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Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of elastic modulus of ceramics at high temperature by thin wall C-ring method

Céramiques techniques (céramiques avancées, céramiques techniques avancées) – Détermination du module élastique des céramiques à haute température par la méthode de l'anneau en C à parois minces

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Foreword

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This document was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

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Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of elastic modulus of ceramics at high temperature by thin wall C-ring method

1 Scope

This document specifies the determination of elastic modulus of ceramics at high temperatures up to 2 100 °C by using the thin wall relative C-ring method. Procedures for test piece preparation, test modes, heat rate, load rates, data collection and reporting are given.

This document applies primarily to ceramic materials including monolithic fine ceramics, refractory materials, whisker and particulate-reinforced ceramic composites. This method is not applicable to super plastic ceramics or ceramics with high creep rate. This test method can be used for material research, quality control and characterization and design data generation purposes.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3611, *Geometrical product specifications (GPS) — Dimensional measuring equipment: Micrometers for external measurements — Design and metrological characteristics*

ISO 7500-1, *Metallic materials — Calibration and verification of static uniaxial testing machines — Part 1: Tension/compression testing machines — Calibration and verification of the force-measuring system*

IEC 60584-1, *Thermocouples — Part 1: Reference tables*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
elastic modulus
ratio of stress to strain, also known as Young's modulus

3.2
C-ring test piece
test piece in the shape of a split ring, prepared by cutting an incision from a thin wall ring

Note 1 to entry: R is the outer radius, r is the inner radius, and b is the width (axial length), as shown in Figure 1.
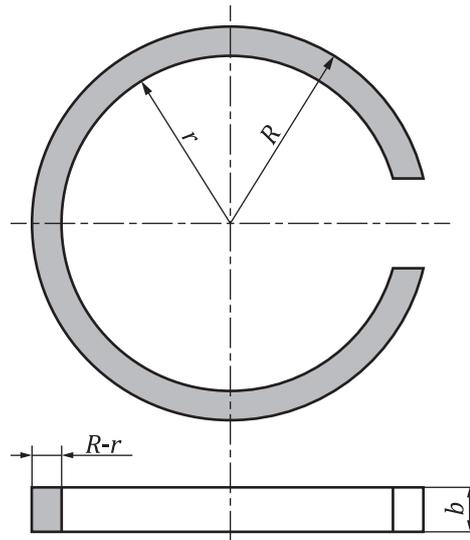
Figure 1 — Schematic diagram of C-ring test piece

3.3
rigid disk
disk which has the same radius and width as the *C-ring test piece* (3.2), but which is much stiffer

3.4
relative C-ring method
testing method for determining the deformation of the C-ring by comparing the crossbeam displacements of the C-ring and the rigid disk under same testing conditions

4   Principle

At ambient temperature, install a C-ring test piece on the fixture and keep the notch at the middle height. Place the fixture on the flat anvil of a mechanical testing machine and apply a symmetrically compressive load, *F*, on the C-ring within its range of elasticity, as shown in Figure 2 a) and b). There is a linear relationship between the load increment, ΔF, and the displacement increment, $\Delta\delta$. The compressive deformation of the C-ring can be directly measured by an accurate inductance micrometer or any other displacement meter at room temperature. The elastic modulus can be obtained from the load-deformation curve and the test piece dimensions.
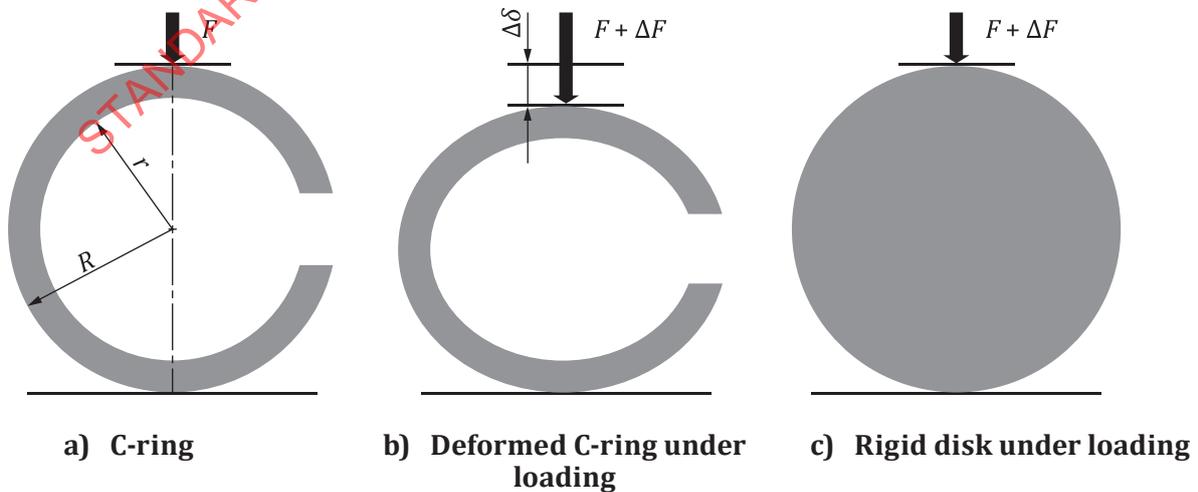

