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DYNAMIC MECHANICAL EVALUATION OF A RUBBER TOUGHENED GRAPHITE–EPOXY COMPOSITE WITH APPLICATION FOR LAMINATE ANALYSIS *

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ABSTRACT

The dynamic mechanical properties of Hexcel F-155 rubber toughened epoxy was evaluated as a pure matrix and as a graphite-fiber composite. The materials were evaluated using a Dynastat mechanical analyzer at from −100°C to 200°C to provide temperature dependent elastic constants for composite laminate analysis. The pure matrix material showed a secondary transition, which has an effect on the mechanical properties of the composite laminate. The experimental results for complex modulus and thermal expansion of the pure matrix and unidirectional laminate were used to provide direct and indirect temperature dependent properties through micromechanics to predict properties of a composite laminate.

INTRODUCTION

The increasing use of fiber re-enforced polymeric composites for precise space structures has necessitated the need for improved knowledge of the thermal–mechanical properties of composite materials. Prediction of the dimensional stability of the structures and compliance over a wide temperature range has become important. Traditional mechanical testing over a wide temperature range is both time consuming and expensive at the material selection stage of design. Traditional mechanical testing also gives no information concerning viscoelastic properties of the materials. Dynamic mechanical analysis of the pure resin and of the composite provides an efficient means to determine the temperature dependent viscoelastic properties. In this study, an IMASS Dynastat was used to study the viscoelastic behavior of a rubber toughened epoxy resin used in a graphite–epoxy composite to provide temperature dependent input parameters for a com-

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posite laminate model. The epoxy studied was Hexcel F-155, a rubber modified epoxy in which the elastomer is intrinsically incorporated into the epoxy matrix as a side chain. The fiber used in the composite was Celion 6000, a 231 GPa modulus PAN graphite fiber. The resin is a thermoset with a 121°C cure temperature, cured under vacuum and 100 lbf in^{-2} pressure for 4 h. The material was tested as a cured resin only, and as an 8 ply $[0]_s$ and $[0,90,\pm 45]_s$ composite. The unidirectional composite was tested in the [0] and [90] degree directions. Dynamic mechanical analysis was conducted at from −100°C to 200°C, or until failure. The resin and the composites were tested in tension with a 1 kg static load, with a 1 kg dynamic sinusoidal load. The dynamic loads frequencies ranged from 0.1 Hz to 100 Hz. Because of the high stiffness of the composite materials, the compliance of the Dynastat load frame was determined and used as a correction factor in calculating the complex modulus, and the in-phase and the out-phase modulus [1]. All composites have a 58% fiber volume. Both the resin and the composite thermal expansion were measured independently using a Harrop Dilatometer.

The basis of composite laminate analysis is that the properties of the individual lamina are well known, and knowing the lamina ply orientation, the mechanical properties of the composite laminate can be determined using the principles of solid mechanics [2]. Composite micromechanics makes use of the constituent material properties of the fiber and the matrix from the fabrication process to predict the mechanical properties of the lamina. For micromechanics to be effective, the properties of the resin, fiber and fiber–matrix interactions must be well documented [3,4].

Direct measurement of the resin and composite thermal expansion, and longitudinal and transverse modulus, as carried out in this study reduces the uncertainty in the prediction of composite mechanical properties from an analysis solely based on micromechanics.

ANALYSIS

A composite micromechanics analysis based on a mechanics of materials approach makes the assumption that the strains in the fiber and the matrix are the same. Overlaying this is the assumption that the fibers are evenly distributed throughout the matrix. This is a good assumption when the strains are in the direction of the fibers, but fails in the transverse direction. Present forms of micromechanics allow no consideration for the temperature dependent viscoelastic properties of the matrix.

