BRITISH STANDARD

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Advanced technical ceramics — Methods of testing monolithic ceramics — Thermomechanical properties —

Part 3: Determination of resistance to thermal shock by water quenching

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ICS 81.060.30

National foreword

This British Standard is the official English language version of EN 820-3:2004. It supersedes DD ENV 820-3:1994 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee RPI/13, Advanced technical ceramics, which has the responsibility to:

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Foreword

This document (EN 820-3:2004) has been prepared by Technical Committee CEN/TC 184 “Advanced technical ceramics”, the secretariat of which is held by BSI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by February 2005, and conflicting national standards shall be withdrawn at the latest by February 2005.

This document supersedes ENV 820-3:1993.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

EN 820 *Advanced technical ceramics — Methods of testing monolithic ceramics — Thermomechanical properties* consists of five Parts:

— Part 1: *Determination of flexural strength at elevated temperatures*

— Part 2: *Determination of self-loaded deformation*

— Part 3: *Determination of resistance to thermal shock by water quenching*

— Part 4: *Determination of flexural creep deformation at elevated temperatures*

— Part 5: *Determination of elastic moduli at elevated temperatures*

Part 4 is a European Prestandard (ENV) and Part 5 is a Technical Specification (CEN/TS).

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1 Scope

This Part of EN 820 specifies the principles of thermal shock testing, and provides a general method for conducting thermal shock tests by quenching into water for both test pieces and components by quenching into water.

NOTE This document does not cover thermal stress developed as a result of steady inhomogeneous temperature within a ceramic body or of thermal expansion mismatch between joined bodies.

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.

EN 623-1, *Advanced technical ceramics — Monolithic ceramics — General and textural properties — Part 1: Determination of the presence of defects by dye penetration tests*

EN 843-1, *Advanced technical ceramics — Monolithic ceramics — Mechanical properties at room temperatures — Part 1: Determination of flexural strength*

EN 60584-1, *Thermocouples — Part 1: Reference tables (IEC 60584-1:1995)*

EN 60584-2, *Thermocouples — Part 2: Tolerances (IEC 60584-2:1982)*

EN 60672-2, Ceramic and glass insulating materials — Part 2: Methods of test (IEC 60672-2:1999)

EN ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories (ISO/IEC 17025:1999)*

3 Principle

A set of test pieces is heated to a given temperature, and then quickly and smoothly transferred to a water bath. The test pieces or components are then inspected for cracks or other damage, either by an appropriate mechanical test to establish whether weakening has occurred, or by using a dye penetrant to detect the presence of cracks (see EN 623-1).

NOTE 1 Dye penetration tests are unsatisfactory for porous or highly microcracked materials.

This thermal shock test determines whether a material or component has a capability of withstanding a water quench through a large temperature difference from high temperature without failure, under the conditions of heat transfer prevailing in such a quenching environment, and for the given geometry and section thickness.

NOTE 2 By agreement between parties an alternative quenching medium may be employed. Details of the medium employed should be incorporated in the report.

If the test pieces for the quench test are available as regular bar shapes, then the inspection after quenching with the mechanical test, such as a flexural test, may be preferred, as it enables the onset of loss of strength with increasing initial temperature to be determined. Sets of at least five test pieces are heated to a series of temperatures above that of the quenching bath, quenched, dried and subjected to a short-term strength test. The temperature drop corresponding to that at which a sudden loss of strength occurs is termed the critical temperature difference, ΔT_c. This temperature difference can be estimated using the first kind of thermal shock parameter, R (see A.3.2), to which it is numerically equal at an infinite rate of heat transfer.

It should be noted that although the flexural strength test method for monolithic ceramics given in EN 843-1 may be employed for testing resistance to thermal shock, because of the small size of the specified test piece an overestimate of the material capability in larger sizes would occur. Larger rod or bar-shaped test pieces specially prepared for the test should be employed if the behaviour of larger sections of material or components is to be assessed, e.g. type A test pieces as described in EN 60672-2. As a general rule, thermal shock test results are more or less independent of test piece diameter when this exceeds about 10 mm.

