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The mechanical strength of a ceramic porous hollow fiber

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ABSTRACT

The mechanical strength of inorganic porous hollow fibers is a critical constraint that limits their wide scale application. Various methods, including 3-point bending, 4-point bending, and diametrical compression are used for the quantification of the mechanical strength. Here, we show that these methods cannot be used in an interchangeable manner. For large sets of alumina hollow fibers, the parameters describing the cumulative probability of failure functions depend on the type of measurement, i.e., 3 or 4-point, the span size, and the measurement geometry. This implies that reporting data on mechanical properties of inorganic hollow fibers requires that extensive information about the experimental details is provided, and that a direct quantitative comparison between datasets is unjustifiable. The mechanical strength of the alumina hollow fibers tends to follow a normal distribution, or log-normal distribution, instead of the often used Weibull distribution. Monte Carlo simulations demonstrate that, especially at small sample set sizes, it is difficult to accurately determine the shape of the probability distribution. However, detailed knowledge of the type and the shape of this distribution function is essential when mechanical strength values are to be used in further design.

1. Introduction

Thin inorganic hollow fibers have large potential to be used as, for example, microfiltration membranes, catalyst supports, and (membrane) microreactors [1]. However, the widespread and large scale application of inorganic fibers is hampered by, in particular, their mechanical properties. Detailed knowledge of these properties is required for an acceptable comparison between different fibers, and quantification of the failure behavior of fibers is of key importance for the design and construction of large area multi-fiber systems.

In ceramic reliability engineering, one often assumes a specific probability distribution. Based on the microstructure of a ceramic — being porous or non-porous— different probability distributions are assumed. In traditional non-porous ceramics the Weibull distribution is mostly used, whereas recently the use of the log-normal or normal distributions are proposed for porous ceramics [2].

In addition to this, many methods are used to assess the mechanical robustness of inorganic fibers; most commonly their flexural or bending strength is determined via a 3-point [3–7] or 4-point bending test [8–11]. Alternatives include burst pressure measurements [12,13] or diametrical compression tests [14,15].

The reported mechanical strength is a direct result of the measurement method and the conditions used. As a result, comparison of strength data presented in literature can be deceptive. In addition to the measurement method, the amount of samples measured and the subsequent statistical analysis are of great importance.

The comparison of a 3-point versus a 4-point bending test is described in literature for various applications, like advanced dense ceramics [16–19] and polymers [20], but not for porous inorganic hollow fibers. In this paper, we demonstrate the pronounced influence of the measurement method on the reported strength value, and why it is crucial to not only report the measurement method, but also sample geometry and sample set size to allow comparison of reported strength values.

2. Theoretical background

2.1. Strength distributions

The result of fracture testing is usually reported as an average strength or mean strength of the measured stress at failure, of a set of N samples.

$$\sigma_i = \frac{\sum i = 1N\sigma_i}{N} \tag{1}$$

A drawback of the average strength is that it contains no information about the spread of the strength values measured. In ceramics, defects are randomly distributed over the sample and they will vary in position,

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size, and severity. As a consequence, the strength of a fiber will vary from fiber to fiber, even for apparent identical fibers. As a measure for this spread often the standard deviation of the mechanical strength data is reported. In addition, if a small amount of samples is measured, the average strength might not be a good representation of the real strength number (this effect becomes more severe at lower sample sizes, e.g. $N < 10$ [21]). Therefore, the common interpretation of fracture tests is based on a statistical models that predict a probability of failure. It is often stated the one is required to measure at least 10–30 samples if the statistical model is known [19,22], others propose that a minimum of 150–200 samples is required for an unknown model [21,23].

The distribution parameters are of utmost importance in the design of components that consist of ceramics. In design, an acceptable probability of failure is selected, and the associated design stress is calculated using the statistical distribution function. Especially the lower tail of the probability distribution strongly affects the design stress [24]. Without appropriate characterization and the use of the correct statistical distribution, the measured strengths cannot be applied in design and might lead to erroneous conclusions. [2,25–27].

2.1.1. Weibull distribution

The Weibull distribution [28] is the generally applied distribution for strength characterization of brittle ceramics with little defects. It is based on the so-called weakest link principle. In the majority of dense ceramics, only few defects are present. If the ceramic fails, it is assumed to fail at its weakest defect. The regular formulation of the Weibull distribution, used in measuring the strength of ceramics, is written as:

$$P(\sigma) = 1 - \exp\left[-\left(\frac{\sigma}{\sigma_\theta}\right)^m\right] \tag{2}$$

where $P(\sigma)$ is the cumulative probability of failure, σ the applied stress, m the Weibull modulus - a measure for the spread of strength data - and σ_θ is the characteristic strength. The characteristic strength and estimate of the Weibull modulus are often obtained via maximum likelihood fitting of the measured strength data. The Weibull characteristic strength depends on the test geometry, such type and size; and it is a value specific to a certain test.

