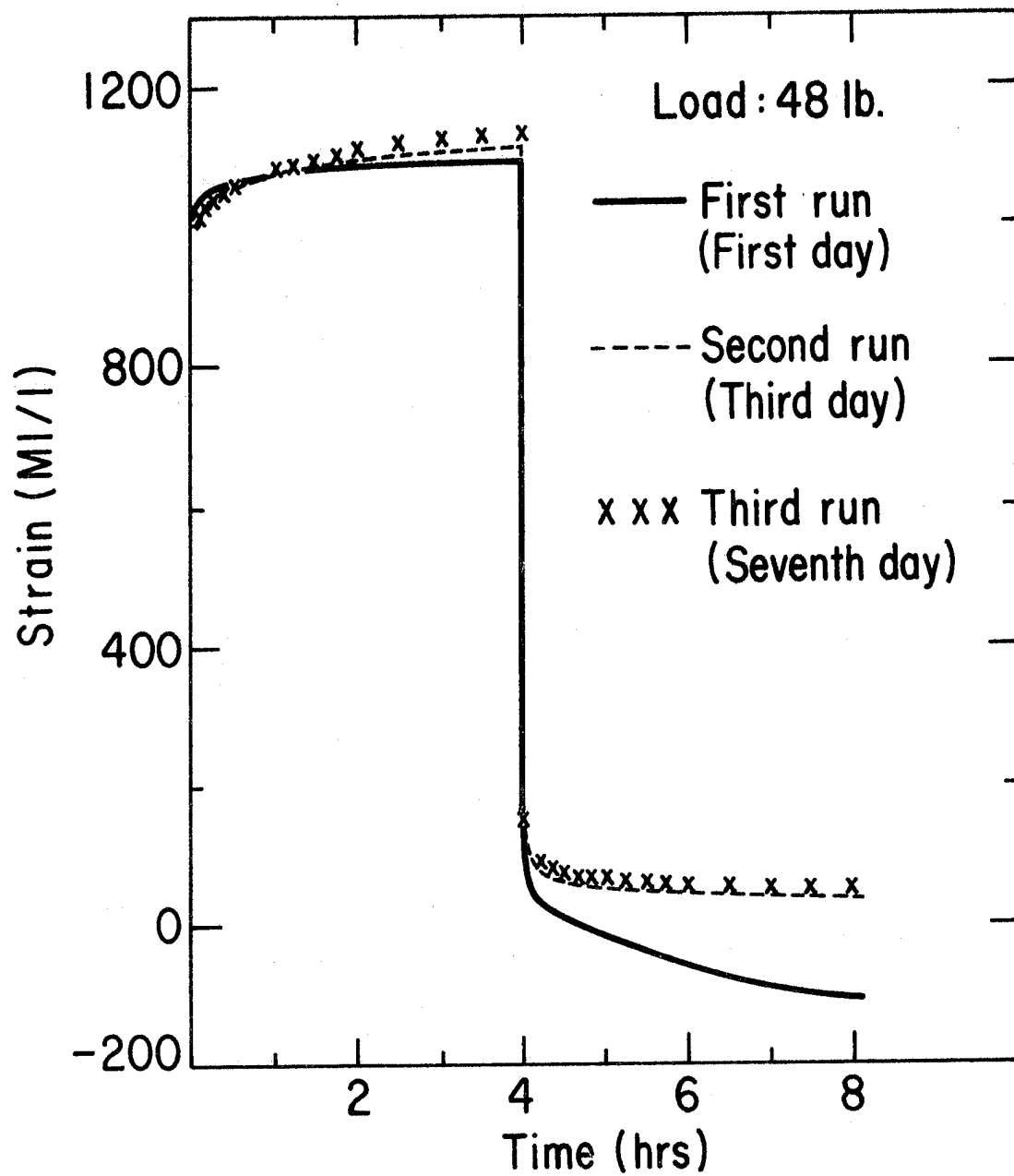


Figure 10. Effect of Humidity on Creep and Recovery

"net creep strain" (i.e. total creep strain minus the initial strain) for different stress levels is shown in Figure 11. The straight lines on the log-log plot indicate that the creep of this composite follows the power law, Equation (2). It also should be noted that by extending the straight line we can predict the creep response for a longer period of time.

Figure 12 shows that the net creep compliance (defined as the ratio of net creep strain to applied stress) for different creep times is essentially independent of stress levels. Since the ultimate stress is approximately 2000 psi the transverse behavior is seen to be linearly viscoelastic under creep conditions practically out to fracture.

Using the data in Figures 8 and 11, we find that the transverse creep compliance of specimen No. 4 is approximately

$$\frac{\epsilon}{\sigma_0} = D(t) \cong [1.0 + 0.1 t^{0.35}] \times 10^{-6} \text{ (psi}^{-1}\text{)} \quad (7)$$

where time (t) is in hours. A plot of this compliance is shown in Figure 13.

Another series of fifteen minute creep tests at various stress levels were made on sample No. 5. It also showed linear response with a slightly larger (5-9%) net creep strain.

#### d. Recovery Results.

Recovery of a linear viscoelastic specimen following removal of a constant load is related very simply to the creep strain. Here we shall use the relation to further check on linearity of Specimen No. 4.

Suppose the constant stress  $\sigma_0$  is applied at  $t = 0$  and removed at  $t = t_1$ . Then the relation between creep and recovery behavior is as shown in Figure 14 [1].

