

BIAXIAL STRENGTH TESTING ON MINI SPECIMENS

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ABSTRACT

Biaxial strength testing of brittle materials using the ball on three balls (B3B) test is a new method for strength testing of disc or plate specimens. The analysis of the stress fields and the calculation of the effective volumes and surfaces for several types of specimens and testing geometries have been performed recently. The ball on three balls testing method has several advantages compared to the common three- or four point bending tests: the results are very stable against small geometrical inaccuracies of the specimen or the test assembly, edge defects are not relevant and there exists only a very low influence of friction. This makes this type of experiment ideal for testing very small specimens. In this paper ball on three ball tests were performed on mini specimens having a volume of several cubic millimetres and less made of several types of advanced ceramic materials. The test results are compared with results gained on larger ball on three ball specimens and on conventional bending specimens. The results are discussed in the framework of the Weibull theory.

Introduction

Fracture of ceramic materials starts in general from crack-like flaws, which are distributed in the material. The size of the largest crack in the specimen determines its strength. A consequence of that behaviour is the well known scatter of strength data of ceramics (the size of the largest defect in different specimens is different). There are two important consequences of this behaviour: The probability of failure depends on the applied load and on the volume of the test specimen. The strength is not given by a single number, but has to be described by a suitable distribution function (fracture statistics) [1-3], which accounts for both effects mentioned above. In most cases this is well done by the Weibull statistics [4, 5]. Examples of exceptions (deviations from the behaviour described by the Weibull statistics) are discussed in [6].

Therefore the measurement of strength has to be performed on the basis of a large set of tests (a large sample). In the standards for strength testing of ceramics, e.g. ENV 843-1 [7], it is claimed that a sample has to contain at least 30 tests. The second important consequence is that strength data can only be compared on the basis of the same (effective) volume (or the same effective surface). This can be ensured, if all tested specimens have the same shape and size, and if they are loaded under identical conditions. The standardised (e.g. ENV 843-1, [7]) and worldwide accepted procedure for the strength testing of ceramics is the four point bending test on rectangular bars (dimensions of 3 x 4 x 45 mm³, distance between supporting rolls 40 mm and between loading rolls 20 mm). Fracture in four point bending starts in the tensile loaded zone at or near the outer fibre, the effective volume is typically around a few percent of the total volume of the specimen.

There are many good arguments for bending testing of ceramics: The machining of rectangular specimens is relatively easy and the testing procedure is quick and not costly. The most important advantage is that the standard is – after a very long and emotional discussion – world wide accepted in the scientific and technical society. The test data evaluation has to be done on the basis of the Weibull statistics. It is standardised in ENV 843-5 [8]. The standards enable a precise determination and evaluation of bending test data and therefore a fair comparison of materials.

But there also exists some disadvantages of the four point bending test: The edges of the rectangular bars are prone to some machining damage and fracture often starts – even if the edges are chamfered - at the edges. Of course such tests are

* In fact the strength depends on an “effective volume” (i.e. the volume under high tensile stresses; for more details see [1, 2]). This also causes a dependency of the loading condition. If, for example, a bar is tested in homogeneous uniaxial tension (tensile test) the effective volume is much higher as if the same bar is tested in bending. The probability of finding a large defect in a large volume is higher than in a small volume. Therefore the (mean) tensile strength of the bars is lower than their (mean) bending strength.

invalid and can not be included in the evaluation of the fracture statistics. The strength of a ceramic material is strongly related to the size and distribution of the defects in the material. The defect population results from details of the production process of the component. Therefore specimens for strength testing should be machined out of the components (and not processed separately) or the component should be tested in one piece. In many cases the size of the components is smaller or even much smaller as the size of the standardised specimen and therefore this is not possible. There have been some efforts to miniaturise the bending test, but with reasonable effort (machining small specimens with a high precision is extremely difficult) and if a low measurement uncertainty is necessary (say less than 5 % of the measurand), the length of the bending bar should not be smaller than about 15 mm, [9, 10]. Therefore other types of tests have to be developed, which make the accurate strength testing of smaller specimens possible. In this paper the ball on three balls (B3B) test is proposed to be a suitable testing method for small plate shaped specimens or components.

In service ceramic components are often biaxially loaded (e.g. during thermal shocks). This stress state is more searching for defects than an uniaxial (bending) state [11]. Biaxial strength testing is therefore more appropriate for these applications as uniaxial testing. Many proposals for biaxial strength testing of disc specimens have been made in the past. A good overview on the methods, their strengths and their drawbacks, is given in the paper of Godfrey and John [11] or in the paper of Morrell et al. [12]. Several advantages are claimed for biaxial flexural testing of discs compared to uniaxial testing (in tension or in bending) including ease of test piece preparation and use for thin sheet materials. An important benefit of this type of test is that the quality of the edges of the disc has no influence on the measurement.

