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Influence of sample size on strength distribution of advanced ceramics

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Abstract

Strength distribution of advanced ceramics is mostly characterized by Weibull distribution function. The question whether the Weibull distribution always gives the best fit to strength data has been being considered in the last years. The sample size affects the reliable decision of discrimination of different distribution functions (e.g. normal, log-normal, gamma or Weibull). In this paper, 5100 experimental alumina strength data and virtual strength data generated by Monte Carlo simulations are used in order to investigate the effect of sample size on strength distribution of advanced ceramics. It is suggested that, at least 150–200 samples should be used for determination of best fitting distribution function with a statistical fallibility of 10%. Extreme Value Analysis performed with the experimental strength data showed that the Weibull distribution fits the data best and difference between the Weibull and Gumbel distributions appear at the tails.

Keywords: B. Failure analysis; C. Strength; D. Al₂O₃; Sample size; Extreme Value Statistics

1. Introduction

Fracture of ceramics initiates from pre-existing crack-like defects and flaws [1,2]. These flaws may be volume flaws that occur during the sintering process of a ceramic material and/or surface flaws that appear during its machining process. The strength of a ceramic is inversely proportional to the square root of the size of the most critical crack in material [3,4]. Therefore, the strength of a ceramic specimen is determined by the existing most critical crack in the volume or on the surface of the part. In 1921, Griffith [5] performed fracture experiments with glass fibers and observed that the fracture stress increases as the fiber diameter decreases. The strength depends on the stressed area or volume of a material because a larger area or volume increases the probability of existence of a critical flaw [6,7]. Here the most critical flaw does not always represent the largest flaw in the material. The size, orientation and position of a crack determine whether a crack is critical or not. The cracks are randomly distributed in the material and the position, size and orientation of the most critical flaw show scattering. Due to this scattering, strengths of ceramics vary

*Tel.: +90 342 211 6789; fax: +90 342 211 6677. E-mail address: serkan.nohut@zirve.edu.tr from components to components, even if identical specimens are tested. Since the strength of ceramics is not a deterministic value, a probabilistic method is recommended for the design of advanced ceramics [8–12].

The Weibull distribution [13], based on the concept of the weakest link, is the most widely used formulation for the strength characterization of ceramics [14,15]. In most cases, it is enough to use two-parameter form of the Weibull distribution for reliable ceramic component design [16].

The question whether the Weibull distribution always gives the best fit to strength data has been investigated in literature in recent years. For example, Danzer [17] performed experiments with small specimens and observed that the Weibull theory is insufficient in estimating the strength behavior because the fracture origins are larger than the effective volume of the specimens [14]. Some possible microstructural reasons may cause deviation from the Weibull distribution. Such deviations occur in ceramics which have multi-modal flaw size distribution, R-curve behavior, subcritical crack growth and internal residual stresses [11,18]. These microstructural activities cause applied stress dependent Weibull modulus. Lu et al. [12] investigated the strength data of Si₃N₄, SiC and ZnO ceramics and reported that the normal distribution fits the strength data of ZnO better than the Weibull distribution. Basu et al. [19] carried out the statistical

analysis of strength data of monolithic ZrO_2 , ZrO_2 – TiB_2 composites, glass and Si_3N_4 by using the probability models of the Weibull, normal, log-normal, gamma and generalized exponential distributions and reported that the gamma or log-normal distribution, in contrast to the Weibull, may more appropriately describe the measured strength data. Nohut and Lu [20] applied involving normal, log-normal and Weibull distributions to the analysis of ten strength data sets of dental ceramics with different compositions and concluded that various microstructures and compositions in the investigated dental ceramics cause their strength distributions deviated from the Weibull distribution.

Fracture experiments (e.g. three-point bending test, fourpoint bending test, ball-on-three balls test) are performed in order to obtain strength data of a ceramic material. The Weibull statistics is applied to strength data of advanced ceramics according to DIN V ENV843-5 standard [21] which suggests at least 30 specimens to be tested for a reasonable determination of Weibull parameters due to high production and machining costs. Danzer et al. [22] investigated the effect of number of specimens on the Weibull parameters of a silicon nitride and concluded that at least 30 specimens should be tested for the determination of Weibull parameters with a reasonable standard deviation. The discrimination of different distribution function of strength of advanced ceramics has been investigated in some articles with 30 samples and some even with less number of specimens [12]. Here the question arises whether 30 strength data is enough for a clear distinction between different distribution functions. In literature, there is no study which investigates the effect of sample size on statistical fallibility in determination of most suitable distribution type due to lack of large experimental data. In this article, 5100 experimental alumina strength data, provided by Lovro Gorjan, Hidria AET [23] is used in order to investigate the effect of sample size on Weibull parameters and on the correct determination possibility of best distribution function. The determination error is given as a function of sample size. Moreover, virtual strength data generated by Monte Carlo simulations is used in order to compare with the experimental results. By random selection of strength values as a sample size from 10 to 100, the distribution of experimental Weibull modulus will be investigated.

For a reliable design with advanced ceramics, low failure probabilities are of interest. When the low failure probabilities are expected, the Extreme Value Statistics should be performed [24]. Extreme value theorem or extreme value statistics deals with the extreme deviations from the median of probability distributions. Unlike from classical statistics which focus on the average behavior of stochastic process, extreme value theory focuses on the extreme and rare events. In the final part, the Extreme Value Analysis of the strength distribution of alumina ceramics is given.

2. Theoretical background

2.1. Distribution Functions

In the design of advanced ceramics, the design is said to be reliable when the failure probability is in the order of approximately 10⁻⁶. This means, for a reliable design, 10⁶ experiments have to be performed in order to catch the tail properties of strength distribution. Since application of 10⁶ experiments is economically not feasible, statistical models are used for the determination of strength distribution by using lower number of experiments. It is expected that the distribution function should predict so low failure probabilities by using limited number of experimental strength data in a reliable manner. In this article, four different distribution functions are used (normal, log-normal, Gamma and Weibull distributions) for fitting the strength of alumina ceramic specimens.

