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Fine ceramics (advanced ceramics, advanced technical ceramics) — Mechanical properties of monolithic ceramics at room temperature — Determination of flexural strength by the ring-on-ring test

Céramiques techniques — Propriétés mécaniques des céramiques monolithiques à température ambiante — Détermination de la résistance à la flexion à l'aide de doubles anneaux

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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) — Mechanical properties of monolithic ceramics at room temperature — Determination of flexural strength by the ring-on-ring test

1 Scope

This document specifies a method for the determination of the nominal equibiaxial flexural strength by the ring-on-ring test of advanced monolithic technical ceramic materials at room temperature.

This document is applicable to materials with a grain size less than 100 µm.

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 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*

ISO/IEC 17025, *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.

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
equibiaxial flexural strength
σ_{rr}
maximum nominal stress at the test piece surface supported by the material at the instant of failure when loaded in linear elastic equibiaxial bending by the ring-on-ring test

Note 1 to entry: The ring-on-ring test for the determination of flexural strength is often referred to as biaxial bending test or equibiaxial bending test (see Reference [1]). These names should not be used without the reference to the ring-on-ring test conditions as there are some other test arrangements for which the term is used or could also be used, such as the ball-on-three-balls test, the punch-on-three-balls test, the ball-on-ring test and the punch-on-ring test.

4 Principle

A round discoidal test piece with a constant thickness is positioned between two concentric rings of different diameters and loaded by an axial force.

The nominal equibiaxial flexural strength is calculated from the axial force which acts during the moment of fracture, the geometry of the test piece and the test arrangement, as well as the Poisson ratio of the test piece material.

5 Significance and use

This test is intended to be used for material development and characterization, quality control and design data acquisition purposes. The strength level determined by the test is calculated on the basis of linear elastic bending behaviour of a round, disk-like thin plate on the assumption that the material being tested is elastically homogeneous and isotropic and shows a linear (Hooke) stress-strain behaviour.

The result obtained from a strength test is determined by a large number of factors associated with the microstructure of the material, the surface finishing procedure applied in preparation of the test pieces, the size and shape of the test piece, the mechanical function of the testing apparatus, the rate of load application and the relative humidity (RH) of the ambient atmosphere. As a consequence of the brittle nature of ceramics, there is usually a considerable range of results obtained from a number of nominally identical test pieces. These factors combined mean that caution in the interpretation of test results is required. For many purposes, and as described in this document, the results of strength tests can be described in terms of a mean value and a standard deviation. Further statistical evaluation of results is required for design data acquisition and can be desirable for other purposes (see ISO 20501).

This method places closely defined restrictions on the size and shape of the test piece and on the function of the test apparatus in order to minimize the errors that can arise as a consequence of the test procedure.

NOTE The basis for the choice of dimensions and tolerances of test pieces and of the requirements of the test jig can be found in ISO 6474-1, ASTM C1499 and Reference [1].

All other test factors are required to be stated in the test report (see Clause 10) in order to allow inter-comparison of material behaviours. It is not possible rigorously to standardize particular surface finishes since these are not absolutely controllable in mechanical terms. The inclusion of a standard grinding procedure (see 7.3) as one of the surface finish options in this method is intended to provide a means of obtaining a minimum amount of residual grinding damage to the test material.

WARNING — The extrapolation of equibiaxial flexural strength data to other geometries of stressing, to single-axial stressing, to other rates of stressing or to other environments should be viewed with caution.

The information about the origin of fracture in an equibiaxial flexural strength test can be a valuable guide to the nature and position of strength-limiting defects. Fractography of test pieces is highly recommended (see e.g. ASTM C1499, Reference [1], Reference [2]) and Annex A. In particular, the test can identify fracture origins as being edge defects (caused by edge preparation), surface defects (caused by surface preparation) or internal defects (caused by manufacturing inhomogeneities such as pores, large grains or impurity concentrations). Not all advanced monolithic technical ceramics are amenable to clear fractography.

6 Apparatus

6.1 Test machine

The test machine shall be capable of applying a force over a ball or a suitable articulation device in that way that the loading ring acts perpendicular to the surface of the test piece so that the two rings (loading ring and support ring) have uniform contact to the face surfaces of the test piece.

