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Abstract. There is an increasing request for zero expansion glass ceramic ZERODUR® substrates being capable of enduring higher operational static loads or accelerations. The integrity of structures such as optical or mechanical elements for satellites surviving rocket launches, filigree lightweight mirrors, wobbling mirrors, and reticle and wafer stages in microlithography must be guaranteed with low failure probability. Their design requires statistically relevant strength data. The traditional approach using the statistical two-parameter Weibull distribution suffered from two problems. The data sets were too small to obtain distribution parameters with sufficient accuracy and also too small to decide on the validity of the model. This holds especially for the low failure probability levels that are required for reliable applications. Extrapolation to 0.1% failure probability and below led to design strengths so low that higher load applications seemed to be not feasible. New data have been collected with numbers per set large enough to enable tests on the applicability of the three-parameter Weibull distribution. This distribution revealed to provide much better fitting of the data. Moreover it delivers a lower threshold value, which means a minimum value for breakage stress, allowing of removing statistical uncertainty by introducing a deterministic method to calculate design strength. Considerations taken from the theory of fracture mechanics as have been proven to be reliable with proof test qualifications of delicate structures made from brittle materials enable including fatigue due to stress corrosion in a straight forward way. With the formulae derived, either lifetime can be calculated from given stress or allowable stress from minimum required lifetime. The data, distributions, and design strength calculations for several practically relevant surface conditions of ZERODUR® are given. The values obtained are significantly higher than those resulting from the two-parameter Weibull distribution approach and no longer subject to statistical uncertainty. [DOI: 10.1117/1.OE.51.12.124002]

Subject terms: ZERODUR®; mechanical load; design strength; lifetime; stress corrosion; ground surface; etched surface; two-parameter Weibull distribution; three-parameter Weibull distribution.

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1 Introduction and Motivation

Glass and glass ceramic materials should withstand very high bending stresses considering the strength of their atomic bonds. In practice bending strength is reduced dramatically by microcracks in their surfaces by orders of magnitude. The microcrack depths limit bending strength to a higher extent than the very type of material.[1] For technical application, it is important to know the statistical variations of crack depths for surface conditions of common use. The crack depth variations are expected to reveal themselves as statistical variations of the breakage stress for specimens representing a given surface condition. In principle one can try to determine variations of microcrack depths in direct observation. But this is a tedious process, and there is always a residual risk that one did not find the deepest crack in the sample investigated. So it is common to perform breakage stress tests with test areas large enough to include a high number of cracks and evaluate the statistical variation of the breakage stress.

Widely in use as statistical distribution is the Weibull distribution with two parameters, characteristic strength σ_0 and Weibull factor λ.[2–6] For many years, strength data for ZERODUR® are given in form of these two parameters just like for other glassy materials.[7,8] Values for σ_0 and λ are listed for a variety of different surface conditions.[7]

However, working with the two-parameter Weibull distribution suffers from two severe drawbacks.

The first is the strong reduction of design strength, if factor of safety considerations basing on the Weibull distribution mathematics are taken into account.[9] This holds especially for broad distributions. By design strength we mean the minimum breakage stress, which a designed structure must withstand for its service life without breaking.

The second drawback is that this distribution can be extrapolated to arbitrarily low values for finite failure probabilities. There is no lower limit, which on the other hand is expected to exist if there is a maximum crack depth for a given surface condition. For surfaces generated mechanically with grinding tools consisting of many small diamonds suspended in a brass carrier, such maximum crack depth must exist. There is a lot of evidence for this fact.[10]

These drawbacks, together with comparatively large statistical uncertainty coming from data sets with about 20 specimens per set, lead to ultra-conservative design strength

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values, making them almost useless in practice.[11,12] There is hardly anything gained with respect to the rule-of-thumb value of 10 MPa as a safe value for undamaged ZERODUR®. So as a rule, higher stress applications have been avoided.

The increasing request for using ZERODUR® with higher stress applications[13,14] has led to a campaign for obtaining much larger data sets for improving their statistical significance.[15–20] For most of them, numbers are sufficiently high to allow a critical review of the two-parameter Weibull distribution's suitability and to test an alternative distribution. Such distribution is the three-parameter Weibull distribution. With its threshold parameter, which can be interpreted as the long sought for minimum breakage stress reflecting the maximum crack depth, it enables a very different approach for determining design strength or life time with much better significance and also much higher allowable stresses.

2 Strength Design with Two-Parameter Weibull Distributions

The strength data of widely used surface conditions with ZERODUR® such as ground with bonded diamond grains D151, D64, or optical polished or etched are described with special values of the two Weibull parameters characteristic strength σ_0 and Weibull factor λ.[7]

They are derived from experiment by fitting a straight line to the data plotted in a Weibull cumulative distribution diagram (see Fig. 1). Its axes are chosen as a logarithm of the experimentally found breakage stresses as x-axis and the double logarithm of $1/(1-F)$ for the y-axis, with F denoting failure probability, which is calculated from ranking of the stress values. Data lying on a perfect straight line follow an ideal Weibull distribution. Usually one also plots confidence bounds to show the statistical significance of the fitted line.

The two-parameter Weibull distribution [Eq. (1)] was used not only because it fits well to the obtained data but also because it allows calculating design strength straightforward.

$$F(\sigma) = 1 - e^{-\left(\frac{\sigma}{\sigma_0}\right)^{\lambda}}. \tag{1}$$

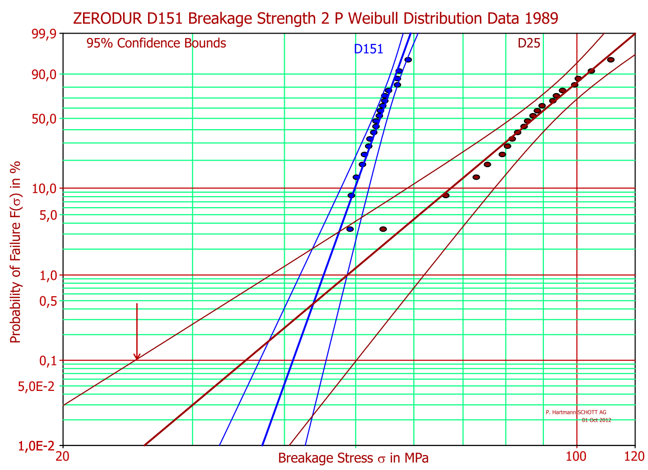


Fig. 1 Breakage stress data for ZERODUR® ground with D151 and D25 diamond tools. Each set consists of 20 specimens. Lines represent two-parameter Weibull distributions with 95% confidence bounds fitted and extrapolated. The arrow points at the lower 95% confidence bound crossing 0.1% failure probability.

As described in detail,[9] it is possible to calculate area up-scaling from the tested area A_T, which is small due to practical reasons to area sizes A_D as are stressed in use of structures.

$$f_A = \left(\frac{A_D}{A_T}\right)^{\frac{1}{\lambda}}. \tag{2}$$

Also factors taking into account failure probability F and fatigue by means of the stress corrosion coefficient n can be given as simple formulae.

$$f_p = \frac{1}{\left[\ln\left(\frac{1}{1-F}\right)\right]^{\frac{1}{\lambda}}} \tag{3}$$

$$f_f = \left(\frac{t_D}{\frac{t_T}{n+1}}\right)^{\frac{1}{n}}. \tag{4}$$

These three factors can be combined to a factor of safety FoS allowing to calculate the minimum breakage stress (design strength) for constant load $\sigma_{B,c}$ from the characteristic Weibull strength σ_0 obtained from experiment.

$$\mathrm{FoS} = f_A \cdot f_P \cdot f_f, \tag{5}$$

$$\sigma_{B,c} \geq \frac{\sigma_0}{\mathrm{FoS}}. \tag{6}$$

A critical review of this approach for ZERODUR® revealed two major disadvantages.

The data sets used for parameter determination consisted of 20 specimens or even less. This means that experimental data cover failure probability only down to about 5%. When designing structures for failure probabilities of 0.1% or even below, extrapolation is needed over more than an order of magnitude. This is not a good fundament for confidence.

The second drawback is the strong reduction of design strength obtained especially for broad distributions with low Weibull parameter λ. Together with several conservative choices in the overall design calculations, this leads to design strength so low that one gains hardly anything over the very conservative value of 10 MPa, which has been used as a rule-of-thumb value in the past being safe but also preventing any application for higher loads.

Figure 1 demonstrates why this method is misleading. Two data sets, each with 20 specimens, are plotted in a Weibull diagram. Both sets represent ZERODUR® surfaces ground with bonded diamond grains. D151 denotes a distribution of diamond grains with sizes around 150 μm, D25 of sizes smaller than 40 μm. The least squares fitted lines are shown as well as 95% confidence bounds. The coarse grain D151 distribution has a steep slope. D25 lies higher and is obviously flatter. This follows the general observation that finer grains leaving smaller microcracks in surfaces lead to higher positioned but flatter distributions.

The diagram shows a crossover of the extrapolated lines at failure probability of about 0.5%, which is strange and in contradiction to practical experience proving finer grain ground surfaces to be stronger. Moreover there is no reason at all why tools with smaller diamond grains should cause deeper microcracks than those with larger diamonds. To

Table 1 Design strength $\sigma_{B,c}$ for constant load calculated with the two-parameter Weibull distribution approach. Characteristic strength and Weibull parameter are given for two ground ZERODUR® surface conditions D151 and D25. Safety factors reduce allowable stress to very low values especially for the finer ground D25 surface, which is expected to be the stronger one.

ZERODUR® ground with	D151	D25
σ_0 Characteristic strength in MPa	54.8	93.2
λ Weibull factor	30.1	11.5
f_A Scaling factor for area ratio 4000:1 (1 m^2 loaded area to 2.5 cm^2 test area)	1.317	2.057
f_P Scaling factor for failure probability of 0.1%	1.258	1.823
f_f Fatigue factor for stress corrosion constant $n = 30$ and 10 y constant load duration	1.775	1.775
Factor of safety $f_A \cdot f_P \cdot f_f$	2.941	6.657
$\sigma_{B,c}$ Design strength in MPa	18.6	14.0

make things even worse, established procedures in design of critical structures require taking not stress values for a specified failure probability from the extrapolated Weibull line but from the lower confidence bound.

In the D25 example, one ends up with design strength of ca. 24 MPa for 0.1% failure probability and 95% confidence whereas the lowest experimental value lies at ca. 54 MPa. Uncritical belief in extrapolation could result in breakage stresses close to zero for very low but finite failure probabilities. This is also in contradiction to practical experience and to results of microcrack depth investigations,[10] where crack depth limits have been confirmed, which should manifest themselves as minimum breakage stresses.

Table 1 shows results of design calculations obtained with experimental parameters of D151 and D25 basing on the Weibull model-derived Eqs. (2) to (6). The area ratio has been chosen as 4000:1, which corresponds to a stressed area of 1 m^2 the test area being 2.5 cm^2, failure probability as 0.1% and stress corrosion coefficient n as 30 the lowest value found for ZERODUR®.[18] The applied load has been assumed to be constant for a 10-year period.

The resulting design strengths are so low that experimental parameter determination and analysis turn out to be not worthwhile at all. Considering lower confidence bounds makes things even worse. In the end such design analyses were discarded in practice, extremely low design strengths were used and applications demanding higher strength values were avoided.

