

THE MINIATURISATION OF THE 4-POINT-BEND TEST

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Abstract—Strength measurements are becoming increasingly important for electroceramics. Bending of specimens small enough to be cut out of small electroceramic components may be one possibility. Therefore the miniaturisation of the 4-point bend-test for ceramic specimens is now being attempted. In this paper the errors in determining the flexural strength arising from the test principle itself, plus the geometry and measuring inaccuracies are calculated and expressed as a function of the outer span length. Contact pressure and a tolerable total measuring inaccuracy determines the dimensions of miniature specimens and fixtures. The possibilities of appropriate specimen preparation are also investigated.

Ceramic materials show a volume (i.e. a specimen size) dependence of strength which is described by Weibull's statistical theory. The applicability of the miniature bend-fixtures is demonstrated by measuring this volume effect.

Keywords—Electroceramics; Miniaturisation tests; Weibull statistics.

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Abstract—Strength measurements become more and more important for electroceramics. Bending specimens small enough to be cut out of the usually small electroceramic components may be a simple possibility. Therefore the miniaturisation of the 4-point bend-test for ceramic specimens is being analysed. The errors in determined flexural strength arising from the test principle itself, geometry and measuring inaccuracies are calculated and expressed depending on the outer span length. Contact pressure and a tolerable total measuring inaccuracy motivates dimensions of miniature specimens and fixtures. The possibilities of appropriate specimen preparation are investigated.

Ceramic materials show a volume, i.e. specimen size, dependence of strength which is described by Weibull's statistical theory. The applicability of the miniature bend-fixtures is demonstrated by measuring this volume effect.

NOMENCLATURE

b = specimen's breadth
 E_1, E_2 = Young's moduli of two different materials
 F = load at failure
 h = specimens height
 l, L = inner and outer span length
 m = Weibull exponent
 M_b = maximum bending moment
 p = contact pressure
 P_F = probability of failure
 r = roller radius
 V, V_0, V_E = volume, normalising volume, effective volume
 W_{\max} = maximum section modulus of bend specimen
 x, y, z = rectangular coordinates
 $\varepsilon, \varepsilon_i, \varepsilon_{\text{ran}}$ = total error, error caused by one single source, random error
 ν = Poisson's ratio
 μ_k, μ_r = coefficient of kinetic and rolling friction
 σ = stress
 σ_0, σ_y = characteristic strength, yield strength
 $\sigma_b, \sigma_{x,i}$ = maximum flexural stress by simple beam theory and actual stress

INTRODUCTION

To gain uniaxial strength data of ceramic materials, flexural testing of beams with rectangular cross-section in 4-point geometry is a simple and widely used standardised method [1]. Some ceramic materials -those for structural applications, where high mechanical loads are

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expected- have been designed to exhibit high strength values. Often components from such ceramics are large enough to cut out specimens of the required size. Another class of ceramic materials is designed to exhibit optimised electric or magnetic properties. Despite of the electric or magnetic loading, such components may fail due to mechanical stresses building up as a consequence ohmic heating. Therefore emphasis has to be put on improving and measuring their mechanical properties. Since electroceramic components are usually too small for commonly used flexure specimens to be cut out of them, strength testing has to be performed by other test methods, e.g. by laser heating of a disc-specimen, as proposed by Schneider [2], by component testing [3], or by testing bending specimens that are small enough. In this work the last method is pursued because of the fact the 4-point bend-test is well investigated [4-6] and to be carried out easily. Small specimens cut out from different locations of a large component will furthermore allow a locally resolved determination of strength.

The down-scaling of the common 4-point bend-test set-up is limited by several factors. The contact pressure between loading rollers and specimen should not exceed the yield strength of the fixture material or the compressive strength of the tested ceramic. Systematic errors in determined flexural strength arise as a consequence of an inadequate description of the testing principle. They could be taken into account on an average when the flexural strength is calculated. Random errors occur due to inexact knowledge of span lengths, specimen geometry and force. Based on the detailed work by Baratta et al. [4] the errors are evaluated depending on specimen size and span length. An acceptable total error of $|\varepsilon| \approx 5\%$ motivates the size of the test set-up. Such a testing device has been built.

A different problem originates in the defect controlled failure which is typical for ceramics and also the reason for the volume dependence of strength. Ceramic specimens break due to inherent defects. The tensile surface of the specimens has to be prepared in such a way that no defects that may cause fracture are introduced. The volume dependence of strength, as described by Weibull's theory [7], will be measured with two specimen sizes. Together with failure origins of the same type in both specimen sets this volume dependence should prove the fitness for purpose of the miniature 4-point bend fixtures.

LIMITATIONS AND ERRORS IN FLEXURAL TESTS

The errors associated with flexure tests have been analysed and calculated in an exemplary way by Baratta et al. [4]. Their objective was to define conditions to minimise the error for an approximately given specimen size. Basing on their calculations we want to determine how the total error depends on span lengths and specimen size in order to find out the minimal specimen size which can be tested without putting up with an unacceptable error.