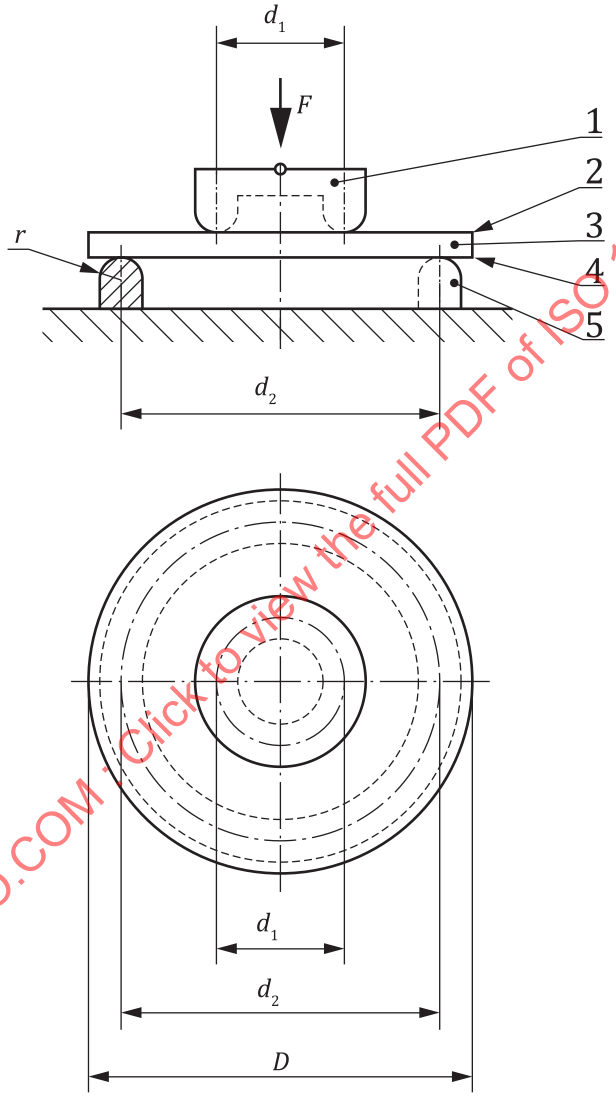
The machine shall be capable of applying the force shock-free and with controlled force or displacement. The test machine shall be equipped for recording the maximum force applied to the test piece. The accuracy of the test machine shall be in accordance with ISO 7500-1, grade 1.

6.2 Test jig

The test jig for the ring-on-ring test shall be as specified below in order to minimize misalignments and frictional forces applied to the test piece.

NOTE 1 The precise construction of the test jig is not fixed in this document but the function is.

A schematic test setup for the examination of the nominal equibiaxial flexural strength by the double-ring flexural test is shown in Figure 1.



Key

1	loading ring	d_1	contact diameter of the loading ring
2	thin adhesive film on the test piece	d_2	contact diameter of the support ring
3	test piece	D	diameter of the test piece
4	rubber film under the test piece	F	force applied
5	support ring	r	contact radius

Figure 1 — Example of a test setup for the determination of flexural strength by the ring-on-ring test

The test jig shall consist of two concentric rings of different diameters, namely the support ring (large), and the loading ring (small), see Figure 1.

The following standard dimensions shall be observed:

— contact diameter of the loading ring, d_1: (12 ± 0,1) mm;

— contact diameter of the support ring, d_2: (30 ± 0,1) mm;

— radius of curvature of the rings in the region of contact with the test piece surface, r: (2 ± 0,2) mm.

NOTE 2 This test setup has been used for decades to characterize the stress of advanced technical alumina ceramics for orthopaedic implants (see ISO 6474-1).

A suitable centring device or procedure for the test jig shall be used ensuring a centred positioning of the loading ring, the test piece and the support ring to one another within 0,2 mm.

Either the support ring or the loading ring shall be fixed to the punch of the test machine used so that an adjustment of the rings to the test piece is also possible if the test piece is not perfectly coplanar flat.

