Association Connecting Electronics Industries

3000 Lakeside Drive, Suite 309S
Bannockburn, IL 60015-1249

Number	
2.4.52	
Subject **Fracture Toughness of Resin Systems for Base Materials**	
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Originating Task Group **IPC 3-11**	

IPC-TM-650
TEST METHODS MANUAL

1 Scope This test method establishes a procedure for characterizing the toughness of the resin system materials used in making laminates for the fabrication of printed wiring boards. The single-edge-notch bending (SENB) geometry is used to determine the critical-stress-intensity factor, K_{1c}, and the energy per unit area of crack surface or critical strain energy release rate, G_{1c}, at fracture initiation. This method assumes linear elastic behavior of the cracked specimen, so there are corresponding restrictions on the linearity of the load-displacement diagram. Use of this test method for printed wiring board laminate materials or other composites may not yield comparative results.

2 Applicable Documents

2.1 ASTM Standards

D638 Test Method for Tensile Properties of Plastics

D4000 Classification Systems for Specifying Plastic Materials

D5045 Standard Test Methods for Plane-Strain Fracture Toughness and Strain Energy Release Rate of Plastic Materials

E399 Test Method for Linear-Elastic Plane-Strain Fracture Toughness K_{1c} of Metallic Materials

E691 Practice for Conducting an Inter-Laboratory Study to Determine the Precision of a Test Method

3 Terminology

3.1 Terms and Definitions (reference ASTM E399)

3.1.1 Compact Tension Specimen geometry consisting of single-edge notched plate loaded in tension.

3.1.2 Critical Strain Energy Release Rate (G_{1c}) Toughness parameter based on energy required to fracture.

3.1.3 Plane-Strain Fracture Toughness (K_{1c}) Toughness parameter indicative of material fracture resistance.

3.1.4 Single-Edge Notched Bend Specimen geometry consisting of center-notched beam.

3.1.5 Yield Stress The stress at fracture (slope of stress-strain curve is not required to be zero).

4 Test Samples

4.1 Sample Construction The preferred finished sample is a block of pure resin, free of contaminants and fully cured (not partially cured, not over-cured). Note: DSC may be used to evaluate a received sample's degree of cure. TGA may be used to check for the presence of residual solvents or other contaminants.

A heated hydraulic press may be required to prepare the sample. Attachment A is a method for making compression molded thermoset neat resin castings. Size and occurrence of voids within the sample should be kept to an absolute minimum (maximum void dimension 25 µm [0.001 in]; maximum 5 voids/cc). Specimen block may be ground down to the desired dimensions, and a mold **shall not** be used. Default specimen dimensions should be 3.50 mm ± 0.05 mm thick, 12.7 mm wide (in general, the nominal width can be between 2X to 4X the thickness, but should be consistent) and 55.88 mm long (length should be 4.4 times the width). However the absolute minimum thickness is 2.5 times the square of the conditional or trial K_{1c} (K_Q) divided by the yield stress (σ_y) of the material for the temperature and loading rate of the test.

The above should ensure that the sample is wide enough to ensure plane strain and sufficiently thick to avoid excessive plasticity in the ligament. If non-linearity in loading still occurs, the width can be increased up to 4 times the thickness of the specimen. Polishing the sample (minimum 600 grit) is recommended to promote yielding in the tensile test, rather than brittle fracture. Each of the thickness and width dimensions of the specimen should be measured in at least 3 locations to an accuracy of 0.1% and both dimensions **shall** be accurate to within 1% of nominal. The average of these measurements will be used in the calculations. At least 10 samples of each material are recommended for testing, allowing up to 5 samples for developing sufficient skill in initiating consistent cracks and subsequently at least 5 samples meeting Section 4.2 criteria for acceptable fracture toughness measurements.

4.2 Sample Preparation The required square notch is cut in the center of the sample width, within ± 0.25 mm, using a diamond saw or similar stress-free method to form a smooth bottom of the notch. The slot width should be 0.25 mm [0.00984 in.].