An alternative representation of the probability of failure is the more general equation,

$$P(\sigma, V) = 1 - \exp\left[-\int_V \left(\frac{\sigma}{\sigma_0}\right)^m dV\right] \tag{3}$$

where $P(\sigma, V)$ is the cumulative probability of failure, V is the volume of the component, σ is the applied stress, m is the Weibull modulus and σ_0 is the Weibull material scale parameter. If the integration in the above-mentioned equation is carried out the equation reduces to:

$$P(\sigma) = 1 - \exp\left[-kV\left(\frac{\sigma}{\sigma_0}\right)^m\right] \tag{4}$$

where k is a dimensionless constant that accounts for test specimen geometry and stress gradients. In general, k is also a function of the estimated Weibull modulus. The product kV is often referred to as the effective volume. Using this effective volume, the characteristic Weibull strength σ_θ can be converted into the Weibull material scale parameter using a relation such as

$$(\sigma_0)_V = (kV)^{1/(m)_V}(\sigma_\theta)_V \tag{5}$$

This approach is discussed in detail in various references [22,29,30]. Calculation of the effective volume of a porous ceramic hollow fiber with its defects, pores, and macrovoids- is problematic as the volume under stress is nearly impossible to estimate.

Mechanical strength testing combined with Weibull analysis are broadly standardized for dense ceramics, for example in ASTM: C1161 [22] or DIN 843-5 [31]. Most methods recommend to measure at least 30 samples in order to accurately estimate the Weibull modulus and characteristic strength. The Weibull distribution, depending on the weakest link theory with non-interacting defects, is questioned to be suitable for certain ceramic strength data [26,32–35]. For example Danzer et al. [26] demonstrate that in certain situations a deviation of the Weibull distribution is expected; when the material exhibits a multi-model flaw size distribution (e.g., porosity), when defects interact, when R-curve behavior is observed or when subcritical crack growth is likely [36]. Inorganic porous hollow fibers prepared by non-solvent induced phase inversion (NIPS) have a high defect density with a large range of defects such as pores, large finger like voids and agglomerates. This results in a questionable applicability of the Weibull model and its underlying assumption. Therefore, other models are also evaluated.

2.1.2. Normal distribution

The normal distribution is one of the mostly used distributions in sciences for real-valued random variables whose distributions are not known. Therefore, a normal distribution is used to describe the strength of brittle ceramics, in particular when these materials show a roughly symmetrical distribution and when the amount of defects is large [33,37,38]. Its parameters $\bar{\sigma}$ and α represent the mean and standard deviation of the distribution. The cumulative probability is given by:

$$P(\sigma) = \frac{1}{2}\left[1 + \mathrm{erf}\left(\frac{\sigma - \bar{\sigma}}{\alpha\sqrt{2}}\right)\right] \tag{6}$$

2.1.3. Log-normal distribution

Lu et al. proposed the log-normal distribution for porous ceramics with high porosity. This assumes that the probability of a flaw being critical depends on a lot of factors such as size, shape and pore-grain interaction [2,21]. The failure probability can be estimated by $p = \prod p_i$, where p_i is the failure probability of the i-th factor of influence. Via $\ln p = \sum \ln p_i$ this results in an overall probability that follows the log-normal distribution [39]. The cumulative probability of a log-normal distribution is:

$$P(\sigma) = \frac{1}{2}\left[1 + \mathrm{erf}\left(\frac{\sigma - \bar{\sigma}}{\alpha\sqrt{2}}\right)\right] \tag{7}$$

If data is log-normally distributed with parameters $\bar{\sigma}$ and α, the logarithm of the data is distributed with the mean $\bar{\sigma}^* = \exp(\bar{\sigma} + \alpha^2/2)$ and multiplicative standard deviation $s^* = \exp(2\bar{\sigma} + \alpha^2)[\exp(\alpha^2) - 1]$. Analog to the additive transformation for the normal distribution $\bar{\sigma} \pm \alpha$, the multiplicative transformation of the lognormal distribution can be expressed as $\bar{\sigma}^* \cdot / s^*$, where ”·/” indicates ”times or divided” by [39].

2.2. Minimum information criterion

The unknown parameters of the proposed distribution functions are obtained by maximum likelihood method. Eq. (8) shows the likelihood of a probability density function, where σ_i is the strength of the i-th sample, N is the total number of samples, $\mathcal{L}$ is the likelihood, and $f(\sigma_i)$ is the probability density function (pdf) of the proposed distribution [33].

$$\ln(\mathcal{L}) = \sum_{i=1}^{N} \ln f(\sigma_i) \tag{8}$$

To compare the proposed models, the Akaike information criterion (AIC) [40] is used; which is an estimate for the distance between the true and the estimated distribution, and is defined as: