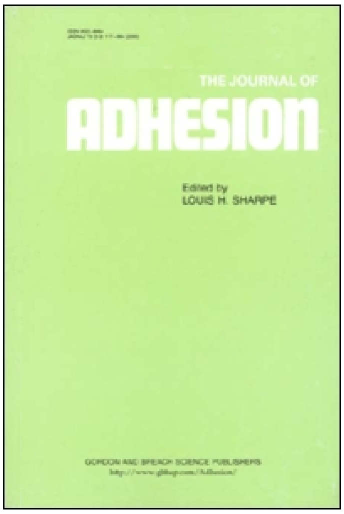

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Measurement of Adhesive Bond Properties Including Damage by Dynamic Mechanical Thermal Analysis of a Beam Specimen*

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A method to obtain mechanical properties using a bonded (double) cantilever beam (or three point bend) specimen loaded in a manner to produce pure shear in the adhesive layer is reviewed. A revised mathematical solution which allows for easier interpretation of optimum beam dimensions to the one originally developed by Moussiaux, Cardon and Brinson for the static case is presented. An extension of this solution for a fixed/fixed viscoelastic beam under steady state oscillations developed by Li, Dickie and Morman is also discussed. Previous results using a vibrating beam to determine the complex viscoelastic properties of a bonded beam are reviewed. These results demonstrate conclusively that dynamic mechanical thermal analysis (DMTA) measurements discriminate differences in surface treatments and various environmental conditions. New measurements are presented that indicate the DMTA procedure can be used to quantify damage simulated by imbedded flaws in beams. The procedure is also shown to assess the effects of both humidity and corrosive environments on lap specimens. It is suggested that this technique may ultimately provide a method to quantify the amount of hidden damage in an adhesive joint subjected to fatigue, moisture or corrosive environments.

KEY WORDS Adhesive pure shear properties; three-point bend specimen; dynamic mechanical thermal analysis; damage; humidity; corrosion.

INTRODUCTION

A simple but accurate method for the reliable evaluation of the mechanical properties of adhesives has long been needed for the proper design of adhesively-bonded structures. Bonded structures often have intricate shapes which are not amenable to closed-form mathematical analysis. They are, however, quite easily analyzed using

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finite element methods (FEM) but this method requires the input of mechanical properties of the adhesive. The needed properties are frequently determined using lap shear or other specimens which have multi-axial stress states within the bond line. Properties so determined are only averages and are not necessarily representative of those in the joint to be designed. For these reasons, scientists and engineers have been seeking specimen geometries which would have only pure stress states (tensile or shear) in the adhesive layer. An equally important consideration is to have a simple but accurate method of measuring strains or deformations in the adhesive layer. Under these circumstances, the true mechanical properties of the adhesive bond can be determined.

Several candidate specimens for pure shear have been proposed such as the Iosepescu, the Arcan and a cone-and-plate torsion bar with a geometry similar to a cone-and-plate viscometer. A review of these methods is given in Reference 1. Basically, each specimen is designed to have a uniform shear stress (or strain) over the bonded area with no peel stresses present. Shear stress can then be found by simply dividing the load by the contact area and the shear strain can then be found by dividing specimen elongation (or rotation) by the adhesive thickness. The shear modulus can be found using the elementary definition of stress divided by strain. These specimens work well but require precisely machined mating parts and are often not practical for routine applications where a large volume of testing is necessary, such as property determination for varying environmental conditions.

An alternate bonded cantilever (or three-point bend) specimen has been proposed which eliminates the need for precision machining and provides a simple method by which adhesive properties under a variety of environmental conditions can be evaluated by measuring beam deflections.[1-6] The static case for elastic adhesives was investigated by Brinson *et al.*[1-4] while the dynamic and viscoelastic case has been investigated by Dicke *et al.*[5,6] In the former, shear properties are shown to be obtainable from either strain measurements within the bondline or by measuring beam deflections. In the latter, the closed-form solution for shear stress and beam deflection for the bonded cantilever beam developed by Moussiaux[3] was modified for a fixed/fixed beam. The modified solution was used to analyze measurements of viscoelastic properties of bonded beams determined using a dynamic mechanical thermal analyzer (DMTA). The same approach was also used to study the effects of various environmental conditions and various surface treatment conditions.

The purpose of the investigation reported herein was to determine if the DMTA approach for a fixed/fixed beam could be used to evaluate damage in an adhesive bond and if the approach could also be used to evaluate the effects of humidity and corrosion on an adhesive bond. A side result of the present investigation was that a modification of the solution of Moussiaux[3] was found which allows for easier optimization of specimen dimensions for different adhesive/adherend combinations.