a) **C-ring**   b) **Deformed C-ring under**   c) **Rigid disk under loading**
                                loading

Figure 2 — Schematic diagram of C-ring and rigid disk, loading mode and deformation

At high temperatures, the deformation of the test piece is hardly measured by the displacement meter. The relative method is used to determine the deformation of the C-ring test piece by comparing the crossbeam displacements of the C-ring and the rigid disk piece under identical loads, as shown in Figure 2 c). In a heating furnace, as shown in Figure 3 a), the displacement of the crossbeam is composed of two parts:

a) real deformation of the C-ring test piece $\Delta\delta$;

b) errors of the loading system which are induced by the loading frame, fixture, loading device and crossbeam.

In order to obtain the real deformation of the C-ring just by the displacement of the crossbeam, a rigid disk with the same dimensions as the C-ring shall be introduced as shown in Figure 3 b). The stiffness of the rigid disk is much higher than that of the C-ring piece, so the deformation of the rigid disk under ΔF can be ignored; the measured crossbeam displacement for the rigid disk, $\Delta d'$, only represents the system errors of the test machine. Thus, the difference between the displacements of the C-ring and the rigid disk is equal to the real deformation of the C-ring test piece. The elastic modulus at high temperatures can be obtained by the real deformation and the dimensions of the C-ring test piece.
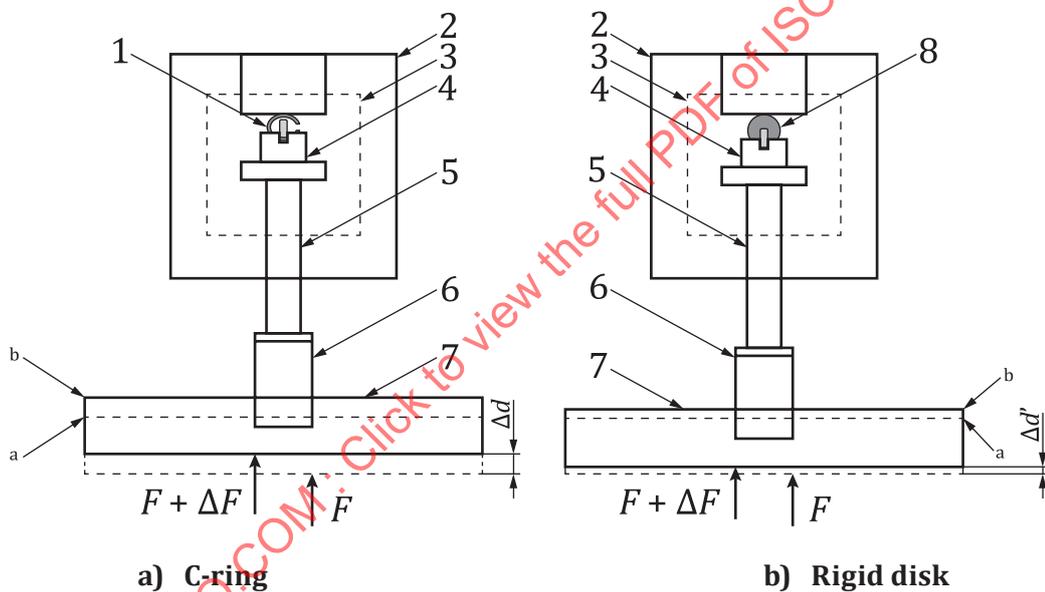
a) C-ring b) Rigid disk

