

# TIE-33 Bending strength of optical glass and ZERODUR®

## 1. Introduction

### 1.1 Application cases and questions

Elements made from optical glass, filter glass or glass ceramics usually do not have to endure high mechanical loads. Built into frames they need only carry their own weight. However, there are applications, where higher mechanical loads will occur either directly from high weight, pressure or accelerations or indirectly from thermal gradients leading to mechanical stress. Such applications may be:

- Vacuum windows (static load of 0.1 MPa pressure)
- Wind channel windows (dynamic pressure loads together with temperature gradients caused by chilling during gas expansion)
- Large thin mirrors with high diameter to thickness ratio (> 6:1)
- Mirrors to be launched to space which as a rule are filigree lightweight mirrors
- Bonding between elements of the same material or to metals

In such cases questions arise

- How strong is the glass or glass ceramic?
- Will it withstand the loads in application during its scheduled lifetime?
- How low is the failure probability?

Such questions have to be translated into physically based technical quantities, which allow measurements, calculations and predictions. This technical information shall give a short overview of methods used and provides a large set of data acquired for SCHOTT optical materials. For detailed presentations of the general theories, please refer to the literature and publications e. g. [1 – 4].

### 1.2 General aspects

The central physical quantity in strength discussion of glass is tensile stress at its surfaces. Breakage will occur when tensile stress surpasses a threshold value, which depends on the depths of the surface flaws. Surface flaws in this context mean subsurface micro cracks extending into the bulk material ending in a tip with atomic dimension cleavage. They must not be confused with surface roughness, which can be measured with a mechanical stylus.

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A piece of glass with surface flaws will not break when there is no tensile stress. Neither will it break when there is tensile stress but there are only few and small flaws in the surface. The quantity bending strength means the maximum tensile stress a glass item with well-defined geometry and surface condition can endure in the application environment during the intended lifetime with acceptable low failure probability.

Tensile stress may cause a micro crack to grow by opening the crack and ripping atomic bonds apart at the tip of the crack. Compressive stress will close a crack not healing it but also not growing it. Glass items can endure much higher compressive stress loads. Such loads are limited in practice by mechanical reaction leading to tensile stress somewhere else at the loaded item's surface. With the special shape of a sphere it is possible to design glass windows, which can endure extremely high compressive stress. Such windows are used in deep diving submarine vehicles, for example.

The strength of glass items may degrade with time during their usage. One cause is continuous worsening of the surface condition due to wear. This is the main influence in household glass items but normally not with optical elements. Here the aging effect under tensile load usually called fatigue may be more prominent. This is the growth of already existing surface micro cracks. It has to be emphasized that this effect will occur

only under tensile load above a given minimum. As long as a glass item is stored without load and protected against the environment, no degradation of its strength may be expected.

The growth of micro cracks under tensile load is enhanced in the presence of water be it liquid water or humidity. Water molecules facilitate ripping atomic bonds of silicon and oxygen apart, which are the basis elements of most common glass types. Glass loaded in dry environment especially in vacuum has the lowest fatigue effect.

Considering failure probability the first reaction is usually the requirement zero failure probability. Consequently, no glass items would ever have been used. And this also applies for all other technical objects. The main question is which failure probability is acceptable. Such limits strongly depend on the worth of the glass item and the consequences of the failure. For large astronomical mirrors and airplane windows, the limits will be much lower than for household glassware. The general tendency to reduce failure probability as far as possible leads to the introduction of too many safety factors. In the end, glass items may be excluded from applications, which they could have easily endured. For the design of a glass application under load the acceptable failure probability should be considered and a safety factor should be introduced only at the last stage of the calculations.

### 1.3 Bending strength

Bending strength is not a well-defined material-dependent physical property of glasses or glass ceramics comparable to density or Young's modulus. Due to the brittleness of these materials, strength depends much more on the condition of their surfaces than on the strength of their atomic bonds. Micro cracks or scratches at their surfaces will grow in depths under tensile stress. At low stress they might not grow at all, at higher stress they will grow very slowly in many cases without causing problems within a glass item's scheduled lifetime. After surpassing a certain threshold a crack will speed up in short time to terminal velocity which means that breakage occurs. Within brittle materials there is no compensation mechanism for scratches such as straining which is the case for ductile materials.