Normal distribution is extremely important in statistics and is often used in the natural and social sciences for real-valued random variables whose distributions are not known. The strength distribution of a brittle material without surface preparation shows a symmetrical behavior and therefore normal distribution may be a potential distribution to fit such a data [25,26]. The pdf of a normal distribution is represented as

$$p(\sigma) = \frac{1}{\sqrt{2\pi\alpha}} \exp\left[-\frac{(\sigma - \overline{\sigma})^2}{2\alpha^2}\right]$$
 (1)

where $\overline{\sigma}$ and α^2 are the mean and variance, respectively and can be calculated as;

$$\overline{\sigma} = \frac{1}{n} \sum_{i=1}^{n} \sigma_i$$
 and $\alpha^2 = \frac{1}{n} \sum_{i=1}^{n} (\sigma_i - \overline{\sigma})^2$ (2)

The log-normal distribution is a distribution of a random variable whose logarithm is normally distributed. Thus, its pdf can be written as

$$p(\sigma) = \frac{1}{\alpha \sigma \sqrt{2\pi}} \exp\left[-\frac{(\ln \sigma - \overline{\sigma})^2}{2\alpha^2}\right]$$
 (3)

If a data is distributed lognormally with parameters $\overline{\sigma}$ and α , then the logarithm of the data is distributed with a mean $m = \exp(\overline{\sigma} + \alpha^2/2)$ and variance $v = \exp(2\overline{\sigma} + \alpha^2)$ [$\exp(\alpha^2) - 1$].

The gamma distribution, like the log-normal distribution, is an alternative to analyze highly skewed data. The general formula for the probability density function of the gamma distribution is;

$$p(\sigma) = \frac{1}{\rho^k} \frac{1}{\Gamma(k)} \sigma^{k-1} e^{-(\sigma/\theta)} \tag{4}$$

where k is the shape parameter, θ is the scale parameter and Γ is the gamma function which has the formula

$$\Gamma(a) = \int_{0}^{\infty} t^{a-1} e^{-t} dt \tag{5}$$

Weibull distribution is a type of Extreme Value Distribution (EVD) and it uses Extreme Value Statistics (EVS). The problem of modeling rare events is applied in many areas where such events can have very negative dangerous consequences. Unlike from classical statistics which focus on the average behavior of stochastic process, extreme value theory focuses on the extreme and rare events. Assume that we have a vector of samples $\{X_1, X_2, X_n\}$ from an arbitrary population. The maximum value from the sample vector is selected from the parent distribution by the operator, max $\{X_1, X_2, X_n\}$. Dealing with the maximum values

is a kind of studying the tails of parent distribution and relevant to the right tail of the distribution. For the left tail, the minimum values are used [27].

Extreme value statistical models are used when the tails are of interest. Three main extreme value statistics are used for the distribution of maximum or minimum of the sample for infinite number of samples [28–31].

• Type I-Gumbel Distribution

$$G(x) = \exp(-\exp^{-(x-\mu)/\sigma})$$
 (6)

• Type II-Frechet Distribution

$$G(x) = \begin{cases} \exp(-((x-\mu)/\sigma)^{-\xi}) & \text{for } x \ge \mu \\ 0 & \text{otherwise} \end{cases}$$
 (7)

• Type III-Weibull Distribution

$$G(x) = \begin{cases} \exp(-((\mu - x)/\sigma)^{\xi}) & \text{for } x \le \mu \\ 0 & \text{otherwise} \end{cases}$$
 (8)

where G(x) is the probability distribution, μ is the location parameter, σ is the scale parameter and ξ is the shape parameter [32]. Unlike from Gumbel distribution which is unbounded in the direction of extreme value, the Weibull distribution is bounded in the direction of extreme value [33–35]. The Frechet distribution does not possess finite moments and therefore is not suitable for real experimental data due to its unlimited extension incompatible with a minimum lower bound value of the strength. In the last part of this article, the Frechet distribution is still used in order to verify the above expression.

The most widely used formulation of Weibull distribution which is used in the design of advanced ceramics is written as [11,12]

$$P(\sigma, V) = 1 - \exp\left[-\frac{V}{V_0} \left(\frac{\sigma}{\sigma_0}\right)^m\right]$$
 (9)

where $P(\sigma, V)$ is the cumulative failure probability of a ceramic component due to flaws, V is the volume of the component, V_0 is the unit volume, σ is the uniaxial applied stress, m is the Weibull modulus, and σ_0 is the characteristic stress at which the failure probability is 63.2% for a specimen with $V=V_0$. Although in most of the ceramic articles it is stated that m alone describes the scatter of strength, from statistical point of view, characteristic strength has also an effect on the scatter of strength data due to the fact that the variance (or standard deviation) of the Weibull distribution, i.e., the scatter of it, depends on both shape and scale parameters [24].

Then, if the volume of the specimen is assumed to be equal to the unit volume, its probability distribution function (pdf) can be written as;

$$p(\sigma) = \frac{m}{\sigma_0} \left(\frac{\sigma}{\sigma_0}\right)^{m-1} \exp\left[-\left(\frac{\sigma}{\sigma_0}\right)^m\right]$$
 (10)

Eq. (9) represents the so called two-parameter Weibull distribution function. There is also a more comprehensive form of the function proposed by Weibull, the so called three-parameter Weibull function, in which the stress σ is replaced by $(\sigma - \sigma_{th})$. Three-parameter Weibull function is a more conservative function [36] when the very low failure probability, "extreme value for minima" is not of interest. In this form of Weibull function, there are three parameters which control the failure probability of a ceramic component, namely σ_0 , m and σ_{th} . σ_{th} is the threshold stress below which no failure occur. Threshold stress is also a measure of scatter of strength of identical ceramic specimens [37].

In most practical and academic applications, two-parameter Weibull distribution is a more preferred function. The first reason of the popularity of using two-parameter Weibull distribution function for fitting the failure strength of advanced ceramics is the ease of determination of Weibull parameters. On the other hand, determination of parameters of three-parameter Weibull distribution is more complicated [38,39]. Moreover, since the number of samples in practical applications is small, the fitting of threshold stress is not stable and based on the sample size [11,40,41].

Normally it is expected that three-parameter Weibull distribution fits the data better than two-parameter Weibull distribution does due to the fact that the number of fitting parameters is higher and with more parameters better fitting can be achieved. However this is not always valid in all cases [17,42,43]. Curtis and Juszczyk [42] fitted the strength data of toughened glass and phosphate-bonded investment by two- and three-parameter Weibull distribution models and reported that the results were similar for chemically toughened glass. The three-parameter Weibull distribution was found to be more reliable for phosphate-bonded investment. Papargyris [43] used Remblend China clay strength data for the determination of fitting performance of two and threeparameter Weibull distribution and according to the computed correlation coefficients they found out that two and threeparameter Weibull distribution provided similar results for a range of specimen numbers starting from 29 and going up to 144. Due to difficulties in the parameter estimation and questionable positive effect on better fitting ability of three-parameter Weibull distribution, two-parameter Weibull distribution is used in this article.

2.2. Akaike information criterion (AIC)

The Akaike Information Criterion (AIC) measures the goodness-of-fit of an estimated statistical model by linking the likelihood to a distance between true (experimental) and assumed distributions [44]. The AIC index which has been used in a number of areas as an aid to select between competing models is defined as

$$AIC = -2 \ln \circ L + 2k \tag{11}$$

where k is the number of parameters to be fitted (for example, k=2 for a two-parameter Weibull distribution), $\ln \circ L$ is the maximized log-likelihood for a given model and can be

calculated by

$$ln L = \sum_{i=1}^{n} ln f(Y_i)$$
(12)

where *n* is the number of data and $f(Y_i)$ is the pdf of an estimated distribution. The *AIC* values can be directly compared, preferring the distribution which gives the smallest value and the difference in *AIC* values $\Delta AIC > 1.5-2$, corresponds to a reliable indication that one distribution is superior to another [16,45].

2.3. Anderson–Darling test (A–D test)

The Anderson–Darling test is a statistical test which checks whether a sample of data comes from a population with a specific distribution [46]. It is a modification of the Kolmogorov–Smirnov (K–S) test which is distribution free in the sense that the critical values do not depend on the specific distribution being tested [47]. The Anderson–Darling test makes the use of the specific distribution in calculating critical values. This has the advantage of allowing a more sensitive test. The critical values for different types of distribution functions (e.g. exponential, extreme-value, Weibull, gamma, logistic, Cauchy) are available in the literature [48–50]. Tests for the log-normal distribution can be implemented by transforming the data using a logarithm and using the above test for normality. A test for the two-parameter Weibull distribution can be obtained by making use of the fact that the logarithm of a Weibull variation has a Gumbel distribution

The simplest formula for the Anderson–Darling statistic *A* of the ordered data

$$A^2 = -n - S \tag{13}$$

where

$$S = \sum_{i=1}^{n} \frac{2i-1}{n} \left[\ln F(Y_i) + \ln(1 - F(Y_{n+1-i})) \right]$$
 (14)

F is the cumulative distribution function of the specified distribution and n is the sample size. The hypothesis regarding the distributional form is rejected at the chosen significance level (α) if the test statistic, A^2 , is greater than the critical value.

3. Materials and methods

3.1. Alumina specimens

The strength data of 5100 alumina samples was provided by Lovro Gorjan, Hidria [23]. Alumina specimens were manufactured by injection-molding of powder containing 96 wt% alumina (Martoxid MR 32 and MR23, Martinswerk, Bergheim, Germany) and 4 wt% steatite (MgSiO₂ powder, Hidria AET, Tolmin, Slovenia) [23]. Paraffin wax was used as a binder. The binder was removed by a thermal debinding process with a capillary extraction agent (highly porous alumina) and the specimens were sintered at 1640 °C for 3 h [51] (Fig. 1).

3.2. Experimental setup

The flexural strength of alumina at room temperature was measured by using a 4-point bending test according to ASTM C 1161-94.

4. Results and discussion

In this paper, 5100 experimental alumina strength data and virtual strength data generated by Monte Carlo (MC) method are used for reliability analysis and compared with each other. In order to determine which type of Weibull distribution will be used for the data generation, the experimental strength data is fitted first by two-parameter and three-parameter Weibull distribution functions. In Fig. 2, the probability density function versus strength is given for two-parameter and three-parameter Weibull distribution functions. Fitting by two- and three-parameter Weibull distribution gives almost the same results.

When A–D Test is applied, the *A* statistic values were computed as 1.3328 and 1.3968 for two-parameter and three-parameter

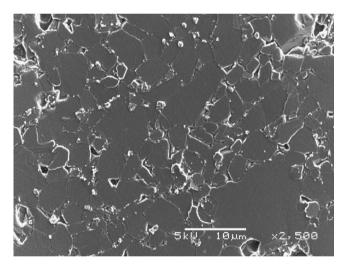


Fig. 1. Scanning electron microscopy images of the alumina specimen surface which was thermally etched for 1 h at $1400\,^{\circ}$ C.

