Designation: D3433 – 99 (Reapproved2005)

Standard Test Method for Fracture Strength in Cleavage of Adhesives in Bonded Metal Joints[1]

This standard is issued under the fixed designation D3433; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method(**1**, **2**, **5**, **6**, **9**)[2] covers the determination of fracture strength in cleavage of adhesives when tested on standard specimens and under specified conditions of preparation and testing (Note 1).

1.2 This test method is useful in that it can be used to develop design parameters for bonded assemblies.

NOTE 1—While this test method is intended for use in metal-to-metal applications it may be used for measuring fracture properties of adhesives using plastic adherends, provided consideration is given to the thickness and rigidity of the plastic adherends.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

A167 Specification for Stainless and Heat-Resisting Chromium-Nickel Steel Plate, Sheet, and Strip
A366/A366M Specification for Commercial Steel (CS) Sheet, Carbon, (0.15 Maximum Percent) Cold-Rolled (Withdrawn 2000)[4]
B36/B36M Specification for Brass Plate, Sheet, Strip, And Rolled Bar
B152/B152M Specification for Copper Sheet, Strip, Plate, and Rolled Bar
B209 Specification for Aluminum and Aluminum-Alloy Sheet and Plate
B265 Specification for Titanium and Titanium Alloy Strip, Sheet, and Plate
D907 Terminology of Adhesives
E4 Practices for Force Verification of Testing Machines
E399 Test Method for Linear-Elastic Plane-Strain Fracture Toughness K_{Ic} of Metallic Materials

3. Terminology

3.1 *Definitions:* Many of the terms used in this test method are defined in Terminology D907.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *crack-extension force, G,*—the system isolated (fixed load-displacement) loss of stress field energy for an infinitesimal increase, d A, of separational area. In equation form,

$$G\,dA = -dU_T \tag{1}$$

where U_T = total elastic energy in the system (component or test specimen). In the test specimens of this method, the crack front is nearly straight through the specimen thickness, B, so that $dA = B\,da$, where da is an infinitesimal forward motion of the leading edge of the crack. Completely linear-elastic behavior is assumed in the calculations (See Annex A1) of G used in this method, an allowable assumption when the zone of nonlinear deformation in the adhesive is small relative to specimen dimensions and crack size.

3.2.1.1 When the shear stress on the plane of crack and forward to its leading edge is zero, the stress state is termed "opening mode." The symbol for an opening mode G is G_I for plane-strain and G_1 when the connotation of plane-strain is not wanted.

3.2.2 *opening mode fracture toughness,* G_{1c}—the value of G just prior to onset of rapid fracturing when G is increasing with time.

3.2.3 *opening mode crack arrest toughness,* G_{1a}—the value of G just after arrest of a run-arrest segment of crack extension.

3.2.3.1 It is assumed that the dimensions of the part containing the crack are large compared to the run-arrest segment

[1] This test method is under the jurisdiction of ASTM Committee D14 on Adhesives and is the direct responsibility of Subcommittee D14.80 on Metal Bonding Adhesives.

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[2] The boldface numbers in parentheses refer to the references at the end of this test method.

[3] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[4] The last approved version of this historical standard is referenced on www.astm.org.

which precedes crack arrest and that the quasi-static stress field enclosing the crack tip just after crack arrest can be assumed in calculating G_{1a}.

4. Summary of Test Method

4.1 This test method involves cleavage testing bonded specimens such that a crack is made to extend by a tensile force acting in a direction normal to the crack surface.

4.2 Load versus load-displacement across the bondline is recorded autographically. The G_{1c} and G_{1a} values are calculated from this load by equations that have been established on the basis of elastic stress analysis of specimens of the type described below. The validity of the determination of G_{1c} and G_{1a} values by this test method depends upon the establishment of a sharp-crack condition in the bondline in a specimen of adequate size. This test method will measure the fracture strength of a bonded joint which is influenced by adherend surface condition, adhesive, adhesive-adherend interactions, primers, adhesive-supporting scrims, etc., and in which of the above possible areas the crack grows.

5. Significance and Use

NOTE 2—Crack growth in adhesive bond specimens can proceed in two ways: (*1*) by a slow-stable extension where the crack velocity is dictated by the crosshead rate or (*2*) by a run-arrest extension where the stationary crack abruptly jumps ahead outrunning the crosshead-predicted rate. The first type of crack extension is denoted flat; the second type peaked because of the appearance of the autographic record. The flat behavior is characteristic of adhesives or test temperatures, or both, for these adhesives where there is no difference between initiation, G_{1c}, and arrest, G_{1a}. For example, the rubber modified film adhesives tested above − 17.8°C (0°F) all exhibit flat autographic records. Peaked curves are exhibited for all modified materials tested below −73°C (−100°F) and in general for unmodified epoxies.

