
Sodobna tehnična keramika - Monolitna keramika – 1. del: Splošni postopki za izvajanje korozijskih preskusov

Advanced technical ceramics - Monolithic ceramics - Part 1: General practice for undertaking corrosion tests

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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Management Centre: rue de Stassart, 36 B-1050 Brussels

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Foreword

This document (prEN 12923-1:2004) has been prepared by Technical Committee CEN/TC 184 “Advanced technical ceramics”, the secretariat of which is held by BSI.

This document is currently submitted to the CEN Enquiry.

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1 Scope

This Part of EN 12923 describes general procedures to be employed when undertaking corrosion tests on advanced technical ceramics. The mechanisms of chemical attack on advanced ceramics are widely varied, and depend on the chemical and phase composition and the phase morphology of the material, as well as the corrosive conditions imposed. For any particular engineering application it is usually necessary to model expected conditions of use in order to obtain quantitative data on ability to withstand the proposed end-use conditions.

This standard is not restricted to specific material types, nor does it prescribe particular test conditions or test period. The actual testing requirements may be very specific, in order to investigate for example the suitability of a range of materials for a given application in which certain specified conditions occur. This standard provides methods for undertaking the assessment of the effect of corrosion, and provides guidance on practical issues related to undertaking the tests.

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 623-4, *Advanced technical ceramics — Monolithic ceramics. General and textural properties — Part 4: Determination of surface roughness*

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

EN 843-4, *Advanced technical ceramics — Monolithic ceramics. Mechanical properties at room temperature — Part 4: Determination of hardness*

ENV 1006, *Advanced technical ceramics — Methods of testing monolithic ceramics — Guidance on the sampling and selection of test pieces*

ENV 12923-2, *Advanced technical ceramics — Monolithic ceramics — Part 2: General oxidation test*

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

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

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

ISO 3611, *Micrometer callipers for external measurement*

ISO 6906, *Vernier callipers for external measurement*

3 Definitions

For the purposes of this standard the following definitions apply:

3.1**corrosion**

process of degradation induced by chemical attack by a surrounding medium on a ceramic body

3.2**oxidation**

process of reaction of a ceramic material with oxygen in the surrounding atmosphere, including any internal reactions as a result of the presence of open porosity or of diffusion of ions to or from the ceramic surface

4 Significance and use

Advanced technical ceramic materials are widely regarded as being generally resistant to corrosion, and many types find applications in highly corrosive conditions where other materials are not viable. However, this is not always the case, and the selection of the most appropriate material requires some form of assessment to provide assurance that it has adequate resistance to the conditions to which it is to be exposed.

The rate of chemical attack is determined by:

1. The chemical nature, phase composition, phase distribution and degree of continuous porosity in the material;
2. The temperature, pressure, composition, concentration and flow rate of the corroding medium, and whether these are constant or vary with time;
3. The mechanical forces applied to the material in terms of internal stress condition and the degree of surface abrasion or wear due to contact with other surfaces, the presence of abrasive particles or the dissolving effect of the corroding medium itself.
4. The period for which the test is performed, because it cannot be assumed that the rate of attack is constant with time. Adjusting test conditions to accelerate the corrosion process and extrapolating corrosion rates to times longer than that of the test should not be done.

These factors need to be carefully selected, clearly specified and reported in any corrosion test.

The undertaking of corrosion tests is normally for two principal purposes:

1. To simulate performance in an application, which will require careful consideration of all factors pertaining to the conditions under which corrosion is occurring, and which will require these to be modelling in the testing environment
2. To provide a comparative measure of performance of a range of materials under defined corrosion conditions

This standard provides a basis for undertaking corrosion tests and details the criteria which might be considered for determining whether attack has taken place and has significant consequences for subsequent use of a material. Since corrosion is dependent on a wide range of parameters, this standard does not prescribe particular environments or durations of attack, but provides guidance on the key factors that need to be considered or specified in undertaking tests and gives a general framework for conducting tests in a meaningful manner.

5 Criteria for determining corrosive attack

Chemical attack is manifest in a number of ways:

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1. change of dry mass;
2. change of section thickness;
3. change of colour;
4. penetration of corrodent into the material rendering the surface open porous;
5. development of surface skins of altered composition;
6. development of a surface skin of reaction product;
7. change of surface finish;
8. change in strength;
9. change of hardness or wear resistance.