The crack itself is initiated on each sample with a new 0.23 mm [0.009 in] thick ultra-sharp carbon steel razor blade (example: **http://www.mcmaster.com/#3962a4/=3qpeql**). It is recommended that the blade be refrigerated or cooled in liquid nitrogen or in dry ice shortly before use. The razor blade is then carefully tapped using a small weighted hammer with sufficient force and control for the crack to initiate on the first or second try. A new, cool or cold razor blade is recommended for reducing the force needed for crack initiation. A few specimens in every test lot should first be sacrificed for operator practice at crack initiation, precisely determining the hammer force needed for that sample lot to avoid only making indentations. The depth of the natural crack generated by tapping **shall** be a least twice the width of the machined notch (3X the width of the notch is ideal).

The total depth of the notch plus the depth of the crack **shall** be half the thickness of the sample, within ± 5 percent. Therefore the depth of the square notch should be 45 percent of the sample width minus 0.75 mm, ± 0.13 mm. The crack **shall** be sufficiently sharp to ensure that a minimum value of toughness is obtained during the subsequent 3-point bending. The actual depths are measured after fracture within 0.5 % accuracy at three locations; at the center of the crack front, and at the end of the crack front on each surface of the specimen. The average of these three measurements, which should be fairly uniform, **shall** be used in the calculations. Cracks or breaks should be resin-resin, not between resin and filler.

5 Equipment/Apparatus or Material

5.1 Test Machine

5.1.1 The testing machine used **shall** be a constant displacement rate device; an electromechanical screw-driven machine, or a closed loop feedback-controlled servo-hydraulic load frame. The stationary and moving rollers used for the 3-point loading (typically two under each end, and one on top in the middle of the specimen block opposite the crack) **shall** each be large enough to avoid excessive indentation of the plastic, however the roller diameter should not exceed the overall thickness of the specimen.

5.2 Displacement Measurement

5.2.1 The displacement measurement using an internal displacement transducer having sufficient precision **shall** be performed using the machines stroke or position transducer. The fracture test displacement data **shall** be corrected for system compliance, loading pin penetration and specimen compression by performing a calibration of the testing systems as described in ASTM D-5045.

5.2.2 The displacement measurement using an external displacement transducer having sufficient precision **shall** be performed with the transducer located between the top and bottom plates, and as close as possible to the load point on the specimen to ensure displacement accuracy.

5.3 Yield Stress

5.3.1 The yield stress, σ_y, is determined by the material's maximum load in an uniaxial tensile test. Using a constant stroke rate uniaxial tensile test, the loading time to yield **shall** be within ± 20 percent of the actual loading time observed in the fracture test. A zero slope to the stress-strain curve is not required. If a tensile test cannot be performed, then use 0.7 times the compressive yield stress as an approximation.

6 Procedure

6.1 Test Preparation The specimens and all testing **shall** be performed at 23 °C ± 3 °C. The actual temperature of the specimen **shall** be recorded. The relative humidity should be between 30 % and 60 % RH, and **shall** be recorded.

6.2 Displacement Correction Specimen **shall** be identical to the specimen prepared for fracture testing, except without the notch or crack in the middle. This specimen **shall** be used for single notch bend testing (reference ASTM D5045).

6.3 Testing

6.3.1 The notched specimen that has been pre-cracked is subjected to loading at a loading rate of 5.0 mm per minute.

6.3.2 The test is performed and the load versus loading point displacement curve is obtained. In the ideal case, there is an abrupt drop of load to zero at the instant of crack growth initiation. If this occurs, then determine the trial K_{1c} or K_Q from the maximum load. Typically there will be a noticeable deviation from linearity prior to fracture.

6.3.3 The load corresponding to a 2.5 % apparent increment of crack extension is established by a specified deviation from the linear portion of the record. The K_{1c} value is calculated from this load by equations that have been established on the basis of elastic stress analysis on other specimens sufficiently large to show linear elastic behavior.

6.3.4 After breaking, the interface should appear smooth and glossy. A layered or hazy surface indicates deformation as a failure mechanism rather than cracking, and invalidates the test results. After testing, measure the depth of the crack from the notch depth at three locations along the width of the sample. Also measure the depth of the crack from the top of the sample (at the same three locations).

