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BSI Standards Publication

Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for thermal fatigue of fine ceramics substrate

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National foreword

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Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for thermal fatigue of fine ceramics substrate

Céramiques techniques — Méthode d'essai de la fatigue thermique des substrats des céramiques techniques

Reference number
ISO 17841:2015(E)

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Foreword

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The committee responsible for this document is ISO/TC 206, *Fine ceramics*.

INTERNATIONAL STANDARD ISO 17841:2015(E)

Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for thermal fatigue of fine ceramics substrate

1 Scope

This International Standard describes the test methods for determining thermal fatigue property of fine ceramic substrates applied to power modules, electronic control unit and similar devices.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

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

ISO 13385-1, *Geometrical product specifications (GPS) — Dimensional measuring equipment — Part 1: Callipers; Design and metrological characteristics*

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

3 Terms and definitions

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

3.1
ceramic substrate
electrical circuits substrate which joined the metal plate to ceramics, applied to power modules and similar devices

3.2
thermal cycling
temperature which is simply and cyclically repeated between a specific high value and a specific low value

3.3
four-point bending strength
maximum value of stress generated in a test specimen obtained by a four-point bending test

3.4
residual strength ratio
value ($\sigma_b / \overline{\sigma_{b0}}$) of four-point bending strength of a test specimen exposed to thermal cycling (σ_b) divided by the mean four-point bending strength of test specimens not exposed to thermal cycling ($N = 0$) ($\overline{\sigma_{b0}}$)

4 Testing machine and equipment

4.1 Thermal cycling testing equipment

Two types of thermal cycling equipment may be used. Type 1 provides two chambers one at a high temperature and the other at a low temperature and a means of moving the test specimen from one to the other within a specified time period. Type 2 provides a single chamber in which the temperature can be changed. The equipment shall be able to maintain the specified temperatures. Attachments and supports used with the test specimens shall have low thermal conductivity and the test specimens shall be thermally insulated. Test specimens shall be arranged so as not to disturb the circulation in the chambers and between specimens when multiple specimens are tested simultaneously.

4.2 Four-point bending strength testing equipment

This testing equipment shall be so constructed that compressive stress can be applied to a test specimen at a constant crosshead speed. The testing machine shall be equipped with an apparatus measuring or indicating the load with an accuracy of at most 1 % of the maximum load.

4.3 Supports for four-point bending strength testing

Four-point flexure configurations measured four-point bending strength shall be used as illustrated in Figure 3. Supports wherein a testing specimen is supported as supporting points, or a load is applied to the specimen, shall be of the same shape in left and right and have a length exceeding a width of the specimen. The material of the supports shall be at least 147 GPa in elastic modulus, and free from plastic deformation and being broken midway through testing. The diameter of supports shall be 4,0 mm to 6,0 mm, and its surface roughness shall be at most 0,40 µm *Ra* as specified in ISO 4287. Also, supports should be able to be rotated and not to disturb the transformation of the test specimen by load.

4.4 Micrometre callipers

Micrometre callipers shall be those for external measurement specified in ISO 3611 or shall have better precision.

4.5 Vernier callipers

Vernier callipers shall be those for external measurement specified in ISO 13385-1 or shall have better precision.

5 Test specimens

5.1 Test specimen form and dimensions

5.1.1 General

The shape of test specimens shall be two metal plates joined respectively to the front and back of a fine ceramics plate, i.e. a total of four metal sheets, as shown in Figure 1. The standard dimensions of the fine ceramics shall be: total length h = 40,0 mm, width w = (10,0 ± 0,5) mm, and metal plate dimensions shall be: total length h_m = 17,0 mm, width w_m = (8,0 ± 0,5) mm. Metal plate spacing shall be d = 2 mm. The four corner portions of metal plates shall be chamfered to R = 1,0 mm, as shown in Figure 1. Any dimensions differing from the standard dimensions shall be mentioned in the test report.

5.1.2 Fine ceramics plate thickness t and metal plate thickness t_m

Fine ceramics plate thickness t and metal plate thickness t_m shall follow product specifications, but in principle, t and t_m shall be selected from the plate thicknesses listed in 5.1.2.1 and 5.1.2.2.

