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Sodobna tehniļ na keramika - Monolitna keramika - Mehanske lastnosti pri sobni temperaturi ï 5. del: Statistiļ na analiza

Advanced technical ceramics - Mechanical properties of monolithic ceramics at room temperature - Part 5: Statistical analysis

Hochleistungskeramik - Mechanische Eigenschaften monolithischer Keramik bei Raumtemperatur - Teil 5: Statistische Auswertung

Céramiques techniques avancées - Propriétés mécaniques des céramiques monolithiques a température ambiante - Partie 5: Analyse statistique

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Advanced technical ceramics - Mechanical properties of monolithic ceramics at room temperature - Part 5: Statistical analysis

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

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Foreword

This document (EN 843-5:2006) 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of an identical text or by endorsement, at the latest by June 2007, and conflicting national standards shall be withdrawn at the latest by June 2007.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 843-5:1996.

EN 843 *Advanced technical ceramics — Mechanical properties of monolithic ceramics at room temperature* comprises six parts:

Part 1: *Determination of flexural strength*

Part 2: *Determination of Young’s modulus, shear modulus and Poisson’s ratio*

Part 3: *Determination of subcritical crack growth parameters from constant stressing rate flexural strength tests*

Part 4: *Vickers, Knoop and Rockwell superficial hardness*

Part 5: *Statistical analysis*

Part 6: *Guidance for fractographic investigation*

At the time of publication of this Revision of Part 5, Part 6 was available as a Technical Specification.

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.

1 Scope

This part of EN 843 specifies a method for statistical analysis of ceramic strength data in terms of a two-parameter Weibull distribution using a maximum likelihood estimation technique. It assumes that the data set has been obtained from a series of tests under nominally identical conditions.

NOTE 1 In principle, Weibull analysis is considered to be strictly valid for the case of linear elastic fracture behaviour to the point of failure, i.e. for a perfectly brittle material, and under conditions in which strength limiting flaws do not interact and in which there is only a single strength-limiting flaw population.

If subcritical crack growth or creep deformation preceding fracture occurs, Weibull analysis can still be applied if the results fit a Weibull distribution, but numerical parameters may change depending on the magnitude of these effects. Since it is impossible to be certain of the degree to which subcritical crack growth or creep deformation has occurred, this European Standard permits the analysis of the general situation where crack growth or creep may have occurred, provided that it is recognized that the parameters derived from the analysis may not be the same as those derived from data with no subcritical crack growth or creep.

NOTE 2 This European Standard employs the same calculation procedures as method A of ISO 20501:2003 [1], but does not provide a method for dealing with censored data (method B of ISO 20501).

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 843-1:2006, *Advanced technical ceramics — Mechanical properties of monolithic ceramics at room temperature — Part 1: Determination of flexural strength*

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 843-1:2006 and the following apply.

NOTE Definitions of additional statistical terms can be found in ISO 2602 [2], ISO 3534-1 [3], or other source literature on statistics.

3.1 Flaws

3.1.1
flaw
inhomogeneity, discontinuity or structural feature in a material which when loaded provides a stress concentration and a risk of mechanical failure

NOTE 1 This could be, for example, a grain boundary, large grain, pore, impurity or crack.

NOTE 2 The term flaw should not be taken as meaning the material is functionally defective, but rather as containing an inevitable microstructural inhomogeneity.

3.1.2
critical flaw
flaw acting as the source of failure

3.1.3
extraneous flaw
type of flaw observed in the fracture of test pieces manufactured for the purposes of a test programme which will not appear in manufactured components

NOTE For example, damage from machining when this process will not be used in the manufacture of components.

3.2 Flaw distributions

3.2.1
flaw size distribution
spread of sizes of flaw

3.2.2
critical flaw size distribution
distribution of sizes of critical flaws in a population of tested components

3.2.3
compound critical flaw distribution
flaw distribution which contains more than one type of strength controlling flaw not occurring in a purely concurrent manner (3.2.4)

NOTE An example is when every test piece contains flaw type A and some contain additionally a second independent type B.

3.2.4
concurrent critical flaw distribution
competing critical flaw distribution.

Multiple flaw distribution where every test piece contains representative defects of each independent flaw type which compete with each other to cause failure

3.2.5
exclusive critical flaw distribution
multiple flaw distribution created by mixing and randomizing test pieces from two or more versions or batches of material where each version contains a single strength-controlling flaw population

NOTE For example, each test piece contains defects exclusively from a single distribution, but the total data set reflects more than one type of strength-controlling flaw.

