# Comparison of Laboratory Techniques for Evaluating the Fracture Toughness of Glassy Polymers

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## Synopsis

Several test methods were employed to determine polymer fracture toughness ($\mathcal{G}_{I_c}$, the opening-mode strain energy release rate) at room temperature. The materials used included DGEBA epoxies and those modified by the addition of CTBN elastomers. Double-cantilever beam specimens were used to determine the fracture toughness both of bulk resins and of an adhesive layer bonded between two aluminum half-beams. The adhesive fracture toughness of a 0.025-cm bond was slightly less than the bulk $\mathcal{G}_{I_c}$ value, attributed to the bond thickness effect. Fracture toughness of bulk resins was also evaluated by using both rectangular and round compact tension specimens. The results, when compared with those obtained with the bulk double-cantilever beams, are quite acceptable. The thickness of compact tension specimens, ranging from 0.64 to 1.0 cm, might not give pure plane-strain conditions, and thus some plane-stress contribution to $\mathcal{G}_{I_c}$ should be expected for the tougher materials. Izod impact tests were also carried out to determine sample fracture toughness at high loading rate.

## INTRODUCTION

Many polymeric resins are being considered as matrix materials for fiber-reinforced composites and as high performance adhesives in various aerospace applications. For such applications it is important to understand the mechanical response of the material under loading. Thermosetting materials, such as tetrafunctional epoxies and polyimides, are potential candidates for structural applications because they offer high modulus and strength at elevated temperatures. However, these resins when fully cured are very brittle, since for the intended applications, the materials are designed to perform in their glassy states (i.e., well below $T_g$). It is possible that in many cases the structure would fail primarily by crack propagation, resulting from the growth of flaws and microvoids inherently present in the material because of the processing techniques employed in fabricating structural components. This consideration makes the fracture toughness of glassy polymers a basic mechanical property which needs to be evaluated.

In this report, several laboratory techniques for determining polymer fracture toughness ($\mathcal{G}_{I_c}$, the opening-mode strain energy release rate) were employed for room temperature tests and results were compared. The material used included unmodified epoxies based on bisphenol-A-diglycidyl ether (DGEBA) and those modified by the addition of carboxy-terminated butadiene-acrylonitrile (CTBN) elastomers. These polymer systems were chosen because, while the base epoxy is brittle, the addition of CTBN at various concentrations could lead to two-phase systems exhibiting a wide range of fracture properties. Small CTBN rubber particles dispersed in a brittle matrix are known to toughen the epoxy considerably,[1] and substantial amounts of data are available in the literature for reference and comparison.[2,3]

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## EXPERIMENTAL

In linear elastic fracture mechanics, a specimen is assumed to deform elastically, and the material compliance is a function of the crack length $a$,

$$C_{(a)} = x/P \tag{1}$$

and the specimen geometry. In eq. (1), $x$ is the displacement in the direction of the load $P$. Since the deflection is entirely elastic, the energy absorbed will be

$$\epsilon = Px/2 = P^2C/2 \tag{2}$$

The strain energy release rate $\mathcal{G}$ is defined as

$$\mathcal{G} = \frac{1}{b}\frac{d\epsilon}{da}$$

where $b$ is specimen thickness. As $\mathcal{G}$ reaches a critical value $\mathcal{G}_c$ for fracture to occur,

$$\mathcal{G}_c = \frac{P^2}{2b}\frac{dC}{da} \tag{3}$$

where $dC/da$ is the change in the compliance with the crack length $a$ and can be determined experimentally by measuring $C$ for different "$a$" values. Once an expression for $dC/da$ is available for a given specimen geometry, one can readily determine the critical strain energy release rate or fracture toughness $\mathcal{G}_c$ by measuring the fracture load $P_c$.

