

Evaluation of Methods of Determining Fracture Parameters for a Glassy Polymer: Poly(methyl Methacrylate)

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Synopsis

Fracture criteria for the brittle fracture of a glassy thermoplastic, poly(methyl methacrylate), have been evaluated using three test piece geometries; the double cantilever beam (DCB), three-point bend (TPB), and compact tension (CT). For the DCB good agreement is obtained with published estimates of the fracture parameters using either a compliance calibration calculation for the critical energy release rate, G_{1c} , or a polynomial function for the critical stress intensity factor, K_{1c} . Anomolously high values of G_{1c} or K_{1c} were obtained using the TPB test piece. These high values of K_{1c} may be partially due to the difficulty of "sharpening" the crack, but there is a test piece size effect which also contributes to the over estimation of K_{1c} . For the CT test piece use of either a new compliance calibration for the determination of G_{1c} or a standard polynomial function for K_{1c} , good agreement was obtained with our own DCB and other published data. The range of applicability of the CT test geometry is discussed critically, and with some reservations it is considered suitable for the evaluation of either G_{1c} or K_{1c} .

INTRODUCTION

The fracture of poly(methyl methacrylate) has been studied extensively using several different types of test specimen design. The results obtained from 20 different determinations have been collated, and apparently discordant results rationalized when the effects of crack speed are considered.¹ One of the favored methods for measuring the energy release rate G is the double cantilever beam (DCB) with side grooves which ensures that the crack path lies approximately in the central plane of the test piece. To calculate the surface energy required to propagate the crack, Berry² proposed an effective method of compliance calibration which will be discussed subsequently. However, both the double cantilever beam (DCB) and its tapered version (TDCB) require large test pieces which makes their fabrication difficult especially when limited quantities of new or modified materials have to be evaluated. Our interest was initiated by the necessity of measuring a fracture parameter for experimental rubber modified dental acrylic resins with only limited amounts of material available. Two types of test piece which require less material are the three-point bend (TPB) and the compact tension (CT) which Ting and Cottingham³ used to measure the fracture energy of rubber modified epoxy resins. Thus, the purpose of this report

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is to assess the suitability of the compact tension and three-point bend test pieces for the measurement of fracture parameters. Standard grade commercial poly(methyl methacrylate) (PMMA—"Perspex") was chosen as the material since there is much data available on its fracture behavior and the TPB and CT test pieces can be compared with the DCB using a compliance calibration.

The fracture parameters which will be used in this evaluation are the energy release rate G_{1c} and the stress intensity factor K_{1c} . G_{1c} is defined by

$$G_{1c} = \frac{1}{b} \cdot \frac{dU}{da} \quad (1)$$

where b is the thickness, the crack length is a , and U is the stored elastic energy. For a linear load extension curve the stored elastic energy U is given by

$$U = \frac{1}{2}P\delta = \frac{1}{2}CP^2 \quad (2)$$

where δ is the displacement for applied load P and C is the compliance, $C = \delta/P$. The change in elastic energy with crack extension at constant load P_c is

$$\frac{dU}{da} = \frac{1}{2} P_c^2 \frac{dC}{da} \quad (3)$$

The famous Irwin-Kies equation is readily obtained by substitution of eq. (3) into eq. (1), thus

$$G_{1c} = \frac{P_c^2}{2b} \cdot \frac{dC}{da} \quad (4)$$

where P_c is the critical load at which crack extension occurs. This relationship is the basis for the compliance calibration methods for determination of G_{1c} .

It is not possible to measure directly the stress at a crack tip, but it can be represented by a stress intensity factor K_1 defined by

$$K_1 = \sigma \sqrt{(\pi \cdot a)} \cdot f(a, w) \quad (5)$$

where σ is the nominal or average applied tensile stress, a is the crack length, and w is the width of the specimen. The fracture toughness of the material, K_{1c} is the critical stress intensity factor at which a crack propagates. For many test piece geometries, numerical computations of the polynomial stress functions $f(a, w)$ are available in standard texts, and will be referred to as appropriate in discussion of the present measurements.

MATERIALS AND METHODS

The material employed was homogeneous poly(methyl methacrylate) (Perspex, ICI Ltd.) and specimens were produced according to the designs and dimensions shown in Figure 1. All specimens were tested using an

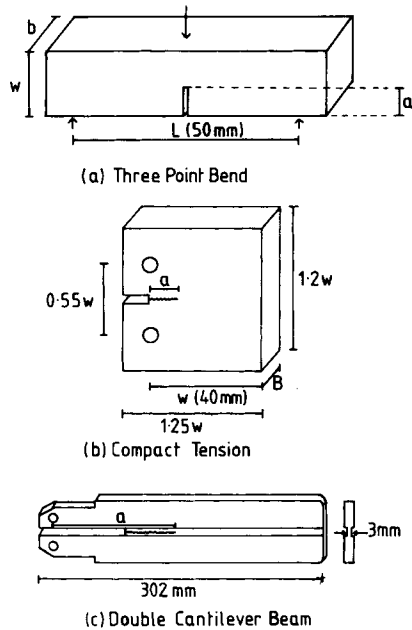


Fig. 1. Specimen geometries for the three-point bend (TPB), compact tension (CT), and double cantilever beam (DCB) test pieces. Dimensions were varied as specified in the text.

Instron 1026 tensile tester at a crosshead speed of 0.5 mm/min. For the DCB specimens a blunt crack was introduced along central guidance grooves, which was then extended by loading the specimen until a crack just started to grow. The load was then removed, leaving a sharp natural crack, which was used as the initial crack length. Because of the size of the specimen, it was possible to obtain a number of measurements of the load required to cause the crack to extend for a range of crack lengths. The guidance grooves prevented gross deviation of the crack from planarity, although at low magnification the fracture surface had a ribbed appearance which we have reported recently.⁴ Much the same test procedure was used for the CT specimens, and with great care it was possible to initiate a crack and make repeated measurements for different crack lengths on the same specimen. The crack travelled in essentially a flat plane across the test piece and guidance grooves were unnecessary. TPB specimens with a variety of dimensions and crack lengths were produced. The initial blunt crack was sharpened with a razor blade and specimens were tested in three-point bending. In every case crack growth and subsequent failure was uncontrollable for the TPB test pieces, and only one result per specimen could be obtained.

RESULTS AND DISCUSSION

The Double Cantilever Beam (DCB)

This type of test piece [Fig. 1(c)] was used many years ago by Berry,² who showed that a compliance calibration allowed determination of fracture surface energies. He represented the stiffness S of the double cantilever beam by the power law

$$S = A_1 a^{-n} \quad (6)$$

where a is the crack length and A_1 and n are constants to be determined by experiment. The values for A_1 and n can be determined from eq. (6) by plotting $\log S$ vs. $\log a$ as shown in Figure 2, and the good straight line fit conforms to eq. (6).

To determine the critical energy release rate, the Irwin-Kies equation (4) is used, which requires that the force P_c required for crack growth and dC/da are measured. The compliance C is the inverse of the stiffness $C = 1/S$, such that

$$\frac{dC}{da} = -\frac{1}{S^2} \frac{dS}{da} \quad (7)$$

Differentiation of eq. (6) gives

$$\frac{dS}{da} = -nA_1 a^{-(n+1)} \quad (8)$$

Substitution of equations (6)–(8) into the Irwin-Kies equation (4) yields the relationship

$$G_{1c} = \frac{P_c^2}{2bA_1} \frac{n}{a^{1-n}} \quad (9)$$

from which the critical energy release rate may be calculated. The average value obtained for G_{1c} is 304 J m^{-2} , which agrees well with Berry's² result of $G_{1c} = 280 \text{ J m}^{-2}$.

