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Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of oxidation resistance of non-oxide monolithic ceramics

Céramiques techniques — Détermination de la résistance à l'oxydation des céramiques monolithiques

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 20509 was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of oxidation resistance of non-oxide monolithic ceramics

1 Scope

This International Standard describes the method of test for determining the oxidation resistance of non-oxide monolithic ceramics, such as silicon nitride, Sialon[1] and silicon carbide at high temperatures. This International Standard is intended to provide an assessment of the mass and dimensional changes of test pieces following oxidation at high temperature in an oxidizing atmosphere, and to assess whether oxidation has a significant effect on the subsequent strength. This test method may be used for materials development, quality control, characterization, and design data generation purposes.

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.

ISO 3611:1978, *Micrometer callipers for external measurement*

ISO 6906:1984, *Vernier callipers reading to 0,02 mm*

ISO 7500-1:—[2], *Metallic materials — Verification of static uniaxial testing machines — Part 1: Tension/compression testing machines — Verification and calibration of the force-measuring system*

ISO 14704:2000, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for flexural strength of monolithic ceramics at room temperature*

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

IEC 60584-2:1989, *Thermocouples — Part 2: Tolerances*

3 Terms and definitions

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

3.1
oxidation resistance
resistance against oxidation of a non-oxide ceramic material due to reaction 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

3.2
flexural strength
maximum nominal stress at fracture of a specified elastic beam loaded in bending

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[1] Sometimes written SiAlON is the acronym for a ceramic that contains silicon, aluminium, oxygen and nitrogen.

[2] To be published. (Revision of ISO 7500-1:1999)

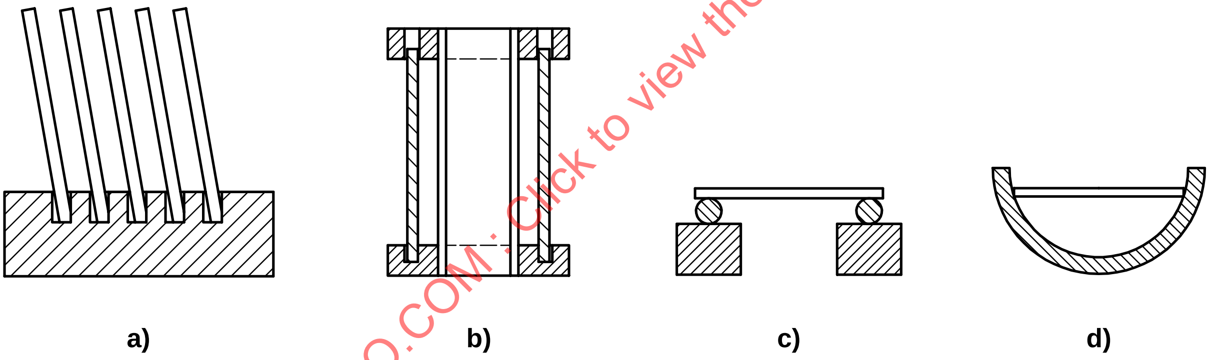
4 Apparatus

4.1 High temperature furnace, e.g. any suitable air atmosphere furnace with a nominal temperature capability of at least 1 500 °C.

The furnace chamber shall have an inlet for a sufficient supply of oxidation gas to ensure that the atmosphere does not stagnate and become oxygen deficient. The temperature shall be capable of being raised to that required for testing at a minimum of 5 °C min^{-1}, of being controlled to better than ± 5 °C at all oxidation temperatures, and of being cooled at more than 5 °C min^{-1} to below 800 °C. Before commencing oxidation tests, the furnace chamber shall be baked out using the same atmosphere as proposed for testing and at a temperature at least as high as the intended oxidation test temperature for a period of at least 10 h in order to remove contaminants.

