Standard Test Method for
Quantitatively Measuring the Effect of Thermal Shock and Thermal Cycling on Refractories[1]

This standard is issued under the fixed designation C 1171; 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 is used for determining the strength loss or reduction in continuity, or both, of prism-shaped specimens which are cut from refractory brick or shapes and subjected to thermal cycling.

1.2 The strength loss is measured by the difference in modulus of rupture (MOR) between uncycled specimens and the specimens subjected to thermal cycling.

1.3 The reduction in structural continuity is estimated by the difference in sonic velocity before and after thermal cycling.

1.4 The values stated in inch-pound units are to be regarded as the standard. The values stated in parentheses are for information only.

1.5 *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:*
C 133 Test Methods for Cold Crushing Strength and Modulus of Rupture of Refractories[2]
C 607 Practice for Coking Large Shapes of Carbon-Bearing Materials[2]
C 769 Test Method for Sonic Velocity in Manufactured Carbon and Graphite Materials for Use in Obtaining an Approximate Young's Modulus[2]
E 4 Practices for Force Verification of Testing Machines[3]
E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[4]

3. Significance and Use

3.1 This test method indicates the ability of a refractory product to withstand the stress generated by sudden changes in temperature.

3.2 Because the recommended furnace temperature of this cycling test is 1200°C (2190°F), this test method may not indicate the ability of a refractory product to withstand cycling at higher or lower temperatures, especially if the existing morphology of the refractory product changes.

3.3 This test method is useful for research and development, as well as for comparing refractory products. The precision should be considered when using this test for specification purposes.

3.4 Ruggedness tests found the following variables to be rugged:

temperature	+5°C
hot spacing	½ to ¾in. (12.77 to 19 mm)
cold spacing	½ to ¾ in. (12.77 to 19 mm)
center vs. end gripping of the bars	
hot hold time	10 to 15 min
cold hold time	10 to 15 min
operator air speed	0 to 2 mi/h (0 to 3.2 km/h)
initially cold or heated samples	
last in, first out (LIFO); or first in, first out (FIFO) removal from the furnace	
sawed or original surface as tensile face during MOR testing	
bar thickness	0.96 to 1.04 in. (24.5 to 26.4 mm)

4. Apparatus

4.1 *Furnace*, capable of maintaining 1200°C (2190°F) with recovery rate of less than 5 min to temperature.

4.2 *Abrasive Saw*, to cut the test specimens.

4.3 *Dryer*, capable of operating at 105°C to 110°C (220°F to 230°F).

4.4 *Tongs or Fork*, for handling hot specimens.

4.5 *Safety Equipment*, such as gloves, face shields, and tinted safety glasses.

4.6 *Alumina Setter Brick*, 90 %, placed 5 in. (127 mm) apart in and outside the furnace.

4.7 *Strength Testing Machine*—Any form of standard mechanical or hydraulic compression testing machine that conforms to the requirements of Practices E 4 may be used.

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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 C08.02 on Thermal Properties.
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[2] *Annual Book of ASTM Standards*, Vol 15.01.
[3] *Annual Book of ASTM Standards*, Vol 03.01.
[4] *Annual Book of ASTM Standards*, Vol 14.02.

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4.8 *Sonic Velocity Machine*—Test apparatus[5] conforming to Section 6 of Test Method C 769.

5. Sampling

5.1 The sampling shall consist of at least two bricks or shapes, or test samples made from monolithic refractories. At least ten test specimens shall be used. An equal number of specimens shall be taken from each of the bricks or shapes.

5.2 Samples should be prefired to a temperature at least as high as the test temperatures.

6. Test Specimens

6.1 Test specimens shall be $1 \pm \frac{1}{32}$ in. by $1 \pm \frac{1}{32}$ in. by approximately 6 in. (25 ± 0.8 by 25 ± 0.8 by approximately 152 mm). Note in the report if other specimen sizes are used. Specimens cut from brick shall have at least one original brick surface. If cut shapes, the specimens shall be taken parallel to the longest dimension. For irregular shapes, all four long surfaces of the specimens may be cut faces. Note this in the report.

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

6.3 Measure the width and depth of the test specimen at mid-span to the nearest 0.01 in. (0.3 mm).

6.4 Specimens should be visually crack- and flaw-free.

6.5 Dry specimens to constant weight at 105 to 110°C (220 to 230°F).

6.6 Carbon-containing samples must be coked according to Practice C 607 and must be wrapped in foil[6] during the cycling procedure. See Fig. 1 for the wrapping technique.

7. Procedure

7.1 Measure the sonic velocity along the length of each test specimen according to Test Method C 769 and divide the specimens into two equal groups on the basis of similar distributions of velocity measurements.

7.2 Determine the cold modulus of rupture (using Test Methods C 133) on one group, using three point loading with a 5-in. (127-mm) span and a loading rate of 175 lbf/min (778 N/mm).

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[5] A commercially available instrument, such as a James V-Meter, Pundit, or equivalent, is an acceptable test apparatus.