The flexural strength σ_b will be calculated as the maximum outer fibre stress according to the simple beam theory [8]:

$$\sigma_b = \frac{M_b}{W_{\max}} = \frac{F \cdot (L-l)}{4 \cdot W_{\max}} \quad (1)$$

F is the maximum load at failure and L and l are the outer and inner span length respectively, see fig. 1. For the section modulus W_{\max} the correct value for a cross-section with two chamfers on the tensile side should be used. The relative error in percent due to each single source (i) is calculated as

$$\varepsilon_i = \frac{\sigma_{x,i} - \sigma_b}{\sigma_b} \cdot 100 \quad , \quad (2)$$

with $\sigma_{x,i}$ the actual stress and σ_b according to eq. (1). The individual errors are supposed to be independent and the total error ε should be the sum of all individual errors. $\varepsilon_i < 0$ indicates an overestimation of flexural strength with the simple beam formula, $\varepsilon_i > 0$ an underestimation.

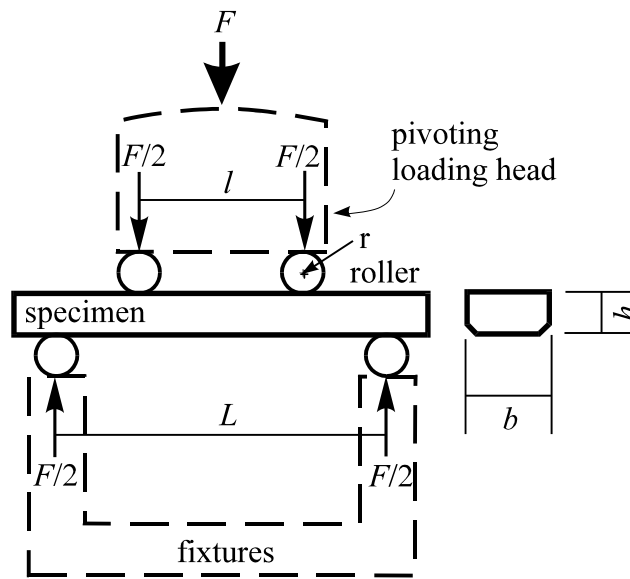


Fig. 1. 4-point bend-test set-up: L outer span length, l inner span length, b specimen's breadth, h specimen's height, r roller radius, F load.

Some considerations about the construction of the fixtures, fig. 1 were made before any calculations started [9]. It was decided to use an inner span of $l = L/3$, because this set-up is easy in handling, especially when dimensions become small, and less sensitive to positioning errors. All four free rollers (hardened steel) are held in place by a guide until a small initial load is applied. Three of them are supported by cambered faces to avoid twisting of the specimen around its longitudinal axis. A pivoting loading head was used. The fixture, loading head and guide were made from steel because of its good machinability which allows sufficient small tolerances.

Limitation contact stress

A limiting factor in down-scaling the 4-point bend test which is likely to be underestimated is the high compressive stress between rollers and specimen or rollers and supporting fixture respectively. To avoid plastic deformation and thus ruining of the fixture or local crushing of the specimen, it should not exceed the yield strength of the steel fixture or the compressive

strength of the tested ceramic. The pressure p depends on the elastic properties of the involved materials (E_1, E_2), the load (or flexural strength σ_b if the volume effect is neglected), the specimen's height h and the roller diameter r [10]:

$$p = h \cdot \sqrt{\frac{\sigma_b \cdot E_1 E_2}{2r \pi L (1 - \nu^2)(E_1 + E_2)}} \quad (3)$$

Two cases for the contact ceramic/roller are considered in fig. 2: the solid line has been calculated for typical values for an electroceramic material ($E_1 = 100$ GPa and $\sigma_b = 100$ MPa) on hardened steel rollers and fixtures with $E_2 = 210$ GPa, the dashed line for a high strength ceramic (e.g. dense SiC with $E_1 = 400$ GPa and $\sigma_b = 1000$ MPa or dense Si₃N₄ with $E_1 = 330$ GPa and $\sigma_b = 1100$ MPa [11]). The dotted line refers to the contact fixture/roller in the latter case. All calculations were made for a specimen height of $h = 1.5$ mm and $\nu = 0.27$. From fig. 2 it follows that crushing of the specimen will not occur before plastic deformation of the fixture. For a steel test-jig and for testing high strength ceramics the outer span length is therefore limited to approximately 12 mm.