REVIEW OF PREVIOUS RESULTS

Cantilever Beam Approach to Property Measurements

The bonded cantilever beam loaded at the end as shown below in Figure 1 will bend in such a manner as to produce a pure shear stress in the adhesive layer. This is easily

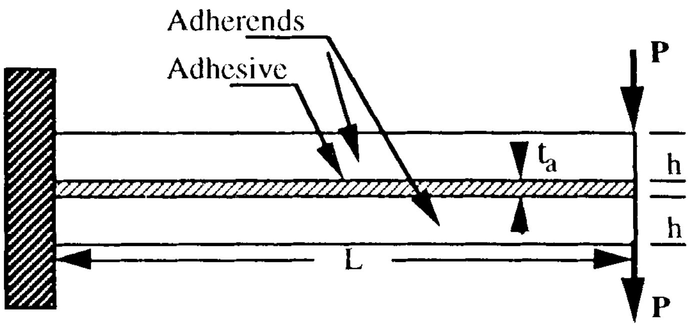


FIGURE 1 Adhesively bonded cantilever beam.

recognized by examining the deformations of the top and bottom adherends and noting that the bottom of the top adherend will deform exactly the same amount as the top of the bottom adherend but in the opposite direction. A detailed examination of the deformed adherends is given in References 3 and 7.

Moussiaux[3] developed a differential equation for the shear stress in the adhesive layer using elementary beam theory into which expressions of moment equilibrium and compatibility of deformations were substituted. The differential equation was then solved to obtain the shear stress distribution in the adhesive layer along the length of the bond line and the standard Euler-Bernoulli beam deflection equation was solved to obtain beam deflections. The resulting equations for adhesive shear stress along the length of the beam and the deflection at the free end were given as:

$$\tau_{xy} = \frac{P}{b\gamma^2(h+2t)}[1 - \cosh(\alpha\xi) + \tanh(\alpha)\sinh(\alpha\xi)] \tag{1}$$

$$\delta = \beta P \frac{L^3}{2Eb(h+t)^3} \tag{2}$$

where τ_{xy} is the shear stress, L is the beam length, $\xi = x/L$ is the non-dimensional distance along the beam, b is the beam width, h is the adherend thickness and $2t = t_a$ is the adhesive thickness. The parameters α, β and γ were found to have the following form,

$$\alpha = \gamma\sqrt{3\frac{G_a}{E}\left(\frac{L}{h}\right)^2 \frac{\left(1+2\frac{t}{h}\right)^2}{\frac{t}{h}}} \qquad \gamma = \sqrt{1 + \frac{1}{3\left(1+2\frac{t}{h}\right)^2}}$$

$$\beta = \left(\frac{h+t}{h}\right)^3\left[4\left(1-\frac{1}{\gamma^2}\right) + \frac{3E}{G_a}\left(\frac{h}{L}\right)^2 + \frac{12}{\alpha^2\gamma^2}\left(1-\frac{1}{\alpha}\tanh\alpha\right)\right]$$

where G_a is the adhesive shear modulus and E is the modulus of the adherend. As may be seen, α, γ and β are functions of the beam geometry and the moduli of the adhesive and adherends. These parameters were found in Reference 3 by forcing continuity at the centerline of the adhesive. Instead, if continuity is enforced at the interface between adherend and adhesive, the following equations for adhesive shear stress along the length of the beam and end deflection are found:

$$\tau_{xy} = \frac{3P}{4bh}[1 - \cosh Ax + \tanh AL \sinh Ax] \tag{3}$$

$$\delta = \beta_b \frac{PL^3}{2Ebh^3} \tag{4}$$

where

$$A = \sqrt{\frac{8}{ht_a}\frac{G_a}{E}} \quad \text{and} \quad \beta_b = \left[1 + \frac{9}{(AL)^2} - \frac{9}{(AL)^3}\tanh(AL)\right]$$

In (3) and (4) all terms are as defined previously except that A and β_b are new parameters related to the specimen geometry and to the properties of the adhesive and adherends. (It should be noted that, in this development, the adhesive thickness is taken as t_a rather than $2t$ as in Moussiaux.)[3] Details of these results can be found in Reference 7 and are being published separately. As shown in Reference 7, optimum beam dimensions are easier to obtain using equations (3) and (4) rather than equations (1) and (2). In either case, it is possible to optimize the beam dimensions such that the shear stress in the adhesive layer is independent of adhesive and adherend moduli and the shear stress can be made to be constant over a large portion of the beam. Using the formulation given in equation (3), a symbolic equation solver such as MATHCAD easily gives the stress distribution along the length of the beam. Figure 2 shows the resulting stress distribution for aluminum bonded with both epoxy and polyurethane as obtained for a typical set of dimensions.[7] As can be seen, the shear stress is uniform over a large portion of the beam for an epoxy adhesive but not for a urethane adhesive (using the same dimensions as in the epoxy beam). The latter could be made more uniform with a different set of dimensions. Moussiaux[3] obtained similar results and verified his calculations with a finite element program. Fior[4] experimentally verified Moussiaux's solution and used a finite element program to examine a number of different loading cases. She showed little difference resulted if a concentrated load of 2P was used only on top of the beam as

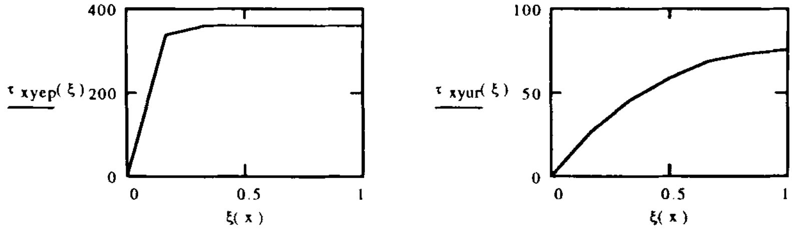


FIGURE 2 Distribution of shear stress along aluminum beam bonded with epoxy (left) and urethane (right).[7]

opposed to a load of P on both top and bottom of the beam as shown in Figure 1. This is important if a three-point bend beam is used rather than a cantilever. The revised solution[7] for shear stresses and deflections given by equations (3) and (4) is shown to give results only slightly different from those of Moussiaux[3] and Fior[4]. Nondimensional plots of a comparison of the two solutions for the shear stress in the adhesive layer and the beam deflection is given in Figures 3 and 4.

DMTA Studies of a Fixed/Fixed Beam

Li, Dickie and Morman[5] extended Moussiaux's solution to the case of fixed/fixed viscoelastic beam (Figure 5) under steady state oscillations using Fourier transforms. The fixed/fixed beam was chosen as it is the required geometry in the DMTA used in their experimental program. The DMTA is designed to give storage and loss moduli and/or damping behavior for monolithic polymeric beams and these quantities are

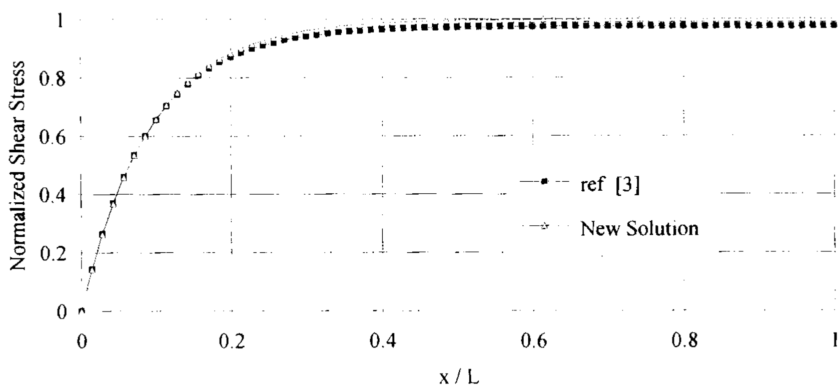


FIGURE 3 Comparison of old and new solution for adhesive shesar stress variation over the beam length.

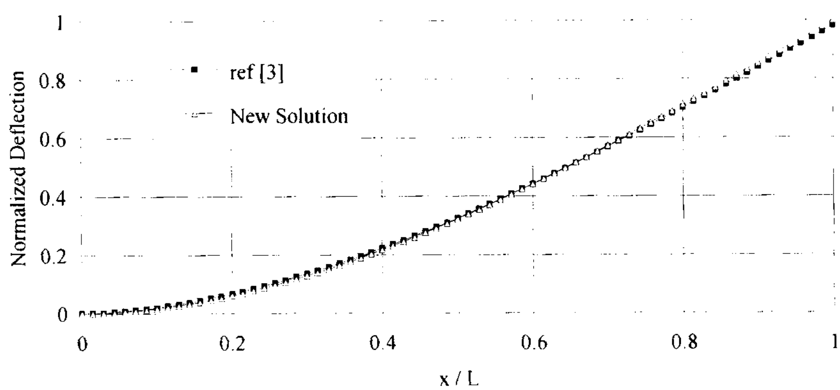


FIGURE 4 Comparison of old and new solution for beam deflection variation over the beam length.