Specimens for biaxial strength testing have in general the shape of a circular disc. Many ceramic components, e.g. ceramic resistors, have the shape of discs or rods and, for these parts, disc shaped specimens offer the opportunity to prepare specimens with a very limited machining effort (or even without machining). The testing methods differ in the way of the load transfer to the specimen. Most common are the punch on ring and the ring on ring test [13], which both produce a radial symmetric stress field in the specimen. This symmetry facilitates the determination of the stresses in the disc. A disadvantage is the not exactly defined contact situation, which may occur due to (small) geometric inaccuracies of specimen (e.g. warping of the disc) and jig and which can not be completely avoided in practice. A not well defined contact situation may cause significant errors in the strength determination.

An alternative testing method proposed recently by the authors is the ball on three balls test (B3B test), where the specimen is supported on three balls and loaded by a fourth ball [14-17]. A sketch of the fixture is shown in **Fig. 1**. The three point support guaranties a well defined three point contact, but the threefold axes of symmetry makes the stress analysis difficult. At the midpoint of the surface plane of the disc opposite to the loading ball a biaxial tensile stress state exists, which is used for the biaxial strength testing of the material. For linear elastic discs this situation has been numerically analysed in a wide range of parameters (geometry of disc and support, elastic constants of disc and support materials, etc.) by Börger et al. [15] for specimens made of ceramic materials (elastic modulus between 100 and 1000 GPa) and for fixtures made of steel or a material with a higher modulus.

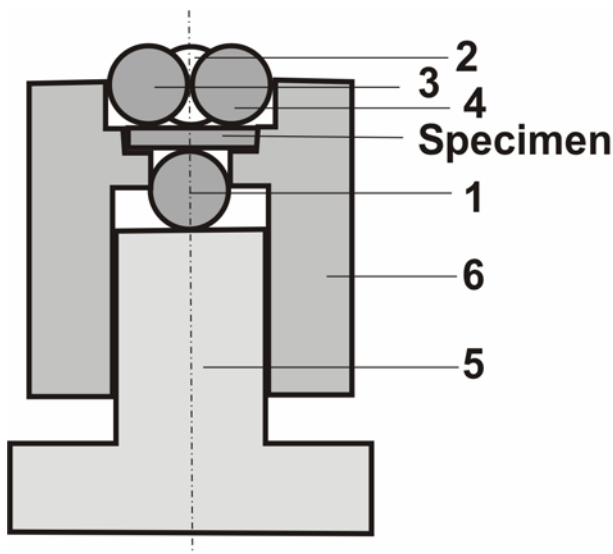


Fig. 1: Experimental set-up of the ball on three balls test [14] and testing procedure: The specimen is centrally positioned over the loading ball (1), which is placed on the stamp (5). Specimen and loading ball are positioned by the guide (6). The specimen is supported by three supporting balls (2-4). All four balls and the specimen are exactly positioned by the guide (6), which has a rotational symmetry and can therefore be machined very accurately with reasonable effort. During testing a preload (typically about 10 % of the estimated fracture load) is applied at first. After that the positioning aid is pushed down and the balls can freely move. The specimen is only fixed by the friction between balls and specimen. Then the load is increased up until fracture. The fracture load can be used to calculate the maximum tensile stress in the specimen at the moment of fracture (i.e. the strength of the specimen).

Of course the stress field has three fold symmetry (for details see [15]). The maximum tensile stress in the specimen is

$$\sigma_{\max} = f \cdot \frac{F}{t^2} \quad , \quad (1)$$

with the maximum load at fracture, F , the disc thickness, t , and a dimensionless factor f , which depends on the geometry of the specimen (thickness, t and radius, R , of the disc), the Poisson's ratio of the tested material and details of the load transfer from the jig into the specimen (the support radius R_a). In general a proper analysis of the stress field needs a precise modelling of the contact situation between supporting balls and specimens, but in a wide range of parameters and with respect to describe the situation in the centre of the disc on the surface opposite of the loading ball, a simple point model for the load transfer is also appropriate. For disc shaped specimens and in the parameter range ($0.55 \leq R_a/R \leq 0.90$; $0.05 \leq t/R \leq 0.60$; $0.2 \leq \nu \leq 0.3$) the analysis can be made using the point loading model and the function f only depends on the dimensionless parameters R_a/R , t/R and ν . The results in form of an analytical equation fitted to the numerical results can be found in [15]. A complete contact analysis has to be performed for specimens having $0.05 > t/R$ or for specimen or fixture materials having modulus smaller as mentioned above.

In a subsequent paper Börger et al. [16] analysed the measurement uncertainties of the B3B test. This test has been recognised to be very tolerant for some out of flatness of the disc and also for small other geometrical inaccuracies or some misalignment. The most significant "geometric" property is the thickness, t , of the disc (determined in the middle of the disc), which enters with $\sigma_{\max} \propto t^{-\beta}$, with $\beta \sim 2 - 3$. Two percent uncertainty in thickness causes about 5 % uncertainty in strength. For all other "geometric" properties (and for the Poisson's ratio) the measurement uncertainty of the strength increases linear with the uncertainty of the property, and the proportionality factor is less than $\frac{1}{2}$. Therefore a measurement uncertainty of one in these properties of less than 10 % would cause an uncertainty in strength of less than 5 %. Furthermore friction is recognised to be much smaller than in the commonly used (three or four point) bending tests. For these reasons the B3B test can also be used to test as-sintered specimens (as sintered specimens show sometimes rough surfaces and are warped) and for the testing of very small specimens (absolute tolerances cause a higher relative uncertainty for small than for large specimens).