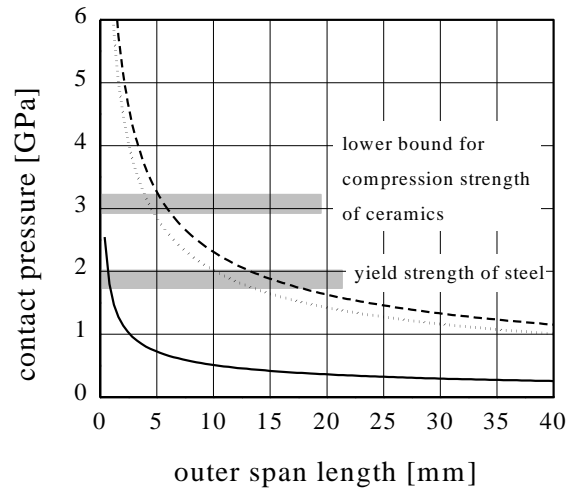


Fig. 2. Contact pressure between ceramic and steel roller (low strength ceramic: solid, high strength ceramic: dashed) and steel roller and steel fixture (dotted).

Systematic errors

Wedging stresses arise from load application only on the surface of the specimen and not over the cross-section, fig. 3a. They lead to additional tensile stresses on the tensile surface. Fig. 3a compares the ideal loading through pure bending moments and the actual situation. Usually only specimens that fracture within the inner span length are used for the evaluation. This procedure will very likely also exclude specimens that fracture in the region where $\sigma_{x,1} < \sigma_b$ (fig. 3a). As a consequence only the positive portion of the error due to wedging stresses will be calculated [4] and taken into account. For height to length ratios $h/L \approx 0.1$ this error is approximately $\epsilon_1 \approx 1.2\%$.

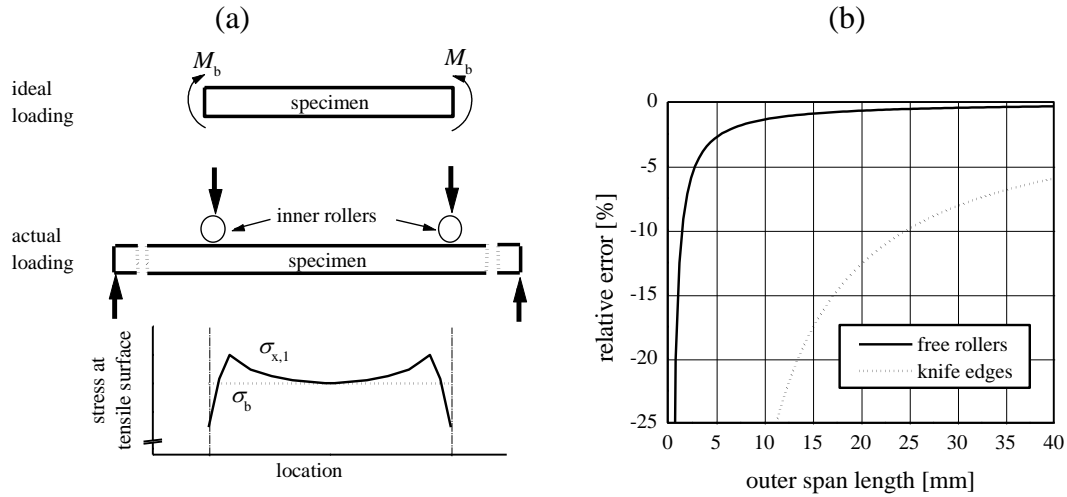


Fig. 3. Systematic errors: (a) Wedging stress: actual stress $\sigma_{x,1}$ on the tensile surfaces with respect to the wedging stress for the actual loading situation (solid), stress σ_b according to the simple beam theory and pure bending (dotted). (b) Error ε_3 due to friction calculated for $\mu_k = 0.5$ (knife edges) and $\mu_r = 0.03$ (free rollers).

Deflection of the specimen during the test leads to a tangential shift of the contact points between rollers and specimen. The contact points on the lower rollers move inwards, those on the inner rollers outwards, thus resulting in a smaller outer and a larger inner span length respectively. The corresponding error depends on the ratio of roller radius to specimen height. It is in the order of $\varepsilon_2 \approx 1.0\%$ if $r/h \approx 1.0$ and $l = L/3$ [4].

Friction at the points of load application gives rise to moments acting out of plane on the beam at this points and to additional axial forces [6]. The corresponding error may be calculated as [4]

$$\varepsilon_3 = -\frac{\mu}{\frac{(L-l)}{2h} - \mu} \cdot 100 \quad . \quad (4)$$

The plot in fig. 3b has been calculated for a specimen height of $h = 1.5$ mm, using the coefficient of kinetic friction $\mu_k = 0.5$ (as a typical value for steel/ceramic and steel/steel pairings [12]) for the case of load application via knife edges and the coefficient of rolling friction for free rollers. The coefficient of rolling friction is typically 15 to 50 times lower than the coefficient of kinetic friction [13, 14], so here the value $\mu_r = 0.03$ is used. The determination of friction coefficients is nevertheless a delicate problem and an uncertainty about their values will always remain. Fig. 3b shows clearly that the error due to friction becomes the more pronounced the smaller the outer span length is. Free rollers are therefore indispensable for small bend-jigs. For $h = 1.5$ mm, $L = 15$ mm and $l = L/3$ eq. (4) gives $\varepsilon_3 \approx -1\%$.

Even more errors may arise from violations of the simple beam theory: if for example the Young's modulus of the tested material is unequal in tension and compression or the maximum deflection is large compared to the beam height. These errors have been discussed

by Baratta et al. [4]. They are an order of magnitude smaller than those discussed above and will be neglected.

Random errors

Random errors arise from deviations of the test-jig geometry from ideal values, from the definite resolution of the screw gauge with which the specimen's dimensions are measured and the accuracy of the load cell. For these errors a maximum magnitude can be calculated.

Wrong span lengths, even if the roller pairs are properly centered with respect to each other, result in an altered bending moment. Eccentric loading with a pivoting loading head will result in a non-constant bending moment between the inner rollers [6]. From the design concept of the used fixtures it follows that the deviations of span lengths and the amount of eccentricity are determined by the clearance of the rollers in the slots of the guide, as shown in the inset in fig. 4a. This clearance is mainly determined by the used machining methods. A good workshop with standard equipment will be able to keep it to $\delta = 0.05$ mm, if not better. The resulting errors ε_4 and ε_5 for $\delta = 0.05$ mm are plotted in fig. 4a depending on outer span length for $l = L/3$. The errors are 25% to 50% larger for $l = L/2$.

Torsion of the specimen around its longitudinal axis is kept to a minimum by cambering three of the four faces that support the roller. The possible misalignment of the rollers is so small (because of the small clearance in the guide) that the resulting error of some tenths percent may be neglected.

For a rectangular cross section eq. (1) reads as follows:

$$\sigma_b = \frac{3F(L-l)}{2bh^2} \quad (5)$$

From eq. (5) is evident that the dimension measurement precision, with which breadth b and height h of the specimens are measured, enters in the error ε_6 . Fig. 4b shows this error ε_6 [4] for several breadths if the specimen's size is measured with a micrometer gauge to 0.01 mm. Neglecting the chamfers will result in an underestimation of strength of about 1%.

Since the precision of the determination of the maximum load is directly translated into the corresponding error ε_7 , the load cell has to be chosen to fit the expected maximum load. An accuracy of 0.1 % of the nominal load will lead to an error $|\varepsilon_7| = 0.1\%$.

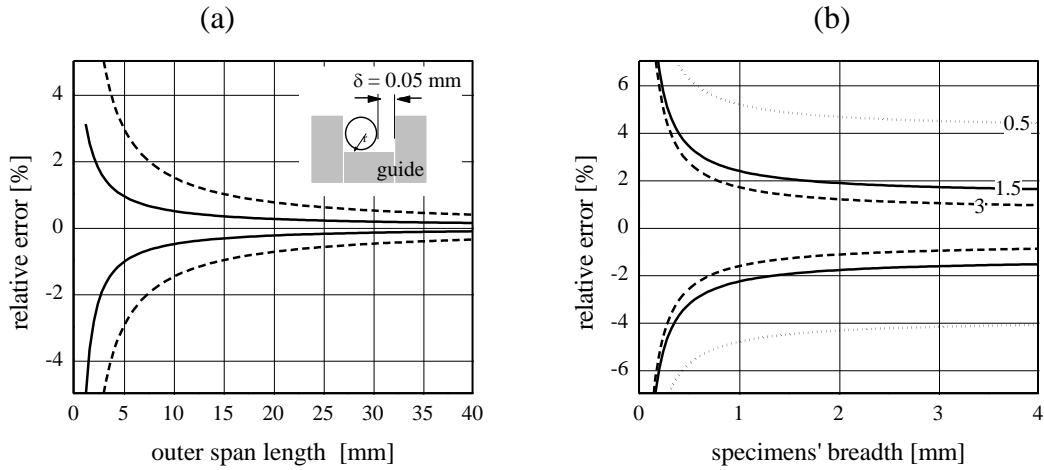


Fig. 4. Random errors. (a) Error due to wrong spans (dashed lines) and eccentric loading (solid lines). (b) Error due to definite (0.01 mm) resolution of the screw gauge depending on specimen's breadth for different indicated heights.

Test jig

Considering the above discussed limitation and errors "small" flexure fixtures with $L = 13$ mm, $l = 13/3$ mm and $r = 1$ mm for specimens with $b = 2$ mm and $h = 1.5$ mm and a length of 15 mm were built. With this configuration the total error may be between $-2.2\% < \varepsilon < 5\%$. This uncertainty is about three times larger (depending on the Weibull exponent) than the statistical uncertainty when 30 specimens are tested. With the usual DIN configuration [1] an error of approximately $-0.8\% < \varepsilon < 2.5\%$ has to be expected.