It should be noted that both peaked and flat behaviors are determined from a crack-length-independent specimen. For other specimens or structures where G increases with a at constant load the onset of crack growth would result in rapid complete fracturing whatever the adhesive characteristics.

5.1 The property G_{1c} (and G_{1a} if relevant) determined by this test method characterizes the resistance of a material to slow-stable or run-arrest fracturing in a neutral environment in the presence of a sharp crack under severe tensile constraint, such that the state of stress near the crack front approaches tritensile plane strain, and the crack-tip plastic region is small compared with the crack size and specimen dimensions in the constraint direction. It has not been proven that tough adhesive systems fully meet this criteria. Therefore, data developed using equations based on this assumption may not represent plane-strain fracture values. Comparison of fracture toughness between adhesive systems widely different in brittleness or toughness should take this into consideration. In general, systems of similar type toughness (**3**, **4**, **7**, **8**, **10**) can be compared as can the effect of environment on toughness of a single system. A G_{1c} value is believed to represent a lower limiting value of fracture toughness for a given temperature, strain rate, and adhesive condition as defined by manufacturing variables. This value may be used to estimate the relation between failure stress and defect size for a material in service wherein the conditions of high constraint described above would be expected. Background information concerning the basis for development of this test method in terms of linear elastic fracture mechanics may be found in Refs (**6**) and (**7**).

5.1.1 Cyclic loads can cause crack extension at G_1 values less than G_{1c} value. Furthermore, progressive stable crack extension under cyclic or sustained load may be promoted by the presence of certain environments. Therefore, application of G_{1c} in the design of service components should be made with awareness of the G increase for a prior crack which may occur in service due to slow-stable crack-extension.

5.2 This test method can serve the following purposes:

5.2.1 In research and development to establish, in quantitative terms, significant to service performance, the effects of adhesive composition, primers, adherend surface treatments, supporting adhesive carriers (scrim), processing variables, and environmental effects.

5.2.2 In service evaluation to establish the suitability of an adhesive system for a specific application for which the stress conditions are prescribed and for which maximum flaw sizes can be established with confidence.

5.2.3 For specifications of acceptance and manufacturing quality control, but only when there is a sound basis for specification of minimum G_{1c} values. The specification of G_{1c} values in relation to a particular application should signify that a fracture control study has been conducted on the component in relation to the expected history of loading and environment, and in relation to the sensitivity and reliability of the crack detection procedures that are to be applied prior to service and subsequently during the anticipated life.

6. Apparatus

6.1 *Testing Machine*, conforming to the requirements of Practices E4. Select the testing machine such that the cracking load of the specimens falls between 15 and 85 % of the full-scale capacity and that is provided with a suitable pair of self-aligning pinned fixtures to hold the specimen.

6.2 Ensure that the pinned fixtures and attachments are constructed such that they will move into alignment with the test specimen as soon as the load is applied.

6.3 For a discussion of the calculation of separation rates see Annex A1.

7. Test Specimens

7.1 *Flat Adherend,* conforming to the form and dimensions shown in Fig. 1, cut from test joints as in Fig. 2, prepared as prescribed in Section 8.

7.2 *Contoured Double-Cantilever Beam (CDCB),* conforming to the form and dimensions shown in Fig. 3.

7.3 The following grades of metals are suggested for the test specimens (Note 3):

Metal	ASTM Designation
Brass	B36/B36M, Alloy 260 **(6)**, quarter hard temper
Copper	B152/B152M, cold rolled, Type 110, hard temper
Aluminum	B209, Alclad 2024, T3 temper, mill finish
Steel	A366/A366M, regular matte finish
Corrosion-resisting steel	A167, Type 304, No. 2B finish
Titanium	B265, Grade 3