For analysis of the elastic properties of a composite, macromechanics require the Young's tensile moduli, normal and transverse to the direction of strain; the shear modulus, and the major Poisson's ratio for the lamina. Micromechanics requires a much larger set of elastic constants for the fiber

TABLE 1

Micromechanic elastic constants

Matrix shear modulus	$G_m = \dfrac{E_m(T)}{2(1+\nu_m)}$
Fiber longitudinal	$G_{12} = G_{12} = \dfrac{G_m}{1 - k_f^{\frac{1}{2}}(1 - G_m/G_{f12})}$
Fiber transverse shear	$G_{23} = \dfrac{G_m}{1 - k_f^{\frac{1}{2}}(1 - G_m/G_{f23})}$
Longitudinal Poisson's ratio	$\nu_{12} = \nu_{13} = k_f \nu_{f12} + k_m \nu_m$
Transverse Poisson's ratio	$\nu_{23} = \dfrac{E_{22}(T)}{2G_{23}} - 1$

and the matrix to provide the reduced set of elastic properties for the lamina. They are E_{11}, E_{22}, G_{12}, G_{23}, and ν_{12} for the fiber and G_m, ν_m and fiber volume for the matrix.

The approach taken here is to reduce the uncertainty in micromechanics by directly measuring the temperature dependent longitudinal moduli. $E_{11}(T)$ was measured by testing a unidirectional laminate in the [0] direction. The transverse moduli, $E_{22}(T)$ was measured from a unidirectional

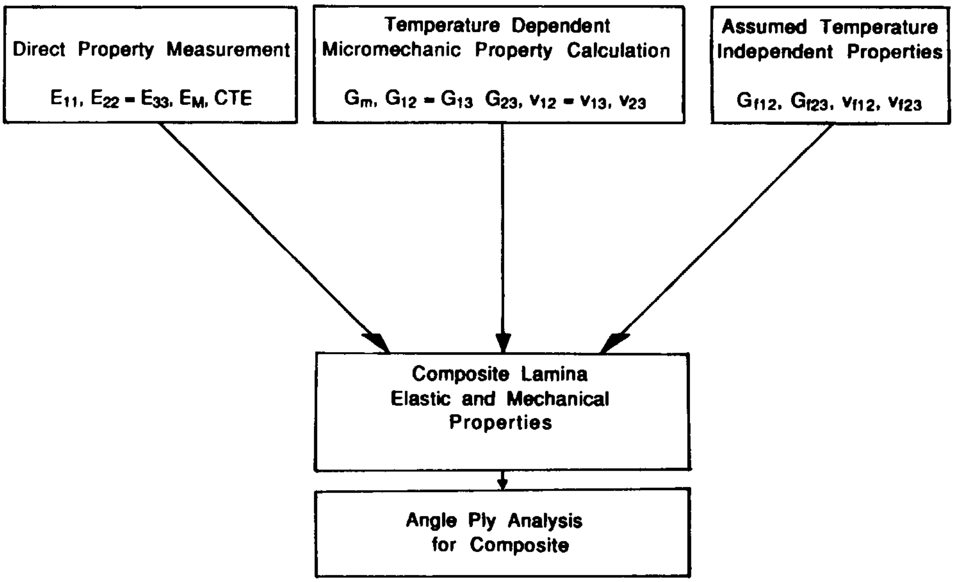

Fig 1. Use of dynamic mechanical analysis for direct and indirect temperature dependent lamina properties.

laminate in the [90] direction. The modulus of the matrix, $E_m(T)$, was similarly tested on a pure resin sample.

The elastic shear properties cannot be directly measured in the Dynastat. To develop temperature dependent shear properties, micromechanics was used in a variation of the approach used by Chamis [3]. The equations are presented in Table 1. The notation used for direction assumes that the unidirectional laminate is orthogonal. The longitudinal fiber direction is subscripted by 1. The subscript 2 denotes transverse in-plane and 3 denotes transverse out-of-plane. The subscript m denotes matrix and f denotes fiber properties.

Two sets of constants were assumed to be temperature independent in this analysis. The first is the Poisson's ratio of the matrix. This was done because no simple means was available for temperature dependent measurement. The Poisson's ratio and shear moduli of the fiber were also assumed to be independent of temperature and were taken from the data of manufacturers. A flow diagram is presented in Fig. 1 to summarize the approach taken. Once the temperature dependent elastic constants of the lamina are defined, macromechanical laminate analysis was used to calculate mechanical properties as a function of temperature.

EXPERIMENTAL

Figure 2 shows the complex, storage and loss modulus of the cured F-155 epoxy resin. Results are shown for a temperature range of from $-100\,°C$ to $50\,°C$ at 1 Hz and 10 Hz. Data from this experimental run were obtained only up to $50\,°C$ when the test sample broke. Independent samples of the pure resin were tested up to higher temperatures and incorporated into the laminate design. From $-70\,°C$ and higher temperatures the pure resin shows a weak dependence of storage modulus with temperature, with the loss modulus being independent. Below $-70\,°C$ the pure resin undergoes a secondary transition associated with the rubber phase. Below this temperature the elastomer cannot freely rotate. This secondary transition sets the lower temperature limit for which the material should be used.

Figure 3 shows the loss and storage modulus results for the unidirectional composite sample in the [0] fiber direction. In the fiber direction, the composite shows very little degradation in stiffness until $20\,°C$ lower than the resin cure temperature. Both the loss and the storage modulus shows the same shape curves with the rubber phase showing no effect on the overall stiffness. From micromechanics, the 20% increase in modulus of the resin from $-70\,°C$ to $-95\,°C$ would only produce a 0.3% change in the E_{11} modulus and one of 11.6% in the E_{22} and E_{33} modulus. The results for the complex modulus were used in the laminate analysis for temperature dependent E_{11}.

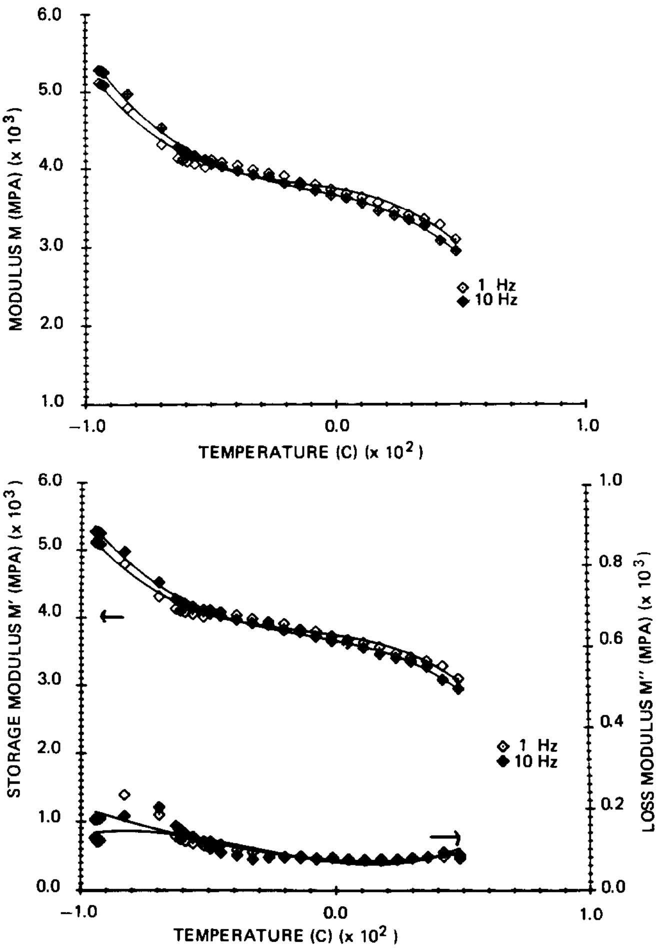

Fig. 2. Dynamic complex, storage and loss modulus of cured F-155 resin.

For verification of the Dynastat results, witness samples from the same composite panel were also tested in an Instron with a 9 in length specimen and 1 in extensometer gauge length. Instron measurements at room temperature averaged 115 GPa compared with 98 GPa from the Dynastat. The lower Dynastat measurements are the results of smaller test samples.