EXPERIMENTAL

Weibull statistics

Structural ceramics are known to show a decrease of characteristic strength with increasing specimen volume. This volume dependence of strength can be explained with fracture statistics concepts. In most cases Weibull statistics describe this effect satisfactory. The probability of failure P_F (Weibull distribution) for an uniaxial but inhomogeneous stress state can be written as [7]:

$$P_F = 1 - \exp \left[-\frac{1}{V_0} \int_V \left(\frac{\sigma}{\sigma_0} \right)^m dV \right] . \quad (6)$$

In terms of flexural strength σ_b and the effective volume V_E eq. (6) reads:

$$P_F = 1 - \exp \left[-\frac{1}{V_0} \left(\frac{\sigma_b}{\sigma_0} \right)^m \cdot V_E \right] . \quad (7)$$

The characteristic strength σ_0 and the Weibull exponent m are a measure for the position and width of the distribution respectively. The effective volume is the volume of a hypothetical tensile specimen which, when subjected to the stress σ_b , would have the same probability of failure, eq. (8):

$$V_E = \int_V \left(\frac{\sigma(x, y, z)}{\sigma_b} \right)^m dV . \quad (8)$$

It permits the comparison of strength values of two different sized and loaded specimens:

$$\frac{\sigma_1}{\sigma_2} = \left(\frac{V_{E,1}}{V_{E,2}} \right)^{1/m} . \quad (9)$$

Eq. (9) will be used to compare the strength values generated with two specimen sizes: standard DIN specimens and small specimens tested on the new developed test-jig.

Materials

Tests were made with the above described specimens (in future referred to as "small") and with standard DIN specimens [1] with $b = 4$ mm and $h = 3$ mm on fixtures with $L = 40$ mm and $l = 20$ mm. All specimens were cut out of larger ceramic plates. Three different materials were investigated: a fine grained dense silicon nitride, a medium grained alumina and a porous barium titanate with a bi-modal grain size distribution. Micrographs of all three materials are depicted in fig. 5. The silicon nitride and alumina were chosen because they are known to show the volume effect [15-17].

All sets were tested under such conditions that sub-critical crack growth [18] was avoided. The small and DIN specimens were investigated with respect to their surface quality, the fracture origins and the coincidence of characteristic strengths according to Weibull's theory.

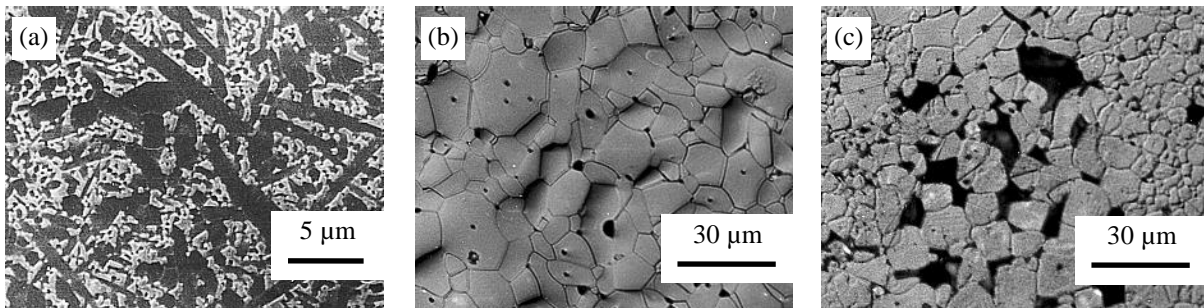


Fig. 5. Micrographs of the tested materials. (a) silicon nitride, (b) alumina, (c) barium titanate.