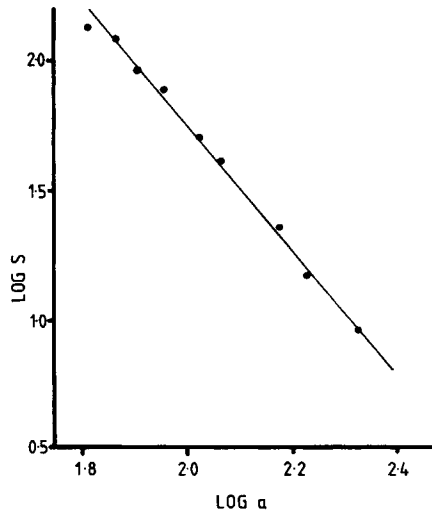


Fig. 2. Linear plot of $\log S$ vs. $\log a$ for the double cantilever beam, where S is the stiffness and a is the crack length.

The double cantilever beam configuration for the determination of fracture parameters has received considerable attention and an alternative and interesting approach is that of Wiederhorn and co-workers,⁵ who show that the stress intensity factor, K_{Ic} , can be computed from

$$K_{Ic} = \frac{P_c a}{b w^{3/2}} \left(3.467 + 2.315 \frac{w}{a} \right) \quad (10)$$

This relationship was also obtained by Gross and Srawley⁶ and by Gillis and Gilman,⁷ who employed different methods of analysis. Equation (10) applies to a double cantilever beam without guidance grooves which restrict the crack path to the center of the test piece. When guidance grooves are present, eq. (10) requires correction. Heady⁸ has found that, for steel, eq. (10) should be replaced by

$$K_{Ic} = \frac{P_c a}{b w^{3/2}} \left(3.467 + 2.315 \frac{w}{a} \right) \left(\frac{b}{b_n} \right)^n \quad (11)$$

where b and b_n are the overall thickness and thickness between the guide grooves, respectively. Using a value of $n = 0.58$ determined by Heady,⁸ the applicability of this equation can be tested by plotting P_c vs. $(3.467a + 2.315w)^{-1}$, which is shown in Figure 3. From the slope of the straight line

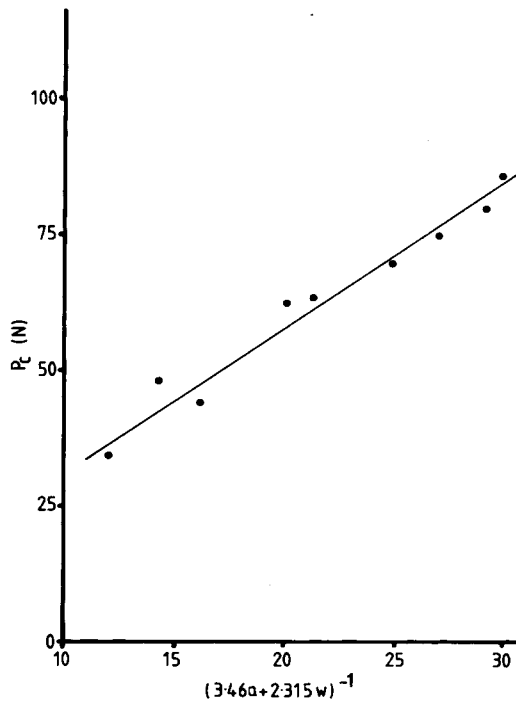


Fig. 3. Plot of P_c vs. $f(a,w)$ showing a linear relationship as predicted by eq. (11).

