Ceramic Materials and Components for Energy and Environmental Applications

Ceramic Transactions, Volume 210

A Collection of Papers Presented at the 9th International Symposium on Ceramic Materials for Energy and Environmental Applications and the Fourth Laser Ceramics Symposium November 10–14, 2008, Shanghai, China

Edited by
Dongliang Jiang
Yuping Zeng
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Juergen Heinrich

The American Ceramic Society

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Preface

The global population growth and tremendous economic development has led to increasing demand for energy from all over the world as well as increasing concern for environment and global warming. The energy efficient and eco-friendly systems and technologies are critically needed for the further global growth and sustainable development. Advanced ceramics are enabling materials for a number of demanding energy efficient and eco-friendly applications in aerospace, power generation, ground transportation, nuclear, and chemical industries. These materials have unique properties such as high strength, high stiffness, long fatigue life, low density, and adaptability to the intended functions. Significant achievements have been made worldwide in the design, development, manufacturing, and application of these materials in recent years and considerable innovative research and technology development is still continuing to address technical and economic challenges.

9th International Conference on Ceramic Materials and Components for Energy and Environmental Applications (9th CMCEE) in Shanghai, China was continuation of series of international conferences held all over the world over the last three decades. The major goal of CMCEE was to bring together academicians, researchers, and end users in various disciplines from all over the world to share knowledge and exchange views leading to industrial applications of these technologies. The current volume contains selected peer reviewed papers from more than 300 presentations from all over the world. The papers in this volume also highlight and emphasize the importance of synergy between advanced materials and component designs. This volume also contains selected papers from 4th International Laser Ceramics symposium which was held during the same time period. We would like to thank organizers and sponsors of this symposium.

We would like to acknowledge the financial support from Chinese Academy of Sciences, Shanghai Municipal Corporation, and Shanghai Institute of Ceramics. Our special thanks to Abhishek Singh from Case Western Reserve University, Cleveland, Ohio for the editing of the manuscripts. We would also like to thank Mr. Greg Geiger, Technical Content Manager of The American Ceramic Society for all

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I. Basic Science, Design, Modeling and Simulation

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FRACTURE STATISTICS OF SMALL SPECIMENS

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ABSTRACT

Strength data of brittle materials show significant scatter. Therefore designing with brittle materials has to be made with probabilistic methods. So far this is done using Weibull statistics, which is based on the weakest link hypothesis. It (implicitly) implies a particular type of defect distribution, which can be observed in many (but not in all) ceramic materials. It is shown that for very small specimens the Weibull assumptions claim unrealistic high densities of flaws. Then the flaws will interact, they are not longer statistically independent and the weakest link hypothesis is not valid. Consequently the Weibull distribution predicts too high a strength for very small specimens.

INTRODUCTION

Fracture of ceramics usually initiates from flaws which are randomly distributed in the material. The strength of the specimen then depends on the length of the major flaw, which varies from specimen to specimen. The strength of brittle materials has to be described by statistical means [1 - 3]. It follows from experiments that the failure probability increases with load amplitude and with size of the specimens [1-5]. The first observation is trivial. The second observation follows from the fact that it is more likely to find a major flaw in a large than in a small specimen. Therefore the mean strength of a set of large specimens is smaller than that of small specimens. This size effect of strength is the most prominent and relevant consequence of the statistical behaviour of the strength of brittle materials.

Weibull developed his statistical theory of brittle fracture on the basis of the weakest link hypothesis, i.e. the specimen fails if its weakest element fails [6, 7]. In its simplest form and for an uniaxial homogenous and tensile stress state, σ, and for specimens of the volume, V, the so called Weibull distribution of the probability of failure, F, is given by:

$$F(\sigma,V)=1-exp\left[-\frac{V}{V_0}\left(\frac{\sigma}{\sigma_0}\right)^m\right] \quad .$$

The Weibull modulus, m, describes the scatter of strength data: the distribution is the wider the smaller m is. σ_0 is the characteristic strength and V_0 is the corresponding reference volume. Of course the probability of surviving (the reliability, R) is: $R=1-F$. Freudenthal [8] showed for sparsely distributed flaws, that the probability of failure only depends on the number of destructive flaws, $N_{c,S}$, occurring in a specimen of size and shape, S:

$$F_S(\sigma)=1-exp(-N_{c,S}) \quad .$$

$N_{c,S}$ is the mean number of destructive (critical) flaws in a large set of specimens (i.e. the value of expectation). Jayatilaka et al. [9] showed, that, for brittle and homogeneous materials, the distribution of the strength data is caused by the distribution of sizes (and orientations) of the flaws.