The B3B test has successfully been applied to describe the mechanical behaviour of several ceramic materials so far [17, 18]. It has been applied for disc specimens made of alumina [17, 18], silicon nitride [18], zinc oxide [18] and barium titanate [17]. In alumina, which has been successfully investigated with a large number of specimens of different size, a pronounced size effect on strength arising from surface flaws could be detected [18]. In silicon nitride the fracture process was caused by volume flaws [18]. In general, it could be demonstrated that the B3B test is easy to perform, as well as the specimen preparation and the testing of as sintered specimens can be done because some buckling of specimens can be tolerated.

The motivation to write this paper arises from our work on electro ceramic components. In fact many billions of electro ceramic components fail per year for brittle failure, but little is known about their strength and their mechanical properties. Due to the increasing miniaturisation electro ceramic components are getting smaller and smaller and their power density increases. This causes higher temperatures in components, big temperature differences in the components, and in consequence, thermal strains and mechanical stresses, which may destroy the component. Many of these components have a plate or disc shape with a diameter of several millimetres or less. Therefore there is a great need to measure strength on such small components or specimens.

In this paper the B3B test is used for strength testing on very small specimens having a diameter of several millimetres and a volume of about 1 mm^3 and less.

Stress analysis

The Weibull theory [4, 5] predicts a size effect on strength, i.e. the strength decreases with increasing effective volume[†]. In order to investigate if the B3B test is suitable for testing of very small specimens this size effect will be determined for three different ceramic materials. Specimens of different size were produced, tested and evaluated according to the Weibull theory [1, 2]. The results were compared with conventional bending tests.

The stress fields of disc shaped B3B test specimens can be found in [15]. But the machining of very small discs turned out to be difficult. Therefore specimens with the geometry of rectangular plates were also produced. In their case the machining was relatively easy since the material could be grinded to a plate (of desired thickness) and than cut into rectangular parallelepipeds. For these rectangular plate specimens the determination of the stresses in the B3B test is missing till now. Therefore a FE analysis for each type of specimen has been performed to determine the factor f (maximum tensile stress) of the specimen.

[†] If strength is determined by surface defects instead of volume effects, it increases with decreasing effective surface.

The stress analysis was performed using the FE-package ANSYS 8.1®. A typical FE model (modelled is the half of the specimen) is shown in **Fig. 2.a**. It contains around 20000 second order brick elements containing 20 nodes each. The area around the load transfer points is modelled with finer elements. The resulting stress distribution is shown in **Fig. 2.b** for the full plate. The cloverleaf shape of the stress field can clearly be recognised. The maximum tensile stress occurs in the middle of the top plane of the specimen opposite to the loading ball.

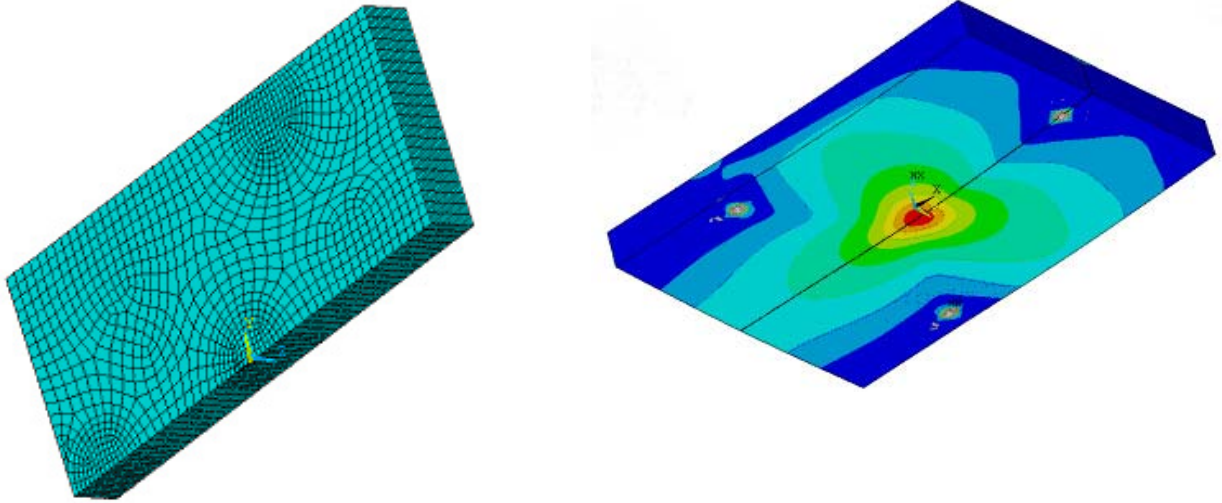


Fig. 2: FE-analysis for the stress field in a rectangular plate specimen tested in the B3B test. a) FE model, b) distribution of the primary principle stress. Parameters: volume of specimen is $10 \times 8.4 \times 0.94 \text{ mm}^3$, Poisson's ratio is 0.25, force is 1N.

