INTERNATIONAL STANDARD

ISO 28704

First edition
2011-08-15

Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for cyclic bending fatigue of porous ceramics at room temperature

Céramiques techniques — Méthode d'essai de fatigue par flexion cyclique des céramiques poreuses à température ambiante

Reference number
ISO 28704:2011(E)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 28704 was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for cyclic bending fatigue of porous ceramics at room temperature

1 Scope

This International Standard spedifies a test method for determining the cyclic four-point bending fatigue of porous ceramics in air at room temperature.

2 Normative references

The following referenced documents are indispensable for the application 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 463, *Geometrical Product Specifications (GPS) — Dimensional measuring equipment — Design and metrological characteristics of mechanical dial gauges*

ISO 1101, *Geometrical Product Specifications (GPS) — Geometrical tolerancing — Tolerancing of form, orientation, location and run-out*

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

ISO 3599, *Vernier callipers reading to 0,1 and 0,05 mm*

ISO 4287, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Terms, definitions and surface texture parameters*

ISO 14704, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for flexural strength of monolithic ceramics at room temperature*

ISO 20507, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Vocabulary*

ISO 22214, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for cyclic bending fatigue of monolithic ceramics at room temperature*

ISO 80000-1, *Quantities and units — Part 1: General*

3 Terms and definitions

For the purposes of this document, the terms and definitions in ISO 20507 and the following apply.

3.1
porous fine ceramics
ceramics with a porosity of typically 30 % to 60 % and a pore diameter of 1 µm to 100 µm, for applications such as filters, catalyst carriers, humidity sensors or molecular sieves, excluding structured honeycomb cellular channels

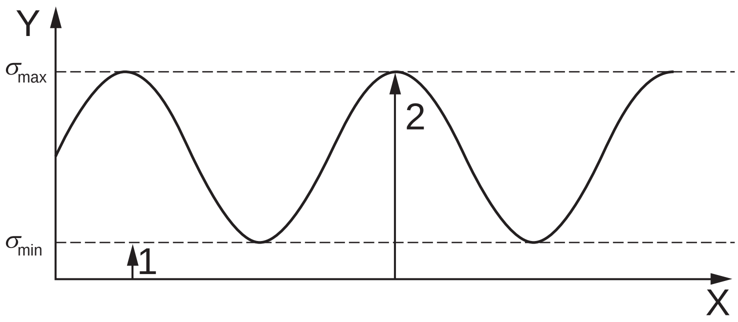
3.2
fatigue test
test wherein a test piece is subjected to repeated stressing to a set stress level, and the number of cycles to fracture is determined

3.3
four-point bending stress
maximum value of stress generated in flexure in a bar-shaped test piece when supported at two support points near its ends and loaded symmetrically by two loading points between them

3.4
cyclic stress
stress which is simply and cyclically loaded between a specific maximum value and a specific minimum value (see Figure 1)

3.5
maximum stress
σ_{max}
maximum value of cyclic stress (see Figure 1)

3.6
minimum stress
σ_{min}
minimum value of cyclic stress (see Figure 1)



Key

X time

Y stress

1 σ_{min}

2 σ_{max}

Figure 1 — Cyclic stress

3.7
minimum to maximum stress ratio
R
ratio of minimum stress to maximum stress

$$R = \frac{\sigma_{min}}{\sigma_{max}} \tag{1}$$

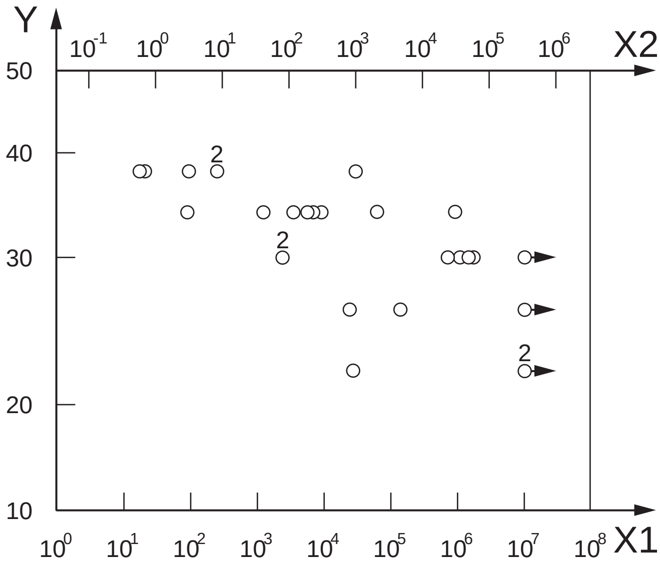
3.8
number of cycles
n
number of cycles of stress in the fatigue test

3.9
number of cycles to failure
N
number of cycles of stress until fatigue failure occurs

3.10
failure time
loading time until fatigue failure occurs

3.11
***S*-*N* plot**
diagram wherein the maximum stress, ***S*,** is taken as an ordinate and the fracture number of cycles, ***N*,** is taken as an abscissa

NOTE An example is given in Figure 2, where the number attached to the data point indicates the number of test pieces and the data with a right-pointing arrow indicates a “run-out”.



Key

X1 number of cycles to failure, N

X2 time to failure (s)

Y maximum stress (MPa)

2 number of superimposed points

Figure 2 — Example of *S*-*N* plot

4 Testing machine and equipment

4.1 Testing machine

A testing machine shall be so constructed that a cyclic bending stress can be applied to a test piece, and a force or moment other than the cyclic bending stress is not applied. The testing machine shall be equipped with apparatus for measuring or indicating the maximum and minimum load, apparatus capable of measuring the number of cycles until the test piece is broken, and a mechanism wherein when the testing machine is stopped for reasons of power failure or others, its automatic reactivation is prevented.

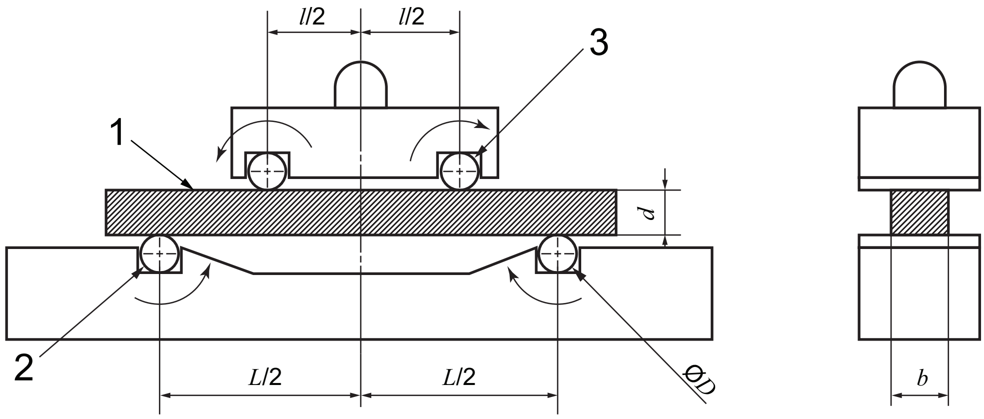
4.2 Loading precision

The fluctuation of the maximum stress shall be within ±1 % of the chosen value, and that of the minimum to maximum stress ratio shall be within ±5 % thereof, as specified in ISO 22214.

4.3 Test fixture

A four-point flexure fixture of the type and function shown in ISO 14704 shall be used (see Figure 3). The use of a semi- or fully-articulating fixture is required to prevent torsional deformation. Bearings supporting the test piece at the support points and loading the test piece at the loading points shall be of equal shape and have a length exceeding the width of the test piece. The material of the bearings shall have an elastic modulus of at least 140 GPa, and shall be free from plastic deformation; the bearing shall never break during the test. The radius of curvature at the tip of the bearings shall be 2,0 mm to 3,0 mm, and their surface roughness, Ra, as specified in ISO 4287, shall be not greater than 0,40 µm.

The test piece and/or its bearings may move during the test. Precautions should be taken where possible to minimise such movement without restricting the flexural displacement, for example, by providing support rollers with restraint against a stop in the correct position.



Key

1 test piece
2 external support
3 internal support
D roller diameter (4 to 6 mm)
L outer span (L = 60 mm ± 0,5 mm)
l inner span (l = 30 mm ± 0,5 mm)

Figure 3 — Four-point flexure fixture

4.4 Micrometer callipers for measurement of external dimensions

Micrometer callipers for the measurement of external dimensions with the same accuracy of 0,01 mm as specified in ISO 3611, or better, shall be used.

4.5 Dial gauge

Dial gauges with the same accuracy of minimum reading of 0,01 mm as specified in ISO 463, or better, shall be used.

4.6 Vernier callipers

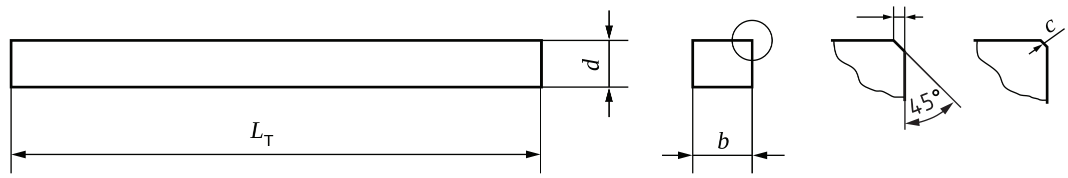
Vernier callipers with the same accuracy of minimum reading of 0,05 mm as specified in ISO 3599, or better, shall be used.

5 Test piece

5.1 Test piece size

The shape of a test piece shall be a beam of uniform rectangular cross-section. Its standard dimensions shall be 70 mm ± 0,5 mm in total length, 8,0 mm ± 0,1 mm in width, and 6,0 mm ± 0,1 mm in thickness. The parallelism between the upper and lower surfaces of the test piece shall be better than 0,02 mm, as specified in ISO 1101. The longer edges shall be rounded or chamfered by an amount of 0,1 mm to 0,3 mm, as given in Figure 4. This chamfering shall be carried out with caution so as not to introduce defects resulting in a fracture initiation site. The chamfering is not necessary when the grain size of material is larger than 0,1 mm. Before carrying out the test, the width and thickness of the test piece shall be measured to the nearest 0,1 mm by using micrometer callipers (4.4).

If, for any reason, different test piece dimensions have to be used, they shall be stated in the test report.



Key

L_T length of test piece (L_T = 70 mm ± 0,5 mm)

b width of test piece (b = 8,0 mm ± 0,1 mm)

d thickness of test piece (d = 6,0 mm ± 0,1 mm)

c chamfer or rounding (c = 0,1 mm to 0,3 mm)

Figure 4 — Test piece dimensions and chamfering of ridge line

5.2 Test piece preparation

The surface shall be finished by grinding with a wheel of grain size 20 µm or smaller. The contamination by wax, if any, should be removed by organic solution or by heating at 773 K (500 °C) for 1 h to evaporate wax.

6 Test method

6.1 Waveform of loading stress

The waveform shall be sinusoidal and the ratio of minimum to maximum stress R = 0,1. The frequency shall be selected by agreement between parties, with 20 Hz being recommended. For other cases, the waveform of the loading stress shall be stated in the test report. A series of tests shall be carried out using the same stress waveform.

6.2 Loading method

6.2.1 The distances between internal support points and external support points shall be measured by a calliper before the tests.

6.2.2 The test piece shall be centrally positioned in the test fixture.

6.2.3 The number of cycles from the start of loading to the end of adjusting to a specific cyclic loading stress shall be as few as possible. In this case, the stress during initial adjusting shall not exceed the specific maximum stress.

6.2.4 The test shall, as a rule, be carried out without a pause on the same test piece from start to end. However, when the test is interrupted for any reason, the number of cycles to the interruption and the duration of interruption shall be recorded.

6.2.5 The fatigue test shall be carried out on at least three test pieces at each of at least three maximum stress levels.

6.3 Number of cycles for interruption

Unless otherwise specified, if the test piece is not fractured by 10^7 loading cycles, the test may be halted, and the result deemed to be a “run-out” and indicated on the plot of results with a right-pointing arrow.

6.4 Reuse of test piece

Do not reuse a test piece which has been used once.

6.5 Recommended test procedure in fatigue test

When the maximum stress and the number of test pieces in a fatigue test are not previously agreed upon between the purchaser and the supplier, the test may be carried out according to the following procedure.

6.5.1 Measurement of flexural strength

In order to obtain the standard value of the maximum stress in a fatigue test, measure the flexural strength of a test piece prior to the fatigue test. Use the test piece of the same shape as that of a fatigue test piece for measurement of flexural strength, and obtain it by the same loading method as that of the fatigue test. Allow the speed of a cross-head in the measurement of flexural strength to be 0,5 mm/min. Calculate the flexural strength from the measured maximum forces of respective test pieces.

6.5.2 Stress level in fatigue test

Determine the maximum stress in a fatigue test by referring to the arithmetic mean value of the flexural strength obtained by the measurement in 6.5.1. Start the fatigue test from a condition wherein the maximum stress is high, and preferably measure the life of at least three test pieces by the same stress. When all the test pieces tested at the same stress are unfailed at the selected number of cycles for test termination, select a higher stress level for the next group of test pieces. Do not carry out tests at a lower stress level. It is preferred that the results of a test programme should contain at least three valid failures at each of at least three maximum stress levels.

6.6 Test environment

The moisture content in the test environment may have an influence on the cyclic fatigue behaviour. For tests in air or other gaseous environments, the test temperature and humidity should be measured and reported at least at the beginning and the end of each test, or hourly if the test duration is longer than 1 h.

7 Treatment of test result

7.1 Maximum stress

Calculate the maximum stress according to the following equation from the measuring values of respective test pieces, and round off to three significant figures in accordance with ISO 80000-1.

$$\sigma_{\mathrm{max}} = \frac{3P_{\mathrm{max}}(L-l)}{2bd^2} \qquad (2)$$

where

σ_{max} is the maximum stress, in magapascals (MPa);

P_{max} is the maximum force, in newtons (N);

l is the distance between internal support points, in millimetres (mm);

L is the distance between external support points, in millimetres (mm);

b is the width of the test piece, in millimetres (mm);

d is the thickness of the test piece, in millimetres (mm).

7.2 Number of cycles to failure

The number of cycles shall be counted by starting from when a load on a test piece reaches a specific testing stress. If the stress is adjusted during the test, the cycles for adjustment shall be included in N. Represent the number of cycles of the test result by, for example, multiple of 10^n such as $2{,}34 \times 10^6$, and round off to three significant figures.

7.3 S-N plot

Draw a S-N diagram by taking the maximum stress or its logarithmic value as the ordinate, and the logarithmic value of the number of cycles to failure as the abscissa. When the life is displayed in time, the number of cycles to failure is scaled on the underside of the abscissa, and the failure time is scaled on the upper side of the abscissa. A point expressing the test result for the test piece that is not broken is marked with a rightward arrow. An example of the S-N diagram is given in Figure 2.

NOTE 1 In the S-N diagram, when at least two points are superimposed, the number of points is clearly described, or arrows are marked by the number of points as shown in Figure 2.

NOTE 2 When the arrow is superimposed on a marked line, the arrow is marked clearly by drawing it upward or downward.

8 Test report

The test report shall include the following information:

a) name and class of material;

b) dimensions (mean values) of the test piece;

c) name of testing machine and its type;

d) test environment, including temperature and humidity;

e) loading conditions (loading method, loading waveform, loading frequency, minimum to maximum stress ratio, and the number of cycles to the moment when a test is temporarily stopped midway and its stop time);

f) list of test results (the maximum stress and the fracture number of cycles);

g) S-N plot;

h) if appropriate, manufacturer's name of the material and its date of manufacture;

i) if appropriate, name of the material, kinds of additive, and sintering method;

j) if appropriate, porosity and mean pore size of the material;

k) if appropriate, chemical composition of material;

l) if appropriate, sampling conditions of test piece made from the material and its machining conditions (when a test piece is heat treated, its conditions are included);

m) if appropriate, mechanical properties of the material, such as flexural strength, elastic modulus, fracture toughness value, etc.;

n) if appropriate, date of test place, and name of the person conducting the test.

ICS 81.060.30

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