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Polymer 42 (2001) 5285–5291

polymer

www.elsevier.nl/locate/polymer

Effective adhesive modulus approach for evaluation of curing stresses

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Received 28 June 2000; received in revised form 21 September 2000; accepted 26 September 2000

Abstract

A study was performed to measure the curing stresses of several epoxy systems using the bimaterial beam approach. In addition, the stress relaxation profile of the adhesive bonded to the metallic strip (bimaterial strip) was compared to the relaxation profile of the bulk adhesive. It was found that the shape of the relaxation curves was consistent for both samples, but that the magnitude of the two curves differed. It is argued that the difference between the curves is a result of curing stress and that the long-term effective modulus as measured from the bonded adhesive should be used to establish the curing stress. Use of an improper modulus is one possible cause of confusion regarding the determination of curing stresses as reported in the literature. © 2001 Thiokol Propulsion. Published by Elsevier Science Ltd. All rights reserved.

Keywords: Residual stress; Viscoelastic modeling; Thermoset cure

1. Introduction

The use of adhesives as engineering materials requires an understanding of their mechanical properties. This includes knowledge of the nature and magnitude of curing stresses formed when the material is used in an adhesive joint. These stresses can superimpose themselves on externally applied stresses and thus cause premature failure of the adhesive bonds [1]. Cure stresses can also negatively impact the fracture properties of the adhesive [2]. Factors that will influence the magnitude of the stresses include volume changes during curing, geometry of the constraining hardware, curing time and temperature, material properties of the system, mechanical deformation, and dimensional changes resulting from thermal fluctuations [3–5].

Curing stresses can result from volume changes that occur during the polymerization process and will depend on the chemical nature of the material and the curing procedure [6]. The adhesive can go through two main transitions during the cure, gelation and vitrification. Gelation corresponds to the formation of an infinite network. Vitrification occurs when the glass transition temperature, T_g, of the adhesive reaches the cure temperature. If the material is isothermally cured above the ultimate glass transition temperature, only gelation will occur. When the material is cured below the ultimate glass transition temperature, it will first gelate and then vitrify. The gelation process results in an equilibrium elastic modulus, whereas vitrification is associated with the transition from a rubbery modulus to a glassy modulus [7]. The change in elastic modulus associated with gelation and vitrification is the dominating factor in the occurrence of the contractive stress rather than just the volume contraction itself [8]. When the adhesive is cured above the ultimate glass transition temperature, curing stresses resulting from volume changes are minimized because of the low magnitude of the rubbery modulus [9,10].

Curing stresses also result as the adhesive joint is cooled from the cure temperature to ambient temperature. Thermal stresses occurring from cooling arise from differential thermal expansions in the joint. The magnitude of these stresses will depend on the material constants of the adhesive including the thermal expansion coefficient, Poisson's ratio, and elastic modulus [4]. These constants depend on the extent of cure, test temperature, and time scale at which they are evaluated. The magnitude of the curing stresses is also affected by how constrained the adhesive is in the joint. If the material is subjected to a one-dimensional constraint, the resulting stress will scale linearly with the dimensional change resulting from thermal expansion and chemical shrinkage. This trend is true if the material behaves according to the following assumptions: material isotropy, incremental linear elastic behavior, history-dependent elastic coefficients, and no viscoelastic behavior. The curing stresses under biaxial constraints will be larger by a factor of

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PII: S0032-3861(00)00725-4

$1/(1 - v)$ where v is Poisson's ratio. The curing stresses for the three-dimensionally constrained system will increase over the uniaxial case by a factor of $1/(1 - 2v)$. The effect of the constraining geometry can be examined by considering an adhesive that is constrained in three-dimensions and is cured above its ultimate glass transition temperature. For this case, the material exhibits elastomeric-like behavior and Poisson's ratio approaches a value of 1/2. The denominator in $1/(1 - 2v)$ approaches zero with the result of a large induced stress for any process that results in shrinkage [3].