Furthermore, attack may not be linear with time, notably if the diffusion path for corroding species increases with increasing corrosion.

The extent of chemical attack may be ascertained using a number of simple criteria, the most important being:

- A. depth of penetration of corrodent;
- B. change of mass;
- C. change of cross-section;
- D. change of strength;
- E. change of surface roughness;
- F. change of hardness;
- G. changes in the chemical composition of the corroding medium.

NOTE Other situations may exist in which it is desirable to record changes in other properties, for example, thermal conductivity and thermal shock resistance, either whilst immersed in the corroding medium, or after extraction from it. Such methods are not specifically included in this standard, and may require special equipment not covered by this standard. Change of colour may be subjective.

Table 1 summarises the areas of validity of using these criteria for various types of corrosive attack. These criteria apply to passive conditions of corrosion, i.e. not when the component is under an externally applied stress.

Table 1 — Appropriateness of methods of assessing corrosive attack

Test type	Aqueous based corrosion	Corrosion by melts	Corrosion by gases and vapours
A. Penetration	Appropriate	May be appropriate	Appropriate in some cases
B. Change of mass	Appropriate, but sometimes inadequate	Inappropriate	Appropriate if non-slugging
C. Change of cross-section	Not always appropriate when change is small	Appropriate	Appropriate
D. Change of strength	Appropriate	May be appropriate if adherent material is removed	Appropriate if non-slugging; may be appropriate if adherent material is removed.
E. Change of surface roughness	Appropriate for slight corrosion	Usually inappropriate	Usually inappropriate
F. Change of hardness	Appropriate	Usually inappropriate	May be appropriate if non-slugging.
G. Change of corrodent composition	Appropriate	Usually inappropriate	Appropriate

6 Interferences

6.1 Test type A: Depth of penetration

6.1.1 For materials subjected to corrodents which result in grain boundary attack and penetration of the corrodent, the depth of penetration may be strongly influenced by the microstructural nature of the original test piece surface. Some as-fired surfaces may have better resistance to penetration than bulk microstructures exposed by machining test-pieces. When possible, testing should avoid as-fired surfaces unless the testing is specifically to evaluate the performance of such surfaces.

6.1.2 The depth of penetration may be variable if the microstructure is inhomogeneous. The greatest depth of penetration is to be recorded.

6.1.3 In materials with substantial pre-existing closed porosity, it may be difficult to determine the true depth of penetration if there is no major change in the appearance of the microstructure. Reliance must be placed on the use of penetrant dyes (see EN 623-1).

6.2 Test type B: Mass change

6.2.1 Accurate determination of change in dry mass may not properly reflect the extent of attack as a result of either the adherence of solid surface deposits or the retention of corrosion products within porosity generated by corrodent penetration. A change in mass therefore indicates that some attack

may have occurred, but other criteria need to be employed to quantify its overall effect on performance.

6.2.2 Assuming that the process of mass gain or mass loss is attributable to the test-piece behaviour, e.g. in an oxidation test (see ENV 12923-2), accurate determination of change in dry mass relies on being able to minimise contact between the test piece and the apparatus in which it is housed for the duration of the corrosion test. If strong reaction occurs between the test piece surface and its support, a significant error in registered mass change may result.

NOTE It may be necessary to perform some preliminary tests to ensure that the supporting material is sufficiently non-reactive with the test material before conducting the required tests. In some atmospheres, for example in low oxygen content atmospheres at high temperatures, the evolution of volatile species from one test-piece can interact with or contaminate adjacent test-pieces. Cases of this type necessitate separately exposing each test-piece to the corroding medium.

6.2.3 Free circulation of corrodent around the test-piece is required. If this is for any reason significantly reduced by the geometrical arrangement of test-pieces and supporting apparatus, rates of corrosion may be reduced as the corroding species become consumed. A very high velocity of corrodent can change the mechanism of corrosion from that expected under low flow rate, and therefore should be avoided unless the high flow rate is a specific requirement of the test.

6.2.4 For very low levels of mass change, the potential error of measurement is large. It may be more appropriate to monitor changes in chemical concentration of dissolved materials in the corrodent medium. In this case an alternative method of assessment should be considered, for example, measurement of change in strength.