6.3.5 Determining the load displacement area or G_{1c} requires an accurate integration of the load versus loading point displacement curve, including an accurate displacement determination using the displacement transducer.

7 Analysis Fracture toughness testing is recommended to be performed at least twice per year.

7.1 Calculation and Interpretation of Results (reference ASTM D5045, Section 9).

7.1.1 In order to establish that a valid K_{1C} has been determined, it is first necessary to calculate a conditional result, K_Q, which involves a construction on the test record, and to then determine whether this result is consistent with the size of the specimen in accordance with 7.1.6. The procedure is given in 7.1.2 through 7.1.8.

7.1.2 Load the specimen and obtain a P (load) versus u (displacement) plot (see Figure 7-1).

Draw a best straight line (AB) to determine the initial compliance, C. C is given by the reciprocal of the slope of line (AB). Draw a second line (AB') with a compliance 5 % greater than that of line (AB). If the maximum load that the specimen was able to sustain, Pmax, falls within lines (AB) and (AB'), use Pmax to calculate K_Q. If Pmax falls outside line (AB) and line (AB'), then use the intersection of line (AB') and the load curve as P_Q. Furthermore, if $Pmax/P_Q$ <1.1, use P_Q in the calculation of K_Q. However, if $Pmax/P_Q$ >1.1, the test is invalid.

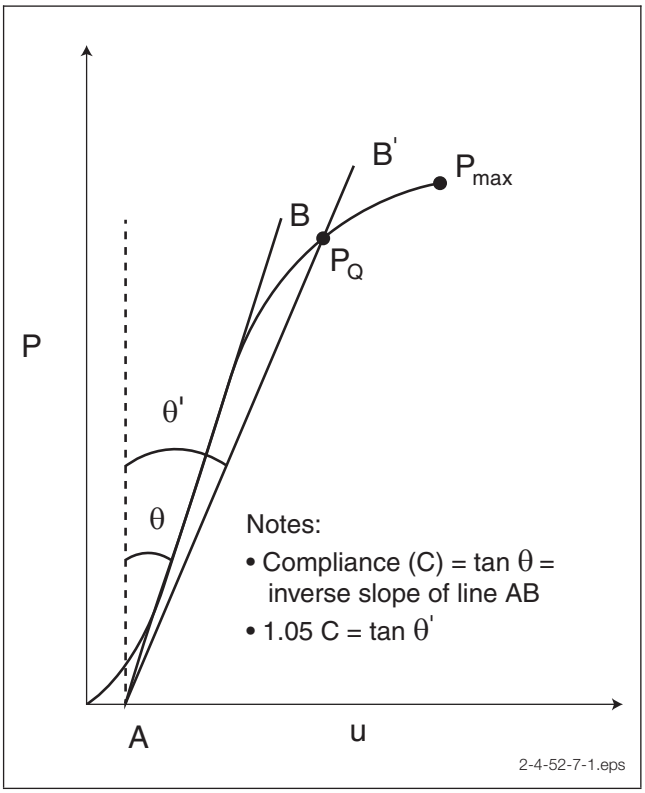

Figure 7-1 Determination of C and P_Q

7.1.3 Calculate K_Q in accordance with the procedure for single edge notch bending in 7.1.4. For this calculation, a value of a, which is the total crack length after both notching and pre-cracking, but before fracture, is best determined from the fracture surface after testing. An average value is used, but the difference between the shortest and longest length should not exceed 10 %. Take care that it is the original crack which is being observed, since slow growth can occur prior to catastrophic fast fracture.

7.1.4 (Reference ASTM D5045, Section A1.4). The general formula for K_Q calculation of bend specimens is given in [Ref. 3]. The general principles of the bend-test fixture are illustrated in Figure 7.2.