4.2 Support or supporting stand, for oxidation tests.

The test pieces shall be supported using techniques that minimize contact area, degree of adhesion and extent of reaction with the test piece (see Figure 1). Preferably this should be done using point or line contact only. Any contact of the supports with the regions of the test piece surfaces to be subjected later to loading roller contact in flexural strength testing shall be avoided. Examples of suitable support methods include the use of a block with drilled holes no more than 3 mm deep such that the test pieces can stand near vertically with a minimum of end and edge contact. The samples can also be situated on horizontal supports on rollers of silicon carbide or mullite, on small diameter platinum wires, either suspended or resting on a clean non-reactive ceramic surface, or on semi-rings which can be cut from ceramic tubes (alumina, mullite, silicon carbide, or silicon nitride).



a) a refractory block with appropriate-sized holes in it, suitable for muffle furnace;

b) a support system based on tubes and discs with holes, suitable for vertical tube furnace;

c) a pair of supported parallel rods spaced near the ends of the test-pieces and with an adequate gap underneath, suitable for a muffle furnace;

d) a test-piece supported by its ends on a ceramic semi-ring.

Figure 1 — Examples of support systems for flexural strength test pieces

NOTE 1 It may be necessary to perform some preliminary assessments to ensure that the supporting material is sufficiently non-reactive as to not significantly contribute to the mass changes in the sample.

NOTE 2 Candidate materials for supporting test pieces include silicon carbide, mullite, platinum wire and alumina. Silicon carbide and mullite may be the most suitable materials. Alumina may react with test pieces, and platinum is inappropriate for non-oxide ceramics containing free metallic species, such as silicon carbide containing silicon.

4.3 Oven, capable of maintaining a temperature of 105 °C to 120 °C.

4.4 Testing machine for flexural strength, capable of applying a uniform crosshead speed. The testing machine shall be in accordance with ISO 7500-1:— Class 1 with an accuracy of 1 % of indicated load at fracture.

4.5 Testing fixture for flexural strength, of three- or four-point flexure configuration in accordance with 5.2 of ISO 14704:2000. The recommended fixture is fully articulated and of the four-point-1/4 point configuration with the two outer bearings at a distance of 40 mm. The corresponding total length of test piece is $\geqslant$ 45 mm.

4.6 Micrometer, such as shown in ISO 3611:1978 but with a resolution of 0,002 mm for measuring the test piece dimensions. The micrometer shall have flat anvil faces such as shown in ISO 3611:1978. The micrometer shall not have a ball tip or sharp tip since these might damage the test piece. Alternative dimension measuring instruments may be used provided that they have a resolution of 0,002 mm or finer.

4.7 Vernier calliper, with a resolution of 0,05 mm or finer for measuring the length of the test piece, in accordance with ISO 6906. Alternative dimension measuring instruments may be used provided that they have a resolution of 0,05 mm or finer.

4.8 Balance, capable of weighing up to 200 g with a sensitivity of at least 0,05 mg.

4.9 Thermocouple, of type R or type S in accordance with IEC 60584-2, permitting the use of the calibration table given in IEC 60584-1.

5 Test pieces

If the strength changes are to be determined, flexural strength test pieces in accordance with Clause 6 of ISO 14704:2000 shall be used. The standard test specimens shall have cross-sectional dimensions of 3,00 mm $\pm$ 0,20 mm thickness and 4,00 mm $\pm$ 0,20 mm width. The length shall be more than 35 mm for 30 mm test fixtures or more than 45 mm for 40 mm test fixtures. All the surfaces shall be machined, and edges shall be rounded or chamfered. Any machining procedure and the surface quality of the test pieces shall be reported. The minimum number of test pieces shall be 10 for each oxidation condition to be tested, plus 10 test pieces as an unoxidized control. Means shall be taken to identify individually, similar test pieces, but shall not be marked or scribed in a way that might affect the result of the test. If strength changes are not to be determined, any test piece, in terms of size and shape, may be used.

Test pieces shall be clean and free from preparation residues and contamination due to handling which can influence the initial mass measurement and/or the oxidation rate. The test-piece cleaning procedure shall be stated in the report.

For materials with no significant open porosity and contaminated by handling, and/or by mounting or machining coolant residues, submerge the test-pieces in ethanol in an ultrasonic bath and agitate for at least 10 min. In order to avoid damage, test pieces shall not be allowed to contact either each other or a hard surface during this operation. For materials containing open porosity, internally entrained organic residues can be removed only by heating in air. The maximum temperature to which this should be done will depend on the material type, but typically a temperature of 500 °C to 600 °C for a least 1 h is required to oxidize carbonaceous residues. Material with intentionally present free carbon shall be treated at a maximum temperature of 350 °C to avoid oxidation.

6 Test procedure

6.1 Measurements of dimensions and mass of specimens

For flexural strength test pieces, measure the width, b, and thickness, h, of each test piece at several places using the micrometer (4.5) with a resolution of 0,002 mm. Measure the overall length, L_T, with the vernier callipers (4.6) with a resolution of 0,05 mm. For other shapes of test piece, measure relevant dimensions at several different places (e.g. diameter and thickness of a disc). Wash and degrease the test pieces (see Clause 5). Place in the oven (4.3) and heat to a temperature of 105 °C to 120 °C until their mass is constant. Remove and store in a desiccator. When cooled to room temperature, weigh each test piece to the nearest 0,05 mg using the balance (4.7). Store in the desiccator until tested.

6.2 Baking out in the oxidation furnace

Unless used for a similar measurement immediately prior to the test, pre-condition the furnace (4.1) and the test piece support system (4.2) at a temperature similar to or greater than that intended for the oxidation test under the intended flowing gas atmosphere. The maximum temperature shall be maintained for at least 10 h.

6.3 Oxidation test

6.3.1 Materials with high oxidation resistance

6.3.1.1 Place the test pieces on their supports (4.2) in the centre of the hot zone of the furnace (4.1) ensuring sufficient space between test pieces and their supports for adequate circulation of air. Ensure that contact with supports is minimized (see Figure 1). The contacts shall always be at locations outside the outer span used for the flexural test. The minimum spacing between test pieces as well as that between a test piece and furnace furniture shall be 5 mm.

NOTE 1 It is preferred that each batch of a least 10 test pieces per oxidation condition is exposed at the same time in the same facility. Separated exposure at separate times may result in slightly different results.

NOTE 2 The minimum spacing between components or test-pieces under test should be increased with increasing component or test-piece size to ensure unimpeded gas flow between neighbouring oxidizing surfaces.

6.3.1.2 Position a type R or type S thermocouple (4.8) in accordance with IEC 60584-2 adjacent to the test pieces for the purposes of monitoring test piece temperature during the oxidation period. Close the furnace.

6.3.1.3 Supply the oxidizing gas at a rate sufficient to provide atmosphere circulation within the furnace cavity and around the test pieces such that stagnation and oxygen depletion is avoided, but not at such a rate that results in inhomogeneous or fluctuating furnace temperature. For testing in normal air, a natural flow of the air through the furnace cavity shall be facilitated. Note that a gas flow rate is recommended of between typically 0,5 and 50 volume changes per hour, but not less than 0,1 changes per hour.

6.3.1.4 Heat the furnace to the test temperature as indicated by the measuring thermocouple adjacent to the test pieces. Maintain this temperature to within 5 °C for the required oxidation period. Cool the furnace at the maximum rate of cooling of the furnace and carefully remove the test pieces from their supports and place in a desiccator. To avoid contamination, do not touch the test pieces with naked fingers until after the final weighing. Ensure that loose deposits on the test piece surface are retained intact as far as possible.

6.3.2 Materials with low oxidation resistance

The procedure outlined in 6.3.1 can be also used in the testing of materials with low oxidation resistance or those producing low melting oxidation products, such as B_2O_3 formed during the oxidation of borides. Some modifications are suggested for the samples that react significantly with or stick to the support fixture. In these cases, it is recommended to use a system supporting each individual sample, such as the semi-rings [Figure 1 d)], so that the sample and the support can be weighed together before and after the test.

Additionally, the samples should be inserted into a furnace, preheated to the test temperature, and then air-quenched after the test to retain the high temperature condition of the surface layer for room temperature microscopic evaluation. Nevertheless, the quenching of the samples can affect the strength as a result of thermal shock.