4 Apparatus

The apparatus shall consist of:

a) temperature-controlled oven capable of maintaining a set of test pieces at a given temperature ± 5 °C;

b) suitable test piece holder capable of being transferred rapidly from the oven to the quenching medium within 0,5 s;

c) water bath controlled at 20 °C ± 2 °C and of sufficient volume that the net temperature rise after quenching the test pieces is less than 5 °C. There shall be a grid near the bottom of the water bath to prevent hot test pieces from resting directly on the bottom.

The temperature of the test pieces in the oven shall be recorded by use of a suitable thermocouple manufactured in accordance with the manufacturing tolerances stated in EN 60584-2, allowing the use of the reference tables in EN 60584-1 or, alternatively, calibrated in a manner traceable to the International Temperature Scale ITS-90.

NOTE The test piece holder may contain several test pieces. Alternatively, test pieces may be lowered or dropped individually from the oven into the quenching medium in accordance with individual circumstances. Care should be taken that no mechanical damage occurs to the test pieces as a result of transfer to the cold bath.

Where a flexural strength test is used, the test jig employed for rod or bar-shaped test pieces and the calculations of strength shall conform to the principles given in EN 843-1.

5 Test pieces

Test pieces shall either be specially prepared as rods or bars, or may be in the form of complete components where appropriate.

Type A: Rod or bar-shaped test pieces either as-fired or with a specified surface finish in accordance with the requirements of the thermal shock test in EN 60672-2.

Rod-shaped test pieces shall be (10 ± 1) mm diameter, uniform to within ± 0,1 mm, and of length at least 120 mm.

Bar-shaped test pieces shall be (10 ± 1) mm x (10 ± 1) mm, uniform to within ± 0,1 mm, and of length at least 120 mm. The edges shall be chamfered.

Type B: Bar-shaped test pieces either as-fired or with a specified surface finish in accordance with the requirements of EN 843-1, size B.

The dimensions shall be (3 ± 0,2) mm x (4 ± 0,2) mm x at least 45 mm. The edges shall be chamfered.

Type C: Complete components in appropriate finished condition.

Other sizes and shapes of test-piece are permitted subject to agreement between parties. Full details of shape and dimensions shall be recorded in the report.

NOTE 1 Test pieces in accordance with Type A will produce results which are applicable to, and give a ranking of materials performance appropriate for, larger components. Test pieces in accordance with Type B will require rather higher quenching temperature differences in order to induce failure. Materials comparisons using this method may be valid for small components of comparable size, but it is possible that it will not correctly rank materials performance for larger or smaller cross-sections of components. Test pieces in accordance with Type C give results which are representative of severe quenching shock for that size, shape and manufacturing method for a specific component.

NOTE 2 The ends of rod or bar test-pieces may be more prone to initiate failure than the central regions. Care should be exercised over the quality of finish on the ends of bars, which should be of equivalent form and dimensions for a valid materials comparison.

NOTE 3 The edges of square or rectangular bars are more prone to initiate failure than the flat or curved surfaces. Chamfering of test-bar edges is critical, and the same size chamfers should be used on all bars for a valid comparison of materials.

If a dye penetration method of crack detection is to be employed, at least 18 test-pieces shall be prepared, permitting three test pieces to be used at each of five test temperatures, plus three as an unshocked control. If a strength test method of damage detection is to be employed, at least 30 test-pieces shall be prepared, permitting five test pieces to be used at each of five test temperatures, plus five as an unshocked control.

6 Procedure

Clean and dry the test-pieces at (120 ± 10) °C for 2 h in an oven. Allow to equilibrate at room temperature before testing.

If a dye penetration test is to be employed, conduct the test on three test pieces selected at random from the prepared batch in accordance with EN 623-1. Inspect for the presence of any damage or cracks.

NOTE This test is inappropriate if the material is found to be significantly porous or contains cracks in the as-received condition. Thermal shock damage in such cases can be reliably assessed only through the use of strength test.

If a strength test is to be employed, fracture five control test pieces selected at random from the batch, in accordance with EN 60672-2 (Type A test-pieces) or EN 843-1 (Type B test pieces) using either three or preferably four-point bending. Calculate the individual and mean strengths.