5.1.2.1 Fine ceramic sheet thickness *t*

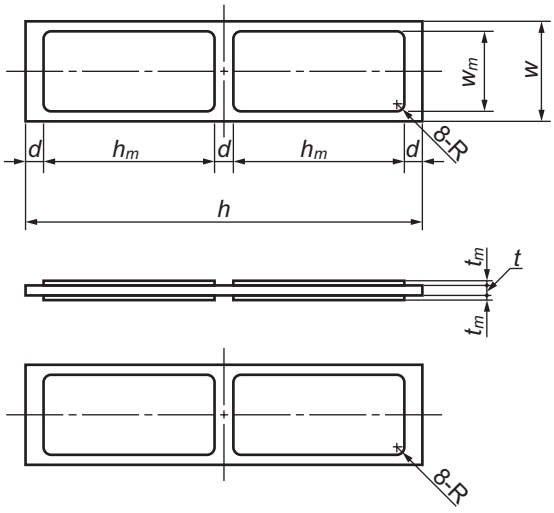
Thickness t = 1,50 mm; 1,00 mm; 0,787 mm; 0,635 mm; 0,381 mm; 0,32 mm; 0,254 mm; 0,2 mm

5.1.2.2 Metal plate thickness t_m

Thickness t_m = 0,6 mm; 0,4 mm; 0,3 mm; 0,25 mm; 0,2 mm; 0,125 mm

5.2 Number of test specimens

The number of test specimens shall be 10 or more for each thermal cycle measuring four-point bending strength.



Key

h length of ceramics (h = 40,0 mm)
w width of ceramics (w = 10,0 mm ± 0,5 mm)
t thickness of ceramics (t = 0,2 mm to 1,50 mm)
h_m length of metal plate (h_m = 17,0 mm)
w_m width of metal plate (w_m = 8,0 mm ± 0,5 mm)
t_m thickness of metal plate (t_m = 0,125 mm to 0,6 mm)
d metal plate spacing (d = 2 mm)
R chamfering (R = 1mm)

Figure 1 — Test specimen dimensions

6 Testing methods

6.1 Test specimen dimension measurements

The horizontal and vertical lengths and thickness of the specimen shall be preliminarily measured in a precision of 0,01 mm by using micrometre callipers for external measurement specified in ISO 3611, Vernier callipers specified in ISO 13385-1 or a measuring instrument at least equal in precision thereto.

6.2 Thermal fatigue (thermal cycling) testing methods

Thermal fatigue (thermal cycling) testing shall be carried out after test specimen dimensions are measured.

6.2.1 Thermal fatigue (thermal cycling) conditions

Thermal cycling (thermal cycling) conditions are established by combinations of low temperature T_A, high temperature T_B, exposure time t_1, transfer time t_2, temperature recovery time t_s, and number of cycles in thermal fatigue N.

6.2.2 Low-temperature tank inside T_A and high-temperature tank inside T_B

Low-temperature tank inside T_A and high-temperature tank inside T_B follow product specifications (see Figure 2). T_A and T_B shall be also each selected from the testing temperatures listed in 6.2.2.1 and 6.2.2.2 respectively.

6.2.2.1 Low-temperature tank inside T_A

T_A = −65 °C ± 2 °C, −55 °C ± 2°C, −40 °C ± 2 °C, −25 °C ± 2 °C, −10 °C ± 2 °C, −5 °C ± 2 °C, +5 °C ± 2 °C

6.2.2.2 High-temperature tank inside T_B

T_B = +200 °C ± 2 °C, +175 °C ± 2 °C, +150 °C ± 2 °C, +125 °C ± 2 °C, +100 °C ± 2 °C, +85 °C ± 2 °C, +70 °C ± 2 °C, +55 °C ± 2 °C, +40 °C ± 2 °C, +30 °C ± 2 °C

6.2.3 Number of cycles in thermal fatigue N

Number of cycles in thermal fatigue follows product specifications. Additionally, standard N shall be 0, 10, 100, 300, 1 000, and 3 000 cycles, and one or multiple N are selected from these cycles. Any other N number different from the standard values shall be mentioned in the test report.

6.2.4 Each exposure time t_1 to low temperature and high temperature

Exposure time t_1 shall account for the heat capacity of the test specimen (see Figure 2). Additionally, standard t_1 shall be 30 min. Any other value of t_1 different from the standard values due to the heat capacity of the test specimen shall be mentioned in the test report.