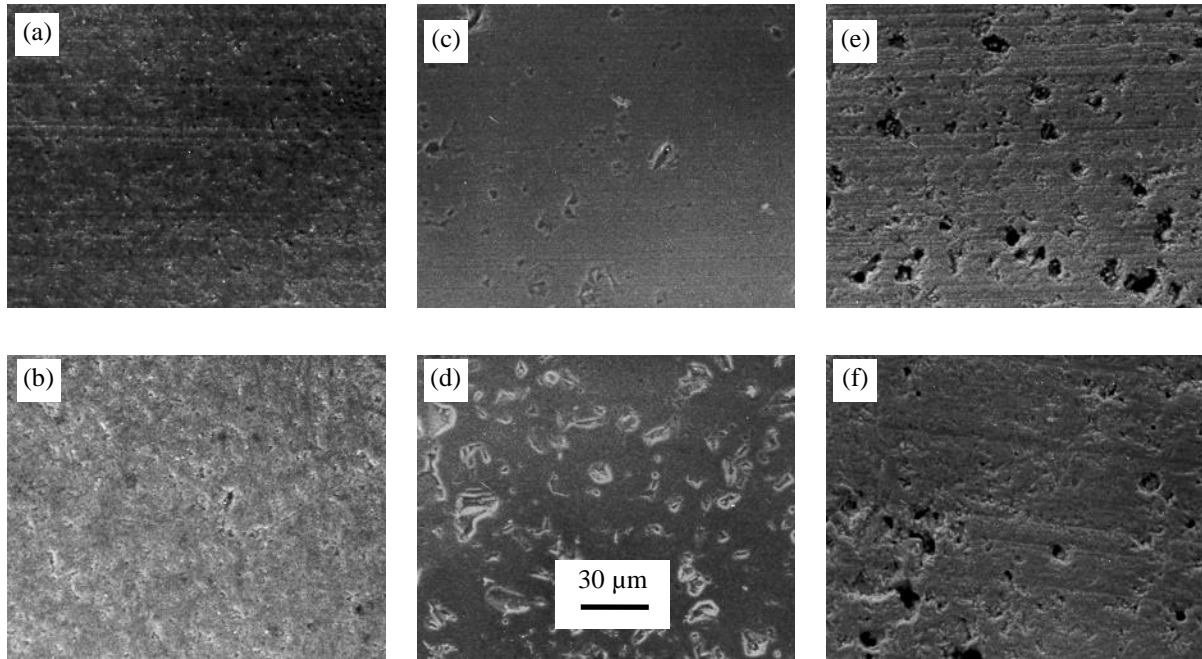


Fig. 6. Tensile surfaces, upper row: DIN specimens, lower row: small specimens. (a), (b) silicon nitride, (c), (d) alumina, (e), (f) barium titanate. The magnification is the same for all pictures.

Specimen preparation

In strength testing of ceramics it is assumed that failure starts from material inherent defects and not from artificially introduced surface defects [1]. Therefore special attention has to be paid to the preparation of the tensile surfaces of the specimens.

All specimens were manufactured by grinding with diamond tools. SEM pictures of tensile surfaces were used to compare the surface finish of the two specimen sizes. DIN-specimens were ground as proposed [1] with a D15 diamond grit finish. The resulting surface quality was used as a control reference for the small specimens. Small specimens were, in a first attempt, ground in the same way. If no comparable result could be obtained, the tensile surfaces were further polished with a 9 μm diamond paste. The resulting tensile surfaces are shown in fig. 6. On the DIN silicon nitride specimens machining grooves can be seen parallel to the specimen's longitudinal axis. The small silicon nitride specimens have no grooves because of a 9 μm polishing step after grinding. The alumina DIN specimens show grooves and some break-out. In the case of the small specimens it was impossible to avoid the large amount of break-out on the surfaces with the preparation means provided. The surfaces of both barium titanate sizes are comparable with grooves (slightly curved on the small specimens due to polishing) and pores.

Fractography

To assure that small and DIN specimens failed due to inherent defect populations the fracture surfaces were investigated in a SEM. Most of the silicon nitride and the barium titanate specimens -both DIN and small ones- failed due to inherent defects, sometimes large pores, sometimes poorly sintered, porous regions. About half of the alumina DIN-specimens also

fractured at pores, but only surface defects could be identified in the small alumina specimens. Examples for fracture origins in all three materials can be seen in fig. 7. From the viewpoint of failure origins the requirements for the presence of the volume effect, eq. (9) are fulfilled for the silicon nitride and the barium titanate but not for the alumina.

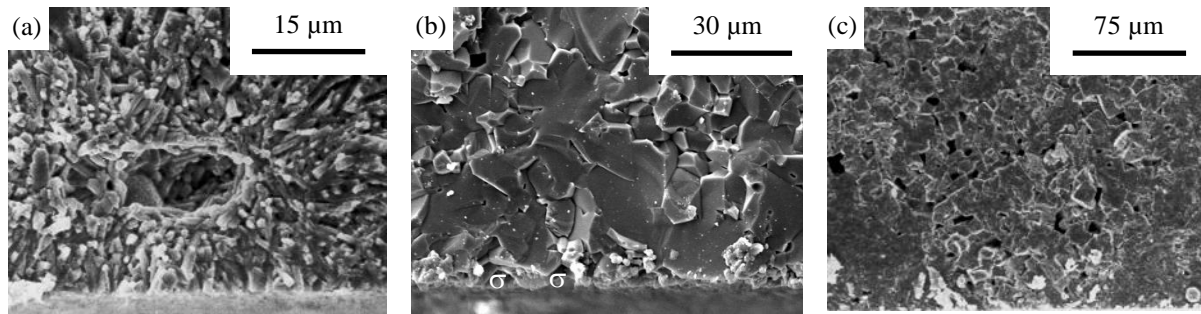


Fig. 7. Fracture origins. The tensile surface is at the bottom of the pictures. (a) silicon nitride: large pore, (b) alumina: surface defects, indicated by arrows, (c) barium titanate: coarse grain zone with increased porosity.

