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

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National foreword

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Contents

Foreword

This document (EN 1006:2009) 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of an identical text or by endorsement, at the latest by January 2010, and conflicting national standards shall be withdrawn at the latest by January 2010.

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Introduction

Advanced technical ceramics have a wide range of applications and functions and, in the as-manufactured condition, have characteristics which require inspection by a variety of techniques not commonly adopted for other classes of material, e.g. mechanical proof testing.

1 Scope

This European standard gives guidance on selection of test-pieces for the evaluation of properties. Important factors requiring attention in the preparation of test samples from large components or blocks of material are also described.

2 Terms and definitions

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

2.1
batch
population of manufactured units of a single type, grade, size and composition, manufactured under essentially the same conditions at the same time, from which a sample is to be taken for inspection and/or testing to determine the conformance with acceptability criteria

NOTE Sometimes referred to as a 'lot'.

2.2
sample
sample consists of one or more manufactured units taken from a batch, these being selected at random without regard for their quality

2.3
sample size
number of units in a sample

3 Selection of test-pieces

3.1 General

The basis of any inspection of any material or batch of manufactured units is to obtain sound information on their fitness for purpose (quality). Advanced technical ceramics are diverse in material, format and application as are the methods devised to test their fitness for purpose. Before arranging any inspection or testing scheme it is wise to consider in depth the nature of the material, its final format in relation to test-pieces required for tests, the accuracy of test methods and the failure criticality in its application.

NOTE It is not the purpose of this European standard to define criteria for fitness for purpose. This is subject to agreement between parties.

3.2 Material homogeneity and anisotropy

3.2.1 Most advanced technical ceramic materials are made by powder technology processes involving the formation of a rigidized powder mass (e.g. pressing, slip casting, etc.) before subjecting this to a densification process (e.g. sintering, reaction bonding, hot pressing). The homogeneity and isotropy of the rigidized powder mass and the control imposed during the subsequent densification process can exert a considerable influence on the homogeneity of the final densified product. Consequently, attributes can vary from one place to another within a component or between components of the same batch.

3.2.2 One of the principal sources of a variation of attributes is density, arising from inhomogeneity of unfired (green) density, which has a subsequent significant effect on many mechanical properties. Large localised variations in unfired density are usually manifest as excessive distortion in firing, porous regions, or cracking. Other varying attributes are grain size (usually resulting from varying heat

treatment conditions between components) and chemical composition (usually resulting from inhomogeneous interaction between initial powder particles or between particles and the atmosphere, perhaps involving the migration of species).

3.2.3 Material inhomogeneity is most prevalent in large components or blocks of material, or in components requiring special firing conditions. It is frequently met with during material development, but is usually minimised during commercial product development.

Material anisotropy is sometimes encountered in materials which have some form of directional microstructure. This may result, for example, from a combination of the initial powder particle shape and the rigidizing process to make a green shape, or during firing if uniaxial hot pressing is employed. Attributes subsequently determined can be dependent on the direction in which a test-piece is cut and in which the property or characteristic is determined.

3.3 Test method accuracy

3.3.1 Most test methods specifically developed for advanced technical ceramics have associated with them a possible uncertainty of result determined by the accuracy of individual contributions from each parameter involved in the measurement. The potential uncertainties arising shall be taken into account when examining the consistency of a parameter within a batch of units or between batches, or examining whether it meets a given specification level.

3.3.2 It should be noted that in cases where the scatter of results of a test is similar to or less than that attributable to the accuracy of test method, the test is clearly unable to distinguish between individual test-pieces or samples. An improvement of the accuracy level of the chosen test or an alternative test method should be sought.

3.3.3 Certain tests for advanced technical ceramics produce a wide scatter of results as a consequence, for example, of the influence of occasional flaws or other defects, e.g. a strength test or dielectric breakdown test. The results from such tests shall be treated statistically (see e.g. EN 843-5 for strength tests), and the confidence level of the mean result or other parameters should be calculated such that the degree of discrimination between results from different batches, or between a set of results and a specification value, is clearly understood.

3.4 Sampling schemes for individual manufactured items

3.4.1 Sampling schemes are conventionally divided into those for inspection by attributes, e.g. ISO 2859-1, or by variables, e.g. ISO 3951 (all parts).

NOTE See the Bibliography for these and other ISO standards on statistics.

The selection of an appropriate scheme should be subject to agreement between parties.

3.4.2 Inspection by attributes consists of examining a sampled unit and deciding whether or not it achieves an appropriate criterion. A decision on the fitness for purpose of a batch is by counting the number of non-achieving units in the batch sample. An example might be the presence of cracks (see EN 623-1) or surface blisters in an as-fired ceramic component. Sampling plans for inspection by attributes are given in ISO 2859-1.

3.4.3 Inspection by variables involves the measurement of a property or properties using a recognised test method producing numerical values for each unit in the sample. These values are used in conjunction with the sampling plan to decide on the fitness for purpose of a batch compared with a pre-set criterion. Typical properties measured in this type of inspection are density (see EN 623-2) and flexural strength (see EN 843-1). Schemes for sampling by variables are given in ISO 3951 (all parts).

NOTE ISO 5022 contains methods of sampling for shaped refractory products which can have some relevance to some types of advanced technical ceramic components.

3.4.4 Some tests involving determination of properties may be used to inspect by attributes by placing an upper or lower acceptance limit on the design value, e.g. a lower limit on density in any unit. The permitted deviation from the design value, based on known uncertainties in test result from manufacturing, selection and testing, should also be defined, e.g. if the known uncertainty in measurement of density is ± 20 kg m^{-3} at the 90 % confidence level, a unit would be deemed to have a satisfactory attribute if the measured density is not more than 20 kg m^{-3} below the selected design value.