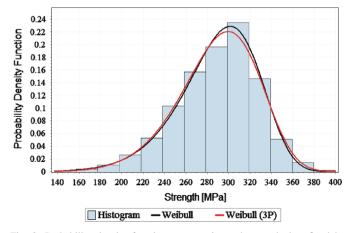


Fig. 2. Probability density function vs. experimental strength data fitted by tw0-parameter and three-parameter Weibull distribution.

Weibull distributions, respectively. This shows that two-parameter Weibull distribution provides a better fit. In Fig. 3, the Weibull plot of the experimental strength data is represented where the red dashed line shows the linear Weibull plot.

In this part of the discussion, the fitting capability of any distribution to all data is investigated. When only the lower tail of the distribution, i.e. minima values, is analyzed, it is seen in Fig. 3 that, the two-parameter Weibull distribution shows a deviation and separates from the straight line tending asymptotically to a certain value, the so-called Weibull location parameter. Therefore using three-parameter Weibull distribution would be more suitable when very low failure probabilities is of interest. However, since two-parameter Weibull distribution is more commonly used in practical design procedure, here the two-parameter Weibull distribution is used for comparison with other distribution functions.

The Weibull parameters of the experimental data, calculated by using the Maximum Likelihood method are, m=9.048 and $\sigma_0=305.5$ MPa. These Weibull parameters will be taken as true Weibull parameters ($m_{\rm true}$, $\sigma_{0,\rm true}$) while generating the virtual strength data. For the generation of N number of strength data with MC method, N failure probability values are assigned by using two-parameter Weibull distribution randomly in the range between 0 and 1 and N strength data are generated by using the following equation;

$$\sigma_i = \sigma_0 \left(\frac{V_0}{V} \ln \left(\frac{1}{1 - F_i} \right) \right)^{1/m} \tag{15}$$

where 0 < i < N. By putting the experimental Weibull parameters into Eq. (15), 5100 virtual strength data is generated. The Weibull plot of the virtual strength data is shown in Fig. 4.

Since two-parameter Weibull distribution was used for the strength data generation by MC, the points in Fig. 4 fit very well to a straight line.

In Table 1, the AIC values of experimental and MC data fitted by Weibull, normal, log-normal and Gamma distributions are given.

Fitting both data with Weibull distribution gives the lowest AIC value and this shows that the experimental and MC strength data are both Weibull distributed.

For the investigation of effect of sample size on determination of best fitting distribution function, a computer code was written in MATLAB. Let us assume that there are *j* different

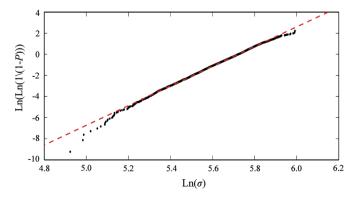


Fig. 3. Weibull plot of experimental alumina strength data.

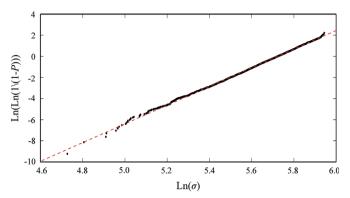


Fig. 4. Weibull plot of MC alumina strength data.

Table 1 AIC values of experimental and MC data fitted by Weibull, normal, log-normal and Gamma distributions.

| | Experimental Data | MC Data | |
|------------|-------------------------|---------------------|--|
| Weibull | 5.124 × 10 ⁴ | 5.146×10^4 | |
| Normal | 5.144×10^4 | 5.181×10^4 | |
| Log-normal | 5.194×10^4 | 5.247×10^4 | |
| Gamma | 5.174×10^4 | 5.221×10^4 | |

sample groups. Each group contains the number of strength data from n=10 to 300. In each sample group, strength data are randomly selected from the experimental and MC data set and then ranked in an ascending order. Then by using Eqs. (8) and (9), the most suitable distribution function is determined. This procedure is repeated 10,000 times and the percentages of best fitting of Weibull, normal, log-normal and Gamma distributions are calculated.

In Fig. 5(a and b), the percentages of best fitting functions of 10,000 simulations are given as a function of sample size for experimental and MC data, respectively.

For the sample size of 10, the probability for normal distribution to be the best fitting function is lower than lognormal and Gamma distributions. For the sample sizes lower than 50, the percentage of normal distribution increases and for sample sizes larger than 50, it again decreases. If the sample size is higher than 50, the probability of Gamma and lognormal distributions functions to be the best fitting function is almost zero. The ratio of the number of simulations for which the Weibull distribution appears to be the most suitable distribution to 10,000 simulations increases continuously as the sample size increases. For the sample size of 300, more than 90% of 10,000 simulations are fitted with Weibull distribution best. For the MC data the same behavior is observed. However, the weight of Weibull distribution converges to almost 100% faster since the deviation of this data from perfect Weibull distribution is smaller than experimental data. Here it is known that both the experimental data and MC data are Weibull distributed. When only 30 samples are used for the discrimination of different distribution functions, the percentage of Weibull distribution is about 70% according to experimental data. This means that the probability of making

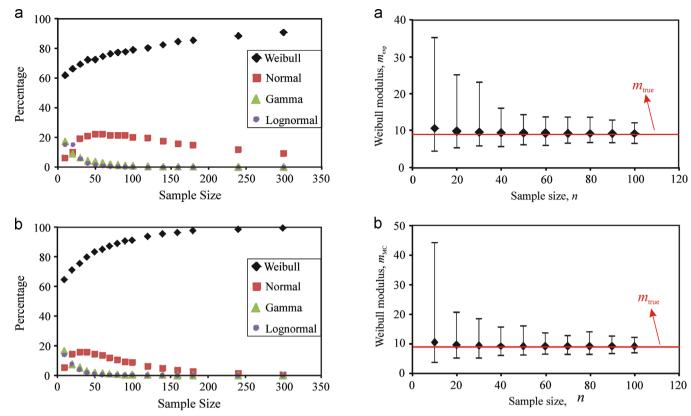


Fig. 5. Percentage of Weibull, normal, log-normal and Gamma distributions from 10,000 simulations for (a) experimental data and (b) MC data.