Strength

To allow a proper statistical evaluation thirty specimens of each kind were tested (with one exception: small alumina: 13). High strength materials were tested on a servohydraulic machine with a constant loading rate, low strength materials on a small universal testing machine with constant crosshead speed. The Weibull parameters were determined with the maximum likelihood method. The characteristic strengths and Weibull exponents of all materials, together with the corresponding 95%-confidence intervals are summarised in table 1. Details about testing conditions are also included.

Weibull diagrams and volume effect on strength according to eq. (9) are plotted in fig. 8.

DISCUSSION

Silicon nitride

The data of both sets of specimen sizes fit nicely to straight lines in the representation in fig. 8a, indicating that they can be described using Weibull statistics. Chi-square tests for both sets showed that the samples cannot be distinguished from their populations. The slopes of the lines (representing the Weibull exponent m) are nearly identical indicating, that the same defect population was responsible for failure. The characteristic strength of the DIN specimens is smaller than that of the small specimens. This can be explained by the defect controlled failure characteristic of ceramics: it is more likely to encounter a large defect which leads to a low strength value in a large specimen than in a small one. The volume dependence of the characteristic strength is plotted in fig. 8b. The solid line represents eq.(9), the diverging dashed lines the propagation of the statistical uncertainty for the 95% confidence interval. The error bars correspond to the measurement errors discussed above. The characteristic strength of the small specimens lies within the scatter band of the prediction

based on the DIN specimens values. The volume effect which has already been measured for silicon nitride [15, 16] is thereby confirmed. Thus the small test set-up is suitable for strength measurements.

Table 1: Results of strength measurements

Material	loading conditions	σ_0 [MPa] (95% confidence interval)	m (95% confidence interval)	eff. volume [mm³] (95% confidence interval)
Si ₃ N ₄ , DIN	100 MPa/s	802 (791 - 814)	23 (18 - 29)	5.12 (4.20 - 6.82)
Si ₃ N ₄ , small	1 mm/min \cong 100 MPa/s	882 (871 - 893)	28 (21 - 34)	0.24 (0.20 - 0.32)
Al ₂ O ₃ , DIN	1 GPa/s	398 (391 - 406)	17 (13 - 21)	6.98 (5.73 - 9.29)
Al ₂ O ₃ , small	15 mm/min \cong 1 GPa/s	429 (420 - 439)	25 (15 - 33)	0.27 (0.20 - 0.45)
BaTiO ₃ , DIN	2 mm/min \cong 10 MPa/s	93 (91 - 94)	21 (16 - 26)	5.60 (4.60 - 7.46)
BaTiO ₃ , small	0.2 mm/min \cong 10 MPa/s	89 (88 - 91)	21 (15 - 25)	0.33 (0.27 - 0.44)

Alumina

The alumina data too may be well described by Weibull statistics. The Weibull exponents differ apparently, fig. 8c, but since the confidence intervals overlap the difference is not significant. Although the volume effect of strength has been measured for alumina [17] it does not appear in our case as can be seen in fig. 8d. In all probability the strength distributions of small and DIN specimens do not match. This is confirmed by the investigations of failure origins and tensile surfaces. The small specimens seem to have fractured due to an artificial surface defect population - the large break-out regions, observable in fig. 8d. A large fraction of the DIN specimens failed due to inherent defects. This example shows clearly, how important -and also how difficult- an appropriate specimen preparation is.

Barium titanate

Each barium titanate data-set can be fitted well by a Weibull distribution. Both sets have the same Weibull exponent, but the strength of the DIN specimens is higher than the strength of the small specimens, fig. 8e. Therefore the expected volume effect does occur. The diagram in fig. 8f with errors-bars depicting the experimental error shows that the strengths of both specimen sizes are actual the same. The fracture origins are mostly large regions composed of large grains with an increased porosity, see also the micrograph in fig. 8c.