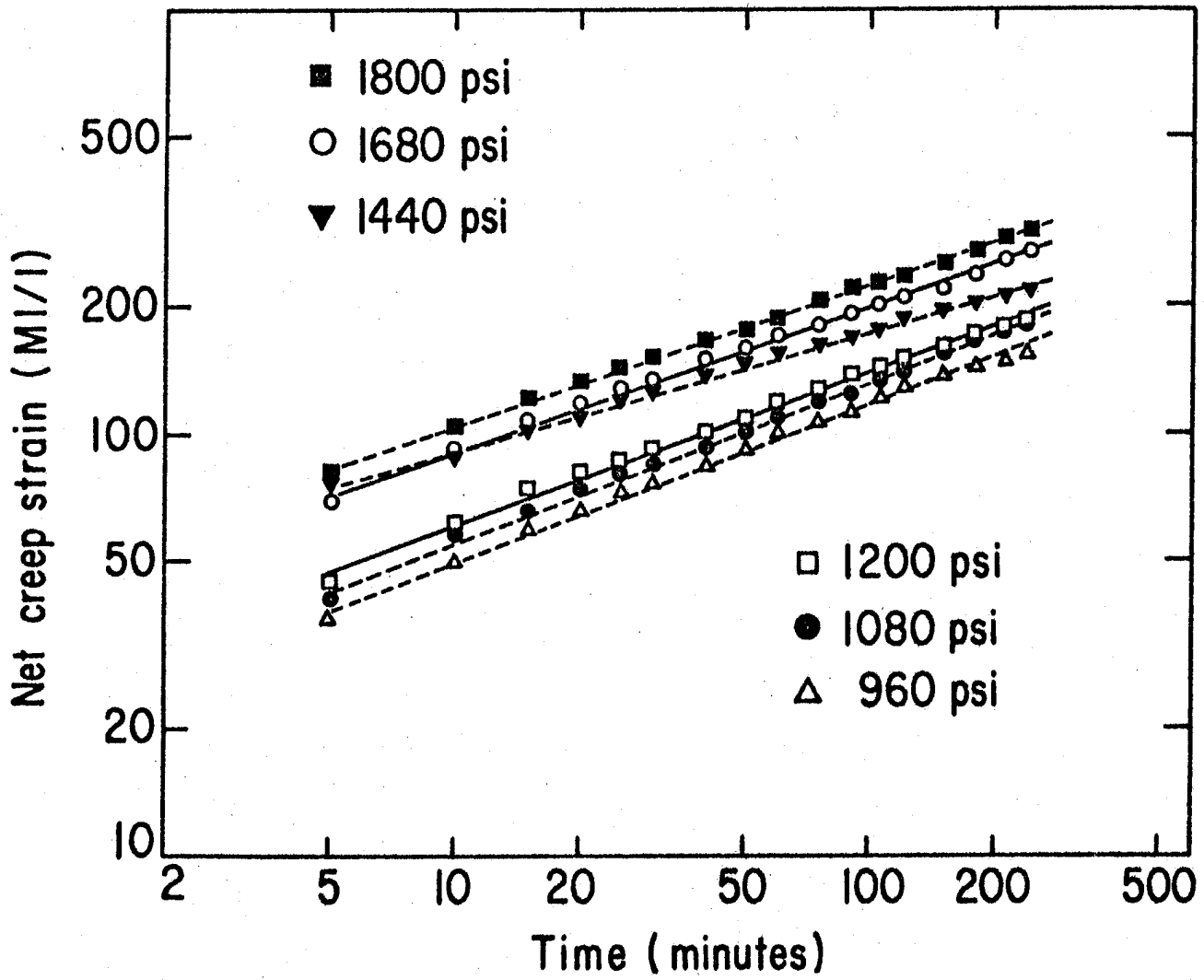


Figure 11. Net Creep Strain for Different Stress Levels

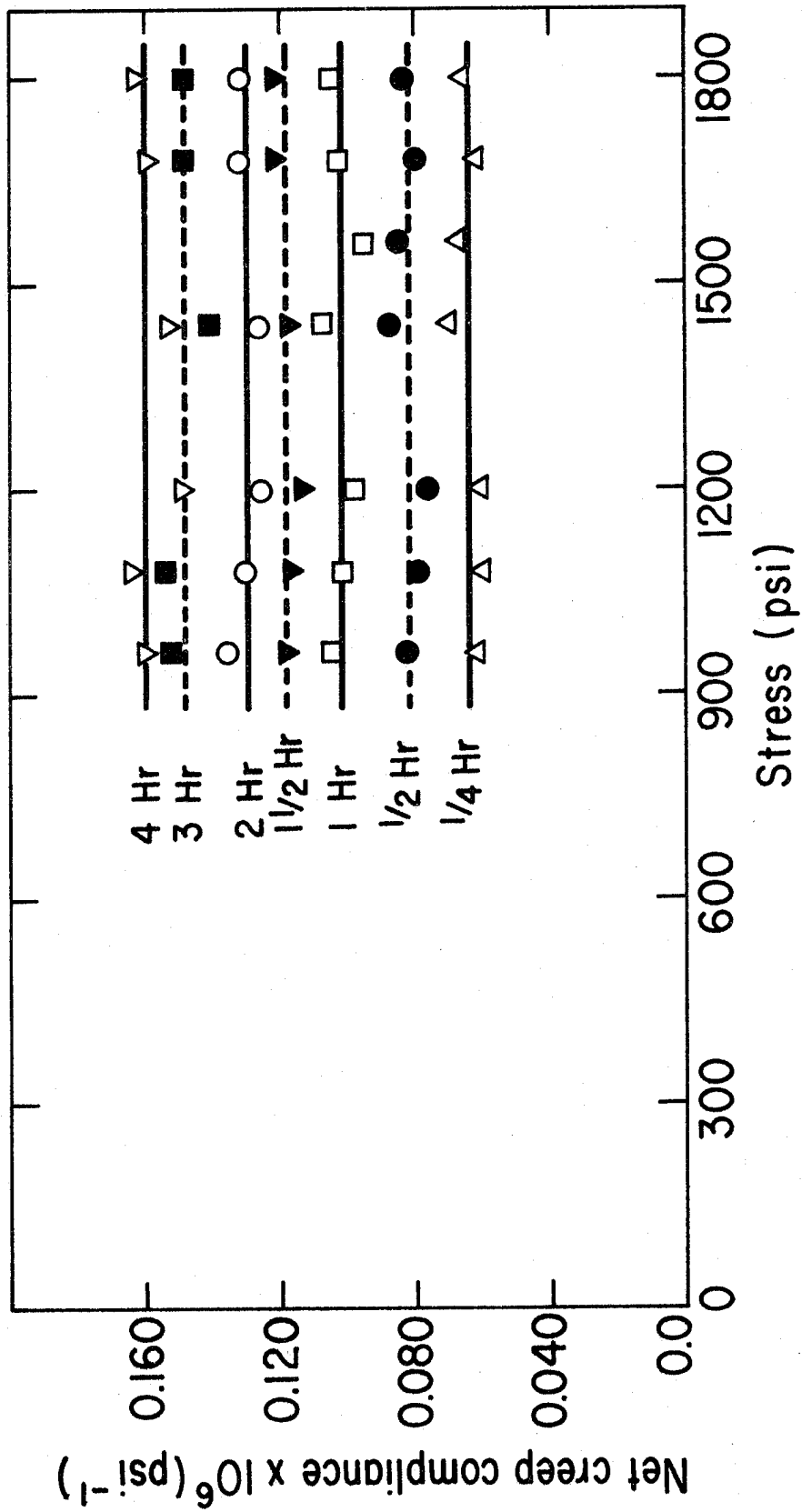


Figure 12. Effect of Stress Level on the Creep Compliance

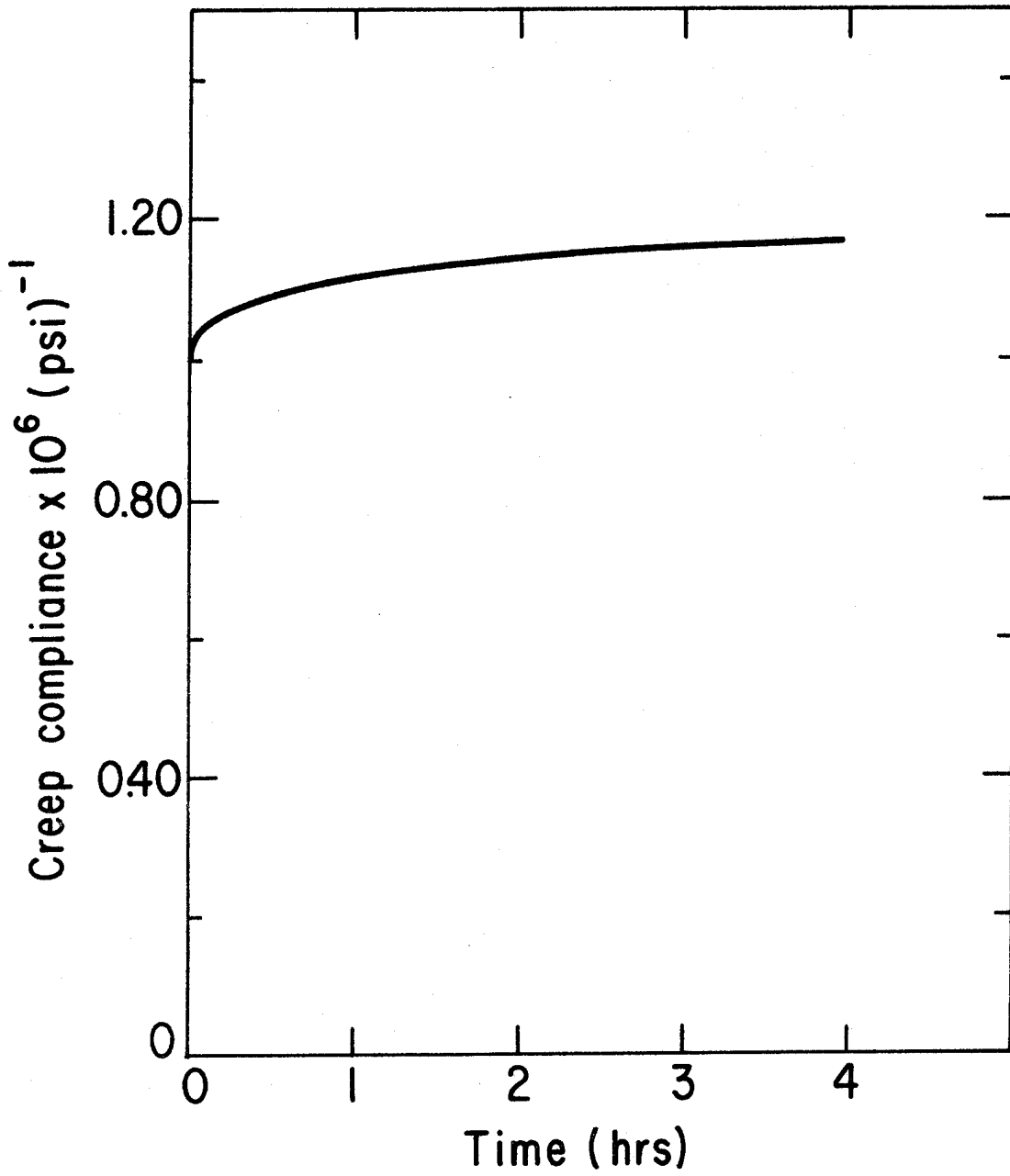


Figure 13. Transverse Creep Compliance

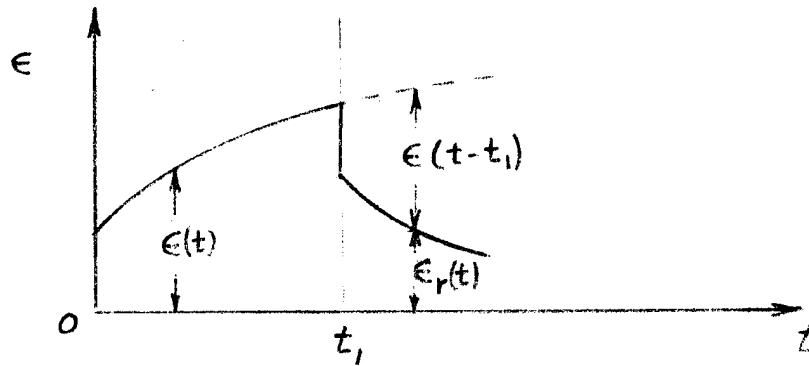


Figure 14. Relation Between Creep and Recovery Strain

From Figure 14 it is seen that

$$\epsilon_r(t) = \epsilon(t) - \epsilon(t - t_1) \quad : \quad t \geq t_1 \quad (8)$$

where  $\epsilon_r(t)$  is the specimen strain following removal of the stress and  $\epsilon(t)$  is the creep strain that would have existed for  $t > t_1$  had the stress not been removed. In order to extend our creep data beyond  $t_1$  we assumed that the power law ( 2 ) is applicable.

Measured recovery curves are compared with the predicted recovery curves for the different stress levels of 1080, 1440, and 1800 psi in Figures 15 through 17, respectively. In order to show sufficient detail we have subtracted the large initial strain  $\epsilon(0)$  from the creep data. Equation (8) is then applied in the form

$$\epsilon_r(t) = [\epsilon(t) - \epsilon(0)] - [\epsilon(t - t_1) - \epsilon(0)] \quad (9)$$

As the stress approaches the ultimate value of 2000 psi, the difference between predicted and measured recovery strain increases. However, compared to the total strain experienced in creep, this difference is quite small.