3.4.5 In either type of inspection, it should be emphasised that the samples chosen have a random chance of selection, i.e. not selecting the most conveniently accessible units, and that the data provided also include elements of test method uncertainty (see ISO 5725 (all parts)). The latter is assumed to be reduced to a minimum by the standardisation of methods, but nevertheless is still inevitably present to varying degrees in most tests for advanced technical ceramics. Equally, it should be noted that sampling schemes do not guarantee that the test data are truly indicative of the quality of the sampled batch. They indicate only a probability, and thus contain an element of risk that a sample apparently meeting a given criterion may have been selected from a batch which overall does not, or that a sample apparently not meeting a given criterion may have been selected from a batch which overall does so. The important point is that the level of risk is calculated and known. The degree of risk may be different for supplier and customer but decreases with increasing severity of inspection, either by increased sample size or by testing more than one attribute and/or property.

3.4.6 Sampling for production consistency should be made at a time during production when it is known from process indications that the product is likely to have stable attributes. This clearly does not apply to small batch supplies of ceramic components.

3.5 Sampling attributes of physically large units or blocks of material

3.5.1 In some circumstances, test-pieces will need to be cut from large units or from supplied blanks or blocks of material. Consideration should be given not only to the position from which the test-pieces can be cut, but also the means for doing this, and the relationship the cut test-pieces have to the unit or block as a whole.

3.5.2 Many ceramic materials when supplied in an as-fired and unmachined condition possess a surface skin, which may be of different composition or have other different attributes from the bulk material exposed by cutting. Recognition shall be given to this factor, since it can influence the result of a test in various ways. Some examples of factors to consider include:

a) the skin may be impermeable, but the bulk not;

b) the process of cutting may change the mechanical condition of the test material, and relieve undetectable internal stresses;

c) the material may be structurally an isotropic, but test-pieces may be prepared only with orientations which are not relevant to the important attributes of the unit as a whole.

Even if the test unit or material has no discernibly different skin, cutting still may introduce flaws which are different to those pre-existing in the unit or block original surface. In addition, flaws internal to the unit or block, which shall have no influence on performance of the unit, may become exposed or positioned such that they influence the result of the test being applied.

3.5.3 Clear definition of cutting positions and methods shall be agreed between parties, and the potential consequences of the actions understood as far as practicable. Full records of the cutting scheme and test-piece preparation shall be recorded and shall be reported as part of the report on the assessment of attributes.

3.6 Relevant evaluation criteria for ceramic components

3.6.1 The fitness for purpose of a ceramic component may be defined by a number of criteria related to that purpose. Some typical ones are:

a) correct dimensions, within tolerances specified;

b) freedom from surface defects which would impede function;

c) freedom from cracks or edge chips which would weaken the component;

d) correct quality of surface form or finish (see EN 623-4);

e) adequacy of mechanical properties;

f) adequacy of other property attributes.

3.6.2 A batch of components should be sampled and its quality determined according to the criticality of that attribute. For example, one or more dimensions may be critical to permit the component to fit into another unit. In such a case, until confidence in dimensional consistency is acquired, especially if that dimension is "as-fired", it might be appropriate to check all units for those dimensions. When the consistency of preparation of the batch is understood, and it is found that the frequency of occurrence of units outside the specified tolerance is small, then a sampling scheme may be invoked to select a small proportion of units for checking.

3.6.3 Some attributes might always be tested on all components, especially when these occur randomly and with sufficient frequency for concern, and the test can be performed non-destructively. For example, dye penetration tests (see EN 623-1) might be undertaken routinely on all units in order to inspect for cracks or other surface defects such as porous regions. Flatness is another such example. Mechanical proof testing to ensure mechanical reliability can also be undertaken, but the basis for undertaking such testing shall be clearly defined and be shown to be effective (see annex A).

3.6.4 Whatever the circumstance, the rules for sampling units, the number or proportion of items in a batch to be tested and the criteria by which the batch is to be deemed fit for purpose should be by agreement between parties to the inspection. It is normally advisable to base inspection schemes on occasional sampling, or sampling by simple percentage, and a recognised statistically based sampling scheme should be used. Such schemes are based on the theory of probability, and on the hypothesis that the values of measured attributes of the selected units are distributed normally about the mean.

Annex A
(informative)

Mechanical proof-testing

A.1 In mechanically critical situations where the risk of failure might incur considerable loss, mechanical proof tests might be specified for use on all units. Proof testing relies on the assumption that those units with flaws which might be large enough to cause fracture in service can be removed from a batch by mechanically overloading each unit before it is used, and hence the units which survive the proof loading should survive in service to a level defined by the degree of overload applied. In performing such a test it has to be recognised that unless the loading applied to the component is distributed in exactly the same way that the component would see in practice, there is a risk that critical areas are inappropriately stressed. Further, the loading should be applied quickly and smoothly without overshoot, and then removed in the same manner, in order to minimise the risk of further damage being obtained in the test. The risks associated with proof testing are principally concerned with the assumption that the fracture-producing flaw distribution does not change in service. If it does, e.g. as a result of abrasion, corrosion, or impact, then proof-testing before service does not guarantee survival in service. Despite this, there remains the potential advantage of proof-testing in guaranteeing the surviving batch quality as it is put into service.

A.2 Careful consideration of proof-testing is required before specifying that it should be done. Notably, factors such as means of applying proof loads, damage to joints or connections to the unit, and controlling the environment surrounding the unit during test should be considered.

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