Fig. 6. Effect of sample size on Weibull modulus of (a) experimental data $m_{\rm exp}$ and (b) MC data $m_{\rm MC}$.

an error in distribution type discrimination is almost 30%. According to Fig. 5(a), it can be said that at least 150–200 specimens should be used for a relative reliable discrimination of different distribution functions.

In Fig. 6(a and b), effect of sample size on Weibull modulus of experimental data and MC data are represented respectively. The range of values that Weibull modulus takes for each sample size from 10,000 simulations is shown by error bars. For both data, the average Weibull modulus of calculated by averaging the Weibull modulus of 10,000 simulations converges to the true Weibull modulus ($m_{\text{true}} = 9.048$) as the sample size increases. However, for example if 10 specimens are used for the determination of Weibull parameters of experimental data, the Weibull modulus takes values between 5 and 35. For the MC data, the Weibull modulus takes even larger values ≈ 45) for sample size 10. Although the average Weibull modulus for MC data converges to the true Weibull modulus rapidly, the overestimation of Weibull modulus is larger than experimental data if 10 ceramic specimens are used for the Weibull parameter determination.

It is observed in Fig. 6(a and b) that, the probability of overestimation of Weibull modulus is higher than the underestimation of Weibull modulus for small sample sizes. For sample size 100, the overestimation and underestimation of Weibull modulus becomes almost same. When the Weibull modulus is underestimated, the failure probability of a ceramic component is overestimated. This means that the ceramic component design is in the reliable region. However, the

overestimation of Weibull modulus causes underestimation of failure probability and this may cause catastrophic failures of ceramic components. As the sample size increases, the ratio of overestimation of Weibull modulus decreases. Therefore, the increase of sample size does not only increase the prediction accuracy of the Weibull modulus but also increases the reliability of ceramic design. According to experimental data, it can be said that for sample size of 60, the overestimation is tolerable.

In Fig. 7, the change of coefficient of variation (COV) as a function of sample size for Weibull modulus is represented. The coefficient of variation is defined as the ratio of standard deviation to the mean and is a useful statistic for comparing the degree of variation from one data series to another, even if the means are drastically different from each other. The advantage of COV is that it is unitless and it calculated the variation of a parameter according to the relative mean.

As shown in Fig. 7, COV the decrease of COV as a function of sample size for experimental and MC data is almost same. The relationship between modulus COV and sample size n is

$$COV = 1.088 \, n^{-0.57} \tag{16}$$

Fig. 8(a and b) shows the effect of sample size on characteristic strength of experimental and MC data respectively. For all sample sizes, the average characteristic strength is almost equal to true characteristic strength.

The deviation of characteristic strength for sample size 30 is \pm 20 MPa. When the failure probability formulation is

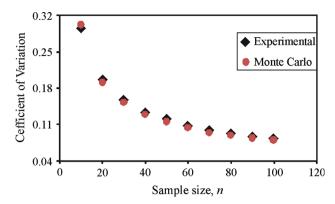
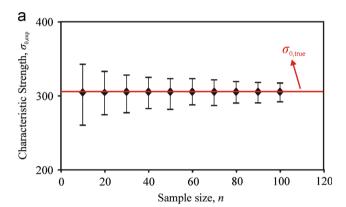


Fig. 7. Effect of sample size on Coefficient of Variation (COV) of experimental modulus $m_{\rm exp}$ and MC modulus $m_{\rm MC}$.



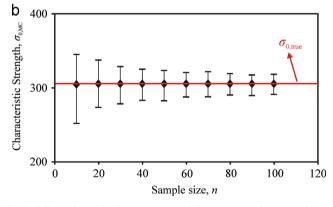


Fig. 8. Effect of sample size on characteristics strength of (a) experimental data $\sigma_{0,\rm exp}$ and (b) MC data $\sigma_{0,\rm MC}$.

considered, a deviation in Weibull modulus has a higher effect than a deviation in characteristic strength. Therefore the effect of sample size on Weibull modulus is of higher interest. In Fig. 9, the change of *COV* of characteristic strength as a function of sample size is given. As the sample size increases, the deviation of characteristic strength from the mean value strongly decreases.

The relationship between COV of characteristic strength and sample size n is

$$COV = 0.119 \, n^{-0.5} \tag{17}$$

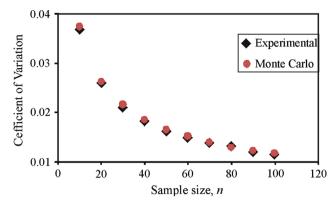


Fig. 9. Effect of sample size on Coefficient of Variation (COV) of characteristic strength.

The Weibull modulus of 10,000 simulations of experimental data with sample sizes 10, 50 and 100 were fitted with different distribution functions and it was found out that log-normal distribution shows the best fitting (see Fig. 10(a–c)).

As the sample size increases, the distribution of Weibull modulus converges to normal distribution. This verifies the behavior given in Fig. 6(a) that for sample size of 100, the overestimation and underestimation rates become almost equal to each other.

The experimental data with sample sizes 10–300 was fitted with log-normal distribution function and the mean and standard deviation of log-normal distribution as a function of sample size are given in Fig. 11(a and b). As expected, when the sample size increases, the standard deviation decreases.

In Fig. 11(c), the cumulative probability of true Weibull modulus, calculated from log-normal distribution of Weibull modulus, is given for each sample size. As the sample size increases, the cumulative probability approaches to 0.5. This means that the distribution of Weibull modulus converges to normal distribution.