6.4 Selecting test conditions

Test conditions (temperature, atmosphere, duration, etc.) shall be selected according to the technical requirements for undertaking the test and on agreement between parties.

NOTE 1 The test condition recommended for silicon nitrides and Sialons is 1 300 °C for 100 h or 200 h, and that for silicon carbides and advanced grades of silicon nitrides is 1 400 °C for 100 h or 200 h. Such conditions provide a means of readily distinguishing performances of similar materials.

NOTE 2 The oxidation behaviour of other non-oxide ceramics (such as borides, carbides, nitrides and silicides) varies widely, and the test conditions should be selected on the basis of preliminary experiments. Test results should be documented carefully in the report.

6.5 Measurements of mass and dimensional changes

Weigh the test pieces individually with their adherent oxidation products to the nearest 0,05 mg. Weigh any loose spallation products separately. If spallation products from individual test cannot be weighed separately, weigh them altogether. If appropriate, remeasure the external dimensions of the test pieces for the determination of dimensional changes.

NOTE It is generally not realistically possible to measure spallation that is adherent to or reacting with furnace or test piece support parts.

6.6 Measurements of flexural strength

Measure the flexural strength of the oxidized and the control test pieces in accordance with Clause 7 of ISO 14704:2000. If the nature of the oxidized surface has to be changed in order to undertake the strength tests this must be mentioned in the report.

NOTE 1 A fully articulating fixture should be used for flexural strength measurements of the oxidized specimens because they may not meet the parallelism requirements given in ISO 14704 for use of a semi-articulating fixture.

NOTE 2 A semi-articulating fixture may be used if the parallelism requirements are satisfied. One surface of an as-oxidized part may be machined to help minimize twisting or warping effects. The machined surface should be placed in contact with the inner bearings (specimen compression side) during testing.

6.7 Particular features

Record any particular features associated with the condition of the oxidized surfaces, the appearance of fractured cross-sections, etc.

NOTE Phase analysis of the oxide layer using an X-ray diffraction technique and microstructure observation of the oxide layer in the cross section using scanning electron microscopy may be useful characterization methods.

7 Calculations

7.1 Flexural strength

If flexural strength has been measured, compute the flexural strength using the relevant formula for the jig type, whether three-point or four-point bending, as in ISO 14704. Compute the average strength and standard deviation for the control batch and for each oxidation condition. Report the outer and inner spans and whether semi-articulating or fully articulating.

7.2 Mass change

If the mass change is required, compute the mass change per unit nominal surface area of test piece (C) in accordance with the formula:

$$C = \frac{W_\mathrm{f} - W_\mathrm{i}}{A} \qquad (1)$$

where

W_f and W_i are final and initial mass of test piece respectively, expressed in grams;

A is the nominal external surface area of test piece based on initial dimensions expressed in square meters.

Compute the average result and the standard deviation for each oxidation condition employed.

$$\overline{C} = \frac{\sum_{i=1}^{n} C_i}{n} \tag{2}$$

$$S = \sqrt{\frac{\sum (C_i - \overline{C})^2}{n-1}} \tag{3}$$

where

$\overline{C}$ is the average value;

n is the total number of specimens;

S is the standard deviation.

NOTE If the material is porous and has oxidized internally, it may be more appropriate to express the oxidation behaviour as a mass change per unit volume, or as a percentage mass change. In both cases, these calculations will be averages for the test piece, and will not reflect spatial variations in oxidation behaviour within the test piece.

7.3 Nominal dimensional change

If the change in dimensions is required, compute the absolute changes in the linear dimensions b and h, and express them in mm:

$$\Delta h = h_\mathrm{f} - h_\mathrm{i} \tag{4}$$

$$\Delta b = b_\mathrm{f} - b_\mathrm{i} \tag{5}$$

where

h_f is the final test piece thickness, expressed in millimetres;

h_i is the initial test piece thickness, expressed in millimetres;

b_f is the final test piece width, expressed in millimetres;

b_i is the initial test piece width, expressed in millimetres.