6.2.5 Transfer time t_2

Transfer time t_2 is the time for transferring test specimen from the low temperature tank to the high temperature tank, or from the high temperature tank to the low temperature tank, and should be kept as short as possible due to the most severe temperature change for the test specimen (see Figure 2). Standard t_2 shall be selected from the following group. Any other value of t_2 different from the standard values shall be mentioned in the test report.

a) $t_2 < 10$ s

b) 10 s ≤ $t_2 < 1$ min

c) 1 min ≤ $t_2 < 2$ min

6.2.6 Temperature recovery time t_s

Temperature recovery time t_s is the time elapsed from test specimen placement in a tank to settling of the temperature in a tank to T_A or T_B, and t_s shall be within 20 % of t_1 and is included in exposure time t_1 (see Figure 2). Any other value of t_s different from the standard values shall be mentioned in the test report.

6.2.7 Single cycle

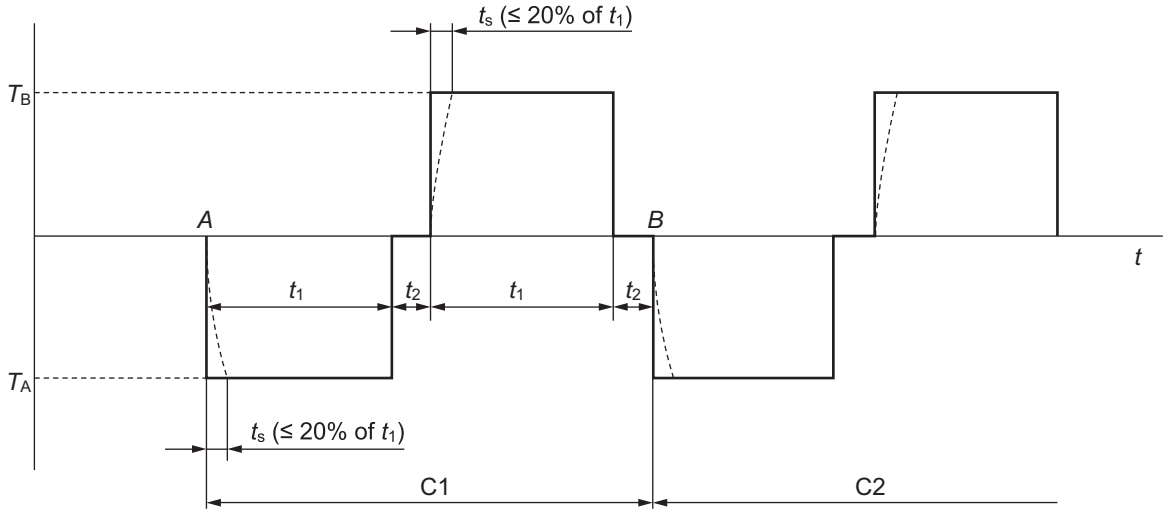
A single cycle comprises two exposure times t_1 and two transfer times t_2, as shown in Figure 1. Thermal cycling shall start in the low temperature tank, and shall end in the high temperature tank. The following sequence shall be repeated.

a) Place the test specimen in the low temperature tank and maintain for the interval t_1. Take t_s as the time from test specimen placement in the low temperature tank to settling of the temperature in a tank to low-temperature tank inside T_A.

b) Transfer the test specimen from the low temperature tank to the high temperature tank within the duration of t_s.

c) Place the test specimen in the high temperature tank and maintain for the interval t_1. Take t_s as the time from test specimen placement in the high temperature tank to settling of the temperature in a tank to high-temperature tank inside T_B.

d) Transfer the test specimen from the high temperature tank to the low temperature tank within the duration of t_s.



Key

t time
C1 Cycle 1
C2 Cycle 2
A start time of Cycle 1
B start time of Cycle 2

Figure 2 — Thermal cycling

6.3 Testing methods of four-point bending strength

Four-point bending strength shall be carried out after thermal fatigue (thermal cycling) testing is completed.

6.3.1 Distance between inner fulcrums (inner span) and distance between outer fulcrums (outer span)

In a four-point bending test for a four-point bending strength, a distance between outer supporting points *L* shall be 30 mm ± 0,5 mm, and a distance between inner supporting *l* shall be 10 mm ± 0,5 mm as shown in Figure 3. The distances shall be measured before tests by Vernier callipers.