B3B tests produce a biaxial stress state (in the middle of the specimen, where the maximum occurs). To compare data of B3B tests with bending test data an equivalent stress has to be determined, which accounts for the more damaging loading in biaxial testing compared to uniaxial testing [1]. Although the proper definition of such a stress is still in discussion, the principle of independent action (PIA) [23] is often used. The PIA equivalent stress is

$$\sigma_{eq,PIA} = \left(\sigma_I^m + \sigma_{II}^m + \sigma_{III}^m \right)^{1/m}, \quad (2)$$

where σ_I , σ_{II} and σ_{III} are the principle tensile stress components. For a biaxial stress state it holds: $\sigma_{eq,PIA} = 2 \cdot \sigma_I^{1/m}$, m is the Weibull modulus.

For each type of B3B test (defined by the specimen geometry and the loading radius) the stress field, the effective volume and the effective surface has also been determined to make a comparison of data possible.

Investigated materials and specimens

Alumina Ceramic

The investigated material (AL23) is a typical commercial 99.7 % pure Al_2O_3 ceramic produced by FRIATEC GmbH, Germany. It was delivered in form of discs (radius 10 mm, thickness 3 mm). It has a bimodal grain structure (**Fig. 3.a**). Large grains having a diameter around 20 - 30 μm are surrounded by smaller grains with a diameter of a few micrometers. Some mechanical properties are listed in **Table 1**.

For that material the as-sintered surface was used as tensile loaded surface of the specimens. Therefore the delivered discs could be used as B3B specimens without any further machining. In the case of machined specimens, special care was given to keep the as-sintered surface in its original condition. To produce thinner discs the tensile top plane of the disc (opposite to the loading ball) remained just as it was, but the bottom plane was grinded that the disc gets the desired thickness. Discs with a smaller radius were drilled out of the delivered discs and then thinned in the way described above. For the production of rectangular plate specimens the delivered discs were first thinned and then cut in rectangular plates of desired size using a diamond grinding wheel. Bending specimens (size $13.3 \times 2 \times 1.5 \text{ mm}^3$) were cut out of the delivered discs. In their case also special care was given to keep the tensile surface in its as sintered condition.

An overview about the produced sets of specimens is given in **Table 2**. The smallest B3B specimens had a volume of less than 0.7 mm^3 and the largest of about 1000 mm^3 . In total 14 different types of specimens (315 test pieces) were tested.

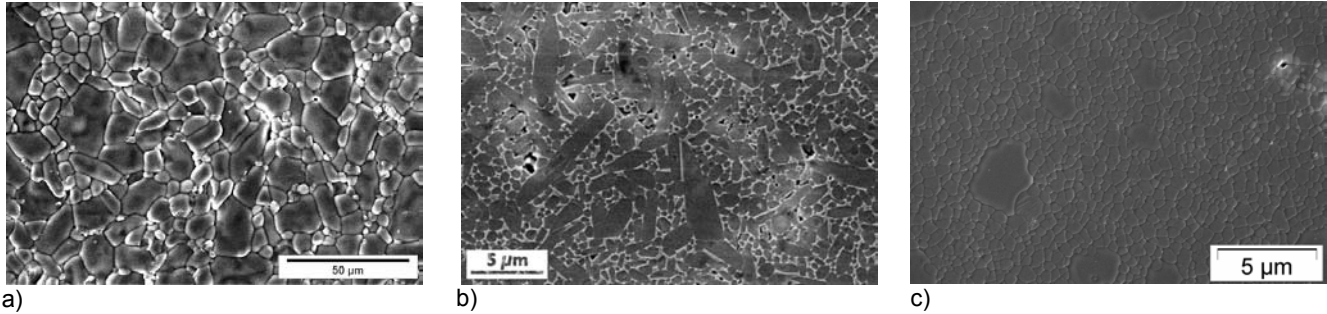


Fig. 3: Micro structures of the investigated materials. a) alumina (SEM picture of the as sintered surface), b) silicon nitride (SEM picture of a polished section) and c) zirconia.

Silicon nitride ceramic

The investigated material is a typical commercial gas pressure sintered silicon nitride ceramic with $\text{Al}_2\text{O}_3 - \text{Y}_2\text{O}_3$ additions. It has a needle like grain structure (**Fig. 3.b**), a high strength and reasonable fracture toughness. It has been produced by CeramTec, Plochingen, Germany (trade name SL200). The material was provided in form of plates with dimensions of $47 \times 11 \times 102 \text{ mm}^3$. This material (the same production lot) is used as a reference material in the ESIS reference material testing program. For more details see [21] and also the paper of Lube and Dusza, [22]. Some mechanical properties are listed in **Table 1**.

Large disc specimens (diameter 43 mm) were machined out of the plates by grinding the plates to the desired thickness. The thinned plates were cut in octagonal pieces and then round grinded to the desired diameter. Rectangular plate specimens were machined as described above for the alumina specimens, but in the case of this material both surfaces were machined. Bending specimens of three different sizes were machined following the instructions of the standards [7]. For all types of specimen special care was given not to damage the tensile loaded surfaces.

The smallest B3B (rectangular plate) specimen had a volume of less than 2 mm^3 and the largest B3B specimen had a volume of more than 5000 mm^3 . In total 9 different types of specimens and 151 test pieces were tested.