A Weibull distribution of strength will be observed for flaw populations with a monotonically decreasing density of flaw sizes. Danzer et al. [10 - 12] extended these ideas to flaw populations with any size distribution and to specimens with an inhomogeneous flaw population. On the basis of these ideas a direct correlation between the flaw size distribution and the scatter (statistics) of strength data can be defined.

The Weibull distribution is the state of the art statistics in the mechanical design process of ceramic components [1 - 3]. Strength testing and data evaluation are standardised. A sample of at least 30 specimens has to be tested. The range of "measured" failure probabilities increases with the sample size [3, 13] and is - for a sample of 30 specimens - very limited (it is between 1/60 and 59/60). To determine the design stress, the measured data have to be extrapolated with respect to the volume and to the "tolerated" failure probability. This often results in a very large extrapolation span [3].

In this paper the Weibull theory is applied to very small specimens. The analysis follows the ideas presented in [13]. The relationships between flaw population, size of the fracture initiating flaw and strength are discussed. It is shown that a limit for the applicability of the classical fracture statistics (i.e. Weibull statistics based on the weakest link hypothesis) exists for very small specimens (components).

FRACTURE STATISTICS AND DEFECT SIZE DISTRIBUTION

The function $N_{c,S}(\sigma)$ is obtained by integrating the local density, $n_c(\sigma,\vec{r})$, of destructive flaws

$$n_c(\sigma,\vec{r}) = \int_{a_c(\sigma)}^{\infty} g(a,\vec{r})\,\mathrm{d}a$$

over the volume of the specimen: $N_{c,S} = \int n_c \mathrm{d}V$ [3, 8 - 10]. For simplicity, but without loss of generality [8], it is assumed that size and orientation of a flaw are described by a single variable (the flaw size, a). The frequency distribution of the density of flaw sizes, $g(a,\vec{r})$, may depend on the position vector, $\vec{r}$. A local fracture criterion (e.g. the Griffith criterion, [1, 2]) correlates stress amplitude and flaw length: the critical flaw size is the smallest flaw length, which – under the action of the stress – causes failure (the size of the smallest destructive flaw). Since a_c depends on the magnitude of the applied stress, so do the values of n_c and also $N_{c,S}(\sigma)$. For a homogeneous material loaded under uniaxial homogeneous tension the volume integral is trivial. For a flaw population with relative frequencies decreasing with a negative power the flaw size, a,

$$g(a) = g_0 \cdot (a / a_0)^{-r}$$

a Weibull distribution (eq. 1) occurs [9]. This function has only two independent parameters: the exponent $(-r)$ and the coefficient ($g_0 \cdot a_0^{\,r}$). Using these assumptions and after some algebra the density of destructive flaws in terms of a critical flaw size is: $n(a_c) = (a_c \cdot g(a_c)) / (r-1)$. The critical flaw size can be defined using the Griffith/Irwin criterion [1 - 3]:

$$a_c = \frac{1}{\pi} \cdot \left(\frac{K_{Ic}}{Y \cdot \sigma} \right)^2 \quad .$$

K_{Ic} is the critical stress intensity factor (the fracture toughness) and Y is a dimensionless geometric factor. Inserting in the above expression analytical equations for the Weibull parameters results: The Weibull modulus is only related to the path of the flaw size distribution:

$$m = 2 \cdot (r-1) \quad .$$

The second parameter in the Weibull statistics is: $V_0 \cdot \sigma_0^m = \left((r-1)/g_0 \cdot a_0\right)\left(K_{Ic}/Y\sqrt{\pi \cdot a_0}\right)^{-(\ \ )}$.
In the following a material behaving in the way as described above (eq. 1, eq. 4, etc.) is called "Weibull material".
A Weibull type strength distribution also may arise for inhomogeneous stress and non uniaxial stress states (then the volume has to be replaced by an effective volume, [1 - 3]). If failure is caused by surface flaws, the volume has to be replaced by the surface [1, 3, 12].
Danzer et al. have discussed the influence of other types of flaw populations (e.g. of bimodal distributions) on strength [11, 12]. In these cases the Weibull modulus might depend on the applied load amplitude and on the size of the specimen. Then the determination of a design stress in the usual way may become problematic. A stress and size dependent modulus occurs for materials with an R-curve behaviour [11] and may also be caused by internal stress fields [11].
It should be noted that on the basis of a small sample size, e.g. only 30 specimens, it is not possible to differentiate between a Weibull, a Gaussian, or any other similar distribution functions, as shown by Lu et al. [14] using statistical measures or by Danzer et al. [12] using Monte Carlo simulations. This is caused by the inherent scatter of the data and the difference between sample and true population. The ultimate test for the existence of a Weibull distribution is to test a material on different levels of (effective) volumes.