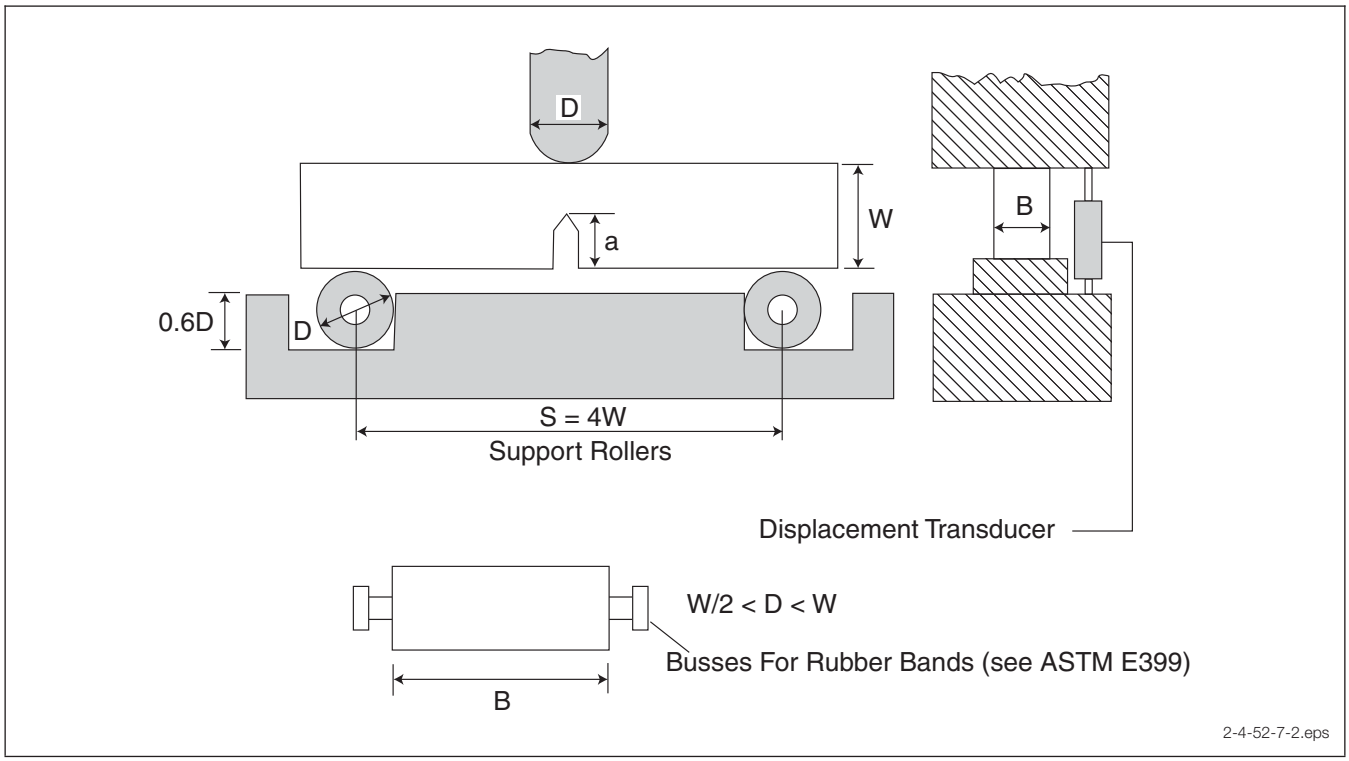

Figure 7-2 Bending Rig with Transducer for Single-Edge-Notch Bending (SENB)

This fixture is designed to minimize frictional effects by allowing the support rollers to rotate and move apart slightly as the specimen is loaded, thus permitting rolling contact. Thus, the support rollers are allowed limited motion along the plane surfaces parallel to the notched side of the specimen, but are initially positively positioned against stops that set the span length at 50 mm, and are held in place by low-tension springs (such as rubber bands). For the bend specimen, the displacements will be essentially independent of the gauge length up to a gauge length of W/2. For bend specimens with S/W = 4, K_Q in units of MPa • $m^{1/2}$ is as follows:

$$K_Q = (P_Q/BW^{1/2})\, f(x)$$

where $(0 < x < 1)$:

$$f(x) = 6x^{1/2}\,(1.99 - x(1-x)(2.15 - 3.93x + 2.7x^2))\,/\,(1 + 2x)\,(1 - x)^{3/2}$$

and:

P_Q = load as determined in 7.1.2, kN,

B = specimen thickness, cm,

W = specimen depth (width), cm,

a = crack length, cm

and

x = a/W.

Tabulated values of $f(x)$ are given in Table 7-1.

Table 7-1 Calibration Factors SENB[A] *S/W* = 4

a/W	*f (x)*	Φ	ψ	η_e
0.450	9.14	0.274	45.8	2.00
0.455	9.27	0.272	46.7	2.00
0.460	9.41	0.269	47.6	2.01
0.465	9.55	0.266	48.5	2.01
0.470	9.70	0.263	49.5	2.02
0.475	9.85	0.260	50.4	2.02
0.480	10.00	0.257	51.4	2.03
0.485	10.16	0.254	52.5	2.03
0.490	10.32	0.252	53.5	2.03
0.495	10.48	0.249	54.7	2.03
0.500	10.65	0.246	55.8	2.03
0.505	10.82	0.243	57.0	2.03
0.510	10.99	0.241	58.2	2.04
0.515	11.17	0.238	59.4	2.04
0.520	11.36	0.236	60.7	2.04
0.525	11.54	0.233	62.1	2.04
0.530	11.74	0.230	63.5	2.04
0.535	11.94	0.228	64.9	2.04
0.540	12.14	0.225	66.4	2.04
0.545	12.35	0.223	67.9	2.04
0.550	12.56	0.220	69.5	2.05

[A]Values calculated using A. Bakker, Compatibility Compliance and Stress Intensity Expressions for the Standard Three-Point Bend Specimens. Paper submitted for publication in International Journal of Fatigue and Fracture of Engineering Materials and Structures (March 1989).

7.1.5 For the bend specimens calculate G_Q [=] kJ/m2 from the corrected energy, U, as follows:

$G_Q = U/(BW\Phi)$ or $G_Q = \eta_e U/(B(W - a))$

Values of η_e are given in Table 7-1. The energy calibration factor, Φ, is defined as:

$\Phi = C/(dC/d(A/W))$

and **shall** be computed from the following:

$\Phi = (A + 18.64)/(dA/dx)$

where:

$A = [16x^2/(1 - x)^2][8.9 - 33.717x + 79.616x^2 - 112.952x^3 + 84.815x^4 - 25.672x^5]$,

and:

$dA/dx = [16x^2/(1 - x)^2][-33.717 + 159.232x - 338.856x^2 + 339.26x^3 - 128.36x^4]$

$+ 16[8.9 - 33.717x + 79.616x^2 - 112.952x^3 + 84.815x^4 - 25.672x^5]\{[2x(1 - x) + 2x^2]/(1 - x)^3\}$

Values of Φ are given in Table 7-1.

7.1.6 (Reference ASTM D5045, Section 9.1.3) Check the validity of K_Q via the size criteria. Calculate 2.5 $(K_Q/\sigma_y)^2$ where σ_y is the yield stress. If this quantity is less than the specimen thickness, B, the crack length, a, and the ligament (W - a), then K_Q is equal to K_{1c}. Otherwise the test is not a valid K_{1c} test.

NOTE: Use of a specimen with too small a thickness, B, will result in K_Q being higher than the true K_{1c} value while a small (W - a) will result in a K_Q value that is lower than the true K_{1c} value. The net effect may be close to the correct K_{1c} but unfortunately in an unpredictable way, since the dependence on B cannot be quantified.

7.1.7 For the recommended specimen dimensions of W = 2B and a/W = 0.5, all the relationships of 7.1.6 are satisfied simultaneously. In fact, the criterion covers two limitations in that B must be sufficient to ensure plane strain, but (W - a) has to be sufficient to avoid excessive plasticity in the ligament. If (W - a) is too small the test will often violate the linearity criteria. If the linearity criterion is violated, a possible option is to increase W for the same a/W and S/W ratios. Values of W/B of up to 4 are permitted.