3 Larger Data Sets

3.1 *Measurement Campaign*

In the last years, SCHOTT performed a large measurement campaign in order to improve the data basis for practical surface conditions of ZERODUR® mainly being surfaces ground with bonded diamond tools.[15–20] In order to specify a tool, its diamond grain size distribution is used. The most common tool is D151, a comparatively coarse grain tool for efficient shaping. Tools with finer diamond grains have been investigated (D64 and D25), since they are used for achieving higher strength surfaces. Finally surfaces have been measured, which were etched in order to obtain much higher strength values.

D64 and D151 are the denominations of grain size distributions according to the FEPA specification of diamond abrasives. FEPA is the federation of European producers of abrasives.[21] D151 stands for a range of grain sizes limited by two sieves with mesh widths of 125 and 150 μm according to the international standard ISO 6106.[22] D151 corresponds to US mesh number 100/120. D64 specifies a range of 53 to 63 μm (US mesh number 230/270). The microgrit D25 used in this investigation had a maximum diamond grain size of 40 μm.

Etching had been done according to the procedure, which is commonly used at SCHOTT. Each sample was etched in batches of 12 specimens. The layers removed from the surfaces were measured from the specimens' thickness change.

3.2 *Experiments*

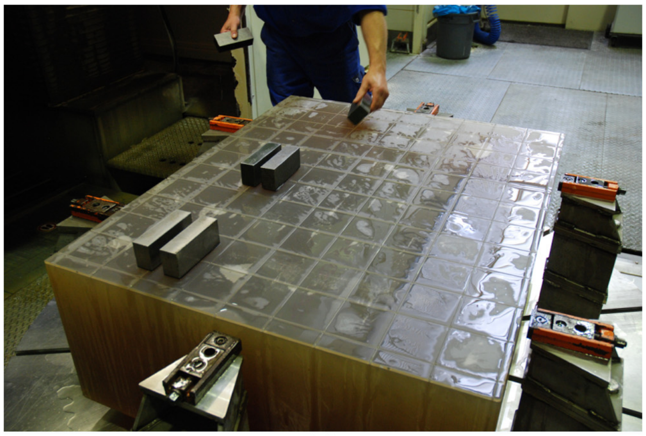
In order to obtain largest possible sets of specimens with equal preparation process while keeping costs lowest possible 100×100 mm^2 tiles of ZERODUR® have been produced from plates cut off from a large round block with diameter 1.5 m yielding 148 specimens at maximum or from a rectagular 1×1.2 m^2 block with 99 specimens (Fig. 2). 10-mm-thick plates lying on top of the original block were ground down to 6 mm. While approaching the final thickness, special care was taken to remove any subsurface microcracks from the preceding process. For the D25 specimens, an intermediate step with D64 was introduced. After finishing surface grinding, the plate was cut in a rectangular pattern with a diamond wheel so that tiles could be separated by hand.

Grinding kinematics was optimized for best homogeneous surface preparation. As a monitoring measure, the relative position of each tile in the original plate was recorded. The spacial pattern of the tiles' breakage stresses per plate was inspected for obvious inhomogeneity. The procedure is described in more detail in Refs. 15 and 18.

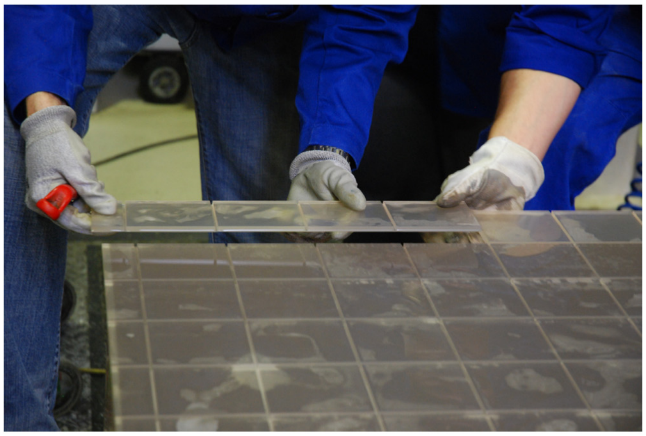
All measurements have been performed employing the ring-on-ring method described in the European standard EN 1288-5[23] with an R 45 adapter (Fig. 3). This method is preferred since it provides a very homogeneous stress field across the load ring area and radial and tangential stresses are almost equal. From the edge of the load ring the stress field decreases sharply to the specimens edges. This reduces breakages outside of the load ring very efficiently. If occurring such specimens would have to be discarded. The square specimen lies on the support ring of radius 45 mm and is bent by a force applied with the help of a ring with radius 9 mm, increasing with load rate $\dot{\sigma}_r = 2$ MPa. Breakage origin locations were recorded. Only specimens, which broke within or at the load ring, have been used, according to the requirements of European standard EN 1288-5.

4 Results

The original data had been evaluated using the two-parameter Weibull distribution. Closer reexamination of D25 data showed that at the low end, several specimens had to be removed because they were obviously outliers.[18]

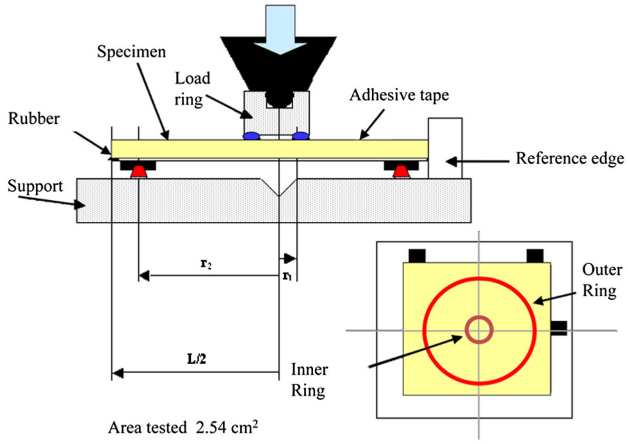


(a)

