A PRACTICAL TEST METHOD FOR MODE I FRACTURE TOUGHNESS OF ADHESIVE JOINTS WITH DISSIMILAR SUBSTRATES

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ABSTRACT

A practical test method for determining the mode I fracture toughness of adhesive joints with dissimilar substrates will be discussed. The test method is based on the familiar Double Cantilever Beam (DCB) specimen geometry, but overcomes limitations in existing techniques that preclude their use when testing joints with dissimilar substrates. The test method is applicable to adhesive joints where the two bonded substrates have different flexural rigidities due to geometric and/or material considerations. Two specific features discussed are the use of backing beams to prevent substrate damage and a compliance matching scheme to achieve symmetric loading conditions. The procedure is demonstrated on a modified DCB specimen comprised of SRIM composite and thin-section, e-coat steel substrates bonded with an epoxy adhesive. Results indicate that the test method provides a practical means of characterizing the mode I fracture toughness of joints with dissimilar substrates.

KEYWORDS: Adhesive Joints, Fracture Toughness, Dissimilar Materials

1. INTRODUCTION

Under the auspices of the United States Council for Automotive Research (USCAR) and the Federal Government, considerable research efforts are being undertaken to increase fuel efficiency and decrease harmful emissions of automobiles. Two obvious approaches to accomplish these goals are (1) to increase the efficiency of the engine and drivetrain and (2) to reduce the weight of the automobile. Research activities are underway to improve engine efficiency through redesign or replacement with, for example, fuel cells or hybrid systems. On the other hand, the traditional approach taken to reduce vehicle weight has been by reducing vehicle size. Obviously, consumer concerns over comfort and safety, whether real or perceived, limit the opportunities here. Consequently, the emphasis has switched to lightweight materials as a means for saving weight without sacrificing vehicle size. Many potential alternative materials are being considered. The most prevalent materials include aluminum, polymer composites, and high strength steels.

One challenge that arises when considering alternative materials is joining. For example, polymer composites do not perform well, in general, when mechanically fastened and obviously cannot be welded like steel. Fortunately, structural adhesive bonding facilitates the usage of these materials. In fact, adhesive bonding has attributes that make it an attractive alternative for materials that can be joined by traditional methods. Adhesive joints are lightweight, have lower stress concentrations than mechanical fasteners, and reduce the potential for corrosion. They provide for added design flexibility, increasing the opportunity for part consolidation as well as for joining dissimilar materials.

Seemingly at odds with the potential benefits of structural adhesives is their current usage. Most adhesives in production vehicles today are either for non-structural or semi-structural applications. This stems from a lack of understanding and predictive methodologies for the long-term performance of the bonds.

In 1992, the Oak Ridge National Laboratory began a cooperative research effort with the Automotive Composite Consortium's (ACC's) joining work group to develop enabling technologies that overcome hurdles in adhesive bonding of current and future automotive materials. The tasks of this research effort were reasonably broad. However, one of the primary goals was to establish a set of standardized test practices for evaluating automotive-grade adhesives with a variety of substrates. These test practices are intended to be used by automotive companies and suppliers to provide material property data for designers. Fracture-based test methods were chosen as the initial focus for the test development activities. This paper describes a relatively focused aspect of that work – procedures that were developed to characterize the mode I fracture toughness of adhesive joints where the two bonded substrates have different flexural rigidities due to either geometric and/or material considerations.

2. MODE I FRACTURE TESTING

2.1 The Double Cantilever Beam Test Mode I fracture toughness defines a material's resistance to crack propagation while under tensile forces normal to the crack surface. Several standard methods exist for testing composites, metals and plastics in mode I fracture. For composite delamination and adhesive joint studies, most methods are based on the double cantilever beam (DCB) specimen [1]. In its simplest configuration, the DCB specimen geometry consists of a uniform thickness rectangular specimen with a crack starter at one end. The specimen is visualized as two cantilevered beams, fixed at the crack tip. Opening load is introduced to the specimen, through piano hinges or end blocks with clevis holes, by specifying a constant-rate opening displacement. As the crack extends, the compliance of the specimen increases. The mode I fracture toughness can be determined from load, displacement, and crack length measurements according to the relationship

$$G_{lc} = \frac{P_{cr}^2}{2b} \frac{dC}{da}$$
[1]

where G_{lc} is the mode I critical energy release rate, P_{cr} is the load required to extend the crack, b is the specimen width, a is the crack length measured from the load line, and C is the compliance defined as the load-line deflection divided by the load. Several data analysis schemes can be used for determining G_{lc} including elementary beam analysis, area method, and empirical [1]. For each data reduction scheme, multiple measurements for fracture toughness can be obtained from each specimen.

