JOURNAL OF MATERIALS SCIENCE 32 (1997) 1903–1907

The validity of Weibull estimators – experimental verification

N. ORLOVSKAJA
Institute of Problems of Materials Science, 3 Krzizanovskogo, 252180 Kiev – 180, Ukraine
H. PETERLIK*, M. MARCZEWSKI, K. KROMP
Institute of Materials Physics, University of Vienna, Boltzmanngasse 5, A-1090 Vienna, Austria

A sufficiently large number of bending tests of a recrystallized silicon carbide ceramic was performed, which gave a fundamental set of strength values. From this fundamental set, arbitrary subsets of size *M* were chosen by a Monte-Carlo procedure and the parameters of the two-parametric Weibull distribution were calculated by the maximum likelihood method. The dependence of the statistical distribution of the two parameters, obtained by this procedure, on the size of the subset *M* was investigated. It was found that the variation coefficient of the scale parameter could be well described by the equations given in the literature, whereas the variation coefficient of the Weibull modulus was much higher. It has been shown that the reason for this behaviour is that the distribution of flaws and therefore the strength of the material does not perfectly obey the Weibull statistics, for which the theoretical equations were derived. Thus, in real ceramics the numerical value for the Weibull modulus obtained from a certain number of experiments is even more indeterminate than described by the theoretical solution.

1. Introduction

The Weibull distribution has been widely used to describe the statistical behaviour of the fracture of ceramics [1]. It is based on the "weakest-link hypothesis", which means that the most serious flaw controls the strength. If the flaw sizes of the large pores, i.e. those which are responsible for failure, are distributed according to a power law, the strength values are distributed according to the Weibull distribution [2]. Predominantly, the two-parameter Weibull distribution is used, with the scale parameter, σ_0, describing the strength, and the Weibull modulus, *m*, which characterizes the width of the strength distribution. These parameters have to be determined by a limited number of experimental tests, depending on the money and the time of the producer and the the testing institution. As the exact values can only be determined by an infinite number of tests, there had been considerable interest in the precision of the determination of the parameters by a certain, limited number of experiments [3–9]. Another point of interest was to investigate the dependence on different evaluation procedures, e.g. linear regression, moments method or maximum likelihood [3–9].

Recently, for a known Weibull modulus, *m*, an exact solution was found for the dependence of standard deviation on the number of tests, if evaluated by the maximum likelihood procedure [10]. The general solution for unknown *m* was approximated by a computer simulation and can be well described by multiplying the special solution with a constant factor close to 1 [10]. Furthermore, it was shown that the biasing results only from the way of adding different values from an asymmetric distribution, hence each single measurement is statistically correct and unbiased. In this context, biasing shows only the degree of the asymmetry of the distribution.

In this work, the idea of testing the statistical behaviour by investigating subsets of a set of numerous bending tests of a real ceramic material was adopted [11]. Furthermore, we calculated the statistical behaviour, i.e. the standard deviation and the biasing, by the complete distribution of arbitrary subsets of dimension *M* of a sufficiently large fundamental set of bending tests.

2. Experimental procedure

The material investigated was a recrystallized silicon carbide (RSiC). This material has a high strength in relation to its density and is nearly free of second phases (sintering aids) except a rest of free silicon (< 2 wt %). Thus the material is high creep resistant. A high thermal conductivity and a low thermal coefficient of expansion allow this material to be applied for kiln furniture in the ceramic industry. The name

* Author to whom all correspondence should be addressed.

0022–2461 © 1997 Chapman & Hall

"recrystallized silicon carbide" is misleading, because the material is produced by an evaporation–condensation process. Small SiC grains ($< 5\ \mu m$), silicon powder and coarse SiC grains ($> 50\ \mu m$) are mixed with an organic binder, so that slip casting can be applied. During sintering of the green bodies at 2200 °C small particles partly evaporate and condense, filling gaps and building up necks between the large particles. The process has the advantage that almost no shrinkage takes place, but it results in a (partly open) porosity of up to 20%. Two plates of the same material but of different charges were investigated, one for the Weibull distribution tests with a mean density of $2.74\ g\,cm^{-3}$ and the other for the determination of the subcritical crack growth parameters with a mean density of $2.81\ g\,cm^{-3}$. The distribution of pores is usually described by a two-parameter power law, from which the two parameters of the Weibull-distributed strength may be derived [12].