From an engineering approach, measuring the stress associated with the curing process is desirable. A number of experimental approaches have been used to make these measurements and include wafer deflection [11], photoelasticity [12], strain gage [5,13,14], bimaterial strip [1,14–17,19], impulse viscoelasticity [18], and cantilever methods [10,20]. In addition, mathematical methods have been used to estimate curing stresses in adhesive joints. On the practical front, most of these analyses are elastic and make no allowance for stress relaxation of the adhesive [20].

Of particular interest to this work is the bimaterial strip approach. This method is similar to the analysis of the bimetallic strip developed by Timoshenko [21]. If a layer of adhesive bonded to a metallic strip contracts or expands, the strip will curve in response. The extent of curvature is a measure of the stress state in the adhesive if linear limits are maintained. Inoue and Kobatake also developed a relation to describe the residual stress for a composite strip as a function of the radius of curvature [22]. Their derivation differed from that of Timoshenko in that each filament in the bimaterial strip is assumed to be cemented tightly instead of only at the ends.

In this work, we utilize a bimaterial strip along with the relation of Inoue and Kobatake to measure the curing stress as a function of an equilibrium modulus. Afterwards, a dynamic mechanical analyzer (DMA) is used to establish the stress relaxation profile of the bimaterial strip. The results are compared to the relaxation profile of the bulk adhesive. Finally, emphasis is placed on the role of the measured modulus and how it relates to the curing stress.

2. Experimental

2.1. Materials

The epoxy resins used in the study were TIGA 321® manufactured by Resin Technology Group (RTG) and EA 946® manufactured by Dexter-Hysol. In addition, a simple epoxy system of 34 parts by weight Epon® 828, 55 parts by weight Versamid® 140, and 4 parts by weight TS-720 Cab-O-Sil® was considered.

2.2. Preparation of bimaterial strips

The epoxies were vacuum mixed and then coated on a brass strip having the dimensions of 5.79 × 0.635 × 0.013 cm. The adhesive thickness was controlled to 0.005 cm. After coating with the adhesive, the bimaterial strip was placed at 41°C for 48 h and then rapidly cooled to 22°C for the TIGA 321 and EA 946 adhesives. This cure cycle was chosen because it duplicates the cure schedule of these adhesives as they are used on the nozzle of the Space Shuttle booster motor produced by Thiokol Propulsion Group. The Epon® 828 + Versamid® 140 epoxy was cured at 121°C for 15 h and then rapidly cooled to 22°C.

2.3. Curing stress

After the bimaterial strip was cured and cooled to ambient temperature, it began to curve. The deflection of the center of the strip, δ, was optically measured and related to the radius of curvature, ρ, by the following relation

$$\delta = \frac{l^2}{8\rho} \tag{1}$$

where l is the length of the bimaterial strip and it is assumed that $\delta \ll \rho$.

2.4. Stress relaxation

Recently, a DMA was used to measure the stress-free temperature of a bimaterial strip [23]. In this work, a Rheometrics RSA II DMA was used to measure the stress relaxation profile of both the bimaterial strip and bulk adhesive using a 3-point bending setup. The bimaterial strip is placed with the concave surface facing up and the loading arm is placed at the center of the strip. During testing, a step strain of 0.1% was applied to the sample. The choice of 0.1% strain was well within the linear viscoelastic regime for these samples. After application of the step strain, the loading arm was able to monitor the applied force as a function of time. This stress relaxation test was repeated with the concave surface of the bimaterial strip facing down and no difference in the relaxation profile was observed. A similar approach allowed the stress relaxation profile to be evaluated for the bulk adhesive molded into thin strips. The DMA was equipped with an oven that allowed these measurements to be carried out over a relatively wide temperature range.

2.5. Coefficient of thermal expansion

The coefficient of thermal expansion was evaluated on the bulk adhesive using a Perkin–Elmer Thermal Mechanical Analyzer (TMA) 7. The instrument was outfitted with a flat tipped expansion probe. Samples were tested from −75 to 150°C at 10°C/min.

2.5.1. Glass transition temperature

The onset T_g of the adhesive was measured using a Mettler-Toledo differential scanning calorimeter (DSC) 821. Samples were tested from −75 to 150°C at 10°C/min.

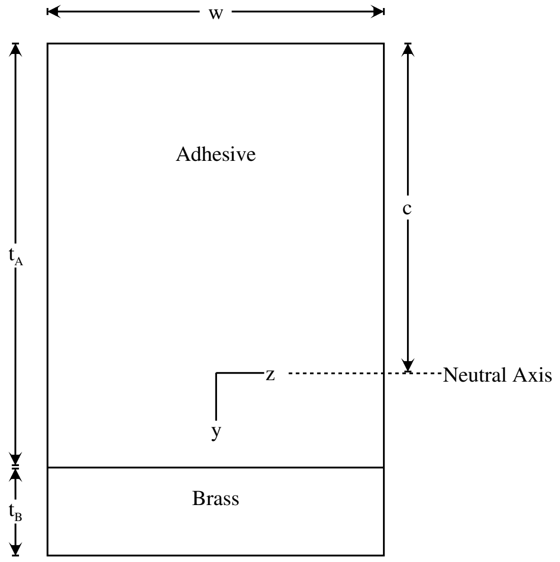

Fig. 1. Coordinate systems of adhesive bonded to a brass shim as viewed axially through the cross-sectional area.

3. Results and discussion

3.1. Stress relaxation

In a typical 3-point bending test, the relation between the deflection at the center of the beam, δ, and the applied load, P, is

$$\delta = \frac{PL^3}{48EI} \tag{2}$$

where L is the length of the support span, E is Young's modulus of the beam, and I is the moment of inertia for the beam [24]. In the case of the bimaterial strip, the measured modulus as given by Eq. (2) will depend on the moduli of both the adhesive and the metallic strip. If the effective modulus of the bimaterial strip is viscoelastic, Eq. (2) changes. For a stress relaxation test, the displacement term will remain constant, but the load becomes a function of time, as does the flexural rigidity term, EI.

The flexural rigidity of the bimaterial strip depends on the viscoelastic behavior of both the brass shim and adhesive. For the experimental times considered, the modulus of the brass shim can be assumed constant with the time-dependent contribution to the measured modulus coming solely from the adhesive. The moment of inertia will in turn depend upon the neutral axis plane of the composite strip, which is also changing with time. For a stress relaxation test, Eq. (2) becomes

$$EI(t) = \frac{P(t)L^3}{48\delta} \tag{3}$$

where $P(t)$ and $EI(t)$ are the applied load and flexural rigidity as a function of time, respectively.

The change in flexural rigidity of the composite is related to the individual contributions from both the brass and adhesive. Fig. 1 shows the cross-section of the bimaterial beam. The x-coordinate direction is perpendicular to the plane of the paper and the y-coordinate is defined as positive towards the bottom of the page. For a beam made of two materials, the bending moment, M, is

$$M = \int_A \sigma_{xA} y \, dA + \int_B \sigma_{xB} y \, dA \tag{4}$$

where σ_{xA} is the stress of the adhesive acting in the x-direction, σ_{xB} is the stress of the brass shim acting in the x-direction, y is the distance from the neutral axis plane, and dA is the differential change in area. The subscripts A and B will denote properties relating to the adhesive and brass, respectively. Making use of Hookes law, $\sigma = E\epsilon$, and the strain in the x-direction, $\epsilon_x = y/\rho$, Eq. (4) becomes

$$M = \frac{1}{\rho}\left[E_A \int_A y^2 \, dA + E_B \int_B y^2 \, dA\right] \tag{5}$$

If the observed moment of the bimaterial beam, $M = EI/\rho$, is used, Eq. (5) becomes

$$EI = E_A I_A + E_B I_B \tag{6}$$

where $I = \int y^2 \, dA$.Using the parallel axis theorem gives

$$I_A = \frac{1}{12} w t_A^3 + t_A w \left(c - \frac{1}{2} t_A\right)^2$$

$$I_B = \frac{1}{12} w t_B^3 + t_B w \left(t_A + \frac{1}{2} t_B - c\right)^2$$

where c is the distance from the top of the section to the neutral axis, w is the width of the beam, t_A is the thickness of the adhesive, and t_B is the thickness of the brass.

It is also noted that there are no external forces acting on the strip from the x-direction, therefore,

$$\sum F_x = 0 \Rightarrow \int_A \sigma_{xA} \, dA + \int_B \sigma_{xB} \, dA = 0$$

and remembering that $\sigma_{xA} = E_A y/\rho$ and $\sigma_{xB} = E_B y/\rho$, then the summation of forces becomes

$$E_A \int_A y \, dA + E_B \int_B y \, dA = 0 \tag{7}$$

To perform this integral, Fig. 1 is divided into three centroids. The first is the area from the top of the section to the neutral axis. The second is the area from the neutral axis to the adhesive and brass interface. The third is the area of the brass shim. Eq. (7) then becomes

$$E_A w\left[-\frac{1}{2}c^2 + \frac{1}{2}(t_A - c)^2\right] + E_B t_B w\left[t_A - c + \frac{1}{2} t_B\right] = 0 \tag{8}$$

Eq. (8) can be solved for E_A and then substituted into Eq. (6). This along with the use of the flexural rigidity of the composite beam as given by Eq. (3) allows the relaxation modulus of the bonded adhesive to be measured.

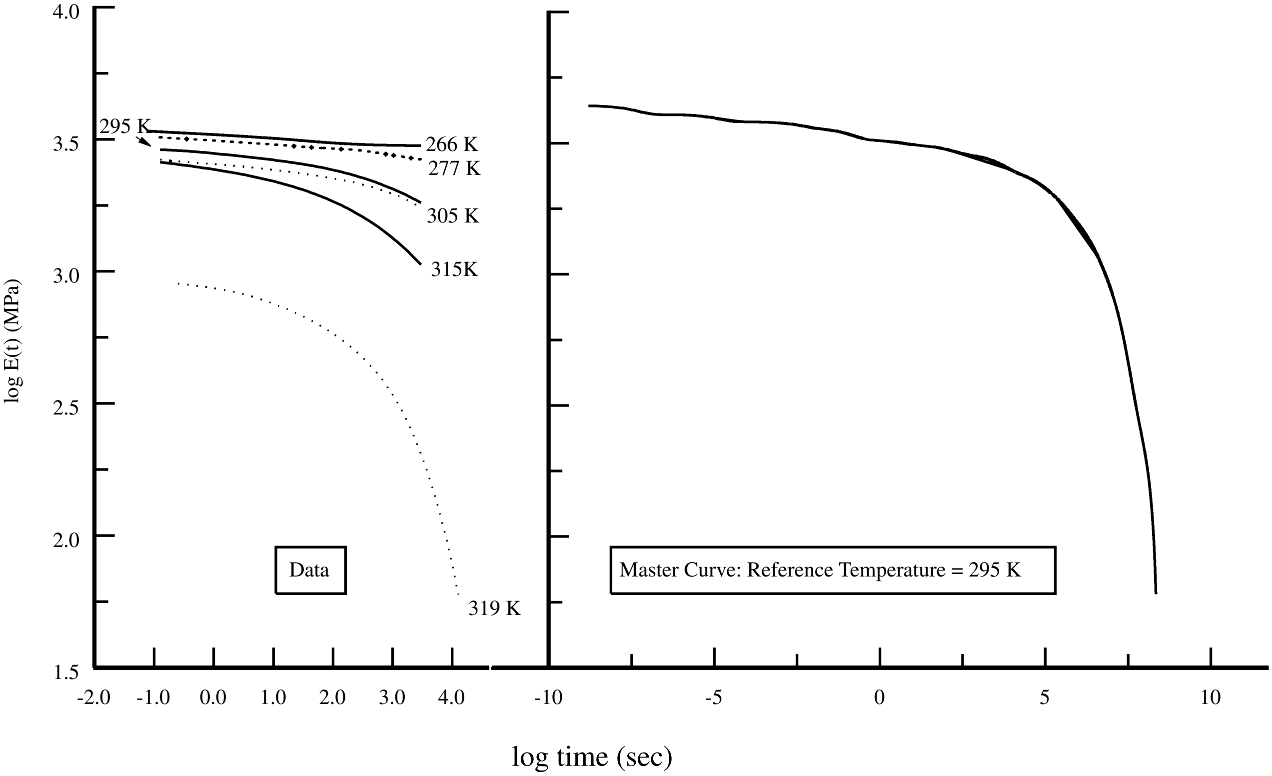

Fig. 2. Master stress relaxation curve for TIGA 321 bonded to a brass shim as measured by 3-point bending test on TMA.

