In-Plane Shear Stress Relaxation Modulus of a Carbon-Epoxy Composite

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SUMMARY: In this paper, a model for the in-plane shear stress relaxation modulus of a carbon-epoxy prepreg composite material is developed. Due to their simplicity, creep tests are used to determine the creep compliances of the material at various temperatures, which are transformed to the stress relaxation moduli through the Hopkins-Hamming method. The results are then fitted into a time shift function and a Prony series. It is shown that the material behaves linear viscoelastically and significant creep/stress relaxation occurs at temperatures higher than 120°C. The model developed is validated through test simulation.

KEYWORDS: Stress relaxation modulus, Viscoelastic material model, Creep test, Carbon-epoxy composites.

INTRODUCTION

Advanced polymer composites are made by reinforcing polymer matrices with fibrous reinforcements. The resulting behavior of the composites then depends upon the property and orientation of the fiber, and the property of the matrix. It is well known that the polymer matrix exhibits viscoelasticity, especially when it is not fully cross-linked, or is loaded at a high temperature or in a humid environment. The viscoelasticity of polymer composites may have significant impact on the design, manufacture and service life of composite structures. For example, the mechanical response of a composite part can be significantly viscoelastic during the later stage of the curing process or when it is reheated to a high temperature for assembly by bonding. Models for stress relaxation modulus need to be established to correctly predict the part distortion during these processes using a viscoelastic stress analysis.

There is relatively a large amount of reported work on viscoelastic analysis of polymer composites; see for example [1-4]. As a result, anisotropic viscoelastic analysis of composite materials is now supported by some commercial finite element packages, such as MSC.MARC. Considerable attempts have also been made to determine the viscoelastic composite material models to be used for the analysis both analytically and experimentally.

For example, White and Hartman conducted experiments to determine the stress relaxation modulus of 3501-6 epoxy resin during cure using DMA (Dynamic Mechanical Analyser) [5]. Also using DMA, Nam and Seferis investigated the viscoelastic behavior of a phenolic-carbon composite material through glass transition and degradation reaction processes in temperature up to 400°C [6]. A comprehensive review on micromechanics models for evaluating viscoelastic properties of composite materials can be found in Reference [7].

In the present work, in-plane shear viscoelastic response of a carbon-epoxy composite material is investigated by isothermal tensile creep tests. The results are processed to fit into a time shift function and a master Prony series representing the in-plane shear stress relaxation moduli of the composites at temperatures up to 150°C. It is shown that the model developed can be used as an input to MSC.MARC to predict the in-plane shear viscoelastic behavior of the composite laminates investigated with reasonably good accuracy.

EXPERIMENTAL WORK

Ideally stress relaxation tests should be used to measure the stress relaxation moduli. However, a stress relaxation test may be more difficult to conduct than a creep test. Therefore, creep tests were used in the present work. The creep compliances determined were then transferred to the relaxation moduli using the Hopkins-Hamming method [8].

Material and Test Apparatus

The material tested was Hexel F593-18 plain weave pre-preg laminates laid-up as $[(45/\text{-}45)_6]_S$ with each ply being 0.208mm thick. The laminate was cut into 250mm long and 25mm wide test specimens. Two 90° rosette strain gauges were bonded to each specimen to measure the longitudinal and transverse strains during the creep test and a thermocouple was placed next to the strain gages to record the temperature history that the specimen experienced. Uniaxial tension was applied by dead weights using the apparatus described below.