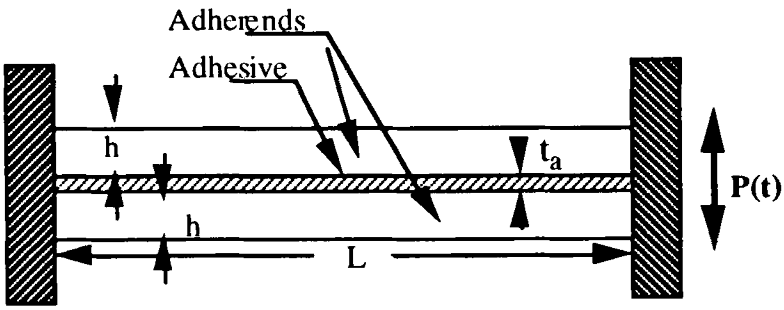


FIGURE 5 Adhesively-bonded fixed/fixed beam in steady state oscillation.

indicative of the polymer investigated. However, DMTA output for a bonded beam will be composed of contributions from both the adherend and the adhesive bond and, therefore, will not give a definitive number for the moduli and damping ratio of only the bonded adhesive layer. The unique feature of the viscoelastic solution of Li, Dickie and Morman[5] is that it does provide a method by which unique moduli and damping properties of the adhesive can be separated from those of the total beam. A detailed description of the procedure is given in Reference 6. It is appropriate to note that the simplification afforded by enforcing continuity at the interface, rather than the middle of the adhesive layer, will result in simplified procedures to obtain adhesive viscoelastic properties using the DMTA. Such efforts are in progress and will be reported subsequently.

Humidity and Corrosion Studies

Li, Dickie and Morman[5] used a DMTA (Polymer Laboratories, Amherst, MA, USA) with a standard driving unit to determine dynamic viscoelastic properties of an electro-galvanized steel (EGS) beam, an epoxy beam and a bonded EGS/epoxy beam each with a length of 14 mm. The geometry and loading of the latter were the same as those shown in Figure 5. Testing frequencies for each beam were 0.1, 1.0 and 10 Hz with an end displacement of 16 μm. Temperatures were scanned from −50°C to 200°C at a rate of 1.5°C/min. Figure 6 shows the storage modulus, E', and the damping factor, $\tan \delta$, obtained for a frequency of 1.0 HZ.

The storage modulus and damping factor for the EGS beam are independent of temperature and are equivalent to the expected values for electro-galvanized steel. The storage moduls and damping ratio of the epoxy beam do vary with temperature and show a glass transition temperature, T_g, at about 73°C. As might be expected, the storage modulus and damping ratio of the bonded EGS/epoxy beam also vary with temperature and indicate essentially the same transition temperature. The EGS/epoxy storage modulus varies less than that of the epoxy but the damping ratio variation is almost the same. Tests of EGS/epoxy beams immersed in water at different temperatures were performed and the different conditions gave different storage modulus and damping ratio traces with varying temperature. Also, a variety of substrates were

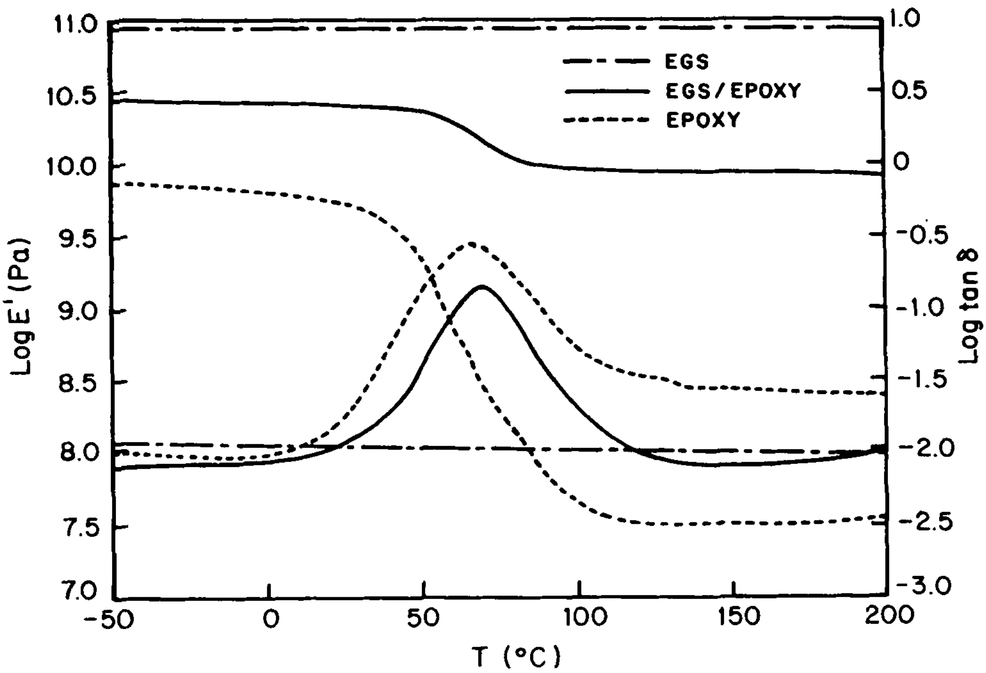


FIGURE 6 DMTA storage modulus and damping factor variation with temperature for a frequency of 1.0 Hz. Reproduced from Reference 5.

