Designation: C 1099 – 07

Standard Test Method for Modulus of Rupture of Carbon-Containing Refractory Materials at Elevated Temperatures[1]

1. Scope

1.1 This test method covers the determination of the modulus of rupture of carbon-containing refractories at elevated temperatures in air.

1.2 The values stated in inch-pound units and degrees Fahrenheit are to be regarded as standard. The values given in parentheses are for information only.

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 and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see Section 5.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

C 583 Test Method for Modulus of Rupture of Refractory Materials at Elevated Temperatures

E 220 Test Method for Calibration of Thermocouples By Comparison Techniques

2.2 *ISO Standard:*

ISO Recommendation 5013 Determination of the Hot Modulus of Rupture of Shaped and Unshaped Dense and Insulating Refractory Products[3]

3. Significance and Use

3.1 The modulus of rupture of carbon-containing refractories at elevated temperatures has become accepted as a useful measurement in quality control testing and in research and development. These measurements are also used to determine the suitability of particular products for various applications and to develop specifications. The sample may undergo some oxidation during the test.

3.2 In 1988, ruggedness testing was conducted on this test procedure. The following variables were studied:

3.2.1 Testing temperature (2525 (1385) versus 2575°F (1413°C)),

3.2.2 Air atmosphere versus argon atmosphere in the furnace,

3.2.3 Hold time prior to breaking the sample (12 versus 18 min), and

3.2.4 Loading rate on the sample (175 (778) versus 350 lb/min (1556 N/min)).

3.3 Resin bonded magnesia-carbon brick containing approximately 17 % carbon after coking where tested in two separate ruggedness tests. Metal-free brick were tested in the first ruggedness test, while aluminum-containing brick were tested in the second. Results were analyzed at a 95 % confidence level.

3.4 For the metal-free brick, the presence of an argon atmosphere and hold time had statistically significant effects on the modulus of rupture at 2550°F (1400°C). The argon atmosphere yielded a lower modulus of rupture. The samples tested in air had a well-sintered decarburized zone on the exterior surfaces, possibly explaining the higher moduli of rupture. The longer hold time caused a lower result for the metal-free brick.

3.5 For the aluminum-containing brick, testing temperature, the presence of an argon atmosphere, and loading rate had statistically significant effects on the modulus of rupture at 2550°F (1400°C). The higher testing temperature increased the measured result, the presence of an argon atmosphere lowered the result, and the higher loading rate increased the result.

4. Apparatus

4.1 *Electrically-Heated Furnace*—An electrically heated furnace should be used. The furnace will contain an air atmosphere.

4.2 *Lower Bearing Edges*, at least one pair, made from volume-stable refractory material (Note 1) shall be installed in the furnace on 5-in. (127-mm) centers.

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[1] This test method is under the jurisdiction of ASTM Committee C08 on Refractories and is the direct responsibility of Subcommittee C08.01 on Strength.

Current edition approved March 1, 2007. Published April 2007. Originally approved in 1992. Previous edition approved in 2002 as C 1099 – 92 (2002).

[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.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

4.3 *Thrust Column*, containing the top bearing edge that is made from the same volume-stable refractory material used for the lower bearing edges, shall extend outside the furnace where means are provided for applying a load.

4.3.1 The lower bearing edges and the bearing end of the support column shall have rounded bearing surfaces having about a ¼-in. (6 mm) radius (Note 2). The lower bearing surfaces may be made adjustable, but must attain the standard span of 5 ± 3⁄32 in. (127± 2 mm). The length of the lower bearing surfaces shall exceed the specimen width by about ¼ in. The load shall be applied to the upper bearing edge by any suitable means. Instrumentation for measuring the load shall be accurate to 1 %.

4.3.2 The thrust column shall be maintained in vertical alignment and all bearing surfaces shall be parallel in both horizontal directions.

NOTE 1—A minimum of 90 % alumina content is recommended as a suitable refractory.

NOTE 2—All bearing surfaces should be checked periodically to maintain a round surface.

4.4 It is recommended that the furnace temperature be controlled with calibrated platinum-rhodium/platinum thermocouples connected to a program-controller recorder (see Method E 220). A thermocouple protection tube is advisable. Temperature differential within the furnace shall not be more than ±20°F (11°C), but the controlling thermocouple shall be placed within ½ in. (13 mm) of the geometric center of a side face of the test specimen when positioned on the bearing edges.

5. Hazards

5.1 Standard safety precautions that are used in high temperature testing should be followed for this test method. This would include use of protective clothing and eyeglasses when handling hot samples. In addition, these tests should be run in an area that has adequate ventilation since there is potential for oxidation of carbon to form carbon monoxide. There may also be organic volatiles present from pyrolysis of pitch and resin.