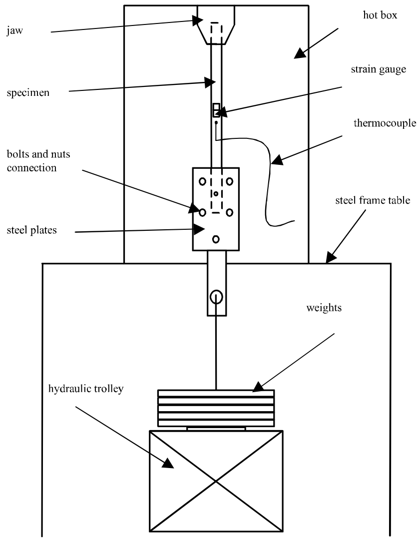

Fig. 1 Schematic diagram of creep test set-up

The creep tests were conducted in a hot box sitting on a steel frame table with a setup sketched in Fig. 1. A mechanical jaw attached to a universal joint fixed to the top of the hot box was used to hold the top end of the specimen. To avoid applying a concentrated load on the specimen, a pair of steel plates connected by five bolts and nuts was clamped on the bottom end of the specimen. Weights were then hung onto the clamping plates to create a uniformly distributed loading along the width of the specimen during test. A hydraulic trolley was placed under the weights to allow almost instantaneous loading when the hydraulic oil pressure was suddenly released.

Experimental Procedure

Since the tests were conducted at relatively high temperatures, a high temperature adhesive for installing strain gauges to the specimen was used. This adhesive is required to cure at three temperature stages (80°C for one hour, 130°C and 150°C for two hours respectively) under 0.2MPa pressure. Such a curing process produced an internal residual stress in the adhesive, and consequently an unwanted adhesive creep during the test. To eliminate the adhesive creep, an annealing process was introduced into the experimental procedure after the strain gauge curing process.

Table 1 Temperature, loading and time of tests

Process		Number of tests	Temperature (°C)	Keeping time (hour)	Applied stress (MPa)	Loading time (hour)
Annealing		all	150	2	0	NA
Creep test	Adhesive	2	150	4	0	NA
	Various temperature	2-3	100	1	10	2
		2-3	120	1	10	2
		2-3	130	1	10	2
		2-3	140	1	10	2
		2-3	150	1	10	2
	Various load	2-3	140	1	6.5	2
		2-3	140	1	10	2
		2-3	140	1	13.8	2

To perform an isothermal creep test, the specimen was preheated by the hot box to the desired temperatures (Table 1) without any loading, and then kept at this temperature for an hour to allow the heat to be transferred to the center of the specimen and to achieve an even temperature distribution in the specimen. Without changing the temperature, the hydraulic trolley was suddenly released and a constant load was then applied onto the specimen for 2 hours and the creep strains were recorded. Details of each of the tests conducted are listed in Table 1.

RESULTS AND DISCUSSION

Creep of Adhesive

Two unloaded tests were conducted to verify the effectiveness of the annealing process in eliminating the adhesive creep during experiment. The specimens were hung in the hot box and heated to 150°C without any loading and kept at this temperature for 4 hours.

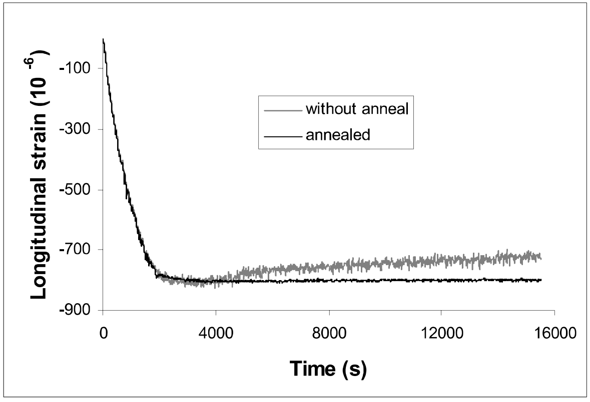

Fig. 2 Adhesive creep strains

Fig. 2 shows the transient strains of the annealed and unannealed specimens respectively. The strain signals were zeroed at room temperature. Due to thermal straining, the longitudinal strain signals shown in Fig. 2 decrease rapidly at the beginning. Once the temperature in the specimen reaches and remains at 150°, the strains are expected to remain unchanged with time if the adhesive does not creep. Such a result is only observed for the curve in dark color in Fig. 2, which represents the creep strain of the annealed specimen. However, the creep strain of the unannealed specimen increases with time, indicating that the adhesive creep occurred during the test. Therefore in subsequent tests the specimen was always annealed after curing the strain gauge.

