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Standard Guide for
High Temperature Strength Measurements of Graphite Impregnated with Molten Salt[1]

This standard is issued under the fixed designation D8377; 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 covers the best practice for strength measurements at elevated temperature of graphite impregnated with molten salt.

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

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
C749 Test Method for Tensile Stress-Strain of Carbon and Graphite
D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants
D8091 Guide for Impregnation of Graphite with Molten Salt
D8289 Test Method for Tensile Strength Estimate by Disc Compression of Manufactured Graphite
E4 Practices for Force Verification of Testing Machines

3. Terminology

3.1 For definitions of terms used in this guide, refer to Terminology D4175.

4. Summary of Guide

4.1 There is currently a set of ASTM standards as stated in 2.1 that can be applied to graphite for different strength measurements (Test Methods C651, C695, C749, D8289). Each of these standards has been developed specifically to provide a method of measurement for graphite as a single material. However, in some applications such as in molten salt reactors, graphite components are submerged in a molten salt. In order to assess the effect of molten salt on graphite components, a method may be necessary for the measurement of strength for graphite specimens both impregnated with molten salt and at elevated temperatures (see Section 6). The objective of this guide is to provide advice on the application of selected standards for graphite specimens impregnated with molten salt and tested at elevated temperatures. This includes transportation of graphite specimens impregnated with molten salt, temperature measurement, equipment for measuring compressive, tensile, or flexural strength at elevated temperature, and safe handling of the molten salt.

5. Significance and Use

5.1 The Molten Salt Reactor is a nuclear reactor which uses graphite as reflector and structural material, and molten salt as coolant. The graphite components will be submerged in the molten salt during the lifetime of the reactor. The porous structure of graphite may lead to molten salt permeation, which can affect the thermal and mechanical properties of graphite. Consequently, it may be necessary to measure the various strengths of the manufactured graphite materials after impregnation with molten salt and before exposure to the reactor environment in a range of test configurations in order for designers or operators to assess their performance.

NOTE 1—Depending upon the salt selected for the reactor, there may be some chemical reaction between the salt and the graphite that could affect properties. The user should establish, prior to following this guide, that

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[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.

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[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.

***A Summary of Changes section appears at the end of this standard**

any interactions between the molten salt and graphite are understood and any implications for the validity of the strength tests have been assessed.

5.2 For gas-cooled reactors, the strength of a graphite specimen is usually measured at room temperature. However, for molten salt reactors, the operating temperature of the reactor must be higher than the melting temperature of the salt, and so the salt will be in solid state at room temperature. Consequently, room temperature measurements may not be representative of the performance of the material at its true operating conditions. It is therefore necessary to measure the strength at an elevated temperature where the salt is in liquid form.

NOTE 2—Users should be aware that a small increase in graphite strength is expected with increasing temperature. Testing at the plant operating temperature will eliminate this small uncertainty.

5.3 The purpose of this guide is to provide considerations, which should be included in testing graphite specimens impregnated with molten salt at elevated temperature.

5.4 For the test results to be meaningful, the test material must have been impregnated at the reactor operating temperature of interest and the strength measurement conducted at the same temperature following this guide. The user must consider the effect of interaction between graphite and impregnated salt on the representativeness of the strength test based upon the heating and cooling stages between impregnation and testing.

NOTE 3—The user may wish to measure the strength of the same test geometry on unimpregnated graphite both at room temperature and at the chosen elevated temperature as a benchmark prior to the testing of the impregnated material.

NOTE 4—The user may wish to demonstrate that the impregnated test specimen does not lose molten salt during the elevated strength test procedure. This should be performed by putting the impregnated specimen through the exact procedure (temperature and duration) without applying any test load, weighing the specimen before and after the procedure. A reduction in mass will indicate loss of molten salt and the user will then need to assess its significance.

6. Test Specimen Preparation

6.1 The specimen shape and size should be prepared in accordance with the corresponding ASTM standard listed in 2.1 for the desired test configuration (compressive, tensile, or flexural strength measurement).

6.2 The test specimen should then be impregnated with molten salt according to Guide D8091.

6.3 After impregnation, test specimens should be stored at room temperature in a glove box with a water content of less than 1 ppm.