[6] Rao-Foil heat resistant foil (item number RA AMS 5592, 24-in. (610-mm) wide, 0.002-in. (0.05-mm) thick), available from Rolled Alloys, 125 West Sterns Rd., Temperance, MI, has been found satisfactory for this purpose.
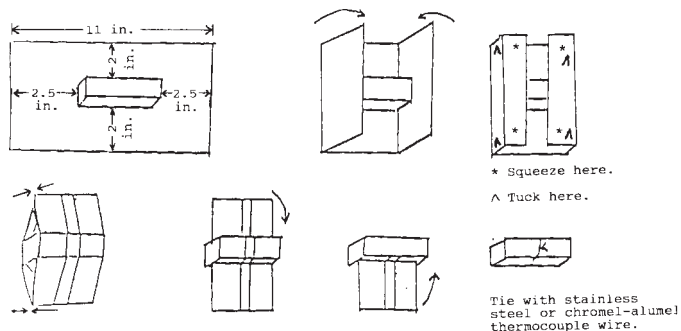

FIG. 1 Foil Wrapping for Prism Shock Test (Not to Scale)

7.3 Preheat the test furnace to the test temperature of 1200 $\pm$ 15°C (2190 $\pm$ 25°F); preheating is usually done the night prior to testing. Use of other test temperatures is allowed and must be included as a deviation in the report.

7.4 Place the test specimens from the remaining group into the furnace spanning the setter brick and allow them to remain there for 10 to 15 min. Then, remove the specimens from the furnace and allow them to cool for 10 to 15 min while spanning the setter brick in ambient air. This is considered one full cycle. Keep the specimens ½ to ¾ in. (12.77 to 19 mm) apart during each 10 to 15 min interval. Repeat for a total of five full cycles. Cycle time in the furnace starts after recovery.

7.5 Measure the sonic velocity (using Test Method C 769) along the length of each cycled test specimen.

7.6 Determine the cold modulus of rupture (using Test Methods C 133) of each cycled test specimen from the second group, using three point loading with a 5-in. (127-mm) span and a loading rate of 175 lbf/min (778 N/mm) or 0.05 in./min (1.27 mm/min).

8. Calculation

8.1 Calculate the percent sonic velocity loss of each specimen as follows:

$$\frac{V_0 - V_F}{V_0} \times 100$$

where:
V_O = original sonic velocity of each specimen, ft/s (m/s), and
V_F = sonic velocity of each specimen after testing, ft/s (m/s).

8.2 Calculate the percent modulus of rupture strength loss of each specimen as follows:

$$\frac{M_0 - M_f}{M_0} \times 100$$

where:
M_O = average modulus of rupture strength of the unshocked specimens from the first group after testing, psi (MPa), and
M_f = modulus of rupture strength of each specimen for the second group after testing, psi (MPa).

9. Report

9.1 Report the individual sonic velocity, modulus of rupture, percent sonic velocity loss, and percent modulus of rupture strength loss values, as well as the average percent sonic velocity loss, the average percent modulus of rupture loss, and the furnace temperature. Report the number of test specimens included in any modulus of rupture calculation. The report shall also list any deviations from standard test requirements.

10. Precision and Bias

10.1 *Interlaboratory Test Data*—An interlaboratory round-robin test was conducted in 1988 among six laboratories on three different types of refractory products. Each laboratory tested two specimens from each of five samples of each of three different types. Each laboratory determined sonic velocity and modulus of rupture strength after cycling to 1200°C.

TABLE 1   Relative Precision

		Modulus of Rupture		
Brick Type	MOR Lost, %	Coefficient Within Lab, %	Variation Between Labs, %	Repeatability Interval, Percent of Average	Reproducibility Interval, Percent of Average
70 % Alumina:					
Specimen 1	20.68	59.43	65.33	166.40	182.92
Specimen 2	19.44	53.09	66.51	148.64	186.23
60 % MgO DB:					
Specimen 1	59.48	25.74	29.36	72.07	82.21
Specimen 2	55.52	19.92	22.18	55.78	62.10
MgO—C:					
Specimen 1	23.58	46.10	96.82	129.08	271.09
Specimen 2	31.49	74.31	83.01	208.07	232.42

		Sonic Velocity		
Brick Type	Sonic Velocity Lost, %	Coefficient Within Lab,%	Variation Between Labs, %	Repeatability Interval, Percent of Average	Reproducibility Interval, Percent of Average
70 % Alumina:					
Specimen 1	14.03	11.62	21.74	32.53	60.87
Specimen 2	13.79	17.37	23.37	48.63	66.96
60 % MgO DB:					
Specimen 1	42.41	14.15	19.14	39.61	53.58
Specimen 2	44.19	7.98	13.85	32.35	38.78
MgO—C:					
Specimen 1	22.64	6.08	78.90	17.03	220.92
Specimen 2	23.75	38.69	94.76	108.33	265.33

The components of variance from this study expressed as standard deviation and relative standard deviation are given in Table 1. Refer to Practice E 691 for calculation of components of variance.

10.2 *Precision*—The results of the interlaboratory study are shown in Table 1. The precision was found to vary based on the type of material tested. A test result should be considered significantly different at a confidence level of 95 % if the repeatability or reproducibility, or both, exceeds the precision data listed in Table 1.

10.3 *Bias*—No justifiable statement of bias is possible since true variables cannot be established by an acceptablereference method.

11. Keywords

11.1 modulus of rupture; refractories; sonic velocity; thermal cycling