3.2. Master stress relaxation curve

If a relaxation spectrum of the adhesive is measured at different temperatures, a master curve can be constructed by shifting the experimental logarithmic curves along the time axis to obtain superposition. The distance shifted represents the logarithm of the shift factor, a_T. Williams, Landel, and Ferry proposed an empirical equation for the temperature dependence of the shift factor [25]. This relation is

$$\log a_T = \frac{-C_1(T - T_g)}{C_2 + T - T_g} \tag{9}$$

where T is the temperature of interest, C_1 and C_2 are experimentally determined constants. In addition, a small correction is required in the form of a vertical shift due to temperature variations and changes in volume resulting from temperature changes [26]. The correction factor is

$$E(T_g, t) = \frac{\rho(T_g)T_g}{\rho(T)T} E(T, t/a_T) \tag{10}$$

where $E(T_g, t)$ is the shifted modulus, $\rho(T_g)$ is the density measured at T_g, $\rho(T)$ is the density at temperature, T, $E(T, t/a_T)$ is the modulus measured at temperature, T, and shifted along the time axis by a_T. Since the contribution from the mass to the density does not change with temperature, Eq. (10) may be rewritten as

$$E(T_g, t) = \frac{V(T)T_g}{V(T_g)T} E(T, t/a_T) \tag{11}$$

where $V(T)$ is the volume of the body at temperature, T, and $V(T_g)$ is the volume of the body at the glass transition temperature.

It has been observed that the length of an isotropic body increases with temperature over a fairly broad temperature

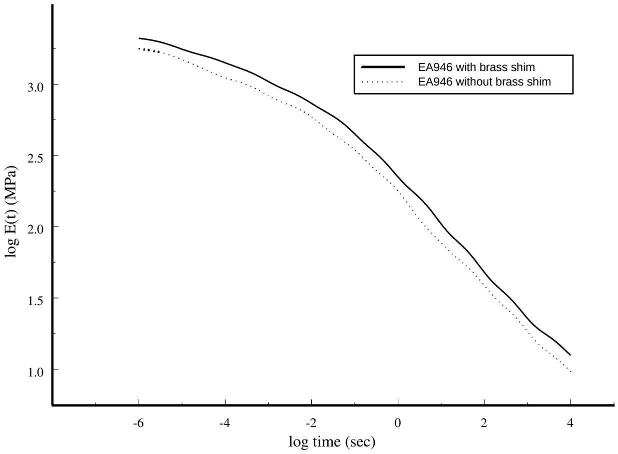

Fig. 3. Master stress relaxation curve for EA 946 for bulk adhesive and adhesive bonded to brass shim.

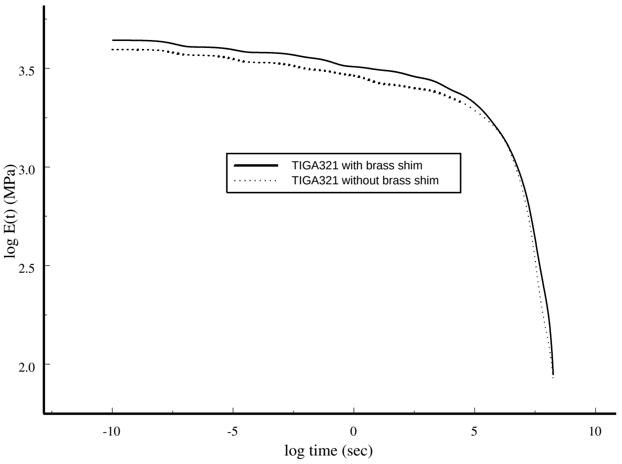

Fig. 4. Master stress relaxation curve for TIGA 321 for bulk adhesive and adhesive bonded to brass shim.