3.2.6
competing failure mode
distinguishably different type of fracture initiation event that results from concurrent (competing) flaw distributions (3.2.4)

3.3 Mechanical evaluation

3.3.1
fractography
analysis of patterns and features on fracture surfaces, usually with the purpose of identifying the fracture origin and hence the flaw type

3.3.2
proof test
application of a predetermined stress to a test piece or component over a short period of time to ascertain whether it contains a serious strength-limiting defect

NOTE This enables the removal of potentially weak test pieces or components from a batch. This procedure modifies the failure statistics of the survivors, such that the two-parameter Weibull distribution is typically no longer valid.

3.3.3
population mean
average of all strength results in a population

3.3.4
sample mean
average of all strength results from a sample taken from the population

3.3.5
strength population
ensemble of fracture strengths

3.4 Statistical terms

3.4.1
bias
consistent numerical offset in an estimate relative to the true underlying value, inherent in most estimating methods

NOTE For the maximum likelihood method of estimation, the magnitude of the bias decreases with increasing sample size.

3.4.2
confidence interval
interval for which it can be stated with a given confidence level that it contains at least a specified proportion of the population of results, or estimates of parameters defining the population

NOTE For example, estimates of Weibull modulus and characteristic strength from a batch of test pieces.

3.4.3
confidence level
required probability that any one estimate will fall within the confidence interval

3.4.4
estimate
well-defined value that is dependent on the variation of strengths in the population

NOTE The resulting value for a given population can be considered an estimate of a distribution parameter associated with the population as a whole.

3.4.5
probability density function
function $f(x)$ is a probability density function for the continuous random variable x if:

$$f(x) \geq 0 \tag{1}$$

and:

$$\int_{-\infty}^{\infty} f(x)dx = 1 \tag{2}$$

such that the probability, P, that the random variable x assumes a value between a and b is given by:

$$P(a < x \leq b) = \int_{a}^{b} f(x)dx = F(b) - F(a) \tag{3}$$

where F is the cumulative distribution function

3.4.6
ranking estimator
means of assigning a probability of failure to a ranked value in a collection of strength values

3.4.7
sample
collection of measurements or observations on test pieces selected randomly from a population

NOTE For example, strength measurements from a batch of similar test pieces.

3.4.8
sampling
process of selecting test pieces for a test

NOTE For the purposes of this European Standard the guidance given in ENV 1006 [4] should be noted.

3.4.9
unbiased estimate
estimate of a distribution parameter which does not contain a bias or which has been corrected for bias

3.5 The Weibull distribution

3.5.1
Weibull distribution
The continuous random variable *x* has a two-parameter Weibull distribution if the probability density function (see 3.4.5) is given by:

$$f(x) = \left(\frac{m}{\beta}\right)\left(\frac{x}{\beta}\right)^{m-1} \exp\left[-\left(\frac{x}{\beta}\right)^{m}\right] \qquad x > 0 \tag{4}$$

$$f(x) = 0 \qquad x \le 0 \tag{5}$$

NOTE 1 This corresponds with a cumulative distribution function as follows:

$$F(x) = 1 - \exp\left[-\left(\frac{x}{\beta}\right)^{m}\right] \qquad x > 0 \tag{6}$$

$$F(x) = 0 \qquad x \le 0 \tag{7}$$

where

m is the Weibull modulus or shape parameter (> 0);

β is the scale parameter (> 0).

NOTE 2 The random variable representing the fracture strength of a ceramic test piece will assume only positive values, and the distribution is asymmetric about the mean. These characteristics rule out the use of the normal distribution amongst others and point to the use of the Weibull distribution or similar skewed distributions. The assumption made in this European Standard is that the Weibull distribution will approximate to the true distribution of strengths observed.

NOTE 3 This European Standard is restricted to the use of the two-parameter Weibull distribution. Other forms, such as the three-parameter method which assumes the existence of a non-zero minimum value for *x*, are outside the scope of this European Standard.

NOTE 4 The population mean $\bar{x}$ is related to β by:

$$\bar{x} = \beta\Gamma\left(1+\frac{1}{m}\right) \tag{8}$$

where

Γ is the gamma function.