Compute the average absolute change and the standard deviation for each oxidation condition.

$$\overline{\Delta h} = \frac{\sum_{i=1}^{n} \Delta h_\mathrm{i}}{n} \tag{6}$$

$$S = \sqrt{\frac{\sum_{i=1}^{n} (\Delta h_i - \overline{\Delta h})^2}{n-1}} \tag{7}$$

$$\overline{\Delta b} = \frac{\sum_{i=1}^{n} \Delta b_{\mathrm{i}}}{n} \tag{8}$$

$$S = \sqrt{\frac{\sum_{i=1}^{n} (\Delta b_i - \overline{\Delta b})^2}{n - 1}} \tag{9}$$

where

$\overline{\Delta h}$ and $\overline{\Delta b}$	is the average value;
n	is total number of specimens;
S	is standard deviation.

8 Test report

The results of oxidation tests shall include the following:

a) name and address of the testing establishment;

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

c) reference to this International Standard, i.e., determined in accordance with ISO 20509;

d) description of the test apparatus used, including details of the type of oxidation furnace, the materials used in construction of the hot-zone, test piece support system used, the materials in direct contact with the test pieces, the heating and cooling rates used, details of the rate of supply of test gas, its composition and moisture content, if appropriate, the type of flexural test, etc.;

e) description of the test material (manufacturer, type, batch number, date of manufacture, etc.);

f) description of the details of the test piece preparation, including nominal dimensions and surface finishing procedures, cleaning procedures, strength test conditions, in accordance with the provisions of ISO 14704;

g) individual determinations of test pieces mass before and after testing, the computed mass change for each test piece at each test condition, the mean and standard deviation for each test condition, expressed in gram per square metre;

h) mass of the collected spallation products, expressed in grams, and the average spallation mass per unit surface area of test piece;

i) individual fracture load in strength tests, expressed in newtons, and computed nominal fracture stress expressed in megapascals (or meganewtons per square metre) for each test piece at each test condition including unoxidized control test pieces, the mean results and standard deviations for each condition;

j) individual results of mean test piece dimensions before and after testing, and the computed size changes at each test condition, the mean results and standard deviation;

k) any comments about the test or the results, including observations on the surface condition of the test pieces following oxidation and the oxidation layer, such as scanning electron micrographs of oxidized layer, phases in the oxidized layer and thickness of oxidized layer, on whether the surface condition was modified in order to conduct strength tests, on the appearance of fracture surfaces, and on the likely interference caused by oxide scale spalling or adhesion to the support system.

Annex A
(informative)

Useful information

A.1 General

Evaluation of oxidation resistance is important when non-oxide ceramics are used in oxidizing environments at high temperatures. Oxidation in non-oxide ceramics usually occurs as a result of substitution of non-oxygen non-metallic species by oxygen, which develops a surface skin of altered composition. Mass change is a common measure to evaluate oxidation resistance. Another measure of oxidation resistance is the strength variation, since the strength-determining flaw can be altered by the generation of oxidation-induced flaws and crack healing. When non-oxide ceramics are used at high temperatures as structural components, oxidation resistance is often evaluated via mass and strength variations.

A.2 Oxidation mechanisms and the formation of oxides

Oxidation of non-oxide ceramics usually occurs when oxygen in air reacts with the ceramic substance. Once the reaction occurs, an oxide layer is often formed on the surface. If the layer is protective, further oxidation is retarded and the oxidation rate is governed by the diffusion rate in the oxidation layer. In this case, the oxidation rate of mass gain should obey a parabolic law and a dense oxide layer is formed on the surface [1]-[12]. However, when the oxide layer is not protective, the reaction at the original surface of the material determines the rate, and the reaction rate exhibits a linear dependence over time [11]-[14]. In some cases, the oxide products may not adhere to the sample, but tend to flake off as a result of disruptive forces caused by volume changes, phase changes and thermal expansion mismatches [13], [14]. For materials with open porosity, such as reaction-bonded silicon nitride and some silicon carbides, oxidation will generally occur through continuous pores that are initially surface connected, although these may become blocked as oxidation proceeds [15]. As a result of oxidation of silicon nitride and silicon carbide, the major phase formed on the surface is amorphous silica that contains the ions originally doped in the bulk [5], [16]. The presence of water vapour influences the oxidation rate [6], [10], [17], and can enhance the crystallization of the oxide layer [18].