Place three (for the dye test) or five (for the strength test) test pieces or components in the oven and heat them slowly to a temperature near to that which is expected to induce failure on quenching. After a period of at least 10 min for stabilization of temperature of the test pieces or components, record the test temperature. Transfer the test pieces smoothly and quickly to the water bath. After a minimum period of two minutes, remove the test-pieces or components from the bath and dry them at (120 ± 10) °C for at least 2 h, cool and allow them to equilibrate at room temperature. If the dye test is to be used, subject them to the test in accordance with EN 623-1 and examine them for the development of surface cracks. If the strength test is to be performed, fracture the test pieces in accordance with EN 60672-2 or EN 843-1 using the same procedure adopted for the unshocked controls. Calculate the individual and mean strengths at this quenching temperature difference.

If cracking and/or a reduction in strength has occurred from the level obtained without thermal shock, select a lower temperature and repeat the test with fresh test pieces. If no cracking or no reduction in strength has occurred, select a higher oven temperature and repeat the test with fresh test pieces. Continue this process until the oven temperature can be determined at which fracture is just initiated, or at which there is a first drop in mean strength by more than 30 % of the initial mean strength (see Figure 1). This temperature is the critical temperature, and the difference in temperature between this and the bath is the critical quenching temperature difference. If the material or component does not show a

sharp decrease in strength at a critical quenching temperature difference, but rather a steady decline in strength with increasing quenching temperature difference, a critical value cannot be reported.

For a material of characteristics which are known approximately, this procedure can be used to locate the critical quenching temperature difference by a series of temperature increments or decrements reducing in size. It is suggested that the process should start with 50 °C or smaller increments or decrements. For a material with unknown characteristics, it may be necessary to commence with larger increments or decrements.

If the strength test has been used, plot a graph of mean fracture load or stress against quenching temperature interval.

By agreement between parties, this procedure may also be used where appropriate as a pass/fail test for given quenching conditions.

7 Test report

The test report shall be in accordance with EN ISO/IEC 17025, and shall include the following:

a) name of the testing establishment;

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

c) reference to this test method, i.e. to EN 820-3; whether the test was using dye penetration or mechanical testing; whether there were any agreed departures from the method in accordance with agreement between parties, such as the use of a quenching liquid other than water;

d) description of the test apparatus used; if strength testing is used, the method adopted using either type A or type B test-pieces;

e) details of the test procedure employed including the sequence of oven temperatures;

f) details of the test material type or components, manufacturing code, batch no., etc.;

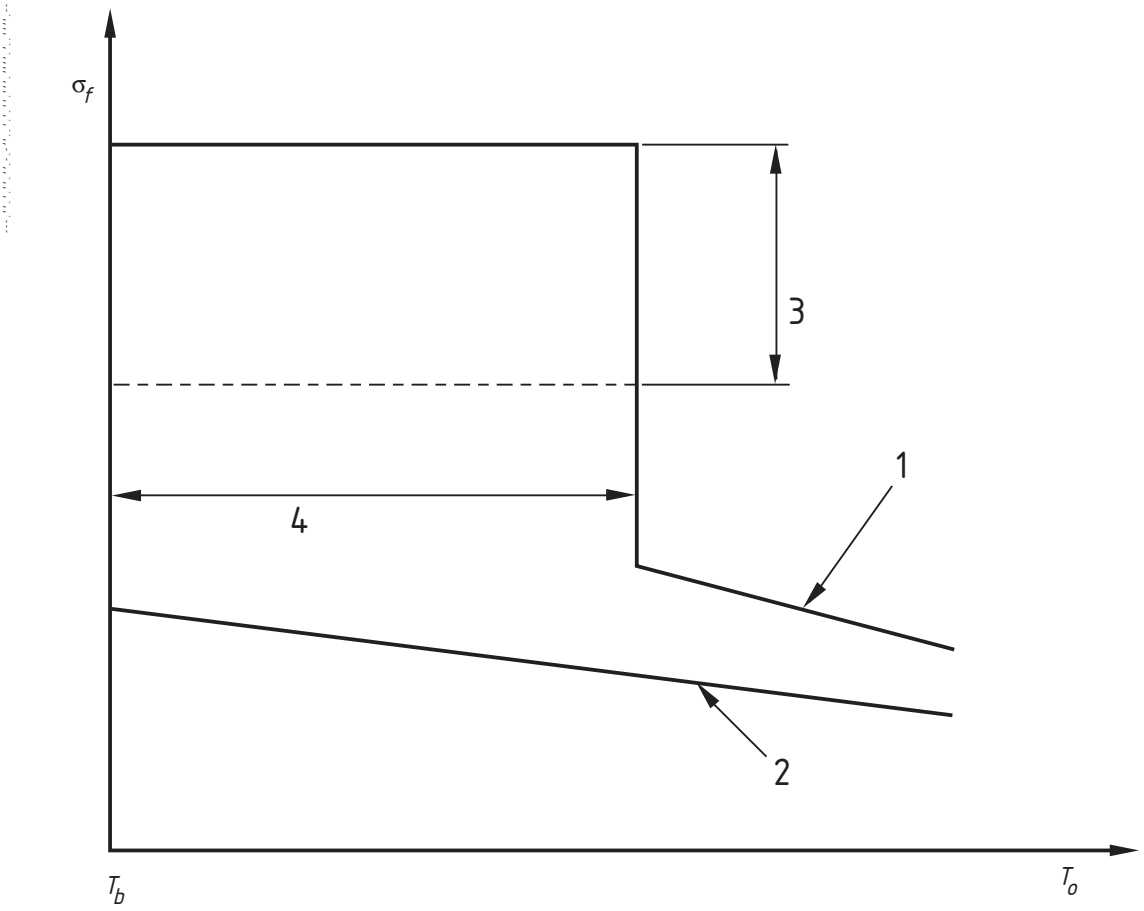
g) dimensions of test pieces or components and details of the surface preparation of the test pieces if appropriate;

