Designation: D8255 – 19

Standard Guide for Work of Fracture Measurements on Small Nuclear Graphite Specimens[1]

This standard is issued under the fixed designation D8255; 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 guide provides general tutorial information and best practice for measuring the work of fracture on manufactured graphite and carbon specimens. Although applicable to all carbon and graphite materials, this guide is aimed specifically at measurements required on nuclear graphites, where there may be constraints on the geometry and/or volume of the test specimen.

1.2 The values stated in SI units are to be regarded as standard. The values given in parentheses after SI units are provided for information only and are not considered standard.

1.3 *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.*

1.4 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C559 Test Method for Bulk Density by Physical Measurements of Manufactured Carbon and Graphite Articles

C651 Test Method for Flexural Strength of Manufactured Carbon and Graphite Articles Using Four-Point Loading at Room Temperature

C695 Test Method for Compressive Strength of Carbon and Graphite

C747 Test Method for Moduli of Elasticity and Fundamental Frequencies of Carbon and Graphite Materials by Sonic Resonance

C749 Test Method for Tensile Stress-Strain of Carbon and Graphite

C769 Test Method for Sonic Velocity in Manufactured Carbon and Graphite Materials for Use in Obtaining an Approximate Value of Young's Modulus

D7775 Guide for Measurements on Small Graphite Specimens

D7779 Test Method for Determination of Fracture Toughness of Graphite at Ambient Temperature

D7972 Test Method for Flexural Strength of Manufactured Carbon and Graphite Articles Using Three-Point Loading at Room Temperature

E4 Practices for Force Verification of Testing Machines

E399 Test Method for Linear-Elastic Plane-Strain Fracture Toughness of Metallic Materials

3. Terminology

3.1 *Definitions:*

3.1.1 *work of fracture,* γ_f (J/m^2), *n*—the total energy required to produce a unit area of fracture surface.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *absorbed energy, n*—the plastic energy absorbed by the system.

3.2.1.1 *Discussion*—This is primarily the work done to extend the crack but can also include other plastic strains related to the specimen, such as microcracking, or the entire system, usually related to the stiffness of the loading frame. These cases are discussed in detail in Section 10.

3.2.2 *total (consumed) energy, n*—the total energy calculated by the load-displacement trace in this type of test.

3.2.2.1 *Discussion*—This is the sum of the elastic energy that leads to elastic deformation of the specimen and the plastic energy, which is primarily the work done to extend the crack.

4. Summary of Guide

4.1 An introduction is provided on the characteristics of nuclear graphite that restrict the number of test methods that are applicable for measuring the Work of Fracture (WoF),

[1] This guide 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.

Current edition approved May 1, 2019. Published June 2019. DOI: 10.1520/D8255-19.

[2] 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.

especially with regard to testing of small irradiated specimens in a shielded facility. This guide takes into account these restrictions and proposes a method for measuring WoF.

4.2 This guide provides the basic principles and experimental setup for the proposed method.

5. Significance and Use

5.1 Structural integrity assessments typically use values of strength and elastic modulus to predict crack initiation in graphite components and there is a suite of ASTM standards (Section 2, Test Methods C651, C695, C747, C749, C769, and Guide D7775) to cover the measurement of these properties.

5.2 The graphite component behavior after crack initiation depends on fracture mechanics parameters, such as fracture toughness and the work of fracture. Test Method D7779 provides the specification and requirements for measuring the fracture toughness of graphite based on linear-elastic stress analysis. Moreover, Test Method D7779 applies to cases where there are no restrictions on specimen size and on applicable machining and specimen preparation techniques.

5.3 Most polycrystalline graphites are non-linear elastic, non-uniform, quasi-brittle materials. For such materials, an effective approach for the determination of fracture properties is the analysis of the global energy balance associated with crack extension, similar to Griffith's theory of brittle fracture. This approach does not have the mathematical complexity of the non-linear elastic fracture and is easier to implement in practice.

