FRACTURE TOUGHNESS TESTING OF HIGH-PERFORMANCE ADHESIVES

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ABSTRACT

Bonding metals to composites for lightweight bridge applications places considerable loading and environmental demands on the adhesives. As part of a program to develop and improve the performance of ambient-temperature-curing adhesives for bridge applications, we are attempting to address three primary problems: 1) the low tensile and peel strengths of the ambient-temperature-curing adhesives suitable for such joints; 2) environmental durability, i.e., resistance to water and humidity; and 3) low fracture toughness. So far, any advances in adhesive formulation that have improved one of the properties generally have reduced performance in the others.

Compact tension specimens of both bulk and bonded adhesives were investigated to evaluate potential and practical fracture toughness for various adhesive formulations. Partitioning the load versus load-line displacement curves allowed both the elastic energy released and the plastic energy dissipated to be measured for each increment of crack growth during successive displacements of the specimen. From these measurements traditional elastic-plastic parameters (G and J) can be measured as well as total fracture resistance (I). We have found that I better characterizes an adhesive's resistance to crack growth than the more traditional parameters.

Results of both bulk adhesive and bonded aluminum compact tension testing are presented.

INTRODUCTION

Although adhesive bonding is extensively utilized for large-scale aerospace structures, its use for engineering structures, such as bridges, is much more limited. The primary problem that has restricted wider use of bonding is the limited performance of easy-to-apply adhesive systems. High-performance aerospace applications have evolved using thin, controlled bond-line, high temperature and pressure autoclave cure processes. Unfortunately, low-temperature-curing adhesives that do not require autoclaves have lacked the combination of strength, thermal stability and resistance to environmental degradation.

We have been studying ways to improve the performance of low-temperature-curing adhesives as part of a program to develop light-weight portable bridge structures. This application demands high strength, high resistance to moisture, and retention of properties over a fairly wide temperature range. Initial screening tests of neat adhesive films showed that many epoxy specimens had quite good strengths [>8000 psi (>55 MPa)](1). Resistance to moisture was less common, with most commercial adhesives failing to retain more than 10% of their dry strength after equilibrium high-humidity exposure. Model adhesives developed during the program to test new chemical approaches to moisture resistance showed much better strength retention after humidity exposure, but seemed to be quite brittle. A prior adhesives screening study showed the same results (2).

The fracture behavior of the candidate adhesives was quantified by analyzing the unloading compliance records of compact tension specimens. This adaptation of a standard fracture toughness test used for metallic materials (3) has proven very useful in guiding our research efforts toward toughening adhesive systems having good moisture resistance. The primary advantage of this test for adhesive development/screening is the relatively small and easily constructed test specimen that still yields accurate results. In addition, the elastic energy release rate (G), the plastic energy release rate (I), and the total elastic-plastic energy release rate (J) can be determined by straightforward analysis of the load versus load-line-displacement curve generated by the test. We have studied both neat adhesive and bonded aluminum specimens in this fashion.

EXPERIMENTAL

Half-plan compact tension specimens (B=0.25 in.) were cast from neat resin in an aluminum mold, as shown in Fig. 1. The specimen dimensions are shown in Fig. 2. A crack-opening displacement (COD) gauge was used to monitor load-line-displacement and the specimen was loaded in an Instron 1125 screw machine.

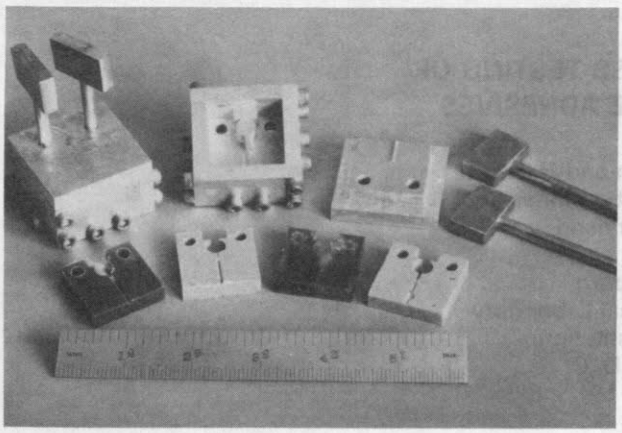


Fig. 1. Aluminum mold and adhesive compact tension specimens.