Oxidation kinetics depend not only on the materials but also the oxidation conditions. The oxidation rates of refractory carbides tend to follow a parabolic rate law under conditions of high oxygen partial pressure and low oxidation temperature, and a linear dependence is found in high temperature oxidation [11]. La_2O_3-Y_2O_3 doped-silicon nitride obeys parabolic oxidation dependence at temperatures $< 1\,450\ ^\circ C$ for short exposure periods, while linear oxidation dependence governs at higher temperatures for longer exposures [12]. A porous (21,5 % porosity) nitride bonded silicon carbide containing 20 % SiC exhibited two oxidation stages: the initial stage corresponds to an incubation period (0 h-10 h), where a continuous oxide film is developed on the surface while allowing internal oxidation at the pore channels; the parabolic oxidation kinetics govern the second stage, where the rate-limiting step is oxygen diffusion through the surface silica layer [19].

A.3 Mechanical properties after oxidation

Since oxidation produces the surface oxide layer and modifies the microstructure of the materials, mechanical properties such as fracture strength, fracture toughness and elastic modulus are often degraded [5]-[9], [13], [14], [20]-[22]. In particular, severe oxidation often causes a significant degradation in flexural strength while slight oxidation sometimes leads to an increase in strength [6]-[9], [22]-[24]. The increase is due to the blunting of machining flaw crack tips by formation of a thin oxide film, and the strength degradation is usually caused by the generation of oxidation pits and the relaxation of residual stresses, which are produced during the machining process [12], [22]. Controlled oxidation of non-oxide ceramics can improve the oxidation resistance and fracture strength; e.g., a substantial increase in room temperature strength was observed when the as-machined samples of hot-pressed silicon nitride were heated in air at moderate temperatures (800 °C to 1 100 °C) [25], [26]. Oxidation induced surface modification improved the strength, oxidation resistance and fracture toughness [27], [28].

Annex B
(informative)

Interlaboratory evaluation of the test method

B.1 General

This International Standard was established based on the Japanese Industrial Standard: JIS R 1609[31] and the European Standard: ENV 12923-2[32]. Therefore, noting the background for these Standards may be useful.

B.2 Round robin for JIS R 1609

In order to establish the Japanese industrial standard JIS R 1609, the round robin was conducted for three years with three participants. The ceramic materials used in the round robin were sintered silicon nitride, sintered silicon carbide and reaction-sintered silicon carbide. The dimensions of test pieces were 3 mm × 4 mm × 40 mm. The oxidation tests were conducted at a temperature of 1 200 °C to 1 400 °C for soaking times of 24 h to 100 h. Mass changes were measured from the weights before and after oxidation tests. Flexural strength was measured in three-point flexure with a span of 30 mm. Oxidation layers of cross section of the specimens were observed with scanning electron microscopes. The results showed the following.

a) In the case of silicon nitride conducted at 1 200 °C for 24 m, an oxide layer of 2 μm to 3 μm was formed on the surface of the material, accompanied with slight weight gain. The flexural strength was, however, significantly degraded and the degradation was 10 % to 40 %. Prolonged soaking time and higher temperature lead to further development of the oxide layer on the surface, while the further degradation in flexural strength was rather insignificant.

b) Sintered silicon carbide has an excellent resistance to oxidation even at 1 400 °C. The mass change was very small within several mg and most of tests were within 1 mg. Although the time-dependences of strength and mass changes differ greatly among the participants, the flexural strength related to the mass change. The slight oxidation with a weight gain of approximately 1 mg leads to the highest strength of approximately 450 MPa in comparison with the initial strength of 400 MPa.

c) Reaction-sintered silicon carbide exhibits a decrease in the flexural strength and a weight increase with oxidation time.