5.4 Work of Fracture, γ_f (J/m^2), is defined as the energy required to form a crack divided by the cross sectional area of the crack. It is assumed that the energy per unit area is constant during crack propagation. In general, components that have an excess of strain energy to the point of fracture, compared to the work needed to extend the crack to full dimension, fail by fast fracture. Any excess energy is converted into kinetic energy through a process that generates stress waves. If the amount of excess energy is sufficiently large, the stress waves will have peak magnitudes greater than the material strength, leading to the initiation and propagation of secondary cracks that could result in the fragmentation of the component.

5.5 However, some components that have less strain energy at the point of fracture than the work needed to extend the crack to full dimension, fail in a quasi-brittle manner and result in stable cracks, crack bridging and distributed micro-cracking. Graphite components are generally tested in their as-manufactured state and fail somewhere between these extremes showing fast fracture with relatively minor amounts of secondary cracking and little tendency to fragment. The change in the WoF and strain rate of graphite components in a reactor environment is important in assessing the component's tendency for secondary cracking and fragmentation.

6. Basic Principles

6.1 A widely used approach to measuring fracture parameters for brittle materials is by means of a compact tension or 3-point bend specimen with a chevron-shaped notch machined in the cross-section.[3,4] Because the crack is initiated at the tip of the chevron at very low applied loads, it is not necessary to make assumptions about the crack initiation process. Furthermore, provided the chevron shape is such that the compliance of the specimen increases continuously as the crack propagates, fast fracture cannot occur under displacement-controlled loading and release any excess energy that cannot be measured during the test.

6.2 During such a test, measurements of the load application point displacement and the load applied can be used to determine the energy being dissipated. Assuming that all the energy is used in the formation of the crack and the area of the crack's surface is known, WoF, γ_f, can be determined. With the above caveats in mind, measurement of the load and the deformation at the load points enables the WoF to be directly evaluated from the energy changes that occur as the crack propagates and so it is not necessary to make assumptions about the values of elastic modulus and strength.

6.3 An example case[5] that follows these basic principles is described in Sections 7 – 9.

7. Test Specimen for Example Case

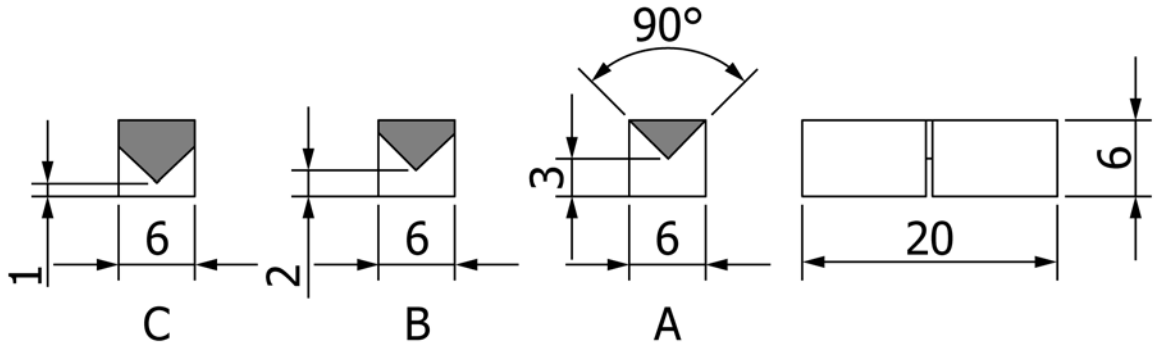
7.1 *Test Specimen Configuration*—The specimens were medium grain graphite rectangular beams with a 90º chevron cut at the mid-point, Fig. 1. In this particular case, the specimen size of interest was a nominal 6 mm × 6 mm × 20 mm beam. However, beams with nominal sizes 10 mm × 10 mm × 50 mm and 20 mm × 20 mm × 100 mm were also tested.

