

SLOVENSKI STANDARD SIST EN 12923-1:2007

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

Hochleistungskeramik Monolithische Keramik Teil 1: Allgemeines zur Durchführung von Korrosionsprüfungen (standards.iteh.ai)

Céramiques techniques avancées - <u>Géramiques_mon</u>olithiques - Partie 1: Pratique générale destinée aux essais de corrosion and ards/sist/2bd85f30-c877-4989-b050-b21265d77afd/sist-en-12923-1-2007

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

Céramiques techniques avancées - Céramiques monolithiques - Partie 1: Pratique générale destinée aux essais de corrosion Hochleistungskeramik - Monolithische Keramik - Teil 1: Allgemeines zur Durchführung von Korrosionsprüfungen

This European Standard was approved by CEN on 25 November 2006.

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This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

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Foreword

This document (EN 12923-1:2006) 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of an identical text or by endorsement, at the latest by June 2007, and conflicting national standards shall be withdrawn at the latest by June 2007.

This document supersedes ENV 12923-1:1997.

EN 12923 Advanced technical ceramics — Monolithic ceramics consists of two parts:

Part 1: General practice for undertaking corrosion tests

Part 2: Oxidation test

At the time of publication of this edition of Part 1, Part 2 was a European Prestandard.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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1 Scope

This part of EN 12923 specifies guidelines 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 the ability to withstand the proposed end-use conditions.

This European Standard is not restricted to specific material types, nor does it prescribe particular test conditions or a test duration. The actual testing requirements might be very specific, for example, in order to investigate the suitability of a range of materials for a given application in which certain specified conditions occur. This European Standard provides recommended 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

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EN 623-4, Advanced technical ceramics — Monolithic ceramics — General and textural properties —
Part 4: Determination of surface roughness
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EN 843-1, Advanced technical ceramics — Mechanical properties of monolithic ceramics at room temperature — Part 1: Determination of flexural strength

EN 843-4, Advanced technical ceramics — Mechanical properties monolithic ceramics at room temperature — Part 4: Vickers, Knoop and Rockwell superficial hardness

ENV 1006, Advanced technical ceramics — Monolithic ceramics — Guidance on the selection of test pieces for the evaluation of properties

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

EN 60584-2, Thermocouples — Part 2: Tolerances

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

ISO 3611, Micrometer callipers for external measurement

ISO 6906, Vernier callipers reading to 0,02 mm

3 Terms and definitions

For the purposes of this document, the following terms and 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: DARD PREVIEW

- i) the chemical nature, phase composition, phase distribution and degree of continuous porosity in the material;
- the temperature, pressure, composition, concentration and flow rate of the corroding medium and whether these are constant or vary with time; 12007
- 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;
- iv) 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:

- 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 modelled in the test environment;
- b. to provide a comparative measure of performance of a range of materials under defined corrosion conditions.

This European 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 European 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 Assessment of corrosive attack

5.1 Introduction

Chemical attack is manifest in a number of ways:

- a. change of dry mass;
- b. change of section thickness;
- c. change of colour;
- d. penetration of corrodent into the material rendering the surface open porous;
- e. development of surface skins of altered composition;
- f. development of a surface skin of reaction product;
- g. change of surface finish;
- h. change in strength;
- i. change of hardness or wear resistance.

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

Table 1 summarizes 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.//standards.iteh.ai/catalog/standards/sist/2bd85f30-c877-4989-b050-

NOTE 1 Other situations might 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 European Standard and might require special equipment not covered by this European Standard. Change of colour might be subjective.

NOTE 2 This European Standard does not lay down any recommended methods for determining the quantity of test piece species dissolved in a corrodent (Method G). Reference should be made to standard analytical practices. Care should be taken in selection of the container material, corrosion of which may influence the results of such an analysis.

5.2 Method A: Depth of penetration

- **5.2.1** For materials subjected to corrodents which result in grain boundary attack and penetration of the corrodent, the depth of penetration might be strongly influenced by the microstructural nature of the original test piece surface. Some as-fired surfaces might 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.
- **5.2.2** The depth of penetration might be variable if the microstructure is inhomogeneous. The greatest depth of penetration is to be recorded.
- **5.2.3** In materials with substantial pre-existing closed porosity, it might be difficult to determine the true depth of penetration if there is no major change in the appearance of the microstructure. Reliance should be placed on the use of penetrant dyes in accordance with EN 623-1.

Assessment method	Aqueous based corrosion	Corrosion by melts	Corrosion by gases and vapours
A. Penetration	Appropriate	Might be appropriate	Appropriate in some cases
B. Change of mass	Appropriate, but sometimes inadequate	Inappropriate	Appropriate if non-slagging
C. Change of cross- section	Not always appropriate when change is small	Appropriate	Appropriate
D. Change of strength	Appropriate	Might be appropriate if adherent	Appropriate if non-slagging; might be

material is removed

Usually inappropriate

Usually inappropriate

appropriate if adherent material is removed

Usually inappropriate

Might be

appropriate if

non-slagging

Appropriate

Table 1 — Appropriateness of methods of assessing corrosive attack

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Appropriate A Usually inappropriate

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Appropriate for slight

corrosion

Appropriate

5.3 Method B: Mass change

E. Change of surface

F. Change of hardness

corrodent composition

roughness

G. Change of

- **5.3.1** Accurate determination of change in dry mass might 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 might have occurred, but other criteria need to be employed to quantify its overall effect on performance.
- **5.3.2** Assuming that the process of mass gain or mass loss is attributable to the test piece behaviour, accurate determination of change in dry mass relies on being able to minimize 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 might result.
- NOTE 1 It might 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.
- NOTE 2 For the particular case of high temperature oxidation tests, further information is given in ENV 12923-2 [1].
- **5.3.3** 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 might 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.

- **5.3.4** For very low levels of mass change, the potential error of measurement is large. It might 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.
- **5.3.5** In cases where material is lost due to spallation, it might be useful to incorporate a plate or tray under the test material to catch debris for later analysis. Caution should be exercised such that the tray does not increase the contact area with the test piece or restrict the flow of the corroding medium.

5.4 Method C: Change of test piece size

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

- 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;
- ii) measurement of section thickness unaffected by corrosive attack. This method involves sectioning and polishing which might change the morphologies of corrosion products and introduce artefacts. 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. SIST EN 12923-1:2007

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5.5 Method D: Strength change 265d77afd/sist-en-12923-1-2007

The close specification in EN 843-1 (see 6.11) for flexural strength testing is designed to achieve results with minimal errors might 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 might 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.

5.6 Method 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 might become soft, or the apparent roughness very large.

5.7 Method F: Change in hardness

The test is appropriate only for light corrosive attack. The measured hardness of corroded surfaces is influenced by the depth of penetration achieved by the corrodent 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 might 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.