4.1. Extreme value analysis of strength distribution of alumina ceramics

In this part of the article, the strength distribution of alumina ceramics is analyzed with more statistical details. The "true distribution" mentioned in the article is basically fitting the 5100 strength data with two-parameter Weibull distribution function. It is called "true distribution" because it is somehow a population for a distribution fitted by using 30 strength data as performed in practical applications. Of course when one million strength data is available, the "true distribution" calculated by using one million strength data will be different than our "true distribution". The results given above are more or less according to the practical concern. In this article, for the first time, the effect of sample size and reliable discrimination of strength distribution is given. As the distribution functions, the most widely used distribution functions were used.

Since the low probabilities (e.g. 10^{-6}) are of interest, the lower tails of the distribution functions should be analyzed. Three main functions are used in order to fit the data of

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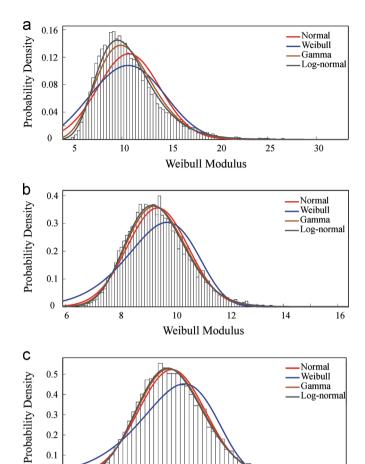


Fig. 10. Distribution of experimental Weibull modulus fitted with normal, Weibull, Gamma and Log-normal distributions for sample sizes of (a) 10, (b) 50, and (c) 100.

Weibull Modulus

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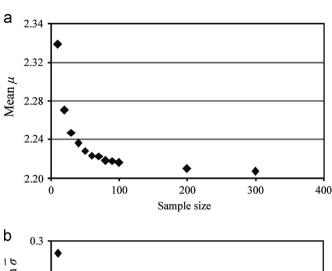
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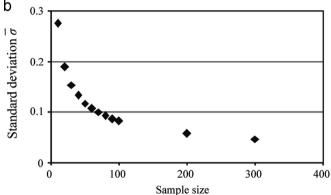
maximum or minimum vector samples. Quinn and Quinn [32] investigated the extreme value distributions of "largest flaws" in the ceramic materials since the "largest flaw" population is not expected to be a symmetric distribution, whether the parent population is Gaussian. When the strength of alumina ceramics is of interest, the extreme value analysis of minimum should be considered due to the fact that ceramic materials fail if the weakest volume element (i.e. the element with the lowest failure strength) fails. In Fig. 12, the probability density function of the strength data fitted by Gumbel, Frechet and Weibull distribution functions is shown.

In order to determine the best fitting extreme value function, as a goodness-of-fit test, A-D test was used with null hypothesis. The A-D statistical test is defined as:

 H_0 : the data follow the specified distribution, H_1 : the data do not follow the specified distribution.

The "null hypothesis" (H_0) is rejected (a candidate distribution is rejected) if the A–D statistics is greater than a threshold value that depends on the distribution itself. In this case $(H_1$ is true), the test is customarily called significant [52]. The





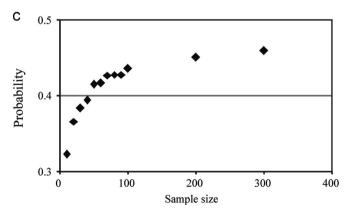


Fig. 11. (a) Influence of sample size on mean of Weibull modulus distribution, (b) influence of sample size on standard deviation of Weibull modulus distribution, and (c) cumulative probability of true Weibull modulus as a function of sample size.

"significance" corresponds to the probability (chosen by the analyst) to reject H_0 when H_0 is true. If P-value $< \alpha$, then H_0 is rejected in favor of H_1 . The choice of α depends on what hypothesis the analyst is interested in and on the criticality of the decision. A significance level of 20% (α =0.2) is conservative enough here.

In Table 2, the A–D Test results are given. As it is seen, the Weibull distribution function is the only function that can fit the strength data with α =0.2. Results given in Table 2 verify the statement given at the beginning of the article that Frechet function is not suitable for the analysis of real experimental strength values.

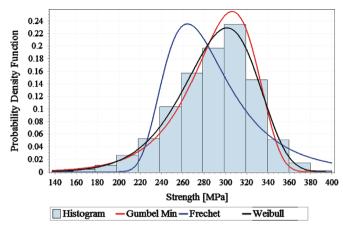


Fig. 12. Probability density function vs. experimental strength data fitted by Gumbel, Frechet and Weibull functions.

Table 2 A–D Test results of fitting strength data by Frechet, Gumbel and Weibull distributions with α =0.2.

| Distribution | Anderson-Darling | | | |
|--------------|------------------|----------------|--------|--|
| | Statistic | Critical value | Reject | |
| Frechet | 232.71 | 1.3749 | Yes | |
| Gumbel | 19.962 | 1.3749 | Yes | |
| Weibull | 1.3328 | 1.3749 | No | |

The PP (Probability-Probability) plot of the three models is given in Fig. 13. A PP-plot represents the cumulative distribution functions of the models against the empirical values. It is seen that the Weibull and Gumbel functions shows a better fit than the Frechet function.

A QQ (Quantile–Quantile) plot is another graphic method for testing whether a dataset follows a given distribution. It differs from the PP-plot in that it shows observed and expected values instead of percentages on the *x*- and *y*-axes. If all the scatter points are close to the reference line, we can say that the dataset follows the given distribution.