The two rings shall be made from hardened steel with a Vickers hardness greater than 500 HV or greater than 40 HRC (Rockwell C-scale), or another material with at least the hardness of the material tested (e.g. hard metal, ceramic) to minimize damage to the rings which can occur during the fracture.

To compensate small deviations of the surface flatness of the discoidal test piece, a thin rubber film with a thickness of (0,6 ± 0,1) mm and a shore A hardness of (60 ± 5) shall be positioned between the support ring and the test piece.

The flatness (and/or waviness) of the loading and the supporting rings shall be equal or smaller than 0,02 mm.

6.3 Calliper or alternative calibrated device for the measurement of the test piece thickness

For the determination of the test piece dimensions, a calibrated calliper or an alternative calibrated device with a resolution ≤ 0,1 % of the dimension to be measured shall be used.

It means that the resolution of the measurement device for the test piece thickness shall be ≤ 0,002 mm and for the test piece diameter ≤ 0,04 mm.

6.4 Device for measuring the temperature and humidity

A device for measuring the temperature with a resolution of at least 1 °C and a humidity measuring device with a resolution of at least 5 % RH shall be used.

7 Test pieces

7.1 General

The test pieces shall be prepared by processes in accordance with the technologies used during the production of the products. They may either be specially processed to, or close to, the final required dimensions specified in 7.2, or may be machined from larger blocks or components.

NOTE 1 The strength of many types of advanced monolithic technical ceramics is strongly influenced by the machining procedure used for the preparation of the test pieces. Low strengths might be caused by grinding with coarse diamond grit sizes and, conversely, very high strengths might be obtained if care is taken in surface preparation. Some materials, especially those containing transformable zirconia, could be markedly strengthened by appropriate grinding schedules. The definition of surface preparation conditions is therefore an important aspect of this test method.

Because of the different surface preparation conditions there are no limits specified for the flatness (and/or waviness) for the test pieces.

If as-fired samples, especially very flat samples, have to be tested (e.g. substrates, semiconductor wafers), there could be some contact issues with asymmetric loading if the dimensions of the sample are not well described. For example, the maximum of total thickness variation (TTV), warp, bow and sori should be defined or at least considered.

On the compressive stressed surface of the test piece (the side of the test piece which is turned to the loading ring), a thin adhesive film shall be fixed to compensate unevenness of the test piece surface and to keep the fracture pieces together after the fracture of the test piece.

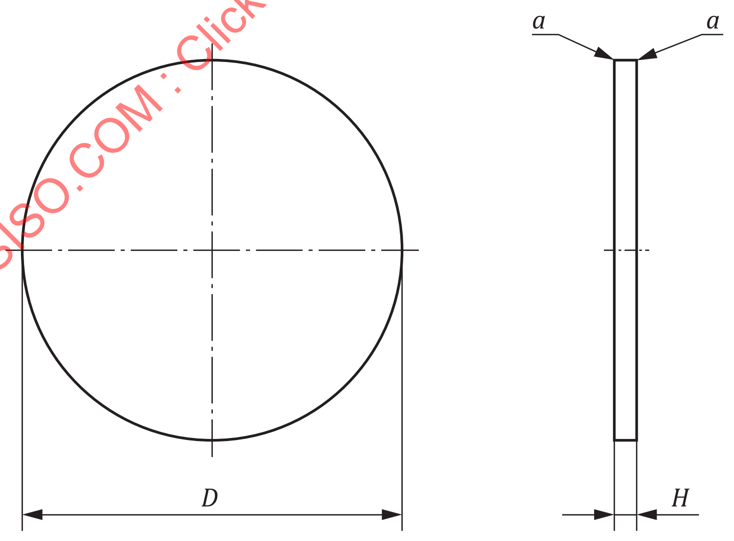
NOTE 2 A first visual fracture analysis is possible immediately after the bending test. The intention of this fracture analysis is to decide whether the fracture origin lies at or inside the loading ring region (regular fracture) or outside the loading ring region (irregular fracture).

7.2 Dimensions and tolerances

Dimensions and tolerances of test pieces shall be as shown in Table 1 and Figure 2 unless otherwise agreed between the parties.