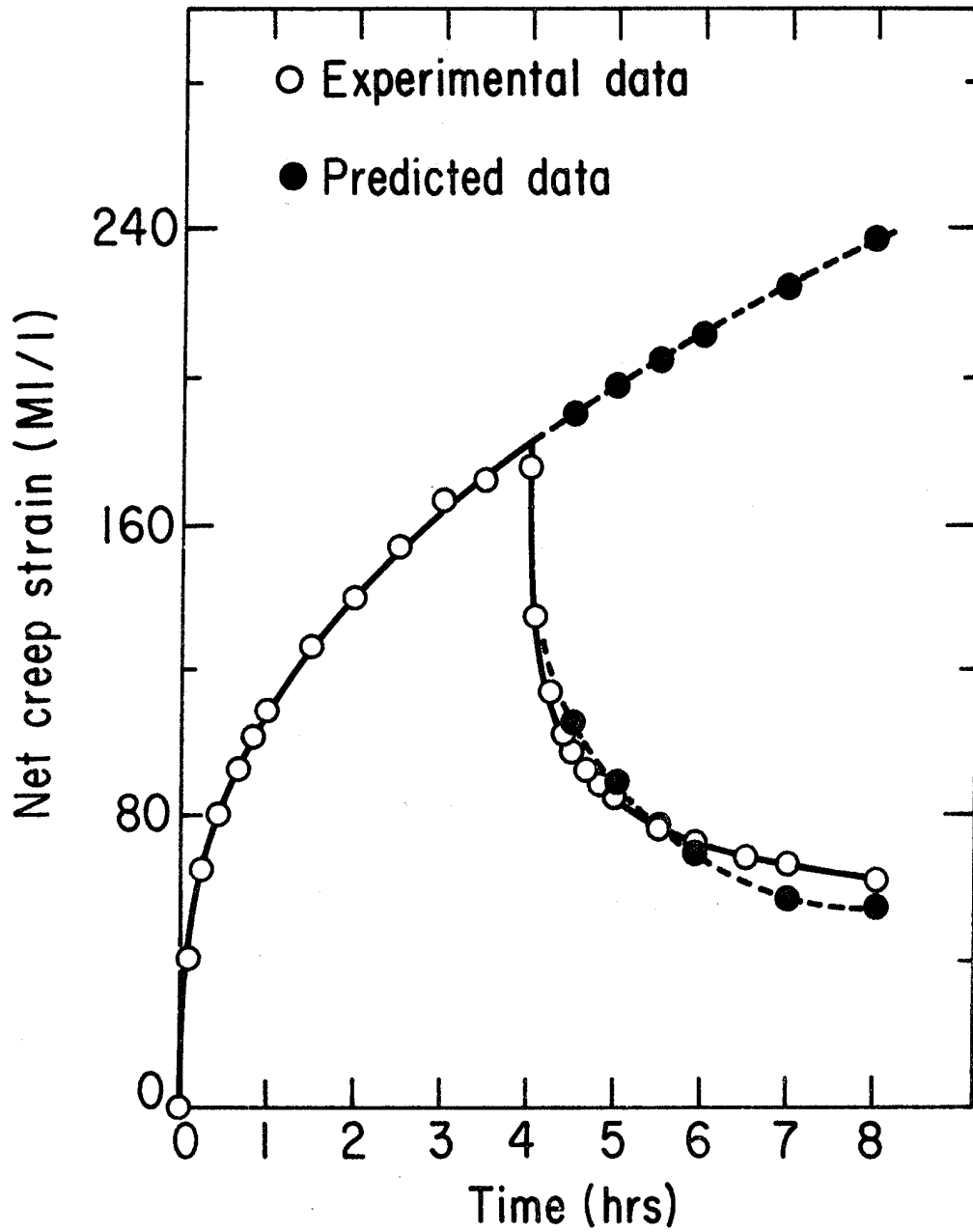


Figure 15. Creep-Recovery Curve (Stress = 1080 psi)

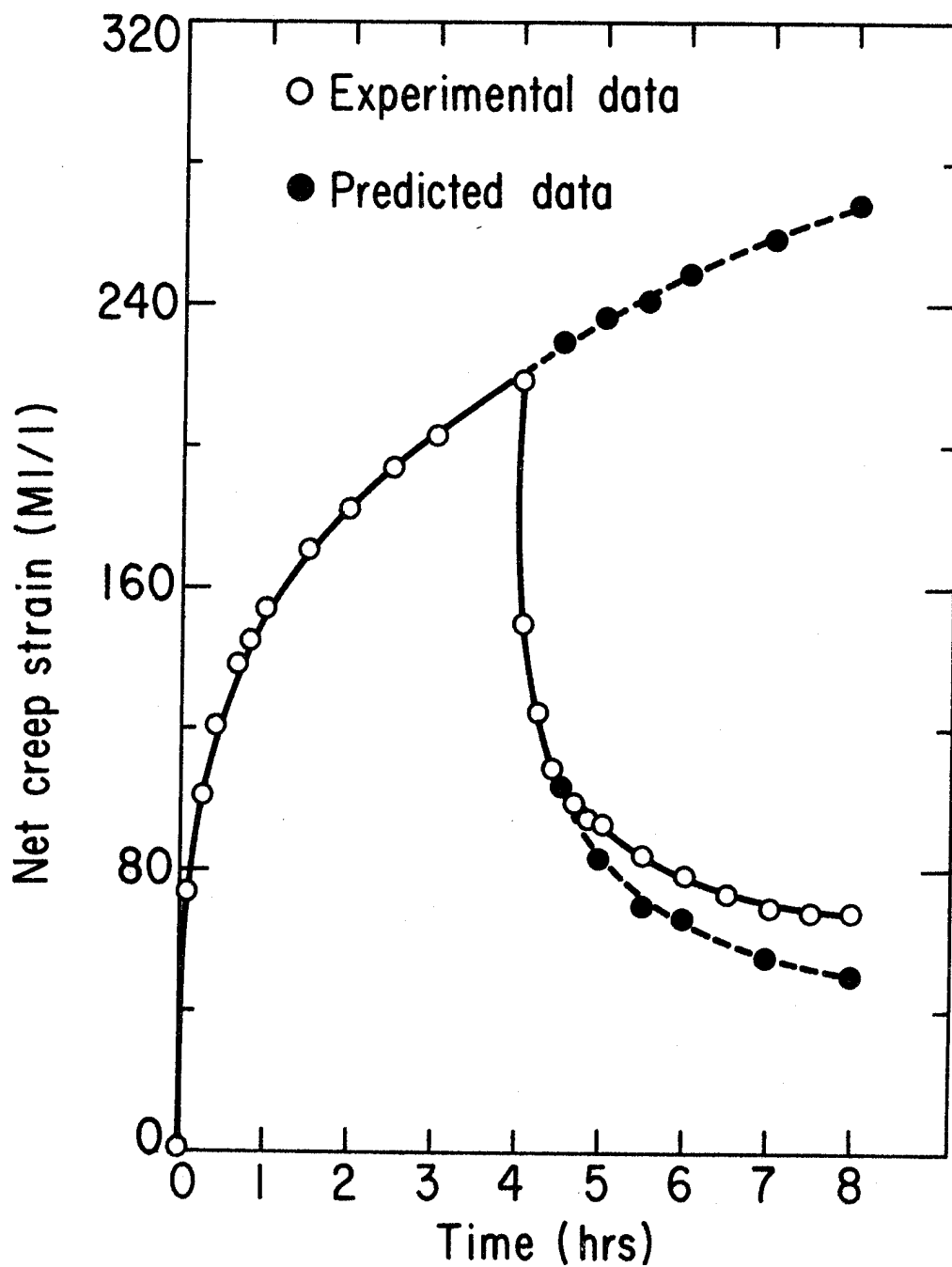


Figure 16. Creep-Recovery Curve (Stress = 1440 psi)