6.4 Hermetic containers should be placed inside the glove box for specimen transfer or movement. The specimen should be placed within a hermetic container when moved from the glove box to a testing machine.

7. Apparatus

7.1 *Testing Machine:*

7.1.1 The testing machine should contain a loading device and a testing compartment. The loading device should provide an appropriate loading force on the specimen. The testing compartment should provide a sealed low-moisture inert gas environment in which to perform the elevated temperature strength measurement.

7.1.2 The loading device should conform to Practice E4 and to the requirements for speed of testing prescribed in the associated ASTM standards listed in 2.1 (Test Methods C651, C695, C749, D8289).

7.1.2.1 The loading device should be qualified by measurements of strength at room temperature using the same assembled machine, pull rods, and grips used for high temperature testing. The specimen shape and size for qualification should be the same as that used during the elevated-temperature tests.

7.1.2.2 Gripping devices and pull rods may oxidize, warp, and creep with repeated use at elevated temperature. This may lead to increased bending stresses in the specimen. Therefore, grips and pull rods should be periodically retested for axiality and reworked when necessary. High temperature alloys or SiC composites may be used to manufacture the gripping devices and pull rods.

7.1.3 The testing compartment provides a sealed space for specimen testing in an inert gas environment. The testing compartment should have a cooling system to maintain the housing at an appropriate temperature. A high temperature furnace is used in the testing compartment to heat up the specimen. Temperature-measuring apparatus should be used to measure the temperature and a vacuum pump should be used to remove air from the testing compartment before filling with inert gas.

NOTE 5—The user may use other testing compartment arrangements to achieve the same test conditions.

7.1.4 The testing compartment is filled with an inert gas such as argon (see 7.4.1). If the testing compartment is not completely sealed, a small positive inert gas pressure should be maintained to ensure that no thermal oxidation of the test material can occur.

7.2 *Heating Apparatus:*

7.2.1 The apparatus for heating of the specimens should provide the temperature control necessary to ensure that the temperature of the specimen is within ±5 °C of the testing temperature.

7.2.2 An electric furnace with temperature control is used to heat up the testing compartment.

7.3 *Temperature-measuring Apparatus:*

7.3.1 The method of temperature measurement must be sufficiently sensitive and reliable to ensure that the temperature of the specimen is within the ±5 °C limits.

7.3.2 Temperature should be measured with thermocouples in conjunction with the appropriate temperature displaying instrumentation.

7.3.3 Thermocouples should be kept clean prior to exposure and during use at elevated temperatures.

7.3.4 Temperature-measuring, controlling, and recording instruments should be re-calibrated periodically.

7.4 *Purity of Inert Gas:*

7.4.1 A high purity 99.9 % inert gas, such as argon, is used to fill the testing compartment after air evacuation.

7.5 *Cooling System:*

7.5.1 A cooling system is used to maintain the compartment housing at an appropriate temperature during testing.

7.6 *Fixture:*

7.6.1 All load-bearing machine and fixture surfaces inside the testing compartment shall have a minimum hardness of 45 HRC and surface finish of 0.4 µm rms maximum at room temperature. Surfaces in contact with the specimen shall be flat to less than 0.005 mm ⁄mm.

7.6.2 All the contact block surfaces inside the testing compartment should be plane and parallel to within 0.005 mm ⁄mm at room temperature.

7.6.3 The fixture inside the testing compartment should be able to perform the elevated temperature test according to the corresponding room temperature standard (2.1 refers).

7.6.4 A safety shield made with high temperature alloy should be used to protect the internal structure from damage caused by fragmentation of the test specimen.

8. Procedure

8.1 A representative testing facility is shown in Fig. 1. A schematic of the test compartment internals is shown in Fig. 2.

8.2 Transfer the test specimen from the glove box to the testing machine in the hermetic container.

8.3 Place the test specimen in the testing compartment, taking care not to introduce non-axial forces while installing the specimen. The user should be wearing protective gloves, clothes, and glasses to avoid direct contact with the specimen.

8.4 Securely seal the testing compartment.

8.5 Evacuate air from the testing compartment using the vacuum pump before back-filling the testing compartment with the inert gas.