tested, again showing that different conditions gave different storage modulus and damping ratio traces.

In a series of studies, Dickie *et al.*[8,9] have explored the effect of humidity and corrosion (alternate periods of salt water and humidity exposure) on the residual bond strength of stressed and unstressed EGS/epoxy bonded lap joints. They found losses in residual bond strength of about 20% after 10 weeks of humidity exposure. EGS/epoxy lap specimens exposed to a corrosive environment were found to exhibit a similar decrease in residual bond strength. Examination of failure surfaces of exposed lap shear specimens showed a central region of cohesive failure surrounded by a peripheral region of apparent interfacial failure; the amount of interfacial failure increased with exposure time.

DAMAGE STUDIES USING DMTA

As previously indicated, the adhesive layer of an end-loaded bonded cantilever beam with appropriate dimensions and adhesive/adherend moduli will be in a state of pure shear. For the case of elastic deformations, the adhesive shear modulus can be obtained by measuring the beam deflection.[3,4] A pure state of shear stress will also be present in the adhesive layer of a bonded fixed/fixed beam and viscoelastic adhesive shear moduli can be obtained using standard DMTA procedures as demonstrated by the results shown in Figure 6. For monolithic polymer beams, it is known that the loss modulus (or $\tan\delta$) is a measure of energy dissipation. For bonded metallic beams, the loss modulus (or $\tan\delta$) will also be a measure of energy dissipation of the total beam including the adhesive layer and/or bonding mechanisms. Should the bond (either adhesive and/or interphase) become damaged due to excessive load, fatigue, moisture of corrosion, it would seem likely that dissipation mechanisms or loss modulus and $\tan\delta$ would change. If so, the procedures outlined earlier and given in detail in References 1–7 could provide an estimate of the degree of damage and perhaps lead to quantification of the remaining life or strength. To confirm this conjecture, a series of DMTA tests were undertaken on beams with simulated flaws and on beams taken from lap specimens which had been exposed to humidity and/or corrosion for extended periods.

EXPERIMENTAL PROCEDURES

Electro-galvanized steel (E60 EZG 60G, from Advanced Coating Technologies, Inc.) platens with dimensions of 0.80 mm × 55 mm × 100 mm were first wiped clean with acetone; then a one-component paste epoxy adhesive (Terokal 4520–34, from Teroson, Inc.) was spread on both mating surfaces with a spatula. The platens were pressed together so as to avoid the inclusion of air bubbles. Adhesive thicknesses were controlled using 0.35 mm metal shims. The platens were clamped and the resulting sandwich was placed in a convection oven and cured for 30 minutes at 180°C. Specimens to be tested were machined from each platen. Bond lines were examined for air bubbles and for uniform thickness. Similarly, platens were made with release paper inclusions. The resulting beams contained centrally-embedded interfacial flaws of 0%, 25%, 50%, 75% and 100%. For the 100% simulated flaw, specimens were tested with a cured but unbonded epoxy layer (but no release paper) between the adherends and with a cured but unbonded epoxy layer (with release paper on each side) between the two adherends. Control samples of metal and epoxy alone were tested to determine if behavior similar to that given in Figure 6 was obtained. It should be noted that the adhesive tested was different from that shown in Figure 6 but similar results were obtained.

Standard lap shear specimens (100 mm × 32 mm, with a 12.7 mm overlap) of electrogalvanized steel were prepared by the same procedure as above except 0.25 mm glass beads were used to control the bond line thickness. These unstressed specimens were exposed to either a pure humidity environment or to a corrosive environment. The humidity environment was 90% RH at 50°C. The corrosive environment was a 24 hr cycle composed of a 15 minute immersion in a 5% aqueous NaCL solution followed by a 105 minute ambient temperature drip dry and 22 hours of exposure to humidity of 90% RH at 50°C. On weekends the specimens were only exposed to the humidity environment so that only 5 corrosion cycles occurred per week. For comparison purposes between humidity and corrosion, each week is defined as five cycles.