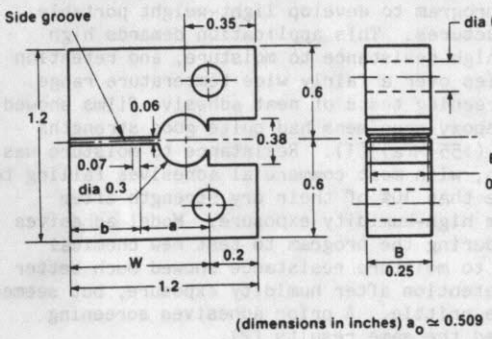
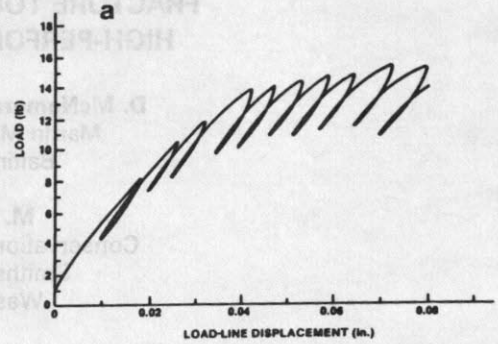


Fig. 2. Dimensions of a 1/2-plan compact tension specimen.

Load-cell and COD data were collected interactively using a personal computer, and the load versus crack-opening displacement curve was plotted simultaneously(4). The specimen was unloaded at appropriate points to determine the compliance and thus the crack length. A typical load versus load-line-displacement curve is shown in Fig. 3. At least three replicate specimens were used for each data point.

Halves of 1/2T-plan compact tension specimens were machined out of aluminum and bonded together with the candidate adhesives. The jig shown in Fig. 4 was used to align the specimens and control their bond-line thicknesses. All the bond-line thicknesses were held in the range of 0.010 - 0.025 in., following typical aerospace bonding practice. Fracture toughness testing was conducted in an identical fashion to the neat adhesive specimens. As before, at least three replicate specimens were tested for each adhesive.



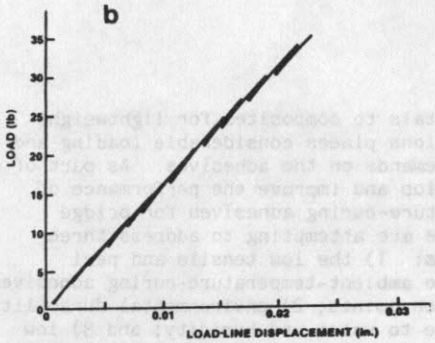


Fig. 3. Load versus load-line-displacement curves obtained from a a) ductile and b) brittle adhesive.

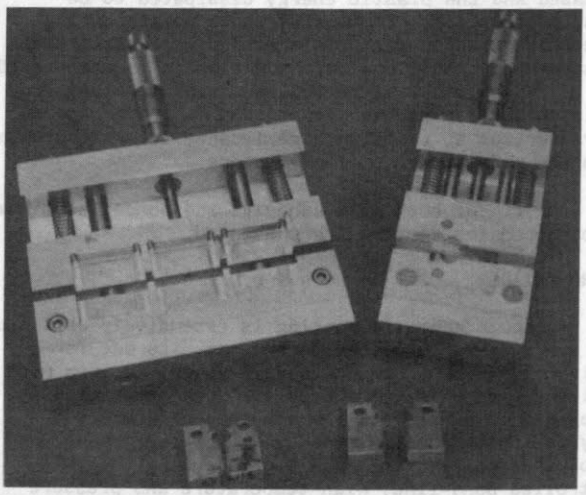


Fig. 4. Alignment jig used to bond aluminum compact tension specimens.

ANALYSIS AND RESULTS

In the unloading compliance test, the area under the load versus load-line-displacement curve represents the fracture energy stored in the specimen as a function of the work done by the external load. It can be used to directly determine J by the simple expression

$$J = 2A/Bb$$

where A = area under the load-COD curve
B = specimen width
b = length of remaining ligament

as derived by Rice for a "one dimensional" specimen such as the compact tension (5). Thus, simple numerical integration of the curve yields values of J which can be directly related to crack length by unloading compliance analysis. Unloading compliance values are used according to the method in ASTM E 668 to derive crack lengths(4). We have compared crack length values measured with a travelling microscope with those calculated by the test method and found good agreement for typical epoxy polymer materials, as shown in Fig. 5.

