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

Céramiques techniques — Méthode d'essai pour la fatigue de courbure cyclique de céramiques monolithiques à température ambiante

Reference number
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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.

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ISO 22214 was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

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

1 Scope

This International Standard specifies a test method for four-point cyclic bending fatigue of fine ceramics that are carried out in the air at room temperature. This test may be used as a pass/fail test for material qualification purposes, or for determination of the overall fatigue behaviour at several different stress levels.

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 31-0:1992, *Quantities and units — Part 0: General principles*

ISO 14704:2000, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for flexural strength of monolithic ceramics at room temperature*

ISO/IEC 17025:2005, *General requirements for the competence of testing and calibration laboratories*

3 Terms and definitions

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

3.1
fatigue test
test where repeated stress is applied to a test specimen, and the number of cycles to fracture is measured

3.2
fatigue failure
fracture due to cyclic loading well below the strength under monotonic loading

3.3
suspending time
time at which test is intentionally interrupted without fracture

3.4
four-point bending stress
maximum value of stress generated in a test specimen when a test specimen is loaded equally by two bearings symmetrically located between two support bearings

3.5
cyclic stress
stress which is simply and cyclically varied between a specific maximum value and a specific minimum value

See Figure 1.

3.6
maximum stress
σ_{max}
algebraic maximum value of cyclic stress

See Figure 1.

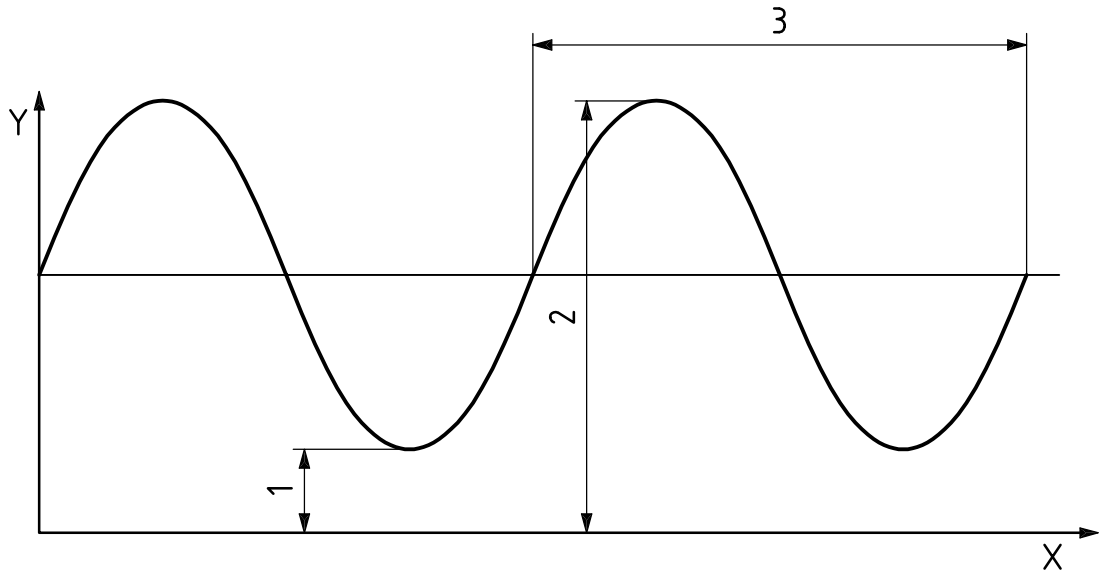
3.7
minimum stress
σ_{min}
algebraic minimum value of cyclic stress

See Figure 1.

NOTE For maximum stress or minimum stress, the sign is considered. A tensile stress is taken as positive, and a compressive stress is taken as negative.

3.8
stress cycle
one period of the cyclic stress

See Figure 1.



Key
X Time
Y Stress
1 σ_{min}
2 σ_{max}
3 One stress cycle

Figure 1 — Cyclic stress

3.9
minimum to maximum stress ratio
R
algebraic ratio of minimum stress to maximum stress