As shown in QQ-Plot in Fig. 14, the main difference between the Weibull and Gumbel distribution functions are at the tails. For high shape parameters (m > 6), the Weibull distribution approaches to Gumbel distribution whereas only the difference in lowest tail of the distribution becomes apparent [24]. Gumbel distribution has heavier tails and therefore is more skewed than Weibull distribution

5. Conclusion

In this article, the effect of sample size on the reliable discrimination of strength distribution of alumina ceramics was investigated by using 5100 experimental data and virtual strength data generated by MC simulations. It was found out that for a reliable discrimination, at least 150–200 strength data should be used. For lower sample sizes, the statistical fallibility increases. According to 10,000 simulations, the distribution of

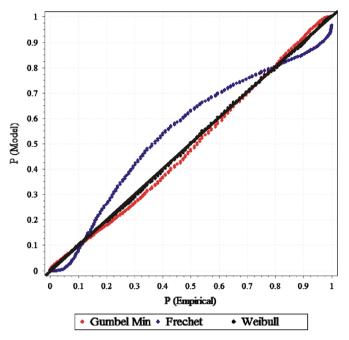


Fig. 13. PP-plot of the strength data fitted by Gumbel, Frechet and Weibull functions.

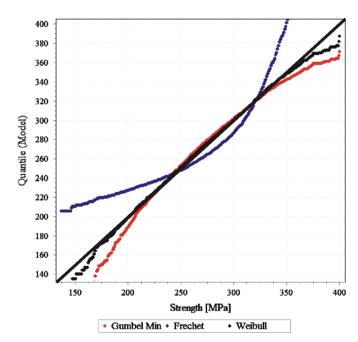


Fig. 14. QQ-plot of the strength data fitted by Gumbel, Frechet and Weibull functions.

Weibull modulus was obtained and fitted by log-normal distribution function. For small sample size, the Weibull modulus is mostly overestimated. As the sample size increases, the distribution of Weibull modulus converges to normal distribution. This means that the probability of overestimation and underestimation of failure probability becomes almost equal to each other. Extreme Value Analysis showed that Weibull is the most suitable extreme value distribution for fitting the strength data of alumina ceramics. For high Weibull

modulus values, the Weibull distribution approaches to the Gumbel distribution but the main difference occurs at the tails.

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References

- [1] A.G. Evans, Structural reliability: a processing-dependent phenomenon, Journal of the American Ceramic Society 65 (3) (1982) 127–137.
- [2] R. Danzer, T. Lube, P. Supancic, R. Damani, Fracture of ceramics, Advanced Engineering Materials 10 (4) (2008) 275–298.
- [3] B.R. Lawn, Fracture of Brittle Solids, second ed., Cambridge University Press, Cambridge, 1993.
- [4] J.H. Andreasen, Reliability-based design of ceramics, Materials & Design 15 (1) (1994) 3–13.
- [5] A.A. Griffith, The phenomena of rupture and flow in solids, Philosophical Transactions of the Royal Society of London A 221 (1921) 163–198.
- [6] D. Munz, T. Fett, Ceramics: Mechanical Properties, Failure Behavior, Materials Selection, Springer Verlag, Berlin, Heidelberg, New York, 1999.
- [7] J.E. Ritter, Predicting lifetimes of materials and material structures, Dental Material 11 (2) (1995) 142–146.
- [8] S. Nohut, G.A. Schneider, Failure probability of ceramic coil springs, Journal of the European Ceramic Society 29 (6) (2009) 1013–1019.
- [9] S. Nohut, A. Usbeck, H. Özcoban, D. Krause, G.A. Schneider, Determination of the multiaxial failure criteria for alumina ceramics under tension–torsion test, Journal of the European Ceramic Society 30 (16) (2010) 3339–3349.
- [10] R. Danzer, A general strength distribution function for brittle materials, Journal of the European Ceramic Society 10 (6) (1992) 461–472.
- [11] R. Danzer, P. Supancic, J. Pascual, T. Lube, Fracture statistics of ceramics—Weibull statistics and deviations from Weibull statistics, Engineering Fracture Mechanics 74 (18) (2007) 2919–2932.
- [12] C. Lu, R. Danzer, F.D. Fischer, Fracture statistics of brittle materials: Weibull or normal distribution, Physical Review E 65 (6) (2002) 067102.
- [13] W. Weibull, A statistical distribution function of wide applicability, Journal of Applied Mechanics 18 (2) (1951) 293–297.
- [14] Z.P Bazant, S.D. Pang, Mechanics-based statistics of failure risk of quasibrittle structures and size effect on safety factors, Proceedings of the National Academy of Sciences USA 103 (25) (2006) 9434–9439.
- [15] L. Dortmans, G. de With, Weakest-link failure predictions for ceramics IV: application of mixed-mode fracture criteria for multiaxial loading, Journal of the European Ceramic Society 10 (2) (1992) 109–114.
- [16] C. Lu, R. Danzer, F.D. Fischer, Influence of threshold stress on the estimation of the Weibull statistics, Journal of the American Ceramic Society 85 (6) (2002) 1640–1642.
- [17] R. Danzer, Some notes on the correlation between fracture and defect statistics: are Weibull statistics valid for very small specimens?, Journal of the European Ceramic Society 26 (15) (2006) 3043–3049
- [18] H. Toshihiko, F. Junpei, Simulation of strength distribution in ground ceramics by incorporating residual stress effect, Journal of Materials Engineering and Performance 17 (2008) 627–632.
- [19] B. Basu, D. Tiwari, D. Kundu, R. Prasad, Is Weibull distribution the most appropriate statistical strength distribution for brittle materials?, Ceramics International 35 (1) (2009) 237–246
- [20] S. Nohut, C. Lu, Fracture statistics of dental ceramics: discrimination of strength distributions, Ceramics International 38 (2012) 4979–4990.
- [21] ENV843-5: in Advanced Technical Ceramics, Monolithic Ceramics; Mechanical Tests at Room Temperature, Part 5-Statistical Analysis, (1997) p. 41.

