

MODELING OF MULTI-STEP NONLINEAR STRESS RELAXATION IN DGEBA/m-PDA EPOXY RESINS

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INTRODUCTION

In a previous publication, Holmes (1997), fragmentation of bare E-glass fibers embedded in an undercured diglycidyl ether of bisphenol-A (DGEBA)/meta-phenylenediamine (m-PDA) epoxy resin system was shown to occur when the stress-strain response of the matrix material is nonlinear. The occurrence of fragmentation in this region violates the linear elastic assumption in "classical" shear-lag models formulated to determine the "interfacial shear strength" or "effective shear stress transferability" at the fiber-matrix interface. Estimates using a strain dependent *secant modulus* instead of the linear elastic modulus in the Cox model suggests that the linear elastic estimate of the interfacial shear strength in dry DGEBA/m-PDA single fiber fragmentation test (SFFT) specimens is at least 15 % too high. These results are consistent with numerical simulations of the single fiber fragmentation test by Feillard (1993) which indicate that the linear elastic approximation overpredicts the number of breaks obtained during a fragmentation test. These researchers found better agreement between the numerical simulation and experimental data by using a *secant modulus* in the Cox model.

Central to understanding the applicability of the strain-dependent *secant modulus* in the Cox Model and establishing a rigorous theoretical stress-transfer model is the development of a nonlinear constitutive equation for the matrix. In the present paper we subject the DGEBA/m-PDA epoxy resin system to multi-step strain deformation profiles analogous to those obtained in the single fiber fragmentation test (SFFT). From this data we develop an empirical nonlinear constitutive equation which accurately accounts for the observed deformation behavior.

EXPERIMENTAL

Sample Preparation. Test specimens without fibers were made according to a procedure described

previously, Holmes (1997), for the preparation of SFFT specimens.

Multi-Step Stress Relaxation Test. The multi-step stress relaxation tests are carried out on a small hand operated loading frame mounted on a polarizing microscope. The image is viewed using a video camera and monitor. The sample is scanned by translating the loading frame under the microscope with a micrometer. The position of the load frame is monitored by an LVDT connected to an A-to-D board in a computer. To determine the strain, two lines are made in the gauge section of the test specimen using a "green" permanent marker. An identifiable point on each line is marked and the location of this point is determined at each strain increment. The location of the point of interest on each line is aligned with a cross hair in the microscope as seen on the video monitor, and the position of the LVDT is digitized into the computer. The load is also monitored during the experiment using a 2,224 N (500 lbf) load cell that is connected to a bridge. The bridge is attached to the same computer via a serial connection. The expected standard uncertainty of the load measurements is 3 % of the load. A custom program was developed to continuously record the load and any LVDT measurements that are made.

RESULTS AND DISCUSSION

A typical load-time curve for the multi-step relaxation of DGEBA/m-PDA is shown in Figure 1 along with various theoretical approximations. The linear elastic and linear viscoelastic approximations grossly over predict the load in the tests specimen at higher strains. This behavior indicates the epoxy resin at higher strains is exhibiting nonlinear viscoelastic behavior. Two nonlinear viscoelastic approximations of the load-time curve are also shown in Figure 1. The time-strain separable nonlinear viscoelastic approximation of the load-time curve was obtained using the strain dependent relaxation modulus shown in equation 1 and the modified Boltzmann Superposition Principle.

$$E\{\varepsilon, t\} = E_0$$

This equation Wagner type of the matrix power law behavior. This predicts the response to nonlinear strains. In an increasing strain to nonlinearity separable non-obtained by a $\theta\{\varepsilon\}$. Until the increasing strain relaxation under predicts increasing strain.

It was shown 12 from 10 min = $\theta_2\{\varepsilon\}$ could when the time relaxation behavior 2 (see Fig 1) form of the class, is used summed, i.e., θ_1 parameter, κ , strain rate of time.

$$E\{\varepsilon, \dot{\varepsilon}, t\} = E_0 \left[1 + E_0 \left(1 + \frac{t}{\tau} \right)^{-\kappa(\varepsilon)} \right]$$

(1997), for the preparation of

Relaxation Test. The multi-step are carried out on a small hand are mounted on a polarizing nage is viewed using a video . The sample is scanned by ng frame under the microscope he position of the load frame is VDT connected to an A-to-D . To determine the strain, two gauge section of the test speci- permanent marker. An identi- line is marked and the location mined at each strain increment. point of interest on each line is hair in the microscope as seen r, and the position of the LVDT e computer. The load is also e experiment using a 2,224 N at is connected to a bridge. The o the same computer via a serial pected standard uncertainty of nts is 3 % of the load. A custom oped to continuously record the ' measurements that are made.

