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Prediction of strength of recrystallized siliconcarbide from pore size measurement

Part II *The reliability of the prediction*

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The distribution of the pore sizes was measured for more than hundred specimens with an total area of 16000 square millimeters of recrystallized siliconcarbide specimens, from which the bending strength values were known. The parameters of the Weibull distribution of the strength were predicted from the distribution of the pore sizes. The reliability of the prediction was calculated by taking out arbitrary subsets of these specimens, corresponding to sub-areas of the total area, and investigating the statistical distribution in dependence on the size of the pores in these subsets. The so obtained mean and the variation coefficient were compared to the results from the mechanical bending tests.

1. Introduction

To describe the fracture behaviour of brittle materials, the Weibull distribution has been widely used [1–3]. The strength values obey a Weibull distribution, if the number of the pores decreases with increasing pore length according to a power law [2, 3]. Thus, the statistical distribution of the flaw dimensions is closely connected to the fracture stresses obtained by mechanical tests. This relationship has been used to predict either the distribution of flaw sizes from fracture experiments [4–8] or to predict fracture stresses from pore size distributions [9–15]. For practical use, the reliability of the measurements is of great interest. For mechanical tests, this has been investigated in the past years. Most works dealt with the precision and the biasing of the measurement of the scale parameter and the Weibull modulus, most of them using computer simulation of experiments [16–22]. If a material perfectly obeys the two-parametric Weibull distribution and its Weibull parameters are known, the reliability of the measurement can be analytically calculated in dependence on the number of experiments performed [23]. For an unknown Weibull parameter, the functional behavior is similar and the variation coefficients of the scale parameter and the modulus were given by simple approximative equations [23] obtained from computer simulations. If a material does not show an unimodal but a bimodal distribution, which is often the case in practice, this considerably increases the variation coefficient of the modulus, but only sligthly affects the scale parameter, as had been verified for recrystallized siliconcarbide (RSiC) [24].

The method proposed in [24] based on a large number of mechanical bending tests considered as the fundamental set, from which arbitrary subsets were taken out. The statistical distribution of these subsets, i.e. the mean and the variation coefficient, was investigated in dependence on the size of the subsets, i.e. the number of experiments performed [23, 24].

Now this method is applied to calculate the reliability of the prediction of the Weibull parameters of the bending strength by a non-destructive measurement of the pore size distribution. The pore size distribution determined from the area of all specimens is considered as the fundamental set. From this fundamental set arbitrary subsets are taken out (representing smaller amounts of area measured) and the Weibull parameters predicted from these areas are calculated. The statistical distribution of these predicted Weibull parameters is then compared to the results obtained from the mechanical bending tests. The knowledge of the application and the limits of this non-destructive method could be important for the development of automatic quality control during industrial processing.

2. Materials and methods

Recrystallized siliconcarbide (RSiC) is a material consisting of a nearly perfect stochiometrical ratio of silicone and carbide and is therefore nearly free of second phases, which are usually required as a sintering aid. This results in a high creep resistance. Together with the high thermal conductivity and the low coefficient of expansion this material is especially designed for the use as kiln furniture. A short description of the material may be found in [15], a precise description of processing and texture in [25].

To determine the pore size distribution, 123 specimens were polished and data about the shape and the size of the pores were collected by digital image processing in the optical microscope. Thus, a total area of more than 16000 square millimeters was measured.

The validity of linear elastic fracture mechanics (LEFM) was assumed and the frequency distribution of pore size $g(a)$ should decrease with an inverse power law with an exponent r and a scaling pore size a_{sc}. Then the fracture probabilities P_f are Weibull distributed with an exponent m and a scaling parameter σ_0 [1, 2]:

$$g(a) = g(a_{sc})\left(\frac{a}{a_{sc}}\right)^{-r} \rightarrow$$
$$P_f = 1 - \exp\left(-\left(\frac{\sigma}{\sigma_0}\right)^{m}\right) \qquad (1)$$