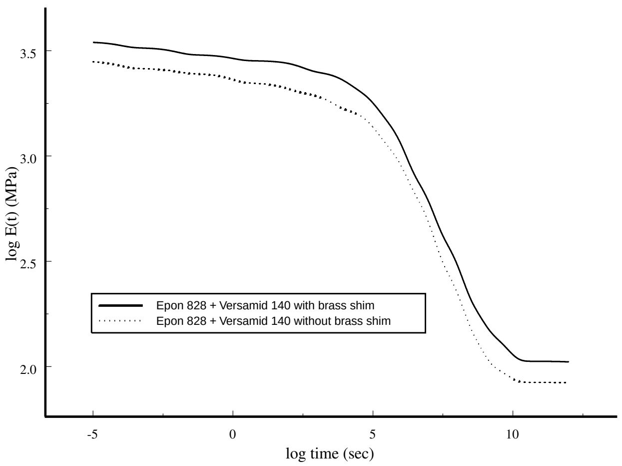

Fig. 5. Master stress relaxation curve for Epon 828 + Versamid 140 for bulk adhesive and adhesive bonded to brass shim.

range as [27]

$$\ln l_{\mathrm{i}}(T) = \ln l_{\mathrm{i}}(T_0) + \alpha(T - T_0) \tag{12}$$

where $l_{\mathrm{i}}(T)$ is the length of the body at temperature, T, $l_{\mathrm{i}}(T_0)$ is the length of the body at some reference temperature, T_0, and α is the linear coefficient of thermal expansion. Also, indicial notation is used. Since $V = l_1 l_2 l_3$, then Eq. (11) when used in conjunction with Eq. (12) becomes

$$E(T_{\mathrm{g}}, t) = \mathrm{e}^{3\alpha(T-T_{\mathrm{g}})}\frac{T_{\mathrm{g}}}{T}E(T, t/a_{\mathrm{T}}) \tag{13}$$

Eq. (13) can be used to shift the relaxation curves to make a master curve. Fig. 2 illustrates a master relaxation curve for TIGA 321 bonded to a brass shim.

3.3. Relaxation curve for bulk adhesive versus bonded adhesive

Figs. 3–5 show the relaxation curves for EA 946, TIGA 321, and Epon 828 + Versamid 140 for both the bulk and bonded resin. The most noticeable features are that the relaxation moduli for the bonded adhesives are of greater magnitude than for the bulk adhesive and are generally separated by a constant. This phenomenon has been observed before when an adhesive modulus has been measured in butt joints [28]. The measured or apparent modulus of the adhesive is of greater magnitude than that of the unbonded material. This is because the adhesive is constrained and the substrates are more rigid than the adhesive [1].

The relaxation curves shown in Figs. 3 and 4 do not show a proper long-term equilibrium modulus. This is because both adhesives are cured at 41°C for 48 h which does not allow either adhesive to completely cure. To prevent additional chemical aging during the stress relaxation test, the test temperatures were restricted to slightly above the cure temperature. A true equilibrium modulus is not reached during the stress relaxation test at the chosen test temperatures but this value will not differ significantly from the measured long-term modulus. The adhesive used to generate Fig. 5 was cured above its ultimate glass transition temperature (87°C). This allowed higher test temperatures to be used and a more proper equilibrium modulus to be measured.

For the curves shown in Figs. 3–5, the effect of the curing stress on the relaxation spectra is of particular interest. Kubat and Rigdahl noted, at conditions of low stress and/or temperature, the following relation for both metals and polymers in a stress relaxation test [29–33]

$$\sigma(t) - \sigma_{\mathrm{i}} = K(t + a)^{-n} \tag{14}$$

where $\sigma(t)$ is the time-dependent measured stress, σ_{i} is the internal stress and is assumed independent of time, t is the time, K, a, and n are constants. The term on the right of the equal sign is a decaying power law that yields a relaxation spectrum. The terms on the left indicate that the resulting relaxation curve is shifted vertically by a constant, which in this case they ascribe to the internal stress. In polymers, relaxation phenomena are often interpreted in