7.1.8 If the test result fails to meet the requirements in either 7.1.2 or 7.1.6, or both, it will be necessary to use a larger specimen to determine K_Q. The dimensions of the larger specimen can be estimated on the basis of K_Q, but generally must be increased to 1.5 times those of the specimen that failed to produce a valid K_{1c} value.

7.2 Displacement Correction for Calculation of G_Q (Reference ASTM D5045, Section 9.2)

Make a displacement correction for system compliance, loading-pin penetration, and specimen compression, then calculate G_{1C} from the energy derived from integration of the load versus load-point displacement curve.

7.2.1 The procedure for obtaining the corrected displacement, u_c (P), at load P from the measured displacement, u_Q (P), is as follows: Use an un-cracked displacement correction specimen prepared from the same material as that being tested. Using the same testing parameters as the actual test, load the specimen to a point at or above the fracture loads observed during actual testing. From the load-displacement

curve, determine ui (P). The corrected displacement is then calculated using u_c (P) = u_Q (P) - u_i (P).

7.2.2 In practice, it is common to obtain a linear displacement correction curve (up to the fracture loads observed during actual testing). This simplifies the displacement correction to be applied to the fracture test. Initial non-linearity due to penetration of the loading pins into the applied specimen should occur during both the calibration test and the actual fracture test. Linearization of the near-zero correction data and the fracture test data can compensate for this initial non-linearity.

7.2.3 The displacement correction must be performed for each material and at each test temperature or rate. Polymers are generally temperature- and rate-sensitive and the degree of loading-pin penetration and sample compression can vary with changes in these variables.

7.2.4 The indentation tests should be performed in such a way that the loading times are the same as the fracture tests. Since the indentations are stiffer, this will involve lower rates to reach the same loads.

7.3 Calculation of G_Q (Reference ASTM D5045, Section 9.3) In principle, G_{1C} can be obtained from the following:

$G_{1C} = (1 - \nu^2) K_{1C}^{\ 2} / E$ [Ref. 2]

but for plastics, E must be obtained at the same time and temperature conditions as the fracture test because of viscoelastic effects. Many uncertainties are introduced by this procedure and it is considered preferable to determine G_{1C} directly from the energy derived from integration of the load versus displacement curve up to the same load point as used for K_{1C} and shown in Figures 7-3 (a and b).

7.3.1 The energy must be corrected for system compliance, loading-pin penetration, and specimen compression. This is done by correcting the measured displacement values, as shown in Figure 7-3 (a and b). Accordingly, if complete linearity is obtained, one form of the integration for energy is as U = 1/2 P_Q (u_Q - u_i), where P_Q is defined in 7.1.2.

7.3.2 Alternatively, it is possible to use the integrated areas from the measured curve, U_Q , of Figure 7-3, a and indentation curves, U_i , of Figure 7-3, b in accordance with 7.3.3 and following.

U = U_Q - U_i [Ref.3, SENB].

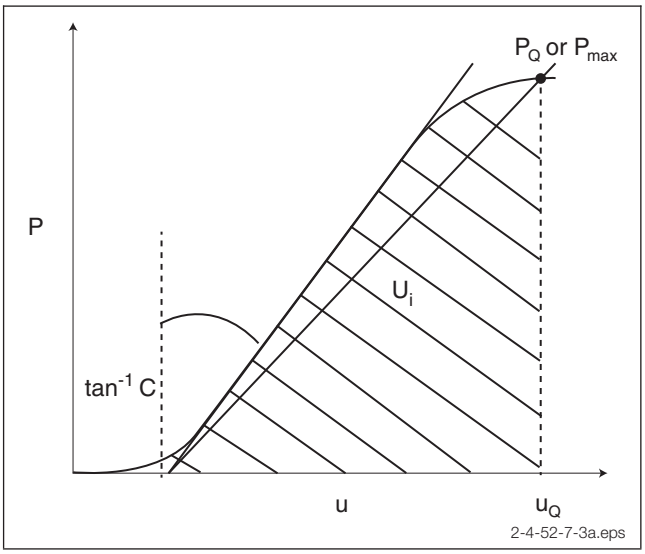

Figure 7-3 (a) Method of Correcting for Indentation; Load - Deflection in Fracture Test