6.3.2 Crosshead speed and fracture load

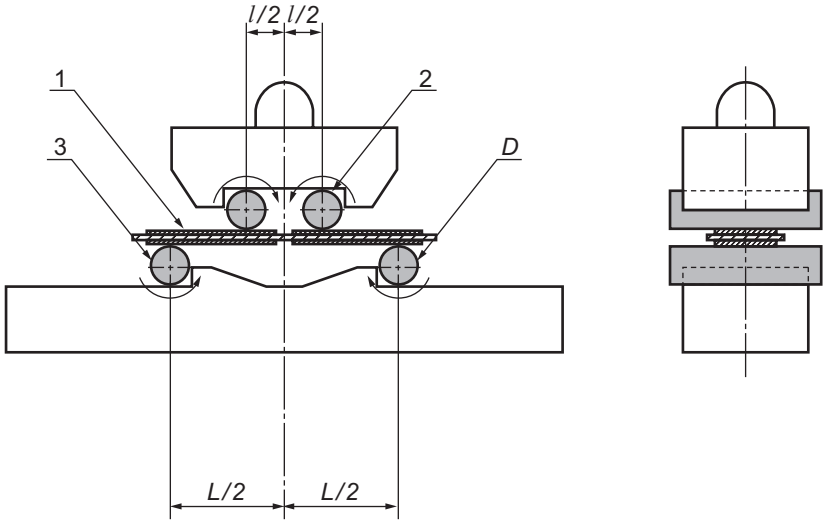
The test specimen shall be loaded with weight at a crosshead speed of 0,5 mm/min, and the maximum load until test specimen fracture shall be measured.

6.3.3 Measurement of load-displacement

Load and displacement shall be measured from the start of the test to the specimen fracture.

6.4 Four-point bending strength testing for virgin specimens not exposed to thermal cycling

Four-point bending strength shall be tested using 10 virgin specimens not exposed to thermal cycling or more for calculation of residual strength ratio.



Key

l distance between inner fulcrums (*l* = 10,0 mm ± 0,5 mm)

L distance between outer fulcrums (*L* = 10,0 mm ± 0,5 mm)

D diameter of fulcrum (*D* = ϕ4 to 6 mm)

1 test specimen

2 inner fulcrums (fulcrums rotatable)

3 outer fulcrums (fulcrums rotatable)

Figure 3 — Four-point bending fixture

7 Calculation of test results

7.1 Calculation of four-point bending strength

Four-point bending strength shall be calculated using Formula (1) from measured values for individual test specimens, and round off to significant figures in accordance with ISO 80000-1.

$$\sigma_b = \frac{3P(L-l)}{2wt^2} \tag{1}$$

where

σ_b is the four-point bending strength (MPa);

P is the maximum load at test specimen breakage (N);

l is the distance between inner fulcrums (mm);

L is the distance between outer fulcrums (mm);

w is the test specimen width (mm);

t is the test specimen thickness (mm).

7.2 Calculation of mean values and standard deviations

The mean value and the standard variation for four-point bending strength shall be calculated using Formulae (2) and (3) from measured values for individual test specimens, and round off to significant figures in accordance with ISO 80000-1.

$$\bar{x} = \sum_{i=1}^{n} \frac{x_i}{n} \tag{2}$$

$$S = \sqrt{\sum_{i=1}^{n} \frac{(x_i - \bar{x})^2}{n-1}} \tag{3}$$

where

$\bar{x}$ is the mean four-point bending strength (MPa);

x_i is the four-point bending strength of individual test specimen (MPa);

S is the standard deviation of four-point bending strength (MPa);

n is the number of test specimens.