Key

1	split ring	6	metallic connection
2	furnace	7	crossbeam
3	heating area	8	rigid disk
4	fixture	a	Original position.
5	supporting bar	b	Final position.

Figure 3 — Schematic diagram of C-ring (a) and rigid disk (b) before and after loading in the furnace

5 Apparatus

5.1 Testing machine

A suitable testing machine capable of applying a force to the test piece at a uniform displacement or loading rate shall be used. The testing machine shall conform with the values in ISO 7500-1. The measuring accuracy shall be 1 % or better and the calibration shall have been recently checked.

5.2 Heating system

5.2.1 General

A furnace shall be capable of heating the test fixture and test piece as well as maintaining a uniform and constant temperature during the test, by which a vacuum environment shall be available for test requirements. If a vacuum chamber is used, and it is necessary to transmit the load through a seal, bellows or a fitting, it shall be verified that load losses or errors are less than 1 % of the applied loads.

5.2.2 Test piece temperature stability

The furnace shall be controlled by a device for maintaining a constant temperature within ±2 °C or better within the working space of the furnace, during the time that the test piece is loaded in the range of elasticity.

5.2.3 Test temperature uniformity

The furnace shall be capable of maintaining the test piece at a uniform temperature. The temperature of the test piece shall not vary by more than 10 °C after a 15-min holding time at the required temperature.

5.2.4 Furnace heating rate

The furnace control device shall be capable of controlling the heating rates of the furnace and preventing temperature overshoots.

5.2.5 Furnace stability

The time for the system to reach thermal equilibrium at the test temperature shall be determined.

5.3 Temperature-measuring and indicating instruments

5.3.1 General

The thermocouple temperature-measuring equipment shall have a resolution of at least 1 °C and an accuracy of 5 °C or better. The optical pyrometer, if used, shall have a resolution of at least 5 °C and an accuracy of 10 °C or better.

5.3.2 Thermocouples

Thermocouples in accordance with IEC 60584-1 shall be used. The thermocouples shall exhibit low thermal inertia (the diameter of the wires shall be not greater than 0,5 mm). The thermocouples shall have a sufficient length within the furnace (with respect to heat conduction along the wires). The measuring thermocouple tip shall be as close as possible to the test piece.

5.3.3 Verification of the thermocouple temperature-measuring system

Thermocouples shall be checked periodically as their output may drift with usage or contamination.

5.4 Vacuuming machine

A suitable vacuuming machine capable of applying a suitable vacuum environment shall be used. The low vacuum degree (<10 Pa) shall be accomplished by a mechanical pump, while the high vacuum degree (<0,1 Pa) shall be accomplished by a molecular pump.

5.5 Data acquisition

Obtain records of the applied load versus crosshead displacement or testing time.

Use either analogue chart recorders or digital data acquisition systems. Recording devices shall be accurate to within 1 % of the selected range of the test equipment, including the readout unit, and shall have a minimum data acquisition rate of 10 Hz, with a response of 50 Hz deemed more than sufficient.

5.6 Dimension-measuring device

Micrometers and other devices with a precision of 0,01 mm according to ISO 3611 shall be used to measure the linear dimensions of the test piece. Alternative dimension-measuring instruments may be used, provided they have a resolution of 0,01 mm or finer.

6 Test pieces

6.1 Test piece size and preparation

6.1.1 General

Test piece geometry shall be $0,8 \leq r/R \leq 0,95$ for the validity of the calculation formulae for the thin-wall ring. The gap of the C-ring shall be sufficiently large so that the ring does not close during the test, but less than one half of the inner diameter, as shown in Figure 1. The width (axial length) of the C-ring shall satisfy two conditions: $0,2 \leq b/R \leq 1,0$ and $1,0 \leq b/(R - r) \leq 5,0$.