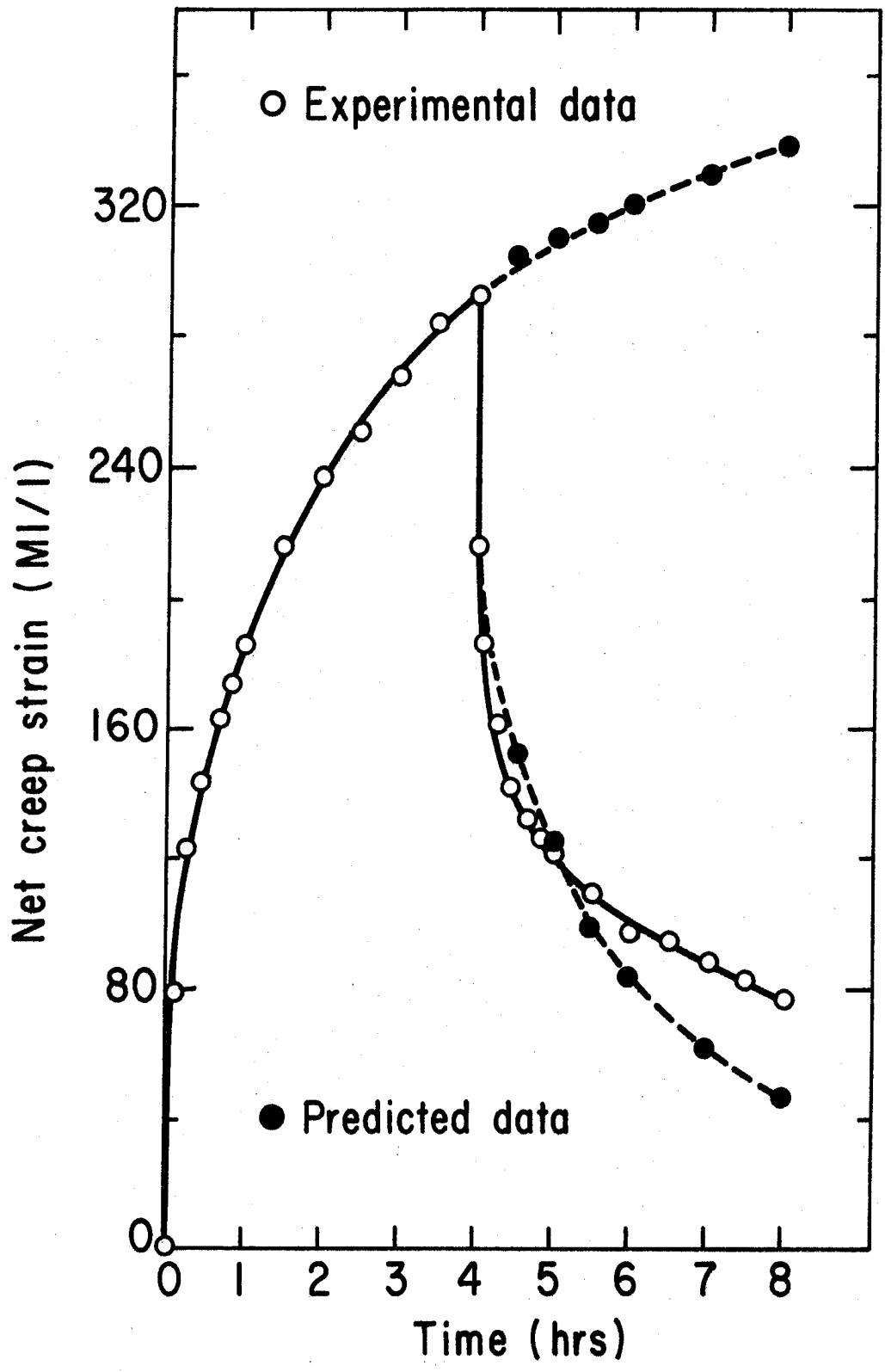


Figure 17. Creep-Recovery Curve (Stress = 1800 psi)

## 5. HIGH TEMPERATURE BEHAVIOR

A preliminary experimental study was made to determine the creep and recovery behavior in the neighborhood of the glass-transition temperature of 130° F.

In Figure 18 is shown typical creep and recovery data, in which the initial strain of  $\epsilon(0) = 292 \text{ MI/I}$  has been subtracted from the creep data. The measured strain after load removal is seen to be significantly greater than predicted by linear theory. Specimen No. 3 was used in this test.

Figures 19 and 20 show a sequence of creep and recovery curves obtained from a series of tests on sample No. 1. It should be pointed out that there is at least one day between each test, and that the strain is referred to the unstressed specimen length existing on the day of the test.

The first test shown in Figure 19 was made under essentially the same environment and loading as the last test in Figure 20. Comparison of these curves shows that the initial strain has increased due to the loading and unloading cycles, while the time-dependent component has decreased.

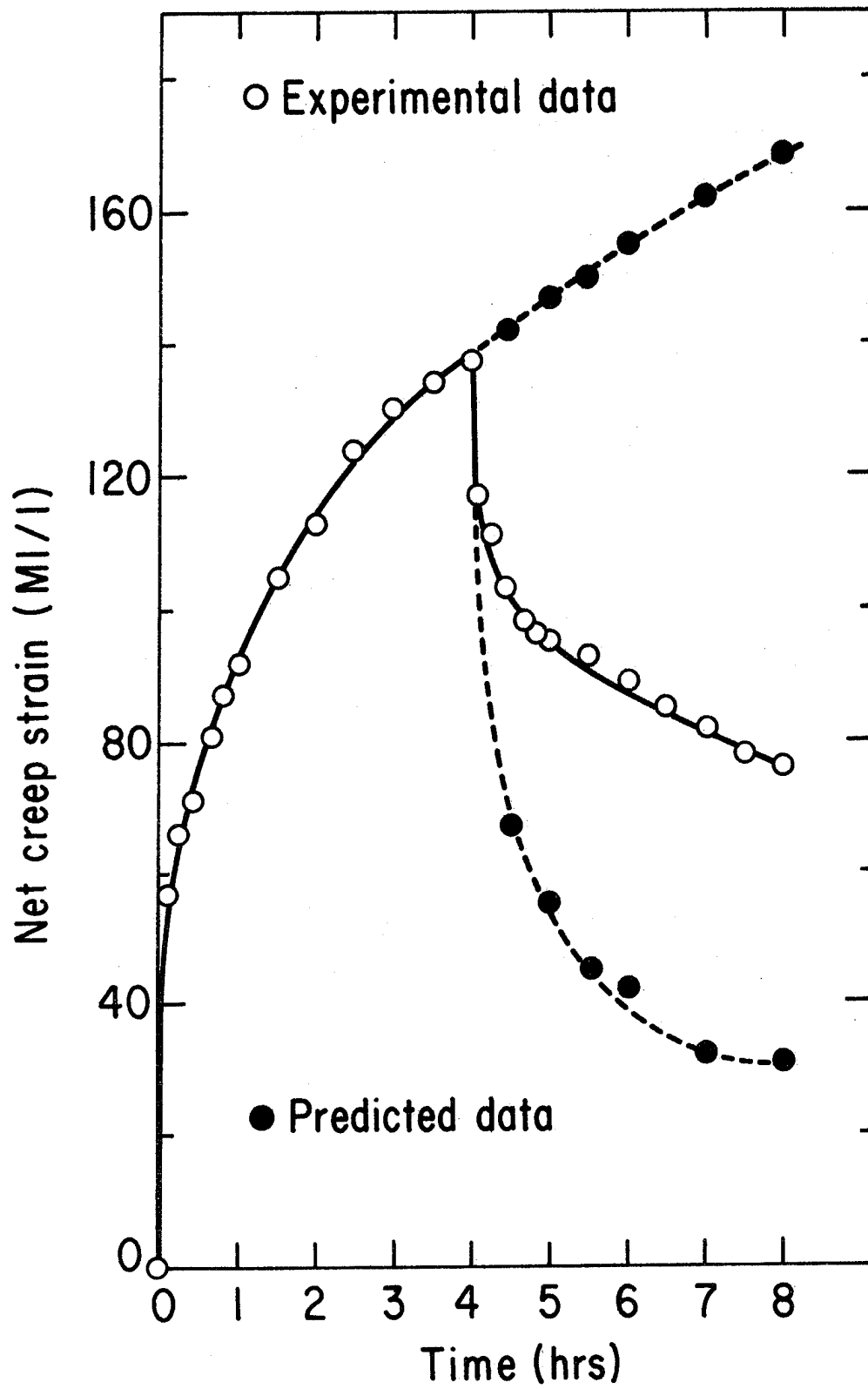


Figure 18. Creep-Recovery Curve (Stress = 300 psi, Temperature = 128° F, Humidity = 34 %)

