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Sodobna tehnična keramika - Monolitna keramika - Mehanske lastnosti pri sobni temperaturi – 5. del: Statistična analiza

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

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Hochleistungskeramik - Monolithische Keramik - Mechanische Eigenschaften bei Raumtemperatur - Teil 5: Statistische Analysis

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Foreword

This document (prEN 843-5:2004) has been prepared by Technical Committee CEN/TC 184 "Advanced technical ceramics", the secretariat of which is held by BSI.

This document is currently submitted to the CEN Enquiry.

This document will supersede ENV 843-5:1996.

1 Scope

This standard describes 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 strictly to be 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 Standard permits the analysis of the general situation where crack growth or creep may have occurred, provided that it is recognised 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 standard employs the same calculation procedures as ISO 20501:2003 (see annex J, Ref. [1]), method A, but does not provide a method for dealing with censored data, Method B.

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, *Advanced technical ceramics — mechanical properties of monolithic ceramics at room temperature — Part 1: Determination of flexural strength*

EN 1006, *Advanced technical ceramics — methods of testing monolithic ceramics — guidance on the selection of test-pieces for the evaluation of properties*

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

ISO 2602, *Statistical interpretation of test results — estimation of the mean: confidence interval*

ISO 3534-1, *Statistics — vocabulary and symbols — probability and general statistical terms*

3 Definitions

For the purposes of this standard the following definitions apply.

Definitions of additional statistical terms may be found in ISO 2602, ISO 3534-1, or other source literature on statistics.

3.1 Flaws

3.1.1
flaw
inhomogeneity, discontinuity or structural feature, e.g. a grain boundary, large grain, pore, impurity or crack, in a material which when loaded provides a stress concentration and a risk of mechanical failure

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

3.2.4
concurrent (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. Thus 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 result 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, and hence 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; in the present case, 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. 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
the function *f(x)* is a probability density function for the continuous random variable *x* if:

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

and:

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

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, e.g. strength measurements from a batch of similar test-pieces

3.4.8
sampling
process of selecting test-pieces for a test. For the purposes of this standard the guidance given in EN 1006 shall 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 Weibull distribution

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

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

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

This corresponds with a cumulative distribution function:

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

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

where: *m* is the Weibull modulus or shape parameter (> 0)

β is the scale parameter (> 0)

NOTE 1 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 standard is that the Weibull distribution will approximate to the true distribution of strengths observed.

NOTE 2 This 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 Standard.

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.

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

$$\Gamma\left(1+\frac{1}{m}\right)=\left(\frac{1}{m}\right)! \tag{8A}$$

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:

$$P_f = 1 - \exp\left[-\left(\frac{\sigma_f}{\sigma_0}\right)^m\right] \qquad \sigma_f > 0 \tag{9}$$

$$P_f = 0 \qquad \sigma_f \leq 0 \tag{10}$$

where: P_f is the probability of failure

σ_0 is the Weibull characteristic strength (at P_f = 0,6321), acting as the scale parameter

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

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

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 I for information on the theoretical relationship between strengths of test-pieces of different stressed volumes or areas.

4 Symbols

The symbols used in this standard are defined below:

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 ${}_0$

C_u upper limit of confidence interval for ${}_0$

D_l lower limit of confidence interval for

D_u upper limit of confidence interval for

f probability density function

F cumulative probability distribution function

g a 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 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 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 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 must 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 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 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 Basis of method

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:

$$L = \prod_{j=1}^{N} \left(\frac{m}{\sigma_0}\right)\left(\frac{\sigma_{fj}}{\sigma_0}\right)^{m-1} \exp\left[-\left(\frac{\sigma_{fj}}{\sigma_0}\right)^{m}\right] \qquad (11)$$

where *N* is the number of fracture data.

This function is maximised 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:

$$\frac{\sum_{j=1}^{N} \sigma_{\mathrm{fj}}^{\hat{m}} \ln \sigma_{\mathrm{fj}}}{\sum_{j=1}^{N} \sigma_{\mathrm{fj}}^{\hat{m}}} - \frac{1}{N}\sum_{j=1}^{N} \ln \sigma_{\mathrm{fj}} - \frac{1}{\hat{m}} = 0 \qquad (12)$$

and

$$\hat{\sigma}_0 = \left[\left(\sum_{j=1}^{N} \sigma_{fj}^{\hat{m}}\right)\frac{1}{N}\right]^{1/\hat{m}} \qquad (13)$$

Equation (12) must be solved numerically to obtain a solution for $\hat{m}$, which can then be used to solve for $\hat{\sigma}_0$ through equation (13). The required fractional accuracy of solution (ε) shall be ≤ 0,001, giving three significant digits in the value of $\hat{m}$.

A computer may be used for this task. The proper implementation of any computer program shall be checked by employing the example data in Annex G for the defined level of accuracy, *ε.*

NOTE 1 The computer programs in annexes A to C incorporate appropriate routines for the interval halving method for numerically solving for $\hat{m}$ and $\hat{\sigma}_0$. They may need to be modified to suit different computer systems.

NOTE 2 As an alternative to the interval halving method, a Newton-Raphson method of solution may be employed. These two methods are known to provide equivalent results within the accuracy requirements of this standard.

6.2 Bias correction

The estimate $\hat{m}$ provided by this method has a bias which gives an overestimate of the true Weibull modulus m. It is necessary to correct it using an unbiasing factor tabulated in annex D. This unbiasing factor has been determined by a Monte Carlo method sampling randomly from a large population with a predetermined true value of *m*, allowing correction of the biased value $\hat{m}$ to the corrected value $\hat{m}_{cor}$:

$$\hat{m}_{cor} = \hat{m}.b \qquad (14)$$