Figure 4 shows the loss and storage modulus in the [90] fiber direction. In the transverse direction the matrix carries directly the tensile load. In this case the transition of the rubber phase is observed. The loss modulus also shows a peak at the T_g of the resin. Results for the complex modulus were used for E_{22} and E_{33} in the laminate analysis.

Figure 5 shows the Dynastat results for the loss and storage modulus for a $[0,90,\pm 45]_s$ composite on the [0] direction. The transition for the elastomeric phase shows a small effect on the overall stiffness of the quasi-isotropic laminant. The peak is observed for the cure temperature T_g at 120 °C and a

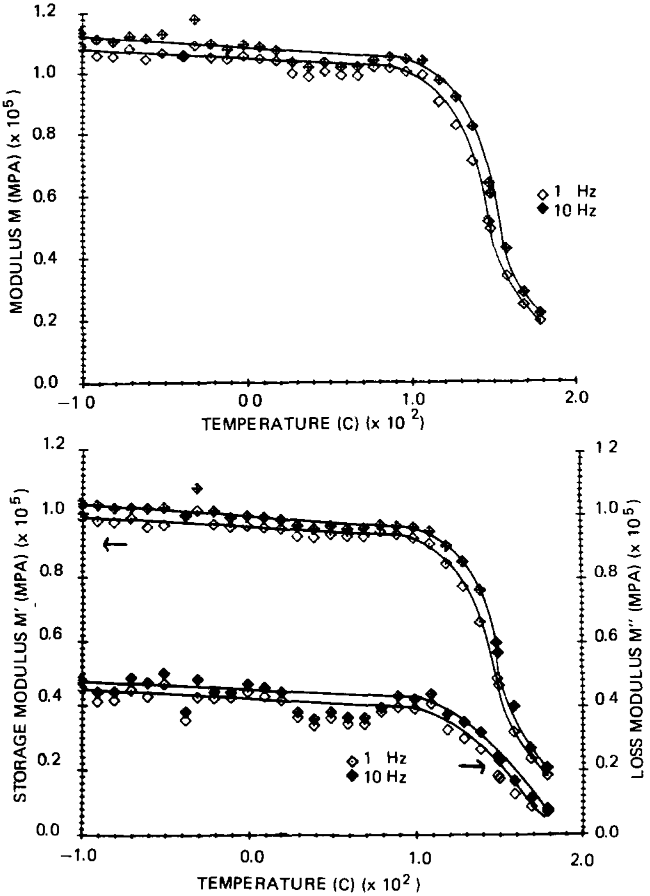

Fig. 3. Dynamic storage and loss modulus of unidirectional F-155 graphite–epoxy composite in [0] direction.

second transition peak is observed in the loss modulus at 170 °C. This second peak is an artifact of the angle ply layup. Because there are fibers normally, tangentially and symmetrically off axis the laminate dissipates energy as viscous work and a higher temperature than T_g results.

Figure 6 shows a comparison between the experimental modulus of the $[0,90,\pm 45]_s$ laminate and the predicted modulus using lamina properties from the Dynastat. The agreement is within 5% between the measured and the predicted values. Also, the predicted measurements show the experimental temperature dependent decrease in moduli as the cure temperature is approached. Overall, the predicted values for modulus is very good. Using directly measured lamina properties and indirectly calculated temperature dependent properties, the set of unknown material properties can predict viscoelastic properties of the composites with greater certainty. The same