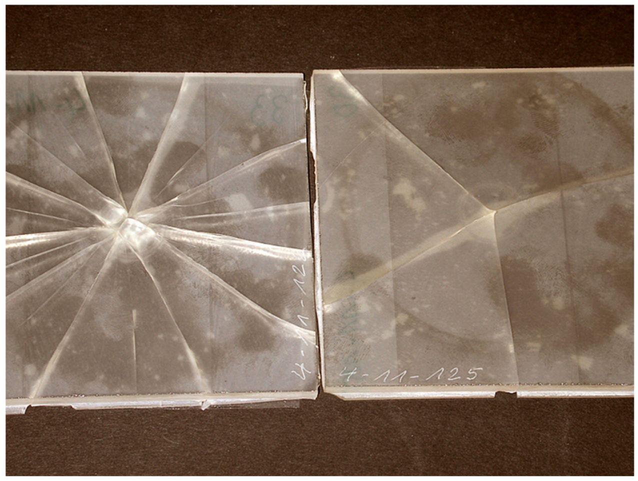


(b)

Fig. 2 (a) Finished ground rectangular plate on its original block ready for separation of the specimen tiles; (b) taking single ZERODUR® specimens.



(a)



(b)

Fig. 3 (a) Ring-on-ring test setup according to the European standard EN 1288-5; (b) two specimens broken at different stresses.

Their recorded locations in the original ZERODUR® plate were close to each other at the same edge. They were discarded as a result of imperfect grinding at the beginning of grinding cycles, when the tool entered the surface, as not representative for the overall surface conditions. With the outliers removed deviations from the Weibull, distribution became clearly visible (Fig. 4).

With this observation in mind, results for D151 and D64 were reevaluated. Also here, deviations of the same kind could be seen clearly now. In the past they probably had been overlooked due to preoccupation with the two-parameter Weibull approach. Hence we deem the existence of deviations to be proven with satisfying statistical significance and see clear evidence for the existence of minimum breakage stresses for each surface condition.

Such data sets are expected to be fitted better with the three-parameter Weibull distribution with threshold stress σ_T, scale parameter η, and shape parameter β.

$$F(\sigma) = 1 - e^{-\left(\frac{\sigma-\sigma_T}{\eta}\right)^{\beta}}. \tag{7}$$

Earlier applications of this distribution showed no significant relevance because either there were still too few specimens in a data sample[24] or investigated materials such as

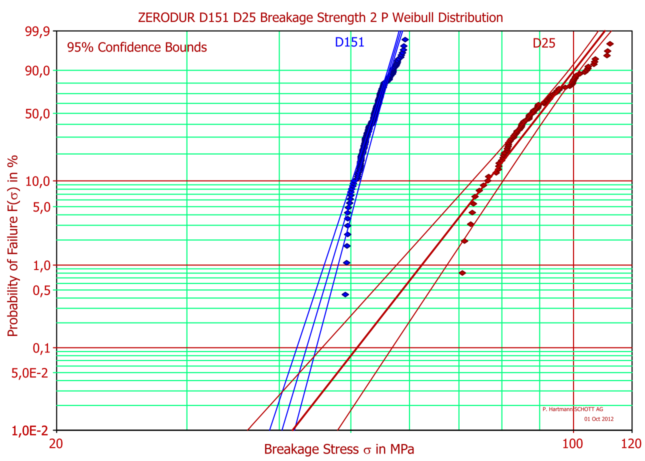


Fig. 4 Larger data sets for breakage stress of ZERODUR® ground with D151 (138 specimens) and D25 (86 specimens) diamond tools. Data fitted with two-parameter Weibull distributions with 95% confidence bounds and extrapolated. Deviations from the two-parameter Weibull distribution at the lower end are obvious.[20]

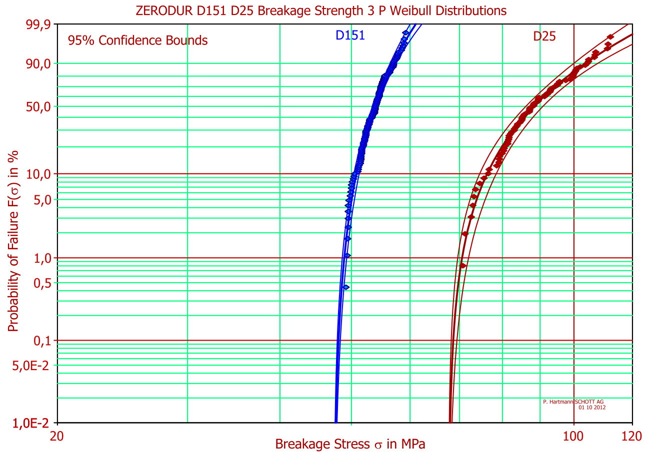


Fig. 5 Breakage stress data for ZERODUR® ground with D151 and D25 resp. diamond tools, same data as in Fig. 4, now fitted with three-parameter Weibull distributions with 95% confidence bounds and extrapolated. The distributions support the existence of minimum breakage stresses for both data sets very clearly.

silicon carbide might lack a maximum flaw size for a given production process.[25] SiC usually breaks due to volume flaws as result from imperfect sintering.

Figure 5 shows the same data as Fig. 4 with three-parameter Weibull distributions fitted, which shows well agreement with fit.[20] The fits deliver values for minimum stresses σ_T of 47.3 MPa (D151) and 67,7 MPa (D25).

These values lie close to the experimental data points thus increasing credibility by avoiding wide extrapolations over empty data space.

The existence of minimum values now proven by experiment removes the need for calculations of low failure probabilities and area scaling factors. The only effect remaining to be taken into account is stress corrosion. This can be done in a straightforward manner.