6. Sampling

6.1 The sample shall consist of five specimens, each taken from five brick or shapes.

7. Test Specimens

7.1 The standard test specimen shall be 1 ± 1⁄32 by 1 ± 1⁄32 by approximately 6 in. (25 ± 0.8 by 25 ± 0.8 by approximately 152 mm). Specimens cut from brick shall have at least one original brick surface perpendicular to the pressed direction. This original brick surface will be the surface in tension during testing. If cut from shapes, the specimens shall be taken parallel to the longest dimension. For irregular shapes, all four long surfaces of the specimen may be cut faces. Note this in the report.

7.2 The test specimens shall be prepared from brick as they are to be used. They shall not be coked prior to testing.

7.3 Opposite faces of the specimen shall be parallel, and adjacent faces shall be perpendicular.

7.4 Measure the width and depth of the test specimen at midspan to the nearest 0.01 in. (0.3 mm).

8. Procedure

8.1 Preheat the furnace to the test temperature and allow it to soak until thermal equilibrium is established.

8.2 Specify the test temperature as 2550 ± 10°F (1400 ± 6°C). Note any deviation from 2550°F in the report.

8.3 Once thermal equilibrium is established, open the furnace door, place one specimen on the lower bearing edges keeping the original brick surface as the tension surface, and close the door as quickly as possible.

8.4 Hold the sample for 15 min ± 30 s. Bring the top bearing edge to bear at mid-span on the specimen, ensure proper alignment of the bearing surfaces, and apply pressure through the loading mechanism until failure of the specimen occurs. The rate of application of the load on the sample shall be 175 ± 17.5 lbf (778.8 N)/min. The resulting rate of increase in bending stress for the standard 1 by 1 by 6 in. (25 by 25 by 152 mm) specimen is 1312.5 ± 131 psi (9.05 ± 0.9 MPa)/min.[4]

8.5 Since opening the furnace door as the specimen is inserted will lower the temperature of the furnace, note the amount of temperature loss, as well as the time it takes for the furnace to reestablish its equilibrium temperature.

8.6 Once the sample has been broken, open the furnace door, remove the broken sample from the lower bearing edges, and place another sample on the lower bearing edges for testing in an identical manner.

9. Calculation

9.1 Calculate the modulus of rupture (MOR) for each rectangular specimen as follows:

$$MOR = 3PL/2bd^2$$

where:
MOR = modulus of rupture, psi or MPa,
P = concentrated load at rupture, lbf or N,
L = span between supports, in. or mm,
b = breadth or width of specimen, in. or mm, and
d = depth of specimen, in. or mm.

10. Report

10.1 Report the following information:

10.1.1 The test temperature,

10.1.2 The five individual test results,

10.1.3 The average modulus of rupture and standard deviation in pounds-force per square inch (or megapascals) for the five specimens, and

10.1.4 List of deviations.

11. Precision and Bias [5]

11.1 *Precision—Interlaboratory Study:* An interlaboratory test program between four laboratories was completed in 1989. Each laboratory received five brick measuring 9 by 4.5 by 3 in. for each of four different materials. The four materials were:

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[4] This rate is 0.151 MPa/s, which is in agreement with the stress rate in ISO Recommendation 5013.

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting RR: CO8-1010.

tar-bonded magnesia brick containing about 5 % residual carbon with no metallic additives; resin-bonded magnesite-carbon brick containing about 20 % residual carbon and no metallic additives; resin-bonded magnesite-carbon brick containing about 20 % residual carbon and an addition of powered aluminum; and resin-bonded magnesite-carbon brick containing about 10 % residual carbon and no metallic additives.

11.2 *Repeatability*—The maximum permissible difference due to test error between two test results obtained by one operator on the same material is given by the repeatability interval and the relative repeatability interval (coefficient of variation). The 95 % repeatability intervals are given in Table 1. Two test results that do not differ by more than the repeatability interval will be considered to be from the same population, and, conversely, two test results that do differ by more than the repeatability interval will be considered to be from different populations.

TABLE 1 Relative Precision

Material Number	Average $\bar{X}$, psi	Standard Within, S_r, psi	Deviation Between, S_R, psi	Repeatability Interval r, psi	Reproducibility Interval R, psi	Coefficient of Variation		Relative Repeatability, r,%	Relative Reproducibility, R, %
						Within Lab, V_r,%	Between Labs, V_R, %		
1	343	60.4	63.8	169	179	17.6	18.6	49.3	52.2
2	522	33.8	46.3	94.6	130	6.48	8.87	18.1	24.9
3	1400	122	109	341	341	8.71	7.79	24.4	24.4
4	390	81.9	81.9	229	229	21.0	21.0	58.7	58.7

11.3 *Reproducibility*—The maximum permissible difference due to test error between two test results obtained by two operators in different laboratories on the same type of material using the same type of test equipment is given by the reproducibility interval and relative reproducibility interval (coefficient of variation). The 95 % reproducibility intervals are given in Table 1. Two test results that do not differ by more than the reproducibility interval will be considered to be from the same population and, conversely, two test results that do differ by more than the reproducibility interval will be considered to be from different populations.

11.4 *Bias*—This test method does not lend itself to a statement of bias.

12. Keywords

12.1 carbon-containing; modulus of rupture; refractories; strength