B.3 Round robin for ENV 12923-2

As a part of the development of the European Standard, an interlaboratory study was conducted among 23 European laboratories. Participants were asked to conduct 200 h oxidation tests in air on two materials, a reaction-bonded silicon nitride at 1 100 °C (high oxidation rate) and a sintered silicon carbide at 1 400 °C (low oxidation rate). Participants were provided with 20 flexural strength test pieces of each material and were asked to make measurements of mass gain, dimensional changes and flexural strength according to the draft procedure for the ENV standard. Preferred support materials were as recommended in the CERANORM project [29]. The results [30] showed the following.

a) Mass change measurement can produce useful data provided that oxidation resistance is not too high relative to achievable measurement accuracy and that test pieces are treated carefully during and after oxidation testing. For RBSN with a mean mass gain per test-piece of 55 mg and negligible interaction problems with support materials at 1 100 °C, the coefficient of variation was 38 %. For SiC with a mean mass gain of only 3 mg and substantial interaction with support materials, the coefficient of variation exceeded 100 %. It was concluded that the principal problem was limited participant capability for weighing to the required accuracy. Although mass change provides a measure of oxidation resistance to many people with an oxidation/corrosion background, it is recommended that a mass change should only be reported as a valid measure of oxidation resistance if it exceeds at least 50 times the balance sensitivity (i.e. 2,5 mg), and if interaction between test material and support materials is negligible. The change may be expressed in mass change (g or %) per unit surface area.

b) When dimensional changes due to oxidation were measured to the nearest 0,01 mm on test pieces machined to the requirement of EN 843-1[33], avoiding surface affected by interaction with support materials, the coefficients of variation for the measurement on RBSN and SiC were 71 % and over 100 %, respectively. Within the limit of measuring accuracy, it is therefore evident that dimensional changes of relatively oxidation-resistant materials such as RBSN and SiC cannot be used as a measure of oxidation-induced damage. A more stringent requirement regarding the accuracy of measurement (cf. 0,01 mm) might have given more consistent results but may have reduced the number of laboratories able to conform to the requirement. Measurements on cross sections of test pieces using microscopic methods (e.g. SEM) could be applied in specific circumstances, but are considered outside the main scope of the standard due mainly to cost consideration. Other less oxidation-resistant materials could well produce measurable changes in test piece dimensions.

c) For SiC, a homogeneous material, the spread of participant mean strength was described by a range about the mean of 15,7 % and a coefficient of variation 5,1 % for as-received material and an average of 24,3 % about the mean and coefficient of variation of 5,7 % for oxidized material. For RBSN, the material used for this work was found to have been supplied as two sub-batches with different porosity levels, the overall coefficient of variation for as-received and oxidized test pieces were 17 % and 20 %, respectively. However, considering data for only one of the sub-batches (high porosity sub-batch) the coefficient of variation was only 4 %. Thus, even with relatively inhomogeneous material, scatter for all laboratories is relatively small. None of the laboratories reported a need to remove rough deposits or oxidation products prior to flexural testing of oxidized test pieces, but extensive test piece/support material interaction did lead to the span length being changed to a non-standard one by one laboratory. It is advisable, therefore, for preliminary tests to be carried out when the magnitude of test material/support material interaction cannot be predicted.

d) The observed scatter in strength data may be due either to random sampling of the batches of test pieces supplied to each participant or equally to differences in test-jig function (nearly all used four-point flexure), or to a “sidedness” to the oxidized test pieces relative to the tensile side in the flexural strength tests (i.e. related to differences in oxidation appearances between top and underside of test pieces supported horizontally in the oxidation furnace). A full analysis based on fractographic analysis has not performed within the scope of the analysis, but might help explain the results obtained.

e) There was no evidence that the oxidation test parameters selected for the study were the cause of any specific contribution to the observed scatter of data. This includes in particular, possible effects of moisture content and heating rate. Also, interruptions in the 200 h test due to equipment malfunction had a negligible effect on measured properties. However, problems were encountered in the test pieces that were close to furnace furniture. Being too close to furnace furniture appeared to cause uneven oxidation on the face of the test piece adjacent to the furnace component. A minimum clearance of at least 5 mm is thus essential. Further, chemical interaction with furnace furniture could not be avoided, so supporting test pieces well outside the 40 mm outer span used for flexural strength testing was shown to be essential.