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Determination of strength and fracture toughness of small ceramic discs using the small punch test and the ball-on-three-balls test

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Abstract

The strength and fracture toughness of small ceramic discs under biaxial flexural load are investigated with two different miniaturized test methods: the small punch test (SPT) and the ball-on-three-balls test (B3B). An alumina ceramic was chosen as sample material. While the specimen geometries are identical for both tests, the experimental set-ups and the stress fields are different. First, the Weibull parameters of strength have been estimated. Second, the fracture toughness is evaluated with an adaption of the surface crack in flexure (SCF) method. The discs are prepared with artificial surface cracks whose sizes have to be measured. The evaluation of stresses and stress intensity factors is based on finite element calculations.

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Keywords: small punch test; ball-on-three-balls test; biaxial strength; fracture toughness; miniaturized mechanical testing

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1. Introduction

Influenced by the manufacturing process ceramic components of different size and shape often vary in their microstructure (grain and flaw size distribution) and surface quality (grinding procedure, scratches), which effects their mechanical behavior. Small and thin ceramic components or components with graded properties cannot be characterized properly using standard size specimens made of bulk material. Standardized uniaxial bending tests are often inadequate for strength measurements as edge defects may be the source of failure. Furthermore the loading of

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real components is rather biaxial than uniaxial. Therefore biaxial flexural tests of ceramics on disc-shaped specimens are particularly favored for many applications (Morell et al. (1999), Börger et al. (2002). They have the advantages of easy specimen preparation and that edge defects do not contribute to failure.

In this study the strength and fracture toughness of small and thin circular discs are investigated with two different miniaturized test methods which apply a biaxial flexural load: (i) the small punch test (SPT) and (ii) the ball-on-three-balls test (B3B test). Both methods were developed independently from each other under different requirements of material characterization, resulting to clearly different designs but identical specimen geometry. So it is worth to compare them. The SPT was developed in the 1980s as minimal invasive in-situ technique with small demand for material to investigate the local degradation and embrittlement of nuclear materials (e.g. ferritic alloys) used in fission and fusion reactor applications (Manahan et al. (1981), Baik et al. (1983)). Over the years the application has been extended to a wide range of different materials, see Rasche (2013). Also investigations with ceramic materials are known, but a closer view shows that publications often deal with metal-ceramic composites and/or ceramics at high temperatures (Li and Watanabe (1999), Xiong et al. (2005)), which means nonlinear material behavior with a measurable amount of plastic deformation. In contrast, the B3B test was especially developed for strength testing of ceramics components, see Börger et al. (2002). Large test series on specimens of very different size demonstrated a relatively cheap and easy specimen preparation and that the test is relatively easy to perform (Danzer et al. (2007)). The three-point-support of the discs enables the determination of the strength distribution of as-sintered specimens with negligible loss of accuracy, which makes this method superior to conventional bending tests with ill-defined contact situations (Bermejo et al. (2012).

The obvious question arises, whether the determination of the fracture toughness K_{Ic} with small ceramic discs is feasible. A first attempt was undertaken with SPT specimens prepared with Vickers indentation cracks (Rasche et al. (2010)). These experiments showed that small discs have the potential to be used as a fracture mechanics specimens, although an underestimation of K_{Ic} of about 20 to 25% compared to standard SEVNB (single edge V-notch beam) results was observed. The new approach we deal with in the actual work is the following modification of the specimen geometry. An artificial surface crack is created in the center of the tensile side of the disc by a Knoop indenter. Analogously to the standardized “Surface Crack in Flexure” (SCF) method for uniaxial bending bars residual stresses caused by the plastically deformed zone around the indent will be removed by a grinding procedure. The fracture load and the initial crack size have to be measured. Necessary stress intensity factor calculations can be performed with three-dimensional finite element simulations of both tests. While this approach was recently published for the B3B test in Strobl et al. (2014), it is presented here the first time for the SPT.