Zirconia ceramic

The investigated material is a commercial zirconia ceramic produced by Morgan Matroc (England). The zirconia is stabilized with yttria and has an average grain size of less than $1 \mu\text{m}$ and some large grains with a diameter around $5 \mu\text{m}$ (**Fig. 3.c**). Some mechanical properties are listed in **Table 1**. The four point bending specimens and the B3B test rectangular plate specimens were cut out of delivered plates (size of $100 \times 40 \times 10 \text{ mm}^3$) performing the same procedure as described above. The smallest B3B specimens had a volume of less than 1 mm^3 . In total 6 different types of specimens and 156 test pieces were produced.

Table 1: Mechanical properties of investigated materials

	<i>Alumina</i>	<i>Silicon Nitride</i>	<i>Zirconia</i>
Young's modulus, [GPa]	380 ^a	305 ^b	200 ^a
Poisson ratio, [-]	0.24 ^a	0.28 ^b	0.30 ^a
Characteristic strength ^c , [MPa]	> 400 ^a	869 ^{b, c}	832 ^{b, c}
Fracture toughness, [$\text{MPa} \cdot \text{m}^{1/2}$]	3.2±0.1 ^{b, d}	4.9±0.9 ^{b, d}	10 ^a

^{a)} Data from the supplier; ^{b)} own measurements, ^{c)} 4 point bending with a 40/20 mm span length according to EN843-1,

^{d)} single edge V-notch beam method (SEVNB; [19, 20]).

Fracture experiments and test results

To test the rectangular plate specimens the guide of the fixture (**Fig. 1**, part 6) had to be modified to align and position the specimen properly. The thickness of every B3B specimen was determined by a digital micrometer with a resolution of $\pm 1 \mu\text{m}$. In the evaluation of the data the precise thickness of every specimen is used. After positioning of the specimen and applying a small preload (around 10 % of the expected fracture load) the positioning aid has been removed. Then the specimen has been fixed between the load ball and the support balls, which can freely move and the load has been increased till fracture occurs. The test is only valid if the fracture origin is in the area opposite to the load ball, where maximum tensile stresses occur (in the work reported here other areas of fracture initiation could not be observed in any case). The bending experiments were performed in analogy to ENV 843-1 [7].

Fig. 4 shows some fractured B3B test specimens. The specimen breaks into two or more pieces. There is a tendency to increase the number of pieces with the stored elastic energy and is therefore proportional to the applied force. A fractographic analysis to identify the fracture origin has been started but is not finished yet (the deadline for the written

contribution in this conference was seven months before the conference!). In the case of alumina the fracture origins seem to be surface flaws. In the case of silicon nitride specimen in any case analysed so far (fracture surfaces of large as well as small specimens were investigated) the fracture origins are glassy regions - often related to iron rich inclusions (**Fig. 5**). The fractographic analysis of the zirconia specimen has not been performed yet.

<i>Sample (Test method)</i>	<i>Specimen dimensions</i>	<i>Number of tested specimens</i>	<i>Characteristic strength [MPa]</i>	<i>Weibull modulus m</i>
Alumina				
A1 (4-point-bending, 13/4,3)	13.3 × 1.3 × 1 mm ³	30	338 [330-346]	14,6 [10.9-17.8]
A2 (B3B-discs)	Ø 20 mm, t=3 mm	30	348 [342-355]	17,3 [13.0-21.1]
A3 (B3B-discs)	Ø 20 mm, t=2.9 mm	15	385 [365-406]	9.5 [6.1-12.4]
A4 (B3B-discs)	Ø 20 mm, t=2 mm	16	383 [370-397]	14.1 [9.2-18.3]
A5 (B3B-discs)	Ø 20 mm, t=1 mm	15	417 [405-430]	17.1 [10.9-22.3]
A6 (B3B-discs)	Ø 20 mm, t=0.5 mm	28	471 [458-484]	12.5 [9.2-15.5]
A7 (B3B-discs)	Ø 10,8 mm, t=3 mm	15	380 [361-402]	9.3 [6.0-12.1]
A8 (B3B-discs)	Ø 10,8 mm, t=1.5 mm	15	414 [397-432]	11.7 [7.7-15.6]
A9 (B3B-discs)	Ø 4,7 mm, t=0.55 mm	30	520 [507-533]	13.5 [10.1-16.4]
A10 (B3B-discs)	Ø 4,8 mm, t=0.26 mm	26	520 [506-535]	13.1 [9.5-16.1]
A11 (B3B-rectangular plates)	2.6 × 2.6 × 0.6 mm ³	30	485 [470-500]	11.0 [8.3-13.4]
A12 (B3B-rectangular plates)	2.6 × 2.6 × 0.31mm ³	30	554 [540-569]	12.8 [9.6-15.6]
A13 (B3B-rectangular plates)	2.6 × 2.6 × 0.26 mm ³	30	553 [537-570]	11.3 [8.5-13.8]
A14 (B3B-rectangular plates)	2,6 × 2,6 × 0.1 mm ³	5	628 [567-704]	10.9 [3.9-15.9]
Silicon Nitride				
S1 (4-point-bending, 80/40)	80 × 8 × 6 mm ³	14	809 [784- 836]	16.5 [10.2-21.4]
S2 (4-point-bending, 40/20)	40 × 4 × 3 mm ³	55	867 [852- 881]	14.3 [11.7-16.6]
S3 (3-point-bending, 40)	40 × 4 × 3 mm ³	30	985 [966-1005]	16.8 [12.6-20.5]
S4 (B3B-discs)	Ø 43mm, t=3.8 mm	7	847 [776- 930]	9.4 [4.3-13.3]
S5 (B3B-rectangular plates)	10.1 × 8.4 × 2 mm ³	6	1094 [1008-1195]	11.5 [4.7-16.6]
S6 (B3B-rectangular plates)	10.1 × 8.4 × 1 mm ³	9	1204 [1176-1235]	28.4 [14.5-38.8]
S7 (B3B-rectangular plates)	10.1 × 8.4 × 0.5 mm ³	9	1281 [1245-1321]	23.7 [12.5-32.4]
S8 (B3B-rectangular plates)	2.6 × 2.6 × 0.5 mm ³	11	1204 [1171-1239]	21.6 [12.4-28.9]
S9 (B3B-rectangular plates)	2.6 × 2.6 × 0.29 mm ³	10	1239 [1199-1281]	19.6 [10.9-26.5]
Zirconia				
Z1 (4-point-bending, 40/20)	40 × 4 × 3 mm ³	15	832 [805- 861]	15.5 [9.7-20.4]
Z2 (B3B-rectangular plates)	10.1 × 8.4 × 1.9 mm ³	27	1083 [1059-1109]	15.6 [11.4-19.2]
Z3 (B3B-rectangular plates)	10.1 × 8.4 × 0.9 mm ³	27	1214 [1186-1243]	15.1 [11.1-18.6]
Z4 (B3B-rectangular plates)	10.1 × 8.4 × 0.5 mm ³	27	1267 [1243-1293]	17.9 [13.1-22.0]
Z5 (B3B-rectangular plates)	2.6 × 2.6 × 0.4 mm ³	30	1013 [984-1043]	11.6 [8.7-14.1]
Z6 (B3B-rectangular plates)	2.6 × 2.6 × 0.2 mm ³	30	1196 [1158-1237]	10.2 [7.6-12.4]