Table 1 — Dimensions and tolerances of the test pieces

Test piece type	Final surface preparation condition	Parameter	Diameter *D* mm	Thickness *H* mm
Standard version	I, II, III (all conditions)	Dimensional range	36 ± 0,5	2,0 ± 0,1
	II, III (machined)	Parallelism tolerance	—	±0,02
	I (as-fired)	Parallelism tolerance	—	±0,05



a) Top view b) Side view

Key

D diameter of the test piece

H thickness of the test piece

a edges: sharp, chamfered or rounded

Figure 2 — Dimensions, chamfers and tolerances of test pieces for the ring-on-ring test

The peripheral surface of the test piece shall be free of visible larger sized outbursts. If this is not the case, the edges shall be chamfered with a thickness ≤ 0,3 mm and a chamfer angle of about 45° or rounded with a radius of ≤ 0,3 mm.

NOTE If the edges are not chamfered or rounded as described above, there is a risk that the test pieces break at the test piece edges and therefore an irregular fracture will occur.

If necessary, and in accordance with the agreement between the parties, test pieces may be used with other dimensions in combination with adapted dimensions of the test jig. But in these cases, conditions (I) to (III) (see 7.3) shall be fulfilled (see ASTM C1499). The deviations of the test piece dimensions shall be recorded in the test report.

The test piece thickness shall conform to the conditions of Formula (1).

$$\frac{d_2}{10} \geq H \geq \left(\frac{2 \times \sigma_b \times d_2^2}{3E} \right)^{0,5} \tag{1}$$

where

d_2 is the contact diameter of the support ring, in millimetres (mm);

H is the test piece thickness, in millimetres (mm);

E is the Young's modulus of test piece, in newtons per square millimetre (N/mm^2);

σ_b is the expected equibiaxial flexural strength, in newtons per square millimetre (N/mm^2).

The diameter, D, and the thickness, H, of the test piece, as well as the contact diameter of the support ring, d_2, shall conform to the conditions of Formula (2).

$$2 \leq \frac{D - d_2}{H} \leq 12 \tag{2}$$

If other dimensions of the test piece or test jig are used, the ratio of the contact diameter of the loading ring, d_1, to the contact diameter of the support ring, d_2, shall conform to the conditions of Formula (3).

$$0,2 \leq \frac{d_1}{d_2} \leq 0,5 \tag{3}$$

7.3 Surface finish

7.3.1 General

This document permits any relevant surface finish described in the following three categories:

— I: as-fired;

— II: grinding/lapping/polishing;

— III: agreed procedure.

The aim of the procedure according to category II is to minimize the damage caused to the test pieces due to the preparation method in order to examine strength more representative of the material than the machining. The recommended procedures cannot be standardized in the conventional sense, because of variation between grinding machines and the different behaviours during grinding of different types of material. The procedures described should thus be treated as guidelines. Furthermore, since there are no methods for accurately measuring surface finish parameters of ceramic materials, special attention should be paid to the quality of the machining.

7.3.2 Surface finishing I: as-fired

The test pieces to be tested in the as-fired (ex-kiln) condition without further surface preparation provided shall have dimensions according to 7.2.

The machining of one side of the test piece is allowed in sintered condition (e.g. to achieve the test piece dimensions and tolerances). This machined side shall be the surface which is stressed in compression during the ring-on-ring test, i.e. that surface which is in contact with the loading ring.

Chamfering or rounding of the edges which is eventually necessary to remove great outburst is allowed either before or after the sintering process (see 7.2).

If the test piece dimensions with their tolerances are not observed, reject the test pieces concerned.

7.3.3 Surface finishing II: grinding/lapping/polishing

The test piece side subjected to tension stress during the strength test shall be grinded and, if necessary, lapped or polished. The final step of machining shall be independent of direction, i.e. parallel grinding is not allowed. The defects caused by the machining step before shall be removed completely during the final machining. During the lapping and polishing processes use abrasives (bound or loose abrasive grains) of less than 30 µm grit size, progressively reducing in size. During this operation a thickness of at least 50 µm shall be removed from the test surface machined in order to remove pre-existing damage from rough grinding or cutting operations. At least 5 µm shall be removed using abrasives of less than 6 µm grit size.