7.3 Residual strength ratio

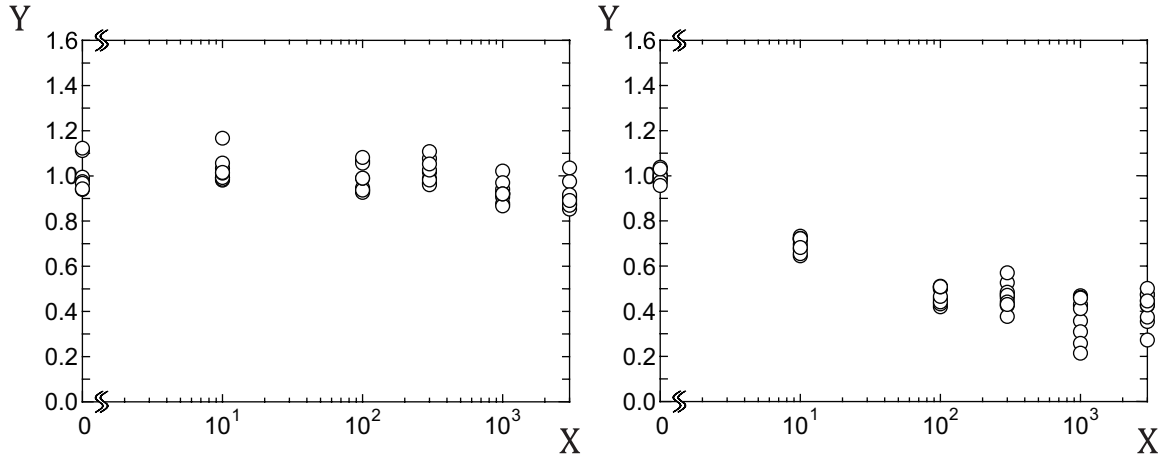
7.3.1 Calculation of residual strength ratio

Residual strength ratio ($\sigma_b / \overline{\sigma_{b0}}$) shall be the four-point bending strength of a test specimen exposed to thermal cycling (σ_b) divided by the mean four-point bending strength of 10 test specimens not exposed to thermal cycling ($\overline{\sigma_{b0}}$) (N = 0), and round off to significant figures in accordance with ISO 80000-1.

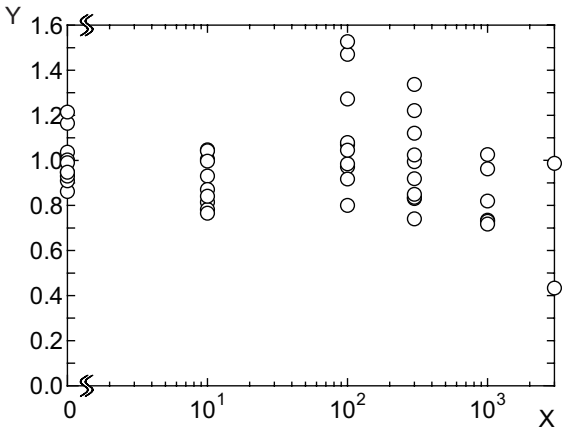
7.3.2 Residual strength ratio – Thermal cycle number plots

Residual strength ratio – number of cycles in thermal fatigue plotted diagram shall be drawn with residual strength ratio $\sigma_b / \overline{\sigma_{b0}}$ on the vertical axis and the log of number of cycles in thermal fatigue N on the horizontal axis.

Figure 4 shows residual strength ratio – number of cycles in thermal fatigue plotted diagram. Figure 4 a) is an example in which residual strength ratio $\sigma_b / \overline{\sigma_{b0}}$ is almost unchanged even as number of cycles in thermal fatigue N increases; Figure 4 b) is an example in which residual strength ratio $\sigma_b / \overline{\sigma_{b0}}$ decreases as number of cycles in thermal fatigue N increases. The decrease in residual strength ratio $\sigma_b / \overline{\sigma_{b0}}$ is caused by cracks initiated/growing in the ceramics of the ceramic substrate by thermal cycling; Figure 4 c) is an example in which residual strength ratio $\sigma_b / \overline{\sigma_{b0}}$ significantly does not decrease as number of cycles in thermal fatigue N increases, but $\sigma_b / \overline{\sigma_{b0}}$ has the large dispersion. The dispersion in residual strength ratio $\sigma_b / \overline{\sigma_{b0}}$ is caused by cracks initiated/growing inside or at the interface of the brazing material in between the ceramic substrate and metal plate by thermal cycling.



a) Residual strength ratio unchanged **b) Residual strength ratio decreasing**



c) Residual strength ratio dispersing

Key

X number of thermal cycles N, cycles

Y residual strength ratio $\sigma_b / \overline{\sigma_{b0}}$

Figure 4 — Residual strength ratio — Number of cycles in thermal fatigue plotted diagram

Additional information on calculation of stress intensity factor at fracture is provided in Annex A.