6.2.5 In cases where material is lost due to spallation, it may be useful to incorporate a plate or tray under the test material to catch debris for later analysis. Caution must be exercised such that the tray does not increase the contact area with the test-piece or restrict the flow of the corroding medium.

6.3 Test type C: Change of test-piece size

Measurement of true test-piece dimensions after corrosion may become uncertain due to the formation of rough layers, pitting, loose scale or loss of scale. The measurement can be approached in two ways:

1. Measurement of overall external dimensions with any adherent surface layer. The uncertainties associated with this method can be large if the corrosion product has non-uniform thickness or partial loss of scale. This method is suited primarily to cases where no scale develops, or where the corrosion product layers are compact, uniform in thickness and adherent to the test material.
2. Measurement of section thickness unaffected by corrosive attack. This method involves sectioning and polishing which may change the morphologies of corrosion products and introduce artifacts. Considerable care should be exercised in test-piece preparation. Provided that sufficient measurements are made, typically at least 10, using a travelling microscope for example, it should be possible to provide a sound statistical assessment of change in cross-section. This method is appropriate to situations where corrosion has caused a loss of material from one surface exceeding 5 µm.

6.4 Test type D: Strength change

The close specification in EN 843-1 for flexural strength testing designed to achieve results with minimal errors may not be achievable on test-pieces after corrosion if surfaces become rough or uneven, or if corrosion deposits cannot be removed without damaging the test piece. The correct alignment function of the test jig may be interrupted. The uncertainty introduced cannot at present be quantified, but needs to be assessed on a case-by-case basis. Provided that this problem is not severe, strength testing remains a sensitive and pragmatic method of determining whether the corrosive attack has caused significant weakening in a test-piece or component.

6.5 Test type E: Change in surface roughness

This test is appropriate if the rate of corrosion is low, and affects principally the surface topography, for example by preferential etching of secondary phases or grain boundaries. It is unsuitable if significant penetration occurs, since surface layers may become soft, or the apparent roughness very large.

6.6 Test type F: Change in hardness

The measured hardness of corroded surfaces is influenced by the depth of penetration achieved by the corroder and/or the depth of depletion of individual species within the microstructure. The formation of additional porosity results in significantly reduced hardness when tested using an adequately large indenter force, typically 10 N or greater. Since corroded surfaces may no longer be flat or adequately polished for accurate hardness measurement, such a test should be used only as an *indicator* of this form of attack, and the likely consequent loss in abrasion or erosion resistance.

Adherent surface deposits or slags render hardness testing irrelevant. The test is appropriate only for light corrosive attack.

6.7 Test type G: Change in chemical composition of corroder

This standard does not lay down any prescribed methods for determining the quantity of test-piece species dissolved in the corroder. Reference should be made to standard analytical practices.

7 Apparatus for laboratory corrosion testing

7.1 Container for corroding medium

7.1.1 The container for the corroding medium shall be resistant to that corroding medium under the conditions of the test, shall be of sufficient volume to allow adequate distance (minimum 5 mm) between test-pieces, and, under conditions in which continuous replenishment of corroder is not provided, that significant depletion of the strength of the corroder during the course of the test does not occur.

NOTE Suitable containers for some simple types of test are listed in Annex A.

7.1.2 The container shall be equipped with suitable safety facilities for sealing against spillage, and if appropriate for maintaining the appropriate pressure. If the pressure is above one atmosphere, ensure that the vessel has been appropriately certified as safe for the intended conditions of use.

7.1.3 Unless the test is under sealed conditions (e.g. in a digestion bomb), the container shall be equipped with suitable inlet and outlet ports for entry and exit of corroding medium as appropriate to requirements.

7.2 Heating device

If the corrosion test is to be performed at elevated temperature, a suitable means of heating the container is required. The temperature shall be capable of being controlled to a constancy of ± 5 K. The temperature of the corroding medium in the region of the test-pieces shall be measured with a separate thermocouple (see 7.3).

7.3 Thermocouple

If the temperature of the test is to be recorded, Type K, Type R or type S thermocouple in accordance with EN IEC 60584-2, appropriately sheathed against corrosive attack.