By this, the parameters of the pores $(g(a_0), r)$ and the ones of the strength (σ_0, m) are related by [2, 3]

$$m = 2\ (r-1) \quad \text{and} \quad \sigma_0 = (m/(2a_{sc}g(a_{sc})V_0))^{1/m} \times K_{lc}\big/\big(Y(\pi a_{ac})^{1/2}\big), \qquad (2)$$

where V_0 is the effective volume, K_{lc} the fracture toughness and Y the shape factor. There are three problems, which have to be solved:

- Firstly, the determination of the fracture toughness K_{lc}. In this work it was measured by an independent procedure (single edge notched beam, notched by a diamond blade with a thickness of 50 microns, resulting in a notch radius of about 30 microns). Five tests were performed according to the German prestandard DIN 51109. The fracture toughness turned out to be 2.05 ± 0.1 MPa $\sqrt{\text{m}}$. A comparison of this test method to others could be found in literature with data obtained by a round robin test for five different brittle materials [26].
- Secondly, the shape factor has to be determined. This problem was treated in [15].
- Thirdly, the frequency distribution has to be known. In [15] the way to calculate the volume distribution from the measured surface distribution was discussed. In this work, the influence of a measurement of different amount of areas and the influence of a pore size distribution, which does not perfectly obey the power law (Equation 1) are investigated.

To calculate the statistical behaviour, histograms were built: The number of pores was collected in bins with an interval length of 50 microns. The number of pores of all specimens was seen as a fundamental set, from which arbitrary subsets (consisting of 5, 10, 20, . . . , up to 100 specimens) were chosen by a random procedure. They represent measurements of the number of pores of smaller areas. By dividing this number through the respective measured area, the corresponding frequency distribution is obtained, from which the Weibull parameters can be calculated according to Equation 1. This procedure is repeated some thousand times and the statistical behaviour of the so obtained Weibull parameters is investigated. Despite the biasing of the Weibull distribution, a symmetric description with mean and variation coefficient is chosen for clarity. The dependence of the predicted mechanical parameters on the size of the subsets, i.e. the respective measured area, has the following importance: The knowledge of the reliability of the method, i.e. the amount of area required to obtain a certain accuracy of the predicted mechanical strength, is important in practice for a use of this method for automatic quality control in industrial processing.

The mechanical parameters predicted from the pore size measurements were calculated by taking into account the different shape factors from the surface and the volume pores [15], which result in a bimodal distribution of the mechanical strength values. This bimodality has been mainly investigated for ceramic fibres [27–31] and not for ceramics. The reason is probably that due to the high scatter of ceramic materials a bimodality could only be seen as statistically significant, if a very large number of mechanical data would be available. This, however, needs a lot of effort and costs.

The predictions by the pore size measurements were compared to results obtained from a bimodal Weibull fit of the mechanical tests using the equation [27, 31]

$$P_f = 1 - \left[(1-\alpha)\exp\left(-\left(\frac{\sigma}{\sigma_{01}}\right)^{m_1}\right) + \alpha\exp\left(-\left(\frac{\sigma}{\sigma_{02}}\right)^{m_2}\right)\right] \qquad (3)$$

as well as to results using the unimodal fit (Equation 1).

3. Results and discussion

Fig. 1 shows the histogram of the frequency distribution in dependence of the size of the pores (the number of pores was counted in bins with a interval length of 50 microns). It can clearly be seen that the decrease in the frequency distribution does not perfectly follow the inverse power law as proposed in Equation 1.

Because the parameters of the frequency distribution were obtained by a fit of the experimental values for large pores, the choice of the upper and lower limiting points for the fit (“upper” and “lower” with respect to the number of pores) influences the resulting parameters of the Weibull distribution. The lower limiting

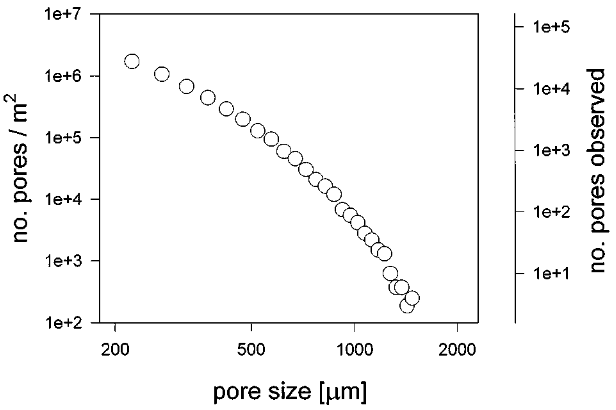

Figure 1 Number of pores counted in the light microscope (right scale) and normalized to area (left scale).