7.2 *Notch Size*—Stable crack growth requires a sufficiently large initial crack length, with the literature suggesting that, for a straight crack front, it should extend to at least half of the beam depth. However, for a very small test specimen as used here, too large a notch would result in the specimen being too delicate to handle, particularly for oxidized specimens. Thus, an important variable is initial crack depth. To investigate this, three separate notch types A, B, and C were tested as shown in Fig. 1. In each case, the notch shape was identical but shifted vertically to provide varying remaining ligament areas of 25 %, 40 %, and 60 %, nominally, of the original beam cross section, (notch types A, B, and C respectively). All notched 6 mm × 6 mm × 20 mm specimens resulted in stable crack growth, but the 25 % remaining ligament area left a very small remaining graphite ligament and may not be appropriate for highly degraded graphite. As it was not certain that the 60 % remaining ligament area would result in stable crack growth for the larger graphite specimens and the literature recommends a deep chevron notch, a remaining ligament area of 40 % was used for the remaining tests. It is recommended that all WoF

[3] Sakai, M., Urashima, K., Inagaki, M., Energy Principle of Elastic-Plastic Fracture and its Application to the Fracture Mechanics of a Polycrystalline Graphite, *Journal of the American Ceramic Society*, Vol 66, No. 12, 1983, pp. 848–874.

[4] Barinov, S. M., Sakai, M., The Work-of-Fractrure of Brittle Materials: Principle, Determination and Applications, *Journal of Materials Research*, Vol 9, No. 6, 1994, pp. 1412–1425.

[5] Tzelepi, A., Ramsay, P., Steer, A. G., Dinsdale-Potter, J., Measuring the Fracture Properties of Irradiated Reactor Core Graphite, *Journal of Nuclear Materials*, Vol 509, 2018.



NOTE 1—The grey region represents the remaining ligament, that is, the connecting region between two halves of the beam.

FIG. 1 Cutting Details of Specimen

measurements start with such an investigation on a limited number of specimens.

7.3 *Test Specimen Preparation*—Standard milling equipment was used for machining the beams to a tolerance of ±0.1 mm. For the small beams, the notch was made using a simple prototype jig with a rotary tool and a 0.5 mm thick diamond cutting wheel, which can be used for specimen preparation in remote-handling facilities. The larger specimens had the chevrons prepared using a miniature table saw. Other types of notching equipment are also acceptable.

8. Apparatus

8.1 *Testing*—The specimens are tested in a testing machine that has provisions for digital recording of force applied to the test specimen versus time and displacement. The testing machine shall conform to the requirements of Practice E4; load cell selection must take into account the relatively low loads to be measured, depending on the material and sample size. The tests are carried out under displacement control, at a speed based on the sample size and compliance. The speeds are chosen such that sufficiently detailed load traces are generated, given the logging speed of the testing machine. It is recommended that the user carries out preliminary tests in order to decide the optimum load speed for the experimental setup. The speeds used to produce the results for this example case were between 0.1 µm⁄s and 5 µm⁄s and the logging speed of the testing machine was 500 registrations per second. The tests are started with the crosshead position above the sample and so exerting no load. The test continues until the sample completely fails and falls from the supports or until the load had dropped to approximately 1 % of the maximum load, as decided by the operator.

8.2 *Fixtures*—A three-point test fixture constructed with high stiffness materials should be used, as specified in Test Method D7972. Different sized test fixtures are used dependent on the specimen size and all incorporate fully articulated rollers to avoid restrictions on the parallelism of the specimen faces. Fig. 2 shows the mounting arrangement for the small nominally 6 mm × 6 mm × 20 mm graphite specimens used in the example case.

8.3 *Dimension-Measuring Devices*—All applicable specimen dimensions should be measured to within 0.05 % and reported as specified in Test Method C559. The area of the chevron notch is determined by marking the chevron notch area after fracture using appropriate image analysis software.