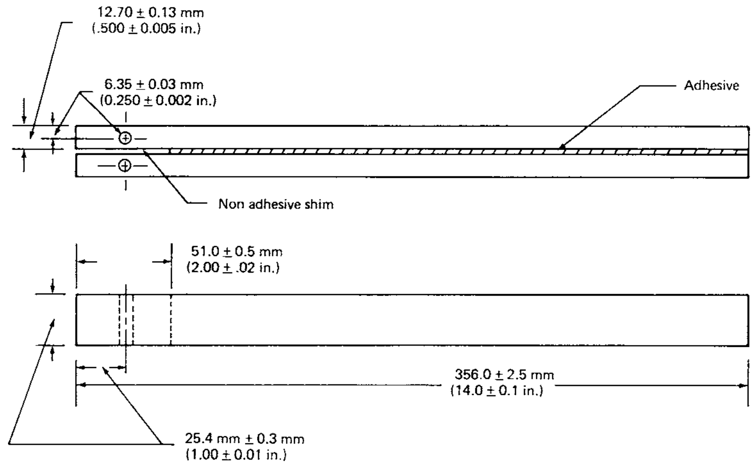


FIG. 1 Flat Adherend Specimen

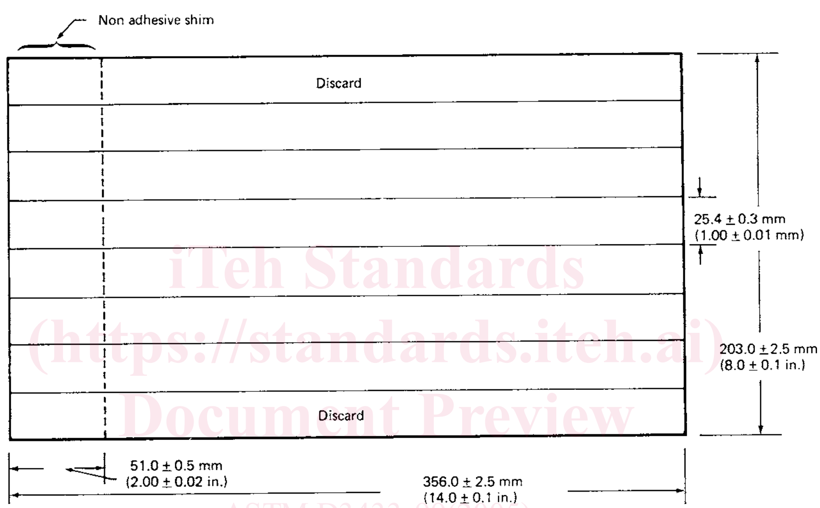


FIG. 2 Test Joint

7.4 Test at least twelve specimens, representing at least four different joints.

NOTE 3—Since it is unacceptable to exceed the yield point of the metal in flexure during test, the permissible thickness of the specimen will vary with type of metal, and the general level of strength of the adhesive being investigated. The minimum permissible thickness in a uniform symmetrical adherend may be computed from the following relationship:

$$h = \sqrt{\frac{6\,Ta}{BF_{ty}}} \qquad (2)$$

where:

h = thickness of metal normal to plane of bonding, mm (or in.),
F_{ty} = tensile yield point of metal (or the stress at proportional limit) MPa (or psi),
T = 150 % of the maximum load to start the crack in the adhesive bond, N (or lbf),
a = crack length at maximum load, mm (or in.), and
B = bond width, mm (or in.).

8. Preparation of Test Joints

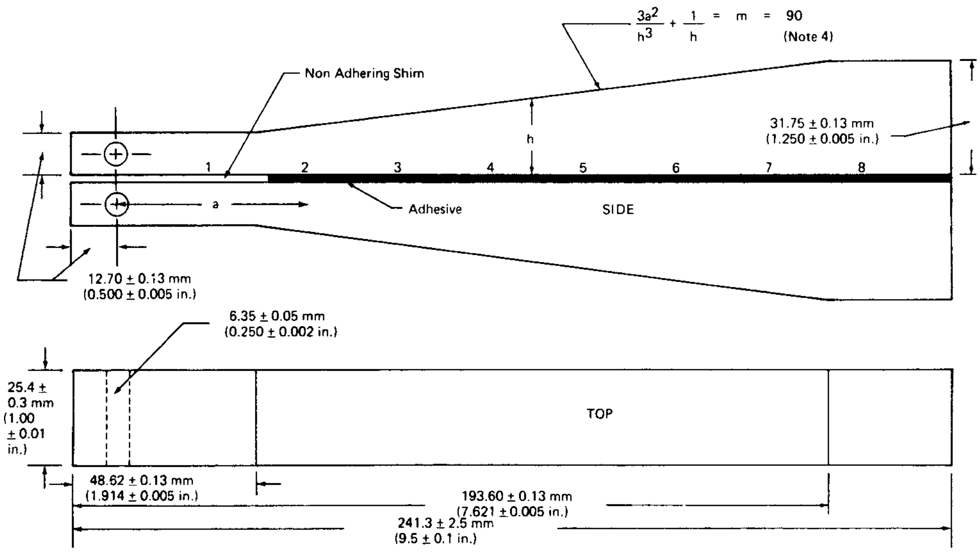
8.1 Cut sheets of the metals or contoured adherends prescribed in 7.1-7.3 and to recommended size (Figs. 2 and 3). All edges of the metal panels and specimens must be flat, free of burrs, and smooth (4.1-µm (160-µin.) maximum) before the panels are surface-treated and bonded. Clean, treat, and dry the sheets or contoured adherends carefully, in accordance with the procedure prescribed by the manufacturer of the adhesive. Prepare and apply the adhesive in accordance with the recommendations of the manufacturer of the adhesive. Apply the adhesive to the faying surface of one or both metal sheets. Then assemble the sheets, faying surface to faying surface in pairs, and allow the adhesive to cure under conditions prescribed by the manufacturer of the adhesive.