### Double-Cantilever Beam

A tapered double-cantilever beam specimen as devised by Mostovoy and Ripling[4] was used to determine the opening-mode fracture toughness $\mathcal{G}_{I_c}$. The explicit form of eq. (3) for this geometry is

$$\mathcal{G}_{I_c} = (4P_c^2/b^2E)[3a^2/h^3 + 1/h] \tag{4}$$

where $E$ is the elastic modulus of the material and $h$ is the beam height measured normal to the crack tip. The test specimen was tapered in such a shape that the quantity in the square brackets in eq. (4) is a constant. Thus, $\mathcal{G}_{I_c}$ becomes independent of crack length $a$ and can be readily determined by measuring $P_c$.

Bulk specimens, as shown in Figure 1(A), were cast and cured in silicone rubber molds. Grooves were then machined along each side to guide the crack through the center path. A saw cut was made at the loading end and a starting crack formed by tapping a fresh razor blade at the end of the saw cut. This procedure led to the formation of a short, "natural" crack having a very small crack tip radius. Specimens were loaded in an Instron with the crosshead moving at 0.125 cm/min until fracture in order to determine $P_c$.

Polymers may also be evaluated for their adhesive fracture toughness using the double-cantilever beam geometry. The specimens were prepared as shown in Figure 1(B). The adherends were 5086 aluminum alloy, cleaned by acid–chromate etching. Because the aluminum has much higher modulus and yield strength as compared to the resin, the adhesive specimens were not tapered as steeply as the bulk specimen of Figure 1(A). The two aluminum half-beams were

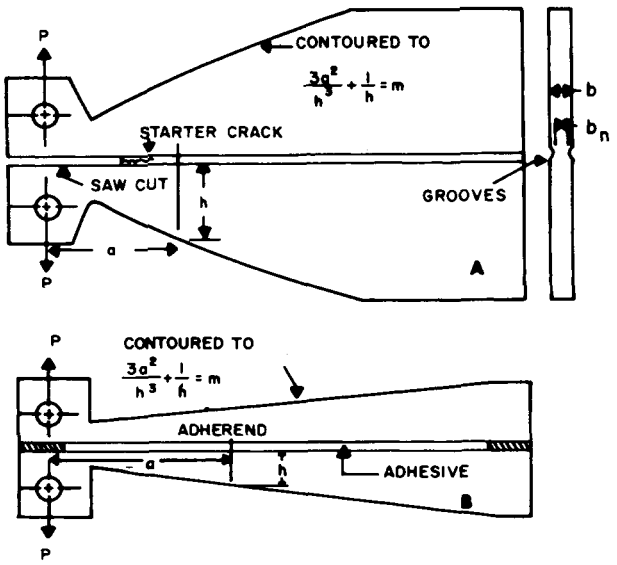


Fig. 1. Double-cantilever beams for bulk and adhesive fracture tests.

clamped together with 0.025-cm Teflon spacers which established the bond-line thickness. One side of the bond was then sealed with pressure sensitive tape and liquid polymer was poured into the resulting cavity for curing. After heat curing, the tape was removed and excess resin machined off. Specimens were tested in a similar way as the bulk specimens. Adhesive $\mathcal{G}_{I_c}$ was calculated using eq. (4) and the measured fracture load $P_c$, except in this case the Young's modulus $E$ for aluminum was used.

## Compact Tension Specimen

The fracture toughness of the bulk polymers was also determined using the standard compact-tension specimen (CTS) shown in Figure 2. Plates were made by casting and then cut into 3.05 × 3.81-cm rectangles. Alternatively, 5.72-cm-diam moldings were made and tested as round compact-tension specimens. The dimension $\overline{W}$ in Figure 2 is 2.5 cm for the rectangular CTS and 3.81 cm for the round ones. Depending on the casting, specimen thickness varied slightly from 0.64 to 1.0 cm. A sharp razor blade was again used to initiate a starting crack before specimens were fractured in the Instron at a rate of 0.125 cm/min. For materials such as CTBN-modified epoxies the deformation zone ahead of the starting crack was of significant size. Consequently, the length of this zone was included as part of the crack length $a$ in calculating $\mathcal{G}_{I_c}$ from the following equation:

$$\mathcal{G}_{I_c} = Y^2 P_c^2 a / E\overline{W}^2 b^2 \tag{5}$$

where $Y = Y(a/\overline{W})$ is a geometrical factor given as

$$Y = 29.6 - 186\,(a/\overline{W}) + 656\,(a/\overline{W})^2 - 1017\,(a/\overline{W})^3 + 639\,(a/\overline{W})^4$$

for rectangular specimens,[5] and

$$Y = 30.0 - 162\,(a/\overline{W}) + 493\,(a/\overline{W})^2 - 664\,(a/\overline{W})^3 + 405\,(a/\overline{W})^4$$