h) individual results for each test piece or component at each quenching temperature interval, i.e. whether cracking was detected in the dye test or strength results in a strength test;

i) estimate of the critical quenching temperature interval, expressed in degrees C, where this is feasible, determined as the temperature at which one of the three dye-tested test pieces shows cracking, or where the mean strength is reduced by at least 30 % compared with the unshocked strength;

j) if a strength test was used, a plot of strength after quenching against quenching temperature interval;

k) any deviations from the procedure described in this document, and any comments on the test or test results.



Key

1 Material showing sharp drop in strength
2 Material showing slow decline in strength
3 30 % loss in strength
4 Critical temperature difference
σ_f Flexural strength
T_b Temperature of bath
T_o Temperature of oven

Figure 1 — Schematic diagram of the typical changes in flexural strength of ceramic test pieces after quenching from various temperatures for materials which show a steady decline in strength, and for materials which show a sharp decline at a critical temperature

Annex A
(informative)

Introduction to thermal shock behaviour

A.1 Causes of thermal stress failure

Because ceramics are non-ductile elastic bodies, thermal stresses derived from temperature gradients through the thickness of a test piece or component cannot be readily relaxed, as for example in ductile metals. If the tensile or shear thermal stress level exceeds the strength of the material, then cracks can develop which degrade the strength of the material or, in extreme circumstances can cause complete fragmentation.

The factors that influence the ability of a ceramic component to withstand degradation of its functional properties by thermal stress are several:

a) geometry of the component, especially the wall thickness and the radii of curvature of exposed corners and faces, which control the rate of heat transfer to the component;

b) thermal conductivity and/or thermal diffusivity of the material comprising the component, which controls the thermal gradients which are set up by the transfer of heat to or from the component;

c) thermal expansion characteristics, which control the levels of thermal strain developed under the temperature gradients established in the component;

d) elastic properties, which control the levels of stress developed by thermal strains;

e) strength of the material at the locations where the tensile or shear thermal stresses develop to high levels, and the density of the defects which are present as crack initiating sites;

f) fracture toughness of the material, which controls the resistance to the propagation of cracks once fracture is initiated;

g) amount and distribution of porosity, which controls the resistance to thermal shock damage through reducing the elastic moduli;

h) surface condition, i.e. roughness, emissivity.

These factors combine to control the survivability of a component in any given environment in which temperature changes rapidly.

A.2 Mechanisms of failure

Creating a thermal gradient in a ceramic body introduces a distribution of thermal strains which cannot exceed the failure strain of the component material at any point if the component is to survive undamaged. If the failure strain is exceeded, then cracks may develop and grow to relieve the strain, and these will tend to initiate and propagate in regions where the thermal strains are in greatest tension or shear. Thermal gradients may arise in practice due to steady localized application of heat to a component, or to the sudden immersion of a component into a hotter or cooler environment.