The specimens were machined to final dimensions of $4 \times 5 \times 45\ mm^3$ and loaded in four-point-bending in a hydraulic testing machine at a stressing rate of about $20\ MPa\,s^{-1}$. The tensile surface was ground and the edges were chamferred according to the European standard EN 843-1 [13].

3. Results and discussion

To obtain the fundamental set of strength values, 137 bending tests were performed. From this fundamental set, 10^5 arbitrary subsets, consisting of M single bending strength values, were chosen by a random procedure and the parameters of the two-parameter Weibull distribution, the scale parameter, σ_0, and the Weibull modulus, m, were calculated by the maximum likelihood method. Then, the dependence of the statistical behaviour, the variation coefficient and the biasing, if the values are arithmetically added, on the subset size, M was investigated.The Weibull parameters obtained by a whole set of 137 values are henceforth called the true values, the scale parameter being $\sigma_0 = 90.73$ MPa and the Weibull modulus $m = 9.36$. Of course, the true values for the parameters can only be obtained in the limit $m \to \infty$ or $M \to \infty$, i.e. an infinite Weibull modulus or an infinite number of experiments. However, the high number of tests will give a quite good approximation and the effects on the variation coefficient and the biasing by this approximation will be small. The theoretical variation coefficient of the scale parameter for this case is below 1%, that of the Weibull modulus being about 7%. Thus, the precision of the numerical results described below may be estimated to be in the range of these deviations.

Now, from the fundamental set, 10^5 arbitrary sets consisting of a certain number, M, of single bending strength values, were obtained. As the number of possible combinations for a certain number $M \leqslant 90$ from the fundamental set far exceeds 10^5, this can be taken as 10^5 independent experiments, consisting of M single bending tests, to determine the Weibull parameters. Hence, in the following, the results from this procedure will be called experimental results. The standard deviation, Δx, of a certain variation x and a number of $N = 10^5$ experiments is given in general by

$$(\Delta x)^2 = \langle (x - \bar{x})^2 \rangle = \frac{1}{N} \sum_{i=1}^{N} (x - \bar{x})^2 \qquad (1a)$$

which is, in the limit $N \to \infty$

$$(\Delta x)^2 = \langle x^2 \rangle - \langle x \rangle^2 \qquad (1b)$$

with

$$\langle x \rangle = \bar{x} = \frac{1}{N} \sum_{i=1}^{N} x_i \qquad (2)$$

and the variation coefficient is obtained by normalizing the standard deviation, $\Delta x / x$. Of course, this definition of the standard deviation and the variation coefficient is symmetric. As the distribution is asymmetric, a description by an asymmetric deviation would be more appropriate, but in this work the symmetric one is chosen for simplicity and for easy comparison with the theoretical results [10]. Owing to this theoretical work [10], the variation coefficient, $\Delta\sigma_0/\sigma_0$, of the scale parameter is given by

$$\frac{\Delta\sigma_0}{\sigma_0} = \alpha^{1/2} M^{-1/m} \times \left\{ \frac{\Gamma(M + 2/m)}{\Gamma(M)} - \left[\frac{\Gamma(M + 1/m)}{\Gamma(M)} \right]^2 \right\}^{1/2} \qquad (3a)$$

with $\alpha = 1.05$ and Γ being the Gamma function. The one of the Weibull moduli is approximated by a power law function,

$$\frac{\Delta m}{m} = 0.04222 + 2.3375\, M^{-0.8836} \qquad (3b)$$

The respective formulas for the biasing, i.e. the deviation, if the single values are arithmetically added, are