5 Lifetime Calculations Including Stress Corrosion

Lifetime calculations have to take into account the fact that glassy materials show some strength degradation when they are exposed to tensile surface stress. This phenomenon is called static fatigue, referring to the fact that it proceeds even under constant stress. Another denomination—subcritical crack growth—refers to its origin, the slow, or in many application cases extremely slow growth of microcracks below the glass surface. As the most important environmental influence, humidity enhances crack growth, which is now being called stress corrosion.[26,27]

The main parameter influencing crack growth velocity is stress intensity factor K, which depends on stress σ being effective at the tip of an initial microcrack with length a and geometrical factor f.[28] This factor f is close to 2 for the microcracks under discussion.

$$K = \sigma \cdot f \cdot \sqrt{a}. \tag{8}$$

Crack growth velocity depends on K following an exponential law over many orders of magnitude. For ZERODUR® it holds for the range from 0.1 mm/s down to 1×10^{-8} mm/s.

$$\frac{\mathrm{d}a}{\mathrm{d}t} = AK^n. \tag{9}$$

The exponent n is mainly influenced by humidity in the environment and thus called stress corrosion coefficient. The factor A crack growth parameter is less important.

The crack growth law with the definition equation for K inserted can be integrated for constant stress.[29,30] Since for lifetime determination, integration is necessary only for the range, where the crack is still short, the stress field at its tip, and geometrical factor f can be assumed to be constant:

$$\frac{\mathrm{d}a}{\mathrm{d}t} = A(\sigma\sqrt{af})^n, \tag{10}$$

$$\mathrm{d}a = A(\sigma\sqrt{af})^n \mathrm{d}t, \tag{11}$$

$$\int_{t_i}^{t_B} \mathrm{d}t = \int_{a_i}^{a_B} \frac{1}{A(\sigma\sqrt{af})^n} \mathrm{d}a. \tag{12}$$

The integral allows calculating lifetime $t_{B,c}$ until breakage (denoted with "B") occurs under constant stress $\sigma = \sigma_c$ load with the initial crack length a_i and crack length a_B just before breakage:

$$t_{B,c} = \frac{2}{(n-2)A}\sigma_c^{-n} f^{-n}\left(a_i^{-\frac{(n-2)}{2}} - a_B^{-\frac{(n-2)}{2}}\right). \tag{13}$$

The second term in the bracket is much smaller than the first term for typical glasses and glass ceramics with $n \gg 10$. So the expression for lifetime $t_{B,c}$ under constant stress load reduces to:

$$t_{B,c} = \frac{2}{(n-2)A}\sigma_c^{-n} f^{-n} a_i^{-\frac{(n-2)}{2}}. \tag{14}$$

Solving Eq. (14) for stress renders the breakage stress for a required minimum lifetime:

$$\sigma_{B,c} = \left[\frac{2}{(n-2)A} t_{B,c}^{-1} f^{-n} a_i^{-\frac{(n-2)}{2}}\right]^{\frac{1}{n}}. \tag{15}$$

Integrating the crack growth law [Eq. 14)] for a constant stress increase rate $\dot{\sigma}_r$ defined by

$$\sigma = \dot{\sigma}_r t \tag{16}$$

as follows:

$$\int_{t_i}^{t_B} t^n \mathrm{d}t = \int_{a_i}^{a_B} \frac{1}{A\dot{\sigma}^n f^n} a^{-\frac{n}{2}} \mathrm{d}a, \tag{17}$$

which yields breakage stress $\sigma_{B,r}$ for constant stress increase rate

$$\sigma_{B,r} = \left[\frac{2(n+1)}{(n-2)A}\dot{\sigma}_r f^{-n} a_i^{-\frac{(n-2)}{2}}\right]^{\frac{1}{(n+1)}}. \tag{18}$$

Equation (15) for breakage stress for constant stress $\sigma_{B,c}$ and Eq. (18) for breakage stress for constant stress increase rate $\sigma_{B,r}$ both contain the expression

$$\frac{2}{(n-2)A} f^{-n} a_i^{-\frac{(n-2)}{2}}, \tag{19}$$

which can be used to combine them, obtaining Eq. (20) delivering constant stress lifetime $t_{B,c}$:

$$t_{B,c} = \frac{\sigma_{B,r}^{n+1}}{\sigma_{B,c}^{n}} \frac{1}{(n+1)\dot{\sigma}_r} \tag{20}$$

from experimentally determined parameters:

- breakage stress $\sigma_{B,r}$ measured with constant stress increase
- experimental stress increase rate $\dot{\sigma}_r$
- stress corrosion coefficient n for the given material in application enviroment

and required design strength $\sigma_{B,c}$.

Equation (20) can be solved for design strength $\sigma_{B,c}$

$$\sigma_{B,c} = \left[\frac{\sigma_{B,r}^{n+1}}{t_{B,c}} \frac{1}{(n+1)\dot{\sigma}_r}\right]^{\frac{1}{n}}. \tag{21}$$

Our proposal for a new deterministic method to calculate lifetime or design strength bases on the use of the experimentally determined minimum breakage stress σ_T for well defined surface conditions in Eqs. (20) and (21) for the quantity $\sigma_{B,r}$, the breakage stress measured with constant stress increase rate $\dot{\sigma}_r$. Together with stress corrosion constant n all quantities are measured or given as specified value (life time or design strength). Using the minimum breakage stress σ_T removes statistical variations and hence the root for large factors of safety as well as the necessity of the area scaling factor since σ_T holds for all areas of the same condition of any size.