A hot ceramic component subjected to sudden chilling of its surface by immersion in a cooler medium has its surface layer subjected to a high tensile stress which is transient in nature. If this stress is

sufficiently high, cracks can develop from surface defects or other fracture initiating sites. Typically, excessive rates of cooling lead to the crazing of the surface by a network of meandering cracks. In a material with a low level of toughness, the cracks may run sufficiently to cause complete fragmentation of the component. In a material with a moderate level of toughness, the cracks may halt, and result in a weaker, but still intact component. In a coarse-grained or porous weak material, the cracking may be minor, and lead to very little overall degradation. Figure 1 illustrates typical behaviour.

A cold ceramic component subjected to sudden heating by immersion in a hotter medium has a thermal stress distribution which is compressive on its surface, but tensile internally. Generally speaking, the tensile stress levels are lower than in the equivalent cooling situation, and thus components tend to be able to survive greater upward thermal shocks than downward ones. Another mode of failure is in shear, close to the surface under high compressive stress, and this results in the production of spalls or flakes which come away from the surface. Similarly, a cold ceramic subjected to localised heating, for example by infrared radiation, a gas flame or a laser, will suffer tensile stresses on the opposite side and around the periphery of the heated zone, with similar results.

Repeated thermal shock cycling can result in the progressive accumulation of mechanical damage if the shock is sufficiently severe.

A.3 Thermal shock parameters

A.3.1 The extent of damage is controlled by a number of physical parameters as summarized in A.1, but especially the thermal expansion coefficient, the toughness and the strength. Resistance to propagation of damage in repeated cycling is controlled principally by the density of cracking and the toughness. To describe the relative performance of different ceramic materials, it is common to find *thermal shock parameters* being employed. The following have been devised from models of downward thermal shock of non-ductile elastic solids and are given in A.3.2 to A.3.5 as examples to illustrate the roles played by various mechanical and thermal properties of the material.

A.3.2 For very rapid thermal shock (instantaneous change of surface temperature):

$$R\frac{\sigma_f(1-\nu)}{E.\alpha} \qquad \text{(A.1)}$$

where

R = thermal shock parameter of first type;

σ_f = fracture strength (typically biaxial strength);

ν = Poisson's ratio (typically 0,25);

E = Young's modulus;

α = thermal expansion coefficient.

A.3.3 For constant rate of heat transfer between the component to the medium:

$$R'\frac{\sigma_f(1-\nu)\lambda}{E.\alpha} \qquad \text{(A.2)}$$

where

R' = thermal shock parameter of second type;

λ = thermal conductivity.

A.3.4 For constant rate of change of surface temperature:

$$R'' = \frac{\sigma_f \ (1 - \nu) a}{E \ . \ \alpha} \qquad \text{(A.3)}$$

where

R'' = thermal shock parameter of third type;

a = thermal diffusivity.

A.3.5 For resistance to loss of strength on thermal cracking:

$$R''' = \frac{E\gamma_f}{\sigma_f^2(1-\nu)} \qquad \text{(A.4)}$$

where

R'' = thermal shock parameter of fifth kind;

γ_f = fracture energy.

A.3.6 It is essential that such parameters, of which the above are the most commonly used for materials intercomparison, are used with caution. In particular, the appropriate property data over the relevant temperature range shall be used to calculate the parameters. The parameters have some value for comparing the potential performance of candidate materials under prescribed conditions. However, their indiscriminate use can give misleading results, especially if the heat transfer coefficient seen in the application for the component is not adequately modelled by the thermal shock parameter chosen. Thus, R' represents a situation of relatively slow heat transfer, where high thermal conductivity is advantageous, while R represents situations of extremely rapid thermal shock, where unless the thermal conductivity is very high, materials are ranked broadly according to thermal expansion coefficient, the lowest values being the most advantageous.

A.4 Performance assessment of components

Because of the complexity of assessing the risks of thermal stress or thermal shock behaviour in ceramics, and also because of the difficulty in accurate prediction of heat transfer conditions, it is often necessary to resort to a pragmatic approach, testing the component under service or simulated service conditions. It is recommended that this process be adopted as it may be more meaningful in the majority of circumstances where conditions are not readily quantifiable.

A conventional method of assessing the relative performance of different materials for severe thermal quenching is to subject them to a water quench test, which is the principle of the method described in Clauses 3 to 7.

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