THE CORRELATION BETWEEN STRENGTH AND FLAW POPULATION

In the following, the relationship between fracture statistics and defect size distribution is discussed for the simple case of tensile tests (uniaxial and homogeneous stress state) on a homogeneous brittle material. The tests are performed on specimens of equal size. It is assumed that the volume of the specimens is: $V = V_0$. The number of tested specimens (the sample size) is X. In each test the load is increased up to the moment of failure. The strength is the stress at the moment of failure. In each sample the strength values of the individual specimens are different, i.e. the strength is distributed.

If data determined in that way are evaluated the specimens are ranked according to their strength, i being the ranking parameter. To estimate the failure probability for an individual specimen an estimation function is used [1, 3, 13]:

$$F_i = (i-1/2)/X \qquad , \qquad i = 1, 2, ? \quad X \quad .$$

Inserting eq. 7 into eq. 2 and making a few rearrangements, we get: $N_{c,S}(\sigma_i) = \ln 2X/(2X - 2i + 1)$. In this way the mean number of critical flaws per specimen (volume V_0; stress σ_i) can be read from the ranking number and the sample size. For the weakest specimen $(i = 1)$ of a sample the estimator for the probability of failing is: $F_1 = F(\sigma_1) = 1/2X$. That specimen contains on average $N_{c,S}(\sigma_1) = \ln 2[X/(2X-1)]$ destructive flaws. For the strongest specimen of the sample $(i = X)$ it holds that: $F_X = F(\sigma_X) = (2X-1)/2X$ and $N_{c,S}(\sigma_X) = \ln 2X$.

A special situation occurs if the strength is equal to the characteristic strength (i.e. for $V = V_0$ and $\sigma = \sigma_0$). Then the probability of failure is $F(\sigma_0) = 1 - 1/e$ and $N_{c,S}(\sigma_0) = 1$ and the density of critical flaws is: $n_c(\sigma_0, V_0) = N_{c,S}(\sigma_0)/V_0 = 1/V_0$. If the calculations made for $\sigma = \sigma_0$ and $V = V_0$ are generalized for any stress value σ_i and for specimens of any volume V the equation reads:

$$g(a_{c,i}) = \frac{r-1}{V \cdot a_{c,i}} \cdot ln \frac{2X}{2X - 2i + 1}$$

The use of this equation opens a simple possibility to determine the frequency distribution of flaw sizes in a wide range of parameters by testing specimens of different volume.
In the following these ideas are applied to describe bending test results of a commercial silicon nitride ceramic.

FRACTURE AND FLAW STATISTICS OF A TYPICAL COMMERCIAL SILICON NITRIDE MATERIAL

A typical commercial gas pressure sintered silicon nitride ceramic is used as model material. Its hardness (HV5) is 15.5 ± 0.3 MPa, the fracture toughness (SEVNB, [15, 16]) is 5.0 ± 0.2 MPa√m, the Young's Modulus is 297 ± 2 GPa and the Poisson ratio is 0.27. More details can be found in [19]. A sample of 30 bending specimens was machined out from large discs (diameter 250 mm, thickness 5 mm). The specimen preparation and the tests were done according to EN 843-1 and evaluated according to EN 843-5 [17, 18]. The characteristic strength of this sample is σ_0 = 871 MPa and the Weibull modulus is m = 14.1. The effective volume [1] of the bending specimens is $V_{eff} = V(m+2)/4(m+1)^2 = 8.5 \cdot 10^{-9}\ \mathrm{m}^3$. This value is used as scaling parameter $V_0 = V$. The Weibull distribution is shown in **Fig. 1.a**. The measured data are nicely distributed around the straight line, which describes the Weibull distribution.