Figure 6 shows life time $t_{B,c}$ depending on design strength $\sigma_{B,c}$ according to Eq. (20) for ZERODUR® ground with D151 bonded diamond grains. As breakage stress $\sigma_{B,c}$ the minimum breakage stress $\sigma_T = 47.3$ MPa found for D151 is used. Experimental stress increase rate $\dot{\sigma}_r$ is 2 MPa/s. In Fig. 7, design strength $\sigma_{B,c}$ is plotted against lifetime $t_{B,c}$ for D151 ground ZERODUR® according to Eq. (21). Figures 8 and 9 show lifetime and design strength for ZERODUR® ground with D25 bonded diamond grains using $\sigma_T = 67.7$ MPa for $\sigma_{B,r}$.

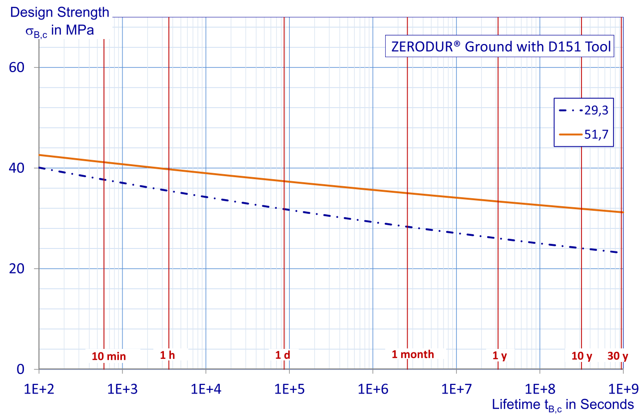


Fig. 7 Design strength $\sigma_{B,c}$ of ZERODUR® structures ground with D151 diamond tool depending on specified life time $t_{B,c}$. Minimum breakage stress $\sigma_T = 47.3$ MPa. Curves reflect two different stress corrosion constant n values 29.3 and 51.7.

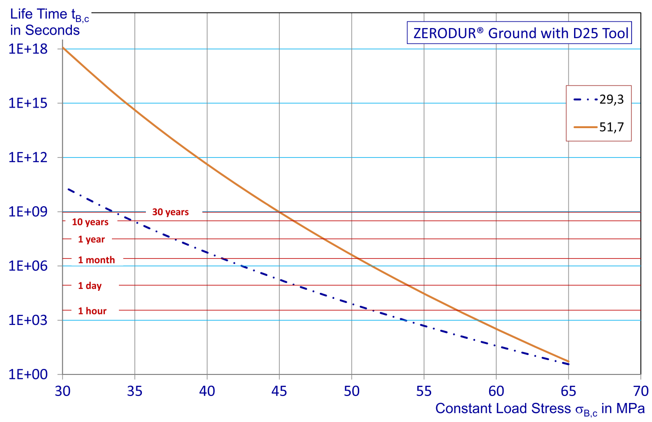


Fig. 8 Lifetime $t_{B,c}$ of ZERODUR® structures ground with D25 diamond tool depending on design strength $\sigma_{B,c}$. Minimum breakage stress $\sigma_T = 67.7$ MPa. Curves reflect two different stress corrosion constant n values 29.3 and 51.7.

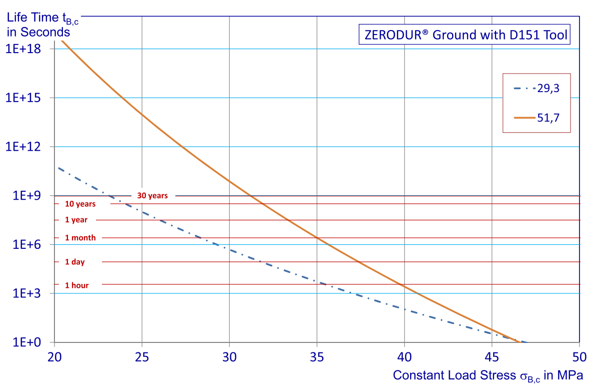


Fig. 6 Lifetime $t_{B,c}$ of ZERODUR® structures ground with D151 diamond tool depending on design strength $\sigma_{B,c}$. Minimum breakage stress $\sigma_T = 47.3$ MPa. Curves reflect two different stress corrosion constant n values 29.3 and 51.7.

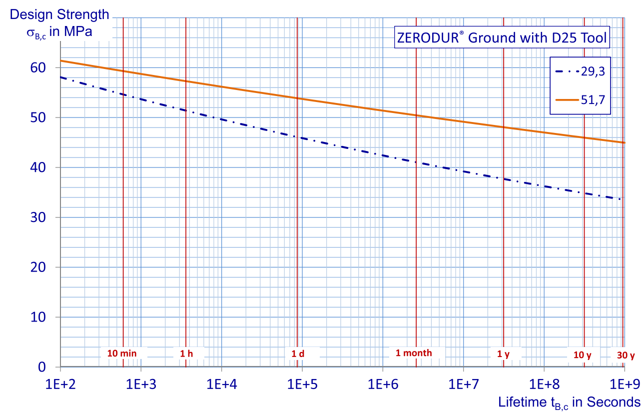


Fig. 9 Design strength $\sigma_{B,c}$ of ZERODUR® structures ground with D25 diamond tool for specified lifetime $t_{B,c}$. Minimum breakage stress $\sigma_T = 67.7$ MPa. Curves reflect two different stress corrosion constant n values 29.3 and 51.7.

Table 2 Design strength $\sigma_{B,c}$ for ZERODUR® ground with D151 and D25 diamond tools calculated with the two different methods presented: the traditional two-parameter Weibull distribution based model as outlined in chapter 2 (columns "2P") and the new method proposed in chapter 4 using the minimum breakage stress obtained from fitting three-parameter Weibull distributions to the data sets (columns "3P"). Application specifications were chosen arbitrarily.

ZERODUR® ground with	D151		D25	
Model Weibull with parameters	2P	3P	2P	3P
$\sigma_{0/T}$ characteristic/threshold stress in MPa	σ_0 54.8	σ_T 47.3	σ_0 93.2	σ_T 67.7
λ/η Weibull factor/scale parameter	λ 30.1	η 7.32	λ 11.5	η 24.4
β Shape parameter	—	3.04	—	2.16
f_A Area factor for ratio 4000:1	1.317	n.a.	2.057	n.a.
f_P Probability factor (0.1%)	1.258	n.a.	1.823	n.a.
f_f Fatigue factor ($n = 30$; 10 years)	1.775	n.a.	1.775	n.a.
Factor of safety $f_A \cdot f_P \cdot f_f$	2.941	n.a.	6.657	n.a.
$\sigma_{B,c}$ Design strength in MPa	18.6	24.0	14.0	38.4

Two curves in each diagram reflect different values for stress corrosion coefficient n: 51.7 a value widely used in the past for ZERODUR® in normal humidity determined with direct crack propagation observation and 29.3, the lowest value found up to now with the method using samples with characteristic breakage strengths determined at different stress increase rates.[18]

Table 2 shows a direct comparison of design strength results obtained from the two different approaches described above. For the first approach, the two-parameter Weibull distribution based model, the same parameters have been chosen as in Table 1, i.e., area ratio 4000:1, failure probability 0.1%, stress corrosion coefficient n as 30 and 10 years constant load. The columns are marked with "2P."

Table 2 contains two additional columns ("3P") with the three-parameter Weibull distribution fit parameters: threshold or minimum breakage stress σ_T, scale parameter η and shape parameter β. Factor of safety considerations used with the two-parameter Weibull based model play no role here. Design strength values in the last row have been calculated using Eq. (21) for specified lifetime of 10 years, stress corrosion coefficient $n = 30$, experimental stress increase rate 2 MPa/s and minimum breakage stress σ_T as listed for D151 and D25.

The gain in allowable stress is very remarkable. For D151 design strength rises from 18.6 to 24.0 MPa, for D25 it is even much more from 14.0 to 38.4 MPa.

The D25 values demonstrate how misleading the extrapolation of the two-parameter Weibull down to low failure probability is. Such discrepancies are to be expected for all high-strength ZERODUR® surface conditions since they are characterised by wide breakage stress distributions.

6 More Ground and Ground and Etched Surface Conditions

In the preceding paragraphs large data set results for ZERODUR® ground with D151 and with D25 were presented and used to demonstrate the findings concerning minimum stresses and how this was utilised for a deterministic approach for obtaining design strength.

Further evaluations have been done reconsidering more data sets, which had been published in the last years,[18] with respect to the new approach. Figures 10 and 11 show the same D151 and D25 data sets as above together with a D64 ground surface and two etched surfaces starting from different preground states D151 and D64. The D64 preground surface was etched removing 73 μm layer thickness lying above the maximum crack depth of about 60 μm, the D151 was etched removing 83 μm lying below the minimum crack depth of about 120 μm.

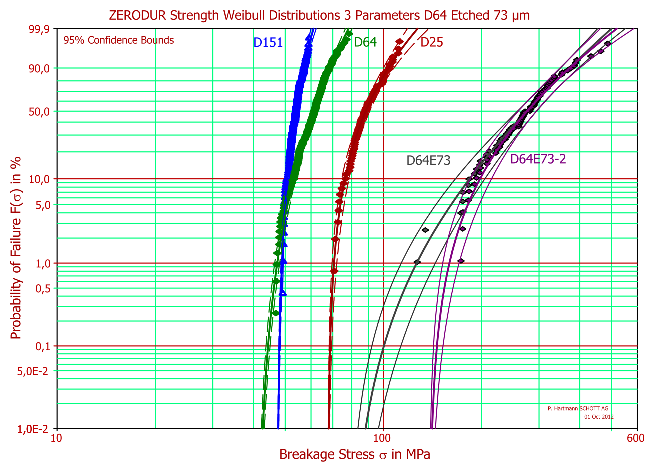


Fig. 10 Breakage stress distributions for ZERODUR® ground with D151, D64, and D25 as already published[20] and additionally one set with specimens ground with D64 and subsequently a layer of 73 μm etched off D64E73. D64E73-2 shows the same data set with two outliers removed. The weakest observed specimen broke at 173 MPa or 127 MPa (outlier)

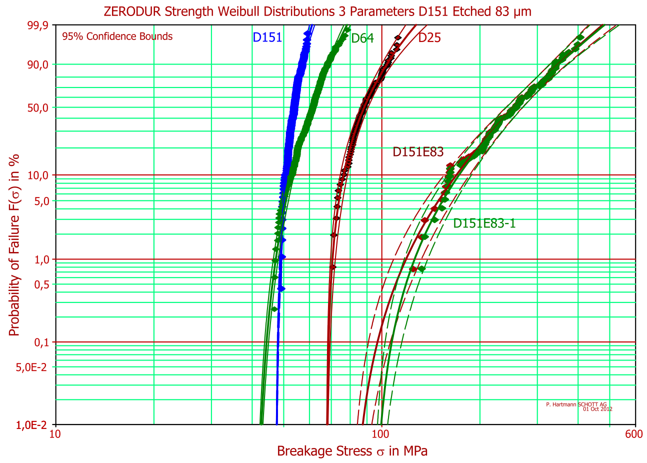


Fig. 11 Breakage stress distributions for ZERODUR® ground with D151, D64, and D25 as already published[20] and additionally one set with specimens ground with D151 and subsequently a layer of 83 μm etched off D151E83. D151E83-1 shows the same data set with one outlier removed. The weakest observed specimen of D151E83 broke at 132 MPa or 125 MPa (outlier)

The D64 ground surface distribution is also well represented by a three-parameter Weibull distribution. However, in contrast to expectations from microcrack depths considerations the minimum stress lies with 40.9 MPa below that of D151 with about double-sized diamond grains. One would have expected the minimum value of D64 lying fairly higher instead of lower than that of D151. An explanation for this phenomenon does not exist up to now.