This document permits two options for the radius and surface treatment of the test piece.

a) Existing tubes: if r and R of these tubes satisfy the requirements above, cut the tube into several rings. Grind and polish the cutting section surfaces of each ring to a parallelism of 0,02 mm or better. Clean the test pieces with alcohol or pure water, then cut a slot with a width from 0,2 r to 0,5 r using a ceramics cutting machine.

b) Other-shaped materials: machine a ring with R = 15 mm, r = 13 mm and b = 8 mm. Grind and polish the cutting section surfaces of the ring to a parallelism of 0,02 mm or better. Clean the test piece with alcohol or pure water, then cut a slot with a width from 0,2 r to 0,5 r using a ceramics cutting machine.

NOTE   The aim of polishing is to minimize the damage created in the test pieces during the preparation process.

6.1.2 Test piece storage

The test pieces shall be handled with care to avoid the introduction of damage after test piece preparation. Test pieces shall be stored separately and not allowed to impact or scratch each other.

6.1.3 Number of test pieces

A minimum of three test pieces is required for the tests.

6.2 Rigid disk preparation

The load-softening temperature of the rigid disk shall be higher than that of the C-ring test piece. In the vacuum environment, graphite is the best choice for the rigid disk. The radius and width of the rigid disk shall be consistent with the outer radius and width of the C-ring test piece, respectively.

7 Test procedures

7.1 Check of vacuuming system

Ensure that there is no leakage in the vacuum system. Use a universal vacuuming machine such as a mechanical pump, molecular pump or diffusion pump to pump the furnace until the set vacuum degree is reached.

7.2 Check of heating system

Ensure that the heating system is functional. Heat the furnace with a speed of 10 °C/min until the set temperature is reached.

7.3 Testing machine and loading speed

Use a universal mechanical testing machine with a crosshead rate of 0,1 mm/min for the C-ring tests and 0,05 mm/min or less for the rigid disk tests. The test piece shall contact well with the supporting and loading anvils.

NOTE   A suitable loading rate depends on the ring size and testing materials. The test can be performed by displacement or load control so that the testing time remains within 100 s for the C-ring test piece.

7.4 Elastic modulus measurement steps

— Step 1: Measure b, R and r of each C-ring test piece at four points of the piece using a calibrated caliper. Compute the mean value of each dimension. The value of r can also be obtained by measuring the wall thickness and R.

— Step 2: Calibrate the displacement of the crossbeam of the testing machine to ensure that the display results are reliable and accurate.

— Step 3: Put each C-ring test piece and fixture in the heating furnace. The middle of the gap shall be at the half height of the ring. The test piece shall contact well with the supporting and loading anvils, as shown in Figure 3 a).

— Step 4: Vacuum the furnace and then heat the furnace up to the testing temperature. Maintain this for 15 min before the loading test.

— Step 5: Apply the test force in the range of elasticity with a rate of 0,1 mm/min. Record the loading increment, ΔF, and the crossbeam displacement increment, Δd, of the test piece. The upper limit of the load shall be lower than half of the fracture load.

— Step 6: Stop heating and cool down. After cooling to room temperature, replace the C-ring test piece with the rigid disk and repeat steps 3 and 4, as shown in Figure 3 b). Record the crossbeam displacement increment, $\Delta d'$, at the same loading increment, ΔF. The real deformation of the C-ring test piece, $\Delta \delta$, at high temperature can be calculated by using $\Delta \delta = \Delta d - \Delta d'$, as shown in Figure 4.

— Step 7: According to Formula (1) the elastic modulus of the testing material at high temperatures can be calculated by using ΔF, $\Delta \delta$ and dimensions of the C-ring test piece.