- [22] R. Danzer, T. Lube, P. Supancic, Monte Carlo simulations of strength distributions of brittle materials: type of distribution, specimen and sample size, Zeitschrift für Metallkunde 92 (7) (2001) 773–783.
- [23] L. Gorjan, M. Ambrozic, Bend strength of alumina ceramics: a comparison based on very large experimental data set, Journal of the European Ceramic Society 32 (2012) 1221–1227.
- [24] E. Castillo, Extreme Value Theory in Engineering (Statistical Modeling and Decision Science), Academic Press, New York, 1988.
- [25] R.H. Doremus, Fracture statistics: a comparison of the normal, Weibull and type I extreme value distributions, Journal of Applied Physics 54 (1) (1983) 193–199.
- [26] B. Stawarczyk, M. Özcan, C.H.F. Hämmerle, M. Roos, The fracture load and failure types of veneered anterior zirconia crowns: an analysis of normal and Weibull distribution of complete and censored data, Dental Materials 28 (5) (2012) 478–487.
- [27] S. Kotz, S. Nadarajian, Extreme Value Distributions: Theory and Applications, Imperial College Press, London, 2000.
- [28] R.A. Fisher, L.H. Tippet, Limiting forms of the frequency distribution of the largest or smallest member of a sample, Proceedings of the Cambridge Philosophical Society 24 (1928) 180–190.
- [29] W. Ledermann, E. Lloyd, S. Vajda, C. Alexander, Extreme Value Theory, Handbook of Applicable Mathematics, Wiley, New York, 1980.
- [30] Z.P. Bazant, D. Novak, Stochastic models for deformation and failure of quasistatic structures: Recent advances and new directions, Computational Modeling of Concrete Structures, in: N. Bicanic, R. de Borst, H. Mang, G. Meschke, (Eds.), Proceedings of the Euro-C Conference, St. Johann im Pongau, Austria, A.A. Balkema Publ., Lisse, Netherlands, (2003) pp. 583–598.
- [31] Z.P. Bazant, D. Novak, Nonlocal model for size effect in quasibrittle failure based on extreme value statistics, Structural Safety and Reliability, Corotis et al. (Ed.), 2001.
- [32] J.B. Quinn, G.D. Quinn, Review: a practical and systematic review of Weibull statistics for reporting strengths of dental materials, Dental Materials 26 (2010) 135–147.
- [33] K.V Bury, Statistical Models in Applied Science, Wiley and Sons, New York, 1975.
- [34] K.C. Kapur, L.R. Lamperson, Reliability in Engineering Design, Wiley & Sons, New York, 1977.
- [35] P. Kittl, G. Diaz, Five deductions of Weibull's distribution function in the probabilistic strength of materials, Engineering Fracture Mechanics 36 (5) (1990) 749–762.
- [36] K. Trustrum, A.D.S. Jayatilaka, On estimating the Weibull modulus for a brittle material, Journal of Materials Science 14 (1980) 1080–1084.
- [37] S.L. Fok, B.C. Mitchell, J. Smart, B.J. Marsden, A numerical study on the application of the Weibull theory to brittle materials, Engineering Fracture Mechanics 68 (10) (2001) 1171–1179.
- [38] C. Przybilla, A. Fernández-Canteli, E. Castillo, Maximum likelihood estimation for the three-parameter Weibull cdf of strength in presence of concurrent flaw populations, Journal of the European Ceramic Society 33 (10) (2013) 1721–1727.
- [39] C. Przybilla, A. Fernández-Canteli, E. Castillo, An iterative method to obtain the specimen-independent three-parameter Weibull distribution of strength from bending tests, Procedia Engineering 10 (2011) 1414–1419.
- [40] M. Ichikawa, Stress state dependence of the shape parameter of the threeparameter Weibull distribution in relation to fracture of ceramics, Engineering Fracture Mechanics 39 (4) (1991) 751–755.
- [41] J. Smart, B.C. Mitchell, S.L. Fok, B.J. Marsden, The effect of the threshold stress on the determination of the Weibull parameters in probabilistic failure analysis, Engineering Fracture Mechanics 70 (18) (2003) 2559–2567.
- [42] R.V. Curtis, A.S. Juszczyk, Analysis of strength data using two-and three-parameter Weibull models, Journal of Materials Science 33 (1998) 1151–1157.
- [43] A.D. Papargyris, Estimator Type and Population Size for Estimating the Weibull Modulus in Ceramics, Journal of the European Ceramic Society 18 (1998) 451–455.
- [44] H. Akaike, A new look at the statistical model identification, IEEE Transactions on Automatic Control 19 (6) (1974) 716–723.

- [45] C. Lu, Y.W. Mai, Y.G. Shen, Optimum information in cracking noise, Physical Review E 72 (2) (2005) 027101.
- [46] T.W. Anderson, D.A. Darling, Asymptotic theory of certain goodness-offit criteria based on stochastic processes, Annals of Mathematical Statistics 23 (2) (1952) 193–212.
- [47] M.A. Stephens, EDF statistics for goodness of fit and some comparisons, Journal of the American Statistical Association 69 (347) (1974) 730–737.
- [48] M.A. Stephens, Asymptotic results for goodness-of-fit statistics with unknown parameters, Annals of Statistics 4 (2) (1976) 357–369.
- [49] M.A. Stephens, Goodness of fit for the extreme value distribution, Biometrika 64 (3) (1977) 583-588.
- [50] M.A. Stephens, Tests of fit for the logistic distribution based on the empirical distribution function, Biometrika 66 (3) (1979) 591–595.
- [51] L. Gorjan, A. Dakskobler, T. Kosmac, Strength evolution of injection-molded ceramic parts during wick-debinding, Journal of the American Ceramic Society 95 (1) (2012) 188–193.
- [52] A. Rinaldi, D. Krajcinovic, Statistical damage mechanics and extreme value theory, International Journal of Damage Mechanics 16 (1) (2007) 57–76.