The samples representing ground and then etched specimens are given twice in each diagram. One sample contains all measured data, the other shows results for distributions with outliers removed. With D64E73 (the E73 means 73 μm etched off) there is evidence for the existence of two outliers.

Table 3 contains results for etched surfaces preground with D151 and D64. Etched D25 were not measured since such combination is not reasonable in practical application. Just as in Table 2 parameters obtained from the same data sets for each surface condition have been used to derive design strength values with the two different methods referred to as two-parameter and three-parameter Weibull distributions based.

The experimentally determined parameters are listed in the first three rows containing characteristic strength or threshold stress and Weibull factor or scale parameter, whichever applies for the given distribution. In order to calculate design strength the same application conditions as in the previous examples have been chosen: area factor 4000, admissible failure probability 0.1% and fatigue affecting the structure for 10 years under constant load with stress corrosion coefficient $n = 30$.

Comparison between the two-parameter Weibull based method and the new proposed one using fatigue adjusted threshold strength demonstrates how far results of the first method lie away from any reasonable real design strength and from obtained experimental values. The weakest observed specimen broke at 125 MPa including even those discarded as outliers. The traditional model results lie more than a factor of 10 below this value.

For analyzing the data sets for etched surface conditions D151E83 and D64E73, threshold stresses have been used including and excluding outliers. The latter ones are marked with brackets.

The minimum improvement gained in design strength (see last row of Table 3) with the new method is a factor 4.5 increase from 9.2 to 41.4 MPa for D151 and a factor 6.8 from 5.9 to 40.1 MPa for D64. Comparing design strength analyzed with the new method for ground surfaces with that for preground surfaces with additional etching applied one finds an improvement of 73% from 24.0 MPa to 41.4 MPa for D151 with etching layer 83 μm (compare $\sigma_{B,c}$ of "3P" for D151 in Table 2 and 3) and 77% from 22.6 to 40.1 MPa for D64 with etching layer 73 μm. The D64 value of

Table 3 Design strength for ZERODUR® ground with D151 and 83 μm etched off and with D64 and 73 μm etched off. In the first column for each surface condition two-parameter Weibull based evaluations ("2P") have been performed using the same application parameters as in Tables 1 and 2.

ZERODUR® preground/etched	**D151 E83**		**D64 E73**	
Model Weibull with parameters	2P	3P	2P	3P
$\sigma_{0/T}$ characteristic/threshold stress in MPa in brackets: with outliers removed	σ_0 281.8	σ_T 79.9 (94.1)	σ_0 303.1	σ_T 77.5 (138.5)
λ/η Weibull factor/scale parameter	λ 5.34	η 199.2	λ 4.51	η 223.3
β Shape parameter	—	2.81	—	3.03
f_A Area factor for ratio 4000:1	4.727	1	6.290	1
f_P Probability factor (0.1%)	3.646	1	4.625	1
f_f Fatigue factor ($n = 30$; 10 years)	1.775	n.a.	1.775	n.a.
Factor of safety $f_A \cdot f_P \cdot f_f$	30.587	n.a.	51.649	n.a.
$\sigma_{B,c}$ Design strength in MPa in brackets: with outliers removed	**9.2**	**41.4 (49.0)**	**5.9**	**40.1 (73.0)**

22.6 MPa has been calculated with the same assumptions as used in Tables 1 to 3 and the threshold stress value of 40.9 MPa.[20] Removing outliers leads to higher design strength values especially for D64E73 (73.0 MPa). This shows a significant influence of single or few data points at the distributions lower end being more pronounced for the etched specimens' sets. In order to obtain reliable threshold stress values with fitting three-parameter Weibull distributions care has to be taken to check the lowest data points of being outliers. It should be pointed out, that calculating lifetime or design strength of etched items according to Eqs. (20) and (21) most probably will lead to significant underestimation. The microcrack propagation model is not valid anymore for such surfaces, since crack tips will not be sharp down to atomic level but rounded. This is expected to reduce stress concentration and hence effectiveness of stress corrosion. Etched items therefore will not be only stronger from the beginning of loading but will also maintain their strength longer than ground items.

Two additional etched specimens data sets already published[16] have been evaluated, D64E91 and D151E181, using the three-parameter Weibull distribution. However, the number of specimens of set D64E91 with 39 is too small to observe a deviation from the two-parameter distribution fit. Most of the originally 109 specimens had broken outside the load ring area and therefore had to be discarded. With the D151E181 sample it is not possible to fit a three-parameter distribution with a reasonable threshold at all. Both sets represent very deeply etched surfaces with layers taken off much thicker than the maximum crack depths expected for the just ground surfaces. Such etching changes surface characteristics dramatically. So it is not really surprising that they cannot be described with the same statistical distribution as the just ground surfaces. A fact that should be noted anyway is that for all surfaces, which were etched deep enough to achieve much higher experimental breakage stresses, no specimen was found to lie lower that 125 MPa. Also important for practical purposes is to make sure taking off a minimum layer thickness with etching. If this layer is much thinner than the maximum crack depth of a given ground surface state etching has no strength increase effect at all.[18]

7 Conclusion

The use of the three-parameter Weibull distribution leads to a much better representation of experimental data for ZERODUR® breakage stresses for ground and moderately etched surfaces. Its third-parameter threshold stress is a plausible equivalent to the maximum depth of subsurface microcracks, which is to be expected from their originating process grinding with small diamonds with well defined and limited size distributions. The experimentally proven minimum stresses for given surface distributions allow lifetime or design strength calculations without the need for safety factors reflecting area effects, failure probability, or statistical uncertainty. The effect of stress corrosion is taken into account. The presented method enables higher loads to be applied with reliably calculable lifetime prediction and thus can be valued as breakthrough with respect to the traditionally used method.

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