8.6 Heat up the testing compartment to the testing temperature.

8.7 *Temperature Control:*

8.7.1 For the duration of the test, do not allow the difference between the measured temperature and the nominal testing temperature to exceed the ±5 °C limit.

8.7.2 If necessary, the hold/soak time at temperature prior to the start of the test should be governed by the time necessary to ensure that the specimen has reached equilibrium and that the temperature can be maintained within the limit.

8.8 Apply the load on the test specimen while intact. The load and displacement from the specimen should be recorded.

8.9 Measure strength according to the corresponding test standard.

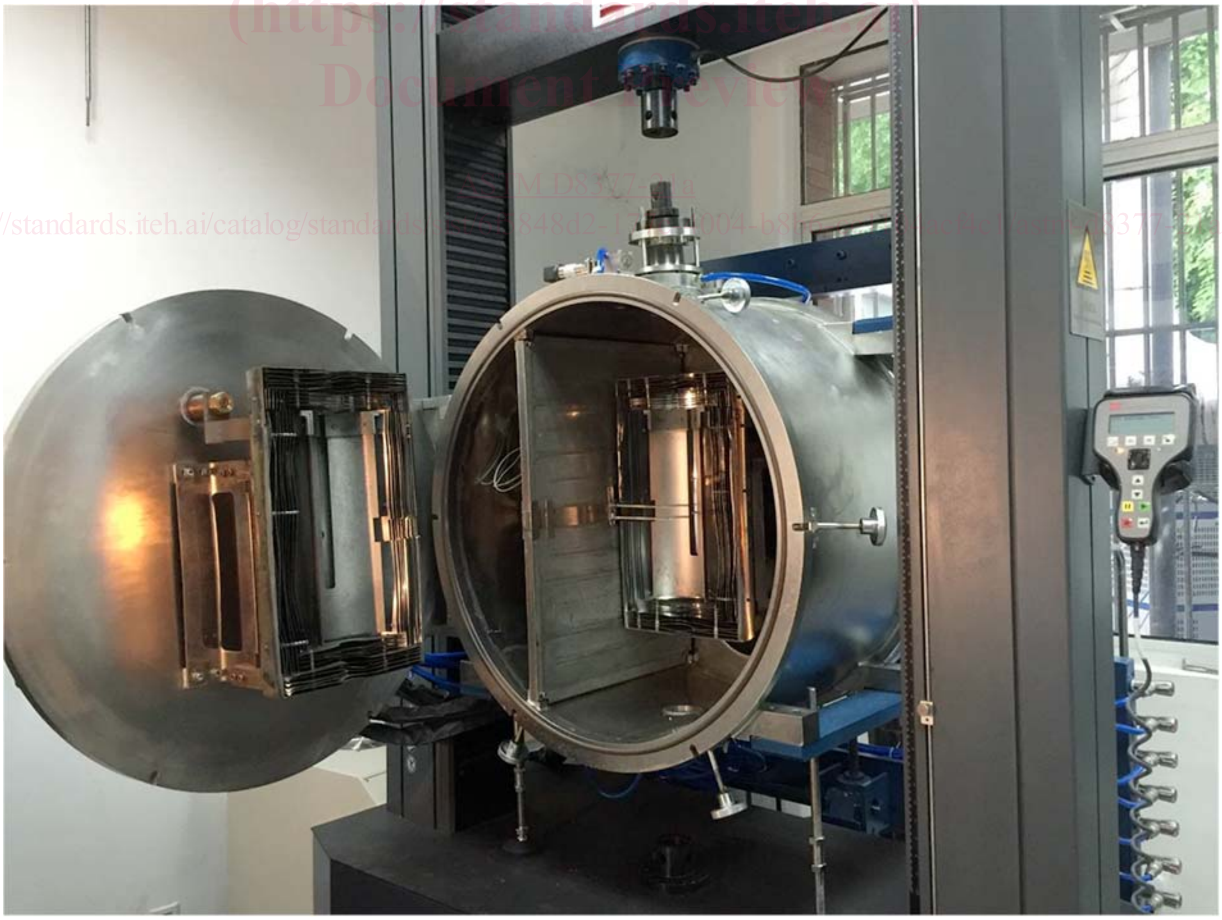

FIG. 1 Overall View of a Representative Testing Compartment and Loading Device