Fig. 4: Fractured B3B test specimens: a) discs of alumina specimens having a different size, b) silicon nitride specimen; the number of pieces, in which the specimen breaks, increases with the stored elastic energy and c) rectangular plate specimens made of zirconia ceramic.

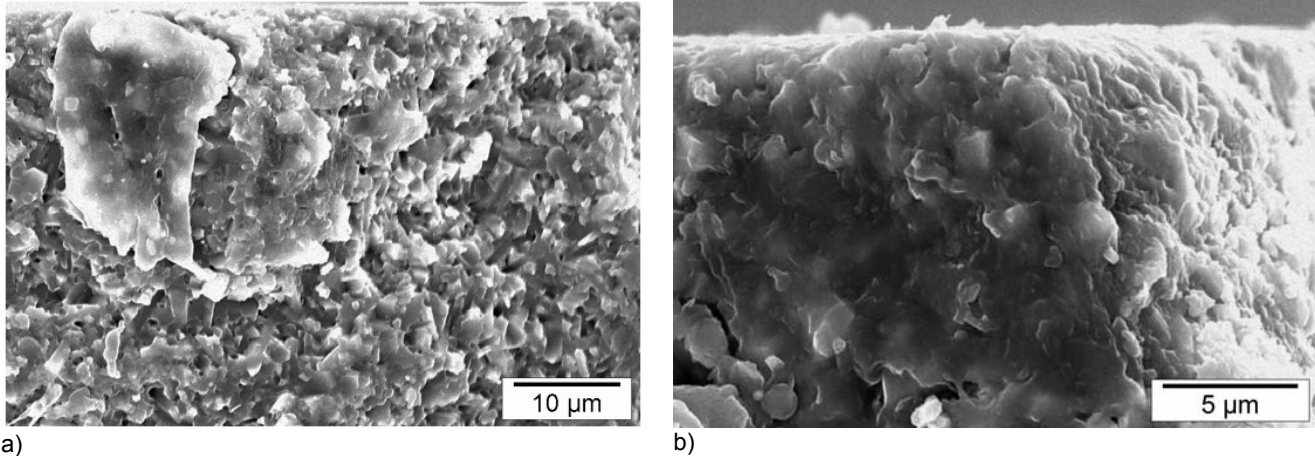


Fig. 5: Fracture origins in silicon nitride specimens: a) origin in a bending specimen and b) in a “small” ($2,6 \times 2,6 \text{ mm}^2$) rectangular plate specimen. The origins are glassy regions in the microstructure.

The data were analysed in the framework of conventional Weibull analysis [1, 2]. The characteristic strength and the Weibull modulus were determined for each set of data separately; i.e. the characteristic volume was set equal to the effective volume: $V_0 = V_{eff}$. It should be noted, that the width of the 90 % confidence interval (for the characteristic strength, the effective volume and the effective surface) depends on the size of the sample and on the Weibull modulus [8]. The test results are summarised in **Table 2**. For the characteristic strength the first principle stress (and not the equivalent stress according to the PIA criterion) is reported.