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

3.10
number of cycles
number of cycles of stress in fatigue test

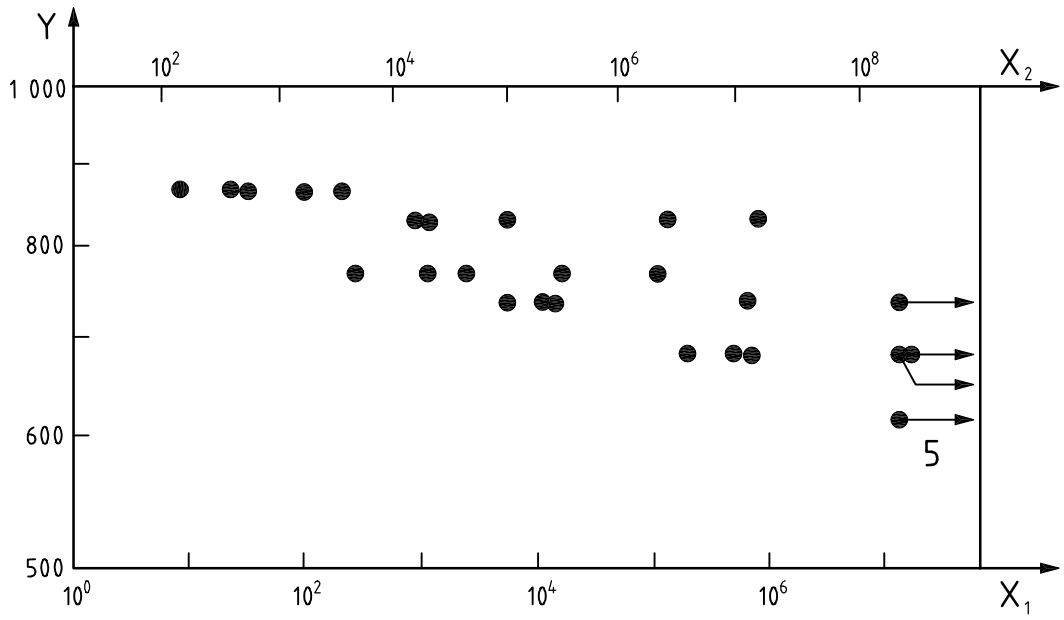
3.11
number of cycles to failure
N
number of cycles of stress until fatigue failure occurs

3.12
time to failure
T
loading time until fatigue failure occurs

3.13
S-N **plot**
diagram where maximum stress is taken as the ordinate and the number of cycles to failure as the abscissa

See Figure 2.

NOTE When the test is interrupted, the result is plotted as the maximum stress and the number of cycles at the suspending time.



Key

X_1 Number of cycles to failure (N)
X_2 Time to failure (s)
Y Maximum stress (MPa)
5 Number of superimposed points

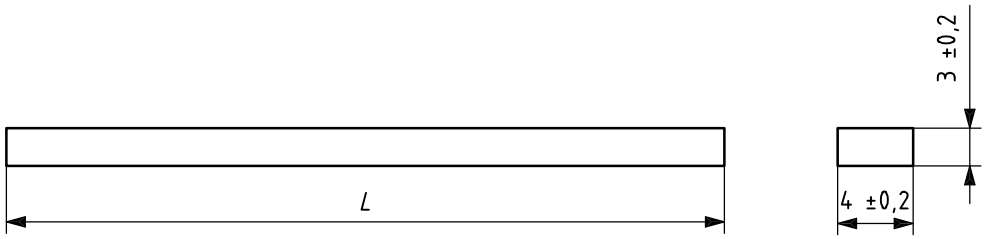
Figure 2 — Example of *S-N* plot

4 Test specimens

4.1 Test specimen size

The test specimen dimensions and tolerances, which are shown in Figure 3, shall be in accordance with ISO 14704. Cross-sectional tolerances shall be 0,2 mm. The use of test specimens having twisting or warpage, which may lead to uneven contact, is not permitted.

Dimensions in millimetres
Edge chamfers at (0,12 ± 0,03) mm × 45° ± 5° or rounded at (0,15 ± 0,05) mm
The parallelism tolerance on opposite longitudinal faces is 0,015 mm



Key

L Specimen length ($\geqslant$ 35 mm for 30 mm test fixture but $\geqslant$ 45 mm for 40 mm test fixture).

Figure 3 — Test specimen

4.2 Specimen preparation

Machining for surface preparation of test specimens shall be based on the procedure described in ISO 14704. The surface roughness should be 0,20 µm Ra or smaller, as defined in ISO 4287, and be reported. The width and thickness of the test specimen shall be measured according to ISO 14704.

4.3 Number of test specimens

The number of identical test specimens that should be prepared depends on the purpose of the fatigue tests. If test conditions for a pass/fail test are set, it is recommended that at least three test specimens be prepared. If the determination of an S-N plot is required, at least 15 test specimens should be prepared, of which three to five are used to determine the flexural strength under monotonic loading based on the testing procedure described in ISO 14704 and the remainder are used to determine the number of cycles to failure for at least three different stress levels.