point, because the restricted number of pores of very large size leads to an increase in the scatter. The upper limit, because the slopes, which are necessary to calculate the Weibull modulus, decrease as a consequence of the curvature of the frequency distribution. Fig. 2 shows the influence of a variation of the limiting point at large pore sizes (lower limit). The lower limiting point for the fit varied from 3 to 45 pores, which is equivalent to a lower cut length varying from 1450 to 1050 microns, and the upper limiting point was kept fixed at 850 microns. The choice of this lower limiting point only slightly affects the calculated mechanical parameters. The symbols in these diagrams indicate the predictions from the pore size distributions and the lines the data from the mechanical tests for comparison, respectively: In the left diagram the scale parameter σ_0 from the bimodal fit for the volume pores according to Equation 3 is shown as solid line, from the unimodal fit according to Equation 1 as dashed-double dotted line, whereas the right diagram exhibits the corresponding results for the Weibull modulus.

Contrary to this, the effect of a variation of the upper limiting point of the fit is relatively large, which is a consequence of the increasing curvature of the frequency distribution. In this diagram, the lower point was kept fixed at 1300 microns and the upper limiting point was varied from 450 to 1000 microns (corresponding to a number of 3200 to 67 pores measured). There seems to appear a plateau value for the fit of the scale parameter.

The first of these plateau values was used in all following calculations for the investigation of the statistical behaviour. This chosen interval for the fit ranged from 800 to 1300 microns, corresponding to a measured pore number of about 300 to 10, respectively. The intention was on the one hand that the number of pores for the fit should be as high as possible, in particular for

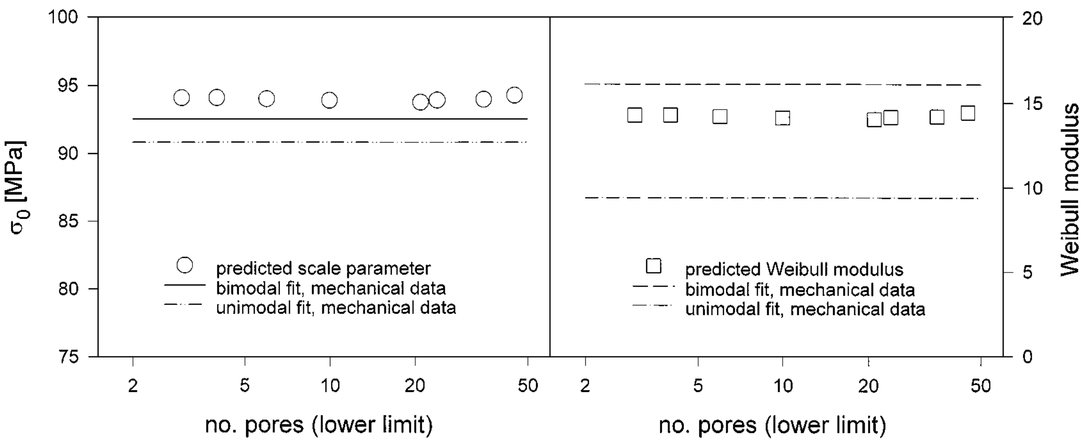

Figure 2 Influence of the lower limiting point chosen for the fit on the resulting Weibull parameters. Symbols: values predicted from pore size distribution. Lines: values measured by mechanical tests, evaluated by either unimodal (Equation 1) or bimodal (Equation 3) Weibull distributions.