$$\frac{\langle \sigma_0 \rangle}{\sigma_0} = M^{-1/m} \frac{\Gamma(M + 1/m)}{\Gamma(M)} \qquad (4a)$$

$$\frac{\langle m \rangle}{m} = 1 + 2.1049\, M^{-1.1} \qquad (4b)$$

Fig. 1 shows the variation coefficient of the scale parameter, i.e. the standard deviation normalized to the true value, $\Delta\sigma_0/\sigma_0$. The circles are the variation coefficients from the distribution of 10^5 experiments described by Equations 1 and 2, the line is the theoretical variation coefficient from analytical results and computer simulations, see Equations 3 and 4. It can be seen that the experimental variation coefficient is in perfect agreement with the theoretical one, which is valid for a material that would exactly obey the Weibull theory. In Fig. 2, the biasing, i.e. the degree of the asymmetry of the distribution, can be seen. The circles are obtained by the arithmetic mean of 10^5 experimental values, the boxes by a Weibull-motivated mean [10, 14]

$$\bar{\sigma}_0 = \left(\frac{1}{N} \sum_{i}^{N} \sigma_0^m \right)^{1/m} \qquad (5)$$

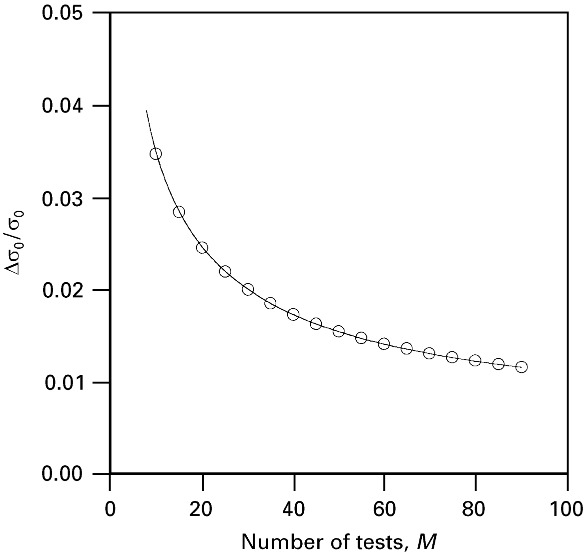

Figure 1 The variation coefficient of the scale parameter, $\Delta\sigma_0/\sigma_0$, in dependence on the number of tests M. ○: Obtained by the distribution of the experimental values, line: theoretical solution for materials perfectly obeying the Weibull statistics [10].

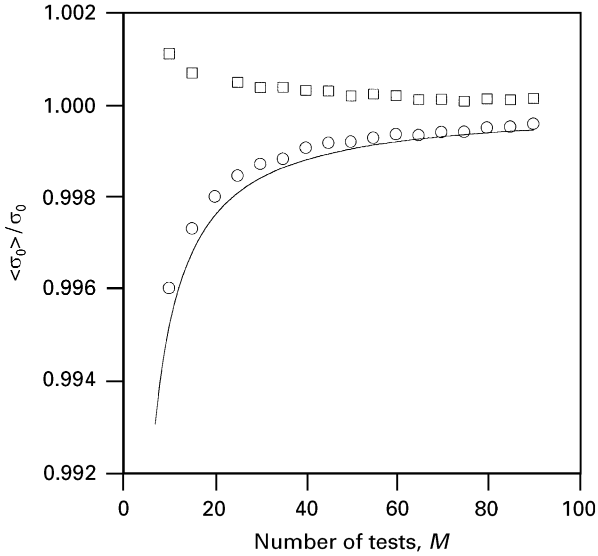

Figure 2 The biasing of the scale parameter. ○: Obtained by arithmetically adding the single measured values (conventional mean value), □: obtained by a Weibull motivated mean value (Equation 5), —: theoretical solution for materials perfectly obeying the Weibull statistics [10].