With brittle materials number and depths of micro cracks in the surface are of much higher importance than the actual material itself. Breakage stress of glasses and glass ceramics calculated purely from the strength of their atomic bonds may lie higher than 10 GPa (1 Giga-Pascal =  $10^9$  N/m<sup>2</sup>). For glass items exposed to normal working environment strength drops by several orders of magnitude to the lower single-digit Mega-Pascal range. Glass ceramics being somewhat stronger can be used up to 10 MPa without special considerations if they do not show obvious serious surface damage.

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The importance of the surface condition compared with that of the material type is underlined by the fact that the difference in strength between coarsely ground and highly polished surfaces may be higher than a factor of ten, whereas the difference between brittle materials with strongly varying composition will be only a factor of three at the same surface preparation.

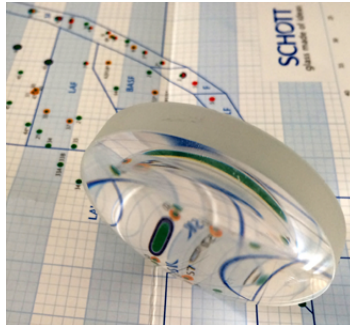


Figure 1: Examples for different surface conditions  
Left: Lens with polished faces with ground lateral face. Right: ZERODUR® lightweight mirror back face.

### 1.4 Surface conditions

Generally one has to distinguish between two types of surface conditions: Polished or etched surfaces, where only few isolated cracks may be present and ground or lapped surfaces with many micro cracks per area.

For isolated cracks in principle it would be possible to calculate strength based on deterministic fracture mechanics considering the growth of a crack under the given conditions. Such calculation is quite involved; in many cases the necessary material parameters are not known precisely enough. The initial crack depth is also not known and hard to determine in practical cases. This is especially problematic since very small variations in initial crack depth lead to huge variations (orders of magnitude) in the results.

With ground surfaces a different approach becomes necessary. The distributions of micro crack depths require a treatment with statistical methods. Commonly applied is the Weibull distribution. It is based on the weakest link ansatz. This corresponds to the statistical distribution of the deepest cracks.

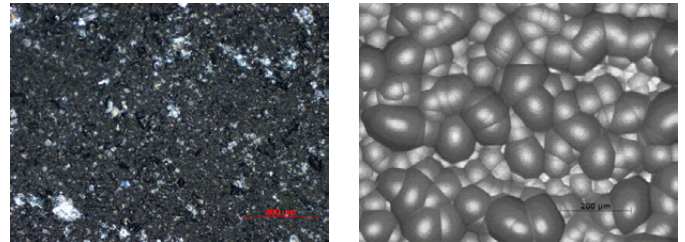


Figure 2: Surfaces of ZERODUR®, Pictured width ca. 0.9 mm  
Left: Ground with D151 bonded diamond grains. Right: strongly etched

Optical elements consist of functional polished faces affecting refraction or reflection and of usually ground lateral or back faces. The ground faces are the weaker ones. Moreover, tensile stress caused by transport, handling and application usually occurs at these faces. Therefore this technical information concentrates on the strength of ground faces or ground faces etched for strength enhancement.

## 2. Breakage stress measurement

The breakage stress measurement setup and the specimens' preparation must assure that the results obtained are valid for the specific surface condition under investigation. Specimens used are disks or plates, which are loaded with increasing force until they break. It is important to know the tensile stress at the time and location where the breakage occurs. Since it is not possible to exert a homogeneous stress field all over the specimens' area, care must be taken that the breakage origin

lies in the investigated surface where stress is known sufficiently precisely [5, 6].

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In addition to the surface to be investigated, specimens have another weak element – the edges. The experimental setup should be chosen in a manner that avoids breakages starting at the edge. Specimens broken in such way must not be included in the data set, which shall be representative for the investigated surface.

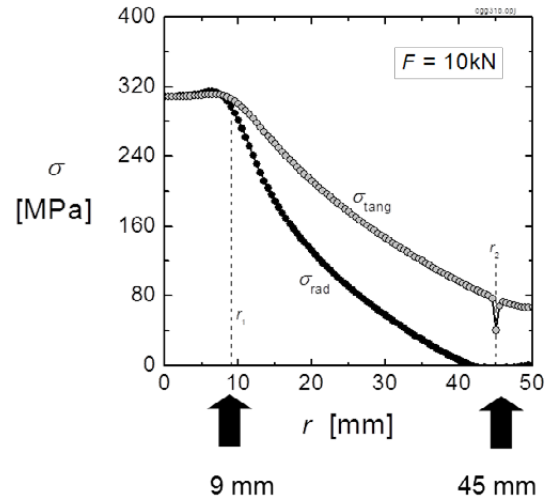
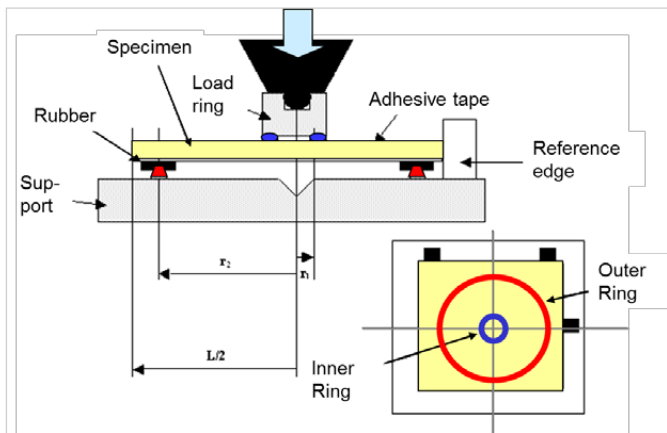


Figure 3: Left: Ring-on-ring breakage measurement setup according to the European standard EN 1288-5 for quadratic specimens. Right: Stress as a function of distance from 9 mm radius load ring center with 45 mm diameter support ring.

The ring-on-ring breakage measurement setup according to the European standard EN 1288-5 [7] provides a very homogeneous central stress field with well-defined diameter, see Figures 3 and 4. Across the load ring area radial and tangential stresses are almost equal. From the edge of the load ring stress drops sharply in direction towards the specimen's edge. With ground surfaces specimens break with origins only within the load ring area (Figure 4).

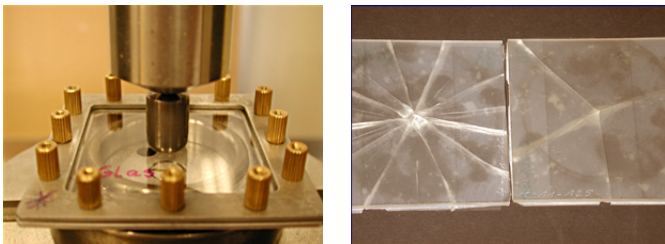


Figure 4: Left: Ring-on-ring setup with specimen. Right: Two specimens broken regularly within the load ring area at higher stress (left) and at lower stress (right).

Preparation of specimens must be conducted such to assure the absence of residual micro cracks in the surface to be investigated as a result of preceding grinding steps with coarser grains. As a rule the final layer should be ground off by a thickness at least 3 to 4 times that of the maximum micro crack depth of the preceding step. If the difference in grain size is large, an additional grinding process with intermediate grain size may become necessary.

Another test method used to determine breakage stress is the four-point bending setup with a homogeneous stress field between the load edges (Figure 5). For a detailed description refer to DIN EN 1288-3 [8]

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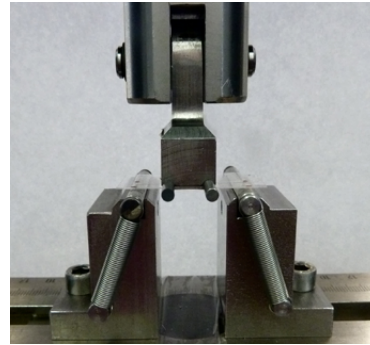
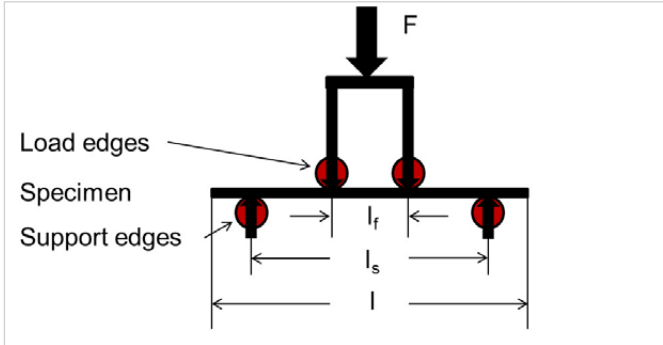


Figure 5:  
Four-point bending setup.

However, if surface breakage stresses are to be measured, special care must be taken to register breakage origins. The four-point bending setup is highly prone to starting

breakages from the edges, especially when they have been made introducing larger flaws e.g. with coarse cutting wheels see Figure 6 [9].

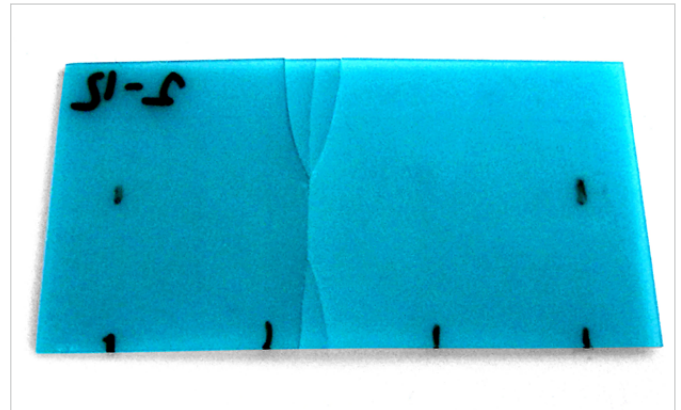
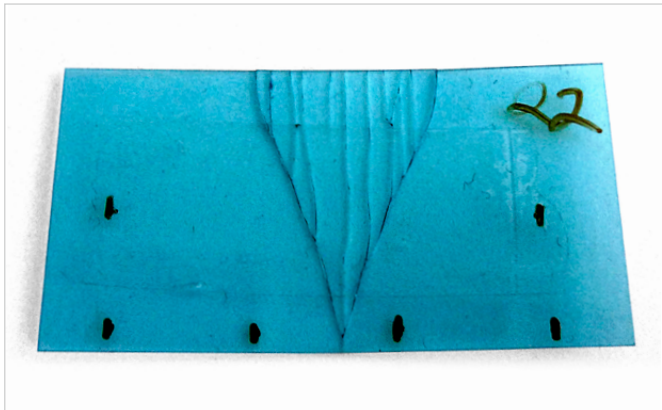


Figure 6: Left: Specimens with breakage origin at the edge. Right: With origin within the face. The origin location can be detected by the fact that bifurcations of cracks always lead away from the origin.

### 3. Evaluation of measurements – Weibull distribution

The most common way of evaluating and presenting the results of breakage measurement results employs the Weibull plot based on the Weibull statistical distribution [10, 11, 12, 13, 14].

The data obtained are sorted according to their size:  $\sigma_B(1) < \sigma_B(i-1) < \sigma_B(i) < \sigma_B(i+1) < \sigma_B(i=N)$  with  $N$  data in total. The relative nominal failure probability for each breakage value  $\sigma_B(i)$  is calculated according to the formula

$$F(\sigma_B(i)) = \frac{i-0.3}{N+0.4} \quad (1)$$

The y-value  $y_i$  in the Weibull plot is calculated from the failure probability  $F(\sigma_B(i))$  according to

$$y_i = \ln \left( \ln \left( \frac{1}{1-F(\sigma_B(i))} \right) \right) \quad (2)$$

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They are plotted against the x-value  $x_i = \sigma_B(i)$ , which also are represented on a logarithmic scale. For easier reading the scale of the y-axis is readjusted to render failure probability in percent. The advantage of this plot is that a set of specimens that exactly follows a two-parameter Weibull distribution lies on a straight line. The two-parameter (2P) Weibull distribution is given by Formula 3.

$$F(\sigma) = 1 - e^{-\left(\frac{\sigma}{\sigma_c}\right)^\lambda} \quad (3)$$

It is characterized by the parameter  $\sigma_c$ , called characteristic strength, which gives the breakage stress for failure probability of 63.2% and  $\lambda$ , the Weibull slope parameter, which is a measure of the distribution's width and of its slope in the Weibull plot.

### 4. Design strength calculation based on 2P Weibull distribution

Design strength is the minimum stress, which a designed structure shall withstand for its service life in a given environment with an allowable failure probability. Such design strength can be calculated on the basis of the parameters of the 2P Weibull distribution.

This distribution fits many data sets well, and it allows straightforward calculation of design strength [15]. The design strength  $\sigma_D$  is just the characteristic strength  $\sigma_c$  divided by a factor of safety FoS consisting of three factors  $f_A$ ,  $f_p$  and  $f_f$ .

$$\sigma_D = \frac{\sigma_c}{FoS} \quad (4) \quad FoS = f_A \cdot f_p \cdot f_f \quad (5)$$

The factor  $f_A$  is the scaling law for the area. It takes into account the difference of the tested area  $A_T$  and the practically loaded area  $A_D$ . The factor  $f_p$  yields the design strength reduction for failure probability specified different from 63.2% the definition value for  $\sigma_c$ .

$$f_A = \left(\frac{A_D}{A_T}\right)^{\frac{1}{\lambda}} \quad (6) \quad f_p = \frac{1}{\left(\ln\left(\frac{1}{1-F}\right)\right)^{\frac{1}{\lambda}}} \quad (7)$$

$f_f$  yields the reduction due to fatigue, which is characterized by the stress corrosion coefficient  $n$ .  $t_D$  denominates the glass structure's service lifetime and  $t_T$  the test duration. The index eff (effective) takes into account that stress loads may vary with time.

$$f_f = \left(\frac{t_{D,eff}}{t_{T,eff}}\right)^{\frac{1}{n}} \quad (8) \quad f_{f,constant\ load} = \left(\frac{t_D}{t_T}\right)^{\frac{1}{n+1}} \quad (9)$$

The design strength calculation based on the two parameter Weibull distribution has the disadvantage that for common practical cases, it leads to strength values so small that they reach the single digit MPa range. In the end such analysis leads to no benefit at all since one could use the established safe values of 4 – 6 MPa for glass and 10 MPa for glass ceramics anyway. Uncritical usage of this method usually leads to considerable underestimation of real strengths.

Moreover extrapolation to an allowable failure probability for high quality surfaces (polished or etched), with their typically low slope parameter  $\lambda$ , can lead to lower nominal strength values than for ground surfaces, which contradicts common sense and experience. Evidence has been found that the two parameter Weibull distribution does not correctly describe breakage stress distributions for homogeneously ground surfaces below failure probability of about 5% [16].

### 5. Three parameter (3P) Weibull distribution

Just recently it has been shown, that experiments with a sufficient number of specimens support the use of the three parameter Weibull distribution (Figure 7):

$$F(\sigma) = 1 - e^{-\left(\frac{\sigma-\gamma}{\eta}\right)^\beta} \quad (10)$$

The third parameter  $\lambda$  delivers a threshold breakage value which can be interpreted as a minimum strength  $\sigma_T$ . This observation fits well with the fact that grinding of brittle materials

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with grains restricted to an upper size limit cannot produce arbitrarily deep micro-cracks but cracks which do not exceed a limit given for the grinding process. The threshold value makes considerations obsolete referring to lower failure probability. Below the threshold it will remain zero for any stress value. Also scaling of strength with area is no longer necessary. It does not matter how large the area is if it is ground with the same given process. The maximum crack depth is independent of the area size. With the introduction of the three parameter Weibull distribution it is now possible to use considerably higher strength values. The long term strength change due to fatigue can also be taken into account [17, 18].

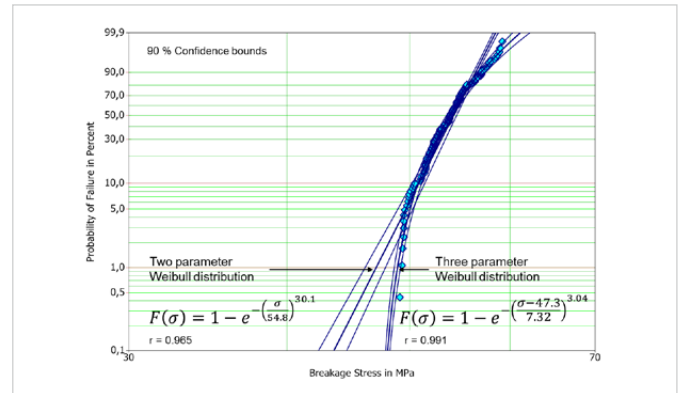


Figure 7: ZERODUR® ground with D151 diamond grains data set with 157 specimens 2P and 3P Weibull distribution fitted.

### 6. Bending strength of optical and filter glasses

The data for optical and filter glasses and also two technical glasses for comparison were acquired in a measurement campaign over the course of the 1980s until the beginning of the 1990s. The parameters determined for the 2P Weibull distribution have been published in the older versions of this document. All data sets have been reevaluated and fitted with Weibull distributions. A 3P Weibull distribution has been done where it could be satisfactorily fitted. This was possible in 15 of 25 cases. Figure 8 shows five selected data sets with fitted Weibull distributions. The y-axis scaling is according to Formula 2.

Table 1 compiles the measurement results: The parameters of the 2P Weibull distribution characteristic strength  $\sigma_c$  and slope  $\lambda$ , those of the 3P Weibull distribution scale parameter  $\eta$ , shape parameter  $\beta$  and minimum strength  $\sigma_T$ .  $n$  is an estimate value for the stress corrosion coefficient. For further information on this quantity see below. HK is the Knoop hardness according to the standard ISO 9385. The number of specimens used for the determination of the data is given as a measure for the statistical significance. For sets of 10 specimens and below 3P Weibull distributions were not fitted, because this number is considered to be too small.

The breakage test procedure was the double ring method according to DIN 52292-1 (now: European standard EN 1288-5) R 30-6 which means 60 mm support ring diameter and 12 mm load ring diameter. This corresponds to an area of 113 mm<sup>2</sup> loaded with homogeneous isotropic tensile stress. Stress increasing rate was 2 MPa/s and the environment room climate with 50% relative humidity.

The strength measurements have been done prior to the optical glass portfolio change in 1998, which was done to replace lead and arsenic containing glass types. Therefore none of the new N-glass types have been measured directly. However, there were many glass types with very minute changes. Only few tenths of a percent of arsenic oxide have been replaced by antimony oxide. Such composition change can be completely disregarded from breakage stress aspects. For these glass types the new glass names are used e.g. SCHOTT N-BK7® in the table whereas in the diagram of Figures 8 and 9 the original glass names are given. Please note that the glass type SFL6 changed to N-SF6. This was already a lead free glass type and can be seen as a representative of the present set of lead free flint glass types.

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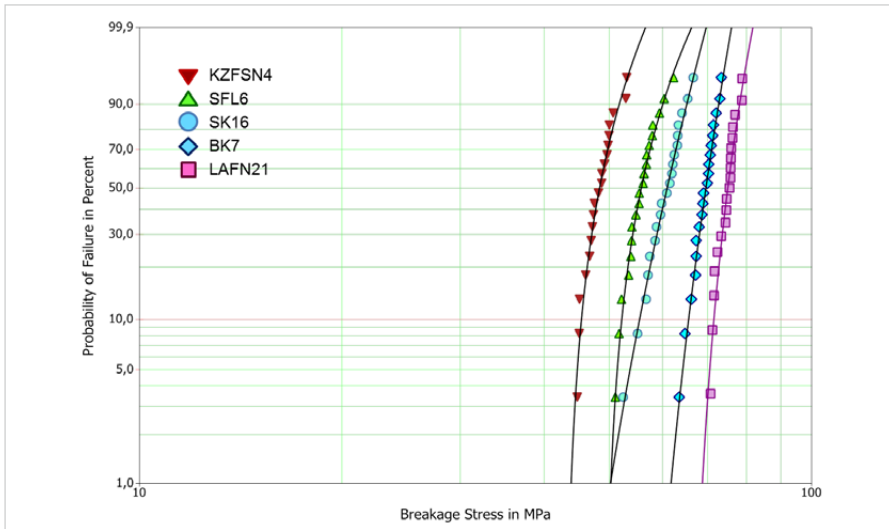


Figure 8: Breakage stress data sets for some optical glasses lapped with silicon carbide fine grain SiC600 with maximum grain size of 19  $\mu\text{m}$ . The lines represent three parameter Weibull distributions fitted to the data sets.

Figure 9 gives three data sets of the glass type BK7 demonstrating the strength increase by going to smaller grains from D64 (bonded diamond grains with 64  $\mu\text{m}$  maximum grain size) to SiC600 (lapping grains with 19  $\mu\text{m}$  maximum grain

size) and then to etched specimens, which lie significantly higher. All three data sets are well represented by a three parameter Weibull distribution. The fit line and the 90% confidence bounds are also plotted.

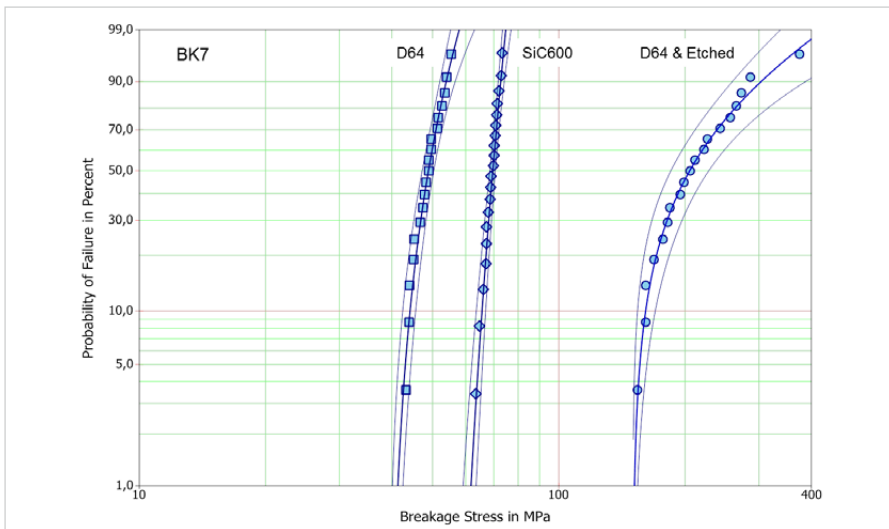


Figure 9: Breakage stress data sets for the optical glass BK7 with three different surface conditions: Ground with bonded diamond grain D64 with maximum grain size of 64  $\mu\text{m}$ , lapped with silicon carbide fine grain SiC600 with 19  $\mu\text{m}$  maximum grain size and D64 ground and subsequently etched. Three parameter Weibull distribution fitted and given with 90% confidence bounds.