5 Testing machine

5.1 Structure of testing machine

A testing machine shall be configured so that cyclic bending stress can be applied to a test specimen and forces or moments other than that related to the cyclic bending stress are not applied. The testing machine shall be equipped with an apparatus for measuring or indicating the maximum or minimum force, an apparatus capable of obtaining the number of cycles until the test specimen fractures, and a mechanism where automatic reactivation is prevented when the testing machine is stopped for reasons other than test specimen fracture (e.g. power failure, etc.).

5.2 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.

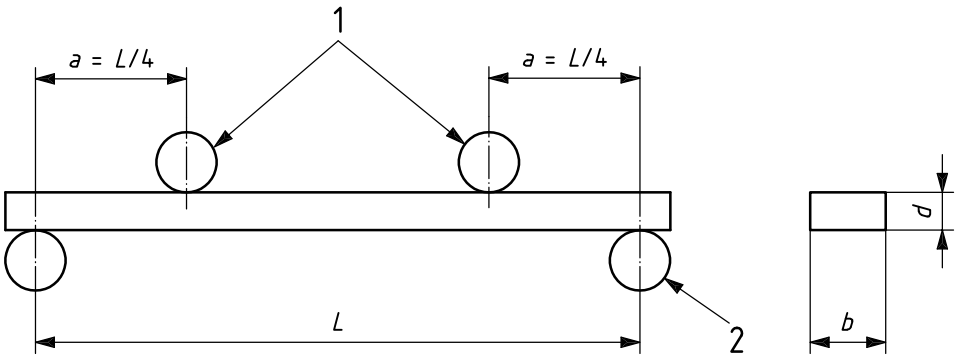
NOTE The scope of ISO 7500-1 does not include dynamic-fatigue machine calibration, but in the absence of a directly applicable standard, the procedure in ISO 7500-1 may be used within this International Standard with the risk of only a small error.

5.3 Test fixture

The four-point flexure fixture specified in ISO 14704 (see Figure 4) shall be used.

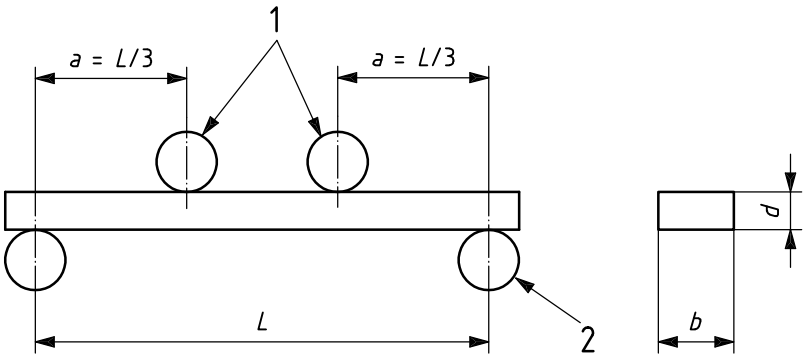
A fixture other than one which is semi-articulating or fully articulating (e.g., one not equipped with free-to-roll bearings) may be used if an articulating fixture is not available. Deviations from the specification described in ISO 14704 shall be reported.

NOTE The influence of friction at the contact between the bearing and test specimen on cyclic-fatigue behaviour is not clear at this time and this International Standard provides some latitude in the use of non-articulating fixtures.



$L = 40$ mm ± 0,1 mm

a) Four-point-1/4 point flexure



$L = 30$ mm ± 0,1 mm

b) Four-point 1/3-point flexure

Key

1 loading bearing
2 supporting bearing
b width of specimen (mm)
d thickness of specimen (mm)

All bearings shall be free to roll.

Figure 4 — Four-point flexure fixture

6 Testing method

6.1 Waveform of loading stress

The recommended stress waveform is a sinusoidal wave of 20 Hz frequency and a stress ratio, $R = 0{,}1$. If other waveforms are employed, the specifics shall be stated in the test report. Regardless of waveform specifics, all tests in a series of fatigue tests shall be conducted using the same waveform.

6.2 Loading method

6.2.1 Either the four-point-1/4 point or the four-point-1/3 point loading configuration specified in ISO 14704 shall be used (see Figure 4).

The ratio of difference between the outer and the inner spans to the test specimen thickness should not be less than 5.

6.2.2 Because the force to achieve the maximum stress is typically not applied upon the first load cycle, it must be slowly increased during subsequent cyclic loading. However, the number of cycles from the start of cycling to that which the final maximum stress is reached should be as few as possible. The peak stress that occurs during this adjustment period shall not exceed the required or specified maximum stress.

6.2.3 The test shall be carried out without a pause of the cyclic loading on the same test specimen from start to end. However, when a test is temporarily stopped during a test for any reason, the number of cycles applied up to the stop, as well as the time duration of the stop, shall be recorded.