Discussion

The results of the strength test are summarized in **Fig. 6** and **Fig. 7**. Plotted is the equivalent characteristic strength (the PIA criterion is used, [23]) versus the effective volume or the effective surface respectively in a double logarithmic plot. Scatter bars are only shown for some selected data (of small samples) to keep the plot clearly arranged. They refer to the 90 % confidence intervals, which results from the sampling procedure [24]. It should be noted that these measurement uncertainties are relatively large for small samples but small for large samples (containing e.g. 30 tests or more). Not indicated is the scatter due to other measurement uncertainties. In a paper of Börger et al. [16] it has been shown that experimental measurement uncertainties increase with decreasing specimen size (especially the specimen thickness has a large potential contribution). But for the used specimen experimental measuring uncertainties should be smaller as 5 % of the strength value in any case and smaller as 1 % in most cases. In general the size of the confidence interval is almost equal to the size of the symbol (but for small samples it is - as shown on some examples - wider).

The data of alumina show an increasing strength with decreasing volume (surface). The dashed line is the prediction bases on the mean Weibull modulus (it is around 12). Starting point of the interpolation line is the bending test result. It can be recognised that the surface flaw model (**Fig. 7**) describe the data more appropriate as the volume flaw model (**Fig. 6**). This observation can be confirmed by microscopy and fractography. The as sintered tensile surface of the specimen shows deep grooves (not sintered grain boundaries) having a depth of 10 µm and more (**Fig. 3.a**). The largest of these grooves in the middle of the tensile top plane of the specimen is expected to be the fracture origin (to give an example for $K_c = 3.2 \text{ MPa}$, $\sigma_f = 640 \text{ MPa}$ and $Y = 1.12$, the critical crack size is around 10 µm; σ_f is the strength and Y the geometric factor). Other types of fracture origins could not yet be found in our fractographic work. This analysis is consistent with the classical Weibull theory.

For the tests on silicon nitride the mean Weibull modulus is around $m = 17$. The full line with the slope $1/m$ indicates the behaviour predicted by the Weibull theory. The data for specimen having an effective volume larger than 10^{-2} mm^3 follow this prediction but the data of smaller specimens does not. For these data an upper limit for strength seems to occur. This is not consistent with Weibull theory. An analogue behaviour can be observed, if the strength is plotted versus surface. The fractographic analysis indicates that volume flaws are fracture origins in all cases analysed so far (see **Fig. 5**). Therefore it can be assumed that the volume flaw model is more appropriate the surface flaw model. In a recent paper of Danzer [25] a threshold for the strength is predicted for very small specimen: following the presumptions of the Weibull theory the density of small flaws (which are relevant for small specimens having a high strength) gets so high, that interaction between flaws becomes possible. Then flaws growth together and this effect restrict the strength. The data presented in this paper are a first experimental hint that this theory is correct.

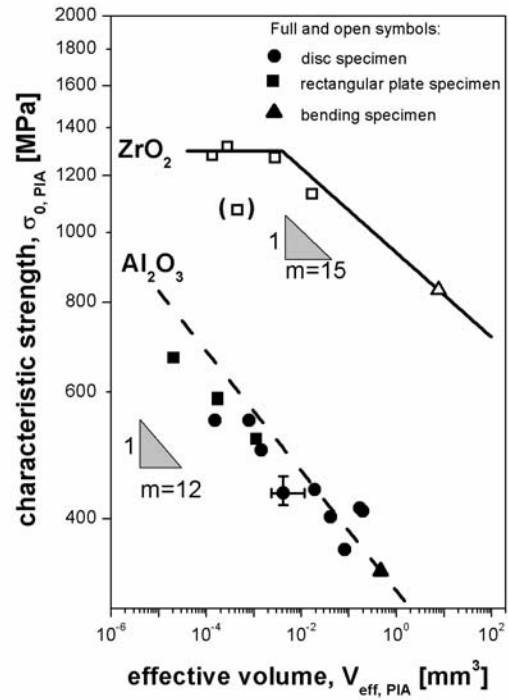
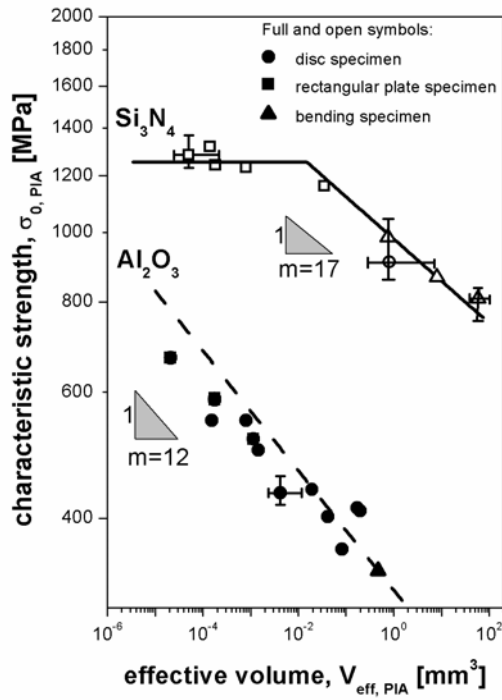


Fig. 6: Characteristic strength versus effective volume in a double logarithmic plot. a) alumina and silicon nitride and b) alumina and zirconia. For the data evaluation the PIA criterion is used.