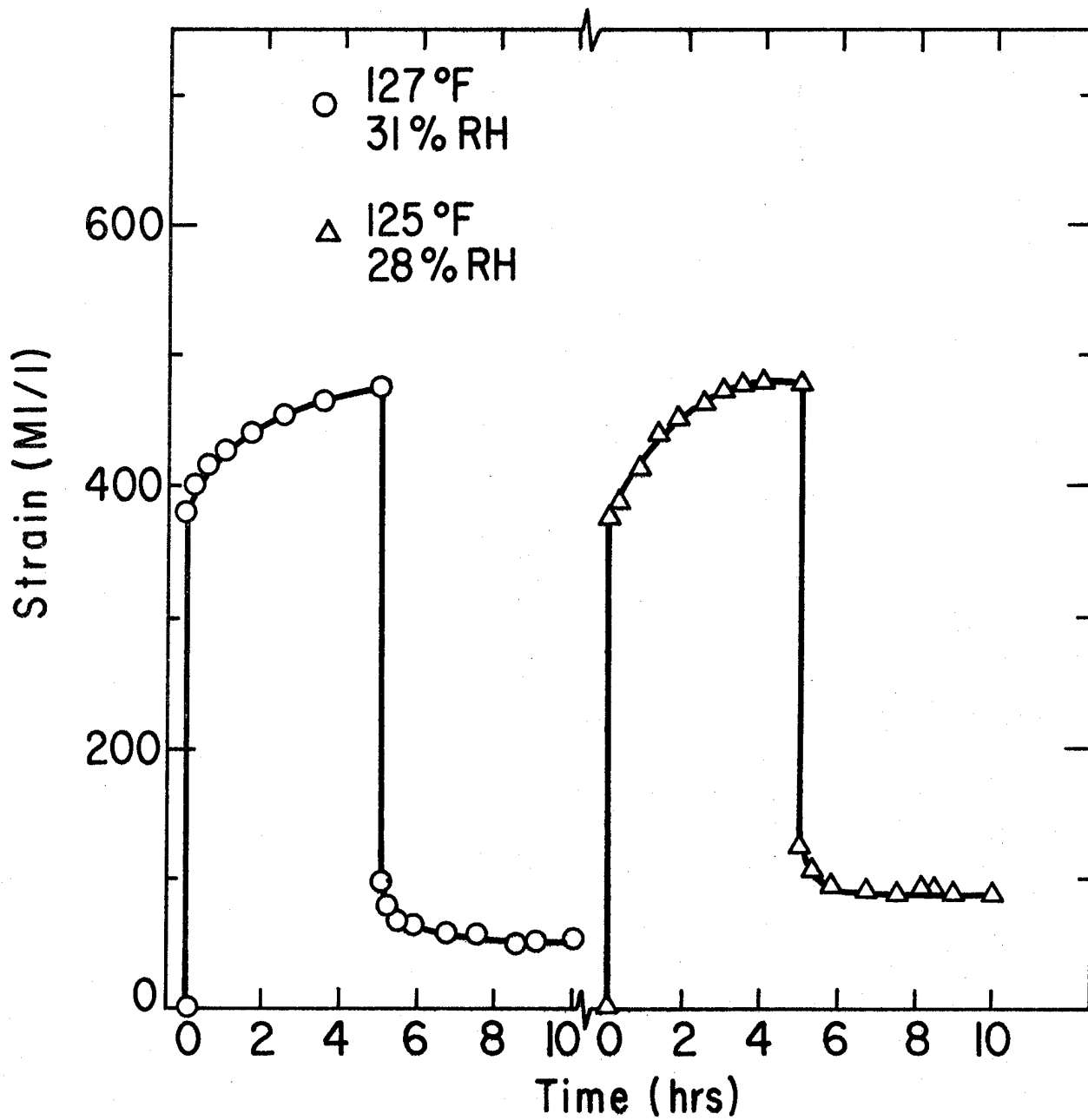


Figure 19. Repeated Creep and Recovery Behavior (Stress = 300 psi)

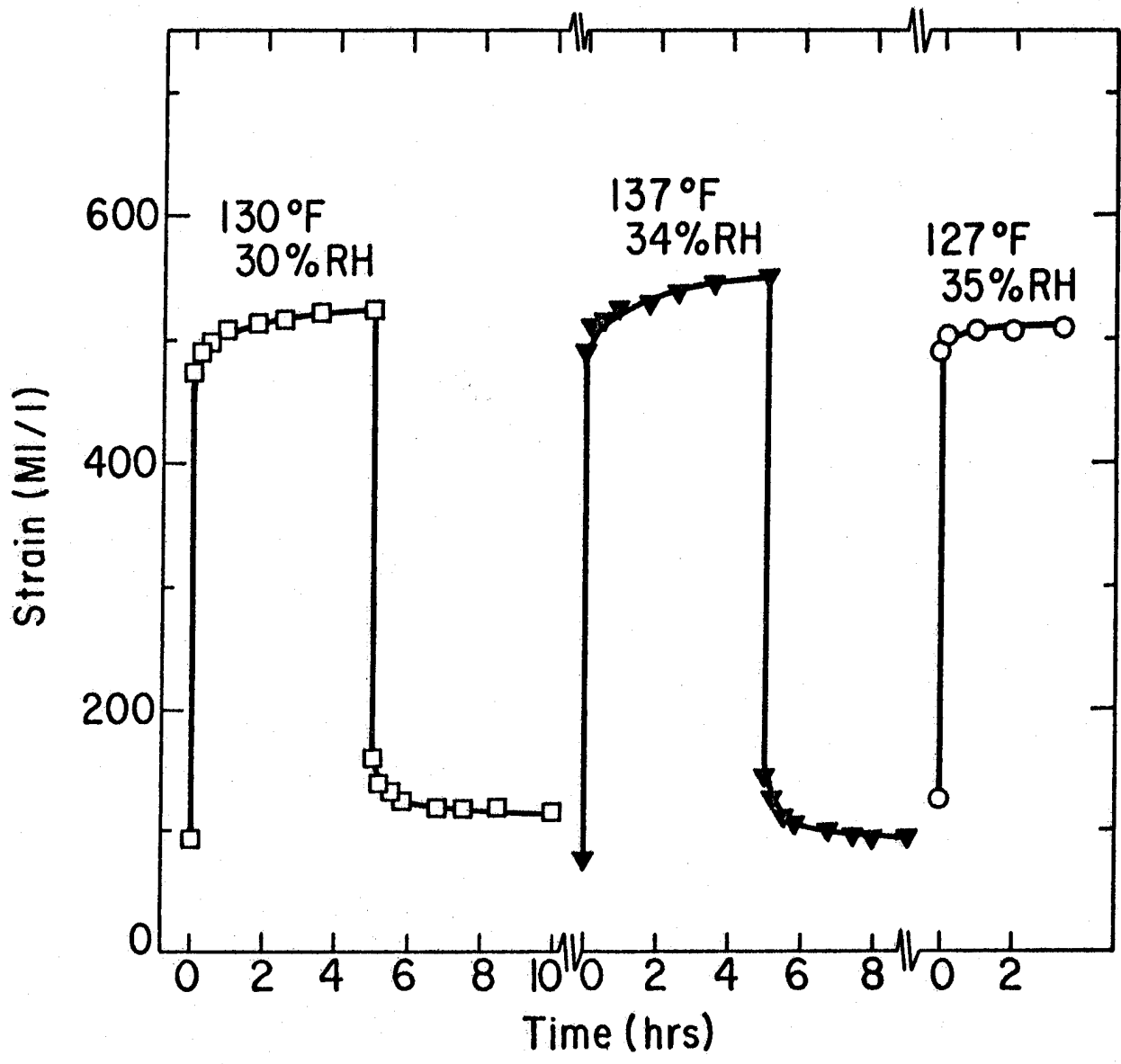


Figure 20. Repeated Creep and Recovery Behavior  
 (Continued from Figure 19)