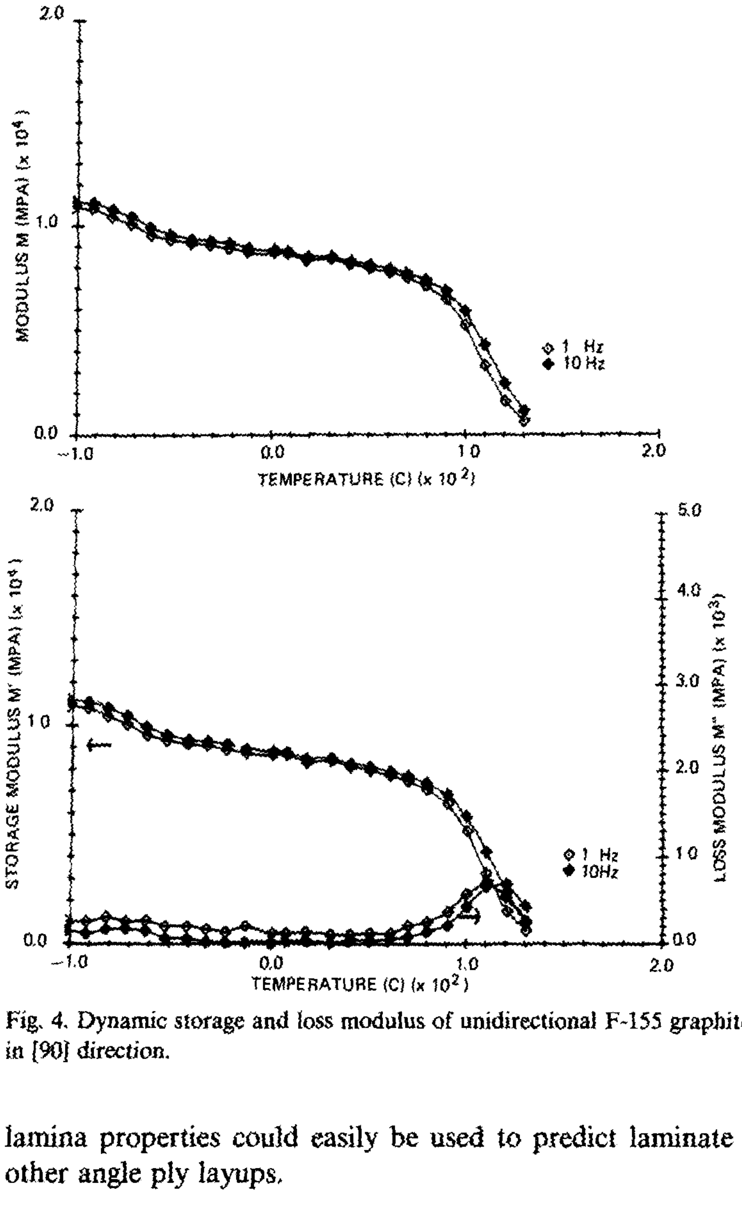

Fig. 4. Dynamic storage and loss modulus of unidirectional F-155 graphite–epoxy composite in [90] direction.

lamina properties could easily be used to predict laminate properties with other angle ply layups.

DISCUSSION

The approach to combining direct temperature measurement of tensile moduli with micromechanics for shear properties for laminate analysis provides good agreement with experimental results. One weakness of the approach presented was that no consideration of the strength of the composite was considered, since low strain levels were used to measure the elastic constants. A corollary of this is seen in the temperatures at which the