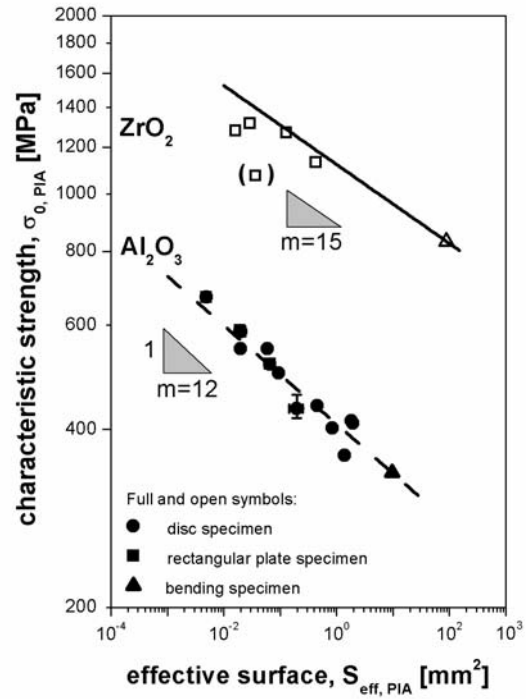
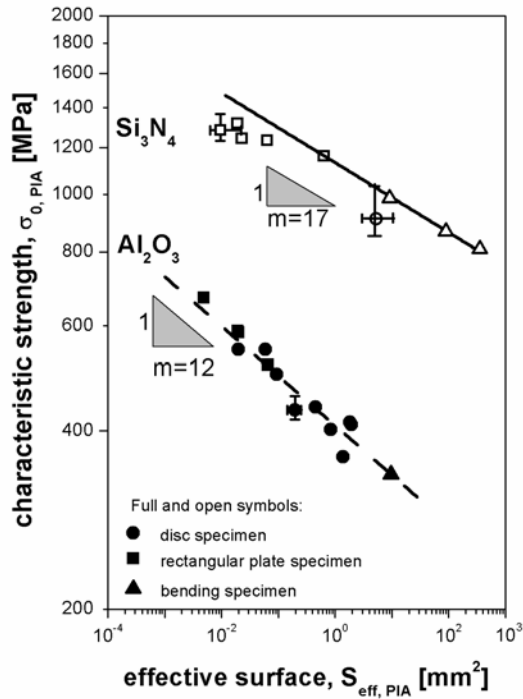


Fig. 7: Characteristic strength versus effective surface in a double logarithmic plot. a) alumina and silicon nitride and b) alumina and zirconia. For the data evaluation the PIA criterion is used.

In the case of the tests on zirconia one of the data points (sample Z5) is definitely outside of the trend determined by the other samples. Microscopic analysis of the top surfaces of the specimen indicates that, in the case of the sample Z5, scratches in the tensile top plane can be found in many cases. The same can not be observed in the specimens of the other samples. Therefore the sample Z5 has been excluded of the further data analysis. It should be noted that the confidence intervals of the data (due to the sampling procedure) is similar to the size of the symbols. The mean Weibull modulus of the other samples is around $m = 15$. For zirconia a similar behaviour as for silicon nitride can be found. For specimens with an effective volume larger than around 10^{-2} mm^3 , the strength follows the trend predicted by the Weibull theory. For smaller specimen the strength does not increase any longer. Up to now a decision between the volume and surface flaw model could not be done because – as stated earlier – a decisive microscopic study has not been performed yet.

Conclusions

- The B3B test is a simple and good applicable testing method for biaxial strength testing of disc or plates. The B3B strength data are in the scatter band of the data determined with other methods, if the (effective) volume or the (effective) surface is similar. Of course the more damaging action of the biaxial stress state (compared to an uniaxial state) has to be taken into account by a suitable definition of the equivalent stress.
- For the materials tested in this work the PIA criterion was appropriate to determine the equivalent stress.
- The B3B strength data show a pronounced size effect of strength.
- For alumina this effect can be explained in the framework of conventional Weibull. The fracture origins are surface flaws.
- For silicon nitride and zirconia a pronounced size effect could also be observed but for very small specimens an upper limit of strength occurs, which is in contradiction to Weibull theory. In principle such a behaviour could result from some machining damage. For silicon nitride it follows from first fractographic results that the fracture origins (in small as well as in large specimens) are volume flaws. Therefore it is probable that machining damage is not the reason for that upper limit of strength. For zirconia a fractographic analysis of our specimens is still missing.
- In a recent paper [25] inherent boundaries for the Weibull theory were analysed. There, an upper limit of strength for very small specimens is postulated for theoretical reasons. The authors believe that the presented experiments are a first experimental hint for the existence of that limit.
- The B3B test is well situated to test very small specimens having a volume of several mm^3 or less.
- It is appropriate to test spherical disc as well as rectangular plate shaped specimens. In principle also plates of other shape can be tested.

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