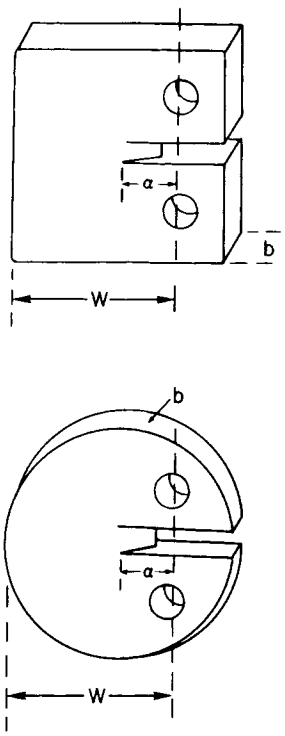


Fig. 2. Compact tension specimens.

for round specimens.[6] The applicability of these relations is normally limited to the range $0.3 \leq a/\overline{W} \leq 0.7$, however.

## Impact Test

The mechanics of impact loading has been examined by Plati and Williams,[7] who showed that from eqs. (2) and (3)

$$\epsilon = \mathcal{G}_c \phi T W \tag{6}$$

where $T$ is the specimen depth in place of $b$ in eq. (3) and $W$ is the specimen width. $\phi = \phi\,(a/T)$ is a dimensionless factor given by

$$\phi = \frac{C_{(a)}}{[dC/d(a/T)]} \tag{7}$$

A graphic representation of $\phi = \phi\,(a/T)$, derived from the results of Plati and Williams,[7] is shown in Figure 3. Equation (6) relates the total energy $\epsilon$ absorbed during an impact failure to fracture toughness $\mathcal{G}_{I_c}$ of the material and may therefore be applied to an impact test.

Standard Izod impact tests were carried out by using an instrumented impact machine. Izod specimens, as shown in Figure 4, were prepared from cast plates of four polymers with different elastomer–epoxy compositions. A razor blade cut was introduced at the bottom of the 45° notch as a starting crack of approximately 0.05 cm depth (Fig. 5). The instrument recorded the impact load and energy as functions of impact time continuously. A typical test record is

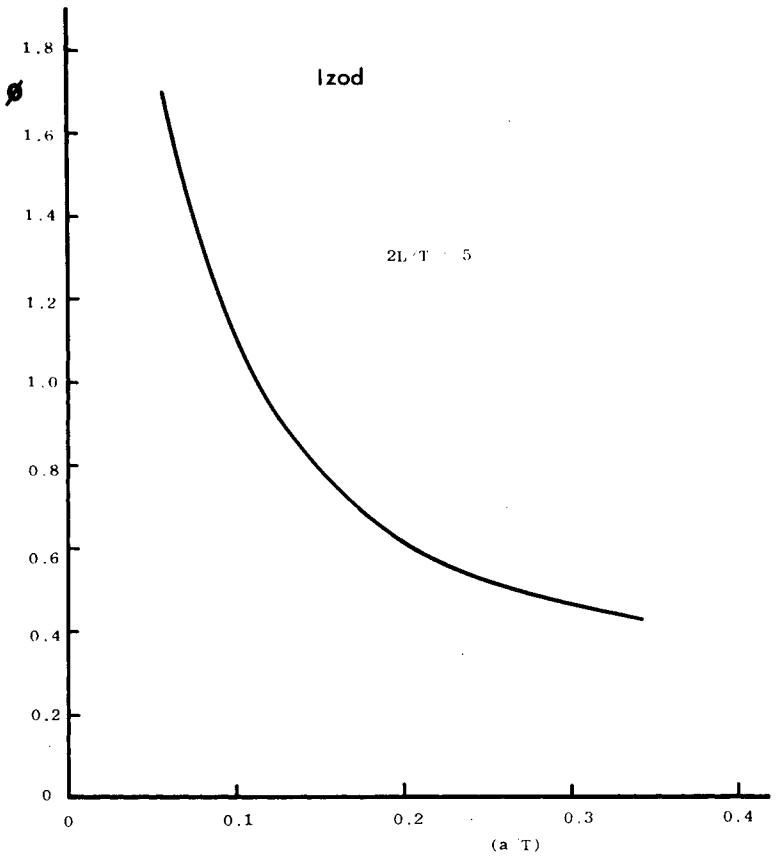


Fig. 3. Graphic expression of $\phi = \phi\ (a/T)$.

shown in Figure 6, in which the time increases from left to right of the horizontal scale. It can be seen that the load-time trace is linear and therefore eqs. (2) and (6) are applicable. The measured impact energy was corrected for kinetic energy effects following the procedure of ref. 7. The data showed that $\epsilon$ indeed followed a linear relationship with $\phi TW$ through the origin as predicted by eq. (6). The slope of a plot of $\epsilon$ vs. $\phi TW$ thus gives the fracture toughness $\mathcal{G}_c$. Figure 7 shows such a plot for one of the four epoxy materials tested.

## RESULTS AND DISCUSSION

The measurements of fracture toughness $\mathcal{G}_{I_c}$ obtained by using both the bulk and the adhesive specimens are shown in Table I. The agreement is quite acceptable, but the adhesive $\mathcal{G}_{I_c}$ seems to be slightly lower than that of the bulk resin. The comparison of these two techniques has been discussed previously.[3] It is possible that the 0.025-cm adhesive bond is not the optimum thickness to give maximum $\mathcal{G}_{I_c}$. Indeed, the fracture behavior of adhesive bonds is known to depend strongly on both the bond thickness and the test temperature.[8] The presence of an adhesive–adherend interface often causes the crack to propagate along that interface which can reduce the measured toughness values since the

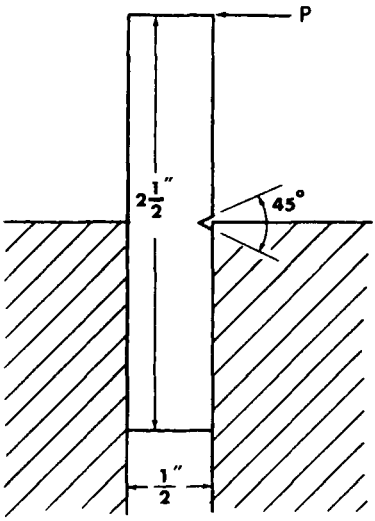


Fig. 4. Izod impact test specimen.

fracture zone is prevented from developing normally. Furthermore, it is not uncommon that volatiles released during the cure of resins are trapped in the confined layer leading to the formation of microvoids within the adhesive bond. This may also contribute to a reduced fracture toughness in the adhesive case.

The CTS test results are also included in Table I. The result from the round

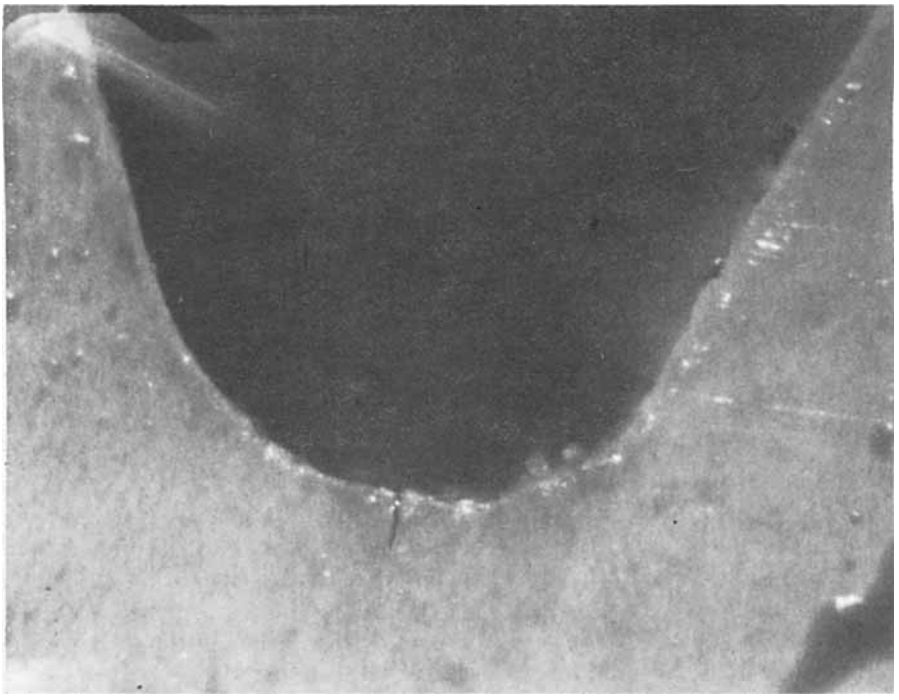

Fig. 5. Precrack at the bottom of the 45° notch of an impact specimen.

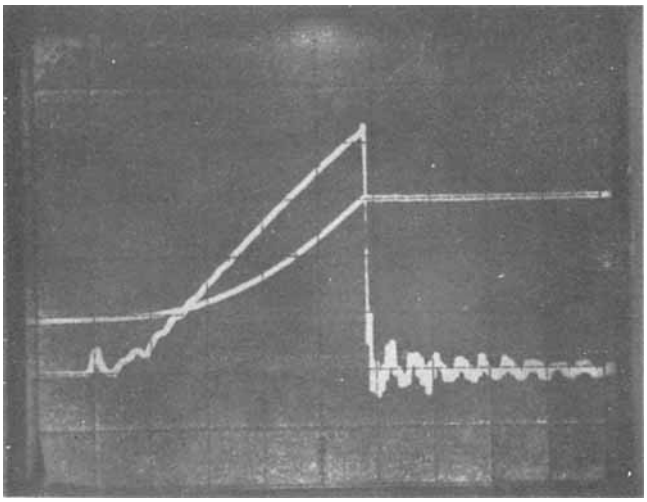


Fig. 6. Impact load and energy recorded as functions of impact time, which increases from left to right (0.2 msec/division).

CTS, although limited, seemed to agree well with those from the rectangular ones. The CTS results, on the other hand, are generally higher than those obtained with the bulk resin double-cantilever beam specimens. The CTS used here

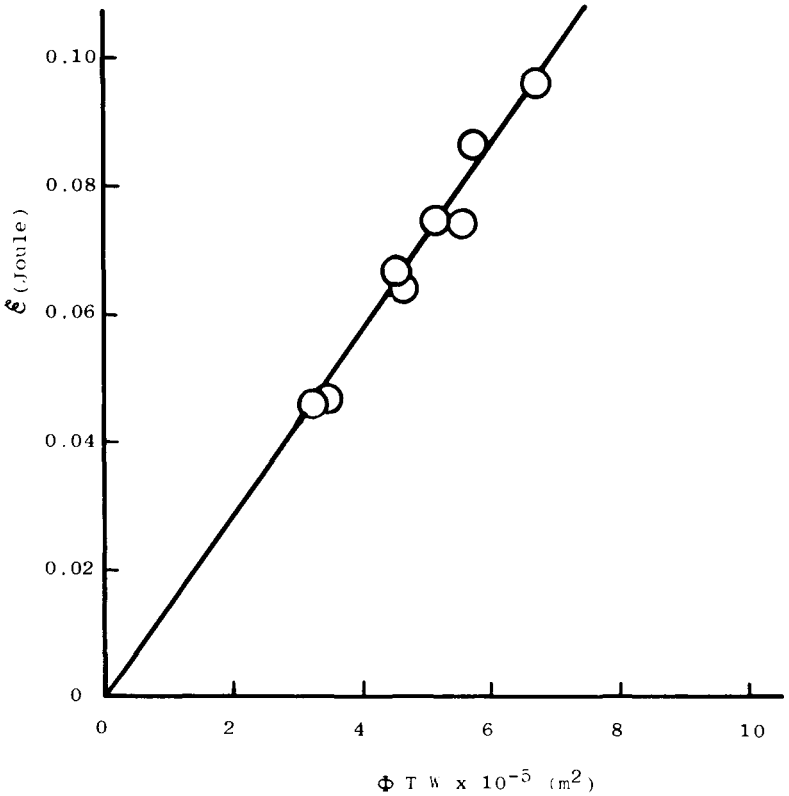


Fig. 7. Linear plot of impact energy vs. specimen cross-sectional area.

TABLE I
$\mathcal{G}_{I_c}$ Values Obtained in Different Tests (kJ/m$^2$)

| Epoxies | Adhesive | Bulk | Rectangular CTS | Round CTS |
|---|---|---|---|---|
| HHPA-cured | 0.116 | 0.136 | 0.181 ± 0.09[a] (7)[b] | — |
| Piperidine cured | 0.121 | 0.154 | 0.187 ± 0.12 (8) | 0.171 |
| 4.5% CTBN | — | 1.60 | 1.35 ± 0.14 (9) | — |
| 4.5% CTBN | 2.07 | 2.14 | 2.20 ± 0.81 (7) | — |
| 10% CTBN | 2.72 | 3.43 | — | — |
| 15% CTBN | 3.52 | 4.12 | 4.78 ± 1.0 (9) | — |

[a] Standard deviation.
[b] Number of specimens.

TABLE II
Comparison of Impact Fracture and CTS Results of $\mathcal{G}_{I_c}$ (kJ/m$^2$)

| Epoxies | Impact | CTS[a] |
|---|---|---|
| base | 0.29 | 0.23 ± 0.04 (6) |
| 8% CTBN | 1.10 | 2.50 ± 0.3 (8) |
| 8% CTBN + 1% solid | 1.38 | 4.10 ± 0.2 (8) |
| 8% CTBN + 5% solid | 2.50 | 5.83 ± 0.9 (8) |

[a] Instron crosshead speed 0.125 cm/min.

might not be thick enough to allow for pure plane strain conditions, although the load-displacement curves were linear.[5] For plane strain conditions to prevail, specimen thickness $b$ should satisfy the condition

$$b \geq 2.5(K_{I_c}/\sigma_y)^2$$

where $K_{I_c}$ is the critical stress intensity and $\sigma_y$ is the yield stress. Based on this criterion, the brittle epoxy specimens tested were sufficiently thick. But the samples toughened with 15% CTBN would require a minimum thickness of 1.68 cm, whereas the test specimens were only 1.0 cm thick. Therefore the plane stress contribution to $\mathcal{G}_{I_c}$ was not negligible in this case, which accounts for the slightly higher values shown in Table I. However, it is felt that this is still a very useful technique for preliminary evaluation of the fracture toughness of new resins, particularly since it requires much less material than, say, a bulk resin double-cantilever beam specimen. In many cases, this also allows one to test more than one compact tension specimen so the results may be more meaningful statistically.

The impact test results are given in Table II, in comparison with those obtained by using the rectangular CTS. It can be seen that the toughness values for the base epoxy were in good agreement. But for the toughened materials the impact test results were significantly lower. This difference is likely due to the difference in loading rate: in impact tests the fracture time was only about 1 msec, whereas the CTS was loaded at a rate of 0.125 cm/min in the Instron. The toughening effect of elastomeric modifiers apparently becomes less pronounced as the loading rate increases. However, it is encouraging to note that both the CTS and the impact test rate the material toughness in the same order. The impact test may be a more useful technique for determining the fracture toughness of glassy polymers, because it more closely simulates the high loading rates which materials encounter in many applications.

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