Chamfering or rounding of the edges which is eventually necessary to remove great outburst is allowed either before or after the sintering process (see 7.2).

Any deviation from the procedure described above shall be agreed between the parties and recorded in the test report.

7.3.4 Surface finish III: finishing by agreed procedure

The test pieces shall be prepared by an agreed procedure between the parties. The preparation of the test pieces shall be followed by conformance to an agreed sequence of abrasives, preparation direction and material removing rates. The test pieces may be annealed or refired after machining. Details of the test piece preparation shall be described or cited in the test report.

NOTE The use of a fine abrasive for a finishing process sometimes cannot completely remove the effects of previous coarser grinding stages. Caution is advised in the interpretation of strength data related to finishing processes.

7.3.5 Check of dimensions

The diameter and the thickness of the test pieces shall be measured to a relative resolution of ≤ 0,1 % (see 6.3) using a suitable length measuring device. The results shall be recorded in the test report.

Measure the diameter in at least three positions distributed equally over the test piece perimeter to three significant numbers and calculate the mean diameter.

Measure the test piece thickness in at least three positions distributed equally over the test piece front surfaces to four significant numbers and calculate the mean thickness. These three measuring values shall not deviate more than 1 % from the mean thickness of the test piece.

NOTE 1 An additional measurement of the parallelism of the test piece surfaces is usually not necessary when using this measuring procedure.

NOTE 2 Measuring the chamfers or rounding of the test piece front surfaces is not necessary because the chamfers and front surfaces do not essentially influence the strength calculations. If there are bad edges, there is only the risk of irregular fracture and then these test pieces are eliminated and not used for the strength calculation.

If test piece dimensions other than those given in Table 1 are used, adopted tolerances shall be fulfilled, namely for test piece thickness ≤ 5 % of the mean test piece thickness, for test piece diameter ≤ 1,5 % of the mean test piece diameter and for parallelism ≤ 1 % of the mean test piece thickness.

The dimensional measurements shall be performed with caution so that damage to the test pieces is avoided.

7.3.6 General requirements

The test pieces shall be cleaned after any machining process using water or an appropriate solvent and shall be dry during the strength test.

Whichever surface finishing routine is applied, full details of the machining procedure shall be described in the test report or the corresponding standard operation procedure (SOP) shall be cited.

7.3.7 Number of test pieces

The minimum number of test pieces shall be 10 for material development, material characterization or quality control. For statistical evaluation of strength data (e.g. Weibull parameters) in accordance with ISO 20501, the minimum number shall be 30.

NOTE Weibull parameters can have a high confidence interval if the number of nominal identical test pieces is considerably less than 30. Then comparisons between batches of a material or between different materials are meaningless because of the uncertainty of the parameters. It is possible, however, to use the common statistical significance or equivalent tests.

7.4 Precautions

The test pieces prepared shall be handled with care to avoid causing damage subsequent to the machining process.

Test pieces finally prepared shall be kept separate at all times and be individually wrapped for transport.

8 Procedure

Record the room temperature of the test environment to the nearest 1 °C and the RH to the nearest 5 % RH (see 6.4). The RH shall not change by more than 10 % during the strength test.

NOTE 1 If massive solar radiation or ventilation is avoided, the above mentioned conditions are usually fulfilled.

Choose the load measuring range on the testing machine (6.1) such that the expected force at fracture is not below the certified measuring range.

Ensure that the test jig is cleaned of fracture debris from previous tests and that the support and the loading rings of the test jig are free from defects (e.g. small burrs and dents).

Position the test piece in turn in the test jig such that the surface of the test piece subjected to the tension stress is in contact with the rubber film (support ring side, see Figure 1 and 6.2). The rubber ring or film is needed to compensate possibly existing unevenness of the test piece surface or of the support or loading rings.

The test piece, support ring and loading ring shall be positioned centrically to each another (see 6.2) in the test jig.