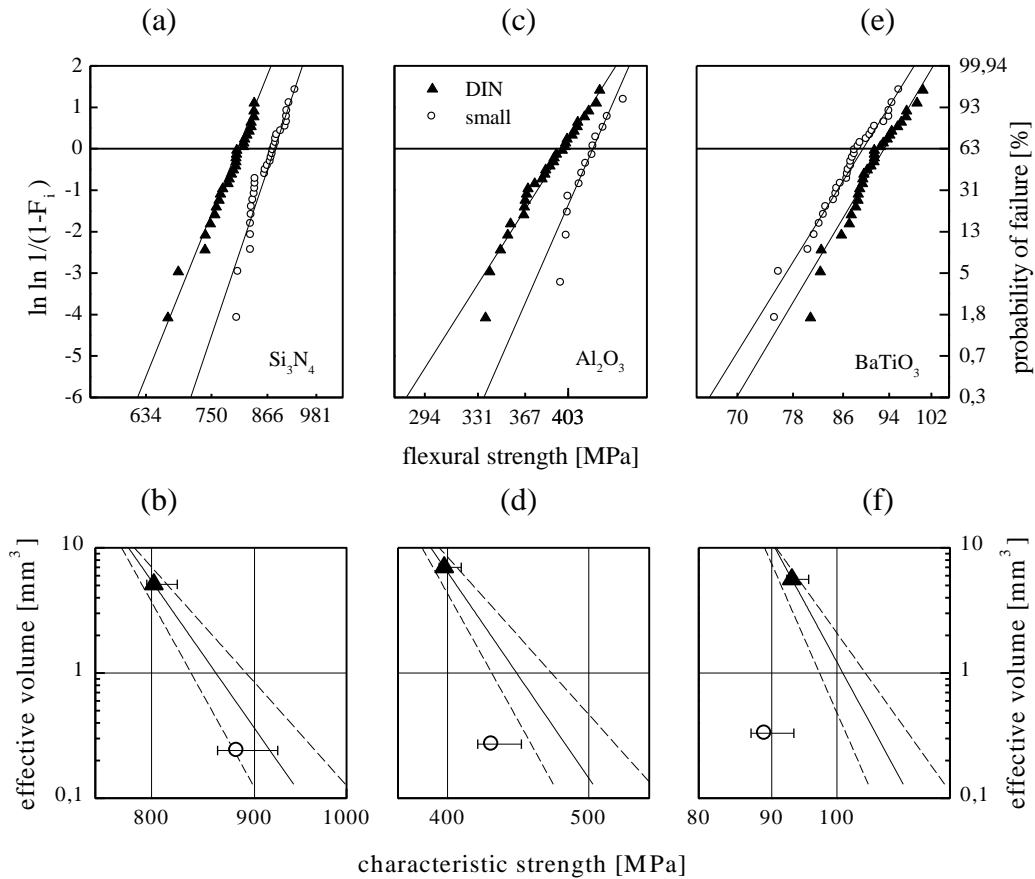


Fig. 8. Weibull plots and volume extrapolations. Triangles indicate datasets from DIN-specimens, open circles datasets from small specimens. (a) silicon nitride, (b) alumina, (c) barium titanate.

Sometimes more than one of these regions is present on the same fracture surface. One of the presumptions for the validity of Weibull statistics is a dilute solution of defects [19] so that they cannot interact. This presumption is not fulfilled in highly porous materials like the tested barium titanate and Weibull statistics do not describe the test results properly.

The data were thus fitted with other distribution functions, a standard Gaussian and a logarithmic Gaussian distribution. Chi-square tests revealed that all three distributions fit the data comparably well. This may be explained by the fact that a small sample of 30 specimens will only cover the peak region of the distribution functions, while the extremities, where the differences between the distributions are more pronounced, are omitted. This is a fundamental problem of fracture statistical evaluations.

CONCLUSION

- The most important limiting factor for bend-test miniaturisation seems to be the contact pressure between fixture (or rollers) and specimen. Using high yield steel fixtures and rollers ($\sigma_y \approx 2000$ MPa) and testing high strength ceramics, these contact stresses limit the outer span length to about 12 mm. If ceramic fixtures and rollers are used an outer span

length of 7 mm or even less (depending on the compressive strength of the fixture and roller material) may be possible. If electroceramics, which usually have low strength values, are tested, even much smaller span lengths may be practicable.

- Load application by free rollers instead of knife edges is necessary in order to minimise frictional forces. For knife edges these forces may be higher than 5% of the applied load even at a span length of 40 mm. In the case of free rollers the frictional forces are much smaller than for knife edges, but they increase strongly for outer span lengths smaller than 5 mm. Friction is an important but difficult determinable source of measuring errors. In the case of $L = 13$ mm it is estimated to produce a systematic error of approximately -1%.
- A possible specimen size is $2 \text{ mm} \times 1.5 \text{ mm} \times 15 \text{ mm}$ for $L = 13$ mm and $l = L/3$. For this configuration the measuring error is in the range of $-2.2\% < \varepsilon < 5\%$ using fixtures and specimens produced in a standard workshop. If the systematic errors are taken into account when the strength is evaluated the error is reduced to $\varepsilon_{\text{ran}} < \pm 3.5\%$. A further reduction of the measuring error for specimens of this size is only possible if highly sophisticated manufacturing procedures for fixtures and specimens are applied.
- Apart from errors due to the test set-up an essential requirement for successful testing of brittle materials on small fixtures is that small specimens can be prepared in such a way that no additional defect populations are introduced through the grinding process. This cannot be assured by simply following preparation directions. A sufficient surface quality is strongly material dependent and has to be checked in each case.
- The good agreement of strength values from DIN and small samples in the case of silicon nitride proves that reliable results can be achieved using the small 4-point bend fixtures.
- Barium titanate, a porous material shows no volume dependence of strength. Fractography and micrographs evoke the assumption, that the material tested here fails due to an mechanism which cannot be included in the Weibull formalism. Until now porous ceramics are generally characterised by Weibull statistics and therefore this seems to be an important new result.

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