The gamma function is sometimes represented by a non-integral factorial:

$$\Gamma\left(1+\frac{1}{m}\right)=\left(\frac{1}{m}\right) \tag{9}$$

3.5.2
Weibull modulus
measure of the width of the Weibull distribution defined by parameter *m* in Equation (4)

3.5.3
Weibull characteristic strength
strength value at a probability of failure of 0,632

NOTE 1 If the random variable representing the strength of a ceramic test piece is characterized by the above equations, then the probability that a test piece will not sustain a nominal stress σ_{nom}, i.e. has a nominal strength $\sigma_f = \sigma_{nom}$, is given by the cumulative distribution function:

$$P_f = 1-\exp\left[-\left(\frac{\sigma_f}{\sigma_0}\right)^m\right] \qquad \sigma_f > 0 \tag{10}$$

$$P_f = 0 \qquad \sigma_f \le 0 \tag{11}$$

where

P_f is the probability of failure;

σ_0 is the Weibull characteristic strength.

NOTE 2 Defined in the above manner, the Weibull characteristic strength depends on the test piece geometry and on the multiaxiality of the stress field applied.

NOTE 3 When testing three-point and four-point bend test pieces from the same population, different values of σ_0 will be derived, reflecting different stressed volumes or surface areas in the two geometries. See Annex A for information on the theoretical relationship between strengths of test pieces of different stressed volumes or areas.

NOTE 4 Caution is needed in the use of Weibull statistical parameters beyond the population from which they are derived.

4 Symbols

For the purposes of this document, the following symbols apply.

A component surface area

A_{eff} effective component surface area

b unbiasing factor for Weibull modulus estimate

C_l lower limit of confidence interval for σ_0, i.e. $C_l = \hat{\sigma}_0 \exp\left(-\frac{t_l}{\hat{m}}\right)$

C_u upper limit of confidence interval for σ_0, i.e. $C_u = \hat{\sigma}_0 \exp\left(-\frac{t_u}{\hat{m}}\right)$

D_l lower limit of confidence interval for $\hat{m}$, i.e. $D_l = \hat{m}/l_l$

D_u upper limit of confidence interval for $\hat{m}$, i.e. $D_u = \hat{m}/l_u$

f probability density function

F cumulative probability distribution function

g function describing the normalised variation of stress over the volume (or area) of a component

i number assigned to an individual strength value of the sample in ascending ranked order

l_u, l_l factors for determining respectively the upper and lower limits of the confidence interval of $\hat{m}$

m Weibull modulus for the population

$\hat{m}$ estimate of m found by the maximum likelihood method

$\hat{m}_{cor}$ value of $\hat{m}$ corrected by factor b to provide an unbiased estimate of m

N number of tested test pieces

P_f failure probability of the test piece

t_u, t_l factors for determining respectively the upper and lower limits of the confidence interval of $\hat{\sigma}_0$

V volume

V_0 unit volume

V_{eff} effective volume

x random variable

$\bar{x}$ population mean of random variable x

α confidence level

β scale parameter

ε fractional accuracy required in determining maximum likelihood estimates of m and σ_0

σ_f strength of test piece

σ_{fi} strength of the i[th] ranked test piece in a population

σ_{fj} strength of the j[th] un-ranked test piece in a population

σ_{nom} nominal stress in test piece at instant of failure, usually taken to be equal to the fracture strength for the purposes of strength assessment

σ_{max} maximum stress in a component against which the stress distribution is referenced

σ_0 Weibull characteristic strength of test pieces

$\hat{\sigma}_0$ maximum likelihood estimate of Weibull characteristic strength of test piece

5 Significance and use

The strength of advanced technical ceramics is not usually a deterministic parameter. It depends on the nature, size and orientations of the flaws within the test piece relative to the stress field being applied. This European Standard applies to most monolithic advanced technical ceramics.

NOTE 1 The Weibull formalism can also be applied successfully in most cases to particulate and whisker reinforced ceramics which fracture in a catastrophic mode. However, in many cases the failure mechanisms in fibre-reinforced ceramic matrix composites preclude its use.

The purpose of this European Standard is to provide unbiased estimates of the parameters of the underlying strength distribution of a population of ceramic test pieces in order to assess numerically the scatter in strengths of the population. There are a number of ways of determining such estimates, including least squares, moments, and maximum likelihood methods. The maximum likelihood method has been found to be the most efficient estimator for small sample numbers based on producing a smaller coefficient of variation of Weibull modulus, *m*, and for this reason it is chosen in this European Standard.