Specimens taken from platens with simulated flaws and specimens taken from lap shear coupons exposed to humidity and/or corrosion were tested in a DMTA (Polymer Laboratories, Amherst MA) with a standard driving unit to determine dynamic viscoelastic properties of bonded EGS beams. The geometry and loading were the same as those shown in Figure 3 and in all cases specimen dimensions were ~ 1.8 mm × 2.5 mm × 32 mm with a distance of 19 mm between clamps. Testing frequencies for each beam were 1.0 and 10 Hz with an end displacement (peak-to-peak) of 16 μm. Temperatures were scanned from − 50°C to 200°C at a rate of 1.5°C/min.

Humidity and corrosion specimens needed for the DMTA were obtained by sectioning the lap coupons. Specimens were taken only from the edge of the overlap (i.e., at the re-entrant corner) with the long dimension, 32 mm, transverse to the coupon (see Figure 7).

DISCUSSION

Simulated Damage Results

Data similar to those given in Figure 4, including E', E'', and tan δ, were obtained for all specimens for each frequency tested. Figures 8 and 9 show the raw storage and loss modulus data obtained for the beams with embedded flaws. As may be observed, both the storage and loss moduli change substantially with the inclusion of simulated flaws. However, it is important to note that the vertical scale for each figure is greatly expanded. For this reason, the loss or damping data outside the transition zone contains a large amount of noise and is not considered to be reliable. Quite obviously, the storage modulus below the T_g and the loss modulus at the T_g change in a predictable manner with changes in the length of the simulated flaw. Also, it is noticed that the T_g, as given by the loss modulus, appears to decrease with increasing simulated flaw length. This may be an effect similar to the apparent shift in T_g of dispersed phases in particulate blends.[10] Figure 10 shows the variation of change in storage, loss modulus, tan δ, and T_g with increasing amounts of debonding. The storage modulus is taken at a temperature of 25°C from Figure 8, while the loss modulus, tan δ and T_g are taken at the point of maximum loss modulus from Figure 9. Two data points are shown

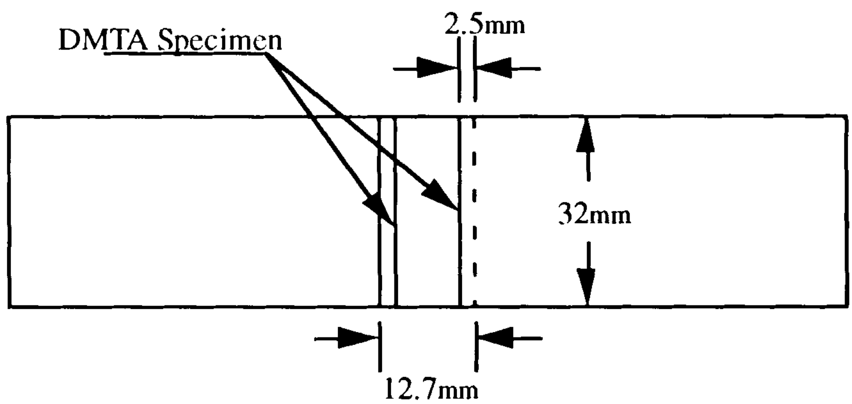


FIGURE 7 Schematic of DMTA specimens taken from lap coupons.

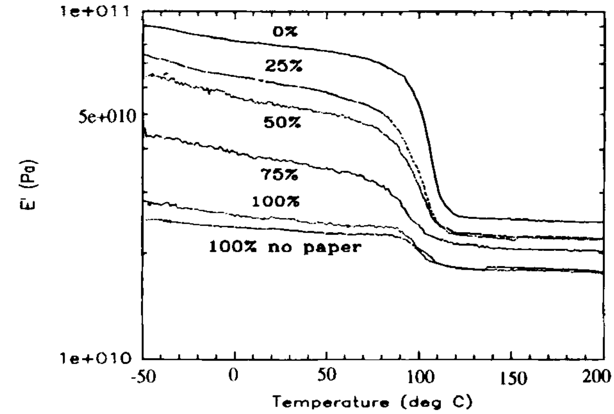


FIGURE 8 Typical storage modulus as a function of temperature for an EGS/Epoxy bonded beam containing simulated flaws. (The percentage shown represents the length of the simulalted flaw varying from no flaw, 0%, to a flaw the length of the beam, 100%).