and the line is the theoretical curve for the results, which should be obtained by the arithmetic mean. It can be seen that the addition according to the Weibull mean leads to an overestimation, whereas the arithmetic mean underestimates the true value. However, for the 30 values recommended by the European Committee for Normalization for the determination of the Weibull parameters, the variation coefficient of the scale parameter calculated by the Weibull-motivated mean is 0.04%, whereas in comparison the arithmetic mean has a variation coefficient of 0.13% for $M = 30$. Thus, if more than one working group has tested a material and only the Weibull parameters (and not the single strength values) are known, an evaluation to obtain a more precise value should be calculated according to Equation 5, but the

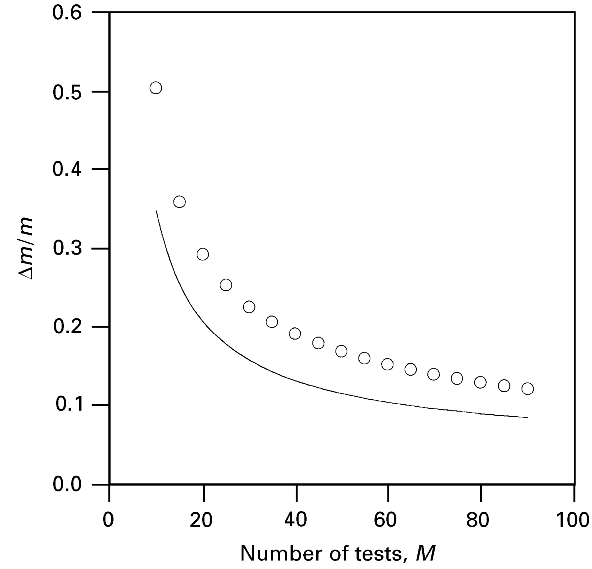

Figure 3 The vatiation coefficient of the Weibull modulus, $\Delta m/m$, in dependence on the number of tests M. ○: Obtained by the distribution of the experimental values, —: theoretical solution for materials perfectly obeying the Weibull statistics [10].

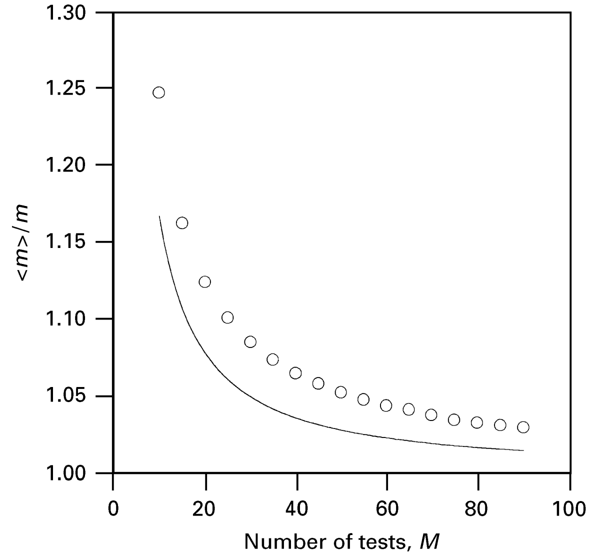

Figure 4 The biasing of the Weibull modulus. ○: Obtained by arithmetically adding the single measured values (conventional mean value), —: theoretical solution for materials perfectly obeying the Weibull statistics [10].

differences between both calculations are usually small.

A completely different behaviour can be observed for the Weibull modulus, m. Fig. 3 shows the variation coefficient, where the results from the experiments (represented by the circles) show significantly higher values than that obtained by the theoretical curve. The same behaviour can be seen for the biasing, i.e. the experimental values are much higher than the theoretical ones. The reason being that the theoretical equations were obtained from a material which perfectly obeys the Weibull theory and thus represent a lower limit for the determination of the Weibull parameters. To prove this assumption, a set of 137 random strength values lying exactly on the Weibull fit were randomly chosen and the procedure as mentioned above was applied, i.e. subsets of a certain size, M, were arbitrarily chosen and their variation coefficient and the biasing were calculated. Then the numerical values exactly follow the theoretical curves, which confirms the assumption that the higher variation coefficient originates from the deviations from the Weibull fit.