2.2 Limitations of the DCB for Automotive Materials The approach described above has been successfully applied in numerous studies involving aerospace-grade composites and adhesive joints. These test methods, however, have limitations that preclude their use for testing adhesive joints comprised of certain automotive materials that are of interest here. Two specific limitations related to substrate failures and dissimilar substrates were addressed in this work.

2.2.1 Substrate Failures As opposed to those in aerospace, typical low-cost composites used in the automotive industry have higher void content and lower fiber volume fraction. Accordingly, they have a lower flexural rigidity that leads, in part, to substrate damage when tested using "standard" DCB geometries (Figure 1). Similarly, DCB specimens comprised of thin-section sheet metal will generally deform plastically prior to or during crack extension. In either case, inelastic contributions to the energy release rate calculation are present and consequently erroneous toughness values will be obtained. The propensity for inelastic effects to occur can be determined from the material and geometry of the substrate, as discussed below.



Figure 1. Flexure failure of the substrate during mode I fracture tests of SRIM composites with traditional DCB geometry.

2.2.1.1 Flexural Stresses in Substrates As opening forces are applied to the DCB specimen, the unbonded portion develops flexural stresses as the strain energy in the specimen increases. If the unbonded portion is assumed to be rigidly fixed at the crack tip, elementary beam theory can be used to determine the flexural stresses, as well as the strain energy release rate, as a function of the applied load. As the load increases the specimen may deform elastically, the crack may extend, or the substrates develop damage (or plasticity in the case of metal substrates). Comparing the critical load for the latter two cases provides an estimate of the minimum required substrate height that will ensure crack extension before substrate damage which is given by:

$$h > \frac{6E(G_{lc}^{est})}{\sigma_{critical}^2}$$
[2]

Where *E* is Young's modulus of the substrate, $\sigma_{critical}$ is the stress at which damage or plasticity in the substrate occurs, and G_{lc} is the mode I critical energy release rate. Since G_{lc} is the property to be determined by the test, it must be estimated from the best available data to determine the height requirement. If the height of the substrate is insufficient, as is the case of the materials reported herein, then the relation suggests that the substrate height be increased. Unfortunately, due to processing limitations, this is not a practical solution for

many of the materials of interest. Additionally, the modulus, critical stress, and critical strain energy release rate are not parameters that can be selected for a given material system.

2.2.2 *Dissimilar Substates* One of the chief advantages of adhesive bonding is the ability to join dissimilar materials. The potential exists for bonding steel to aluminum, steel to polymer composite, or aluminum to steel. However, when the substrates have different flexural rigidities, from geometric and/or material differences, then the DCB specimen does not deform symmetrically and the tensile forces are no longer normal to the crack surface. Consequently, the problem becomes one of mixed-mode (i.e., opening and shearing) fracture. Although this may provide useful data, assuming inelastic effects are avoided and mode-mix determined, it does not permit determination of the mode I fracture toughness which is needed for complete characterization of the fracture envelope.

3. TEST METHOD MODIFICATIONS

3.1 Backing Beams To circumvent the problem of substrate failures, backing beams made of steel or aluminum can be bonded onto the substrates. Equation 2 can be used, with modification to account for the "composite" backing/substrate beam to ensure the stress in the outer fibers of the backing beam does not exceed the yield stress. However, from the authors' experience, aluminum or steel backing beams of 12.7 mm height are generally sufficient and practical. In the unlikely event that the backing beams exhibit any permanent deformation after the test, the data is suspect and Equation 2 should be used to estimate the height required.

Additional advantages arise with the use of backing beams. The resulting displacements are small which minimizes geometric nonlinearities that are often encountered in DCB tests. Consequently, problematic correction factors [2-4] can be avoided. Anticlastic bending effects can be minimized resulting in straighter crack fronts [5]. The backing beam can be machined with clevis holes for convenient application of loads.

3.2 Compliance Matching for Dissimilar Substrates A practical approach to achieving a mode I fracture test for specimens with dissimilar substrates is afforded by the use of backing beams. As stated above, DCB specimens with dissimilar substrates are inherently mixed-mode loading specimens due to non-symmetric flexure of the unbonded (i.e., cracked) portion of the substrates. From a physical standpoint it is argued here that if the heights of the two backing beams are chosen such that symmetric bending is achieved, then conditions for mode I are established. Geometrically, this suggests that the cantilevered portion of each substrate must have the same load-line displacement during loading. Consequently, each portion contributes equally to the work done during the test since, from equilibrium, the same forces are acting on each substrate. The deflection of a cantilever beam, of length a, with a concentrated load, P, at the free end is given by

$$\delta = \frac{Pa^3}{3(EI)}$$
[3]

where *EI* is the flexural rigidity. Clearly, for each substrate to have the same deflection requires both must have the same flexural rigidity as shown in Equation 4.

$$(EI)_{top \ beam} = (EI)_{bottom \ beam}$$
[4]

3.2.1 Determination of the Backing Beam Heights Elementary mechanics of materials can be used to determine the requisite heights in the following manner.

- <u>Step 1</u> Choose the height for one of the backing beams.
- <u>Step 2</u> Using Equation 5, determine the location of the neutral axis, \overline{Y} , of the composite beam which consists of the backing beam with known height and its corresponding substrate (see Figure 2).



Figure 2. Composite beam consisting of backing beam rigidly attached to a substrate

<u>Step 3</u> Calculate the value of *EI* for the composite beam.

$$EI = \frac{wE_{s}h_{s}^{3}}{12} + wE_{s}h_{s}\left(\overline{Y} - \frac{h_{s}}{2}\right)^{2} + \frac{wh_{b}^{3}E_{b}}{12} + wE_{b}h_{b}\left(h_{s} + \frac{h_{b}}{2} - \overline{Y}\right)^{2}$$
[6]

<u>Step 4</u> Determine the height of the backing beam required for the other substrate which will result in a second composite beam of equal flexural rigidity as the first composite beam. This requires iteration on Equations 5 and 6 to solve for h_b with a common spreadsheet program.

3.2.2 Accuracy of the Compliance Matching Approach The compliance matching approach is a practical approximation for minimizing the shearing contribution that is inherent in specimen's with dissimilar substrates. To assess its validity, a finite element analysis was conducted to determine the mode-mix as a function of substrate height for the simplified case — steel bonded to aluminum. The results of this analysis are shown in Figure 3. Using the compliance matching approach, the ratio of the height of the steel to aluminum was calculated as 0.693 for pure mode I. Although the finite element analysis results show the ratio for pure mode I loading to be closer to 0.4, the amount of mode II for a ratio of 0.693 is less than 5 percent. This was considered acceptable.

4. EXPERIMENTS

4.1 Materials The goal of this research effort was to develop testing procedures applicable to a broad range of automotive materials, not to characterize specific materials. Therefore to evaluate the test method, materials were chosen that are believed to represent some of the

most challenging substrates — a standard e-coated, thin-section steel and a glass-fiber, polymer composite. The e-coat, used for corrosion resistance, has a Ford Motor Co. designation of J28. The composite was made from a continuous-strand mat preform infiltrated with an isocyanurate (Dow MM364) resin by the structural reaction injection molding (SRIM) process. The composite is taken as transversely isotropic, although slight differences in modulus are observed in the two principal directions. Fiber volume content is approximately 25 percent. The adhesive chosen for this study was a non-commercial thixotropic epoxy. Aluminum bar was selected for the backing beam material. Dimensions and properties used to determine the backing beam requirements are shown in Table 1.



Figure 3. Finite element analysis depicting mode-mix versus the ratio of substrate heights for a steel-aluminum specimen.

Table 1.	Dimension	and pro	perties of	f substrate	es for b	backing	beam o	determinatio	n
						0			

	Composite Substate	Steel Substrate
Width	12.7 mm	12.7 mm
Height, h	2.54 mm	0.889 mm
Young's Modulus, E	10 GPa	207 GPa

4.2 Backing Beams For convenience, the height of the backing beam for the steel substrate was selected as 12.7 mm. This ensured that the height of the backing beam for the composite substrate will be greater than 12.7 mm, which is considered to be a practical minimum dimension. From Equation 5, the neutral axis, \overline{Y} , for the steel/backing beam combination is 6.06 mm. The flexural rigidity, *EI*, from Equation 6 is 238714 Gpa-mm⁴. The required height

for the aluminum backing beam with the composite substrate which yields the same flexural rigidity is 14.3 mm.