2. Experiment and modeling

2.1. Small-Punch-Test (SPT) and Ball-on-three-balls test (B3B test)

In the SPT the disc-shaped specimen (diameter D, thickness t) is supported and centered by a ring (bore diameter d, drawing radius r) and loaded centrally by a punch with semi-spherical tip (Fig. 1, left). The resulting stress field is axisymmetric. The applied load and displacement of the punch are recorded during the test. In this study a punch tip radius $R_{\mathrm{P}} = 2.5$ mm and a supporting ring radius $R_{\mathrm{a}} = d/2 + r = 2.5$ mm were chosen. The set-up is similar to the punch on ring test and differs from configurations used for ductile materials (e.g. Abendroth and Kuna (2003), CEN (2006)), because no blank holder is used. All tests were carried out in a universal testing machine at Institute of Mechanics and Fluid Dynamics. A detailed description and parametric studies are given in Rasche (2013).

In the B3B test (Fig. 1, right) the disc is centrally positioned over the loading ball and supported by three balls on the upper surface. A circular guide ensures that all four balls of equal Radius $R_{\mathrm{B}} = 2.748$ mm and the specimen are carefully aligned. The three balls are in contact with each other; therefore the loading radius is given by the relation $R_{\mathrm{a}} = 2/\sqrt{3} \cdot R_{\mathrm{B}} = 3.173\ \mathrm{mm}$. The well-defined three-point support guarantees that as-sintered discs with flatness imperfections can be tested properly but leads to a complicated three-dimensional stress field. All tests were carried out in a universal testing machine at Institut für Struktur- und Funktionskeramik. A preload is initially applied before the positioning aid (chock) is removed. Subsequently the load is increased while the supporting balls can freely move and the loading ball is kept fixed. A detailed description and the thorough theoretical treatment is given in Börger et al. (2002) and Börger et al. (2004) or see at the homepage http://www.isfk.at/en/960.

2.2. Material and specimens

The investigated material Rubalit®708S (CeramTec AG, Plochingen, Germany), is a commercial 96% Al_2O_3 substrate ceramics (grain size 3-5 µm, Young's modulus 340 GPa), typically used for electronic circuit carriers. It was delivered in form of laser-cut as-sintered circular discs with $D = 2R = 8$ mm and $t = 0.6$ mm. Two sets of 30 specimens were tested for strength measurements with SPT and B3B in analogy to EN 843-1 (2006). For the fracture toughness evaluation 12 discs were prepared with a Knoop indentation crack of 3 kg (HK 3) in the center of their tensile side. A surface layer of about 32 µm was ground off before testing to remove residual stresses from the plastic imprint. The required layer thickness was estimated following ISO 18756 (2003). An even more accurate advice is given in Strobl et al. (2012).

2.3. Finite element modeling

2.3.1. Stress analysis

Linear elastic finite element calculations were performed with the commercial code ABAQUS in the case of the SPT and ANSYS in the case of the B3B test. While a 3D-model is necessary for the analysis of stresses in the B3B specimen, the SPT can be analysed with a simpler axisymmetric 2D-model. Due to the threefold symmetry in the B3B test, it is sufficient to model only one sixth of the disc, symmetry planes are rotated through 60°. Fig. 1 shows the stress distribution at the surface of the discs for both tests approaches at an applied load of 100 N.

For both experiments the fracture load F can be used to calculate the maximum biaxial stress at the tensile stressed surface of the specimen at the moment of fracture given by the empirical equation (Börger et al. (2002))

$$\sigma_{\max} = f\left(t/R, R_a/R, \nu\right) \cdot F/t^2 , \tag{1}$$

where f is a dimensionless factor which depends on the geometry and the Poisson's ratio ν. It is calibrated by finite element results. When $t = 0.6$ mm, $R = 4$ mm and $\nu = 0.25$ the factors are $f = 2.0$ for the B3B and $f = 1.9$ for the SPT.