However, real materials may not perfectly obey the Weibull distribution. In Fig. 5 the Weibull diagram is depicted, where it can be seen that deviations from the straight line exist, which is the fit from the maximum likelihood function. The circle are (x, y)-pairs with $x = \ln \sigma_b$ being the logarithm of the bending strength and $y = \ln \ln 1/(1 - P_f)$

$$P_f = \frac{n - 0.5}{M} \qquad n = 1 \ldots M \tag{6}$$

which is a frequently used expression for the fracture probability, P_f. The fracture probabilities have to be chosen for a graphical representation or an evaluation by linear regression. They are not required for an evaluation by maximum likelihood method, because they are not arbitrary in this case. From Fig. 5 it is clearly visible that for real ceramics, deviations may exist from the straight line, the Weibull fit. These deviations result in a higher variation coefficient of the Weibull modulus in comparison with the theoretical solutions, whereas the scale parameter is much less affected. This may contribute to the fact that on the one hand the scale parameter has a standard deviation much less than the Weibull modulus, and on the other hand it characterizes a "mean" strength, which is less sensitive than the modulus, which characterizes the uniformity of the flaw distribution and thus the distribution of strength values.

There are numerous possibilities for these deviations, for example, intrinsic differences from different distributions of the defects depending on their size, edge effects or transition from volume to surface flaws. In a recent work [15], deviations were attributed to the R-curve behaviour and the extension of a crack. For our material, RSiC, this can be excluded: in Fig. 6 tests at different stressing rates are shown, from which the subcritical crack extension can be calculated [16]. The circles are the mean of ten bending tests at each loading rate and are nearly at the same level, showing that there exists no considerable subcritical crack extension for this material. The numerical values of the bending strength tests for the subcritical crack extension are higher than that for the Weibull distribution, because these specimens had a higher mean density. But as all strength values at different loading rates are at the same level, R-curve behaviour may be excluded. We doubt, furthermore, the explanation by R-curves for the deviations for two reasons: firstly, the experiments reported by Duan *et al.* [15] lasted only about 0.1 s, and due to this, very high loading rate crack extension should be rather improbable. Secondly, the R-curve was obtained from the crack extension of compact tension (CT) specimens in a range up to 6 mm. As R-curve behaviour characterizes the fracture behaviour of long cracks [17] and the crack extension in bending tests leading to fracture is estimated to be

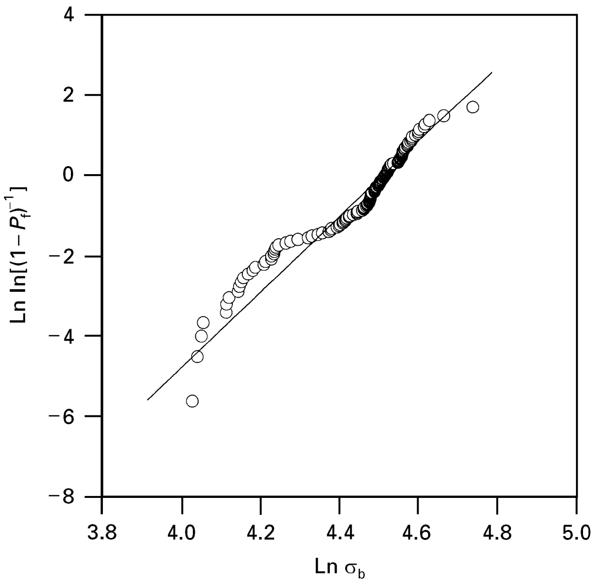

Figure 5 Weibull diagram for the whole sample of 137 measured values (○), showing that the material not perfectly obeys Weibull's law (—).