DISCUSSION

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$$E\{\epsilon, t\} = E_o \exp(-C_1 \epsilon) \left(1 + \frac{t}{\tau}\right)^{-\theta} \quad (1)$$

This equation incorporates a damping function of the Wagner type to model changes in the elastic stiffness of the matrix with increasing strain and a modified power law expression to capture the relaxation behavior. This theoretical load-time curve still over predicts the response of the epoxy matrix at higher strains. In addition, this equation does not capture the change in relaxation behavior of the material with increasing strain. This latter observation is consistent with strain softening being due, at least in part, to nonlinearity in the relaxation process. The non-separable nonlinear viscoelastic load-time curve was obtained by allowing θ to vary with strain, i.e., $\theta = \theta\{\epsilon\}$. Until the nonseparable load-time curve deviates from the actual data, the variation of θ with increasing strain was approximately linear. This form of equation 1 qualitatively captures the change in relaxation behavior with increasing strain, but under predicts the response of the matrix to increasing strain.

It was shown that parameters generated for equation 2 from 10 minutes strain increment data when $\theta_1\{\epsilon\} = \theta_2\{\epsilon\}$ could not predict the response for the matrix when the time between strain increments was increased to 1 hr. Reasonable fits of the multi-step relaxation behavior was obtained by utilizing equation 2 (see Figure 2). In this equation a bi-exponential form of the damping function, Osaki and Laun class, is used and two relaxation regimes are assumed, i.e., $\theta_1\{\epsilon\} \neq \theta_2\{\epsilon\}$. The strain rate dependent parameter, κ , was found to scale with the effective strain rate of the multi-step stress relaxation experi- ment.

$$E\{\epsilon, \dot{\epsilon}, t\} = E_o \left(1 + \frac{t}{\tau}\right)^{-\kappa\{\dot{\epsilon}\}} \left[f_1 \exp(-C_1 \epsilon) \left(1 + \frac{t}{\tau}\right)^{-\theta_1\{\epsilon\}} + f_2 \exp(-C_2 \epsilon) \left(1 + \frac{t}{\tau}\right)^{-\theta_2\{\epsilon\}} \right] \quad (2)$$

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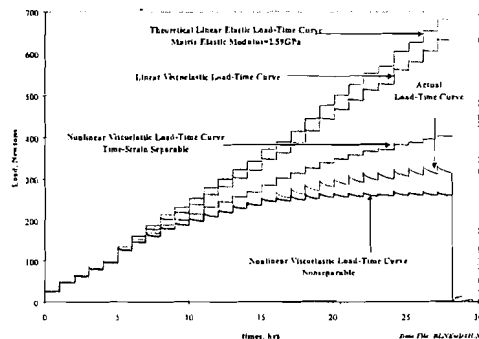


Fig. 1: Approximations of multi-step relaxation behavior in DGEBA/m-PDA epoxy resin strain at 1 hour time increments.

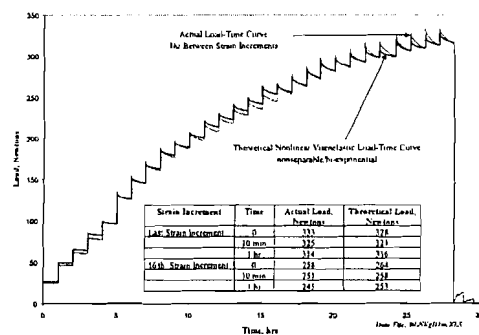


Fig. 2: Theoretical prediction of multi-step relaxation behavior of DGEBA/m-PDA specimen using equation 2.

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ABSTRACT

The last ten or fifteen years *can* be, achieved in the properties. On the one hand and of semi-crystalline multiphase materials (e.g. methods of characterization and sophistication of statistical methods) appears that the need for consequently, the possibility examines these issues and