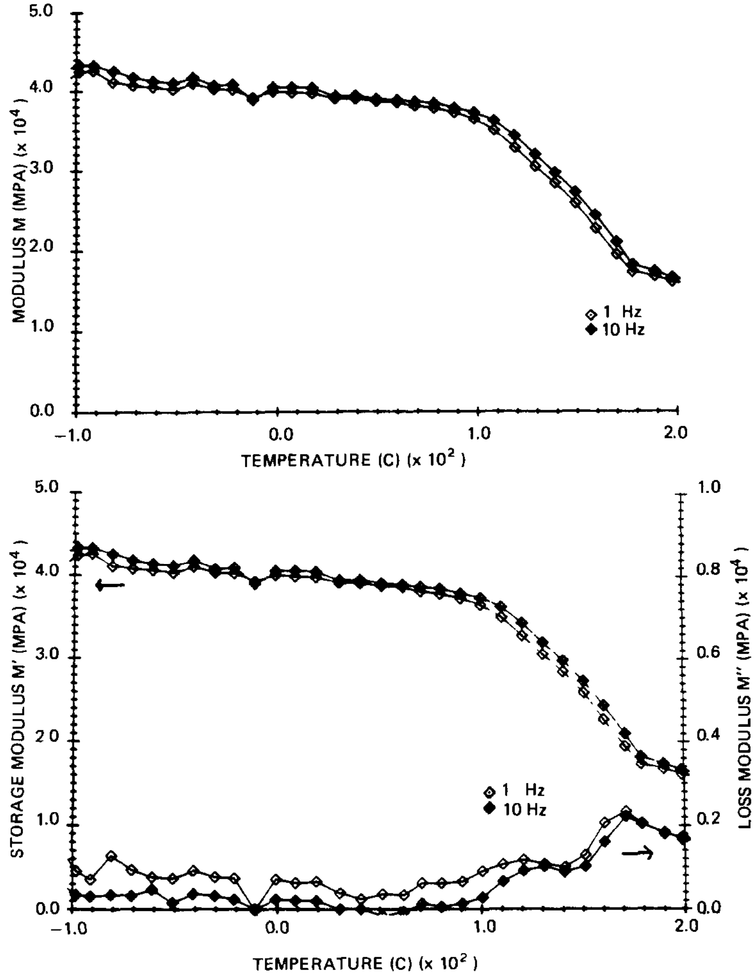

Fig. 5. Dynamic storage and loss modulus of $[0,90,\pm45]_s$ F-155 graphite–epoxy composite in [0] direction.

Dynastat could not continue to make measurements under load control. Even though this analysis was used to predict the modulus of a laminate up to the cure temperature of the matrix, one would expect the ultimate strength to decrease with temperature at high strains.

The assumption that the Poisson's ratio of the matrix was temperature independent has no basis, though it was assumed to be invariant owing to lack of experimental data. One would expect some temperature dependency. The assumption that the Poisson's ratio and shear moduli of the fibers are temperature independent is a good assumption. For graphite fibers, their processing temperatures are much higher than the application temperatures of polymeric composites.

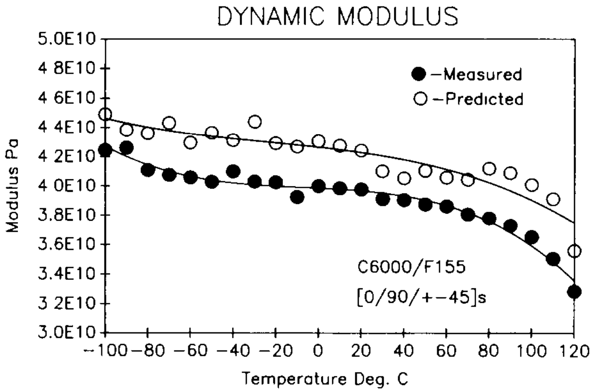

Fig. 6. Comparison between experimental and predicted results for modulus.

This analysis correctly predicts the decrease in modulus of a laminate as the T_g of the resin is approached. It also predicts an increase in modulus when the temperature falls below the secondary transition for the rubber phase. One reason for the scatter in the predicted results is that point values for E_{11} and E_{22} were used at specific temperatures. The use of an algebraic curve fit expression would reduce the scatter and simplify calculations.

CONCLUSIONS

An approach has been introduced to apply dynamic mechanical analysis to composite laminate design. It has the advantage in that it allows cost effective prediction of temperature dependent composite properties with a minimum of experimental testing. This experimental approach also provides viscoelastic property information about the composite and its constituent materials behavior.

ACKNOWLEDGMENTS

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LIST OF SYMBOLS

E lamina Young's modulus
G lamina shear modulus
k_f volume fraction
T_g glass transition temperature
ν Poisson's ratio

Subscripts

f fiber
m matrix

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