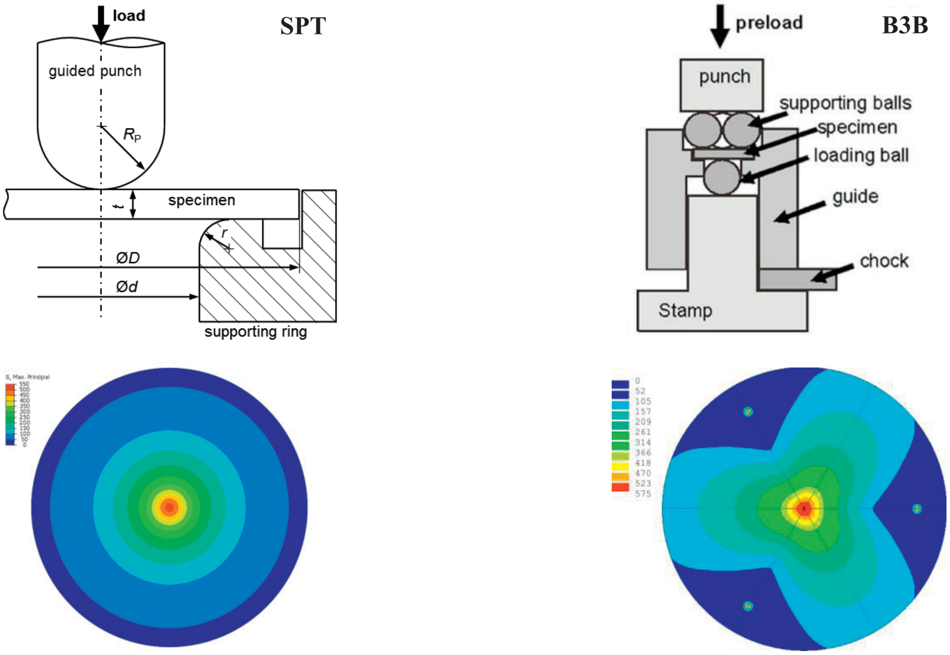

Fig. 1. Schematic of the SPT (left) and the B3B test (right) with contours of first principal stress on the tensile side of the discs ($F = 100$ N).

2.3.2. Stress intensity factor evaluation

The determination of the stress intensity factor along the crack front of the nearly semi-elliptical surface crack needs a more complex 3D model in both cases. Because of two mirror symmetry planes referring to the crack a quarter model is sufficient in the case of the axisymmetric SPT. A full model would be needed for the B3B test, but when the crack plane is oriented in direction of one of the three symmetry planes, as was done in the experiments, only a half model is needed. The crack orientation in the SPT is arbitrary as long as the crack is really centered. Quarter point crack front elements with collapsed element faces and a focused meth were used to model the crack front. The stress intensity factor was obtained by converting the *J*-Integral output of the finite element code into Mode I stress intensity factor K_I under the assumption of plane strain.

2.4. Statistical evaluation of strength measurements

The statistical analysis of the strength measurements was performed according to EN 843-5 (2006). Additionally 4-point-bending-tests (4PB) served as reference. The probability of failure P_f is described with a two-parametric Weibull distribution of failure stress σ_c :

$$P_f(\sigma_c) = 1 - \exp\left(-\left(\frac{\sigma_c}{\sigma_0}\right)^m\right) = 1 - \exp\left(-\frac{V_{\mathrm{eff}}}{V_0}\left(\frac{\sigma_c}{\sigma_{0,V}}\right)^m\right) \tag{2}$$

The characteristic specimen strength σ_0 and the Weibull modulus m have to be determined using the maximum likelihood method. To account for the dependence of the strength on the specimen size the effective volume V_{eff} has been calculated for each type of experiment. The biaxial stress state in the discs was considered by using the principle of independent action (Danzer et al. (2007)).