### Section III

#### EFFECT OF INITIAL TRANSIENTS IN RELAXATION AND CREEP TESTS

For a linear viscoelastic bar, the relation between axial stress  $\sigma$  and strain  $\epsilon$  can be written in terms of the Boltzmann superposition integral [1],

$$\sigma = \int_0^t E(t - \tau) \frac{d\epsilon}{d\tau} d\tau \quad (10)$$

where the "relaxation modulus"  $E(t)$  is defined as the stress response to a unit strain input,

$$\epsilon = \begin{cases} 0 & : t < 0 \\ 1 & : t > 0 \end{cases} \quad (11)$$

The inverse of Equation (10) is written as

$$\epsilon = \int_0^t D(t - \tau) \frac{d\sigma}{d\tau} d\tau \quad (12)$$

where  $D(t)$  is the "creep compliance," and is defined as the strain response to a unit stress input,

$$\sigma = \begin{cases} 0 & : t < 0 \\ 1 & : t > 0 \end{cases} \quad (13)$$

The experimental problem is to measure either  $E(t)$  or  $D(t)$  for various fiber orientations, so as to be able to evaluate principle moduli or compliances. However, in practice, the axial strain or stress cannot be applied instantaneously, and one must wait a certain period of time following application of the prescribed quantity before the response can

be assumed as  $E(t)$  or  $D(t)$ . The usual rule-of-thumb is to wait ten times the transient strain period in the relaxation test and ten times the transient stress period in the creep test.

However, the authors are not aware of any published analysis which shows how conservative or nonconservative this particular waiting period is. The effect of the initial transients will be estimated here for both relaxation and creep tests using typical viscoelastic properties. Inertia effects will be neglected.

Consider the relaxation test first, and suppose that the actual prescribed strain history is a ramp function:

$$\epsilon(t) = \begin{cases} 0 & : t < 0 \\ \frac{t}{t_R} \epsilon_s & : 0 < t < t_R \\ \epsilon_s & : t > t_R \end{cases} \quad (14)$$

where  $\epsilon_s$  is a constant. Substitute this strain into stress (10) and find for  $t > t_R$ :

$$\begin{aligned} \sigma &= \int_0^{t_R} E(t - \tau) \frac{\epsilon_s}{t_R} d\tau + \int_{t_R}^t E(t - \tau) \frac{d\epsilon}{d\tau} d\tau \\ &= \frac{\epsilon_s}{t_R} \int_0^{t_R} E(t - \tau) d\tau \end{aligned} \quad (15)$$

Let  $t - \tau = u$  and  $-d\tau = du$  to find

$$E_R(t) \equiv \frac{\sigma}{\epsilon_s} = \frac{1}{t_R} \int_{t - t_R}^t E(u) du \quad (16)$$

The function  $E_R(t)$  will essentially equal the relaxation modulus at sufficiently long times; we are interested in finding out how much time is necessary by using a typical relaxation modulus. It has been

found that the modified power law [3],

$$E(t) = E_e + \frac{E_g - E_e}{\left[1 + \frac{t}{\tau_1}\right]^n} \quad (17)$$

where

$$\begin{aligned} n &= \text{constant (typically, } 0 < n < 0.5) \\ E_e &= E(\infty) = \text{equilibrium modulus} \\ E_g &= E(0) = \text{glassy modulus} \\ \tau_1 &= \text{time constant} \end{aligned}$$

is a good approximation to polymer data. Equation (16) can now be analytically integrated. For the particular case in which  $t_R/\tau_1 \gg 1$ , corresponding to the use of the modulus

$$E(t) = E_e + \frac{E_g - E_e}{\left(\frac{t}{\tau_1}\right)^n} \quad (18)$$

we find the modulus ratio  $R_E$  for  $t \geq t_R$ :

$$R_E(t) \equiv \frac{E_R(t) - E_e}{E(t) - E_e} = \frac{1}{1-n} \Psi \left[ 1 - \left( \frac{\Psi - 1}{\Psi} \right)^{1-n} \right] \quad (19)$$

where

$$\Psi \equiv t/t_R \quad (20)$$

This ratio is plotted in Figure 21. It is seen that  $R_E$  is within 5% of unity if  $t/t_R \geq 5$ . In turn, the ratio of immediate interest,  $E_R(t)/E(t)$ , is even closer to unity since one can easily show that,