8 Test report

8.1 Items to be reported

The test report shall include the following items:

a) a reference to this International Standard, i.e. ISO 17841;

b) name and class of material;

c) dimensions (mean value) of test specimen;

d) number of test specimens tested;

e) name of testing machine for thermal fatigue and its type;

f) conditions of thermal fatigue test (low temperature, high temperature, transfer time, temperature recovery time, exposure time, thermal cycle number);

g) name of testing machine for four-point bending strength and its type;

h) conditions of four-point bending test (cross-head speed and distance between supporting points);

i) residual strength ratio – number of cycles in thermal fatigue plotted diagram;

j) mean value and standard deviation of four-point bending strength;

k) mean value and standard deviation of residual strength ratio.

8.2 Additional items to be reported

The following items should be supplemented to the report:

a) manufacturer's name of material and its date of manufacture;

b) name of material, kinds of additive, and sintering method;

c) chemical composition of materials;

d) conditions for collecting and processing from material for manufacture of test specimens;

e) conditions for test atmosphere such as temperature, humidity, etc.;

f) type of heating medium of thermal fatigue test;

g) date of test, test place, and name of test person.

Annex A
(informative)

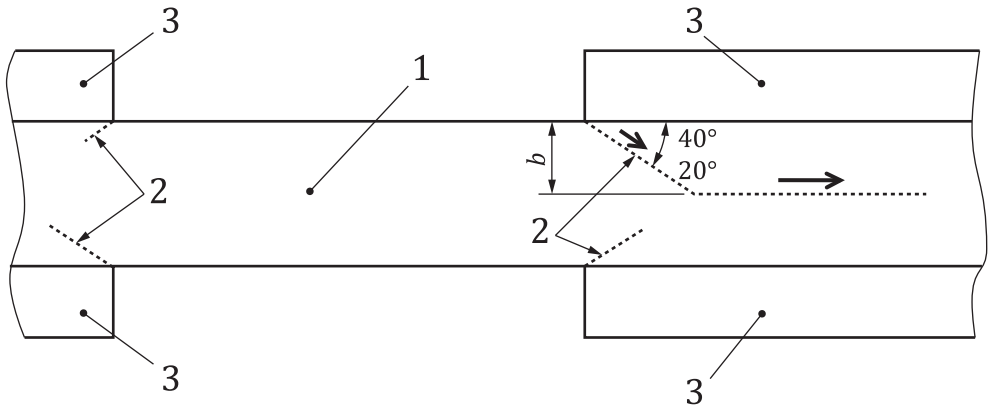
Calculation of stress intensity factor at fracture

A.1 Observation and measurement of cracks initiated/growing due to thermal fatigue (thermal cycling) and calculation of stress intensity factor at fracture

The reason the four-point bending strength of a test specimen exposed to thermal cycling decreases is that the cracks are initiated and grow in ceramics due to thermal fatigue (thermal cycling). As necessary, the stress intensity factor at fracture may be calculated using length of crack on the fracture surface of ceramics, and stress intensity factor – crack depth plots may be drawn. It is possible to examine the condition of the fracture for ceramics substrates.

A.1.1 Observation and measurement of cracks initiated/growing due to thermal fatigue (thermal cycling)

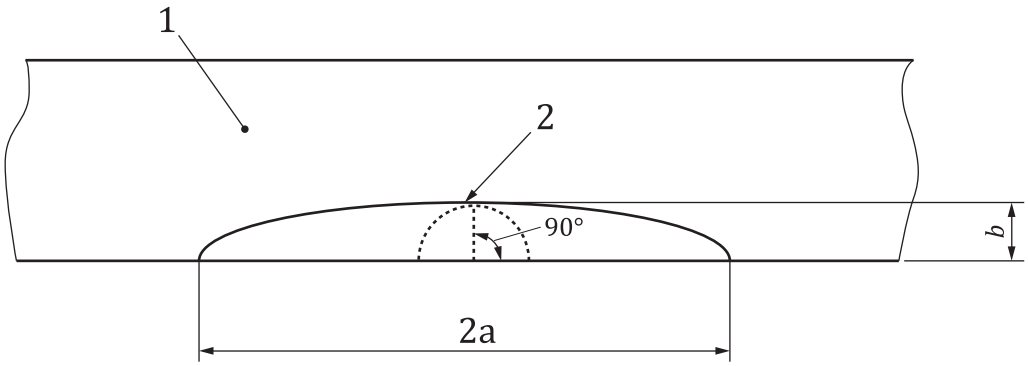
Cracks are initiated and grow in ceramics due to thermal fatigue (thermal cycling). Figure A.1 shows a schematic of crack propagation, as seen from the side of a test specimen. As shown in Figure A.1, cracks are initiated at the ceramics surface close to the edge of the metal plates, and grow from the ceramic surface toward the interior at an angle of approximately 20° to 40°, and then progress parallel to the interface of ceramics and metal plate. After four-point bending strength measurement, semi-elliptical cracks are observed on the fracture surface of ceramics as shown in Figure A.2. For calculation of the stress intensity factor at fracture, the length $2a$ and the depth b of the cracks are measured by perpendicularly projecting to a longitudinal direction of the ceramic substrate using a scanning electron microscope, optical microscope or similar equipment.