3. Results and discussion

3.1. Strength distributions

The measured strength distributions for all experiments are well fitted by a Weibull distribution and are shown in a Weibull plot in Fig. 3. Results are summarised in Table 1. The estimated Weibull moduli are similar, their confidence intervals overlap each other. The obtained strength distributions of the discs are much higher than the strength determined with 4PB. The reason is the nearly thousand times smaller effective volume in the SPT and B3B test. In accordance to this, the fracture origins are located in a small volume at the center of the discs.

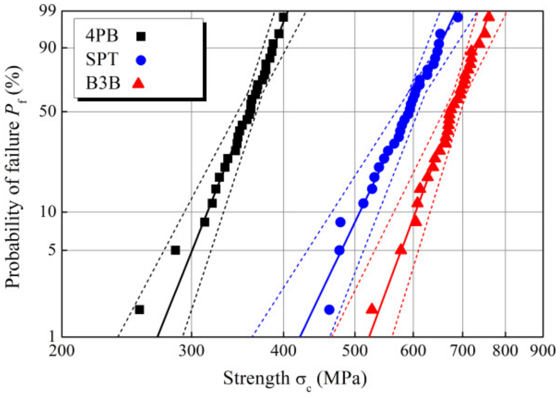

Fig. 2. Weibull plot of failure probability, P_f, versus specimen strength, σ_c, for the 4PB, SPT and B3B samples.

Table 1. Weibull parameters from experiments: Weibull modulus m, characteristic strength σ_0 (90% confidence intervals in square breackets) and the calculated effective volumes V_{eff}.

	4PB	SPT	B3B
m	14.3 [11.3, 18.3]	12.0 [9.4, 15.3]	15.5 [12.1, 19.7]
σ_0 (MPa)	366 [358, 375]	607 [591, 624]	694 [680, 709]
V_{eff} (mm^3)	1.438	0.00340	0.00206

3.2. Fracture toughness evaluation

The specimens prepared with surface cracks fractured at loads less than half the load in strength tests. This is a confirmation that the surface cracks do not compete with distinct smaller natural flaws. Fractographic investigations validate that the fracture origins were the artificial cracks. Instead of three fragments in the strength tests, always two fragments were observed.

The cracks in the SPT are always loaded in mode I to its axisymmetric geometry. Mode I loading was ensured in the B3B test too, because the cracks were orientated in such a way, that they are positioned in one of the three symmetry planes. To avoid measurement errors a carefully alignment of the crack to the cylinder axis of the disc as well as the coaxial alignment of the disc and the loading geometry are necessary. A first analysis of possible errors in the case of B3B is given in Strobl et al. (2014). A detailed analysis of positioning errors for both test methods will be presented in a subsequent paper.

The evaluation of the critical stress intensity factor is based on the fracture load and the crack size measurements on both fracture surfaces. An example of a fracture surface is shown in Fig. 3 (left). Crack sizes were in the range from 70 µm to 100 µm depth and 200 µm to 240 µm length. Table 2 reports the specimen thicknesses after grinding, the measured crack sizes and fracture loads for both tests together with the calculated fracture toughness. For simplicity the crack shape in the finite element models was approximated to be semi-elliptical with the horizontal major semi-axis c and the vertical minor semi-axis a. Fig. 3 (right) shows the calculated stress intensity factor K_I along the angle to the vertical axis in the SPT and B3B specimens for $a/c = 0.75$ and a load of 45 N. The maximum of K_I was found at the surface. The evaluated fracture toughness was 3.48±0.08 MPa·m$^{1/2}$ for the B3B test and 3.67±0.28 MPa·m$^{1/2}$ for the SPT (mean and standard deviation). The values are in good agreement with those obtained with the SEVNB method, which were 3.48±0.19 MPa·m$^{1/2}$ (Rasche et al. 2010).