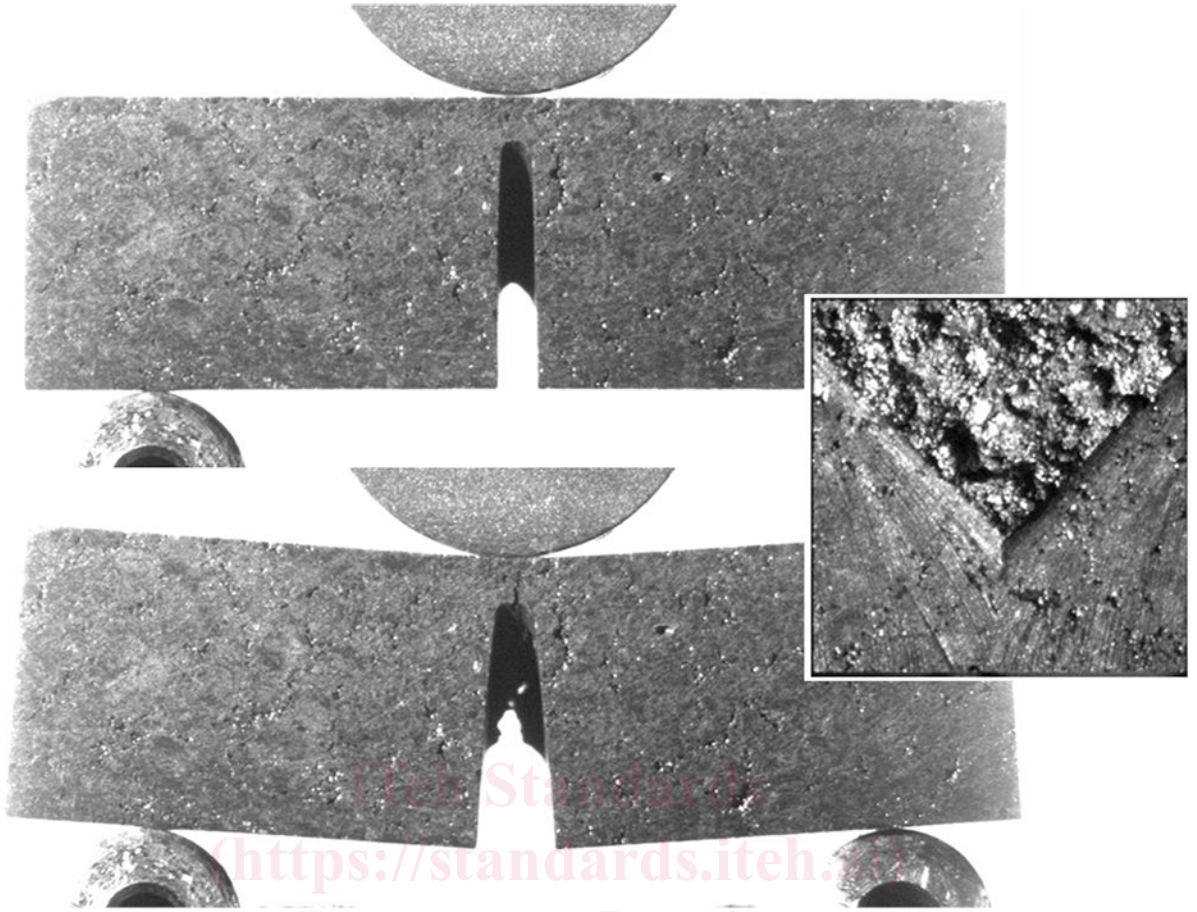
9. Data Analysis

9.1 The load and crosshead displacements were recorded and a typical trace is displayed in Fig. 3 showing the response of a medium-grained near-isotropic graphite. The load values were adjusted to account for the zero load reading prior to contact with the specimen and the zero displacement point was adjusted by extrapolating back the data between 25 % and 75 % of the maximum load. If the stiffness of the specimens is too high, then the test fixture compliance noticeably affects the load trace, as seen by some of the red traces in Fig. 4; in this case, it is recommended to use 50 % to 75 % of maximum load to determine the zero displacement point. The difference in specimen stiffness can be seen in the adjusted traces shown in Fig. 4. For regular beam specimens, the size of the remaining ligament is referenced as the percentage of the beam's total cross sectional area.

9.2 For each data point of the load-displacement curve, the total energy consumed by the specimen/test fixture is determined from the area under the curve, the red area in Fig. 3. The elastic energy, the green area, is subtracted to determine the plastic energy that is absorbed by the specimen; primarily this is the work done to extend the crack, but also includes any work done to create collateral micro-cracking and inelastic, plastic strains. It must be emphasized that the calculation of total energy and elastic energy is performed for every data point of the load trace; the point shown in Fig. 3 is just an example. Typical profiles of the cumulative energy consumed during the tests are shown in Fig. 5. The advantage of this method is that the graph of cumulative energy, as shown in Fig. 5 allows the user to assess whether the test has been extended to completion, that is, whether the plateau of cumulative energy against crosshead displacement has been reached.

9.2.1 Alternatively, the user can simply use the final point of the load-displacement curve, that is to say, calculate the whole area under the curve and then subtract the elastic energy in the same method. In the example load trace of Fig. 3, the final point is at 0.20 mm crosshead displacement.

9.3 After testing, a digital camera is used to image one of the faces of the fracture surface. Using image processing software, a border is manually drawn around the fracture edge



NOTE 1—The small image on the right shows one of the beam halves with the fractured connecting ligament (rough surfaced triangle). The smooth surfaces are the two cuts on one side of the machined groove in the beam.

FIG. 2 Test Specimen Arrangement Pre-Fracture (upper image) and Post-Fracture (lower image)

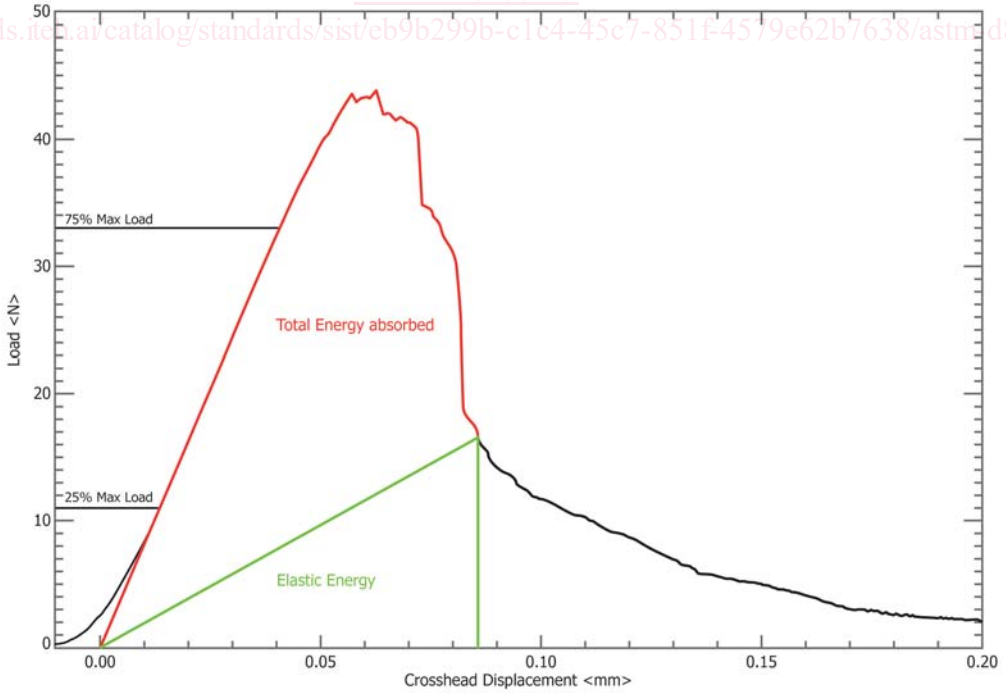


FIG. 3 Data Analysis Method for Example Case