Key

b crack depth
1 ceramics
2 cracks
3 metal plates

Figure A.1 — Schematic of crack growth pattern due to thermal fatigue (thermal cycling), as seen from side of test specimen



Key

2*a* crack length
b crack depth
1 ceramics
2 semi-circular arc crack

Figure A.2 — Model of crack, as seen from fracture surface of ceramics

A.1.2 Calculation of stress intensity factor at fracture

Stress intensity factor at breaking of a ceramic substrate may be calculated using Formula (A.1) using four-point bending strength σ_b determined in four-point bending strength testing, half crack length *a* and crack depth *b*, determined from crack observation, test specimen width *w*, and test specimen thickness *t*, and round off to significant figures in accordance with ISO 80000-1. Refer to A.2 for the correction factor formulae for calculation of stress intensity factor.

$$K = \sigma_b \sqrt{\pi b} \cdot f(\xi) \tag{A.1}$$

where

K is the stress intensity factor (MPam$^{1/2}$);

σ_b is the four-point bending strength (MPa);

b is the crack depth (m);

$f(\xi)$ is the correction factor.

A.1.3 Calculation of mean values and standard deviations

The mean value and the standard variation for stress intensity factor may be calculated using Formulae (A.2) and (A.3) using measured values for individual test specimens, and round off to significant figures in accordance with ISO 80000-1.

$$\bar{x} = \sum_{i=1}^{n} \frac{x_i}{n} \tag{A.2}$$

$$S = \sqrt{\sum_{i=1}^{n} \frac{(x_i - \bar{x})^2}{n-1}} \tag{A.3}$$

where

$\bar{x}$ is the mean stress intensity factor (MPam$^{1/2}$);

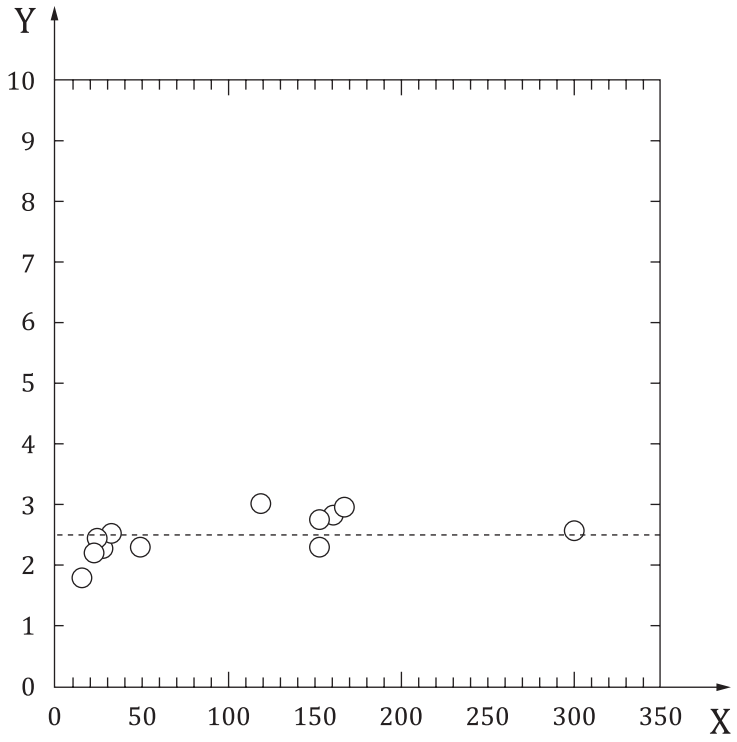
x_i is the stress intensity factor of individual test specimen (MPam$^{1/2}$);

S is the standard deviation of stress intensity factor (MPam$^{1/2}$);

n is the number of test specimens.