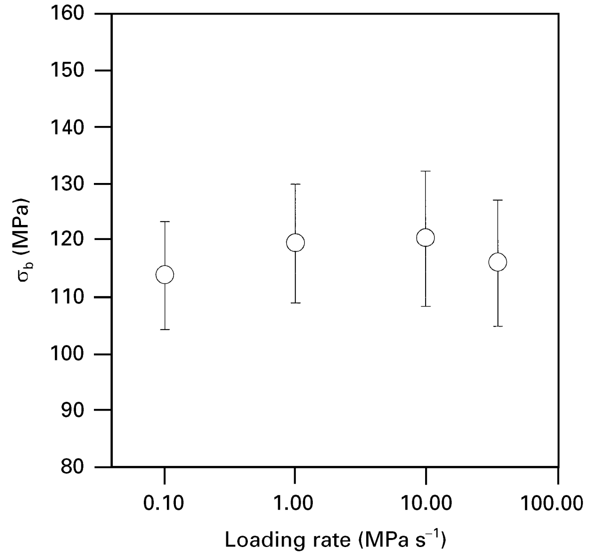

Figure 6 The tests at different loading rates show that there exists no subcritical crack extension for this material, as all values are at the same level.

two orders of magnitude less than in the CT tests (taking the given K_{Ic}-value into account), we assume that the R-curve behaviour is much less pronounced. For bending tests, all fractures occur just at the very beginning of the R-curve.

Therefore, we suggest that different intrinsic distributions of the pores are responsible for the deviation. This is, furthermore, confirmed by pore-size measurements for specimens with different strengths. Fig. 7 shows, as an example, the cumulative probability of the size of 1500 pores, measured by optical microscopy, by using the following rank statistics [18]

$$F(a_i) = \frac{\sum_{j=0}^{i} N_j - 0.5}{\sum_{n=0}^{k} N_n} \tag{7}$$

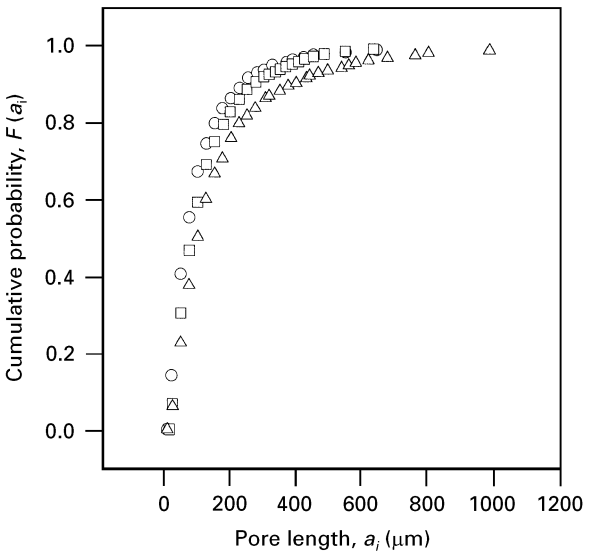

Figure 7 Cumulative probability of the size of 1500 pores for specimens with different porosities, showing that there are intrinsic differences in the pore size distribution. △: porosity 20.1%, □: 17.3%, ○: 16.7%.

with a_i the maximum pore length in the range i and N the number of pores in a particular size group. Results are shown from a plane section measurement of specimens with a porosity of 20.1%, (triangles), of 17.3% (boxes) and of 16.7% (circles). Thus the distribution of pores may depend on the actual porosity and may vary from specimen to specimen. As this has effects on the distribution of the largest pores, i.e. those responsible for fracture, these differences may also be responsible for the deviations from the Weibull statistics. To cope with this, Chao and Shetty [18] used a four-parameter fit for the cumulative distribution function and obtained a better prediction of the measured fracture strength distribution for silicon nitride.

Hence it may be concluded that the pore-size distribution does not follow a power law with two constant parameters. The differences may, of course, be very pronounced for the material RSiC because of its high porosity. We assume that for materials with very small defects in comparison to the specimen's dimensions the deviations from the Weibull fit might be considerably smaller. Henceforth, the influence of different pore-size distributions should not be neglected, but considered as a possible additional uncertainty in determining the Weibull modulus.