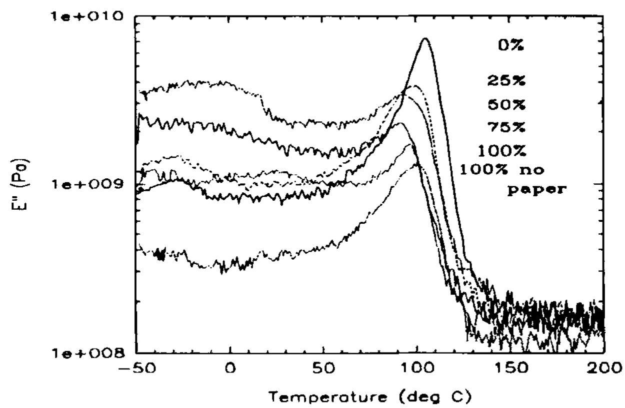


FIGURE 9 Typical loss modulus as a function of temperature for an EGS/Epoxy bonded beam containing simulated flaws. (The percentage shown represents the length of the simulated flaw varying from no flaw, 0%, to a flaw the length of the beam, 100%).

for the 100% non-bonded case in Figure 10. One is for the case of a cured epoxy strip surrounded by release paper sandwiched between the two adherends and the other is for a cured (but not bonded) epoxy strip sandwiched between the two adherends. This tends to show that friction is not a serious factor. Figure 10 clearly shows that the DMTA procedure can discriminate between different amounts of simulated flaws. Therefore, it would seem that the DMTA might be used to detect various amounts of hidden damage due to fatigue, moisture, corrosion or other factors. The following section presents results from our study of possible damage due to humidity and corrosion.

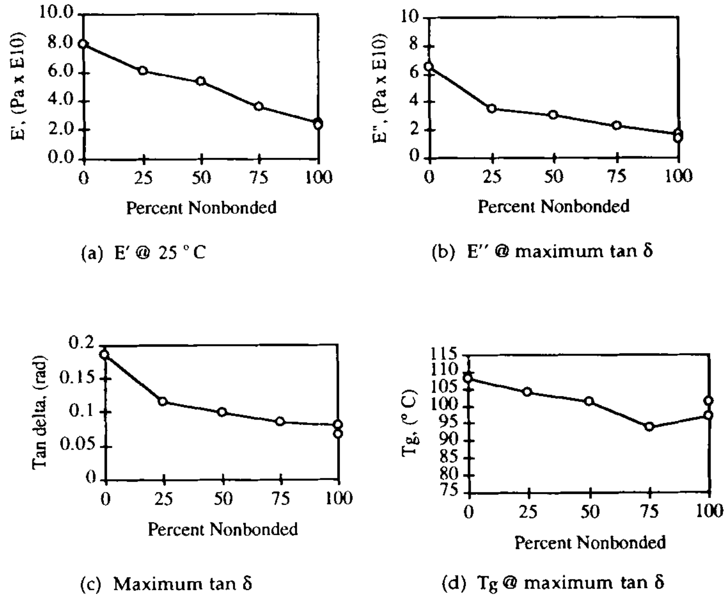


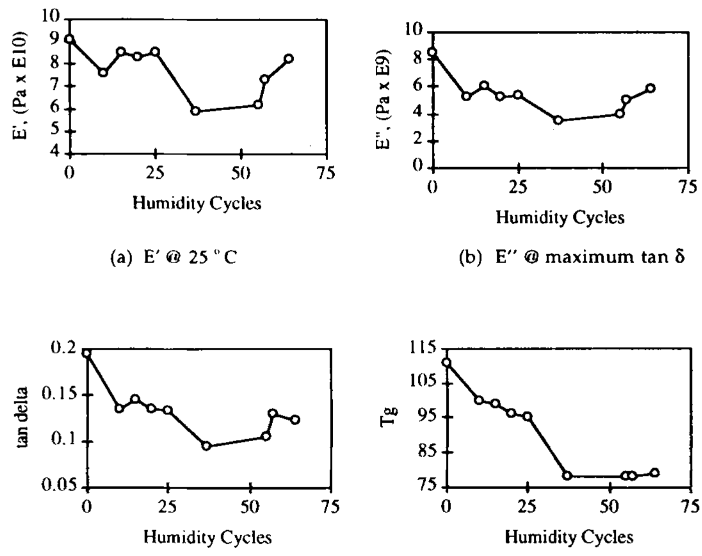
FIGURE 10 Variation of storage modulus, loss modulus, tan δ and T_g with length of simulated flaw for an EGS/poxy beam.

Humidity and Corrosion Results

As indicated earlier, non-stressed single lap specimens were exposed to humidity or corrosive environments over various periods of time. DMTA specimens were sectioned from the lap joints after exposure as discussed previously and as shown in Figure 5. DMTA data similar to that of Figure 8 and 9 was obtained. The results for the variation of storage modulus, loss modulus, tan δ and T_g with humidity and corrosion exposure are shown, respectively, in Figures 11 and 12. The data shown represent exposures as long as approximately 60 days.