an estimate of the fracture toughness was obtained, and a K_{1c} of 0.909 MN $m^{-3/2}$ was calculated. This may be compared with the estimate of the critical energy release rate, obtained from the compliance calibration by using the relationship

$$K_{1c} = \left[\frac{G_{1c} \cdot E}{(1 - \nu)^2} \right]^{1/2} \quad (12)$$

where E is Young's modulus and ν is Poisson's ratio. There are difficulties in deciding on appropriate values of these parameters to use with eq. (12), as mentioned by Marshall and Williams.¹ However, the result will not be very sensitive to the choice of value for Poisson's ratio. Using $E = 3 \times 10^9$ N m^{-2} and $\nu = 0.35$ which are reasonable values and substitution of G_{1c} obtained from the compliance calibration into eq. (12) yields $K_{1c} = 1.019$ MN $m^{-3/2}$, which compares well with the value of 0.909 MN $m^{-3/2}$ obtained using the polynomial function above [Eq. (11)]. Although agreement between the two approaches is reasonable, this could be improved by choice of alternative values for the exponent n and elastic modulus E in eq. (11). A detailed photoelastic analysis⁹ of the stress distribution shows that the side grooves in a double cantilever beam raise the stress by about 22%. However, further experimental work would be required to have more confidence in an alternative value for n .

The Three-Point Bend Test

This test is attractive in that sample fabrication is facile, and relatively small amounts of material are required. The fracture toughness¹⁰ for this test geometry may be obtained using

$$K_{1c} = \frac{P_c L}{bw^{3/2}} \cdot f(a, w) \quad (13a)$$

where

$$f(a, w) = 2.9(a/w)^{1/2} - 4.6(a/w)^{3/2} + 21.8(a/w)^{5/2} - 37.6(a/w)^{7/2} + 38.7(a/w)^{9/2} \quad (13b)$$

and b , L , w , and a are defined in Figure 1.

To determine the fracture toughness, the breaking load P_c has to be measured for a specific crack length a . The applicability of eq. (13) is discussed by Rooke and Cartwright,¹⁰ who give graphs for span length to test piece breadth ratios (L/w) equal to 2 and 4 in the range $0 \leq a/w \leq 0.6$. In this work L/w varied from 2.5 to 8 for a/w in the range $0.2 \leq a/w \leq 0.66$. There was no systematic variation of K_{1c} with the ratio of a/w , which agrees with Davidge and Tappin¹¹ since their surface energy γ_G is also independent of a/w for $a/w < 0.6$. But we found that with increasing L/w the fracture toughness decreased from 1.95 to 1.62 MN $m^{-3/2}$ as shown in Figure 4. The decrease of K_{1c} with L/w does not appear to have been

reported previously and will obviously cause problems with specification of test piece dimensions for a three-point bend test piece. Also, the numerical value of these K_{1c} determinations is higher than those obtained using other geometries as may be seen by inspection of the tabulation compiled by Marshall and Williams¹ and our own results for the double cantilever beam.

When a thinner test piece is used ($b = 3.15$ mm), the calculated fracture toughness is even higher with an average K_{1c} of $2.01 \text{ MN m}^{-3/2}$. To some extent, this thickness effect was unexpected since Parvin and Williams¹² state that there is an absence of any thickness dependence. This is a direct consequence of the limited plastic deformation since for most practical thickness a state of plane strain exists at the crack tip. Williams¹³ discusses the criterion to be used to assess the minimum thickness for valid fracture toughness tests and suggests that the following relationship should be used:

$$b_{\min} = 2.9(K'_{1c} / \sigma_y) \quad (14)$$

where K'_{1c} is an initial estimate of the fracture toughness and σ_y is the yield stress. The factor 2.9 is more conservative than the value of 2.5 given by Knott.¹⁴ The yield stress of poly(methyl methacrylate) is difficult to determine. However, using the value $\sigma_y = 3.7 \times 10^7 \text{ N m}^{-2}$ given by Kitagawa¹⁵ at a temperature of about 25°C the minimum thickness should be 2.1 mm when $K_{1c} = 1 \text{ MN m}^{-3/2}$, a reasonable "average" value from the compilation of Marshall and Williams.¹ Thus for the specimens used there should not have been a thickness effect if this criterion is correct. Also there was no evidence of "shear lips" on the fracture surface which would have occurred if the condition of plane strain is not satisfied across the major portion of the crack front. A thickness effect was also observed with the compact tension test pieces, as will be discussed subsequently.