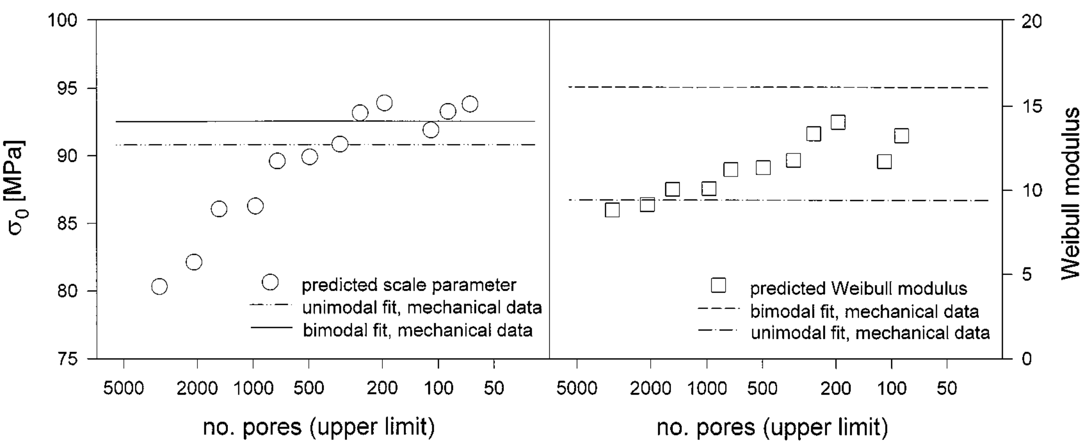

Figure 3 Influence of the upper limiting point chosen for the fit on the resulting Weibull parameters. Symbols: values predicted from pore size distribution. Lines: values measured by mechanical tests, evaluated by either unimodal (Equation 1) or bimodal (Equation 3) Weibull distributions.

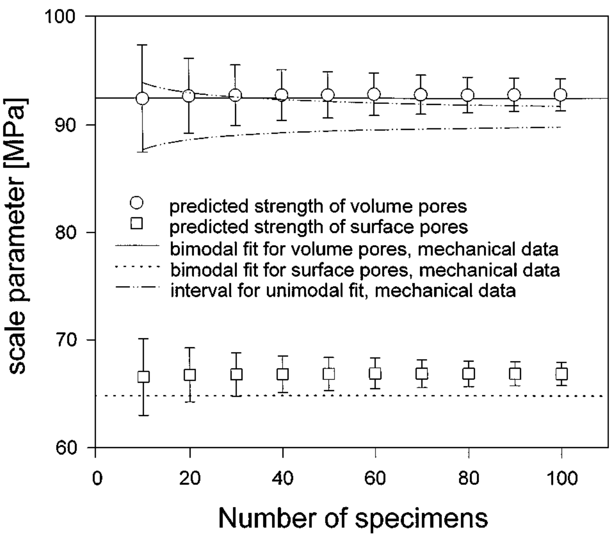

Figure 4 Comparison of predicted strength from pore size measurements (circles: scale parameter of volume pores, squares: scale parameter of surface pores) to the results obtained from the mechanical tests: Solid line: bimodal fit, volume pores, dotted line: bimodal fit, surface pores, dashed-double-dotted line: confidence interval of unimodal fit (mean plus minus one standard deviation).

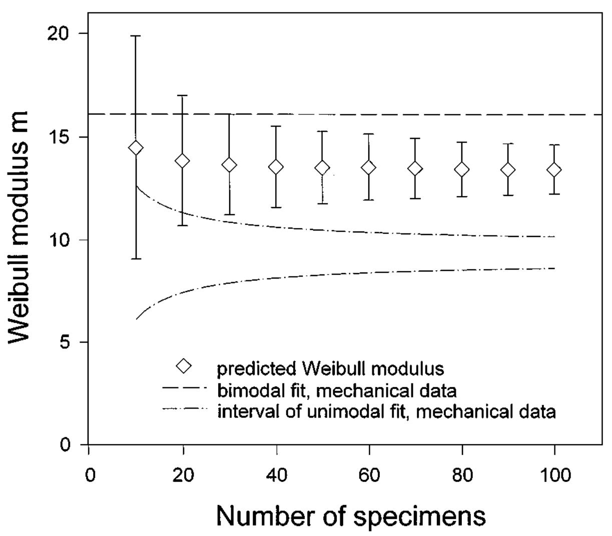

Figure 5 Comparison of predicted Weibull modulus from pore size measurements (diamonds) to the results obtained from the mechanical tests: Dashed line: bimodal fit, dashed-dotted line: confidence interval of unimodal fit (mean plus minus one standard deviation).