Creep under Different Loading Levels

A group of tests was conducted to determine if the viscoelasticity of the material was linear within the temperature range considered. The temperature used for these tests was 140°C and the applied tensile stress levels were set to about 6.5MPa, 10MPa and 13.8MPa respectively.

Shear strain curves obtained at the three different stress levels are plotted in Fig. 3. The level of strain increases with the applied stress. Strains measured at the beginning (time = 0 sec) are the elastic strains representing the instantaneous response of the specimens to the applied stresses. Strains after the initial point are the creep strains and are shown to increase with time under all the three loading levels tested.

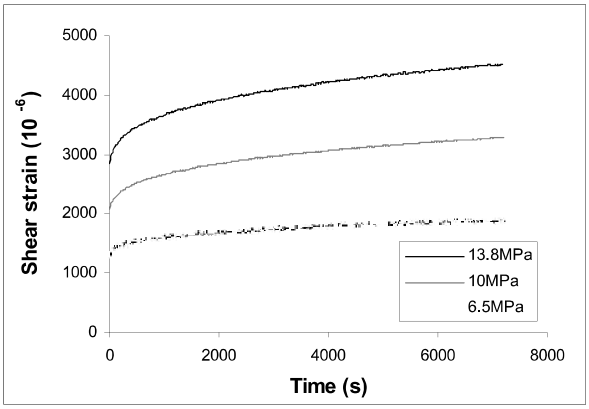

Fig. 3 Strains for laminates under various loads at 140°C

Fig. 4 shows the isochronal stress-strain curves for t=0, 1 hour and two hours respectively. The strains are approximately linearly related to the applied stresses as the isochronal stress-strain curves are almost straight. Therefore the material investigated can be assumed to be linear viscoelastic.

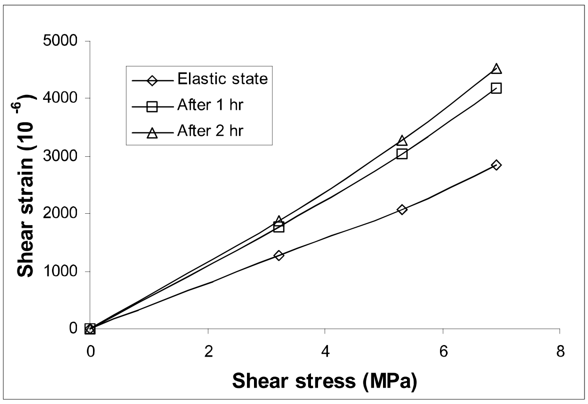

Fig. 4 Isochronal stress-strain curves for laminates loaded at 140°C

Creep at Various Temperatures

More creep tests were then conducted under a tensile stress of 10MPa at five different temperatures: 100°C, 120°C, 130°C, 140°C and 150°C respectively. The longitudinal and transverse strains recorded during the tests were converted to the in-plane shear strain using the procedure documented in ASTM-D3518. The results are shown in Fig. 5 and are averaged over the two or three tests conducted for each test condition. Both the instantaneous elastic shear strain and creep shear strain increase with temperature. Within a time span of about two hours, the material exhibits less than 20% creep strain when the temperature is lower than 120°C and the creep rate gradually increases with the temperature.

Very significant creep (creep strain is 110% of elastic strain) occurs when temperature goes up to 150°C.

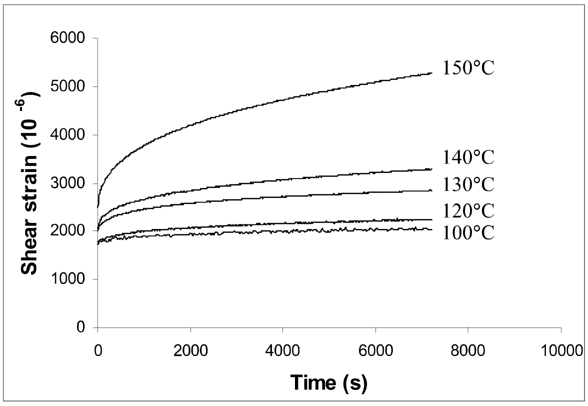

Fig. 5 Transient shear strains at various temperatures

It is straightforward to calculate the creep compliance C(t) from the experimentally determined transient strains $\gamma(t)$ using the following equation:

$$C(t) = \frac{\gamma(t)}{\tau} \tag{1}$$

where τ is the shear stress applied.