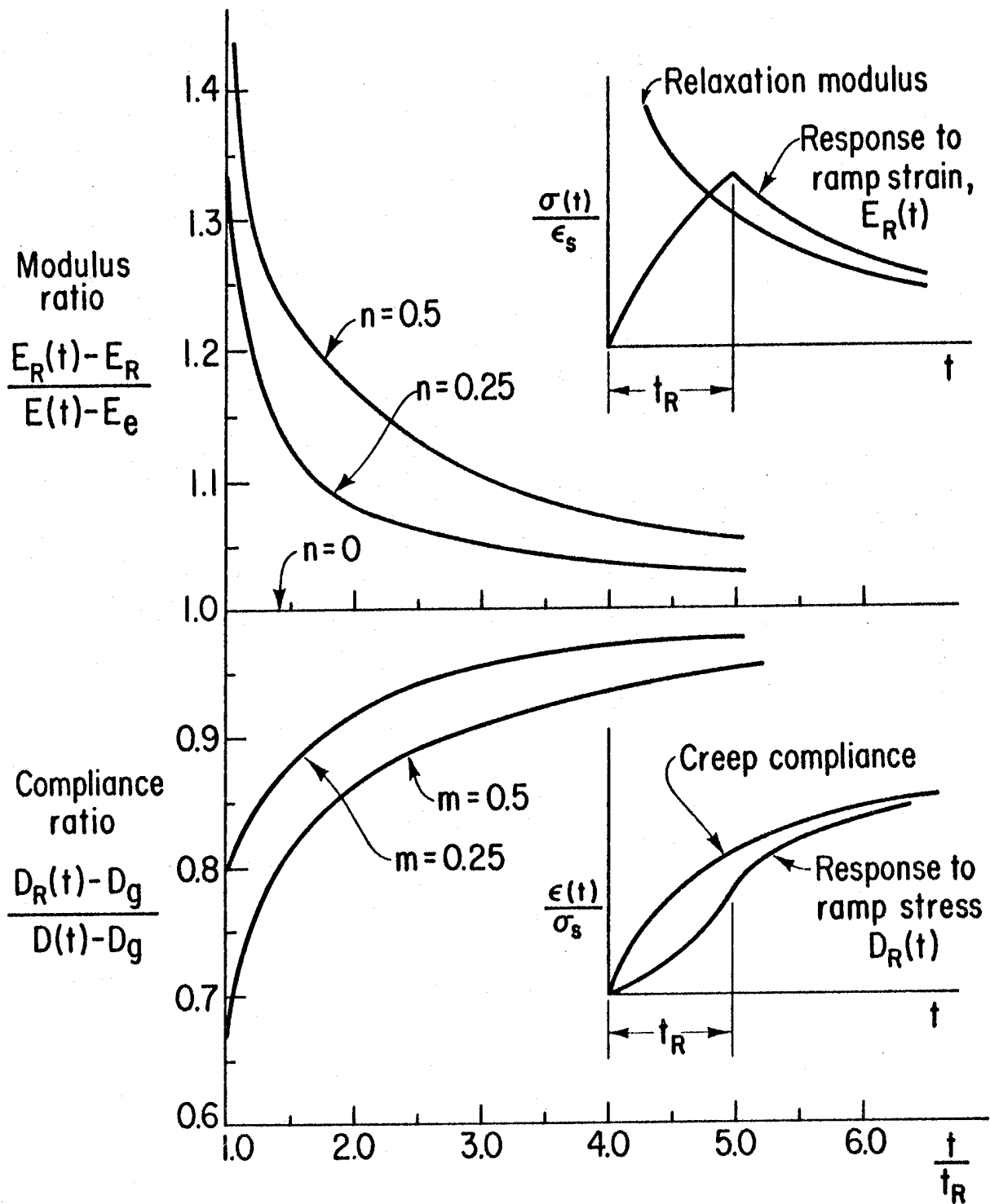


Figure 21. Comparison of Step and Ramp Responses for Relaxation and Creep Tests

$$1 \leq \frac{E_R(t)}{E(t)} \leq R_E(t) \quad (21)$$

Note also that for  $t \geq t_R$ ,  $R_E(t_R)$  is the maximum ratio and is equal to  $1/(1-n)$ . It is also easy to show that when the restriction  $t_R/\tau_1 \gg 1$  is removed, values of  $R_E(t)$  are even closer to unity than those shown in Figure 21.

The effect of initial transient stress in the creep test can be estimated in a similar manner. We assume the ramp stress history,

$$\sigma(t) = \begin{cases} 0 & : t < 0 \\ \frac{t}{t_R} \sigma_s & : 0 < t < t_R \\ \sigma_s & : t > t_R \end{cases} \quad (22)$$

Also the form

$$D(t) = D_g + \frac{(D_e - D_g)(t/\tau_o)^m}{(1 + t/\tau_o)^m} \quad (23)$$

is typical, with

- $m = \text{constant (typically, } 0 < m < 0.5)$
- $D_e = D(\infty) = \text{equilibrium compliance}$
- $D_g = D(0) = \text{glassy compliance}$
- $\tau_o = \text{time constant}$

If  $t/\tau_o \ll 1$ ,  $D(t)$  has the power-law form used earlier in this report,

$$D(t) = D_g + (D_e - D_g)(t/\tau_o)^m \quad (24)$$

Substitution of Equations (22) and (24) into (12) yields for  $t/t_R \geq 1$ :

$$R_D(t) \equiv \frac{D_R(t) - D_g}{D(t) - D_g} = \frac{1}{1+m} \Psi \left[ 1 - \left( \frac{\Psi - 1}{\Psi} \right)^{1+m} \right] \quad (25)$$

where  $\Psi \equiv t/t_R$ , which is plotted in Figure 21. It is to be noted that

$$R_D(t) \leq \frac{D_R(t)}{D(t)} \leq 1 \quad (26)$$

Also, the minimum value of  $R_D$  for  $t \geq t_R$  occurs when  $t = t_R$  and is  $R_D(t_R) = 1/(1+m)$ . Similarly, just as before,  $R_D(t)$  is closer to unity than (25) if  $t/\tau_0$  is not neglected in Equation (23).

On the basis of the above remarks, and the data in Figure 21, we conclude for  $0 \leq n \leq 0.5$  and for  $0 \leq m \leq 0.5$  that the initial transient in both tests will produce less than 5% error in  $E(t)$  and  $D(t)$  if  $t > 5t_R$  (i.e. the ramp response can be assumed essentially equal to these functions for times greater than five times the initial loading period).

All of the data reported in Section II was obtained at times for which the initial transient is negligible.

#### ACKNOWLEDGEMENT

The authors are indebted to Professor E. O. Stitz for providing valuable assistance in the experimental phase of the research program.

#### REFERENCES

1. J. C. Halpin, "Introduction to Viscoelasticity" in Composite Materials Workshop, S.W. Tsai, J.C. Halpin and N.J. Pagano, eds, Technomic Publishing Co., Stamford, Conn. (1967).
2. J.W. Dally and W.F. Riley, Experimental Stress Analysis, McGraw-Hill Book Co., New York (1965).
3. M.L. Williams, "Structural Analysis of Viscoelastic Materials," AIAA J., Vol. 2 (May 1964).
4. W.H. Haslett and F.J. McGarry, "Shrinkage Stresses in Glass Filament-Resin Systems," Modern Plastics, (December 1962).
5. J.J. Hermans, "The Elastic Properties of Fiber Reinforced Materials When the Fibers are Aligned," Koninkl. Nederl, Akademie Van Wetenschappen-Amsterdam, B, Vol. 70 (1967).
6. R. Hill, "Elastic Properties of Reinforced Solids: Some Theoretical Principles," J. Mech. Phys. Solids, Vol. 11, (September 1963).

## APPENDIX

### Stress Analysis of Viscoelastic Composite Materials \*

R. A. Schapery

#### ABSTRACT

This paper deals with foundations and methods of linear viscoelastic analysis of anisotropic composites, including temperature effects. Earlier work on mathematical and thermodynamic bases of constitutive equations and the correspondence principle is reviewed. This principle is then used to relate effective viscoelastic moduli for composites to constituent properties. It is shown that all results from elasticity theory, including upper and lower bounds on moduli, are applicable to viscoelastic materials. Three approximate methods of viscoelastic analysis are reviewed and applied to three problems: pressurization of an orthotropic cylinder, cooling of a glass fiber embedded in resin, and upper and lower bounds on operational and relaxation shear moduli for fiber-reinforced composites. Although realistic material properties are used, these methods enable explicit solutions to be derived without difficulty.

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13. ABSTRACT Tensile creep and recovery response of unidirectional, glass fiber-epoxy specimens <sup>was</sup> studied. In the series of tests reported herein, all loading <sup>is</sup> normal to the fiber axis. The glass transition temperature, T <sub>g</sub> , for the epoxy is determined from thermal expansion measurements normal to the fibers, and the temperature at which the material starts to appreciably soften is found by measuring creep in the presence of a slowly increasing temperature. Linearity of creep and recovery behavior is then studied at room temperature and near T <sub>g</sub> . The material <sup>was</sup> studied at room temperature is found to be approximately linearly viscoelastic out to fracture, but considerable nonlinearity in the form of nonrecoverable deformation appears at temperatures in the neighborhood of T <sub>g</sub> . In a concluding analytical study the influence of initial transients in creep and relaxation tests <sup>is</sup> estimated. This information provides a useful guideline for reducing creep and relaxation test data.			

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