8.2 It is recommended that each "flat adherend" test joint be made with sufficient area to provide at least five test specimens.

9. Preparation of Test Specimens

9.1 For flat adherend test specimens, trim joint area in accordance with Fig. 2. Then cut test specimens, as shown in Fig. 1, from the joints, Fig. 2 (Note 4). Then cut holes for load pins as shown in Fig. 1.

9.2 Contoured double-cantilever specimens are ready for test as bonded.



m = 90

h	a
0.500 (in.)	1.914 (in.)
0.600 (in.)	2.522 (in.)
0.700 (in.)	3.182 (in.)
0.800 (in.)	3.892 (in.)
0.900 (in.)	4.646 (in.)
1.000 (in.)	5.447 (in.)
1.100 (in.)	6.287 (in.)
1.200 (in.)	7.167 (in.)
1.250 (in.)	7.621 (in.)

FIG. 3 Contoured Double-Cantilever Beam Specimen

NOTE 4—Do not use lubricants or oils during the cutting process. For aluminum it is suggested that the specimens be rough cut 3.2 mm (⅛ in.) over-size using a four-pitch band saw traveling at approximately 4.2 m/s (800 ft/min) followed by finish dimensioning to a 1-in. wide 3.2-µm (125-µin.) surface using a five-blade 15-deg carbide fly cutter at 1115 rpm and 0.015 to 0.035-m/s (3 to 7-in./min) feed rate.

10. Procedure

10.1 Test specimens, prepared as prescribed in Section 8, in an atmosphere maintained at 50 ± 4 % relative humidity and 23 ± 1°C (73.4 ± 1.8°F). Tests at other than ambient temperature may be run if desired. It is suggested that specimens be conditioned for a minimum of 10 min and a maximum of 30 min at the temperature of test to assure equilibrium. The manufacturer of the adhesive may, however, prescribe a definite period of conditioning under specific conditions before testing.

10.2 Determine the following test specimen dimensions.

10.2.1 Distance from center of 6.4-mm (0.25-in.) inside-diameter pin holes to close end of specimen.

10.2.2 Width of test specimen, *b*.

10.2.3 Thickness of test specimen 127 mm (5 in.) from pin end and 227 mm (9 in.) from pin end.

10.2.4 Bond line thickness 125 mm (5 in.) from pin end and 227 mm (9 in.) from pin end.

10.3 Load the specimen in the test machine and pin in position using the 6.4-mm (0.25-in.) inside-diameter pin holes. Balance the recorder or chart, or both. Set the test machine at a crosshead separation rate △ chosen to keep time-to-fracture in the order of 1 min, see 6.1 and Annex A1. For example, 2 mm/min (0.08 in./min) gives fracture in 1 min for a CDCB ½-in. wide $m = 90\text{-in.}^{-1}$ aluminum adherend specimen having a 3-in. long starter crack.

10.3.1 The chart recording should be such that maximum load occurs on the record and that at least 13 mm (½ in.) of motion is represented on the abscissa (△) for each 100 mm (4 in.) of ordinate motion (*P*). For load-time records a chart *speed rate* should be used such that the slope of the load versus time record is similar to that specified for load versus load-displacement (for example, 5 mm/min (0.2 in./mm)).

10.4 Apply load to specimen until Point *A* is reached. (See Point *A*, Fig. 4 for flat adherend and Fig. 5, Point *A* for contoured double-cantilever specimen.) Point *A* is the load at which the crack begins to grow rapidly. Then stop loading and follow crack growth curve on the chart. When the load has leveled off at an approximate constant value (the crack has stopped growing), determine and record the following values:

10.4.1 Load to start crack, *L* (max), N (or lbf),

10.4.2 Load when crack stops, *L* (min), N (or lbf), and

10.4.3 Distance from loading end of specimen to the stationary crack tip in millimetres (or inches).

10.5 Repeat 10.4 to yield five determinations on each specimen.

11. Calculation

11.1 *Flat Adherend Specimen:*

11.1.1 Calculate the fracture toughness, G_{1c} (from load to start crack), in joules per square metre or pounds-force per inch as follows: