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# Standard Test Method for Determination of Fracture Toughness of Graphite at Ambient Temperature<sup>1</sup>

This standard is issued under the fixed designation D7779; 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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope\*

1.1 This test method covers and provides a measure of the resistance of a graphite to crack extension at ambient temperature and atmosphere expressed in terms of stress-intensity factor, K, and strain energy release rate, G. These crack growth resistance properties are determined using beam test specimens with a straight-through sharp machined V-notch.

1.2 This test method determines the stress intensity factor, K, from applied force and gross specimen deflection measured away from the crack tip. The stress intensity factor calculated at the maximum applied load is denoted as fracture toughness,  $K_{Ic}$ , and is known as the critical stress intensity factor. If the resolution of the deflection gauge is sensitive to fracture behavior in the test specimen and can provide a measure of the specimen compliance, strain energy release rate, G, can be determined as a function of crack extension.

1.3 This test method is applicable to a variety of grades of graphite which exhibit different types of resistance to crack growth, such as growth at constant stress intensity (strain energy release rate), or growth with increasing stress intensity (strain energy release rate), or growth with decreasing stress intensity (strain energy release rate). It is generally recognized that because of the inhomogeneous microstructure of graphite, the general behavior will exhibit a mixture of all three during the test. The crack resistance behavior exhibited in the test is usually referred to as an "R-curve."

Note 1—One difference between the procedure in this test method and test methods such as Test Method E399, which measure fracture toughness,  $K_{Ic}$ , by one set of specific operational procedures, is that Test Method E399 focuses on the start of crack extension from a fatigue precrack for metallic materials. This test method for graphite makes use of a machined notch with sharp cracking at the root of the notch because of the nature of graphite. Therefore, fracture toughness values determined with this method may not be interchanged with  $K_{Ic}$  as defined in Test Method E399.

1.4 This test method gives fracture toughness values,  $K_{Ic}$  and critical strain energy release rate,  $G_{Ic}$  for specific conditions of environment, deformation rate, and temperature. Fracture toughness values for a graphite grade can be functions of environment, deformation rate, and temperature.

1.5 This test method is divided into two major parts. The first major part is the main body of the standard, which provides general information on the test method, the applicability to materials comparison and qualification, and requirements and recommendations for fracture toughness testing. The second major part is composed of annexes, which provide information related to test apparatus and test specimen geometry.

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1.6 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6.1 Measurement units expressed in these test methods are in accordance with IEEE/ASTM SI 10.

1.7 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.

1.8 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

<sup>&</sup>lt;sup>1</sup> This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.F0 on Manufactured Carbon and Graphite Products.

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## 2. Referenced Documents

- 2.1 ASTM Standards:<sup>2</sup>
- C1161 Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature
- C1421 Test Methods for Determination of Fracture Toughness of Advanced Ceramics at Ambient Temperature
- E4 Practices for Force Verification of Testing Machines
- E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
- E337 Test Method for Measuring Humidity with a Psychrometer (the Measurement of Wet- and Dry-Bulb Temperatures)
- E399 Test Method for Linear-Elastic Plane-Strain Fracture Toughness of Metallic Materials
- E561 Test Method for $K_R$  Curve Determination
- E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E1823 Terminology Relating to Fatigue and Fracture Testing E2309 Practices for Verification of Displacement Measuring

Systems and Devices Used in Material Testing Machines IEEE/ASTM SI 10 Standard for Use of the International System of Units (SI) (The Modern Metric System)

## 3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 The terms described in this test method and E1823 are applicable to the test methods prescribed herein. Appropriate sources for each definition are provided after each definition in parentheses.

3.1.2 *crack depth, a [L], n*—length of the crack in a notched beam specimen, which includes the machined notched length and the crack length which the crack has traveled during testing. Any contributions from crack branching or other secondary cracking are not included in this measurement.

3.1.3 crack extension orientation, n—direction of propagation in relation to a characteristic direction of the graphite specimen. This identification may be designated by a letter or letters indicating the plane and direction of crack extension. The letter or letters represent the direction normal to the crack plane and the direction of crack propagation.

3.1.3.1 *Discussion*—The characteristic direction should be associated with the microstructural grain orientation of the test specimen.

3.1.3.2 *Discussion*—The crack plane can be defined by letter(s) representing the direction of tensile stress normal to the crack plane. And the direction of crack extension can be defined by letter(s) representing the direction parallel to the characteristic grain orientation of the test specimen. As illustrated in Annex A1, the tensile stress direction is notated first, followed by a hyphen, and then the crack extension direction. The legend given in Test Methods C1421 includes the following:

- M =molding direction
- EX = extrusion direction
- AXL = axial, or longitudinal axis (if M or EX are not applicable)
- R = radial direction
- C = circumferential direction

R/C = mixed radial and circumferential directions

3.1.3.3 *Discussion*—For a graphite test specimen of rectangular cross section, R and C may be replaced by rectilinear coordinate axes, x and y, corresponding to two adjacent sides of the test specimen.

3.1.3.4 *Discussion*—Depending on how test specimens are cut from a graphite product, the crack plane may be longitudinal to the forming direction, or circumferential, or radial, or a mixture of these directions as shown in Annex A1.

3.1.3.5 *Discussion*—For the test specimen the plane and direction of crack extension with respect to the applied tensile stress should be recorded. Report the orientation of the specimen and crack propagation direction with respect to the grain direction.

3.1.3.6 *Discussion*—If there is no primary product direction, reference axes may be arbitrarily assigned but must be clearly identified.

3.1.4 crack extension resistance,  $K_R[FL^{-3/2}]$ ,  $G_R[FL^{-1}]$ , or  $J_R[FL^{-1}]$ , *n*—measure of the resistance of a material to crack extension expressed in terms of the stress-intensity factor, *K*, strain energy release rate, *G*, or values of *J* derived using the J-integral concept.

3.1.5 critical crack depth, [L], n—crack depth at which catastrophic fracture initiation occurs, corresponding to the maximum in the applied load.

3.1.6 fracture toughness,  $K[FL^{-3/2}]$ , *n*—property which defines the critical stress intensity factor necessary to initiate a crack for subsequent propagation on further loading.

3.1.7 *R-curve*, n—plot of stress intensity or strain energy release rate as a function of stable crack extension and provides a measure of crack propagation trend in the material. **E561** 

3.1.8 *slow crack growth, (SCG), n*—sub-critical crack growth (extension) which may result from, but is not restricted to, such mechanisms as environmentally-assisted stress corrosion or diffusive crack growth, usually at constant load.

3.1.9 *small crack, n*—being small when all physical dimensions (in particular, with length and depth of a surface crack) are small in comparison to a relevant microstructural scale, continuum mechanics scale, or physical size scale. The specific physical dimensions that define "small" vary with the particular material, geometric configuration, and loadings of interest. **E1823** 

3.1.10 *stable crack extension, n*—crack propagation which provides measurable data of the dependence of stress intensity factor on crack extension and which occurs over some measurable time duration.

3.1.11 stress-intensity factor,  $K[FL^{-3/2}]$ , *n*—magnitude of the ideal-crack-tip stress field (stress field singularity) for a particular mode in a homogeneous, linear-elastic body. **E1823** 

<sup>&</sup>lt;sup>2</sup> 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.

3.1.12 *three-point flexure, n*—flexure configuration where a beam test specimen is loaded at a location midway between two support bearings. C1161

3.1.13 *unstable crack extension*—uncontrollable crack propagation which yields no measurable data of the dependence of stress intensity factor on crack extension.

3.2 Symbols:

3.2.1 *a*—crack depth, including the machined notch (see Fig. 1).

3.2.2 *a/W*—normalized notch depth.

3.2.3 *B*—the specimen width (see Fig. 1).

3.2.4 g(a/W)—geometric function of the ratio a/W.

3.2.5 *L*—test specimen length (see Fig. 1).

3.2.6 P-force.

3.2.7  $P_{max}$ —maximum force.

3.2.8 S—support span (see Fig. A1.2).

3.2.9 W—the specimen depth (see Fig. 1).

## 4. Summary of Test Method

4.1 This test method involves an application of force to a beam test specimen in three-point flexure. The test specimen contains a straight-through notch in the center. The equations for calculating the fracture toughness have been established on the basis of linear-elastic stress analyses.

4.2 Notched Beam Method—A straight-through notch is machined in a beam test specimen. The applied force on the notched test specimen as a function of time and actuator displacement or specimen deflection in three-point flexure, or a combination thereof, are recorded for analysis. The fracture toughness,  $K_{Ic}$ , is calculated from the maximum (fracture) force, the test specimen dimensions, the measured notch depth, and the support span of the test fixture. Calculation of strain energy release rate, G, requires a determination of specimen compliance, and crack length at each load point of the load versus displacement curve. The maximum G derived from the strain energy release rate versus crack growth curve is recorded.

#### 5. Significance and Use

5.1 This test method may be used for guidance for material development to improve toughness, material comparison, quality assessment, and characterization.

5.2 The fracture toughness value provides information on the initiation of fracture in graphite containing a straightthrough notch; the information on stress intensity factor





beyond fracture toughness as a function of crack extension provides information on the crack propagation resistance once a fracture crack has been initiated to propagate through the test specimen.

## 6. Apparatus

6.1 *Testing*—Test the specimens in a testing machine that has provisions for autographic recording of force applied to the test specimen versus time and actuator displacement or deflection of the specimen, or both, in the notch plane. The testing machine shall conform to the requirements of Practice E4.

6.2 *Deflection Measurement*—The deflection gauge should be capable of resolving 0.001 mm. Practices E2309 cover procedures and requirements for the calibration and verification of displacement measuring systems.

6.3 *Recording Equipment*—Provide digital data acquisition for automatically recording the applied force versus displacement.

6.4 *Fixtures*—Use a three-point test fixture constructed with high stiffness materials (see Fig. A1.2). Choose the outer support span, *S*, such that  $5 \le (S/W) \le 10$ . The outer two rollers shall be free to roll outwards from support locations to minimize friction effects. The middle flexure roller shall be fixed. The specimen should overhang each of the outer rollers by a minimum distance equal to the specimen dimension, *W*.

6.5 *Dimension-Measuring Devices*—Measure and report all applicable specimen dimensions to an accuracy of 0.013 mm. Flat, anvil-type micrometers shall be used for measuring test specimen dimensions. Ball-tipped or sharp-anvil micrometers are not recommended as they may damage the test specimen surface by inducing localized cracking. Non-contacting (for example, optical comparator, light microscopy, etc.) measurements are recommended for notch depth measurements. Measure and report the notch depth to an accuracy of 0.0025 mm.

#### 7. Test Specimen

7.1 *Test Specimen Configuration*—The specimen shall have a straight-through machined V-notch with a maximum notch root radius of 0.10 mm. The notch may be sharpened by drawing an industrial razor blade or similar device across the notch tip to encourage stable crack extension from the asmachined notch tip.

7.1.1 The included angle of the razor blade edge should be less than the specimen notch angle. It is recommended that the sharpening process be controlled such that this step is made in a consistent, measurable manner across the width of the notch. Manual sharpening introduces uncertainty in the initial notch depth and may also cause premature failure.

7.1.2 Post fracture inspection of the notch is encouraged, particularly in fine grained graphite, to reveal if notch sharpness consistency has been achieved and to ensure fracture initiation from the notch.

7.1.3 The ease with which a crack initiates from a machined notch depends on both the width of the notch, particularly the notch tip asperity, and the average grain size of the material under consideration. Because typical graphite grades contain