Davidge and Tappin¹¹ estimated the work to fracture from the area under the load deflection curve, which in the case of PMMA is only applicable when the crack length, i.e., $a/w > 0.5$. While most of our test pieces had $a/w < 0.5$ so as to comply with the requirements of Brown and Strawley's computations,^{16,10} in a few cases specimens with $a/w > 0.5$ were used. How-

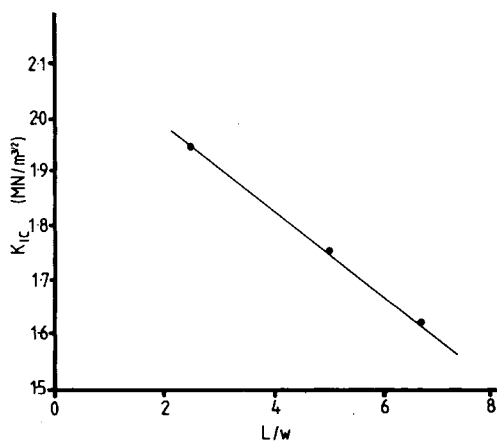


Fig. 4. Fracture toughness K_{1c} as a function of L/w for the TPB test pieces.

ever, even in these cases the crack propagated rapidly across the test piece, and the "tail" region in the load deflection curve was not observed. Unless such a tail is present, it is not possible to use the area under the load deflection curve to measure work of fracture. It should be noted that for the crack to propagate across the test piece, it must move into a region with a very complex stress pattern, due to the application of the central load in line with the crack. Often the crack deviates from its original direction when it enters the region influenced by the applied central load.

The high value of K_{Ic} determined from three-point bend test specimens compared to the DCB test piece is probably related to the difficulty of "sharpening" the crack. The usual practice, which we also followed, involves use of a razor blade but then the crack will not have the same form as those produced by previous fractures such as a double cantilever beam specimen in which the crack is made to grow successively from crack tips produced by terminated growth of the crack.

Compact Tension

This test piece [Fig. 1(b)] is attractive since it is readily fabricated and is conservative with respect to material relative to the double cantilever beam. Also, we have found that with Perspex compact tension test pieces, it is possible to propagate the crack in short increments provided the crosshead of the tensile tester is stopped as soon as any crack growth occurs. This has the advantage that crack growth is from a sharp crack front overcoming a problem associated with the TPB specimens. Several experimental measurements of the force P_c and corresponding crack length a can be obtained from one test piece.

We have developed a simple compliance calibration method for this geometry in which the stiffness S is represented by a power law in terms of the ligament length ($w - a$). Thus

$$S = A_2(w - a)^m \quad (15)$$

and in fact plots of $\log S$ vs. $\log(w - a)$ are linear, as shown in Figure 5, from which A_2 and m can be determined. Differentiating eq. (15) with respect to a gives

$$\frac{dS}{da} = -mA_2(w - a)^{m-1} \quad (16)$$

which with eq. (7) gives

$$\frac{dC}{da} = \frac{m}{A_2}(w - a)^{-(m+1)} \quad (17)$$

Equation (17) can be substituted in the Irwin-Kies equation (4) to obtain

$$G_{Ic} = \frac{P_c^2 \cdot m}{2bA_2(w - a)^{(m+1)}} \quad (18)$$

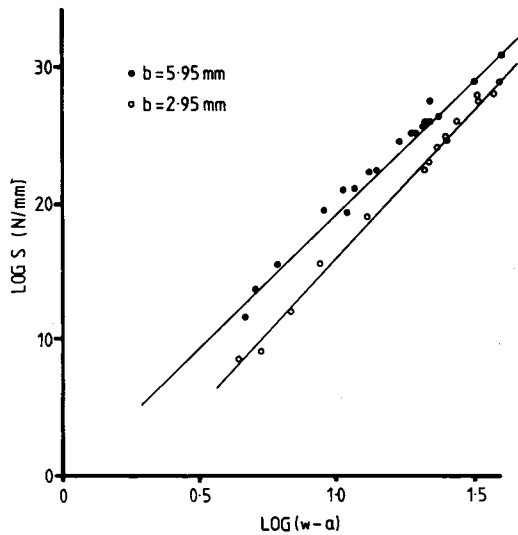


Fig. 5. Linear plot of $\log S$ vs. $\log(w/a)$ for compact tension test pieces of different thicknesses.