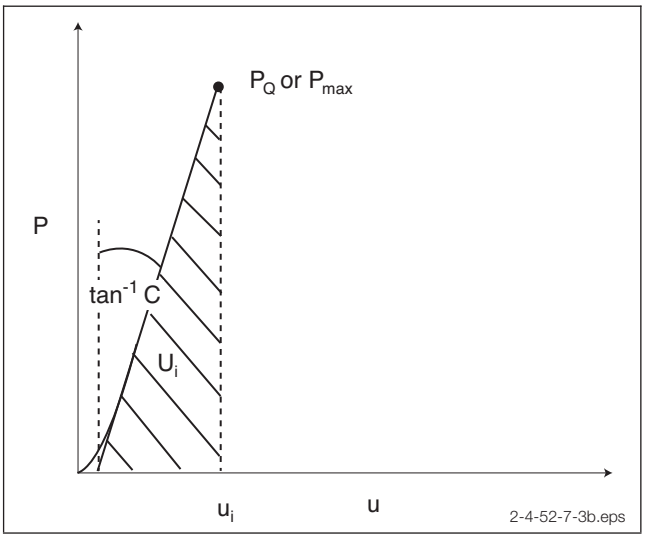

Figure 7-3 (b) Method of Correcting for Indentation; Load - Deflection in Indentation

7.3.3 Calculate G_Q from U in accordance with the procedure given in 7.1.5.

7.3.4 A useful cross check on accuracy may be made using the tensile modulus, E, and Poisson's ratio, v. $E/(1 - \nu^2)$ **shall** be calculated from the corrected compliance, C_c, using the following:

$(E / (1 - \nu^2))$ B $C_c = 2f^2 \Phi = \psi$ [Ref. 4, SENB]

The factors f, Φ and ψ are given in Table 7-1 and Table 7-2 for each geometry. This value of $E/(1 - v^2)$ **shall** be compared with that obtained from K_{1c} 2 $/G_{1c}$. The former value should be the larger, but the difference should be <15 %. The corrected compliance, C_c, is obtained from the measured compliance in the fracture test, C_Q, and the compliance from the indentation test, Ci, in accordance with the following:

$C_c = C_Q - C_i$ [Ref. 5, SENB]

Table 7-2 Calibration Factors Compact Tension[A]

a/W	*f (x)*	Φ	ψ	η_e
0.450	8.34	0.208	28.9	2.64
0.455	8.45	0.207	29.6	2.63
0.460	8.57	0.207	30.4	2.61
0.465	8.70	0.206	31.1	2.60
0.470	8.83	0.205	31.9	2.58
0.475	8.96	0.204	32.7	2.57
0.480	9.09	0.203	33.5	2.56
0.485	9.23	0.202	34.4	2.54
0.490	9.36	0.201	35.3	2.53
0.495	9.51	0.200	35.3	2.53
0.500	9.65	0.199	37.1	2.51
0.505	9.81	0.198	38.0	2.50
0.510	9.96	0.197	39.0	2.49
0.515	10.12	0.196	40.0	2.48
0.520	10.28	0.194	41.1	2.47
0.525	10.45	0.193	42.1	2.46
0.530	10.62	0.192	43.3	2.45
0.535	10.80	0.190	44.4	2.44
0.540	10.98	0.189	45.6	2.43
0.545	11.17	0.188	46.8	2.42
0.550	11.36	0.186	48.1	2.41

[A] *Values calculated using J. A. Knapp, G. S. Leger and B. Gross, Fracture Mechanics Sixteenth Symposium, ASTM, STP 868, 19, pp. 27 - 44.*

7.4 Report List the information required to perform the test and the results obtained in the form of a table. The form to use is provided in Table 7-3.