A.1.4 Stress intensity factor – crack depth plots

Stress intensity factor – crack depth plots may be drawn with stress intensity factor K on the vertical axis and crack depth b on the horizontal axis. Figure A.3 shows an example of a stress intensity factor – crack depth plotted diagram. The broken line in Figure A.3 shows the mean stress intensity factor $\bar{K}$.



Key

X crack depth b, µm

Y stress intensity factor K_I, MPa·m$^{1/2}$

Figure A.3 — Stress intensity factor – crack depth plotted diagram

A.2 Equations for calculation of stress intensity factor

When bending load affects the semi-elliptical surface crack on the test specimen, a formula of stress intensity factor is given by the following Formulae (A.4) to (A.18)[2]. Where, σ_b is four-point bending

strength determined in four-point bending strength testing, *a* and *b* is half crack length and crack depth determined from crack observ*a*tion, *w* and *t* are width and thickness of test specimen.

$$K = H \cdot \frac{\sigma_b \sqrt{\pi b}}{E\left(K\right)} \cdot F\left(\frac{b}{t}, \frac{b}{a}, \frac{a}{w}, \phi\right) \tag{A.4}$$

$$E(K) = \left[1 + 1,464\left(\frac{b}{a}\right)^{1,65}\right]^{\frac{1}{2}}, \left(\frac{b}{a} \leq 1\right) \tag{A.5}$$

$$F = \left[M_1 + M_2\left(\frac{b}{t}\right)^2 + M_3\left(\frac{b}{t}\right)^4\right] f_\phi g f_w \tag{A.6}$$

$$M_1 = 1,13 - 0,09\left(\frac{b}{a}\right) \tag{A.7}$$

$$M_2 = -0,54 + \frac{0,89}{0,2 + \left(\frac{b}{a}\right)} \tag{A.8}$$

$$M_3 = 0,5 - \frac{1,0}{0,65 + \left(\frac{b}{a}\right)} + 14\left(1,0 - \frac{b}{a}\right)^{24} \tag{A.9}$$

$$g = 1 + \left[0,1 + 0,35\left(\frac{b}{t}\right)^2\right]\left(1 - \sin\phi\right)^2 \tag{A.10}$$

$$f_\varphi = \left[\left(\frac{b}{a}\right)^2 \cos^2\phi + \sin^2\phi\right]^{\frac{1}{4}} \tag{A.11}$$

$$f_w = \left[\sec\left(\frac{\pi a}{w}\sqrt{\frac{b}{t}}\right)\right]^{\frac{1}{2}} \tag{A.12}$$

$$H = H_1 + \left(H_2 - H_1\right)\sin^p\phi \tag{A.13}$$

$$p = 0,2 + \frac{b}{a} + 0,6\frac{b}{t} \tag{A.14}$$

$$H_1 = 1 - 0,34\frac{b}{t} - 0,11\frac{b}{a}\left(\frac{b}{t}\right) \tag{A.15}$$

$$H_2 = 1 + G_1\left(\frac{b}{t}\right) + G_2\left(\frac{b}{t}\right)^2 \tag{A.16}$$

$$G_1 = -1,22 - 0,12\frac{b}{a} \tag{A.17}$$

$$G_2 = 0{,}55 - 1{,}05\left(\frac{b}{a}\right)^{0{,}75} + 0{,}47\left(\frac{b}{a}\right)^{1{,}5} \tag{A.18}$$

where

K is the stress intensity factor (MPam$^{1/2}$);

σ_b is the four-point bending strength (MPa);

b is the crack depth (m);

a is the half crack length (m);

w is the test specimen width (m);

t is the test specimen thickness (m);

ϕ is the angle from crack surface to location where stress intensity factor is determined; in this instance, ϕ = 90°;

H is a correction factor;

$E(K)$ is a correction factor;

$f\left(\frac{b}{t}, \frac{b}{a}, \frac{a}{w}, \phi\right)$ is a correction factor.

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[1] ISO 1101, *Geometrical product specifications (GPS) — Geometrical tolerancing — Tolerances of form, orientation, location and run-out*

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