The effects of humidity exposure are shown in Figure 11. Significant changes in all variables were observed with increasing exposure. Strangely, the storage modulus of specimens exposed for over one month is greater than that of some of the specimens exposed for shorter periods of time. The reason for this phenomenon is not known but may simply reflect specimen-to-specimen variation in the course of the interfacial degradation process. Obviously, more tests to assess variability should be conducted. Loss modulus and tan δ appear to show similar behavior while the apparent T_g changes the most over the range of data. On the basis of the results in Figure 11, DMTA testing of exposed samples does appear to provide an indication of damage as a result of humidity exposure.

Changes due to corrosion exposure are shown in Figure 12. The failure process in corrosion has been shown to be more complicated than for humidity with evidence for the establishment of an anodic corrosion site in the adhesive-metal interface[11] and, in the case of specimens exposed under load, an initial gradual loss of strength giving way



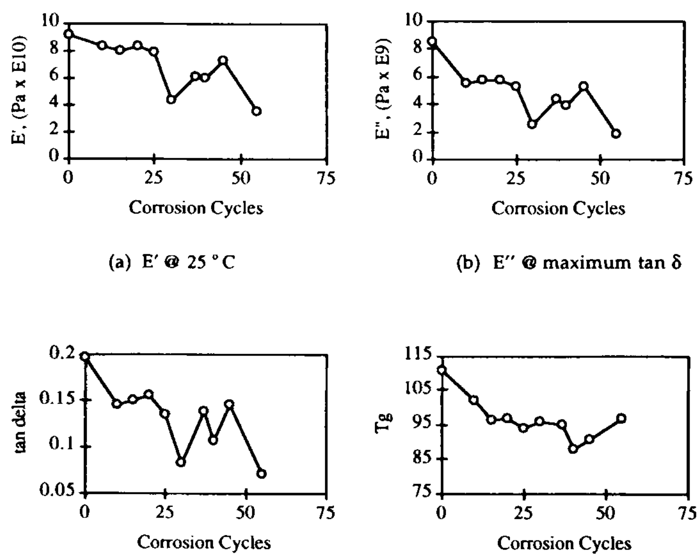
(a) E' @ 25 °C

(b) E" @ maximum tan δ

(c) Maximum tan δ

(d) Tg @ maximum tan δ

FIGURE 11 Variation of storage modulus, loss modulus, tan δ and T_g with humidity.



(a) E' @ 25 °C

(b) E" @ maximum tan δ

(c) Maximum tan δ

(d) Tg @ maximum tan δ

FIGURE 12 Variation of storage modulus, loss modulus, tan δ and T_g with corrosion.

to a rapid and possibly auto-catalytic process leading to spontaneous rupture.[8,9] The initial changes in the dynamic mechanical properties are, however, essentially the same as in humidity exposure. More variability is observed at longer exposure times, but a systematic change of all measured properties is apparent suggesting again that the proposed DMTA method would discriminate changes in mechanical properties with exposure.

SUMMARY AND CONCLUSIONS

A new bonded double cantilever beam (or three-point bend) specimen which provides a simple method of determining adhesive properties through the measurement of strains within the bondline or by measuring end point deflections has been presented. The previous solution developed by Moussiaux[3] using continuity at the beam centerline has been contrasted to a new solution enforcing continuity at the adhesive-adherend interface. The latter leads to less complicated shear stress and deflection equations and should allow optimum specimen dimensions to be found with more ease.

Methods to measure strains in the bondline were not discussed but a new digital imaging micro-measurement system (DIMMS) is under development that will make such measurements possible, perhaps even close to the interface. Details of this procedure are being published separately.

A steady-state, forced-vibration solution developed by Li, Dickie and Morman[5,6] has been discussed and earlier work by the same authors has been presented showing that use of a DMTA on a bonded sandwich beam gives information on the viscoelastic properties of the adhesive bond. The same concept has been used to evaluate simulated damage within the bondline. Also, the DMTA approach has been used successfully to evaluate changes in the viscoelastic properties of a bonded beam due to exposure to moisture and corrosion. From the results, it does appear that DMTA studies on bonded beams may allow the determination of progressive damage due to fatigue, moisture, corrosion or, perhaps, to other environmental parameters. Using a solution to the fixed/fixed beam modified in a manner similar to that for the static case should allow the definitive quantification of the progressive damage state within a bonded beam. However, it should be noted that it may be necessary to build special DMTA equipment that is more accurate and sensitive to small changes in damping behavior.

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