NOTE 2 Use of other methods of estimating *m* and σ_0, such as least squares fitting of a straight line to the ranked data points as performed for the visual inspection (see 7.1), is not permitted by this European Standard because they provide less reliable estimates of *m*.

Many factors affect the numerical values characterising the distribution of fracture strengths. These include:

1. The number of tests taken as an indicator of the population. The reliability of the estimates increases with increasing size of the sample, but there are practical limits to the number of tests that might be employed for cost reasons to be balanced against the improvement in accuracy this produces. It is recommended that the sample size should not be less than 30.

2. The assumption is made that the sample of test pieces can describe the population by having critical flaws representative of the population. It should be recognised that the sampling made from the population shall be on a random basis to reflect fully the true distribution. For example, rejection of part of the population, e.g. by proof-testing, may modify the applicability of two-parameter Weibull statistics.

3. The method of preparation of test pieces for testing. Most test pieces contain more than one inherent flaw type and preparing the surfaces of the test pieces prior to testing, e.g. surface grinding, can add another type of flaw which may change the dominance of the inherent flaws. Concurrent flaw distributions result in competing failure modes which vary in dominance depending on preparation methods.

4. Under identical conditions of testing, two data sets derived from the same population will result in different values of $\hat{m}$ and $\hat{\sigma}_0$ due to the natural scatter in sampling from the population. For the purposes of this European Standard, the values of $\hat{m}$ and $\hat{\sigma}_0$ for the two sets shall be deemed to be equivalent at the same confidence level if the results of one lie within the confidence interval of the other, or vice versa.

It is often the case that concurrent, compound or exclusive flaw distributions exist in a population. These can lead to a bimodal or multimodal distribution of strengths, perhaps with some test pieces failing from one type of flaw, and others from a second type. In such cases a single two-parameter Weibull distribution cannot validly be fitted to the data. This European Standard incorporates a visual inspection method (see 7.1) based on simple data plotting to make the decision whether a Weibull analysis can usefully be made.

NOTE 3 Method B of ISO 20501 [1] deals with the case of 'censored statistics', e.g. where it has been possible fractographically to identify several competing flaw distributions within a batch of test pieces, such that each test can be assigned to a given flaw type. To compute the Weibull parameters associated with each flaw type, it is necessary effectively to suspend the tests which failed prematurely from other flaw types, but include them in the computation on the basis that they contained the flaw type being analysed, but at an unknown strength level. This is known as 'right censoring' (higher data become unknown quantities). An alternative approach is needed in the mathematical analysis.

6 Principle of calculation

6.1 Maximum likelihood method

Once it is determined that a valid two-parameter Weibull distribution can be fitted to the data set being evaluated (see 7.1), the maximum likelihood estimates of Weibull modulus, $\hat{m}$, and characteristic strength, $\hat{\sigma}_0$, can be determined.

The likelihood function L for a single critical flaw distribution is given by the expression:

$$L = \prod_{j=1}^{N} \left(\frac{m}{\sigma_0} \right) \left(\frac{\sigma_{fj}}{\sigma_0} \right)^{m-1} \exp \left[- \left(\frac{\sigma_{fj}}{\sigma_0} \right)^{m} \right] \tag{12}$$

where

N is the number of fracture data.

This function is maximized by differentiating the log likelihood ($\ln(L)$) with respect to m and σ_0, and setting these functions to zero yielding, respectively, estimates $\hat{m}$ and $\hat{\sigma}_0$, for m and σ_0:

$$\frac{\sum_{j=1}^{N} \sigma_{\mathrm{fj}}^{\hat{m}} \ln \sigma_{\mathrm{fj}}}{\sum_{j=1}^{N} \sigma_{\mathrm{fj}}^{\hat{m}}} - \frac{1}{N} \sum_{j=1}^{N} \ln \sigma_{\mathrm{fj}} - \frac{1}{\hat{m}} = 0 \tag{13}$$

and

$$\hat{\sigma}_0 = \left[\left(\sum_{j=1}^{N} \sigma_{fj}^{\hat{m}} \right) \frac{1}{N} \right]^{1/\hat{m}} \tag{14}$$

Equation (13) is solved numerically to obtain a solution for $\hat{m}$, which can then be used to solve for $\hat{\sigma}_0$ through Equation (14). The required fractional accuracy of solution (ε) shall be $\leq$ 0,001, giving three significant digits in the value of $\hat{m}$.

A computer may be used for this task.