6.2.4 Unless the test is being employed for pass/fail purposes, the fatigue test should be carried out on three or more test specimens at each of three or more different stress levels.

NOTE To have meaningful data trends, more than 3 stress levels are preferable. However, there is a tradeoff between testing time and data obtained.

6.3 Number of cycles for suspension

Unless otherwise specified when the test specimen has not fractured after 10^7 cycles, the test may be suspended.

NOTE When 20 Hz frequency is chosen for the loading condition, the 10^7 cycles is equivalent to 5,8 days, approximately.

6.4 Reuse of test specimen

Reuse of a test specimen that has been previously fatigue tested shall be prohibited.

6.5 Recommended test procedure in fatigue test

When the maximum stress and the number of test specimens in a fatigue test are not previously agreed on between the tester and the requester, the test may be carried out according to the procedure described in 6.5.1 and 6.5.2.

6.5.1 Measurement of flexural strength

To obtain a baseline for the maximum stress in a fatigue test, the monotonic flexural strength should be measured in advance. Use flexural strength of test specimens of the same shape and preparation as that of fatigue test specimens. The flexural strength tests shall be conducted using the same fixture as the fatigue test and the testing procedure in accordance with ISO 14704. The mean of the monotonic flexural strength shall be calculated by the standard formulae specified in ISO 14704.

6.5.2 Stress level in fatigue test

For a series of fatigue tests, the first maximum stress level should be chosen such that the maximum stress is less than, but close to, the mean of the monotonic flexural strength. Determine the cycles to failure of at least three fatigue test specimens under this same maximum stress. If none of the fatigue test specimens cycled at this maximum stress level has fractured before the agreed number of cycles for suspending a test, then additional fatigue tests shall be conducted at maximum stress levels greater than the first maximum stress level. When a relation between the maximum cyclic stress and the number of cycles is required, then test specimen fractures at at least three maximum stress levels should be obtained.

6.6 Test environment

Moisture content in the test environment may have an influence on the cyclic fatigue behaviour. For tests in air or another gaseous environment, 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 formulae from measuring values of respective test specimens, and round off to three significant figures in accordance with ISO 31-0.

For four-point flexural test

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

where

$\sigma_{\max}$ is the maximum stress (MPa);

$P_{\max}$ is the maximum force (N);

l is the span of inner bearings (mm);

L is the span of outer bearings (mm);

b is the width of specimen (mm);

d is the thickness of specimen (mm).

7.2 Number of cycles to failure

The number of cycles shall be counted by starting from the time when a force on the test specimen reaches the force necessary to achieve the required maximum stress level. 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, a multiple of 10^n (e.g. $2{,}34 \times 10^6$) and round off to three significant figures.

7.3 *S-N* plot

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

NOTE 1 In the *S-N* plot, when at least two points are superimposed, the number of points is clearly described, or arrows are marked by the number of points.

NOTE 2 When the arrow is superimposed on a marked line, the arrow is so marked as to become clear by drawing it up or down.

8 Test report

The test report shall be in accordance with ISO 17025, unless there are valid reasons for not doing so. The report of the results of a fatigue test shall include the following items:

a) the name and address of the testing establishment;

b) the date of the test, a unique identification of the test report and of each page, customer name and address, signatory of the report;

c) a reference to this International Standard, i.e., determined in accordance with ISO 22214;

d) a description of the test material, batch codes, date of manufacture, as appropriate;

e) dimensions (mean values) of test specimen;

f) sampling conditions of the test specimen taken from the material and its machining conditions (when a test specimen is heat treated, its conditions are included);

g) name of testing machine and its type;

h) loading conditions (loading method, loading waveform, loading frequency, R ratio, and the number of cycles to the stop if a test is temporarily interrupted and the duration of the interruption);

i) a list of test results (the maximum stress and the number of cycles to failure), including those determined from monotonic loading, if undertaken;

j) if necessary, *S-N* plot;

The report of the results of a fatigue test should preferably also include the following items:

k) conditions of the test environment, such as temperature, humidity, gas, etc.;

NOTE When the test environment is controlled, item k) shall be reported.

l) class and name of material, kinds of additives, and sintering method;

m) chemical composition of material;

n) mechanical properties of the material, such as flexural strength, elastic modulus, fracture toughness value, etc.

Bibliography

[1] ISO 4287:1997, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Terms, definitions and surface texture parameters*

[2] ISO 7500-1, *Metallic materials — Verification of static uniaxial testing machines — Part 1: Tension/compression testing machines — Verification and calibration of the force-measuring system*

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