The creep compliances obtained are shown in Fig. 6.

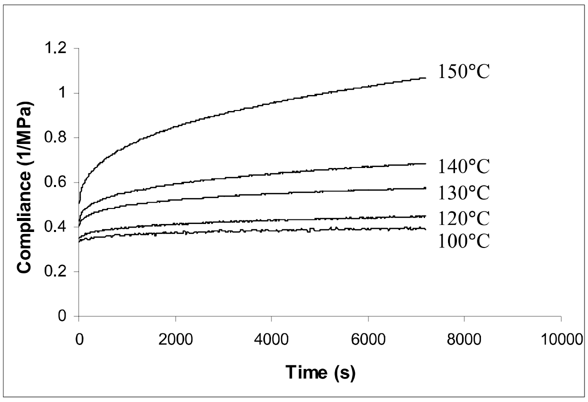

Fig. 6 Creep compliance for various temperatures

MODEL FOR STRESS RELAXATION MODULUS

Theoretical Background

For a linear viscoelastic material subject to both thermal and mechanical loads, the stress relaxation modulus can be described by the hereditary integral model, which is an integral expression of generalized forms of the Maxwell and Kelvin model [9]. It defines the time dependant material behavior in a relative simple form, which is often referred as the Prony series:

$$G(t) = G_0 + \sum_{n=1}^{N} G_n[\exp(-t/\lambda_n)] \qquad (2)$$

where, G(t) is the time dependant stress relaxation moduli, G_0 is long term stress relaxation moduli, G_n and λ_n are the amplitudes and time constants for the Prony series respectively.

For a thermal-rheologically simple material, the effect of temperature on the relaxation moduli can be introduced through the following the time transformation:

$$\xi = \int_0^t \frac{dt'}{\alpha(T, t')} \qquad (3)$$

where, t is time, ξ is the reduced time, and α is the shift function which is a function of temperature T.

Model for In-plane Shear Stress Relaxation Modulus

In the present work, the above in-plane shear creep compliance was transformed to the stress relaxation modulus G(t), through the following transformation:

$$\int_0^t G(\tau)\, C(t-\tau)\, d\tau = t \qquad (4)$$

A numerical solution of Eqn. 4 for tabular data was given by Hopkins and Hamming as:

$$G(t_{n+1/2}) = \frac{t_{n+1} - \sum_{i=0}^{n-1} G(t_{i+1/2})[f(t_{n+1} - t_i) - f(t_{n+1} - t_{i+1})}{f(t_{n+1} - t_n)} \qquad (5)$$

in which

$$f(t) = \int_0^t C(\tau) d\tau \qquad (6)$$

A FORTRAN program was written to read the compliance data from the experimental results and then to solve Eqn. 5 to transform the compliances into the stress relaxation moduli. The results are plotted in Fig. 7.