— Step 8: Turn off the testing machine and finish the test.
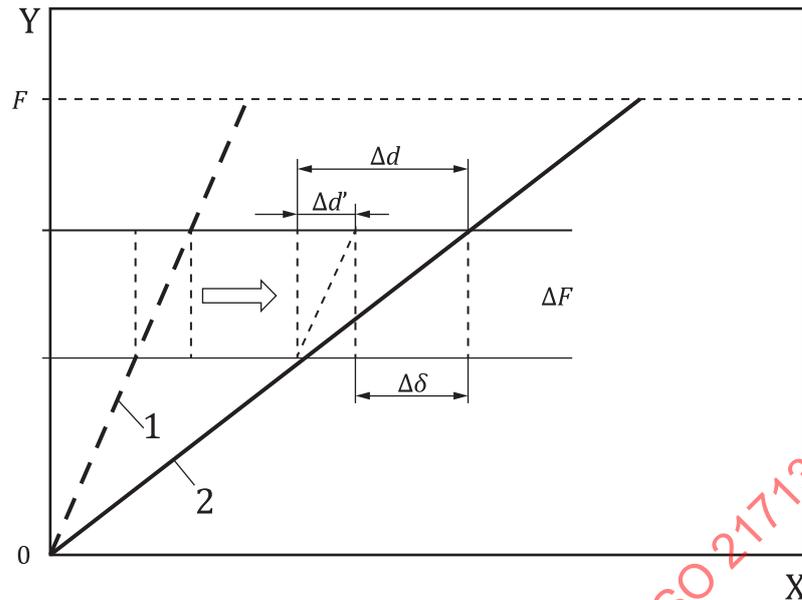
Key

1   rigid disk
2   split ring
X   displacement
Y   load

Figure 4 — Real deformation from two load-displacement curves of a crossbeam for a C-ring and a rigid disk

NOTE   A given test piece can be tested at a series of testing temperatures rising from room temperature to high temperatures to improve the testing efficiency for determining modulus at different temperatures.

7.5   Test validity requirement

The following conditions shall be met for valid results:

a)   Before the loading test, the accuracy of crossbeam displacement shall be calibrated by the micrometer at room temperature, and a relative deviation of less than 0,5 % is acceptable.

b)   The load-displacement curves of the C-ring samples and rigid disk shall be straight and smooth under ΔF with no discontinuous points on these curves. The dimensions of the test piece shall be measured after the test to check their creep deformation.

c)   The testing conditions and environments shall be identical for the C-ring and the rigid disk, except the loading rate. It is recommended that the loading-unloading tests on the test piece are performed three times and the third loading-displacement curve shall be used. This is because the first loading curve sometimes involves a light level of contact deformation.

For the rigid disk, the loading rate shall be low enough to prevent overload or damage to the force sensor.

8 Calculation of results

8.1 Calculation of the elastic modulus

Formula (1) shows the standard formula for elastic modulus:

$$E = \frac{3\pi}{4\,000b} \times \frac{\Delta F}{\Delta \delta} \times \left(\frac{R+r}{R-r} \right)^3 \tag{1}$$

and

$$\Delta \delta = \Delta d - \Delta d'$$

where

E is the elastic modulus, in GPa;

ΔF is the load increment in the scope of elastic deformation, in newtons (N);

Δd is the crossbeam displacement for the C-ring under the load ΔF, in mm;

$\Delta d'$ is the crossbeam displacement for the rigid disk under the load ΔF, in mm;

$\Delta \delta$ is the real deformation of the test piece, and equals the displacement difference between Δd and $\Delta d'$, in mm;

R is the mean outer radius of the C-ring, in mm;

r is the mean inner radius of the C-ring, in mm;

b is the mean width of test pieces, in mm.

8.2 Mean value and standard deviation for elastic modulus

The mean elastic modulus, $\bar{E}$, and the standard deviation, S_E, are given by Formula (2) and Formula (3):

$$\bar{E} = \frac{\sum\limits_{i=1}^{n} E_i}{n} \tag{2}$$

$$S_E = \left[\frac{\sum\limits_{i=1}^{n} (E_i - \bar{E})^2}{n-1} \right]^{1/2} \tag{3}$$

where

E_i is the elastic modulus of the ith test piece;

n is the total number of test pieces.

9 Test report

The test report shall contain at least the following information:

a) a reference to this document, i.e. ISO 21713:—;