7.4.1 Table 7-4 is based on a round robin conducted in 1988 in accordance with E-691, involving four materials tested by nine laboratories. For each material, all the samples were prepared at one source, but the individual specimens were prepared at the laboratories which tested them. Each test result was the average of three individual determinations.

Table 7-3 Testing Summary

Fracture Test Parameters
Testing Laboratory
Materials/orientation
Specimen geometry
Test temperature, °C
Loading rate, m/s
Notching method
Specimen number
Width (W), mm
Crack length from 7.2.2, mm
P_{max}, N
P_{max} loading rate, s
PQ loading time, s
Stable or unstable growth
K_Q, MPa - $m^{1/2}$
Uncorrected energy, J
Corrected energy, J
G_{Ic}, kJ/m^2
Tensile Test Parameters
σy, MPa
σy loading time, s
Validity Checks
Pmax/PQ
2.5 $(KQ/sy)^2$
$E/(1 - \nu^2)$ via C, MPa
$E/(1 - \nu^2)$ via K_Q^2/G_c, MPa

Table 7-4 Precision Statistics from Round-Robin Study in Accordance with Practice ASTM E691

Material[A]	Average	S_x	S_r	S_R	*r*	*R*
A	4.34	0.652	0.235	0.679	0.658	1.90
B	5.70	1.420	0.618	1.510	1.730	4.23
C	3.60	0.692	0.343	0.747	0.960	2.09
D	5.90	1.950	0.944	2.100	2.640	7.39

[A] Material A is values of K_{Ic} for nylon. Material B is values of G_{Ic} for nylon. Material C is values of K_{Ic} for polycarbonate. Material D is values of G_{Ic} for polycarbonate. Units for all columns are as follows: K_{Ic} [=] $MP_a \cdot m^{1/2}$ & G_{Ic} [=] kJ/m^2.

Each laboratory obtained one test result for each material. The following explanations of r and R are only intended to present a meaningful way of considering the approximate precision of this test method. The data in Table 7-4 should not be rigorously applied to acceptance or rejection of material, as those data are specific to the round robin and may not be representative of other lots, conditions, materials, or laboratories.

Users of this test method should apply the principles outlined in E-691 to generate the data specific to their laboratory and materials, or between specific laboratories. The principles would then be valid for such data.

7.4.2 Concept of r and R (Reference ASTM D5045, Section 11.2). If Sr and SR have been calculated from a large enough body of data, and for test results that were averages from testing three specimens, the following information applies.

7.4.2.1 Repeatability, r (comparing two test results for the same material, obtained by the same operator using the same equipment on the same day). The two test results should be judged not equivalent if they differ by more than the r value for that material.

7.4.2.2 Reproducibility, R (comparing two test results for the same material, obtained by different operators using different equipment on the same day). The two test results should be judged not equivalent if they differ by more than the R value for that material.

7.4.2.3 Any judgement in accordance with the above would have an approximate 95 % (0.95) probability of being correct.

7.4.3 Bias There are no recognized standards by which to estimate bias of these test methods.

7.4.4 Keywords (Reference ASTM D 5045, Section 12)

- Critical-strain energy release rate
- Energy-to-break
- Fracture toughness
- Plane-strain fracture toughness

References

(1) Brown, W. F., Jr., and Srawley, J. E., "Plane Strain Crack Toughness Testing of High Strength Metallic Materials," *ASTM STP 410*, ASTM, 1966, p.1.

(2) "Fracture Toughness Testing and Its Applications," *ASTM STP 381*, ASTM, April 1965, p.30.

(3) Srawley, J. E., "Wide Range Stress Intensity Factor Expressions for ASTM E399 Standard Fracture Toughness Specimens," *International Journal of Fracture Mechanics*, Vol. 12, June 1976, p.475.

(4) Newman, J. C., "Stress Analysis of Compact Specimens Including the Effects of Pin Loading," *ASTM STP 560*, ASTM, 1974, p.105.

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(6) Towers, O. L., "Stress Intensity Factors, Compliances and Elastic η_e Factors for Six Test Geometries," *The Welding Institute*, March 1981.