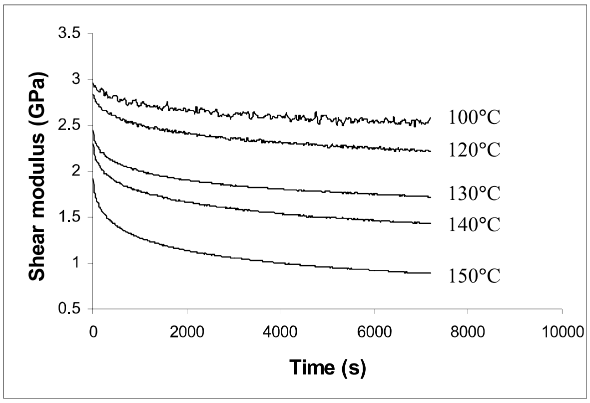

Fig. 7 Stress relaxation moduli for various temperatures

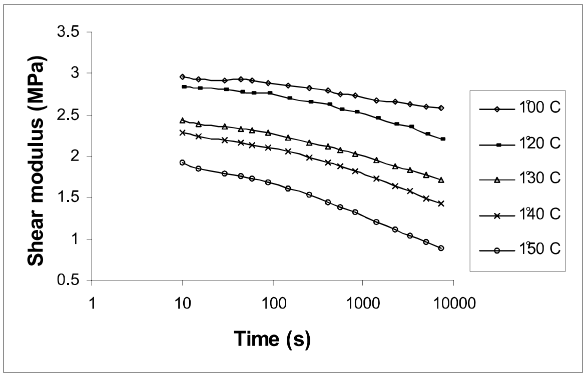

Fig. 8 Stress relaxation moduli in logarithmic time scale

Assuming the material to be thermal-rheologically simple, the stress relaxation curves at various temperatures exhibit an approximate translation shift along a logarithmic time axis. The individual stress relaxation modulus at each temperature (Fig. 7) was then re-plotted in a logarithmic time scale in Fig. 8. Using the shear modulus curve of 100°C as a reference, the other curves were shifted manually along the time axis to the right to form a continuous master curve shown in Fig. 9. The relationship between temperature and the time shifting factor was plotted in Fig. 10 and the shift function α was determined by fitting the points into the following equation:

$$\alpha = 11.672\,x^2 - 19.652x + 7.786 \qquad (7)$$

where, $x = T/100$ and T is temperature.

A nonlinear regression analysis was then conducted to fit the master curve into a five-term Prony series as shown in Fig. 9. Model constants obtained by the fitting are listed in Table 2.

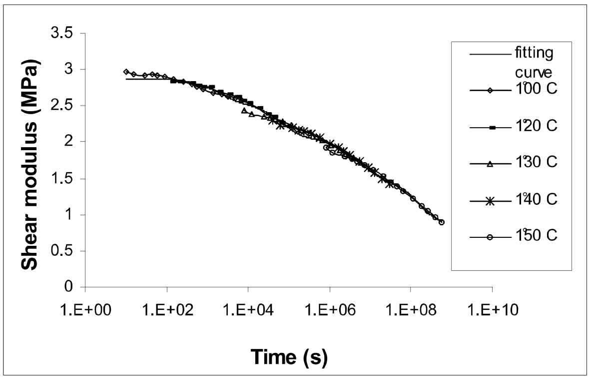

Fig. 9 Master curve of stress relaxation moduli

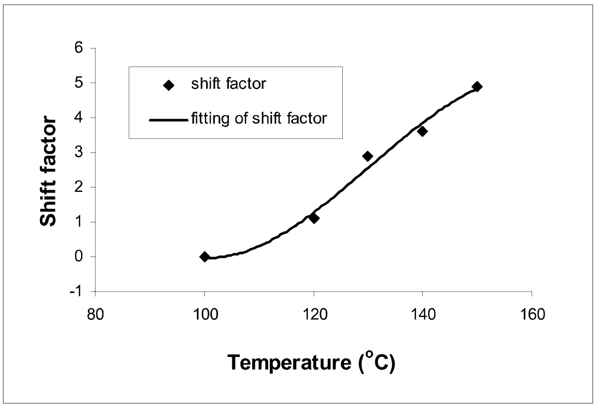

Fig. 10 Time shift factor

Table 2 Relaxation data for the Prony series

n	G_n (GPa)	λ_n (s)
0	0.89592	
1	0.30402	2050.22008
2	0.49028	73252.7885
3	0.50648	4.90159591×10^6
4	0.67819	1.55861669×10^8

Validation

A non-isothermal creep test was conducted to validate the stress relaxation modulus model developed. The specimen used for the validation test was similar to those used for the tests to develop the model, except that a thermocouple was embedded in the middle layer of the composite laminate. Two specimens were used for the test: one was for creep test under an applied stress of 10MPa and the other was hung next to the creep specimen in the hot box for recording the thermal strain which was later deducted from the creep strain recorded. The temperature program applied in the creep test was a hold at 100°C for half an hour, a ramp up to 140°C, and a hold for another half an hour.