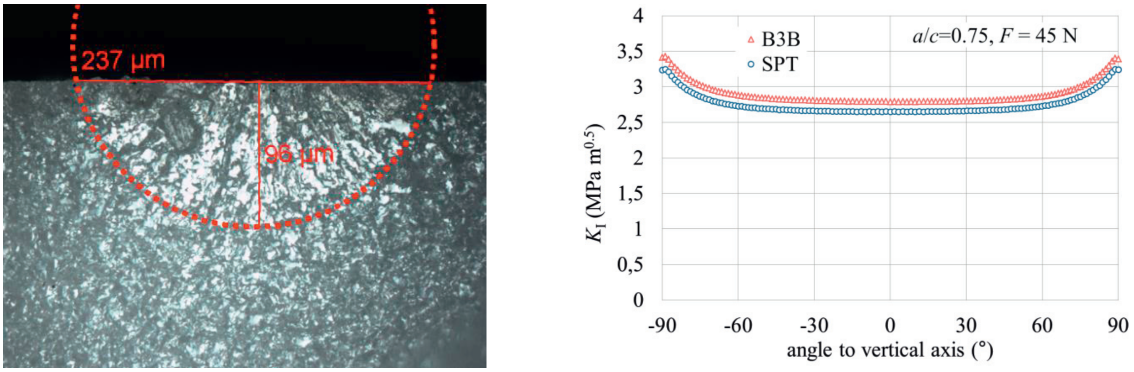

Fig. 3. (Left) Fracture surface of Al_2O_3–specimen P8 with measured indentation crack size. (Right) Stress intensity factor distribution in 0.55 mm thick B3B and SPT specimens modelled with a semi-elliptical surface crack of 80 µm depth and aspect ratio a/c=0.75 loaded with 45 N.

Table 2. Specimen thickness t, crack length $2c$ and crack depth a (average from both crack faces) as well as fracture load F and toughness K_{Ic} for five specimens tested in B3B test and SPT.

	B3B					SPT				
specimen number	P2	P3	P4	P5	P6	P8	P9	P10	P11	P12
t (mm)	0.547	0.545	0.540	0.546	0.549	0.544	0.538	0.543	0.548	0.551
$2c$ (µm)	204	211	231	228	222	236	226	203	229	229
a (µm)	76.8	80.4	85.6	80.8	83.1	97.2	86.3	72.6	96.7	89.3
F (N)	44.2	45.0	43.7	45.0	44.6	50.5	48.7	46.6	44.7	48.1
K_{Ic} (MPa√m)	3.36	3.51	3.57	3.49	3.47	4.04	3.79	3.32	3.53	3.62
	3.48±0.08					3.66±0.28				

4. Summary

The prediction of mechanical failure of small and thin ceramic components should be based on small-sized specimens. In this work, the strength distribution and the fracture toughness of small ceramic discs could be successfully determined by means of the SPT and B3B test. Both methods apply a biaxial stress field in the discs. The B3B test has the advantage of a well-defined three-point support. Therefore it is suited to investigate material samples with as-sintered surface closer to the component geometry. Instead the specimens for the SPT have to be very flat to avoid stress errors resulting from undefined contact situations, but deviations from linear elastic material behavior are detectable more easily, as the specimen deformation is measured during the test. In fact, both test methods can supplement each other for the characterization of brittle materials.

In this work both methods were applied to alumina ceramic discs of same size and preparation. Necessary stress and stress intensity factor calculations were performed with the finite element method. The obtained strength distribution in both tests was Weibull distributed and the characteristic strength was much higher compared to the bigger standard bending test. This is in agreement to the Weibull theory of strength which accounts for the size effect of strength. The new approach presented here is the fracture toughness determination using such small discs. Nearly semi-elliptical artificial surface cracks of only 80 µm depth were created in the tensile stresses center of the discs to act as fracture origin. Due to symmetry reasons the crack is loaded in mode I. The obtained K_{Ic} results from SPT and B3B are in good agreement to fracture toughness results from standardized test methods.

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