the reliability of predictions of smaller areas measured. On the other hand statistical arbitrariness, which occurs if the number of pores counted approaches one, should be excluded. Additionally, the fact that from Griffith theory pore size should be in this range confirms the choice of this interval for the fit. Figs 4 and 5 show the main results: The statistical distribution (i.e. mean and standard deviation) of the predicted values obtained from pore size measurements are shown in dependence on the number of specimens, which correspond to the area required for a certain precision. From each specimen about 130 square millimeters of polished surface were measured. The decrease in the standard deviation of the strength prediction with increasingly measured pore sizes is obvious. The mean of the predicted scale parameters for both volume and surface pores coincides well with the mechanical tests. It should be noted that the reliability of the Weibull parameters from mechanical tests is also limited. This has only been investigated for unimodal Weibull distributions [23]. Therefore, the confidence interval in Fig.4 characterises the unimodal scale parameter range for the mechanical tests, i.e. the mean of the scale parameter plus minus one standard deviation. This interval is similar to the scatter of the pore size predictions, i.e. mechanical testing of a specimen and measuring its pore size distribution lead to a comparable scatter.

The corresponding results for the Weibull modulus are depicted in Fig. 5. The bimodal fit of all bending tests showed a Weibull modulus of 16.1, whereas the unimodal fit resulted only in 9.8. The modulus predicted from pore size measurements lies in between, with a value of 13.4. The dashed-double dotted line characterises the confidence interval (mean plus minus one standard deviation) for the respective number of specimens from the mechanical tests. This confidence interval was calculated for a material, which perfectly obeys a Weibull distribution [23]. In the case of a bimodal behaviour, the variation coefficient of the modulus is significantly higher and could be twice the one of an unimodal distribution [24]. Therefore, one might conclude from Fig. 5 that the scatter of the predictions of the Weibull modulus from pore size measurements seems to be lower than that of the mechanical tests. The situation is, however, complicated by the bimodality in the fracture strength distribution. A bimodal fit of the mechanical data results in two narrower distributions (with higher Weibull modulus) than the description by an unimodal distribution, whereas the predicted value from pore size measurements lies in between. A possible explanation could be that the model is too simplified or the number of large pores observed was not sufficient. Another plausible explanation could be that not all pores have the same shape, which influences the geometry factor in the fracture criterion.

However, considering the large possible scatter, the coincidence could be seen as satisfactory. Due to the high scatter in measuring the Weibull modulus, one should always be careful in interpreting the results, obtained from either mechanical or non-destructive testing. A complete description of the mechanical behaviour of ceramics can only be based on extensive statistics.

4. Conclusion

In the frame of this work it was shown that there exist limits in the possibility to predict the strength of the porous material RSiC by pore size measurements. One limit is that the number of pores does not perfectly obey an inverse power law in dependence on the pore size. The second is that due to the high costs the amount of area, which could be measured, is certainly limited. Both increase the scatter in the prediction of the mechanical parameters in comparison to mechanical testing. The mean and the standard deviation of the predicted parameters are investigated and compared to the ones obtained from mechanical tests. The predicted scale parameter for the volume and the surface pores coincides well with the one calculated by a bimodal Weibull fit of the measured strength data, and the scatter of the mechanical tests and the non-destructive pore size measurement is comparable. Higher deviations were observed for the Weibull modulus, where the scatter was comparable, but the pore size measurement predicted a value for the modulus, which was in between the bimodal and the unimodal fit of the mechanical data. This might be due to several simplifications of the model, a non sufficient statistics of the distribution of large pores or a size-dependent geometry of pores. But the increasing development of suited methods to automatically determine three-dimensional pore size distributions and the subsequent digital image processing offers a lot of possibilities for automatic control during industrial processing and seems to be of great practical interest in future.

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