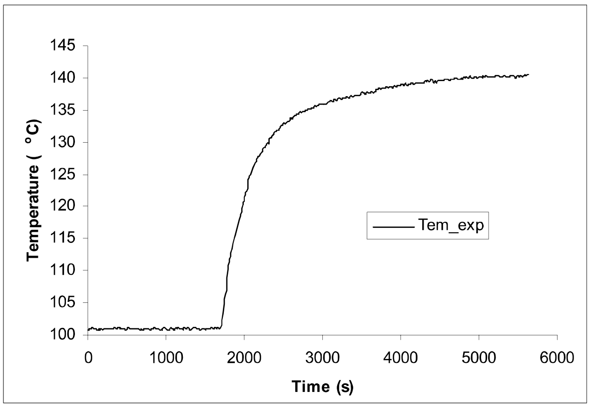

Fig. 11 Temperature program during validation creep test

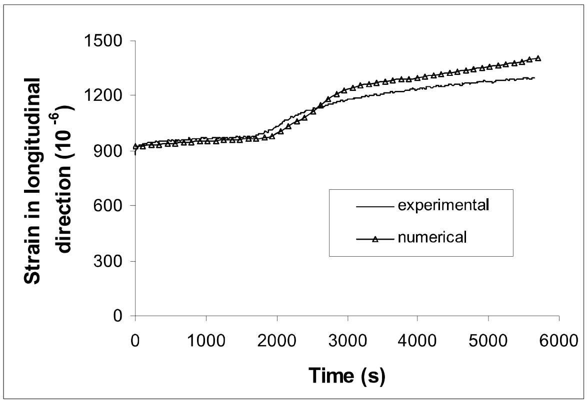

Fig. 12 Comparison of numerical and experimental results

MSC.MARC was used to perform the finite element modeling of the creep test. Due to symmetry, only a quarter of the specimen was included in the model with 20 and 200 elements along its width and length respectively. The temperature applied in the simulation was taken from the readings of the thermocouple embedded in the specimen (see Fig. 11) and a uniformly distributed load was applied instantly to the model and kept constant thereafter.

Fig. 12 compares the numerically predicted and experimentally measured transient strains during the non-isothermal creep test. It shows that there exists a reasonably good agreement between the predicted and experimental strains. The predicted strains are slightly higher than those experimentally measured at 140°C, because the time shift function fitted at 140°C is higher than measured. (see Fig. 10).

CONCLUSION

The in-plane shear relaxation modulus of polymer composites can be measured in a tensile creep test using a simple set-up with reasonable accuracy and consistence. However, attention has to be paid to creep of the adhesive used to bond the strain gauges onto the specimen. Creep was found in the adhesive during initial creep tests. Hence, an annealing process needs to be introduced into the experiment.

The in-plane shear response of ±45^o Hexcel F593-18 prepreg laminates at temperatures up to 150^{o}C is found to be linear viscoelastic. The creep rate is relatively low for temperatures below 120^{o}C and becomes more significant at higher temperatures. At 150^{o}C, the creep strain after two hours exceeds the elastic strain.

The in-plane shear viscoelastic behavior of the material investigated can be expressed by a time shift function and a Prony series with four exponential terms. Numerical simulation of the non-isothermal validation creep test confirms that the material model established in this study predicts a viscoelastic response which is in reasonably good agreement with the experimental one. The model also demonstrates the ability to describe creep behavior under non-isothermal temperature history.

ACKNOWLEDGEMENTS

The authors wish to thank Mr J. Traintafillou, Mr R. Sweeting and Mr M. Crossthwaite of CRC-ACS for their help in setting up the experimental rig and manufacturing the test specimens, and Mr R. Paton of CRC-ACS for his useful comments during the preparation of the manuscript.

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