The British Standard (BS 5447) specifies that only values of G_{1c} within the crack length range $0.45 < a/w < 0.55$ are acceptable. Ting and Cottingham³ proposed that values for G_{1c} would be acceptable if $0.3 < a/w < 0.7$ is satisfied. The results for our experiments are shown in Figure 6 where the calculated G_{1c} is plotted against a/w for the 6 mm thick specimens. This shows that for the particular specimen dimensions used the extended range of crack lengths as proposed by Ting and Cottingham³ is acceptable. Within this range of crack lengths, the average value for G_{1c} was $308 \pm 14 \text{ J m}^{-2}$ for the 6 mm thick specimens and $456 \pm 154 \text{ J m}^{-2}$ for the 3 mm thick specimens. The latter is considerably higher, showing that there is a thickness effect with this test geometry. As with the three-point bend test piece discussed previously, there appears to be a minimum thickness requirement for the compact tension test pieces.*

The fracture surfaces did not have any evidence of shear lips which often occurs when there is an extensive region of plane stress near the edges of a crack. The fracture surfaces appeared at low magnification to consist of ribs which run in the direction of the crack growth and have been described in detail elsewhere.⁴ Thus this thickness effect warrants further study especially since Ting and Cottingham³ also found a similar effect with compact tension test pieces.

A polynomial stress function has been computed for the compact tension test piece¹⁷ and the fracture toughness may be computed from

$$K_{1c} = P/bw^{3/2} [29.6(a/w)^{1/2} - 185.5(a/w)^{3/2} + 655.7(a/w)^{5/2} - 1077(a/w)^{7/2} + 639(a/w)^{9/2}] \quad (19)$$

*The referee states that from his experience on 1-in. CT specimens the minimum thickness needs to be 3/16-in. (4.76 mm) for plastics. From this it can be concluded that our measurements with 6 mm thick CT test pieces yield correct determinations of K_{1c} .

From the plot of K_{Ic} vs. a/w , also shown in Figure 6, it can be seen that eq. (19) yields a constant value for K_{Ic} for crack lengths within the range $0.3 \leq a/w \leq 0.7$ for compact tension test pieces with the dimensions specified in Figure 1. The value for K_{Ic} was found to be $0.95 \pm 0.04 \text{ MN m}^{-3/2}$, which compares well with the values obtained using the double cantilever beam and the average value for K_{Ic} of Perspex from the data tabulated by Marshall and Williams.¹

Concluding Remarks

From the results presented it is clear that the TPB specimen design for measurement of fracture toughness suffers some serious shortcomings. Despite its attraction as an easily fabricated test piece, which requires little material, it cannot at present be used to obtain reliable estimates of the fracture toughness of poly(methyl methacrylate) and by implication related polymers. It has been shown that the results are highly dependent on the specimen geometry chosen. The inability to produce a natural crack is a serious disadvantage and can result in an overestimate of the fracture toughness by as much as 100% and also produces a wide scatter of measurements. With strict specifications of specimen dimensions which are kept constant it may be useful for comparative purposes provided its limitations are fully appreciated.