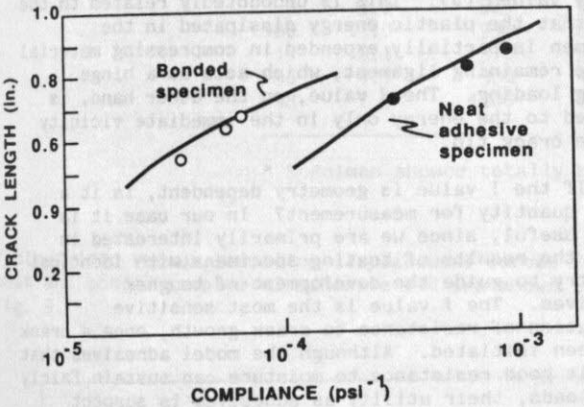


Fig. 5. Compliance versus crack length measurements.

The load-line-displacement record can be further analyzed to separate the elastic energy released and plastic energy dissipated during crack growth. Figure 6 is a diagram of the partioning method used. The area under the curve from one unloading cycle to another is found in two regions, the parallel-sided plastic dissipation region, ΔU_p, and the triangular, elastic energy release region, ΔU_e. These areas represent the energies needed to deform the specimen during crack growth and the potential elastic energy released during crack growth, respectively (6).

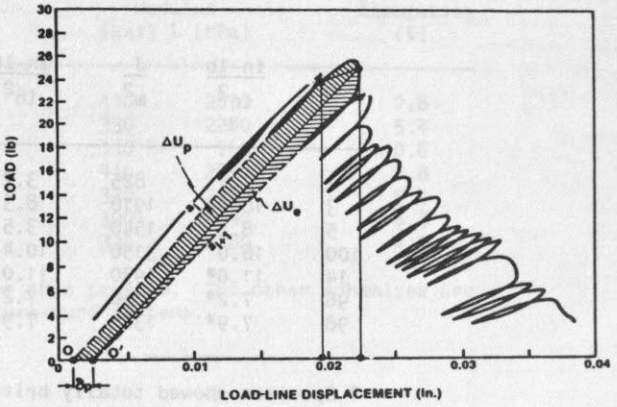


Fig. 6. Elastic plastic partitioning of the load versus load-line-displacement curve.

The increment of crack growth between load-unload cycles is simply the difference in crack length computed for the two cycles. The simple expressions

$$G = 1/B\ (\Delta U_e/\ da)$$

and

$$I = 1/B(\Delta U_p/\ da)$$

are used to calculate G, the elastic energy release rate, and I, the plastic energy dissipation rate, respectively. Values of G calculated in this fashion have shown good agreement with G values determined by the standard method in ASTM E399 (7), as shown in Fig. 7.

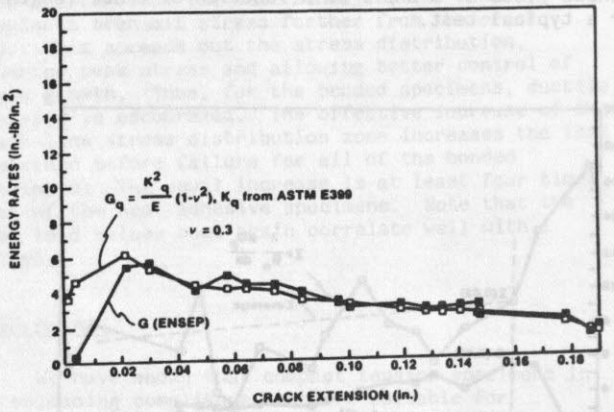


Fig. 7 G values obtained by the energy separation method vs those derived by the method in E399.

Values of J, G, and I calculated in this fashion are shown in Table I for neat adhesive compact tension specimens.