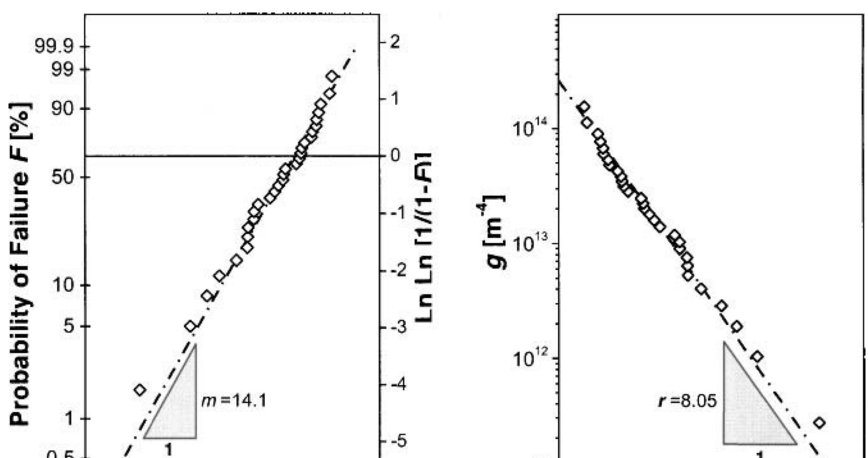

Fig. 1: a) Strength data of a silicon nitride ceramic tested in four point bending (4PB) in a Weibull plot and b) the relative frequency distribution of flaw sizes. The data points were determined by fracture experiments.

By fractographic inspection fracture [20, 21] origins within the volume (inclusions) were found in 3 specimens. In the other 27 specimens no clear evidence for the type of fracture origin was found. It is assumed that all flaws are small surface flaws, i.e. their geometric factor is $Y = 1.12$.

In the following, the strength data are used to determine the frequency distribution function of the defects. Using eq. 5 the size of the critical flaw for the characteristic strength is $a_{c,0} = 26 \cdot 10^{-6}$ m. This value is used in the following for the arbitrary scaling parameter: $a_0 = a_{c,0}$. For the exponent r we get using eq. 6: $r = 8.05$. With these data the factor in eq. 4 is: $g_0 = 3.2 \cdot 10^{13}$ m^{-4}, and therefore all parameters in eq. 4 are determined. The relative frequency distribution of flaw sizes is shown in **Fig. 1.b**. The transformation of strength into flaw size data was done using eq. 8. Again the data are nicely distributed around the straight line. This is characteristic for a Weibull material. An extremely strong dependency of the relative frequency on the critical flaw size can clearly be recognized: although the size of the critical flaws in the samples only varies by a factor of around 2.2 the corresponding relative frequency of the flaw sizes varies by a factor of around 500. That means that flaws with a radius of 21 μm are about 500 times more frequent than flaws with a radius of 46 μm.

SIZE EFFECT ON STRENGTH; APPLICATION TO VERY SMALL SPECIMENS

As discussed in the introduction a size effect on strength exists [1 - 3], which – for a Weibull material - is described by:

$$V_1 \cdot \sigma_1{}^m = V_2 \cdot \sigma_2{}^m$$

The probability of failure in a sample of specimens of volume V_2 is equal to that in another sample containing specimens of volume V_1, if the stresses applied to the specimens are related according to eq. 9. Using the Griffith criterion we get [13]:

$$a_{c,1} / a_{c,2} = (V_1 / V_2)^{2/m}$$

For the specimens with volume V_1 the corresponding relative frequency of flaw sizes at $\sigma = \sigma_0$ is $g(a_1) = g_0 \cdot (a_1 / a_0)^{-r}$ and the density of destructive flaws is $n_c(\sigma_{0,1}, V_1) = 1/V_1$. The analogue holds for specimens of volume V_2.

In **Fig. 2** the diameter ($2a_c$) of the critical flaws (for the characteristic strength of the specimens) is plotted versus the (effective) volume in a double logarithmic scale (eq. 10) using the example of the silicon nitride material described above. The slope of the line is: $1/(m/2) = 1/(r-1) = 1/7.05$. The dashed line describes a typical scaling parameter for the volume of the specimen. For simplicity the edge length of a cube with volume V is taken as the characteristic length: $l = V^{1/3}$. For materials with a modulus $m > 6$, there exists a point of intersection between both lines, which is - in the selected example - at a volume of about $V \approx 4.2 \cdot 10^{-17}$ m^3 (this corresponds to the diameter of the critical flaw of about $2a_c \approx 3.4$ μm).