4. Conclusions

10^5 subsets of a certain size $10 \leqslant M \leqslant 90$ were chosen from a fundamental set of 137 bending strength values and the Weibull parameters were evaluated according to the maximum likelihood method. This corresponds to 10^5 independent experimental test sets, from which the statistical behaviour of the variation coefficient and the biasing (deviation from the true value, if arithmetically added) could be investigated. The following conclusions may be drawn.

1. The variation coefficient and the biasing of the scale parameter may be described by the theoretical solution, which are the lower limits and valid for a material perfectly obeying the Weibull statistics, i.e. pore-size distribution according to a power law and thus strength distribution according to the Weibull statistics.

2. The variation coefficient of the Weibull modulus may be significantly higher than the lower limit described by the theoretical solution, depending on the degree to which the strength distribution of the material deviates from the statistics described by the Weibull theory.

3. The deviations from the Weibull theory are responsible for the difference from the theoretical solutions, as the same procedure as mentioned above, performed with 137 values lying perfectly on the Weibull fit, follows exactly the numerical values of the theoretical solutions.

4. These deviations from the Weibull theory are attributed to intrinsic properties such as different pore distributions, whereas R-curve behaviour cannot play any role, because the material tested is resistant to subcritical crack extension.

Acknowledgements

The financial support by the Austrian research fund (FWF) under contract P8990-TEC, by the Austrian exchange program for middle- and eastern Europe and by the University of Vienna, is greatly appreciated. We thank Mr Heider, Cesiwid, Erlangen, for supplying the material.

References

1. W. WEIBULL, *J. Appl. Mech.* **18** (1951) 293.
2. R. DANZER, *J. Eur. Ceram. Soc.* **10** (1992) 461.
3. J. RITTER, N. BANDYOPADHYAY and K. JAKUS, *Am. Ceram. Soc. Bull.* **60** (1981) 798.
4. L. J. BAIN and C. E. ANTLE, *Technometrics* **9** (1967) 621.
5. D. R. THOMAN, L. J. BAIN and C. E. ANTLE, *ibid.* **11** (1969) 445.
6. K. TRUSTRUM and A. DE S. JAYATILAKA, *J. Mater. Sci.* **14** (1979) 1080.
7. B. BERGMAN, *J. Mater. Sci. Lett.* **3** (1984) 689.
8. B. FAUCHER and W. R. TYSON, *ibid.* **7** (1988) 199.
9. A. KHALILI and K. KROMP, *J. Mater. Sci.* **26** (1991) 6741.
10. H. PETERLIK, *ibid.* **30** (1995) 1972.
11. J. D. SULLIVAN and P. H. LAUZON, *J. Mater. Sci. Lett.* **5** (1986) 1245.
12. A. KHALILI, H. PETERLIK, J. DUSZA and K. KROMP, in "Proceedings of the International Fractography Conference (October 1994), Stará Lesná, edited by L. Parilak *et al.*, Slovakia, Polygrafia SAV Bratislava (1994) p. 72.
13. European standard EN 843-1: "Determination of flexural strength", European Committee for Standardization, rue de Stassart 36, Brussels.
14. H. PETERLIK, *J. Euro. Ceram. Soc.* **13** (1994) 509.
15. K. DUAN, Y. W. MAI and B. COTTERELL, *J. Mater. Sci.* **30** (1995) 1405.
16. European prestandard ENV 843-3: "Determination of subcritical crack growth parameters from constant stressing rate flexural strength tests", European Committee for Standardization, rue de Stassart 36, Brussels.
17. R. W. STEINBRECH, A. REICHL and W. SCHAARWÄCHTER, *J. Am. Ceram. Soc.* **73** (1990) 2009.
18. L. Y. CHAO and D. K. SHETTY, *ibid.* **75** (1992) 2116.

Received 10 August 1995
and accepted 2 July 1996