Table I. Fracture Toughness Tests of Neat Adhesive Specimens

	J		G		I		Peak Load	
	$\frac{\text{in-lb}}{\text{in}^2}$	$\frac{J}{m^2}$	$\frac{\text{in-lb}}{\text{in}^2}$	$\frac{J}{m^2}$	$\frac{\text{in-lb}}{\text{in}^2}$	$\frac{J}{m^2}$	lb	N
1	4.7	825	3.7	650	4.2	735	32	142
3	10.9	1910	8.3	1450	5.8	1020	34	151
5	8.8	1540	3.5	615	18.7	3270	14	62
100	18.0	3150	10.4	1820	27.7	4850	58	258
14	11.0*	1930	11.0	1930	0		36	160
96	7.2*	1260	7.2	1260	0		31	138
98	7.9*	1380	7.9	1380	0		35	156

* Specimen showed totally brittle behavior, G set equal to J.

The value of J is that for a crack extension distance of 0.03 in. This roughly corresponds to the criterion used in metallic specimens for J1c, although the large plastic deformations seen in the adhesive specimens makes the determination of the onset of cracking much more difficult. For specimens that failed in a totally brittle fashion (noted with asterisks), J is given as the total area under the curve at failure. In such cases there is no plastic component. Therefore G = J and I = 0. Figure 3 shows load versus load-line-displacement curves typical of a) ductile and b) brittle failures.

Values of G and I are derived from the energy separation areas as described above, except in the cases of brittle failure. The reported values are the average of the measured G and I values as a function of crack length. The G values are fairly constant during crack growth, and the reported values have a standard deviation <= 20%. The I values are more variable, both from point to point and from sample to sample. Samples with stable crack growths also have a standard deviation of <20%, but samples with very little crack extension are less reproduceable. Figure 8 shows plots of G and I as a function of crack length for a typical test.

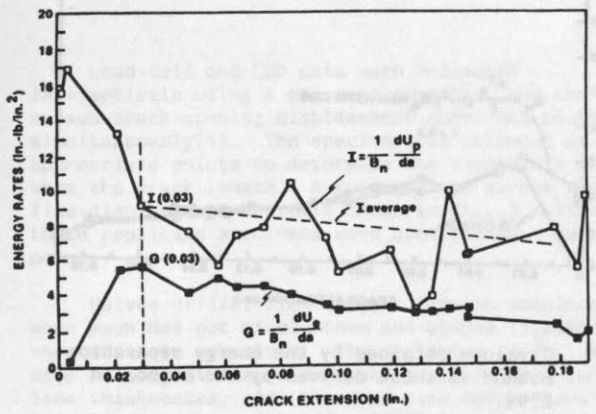


Fig. 8. Elastic energy release rate and absolute plastic energy release rate vs crack extension for adhesive 3.

One feature worthy of note is the lack of correspondence between elongation-to-failure in tensile testing and the fracture toughness values. Table II shows tensile test data from thin films of neat adhesive, measured as described in reference 1. Some of the model adhesive systems show good elongation, e.g., 14 and 96, but exhibit totally brittle fracture.

Another intriguing aspect of the testing is the wide variation in plastic energy dissipation rates (I), which is only partially reflected in the total energy values (J). This is undoubtedly related to the fact that the plastic energy dissipated in the specimen is partially expended in compressing material in the remaining ligament, which acts as a hinge during loading. The J value, on the other hand, is related to the energy only in the immediate vicinity of the crack tip.

If the I value is geometry dependent, is it a valid quantity for measurement? In our case it is quite useful, since we are primarily interested in using the results of testing specimens with identical geometry to guide the development of tougher adhesives. The I value is the most sensitive indication of resistance to crack growth, once a crack has been initiated. Although the model adhesives that exhibit good resistance to moisture can sustain fairly high loads, their utility as adhesives is suspect since they lack any "overload" capacity at present. Without some degree of ductility, the safety margins required to ensure that shock loading never exceeds allowable limits significantly increase the size and weight of joint designs, an unacceptable result. With a sensitive measure of "fracture growth" toughness as a guide, we hope to significantly improve the toughness of our adhesives without severely compromising their strength or moisture resistance.