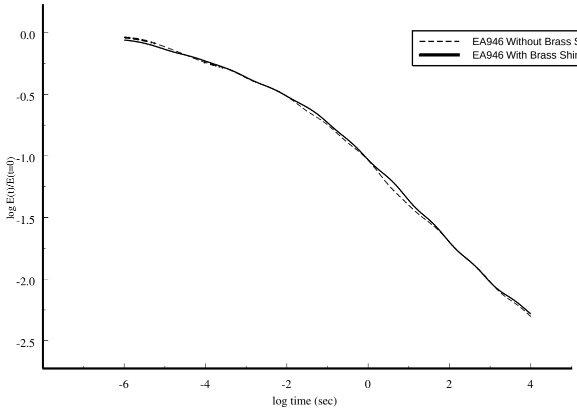

Fig. 6. Normalized master stress relaxation curve for EA 946 for neat resin and adhesive bonded to brass shim.

terms of relaxation time spectra, changes in free volume, etc. That is to say in terms of approaches where internal stress does not appear explicitly. However if the internal stress is included in these interpretations, special care must be taken on how this internal stress is described. White indicates that the internal stress as written in Eq. (14) can refer to two quite different physical phenomena [34]. One type is a result of normal curing stresses found in thermosets. The other type is often used in the field of physical metallurgy to indicate a resistance to deformation. Polymers can behave in a phenomenologically similar manner to that of metals though the responsible mechanisms are almost certainly different. The two types of internal stress are interactive and depend upon the processing conditions.

Normally, stress relaxation can be written as

$$\sigma(t) = E(t)\epsilon_o \tag{15}$$

where $E(t)$ is the time-dependent relaxation modulus and ϵ_o is the applied strain. Instead an effective relaxation modulus as a function of time, $E_{\mathrm{eff}}(t)$, can be defined for a bimaterial strip as

$$E_{\mathrm{eff}}(t) = f(\sigma_i)E_{\mathrm{bulk}}(t) \tag{16}$$

where $f(\sigma_i)$ is a time-independent constant that depends at a minimum on the internal stress of the material and E_{bulk} is the relaxation modulus of the bulk adhesive. Eq. (15) may be rewritten as

$$\log \sigma_{\mathrm{eff}}(t) = \log f(\sigma_i)\, \epsilon_o + \log E_{\mathrm{bulk}}(t)\epsilon_o \tag{17}$$

where $\sigma_{\mathrm{eff}}(t)$ is the effective measured stress for the composite beam as a function of time. The $\log f(\sigma_i)\epsilon_o$ term is time-independent and will shift the relaxation spectrum by a constant. This behavior is seen in Fig. 3 where the lower curve was generated from the bulk adhesive and the upper curve was measured for the adhesive in a stressed state. Similar behavior is seen in Figs. 4 and 5. If both curves in Fig. 3 are normalized by the initial measured modulus, they can be superimposed over each other as seen in Fig. 6. This indicates that a constant is all that differentiates the two different curves.

When evaluating the engineering properties of the adhesive, it is important to consider whether the adhesive is the same in bulk form as when bonded between substrates. Curing stresses are present in greater magnitude when the adhesive is cured in a constrained geometry. This in turn can influence the mechanical properties of the bulk adhesive compared to the bonded material [2]. This behavior was observed by Knollman, who measured a change in shear modulus as the distance from the adhesive/adherend interface increased [35].

3.4. Measurement of curing stress

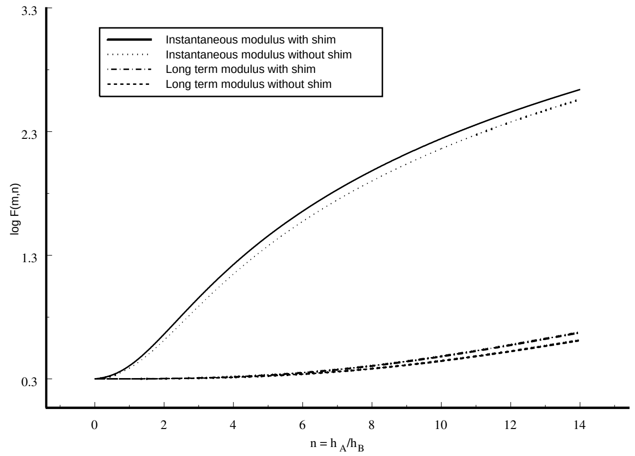

Fig. 7. Structural function, $F(m,n)$, for EA 946 adhesive.

The stress associated with the curing process, σ_i, can be measured for a bimaterial strip using the following relation derived by Inoue and Kobatake [22]

$$\sigma_i = \frac{E_B h_B^3}{12h_A}\frac{1}{\rho(h_A + h_B)}F(m,n) \tag{18}$$

where E_B is Young's modulus of the brass shim and is equal to 110 GPa; h_A the thickness of the adhesive; h_B the thickness of the brass shim; $m = E_A/E_B$; $n = h_A/h_B$; and $F(m,n)$ a structural factor given by

$$F(m,n) = \frac{(1 - mn^2)^3(1 - m) + [mn(n + 2) + 1]^3 + m(mn^2 + 2n + 1)^3}{(1 + mn)^3} \tag{19}$$

In Eq. (18), the dependence of the internal stress on E_A is given by the function $F(m,n)$. As derived, E_A is assigned to the instantaneous modulus and does not allow for any stress relaxation. Under experimental conditions, the measured equilibrium stress will depend on an equilibrium modulus not an instantaneous one. The choice of the adhesive modulus value to be used strongly impacts the resulting measured stress. Examination of Figs. 3 and 4 show that four possible moduli for a given adhesive can be substituted into Eq. (19). Two of the possible choices are the instantaneous moduli for the bulk and bonded adhesive. The other two choices are the long-term moduli for the bulk and bonded adhesive. Fig. 7 shows log $F(m,n)$ for the EA 946 adhesive versus the variation in adhesive to substrate thickness (h_A/h_B) for the four different moduli. When the substrate is much thicker than the adhesive (i.e. $n \longrightarrow 0$), the differences in the four types of adhesive moduli do little to affect $F(m,n)$. However as n approaches the ratio of adhesive to substrate thickness used in this work ($n = 14$), the impact of the modulus choice becomes significant. The best choice for modulus is one of the long-term variety. More specifically, the effective long-term modulus should be used because this is an accurate measure of the bimaterial beam's resistance to deformation. The other measures for the moduli shown in Fig. 7

give an estimate of the possible error if the wrong modulus is chosen. Using an equilibrium effective modulus for EA 946 of 8.9 MPa in Eq. (18) gives a curing stress measure of 0.75 MPa for EA 946. If the long-term modulus for the bulk adhesive is chosen, the calculated curing stress would be underestimated by 25.5%. Either of the short-term moduli would overestimate the curing stress by several orders of magnitude. Using the long term effective moduli, curing stresses of 1.1 and 8.8 MPa were established for TIGA 321 and Epon 828 + Versamid 140, respectively.

It has been pointed out that for a similar chemical system, different results for the curing stress have been obtained by different researchers [13]. The choice of modulus is one possible source of confusion. In some instances, the instantaneous modulus for the bulk material has been used [7,14]. If no other corrections are used, this will lead to an overestimation of the residual stress. In other circumstances, an effective modulus measurement of the adhesive in the test geometry was performed. But again an instantaneous modulus was measured, leading to a possible overestimation of the curing stress [1,23]. Finally, the contribution of the adhesive to the stiffness has often been neglected under the assumption that the adhesive layer thickness and modulus are much less than that of the metallic substrate [7–9,15,17,36]. These assumptions are reasonable as long as the conditions are met. But in checking the assumptions, the effective long-term modulus should be used, especially as the adhesive layer thickness increases.

4. Conclusions

From the presented data, the following conclusions were drawn:

1. The effective modulus of an adhesive in a bimaterial strip will be greater than for the bulk adhesive and may depend on the extent of curing stress.
2. The long-term effective modulus is the most accurate measure for determining the curing stress for a bimaterial strip.
3. Confusion in the literature regarding the curing stress may have arisen from incorrect measures of the adhesive modulus.

Acknowledgements

The author is greatly indebted to D.L. Lamont who substantially contributed to the present work.

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