Obviously the assumption made in eq. 4 (the relative frequency of flaws follows an inverse power law) can only approximate the behaviour of materials for large flaws. It fails for very small flaws: the relative frequency goes to infinity if the flaw size goes to zero: $a \rightarrow 0$, $g(a) \rightarrow \infty$ [13]. At the intersection point in **Fig 2**, the density of dangerous flaws gets so high that the volume of the specimens is completely filled with flaws and, left of that point; the "volume of dangerous flaws" even exceeds volume of the specimens. For obvious reasons this is not possible in real materials.

Another inconsistency is caused by the fact, that the derivation of the fracture statistics (eq. 1 and eq. 2) assumes non-interacting flaws [8]. This will only be true in the case of a low flaw density. If fracture statistics are applied to very small specimens made of a Weibull material the density of dangerous flaws gets high and the interaction between flaws cannot be neglected any longer [22]. For that case it can be assumed that interacting flaws link up. This would cause an upper limit for

Table 1: Strength test on specimens of different size.					
Data set	**4PB**	**A (B3B-test)**	**B (B3B-test)**	**C (B3B-test)**	**D (B3B-test)**
Specimen dimension / mm	45×3.95×2.98	∅ 43; t = 2.484	∅ 20; t= 1.983	∅ 10.8, t = 1.054	∅ 4.7; t = 0.445
Charact. strength / MPa	871	1053	1123	1226	1275
$2a_{c,0}$ / µm	52	36	31	26	24
Weibull modulus / -	14.1 [11 -17]	12.4 [9 – 15]	15.9 [12 – 19]	21.7 [16 – 26]	17.7 [13– 22]
effective volume / 10^{-12} m^3	8500	280	65	4.3	0.6
x_s / µm	2041	654	402	163	84
$x_s / 2a_{c,0}$ / -	39	18	13	6.3	3.5

4PB: four point bending test; B3B: ball on three balls test; ∅ : diameter; t: thickness; $2a_{c,0}$: diameter of the critical flaw corresponding to the characteristic strength, l: reference length (defined to be the third root of the effective volume). Numbers in square brackets are limits of the 90 % confidence interval.

the strength, if the distance between the flaws gets too close, say 2 – 3 times their diameter.
Further strength tests (data sets A- D) were made in biaxial bending on specimens of different size. Specimens were cut from the same plates as used for the bending specimens. Tests and results are described in [19]. Key results are summarised in **Table 1**. The data show a significant size effect, i.e. the characteristic strength is much larger for small than for large specimens (**Fig. 3**). The straight line shows the size effect as predicted by eq. 9 based on the bending test data. Although the data sets A and B are in the 90 % confidence interval of the extrapolation, the sets C and D show a significantly lower strength than predicted.
The behaviour of small specimens is discussed in more detail in [13]. A possible reason for this drop of strength is the fact that machining of very small specimens (as is the case of set D) is very demanding and some machining damage cannot be excluded in this case. Additional damage would cause a reduction of strength as observed in **Fig. 3**. Further possible reasons for the (apparent) deviation of the strength of small specimens from the Weibull behaviour are experimental measurement uncertainties, which become large for small specimens and which are not included in the scatter bars shown in **Fig. 3**. The plotted scatter bars refer to the uncertainties due to the sampling procedure (the sample is different from the underlying population, [12]).
Another reason would be the interaction between flaws, as described above. The last line in **Table 1** shows the ratio of the size parameter (corresponding with the effective volume; it is the length of the edge of a cube with the effective volume) divided by the diameter of the critical Griffith flaw for the characteristic strength. This ratio is larger than 10 for the sets 4PB, A and B. Here an interaction seems not to be likely. But for set D the ratio is as small as about 3. Here some overlapping of local stress field and linking of micro defects may become possible. But at present it is not clear if this really happens or not.
Bazant formulated a statistical theory of fracture for quasibrittle materials [5, 23, 24]. He assumed that there exist several hierarchical orders which can be described by parallel and serial linking of so-called representative volume elements (RVEs). For large specimens (and low probability of failures) the fracture statistics is equal to the Weibull statistics , i.e. if the specimens size is larger than 500 to 1000 times of the size of one RVE. In the actual case this is similar to the diameter of the critical flaw. For smaller specimens the volume effect disappears and the fracture