The fracture testing results for the aluminum bonded compact tension specimens are shown in Table III below. The values of G measured in the bonded specimens were quite similar to the neat adhesive specimen values for the same type of adhesive, and differed significantly only when the crack deviated to the metal/adhesive interface. The values of the I parameter are usually higher for the bonded specimens. Some of this difference may be related to the uncertainty of the measurement. The increased values for adhesives #14 and #96, however, were clearly related to a change in sample behavior.

Table II. Tensile Test Values for Neat Adhesives

	Ultimate Tensile Strength (psi)	(MPa)	Modulus (ksi)	(MPa)	Elongation (%)
1	8960	61.8	410	2830	2.8
3	7650	52.8	330	2280	5.4
5	2160	14.9	110	760	10.6
100	8340	57.5	410	2830	2.8
14*	8830	60.9	330	2280	5.4
96*	10710	73.9	390	2690	6.3
98*	8960	61.8	330	2280	4.9

* Denotes model adhesives developed for this program. The other adhesives are commercially available room-temperature-cure systems.

Table III. Fracture Toughness Test Results for Bonded Specimens.

	J		G		I		Peak Load	
	$\frac{\text{in-lb}}{\text{in}^2}$	$\frac{\text{J}}{\text{m}^2}$	$\frac{\text{in-lb}}{\text{in}^2}$	$\frac{\text{J}}{\text{m}^2}$	$\frac{\text{in-lb}}{\text{in}^2}$	$\frac{\text{J}}{\text{m}^2}$	lb	N
1	4.5	790	4.2	735	19.8	3470	149	663
3	7.1	1240	4.9	860	12.4	2170	138	614
5	3.0	525	3.5	610	11.9	2080	75	334
100	11.0	1930	8.2	1450	40.0	7000	214	952
14	2.1	370	3.8	665	1.2	210	131	583
96	4.1	300	4.0	160	2.7	470	111	494
98	7.9*	1380	7.9	1380	0		111	494

* Specimen showed totally brittle behavior, G set equal to J.

Typical load versus load-line-displacement curves for neat and bonded samples of adhesive #14 are shown in Fig. 9.

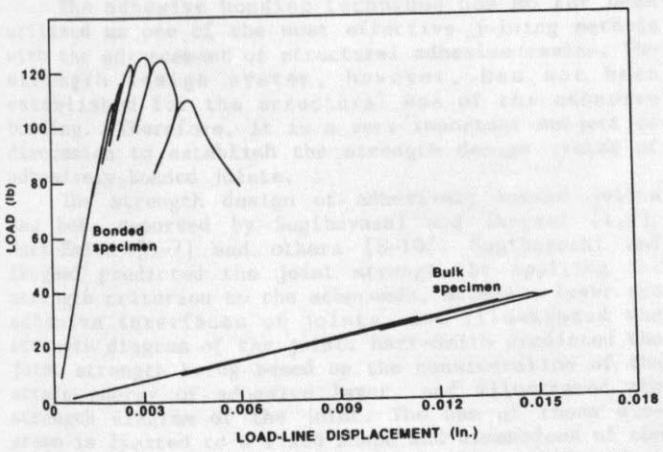


Fig. 9. Load-line displacement curves for neat adhesive and bonded specimen tests using adhesive 14.

These clearly indicate an increase in ductility for the bonded specimen. Since the aluminum adherends are much stiffer than the neat adhesive, the bonded specimens transmit stress further from the crack tip. This spreads out the stress distribution, lowering peak stress and allowing better control of crack growth. Thus, for the bonded specimens, ductile behavior is encouraged. The effective increase of the crack-line stress distribution zone increases the load sustained before failure for all of the bonded specimens. The usual increase is at least four times that of the neat adhesive specimens. Note that the peak load values once again correlate well with I values.

CONCLUSIONS

We have shown that compact tension specimens in an unloading compliance test are suitable for determining fracture toughness parameters for adhesives. Values of the elastic energy release rate, G, are equivalent for neat adhesive and bonded aluminum specimens when the crack proceeds cohesively through the adhesive. The value of the plastic energy dissipation rate, I, although influenced by specimen geometry, provides a sensitive measure of crack growth resistance that is very useful in comparative studies for adhesive bond development.

ACKNOWLEDGEMENTS

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