Place a protective screen (e.g. cloth or acrylic glass) around the test piece to trap the fractured fragments for safety reasons and for subsequent examination.

Select a loading rate such that fracture is obtained in a time period of 5 s to 15 s.

NOTE 2 Selection of loading rate (applied by controlled displacement or force) can be determined by experiment depending on the elastic compliance of the test machine, the stiffness of the test jig and the elastic properties of the test piece.

NOTE 3 The strength of advanced monolithic technical ceramics can be markedly affected by the loading rate. The short time to failure required by this method is intended as a compromise between minimising the effect of the loading rate and the ability of test machines and load measuring range to provide an exact measure of maximum force at fracture.

Apply the selected loading rate and record the maximum force supported by the test piece at the instant of failure.

Retrieve and identify the fracture fragments for later examination.

NOTE 4 For further information on the fracture patterns that can be expected at different fracture energy levels and means to identify the fragments containing fracture origins, see CEN/TS 843-6, ASTM C1322, Reference [2]. and Annex A

Examine the fracture fragments for features of the primary fracture origins.

9 Calculations

Calculate the nominal equibiaxial flexural strength, σ_{rr}, of each test piece using Formula (4) (see also ISO 6474-1 and Reference [1]).

$$\sigma_{rr} = \frac{3 \times F}{2 \times \pi \times H^2} \times \left[(1+\nu) \times \ln\left(\frac{d_2}{d_1}\right) + (1-\nu) \times \left(\frac{d_2^2 - d_1^2}{2 \times D^2}\right) \right] \quad (4)$$

where

σ_{rr} is the equibiaxial flexure strength, in newtons per square millimetre or megapascals (N/mm^2 or MPa);

F is the maximum force at fracture, in newtons (N);

d_1 is the contact diameter of the loading ring, in millimetres (mm);

d_2 is the contact diameter of the support ring, in millimetres (mm);

D is the test piece diameter, in millimetres (mm);

H is the test piece thickness, in millimetres (mm);

ν is the Poisson ratio of the material tested (non-dimensional).

Calculate the mean value and the standard deviation from the fracture strength of the test pieces regularly fractured during the ring-on-ring test.

NOTE It is known that Formula (4) does not necessarily give the true fracture stress at the point of failure but a nominal maximum stress caused by the most highly stressed region in the test piece. For some purposes, such as the statistical evaluation of results, it can be necessary to correct the formula regarding the position of failure (distance inside the test piece from the surface under tensile stress).

If the number of test pieces in a batch is sufficiently high, for example 30 or greater, the statistical analysis should be performed according to the methods defined in ISO 20501.

If the primary fracture origin is more than 0,5 mm outside the contact diameter of the loading ring, then this fact shall be stated in the test report. In this case, however, it shall be assumed for the calculation of the fracture strength that the fracture origin lies inside the contact diameter of the loading ring.

The results of such tests shall be considered when calculating the mean value of equibiaxial flexural strength by the ring-on-ring test.

For a more exact fracture analysis, the maximum stress at the moment of fracture may be corrected for the real position of the primary fracture origin.

10 Test report

The test report shall be in accordance with ISO/IEC 17025 and shall contain the following information:

a) the name and address of the testing establishment;

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

c) a reference to this document, i.e. ISO 17167;

d) a description of the test jig geometry used and, if appropriate, deviations from the standard test jig or the standard test piece dimensions;

e) a description of the test material (material type, manufacturing code, batch number, date of receipt);

f) the preparation method of the test pieces from the material supplied;

g) the category of the surface finish according to the preparation method, i.e. I, II or III;

h) the preparation method of the test piece surface, including details of any test piece pre-treatments;

i) the average ambient test temperature and average RH during the tests;

j) the number of test pieces tested;

k) the diameter and thickness of the test pieces and maximum force at the moment of failure for each test piece;

l) individual nominal equibiaxial flexure strength values for each test piece tested given to three significant figures, including the values of those test pieces that have failed substantially outside the loading ring diameter;

m) unless otherwise agreed, the mean nominal equibiaxial flexure strength and the standard deviation;

n) remarks about the test and the test results, details of any necessary deviations from this document and any incidents.