It must be accepted that whatever approach is used for the measurement of fracture toughness of materials some scatter is inevitable. However, both the DCB and CT test specimens produced results with considerably less scatter than the TPB specimens. More importantly perhaps is that the TPB specimen design results in an overestimate of the fracture toughness, and

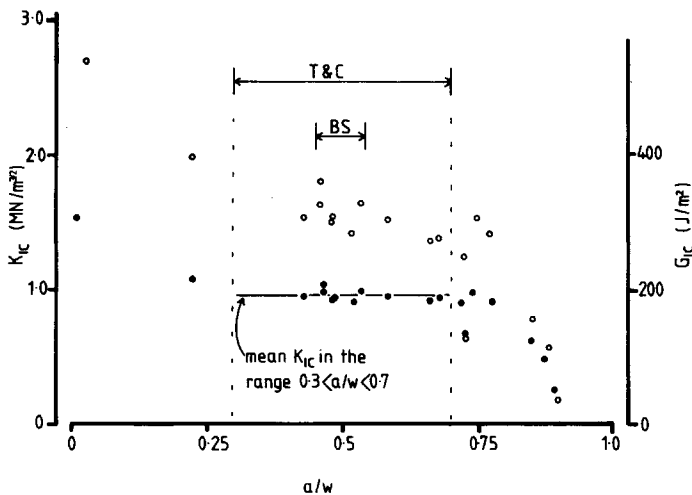


Fig. 6. Variation of K_{Ic} (●) and G_{Ic} (○) with a/w . Indicated also are the limits for a/w proposed by Ting and Cottingham³ and the British Standard recommendation for compact tension test pieces.

in this respect the CT specimen is superior with good agreement for G_{1c} using the compliance calibration method between the CT specimen and the DCB specimen design. Similarly the use of the appropriate polynomial functions for the measurement of K_{1c} produced concordant results between the CT and DCB specimen designs.

Using the relationship between G_{1c} and K_{1c} given in eq. (12), it is possible to convert the values for G_{1c} from the compliance calibration methods to K_{1c} , which allows comparison of the two methods of analysis. As can be seen from the data presented in Table I, the good agreement between the measurement of K_{1c} using the double cantilever beam and the compact tension test piece validates the use of the latter for the measurement of fracture toughness of polymers such as Perspex. Either of the two methods of analysis detailed above may be used to determine the stress intensity factor or, if preferred, the critical strain energy release rate directly. Yet it must be appreciated that on using the compact tension test piece there are limitations on the useful crack length range which can be employed and the thickness effect also needs to be considered.

An additional problem not yet mentioned is the difficulty in measurement of the critical load at which crack growth occurs. In our experiments with Perspex it was possible to differentiate between the load at which nonlinear behavior started to occur and the load at which the crack would start to grow rapidly representing a situation of instability. Stafford et al.¹⁸ proposed that K_{1c} at instability may be the best parameter to use since they claim it is "independent of strain rate." They were able to distinguish between K_{1c} at nonlinearity and K_{1c} at instability using three-point bend test pieces. In our experiments these two parameters were clearly visible for the compact tension test pieces, but it was not possible to obtain separate estimates of K_{1c} for the TPB test. In contrast, Hill et al.¹⁹ observed no detectable deviation from linearity using the CT specimens. It is not clear what gives rise to this deviation from linearity prior to rapid growth, although it has been suggested that this represents the onset of slow crack growth which is difficult to detect. The values given in Table I for the CT test piece used the load at onset of rapid crack growth, i.e., K_{1c} instability, for which good

TABLE I
Comparison of K_{1c} for Perspex

Specimen design	Nominal thickness (mm)	Method of analysis	K_{1c} (MN m ^{-3/2})
DCB	3.00	Polynomial function	0.91
DCB	3.00	Compliance calibration	1.02
CT	3.00	Polynomial function	1.12
CT	3.00	Compliance calibration	1.24
CT	6.00	Polynomial function	0.95
CT	6.00	Compliance calibration	1.03
CT ^a	3.00	Polynomial function	1.12
CT ^a	6.00	Polynomial function	0.99

^aHill et al.¹⁹

agreement was obtained with the results reported by Hill et al.¹⁹ who used specimens of nominally equivalent thickness.

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