



SLOVENSKI STANDARD

SIST ENV 1006:2004

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

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

Hochleistungskeramik - Monolithische Keramik - Leitlinie zur Auswahl von Proben für die Beurteilung von Eigenschaften

Céramiques techniques avancées - Céramiques monolithiques - Guide pour la sélection des éprouvettes destinées à l'évaluation des propriétés

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English version

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

Céramiques techniques avancées - Céramiques
monolithiques - Guide pour la sélection des éprouvettes
destinées à l'évaluation des propriétés

Hochleistungskeramik - Monolithische Keramik - Leitlinie
zur Auswahl von Proben für die Beurteilung von
Eigenschaften

This European Prestandard (ENV) was approved by CEN on 21 November 2002 as a prospective standard for provisional application.

The period of validity of this ENV is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the ENV can be converted into a European Standard.

CEN members are required to announce the existence of this ENV in the same way as for an EN and to make the ENV available promptly at national level in an appropriate form. It is permissible to keep conflicting national standards in force (in parallel to the ENV) until the final decision about the possible conversion of the ENV into an EN is reached.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and United Kingdom.

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Foreword

This document (ENV 1006:2003) has been prepared by Technical Committee CEN/TC 184, "Advanced technical ceramics", the secretariat of which is held by BSI.

This European Prestandard supersedes ENV 1006:1993 with improved attention to the machining of test pieces from larger items and with the addition of an informative annex dealing with general issues of proof testing.

In this European Prestandard the annex A is informative.

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

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ENV 1006:2003 (E)**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.

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1 Scope

This European Prestandard 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 Normative references

This European Prestandard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Prestandard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

ISO 3951, *Sampling procedures and charts for inspection by variables for percent nonconforming*.

NOTE Additional ISO Standards for statistical analysis are listed in the Bibliography.

3 Terms and definitions

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

3.1

batch

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

NOTE Sometimes referred to as a "lot".

3.2

sample

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

3.3

sample size

number of units in a sample

4 Selection of test-pieces

4.1 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 Prestandard to define criteria for fitness for purpose. This is subject to agreement between parties.

4.2 Material homogeneity and anisotropy

4.2.1 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

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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.

4.2.2 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).

4.2.3 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.

4.3 Test method accuracy

4.3.1 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.

4.3.2 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.

4.3.3 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. ENV 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.

4.4 Sampling schemes for individual manufactured items

4.4.1 Sampling schemes are conventionally divided into those for inspection by attributes, e.g. ISO 2859-1, or by variables (e.g. ISO 3591). The selection of an appropriate scheme should be subject to agreement between parties.

4.4.2 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 2589-1.

4.4.3 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.

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

4.4.4 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 $\pm 20 \text{ kg m}^{-3}$ 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.

4.4.5 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). 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.

4.4.6 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.

4.5 Sampling attributes of physically large units or blocks of material

4.5.1 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.

4.5.2 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:

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- 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.

4.5.3 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.

4.6 Relevant evaluation criteria for ceramic components

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

- correct dimensions, within tolerances specified;
- freedom from surface defects which would impede function;
- freedom from cracks or edge chips which would weaken the component;
- correct quality of surface form or finish (see ENV 623-4);