4.3 Specimen Preparation Test samples were prepared by first bonding plates of the SRIM composite and e-coat steel with a teflon-film inserted at one end to provide a crack starter. The size of the bonded panel was nominally 100 mm by 250 mm. The bonded panel was placed in a curing oven for one hour at 150°C for the adhesive to cure. The panel was then sliced into test specimens 12.7 mm by 241 mm, with a crack starter of approximately 25 mm. The trimmed specimens were then bonded to aluminum backing beams, 250 mm long, with an aerospace film adhesive. The resulting test specimen is shown schematically in Figure 4.



Figure 4. Schematic representation of a test specimen with dissimilar substrates and backing beams

4.4 Test Procedures Specimens were mounted in clevis fixtures on a servo-hydraulic test machine. The edge of the specimen was coated with a thin layer of white correction fluid ahead of the crack in order to observe the crack tip extension during loading. Opening forces were applied under displacement control at a rate of 1.27 mm/min until the crack began to grow. For each test specimen, the crack length was visually located prior to testing and monitored for each loading cycle of the test. The crack was allowed to grow approximately 6.35 mm, then the displacement was reversed and the specimen unloaded to approximately 5 percent of the maximum load occurring during crack extension. The extent of crack growth was recorded and the specimen was reloaded for subsequent measurements. Force and displacement data was acquired using a custom LabVIEW application via a National Instruments DAQ card. The process was repeated until the crack growth resulted in separation of the two halves of the specimen. A continuous record of load versus load-line displacement was obtained for each crack extension.

4.4.1 Data Analysis An experimental compliance method using a third-order compliance fit was chosen for this work because it provided the best trade-off between reproducibility and fundamental formulation. The slope in N/mm of the initial linear portion of each loading curve was determined from linear regression. Compliance, *C*, was calculated for each loading cycle and plotted versus crack length. The coefficients for the third order polynomial were determined from internal routines of a commercial spreadsheet program. The change in compliance as a function of crack length, dC/da, was determined through differentiating the polynomial fit. The critical load for each loading cycle was taken as the point at which the load-displacement curve deviates from linearity by approximately 5 percent. The critical energy release rate in mJ/mm² for each loading cycle was then calculated from Equation 1.

5. RESULTS

The specimen exhibited very controlled slow-stable crack growth. The behavior was similar to other mode I experiments conducted on the same set of materials when both substrates were identical. A photograph of the resulting failure surface is shown as Figure 5. Failure

occurred alternately in the e-coat layer of the steel and the resin-rich surface layers of the composite in remarkable periodic fashion. Figure 5 shows that the top half of the specimen has islands of adhesive on the steel substrate, whereas the bottom half has the remaining adhesive on the composite substrate. Bare fibers in the composite are exposed as the resin-rich layer, as well as a few loose fibers, adhere to the adhesive islands on the e-coat substrate. The fracture toughness was essentially constant with respect to crack length as expected since fiber bridging was not significant. The fracture toughness is shown in Figure 6 along with DCB specimens with the identical substrates for comparison. The toughness for the dissimilar specimen is approximately 0.6 mJ/mm², which is slightly higher than the toughness for the steel-steel joint.



Figure 5. Failure surface for mode I fracture of composite-epoxy-steel specimen.

6. SUMMARY

A practical test method for determining the mode I fracture toughness of adhesive joints with dissimilar substrates was developed. The test procedure is based on the use of backing beams to prevent inelastic effects and a compliance matching scheme to establish mode I conditions. The method was successfully demonstrated on specimens comprised of thin-section steel bonded to polymeric composites.

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Figure 6. Fracture toughness as a function of crack length for similar and dissimilar joints. Each condition represents several specimens.

8. REFERENCES

- 1. J.M. Whitney, C.E. Browning and W. Hoogsteden, <u>Journal of Reinforced Plastics and</u> <u>Composites</u>, Vol. 1, 1982.
- 2. E.J. Ripling, S. Mostovoy, and H.T. Corten, Journal of Adhesion, Vol. 3, 1971.
- 3. B. Blackman, J.P. Dear, A.J. Kinloch, and S. Osiyemi, <u>Journal of Materials Science</u> <u>Letters</u>, Vol. 10, 1991.
- 4. J.G. Williams, Journal of Strain Analysis, Vol. 24, 1989.
- 5. D.G. Smith, "Fracture Toughness of Adhesive Joints in Automotive Composites," *Internal Report*, Oak Ridge National Laboratory, August 1993.