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Glass Type	Surface condition	2P $\sigma_c$ [MPa]	2P $\lambda$	3P $\eta$ [MPa]	3P $\beta$	3P $\sigma_T$ [MPa]	n	HK	Sample Size
SCHOTT N-BK7®	SiC 600	70.6	30.4	20.00	7.97	50.6	19.5	610	20
SCHOTT N-BK7®	D 64	50.3	13.3	11.20	2.93	39.0	19.5	610	19
SCHOTT N-BK7®	D 64 etch	234.7	4.1	20.00	7.97	150.1	19.5	610	19
FK52	SiC 600	32.4	6.8	–	–	–	–	400	20
FK52	D 64	27.2	13.8	–	–	–	–	400	20
PSK53A	SiC 600	43.0	14.4	–	–	–	13.6	440	9
N-ZK7	SiC 600	68.9	14.1	–	–	–	26.3	530	10
N-BAK1	SiC 600	62.3	8.2	–	–	–	18.2	530	10
N-SK16	SiC 600	62.2	19.6	27.30	7.96	34.9	21.6	600	20
N-LAK8	SiC 600	70.0	29.9	11.40	4.25	58.4	23.4	740	20
N-LAK9	SiC 600	68.6	9.3	–	–	–	21.6	700	10
LAK10	SiC 600	79.2	7.5	–	–	–	23.2	720	10
LLF2	SiC 600	64.7	18.4	–	–	–	17.5	460	20
F2	SiC 600	57.1	25.0	–	–	–	16.7	420	20
N-BASF64	SiC 600	70.1	23.9	9.87	2.68	59.9	19.0	650	20
LAF2	SiC 600	56.5	20.0	13.00	3.91	43.3	16.9	560	20
N-LAF21	SiC 600	75.9	28.6	11.30	4.63	64.6	22.4	780	20
LASF8	SiC 600	52.0	13.1	–	–	–	22.7	–	10
LASFN30	SiC 600	79.5	24.8	–	–	–	21.9	770	20
SF5	SiC 600	55.3	10.6	–	–	–	16.7	410	10
SF6	SiC 600	49.2	5.4	–	–	–	16.9	370	10
N-SF6*	SiC 600	57.3	21.9	7.81	2.44	49.1	14.6	570	20
SF57	SiC 600	39.1	14.7	–	–	–	16.4	350	10
SF58	SiC 600	32.8	19.5	18.50	10.70	14.3	14.6	320	20
N-SF66	SiC 600	47.6	29.1	8.05	4.01	39.4	22.7	440	38
N-SF66	D151	37.5	4.8	21.95	2.29	15.1	22.7	440	47
N-SF66	Opt. Polish	142.9	7.6	–	–	–	22.7	440	22
KZFSN4	SiC 600	49.4	25.5	6.37	2.51	42.8	26.3	450	20
IRG11A	D64	73.9	9.3	2.33	20.5	53.0	16.7	700	23
IRG11A	Opt. Polish	707.6	2.5	0.91	352.9	323.2	16.7	700	18
IRG25	SiC 600	14.2	16.1	–	–	–	–	–	8
IRG25	SiC 500	14.6	9.1	–	–	–	–	–	10
IRG25	Opt. Polish	17.6	6.0	–	–	–	–	–	10
UG11	SiC 600	63.4	4.4	12.20	3.13	49.1	17.7	–	18
KG 3	SiC 600	61.4	11.3	9.98	1.85	51.8	24.2	–	13
BG60	SiC 500	33.0	25.8	–	–	–	–	360	53
BG60HS	SiC 500	32.5	17.8	–	–	–	–	360	56
S8612	SiC 500	54.2	20.9	–	–	–	–	–	50
Duran®	SiC 600	75.9	19.2	8.57	1.39	66.4	30 [30]	–	16
Float	SiC 600	81.0	35.2	6.61	1.96	74.0	18.1 [29]	–	19

Table 1: 2P and 3P Weibull parameters, estimated stress corrosion constants and Knoop hardness for optical glasses. All glass types where the change to lead and arsenic free versions needed only very small changes in composition are denoted with the leading N in order to indicate that the strength values are also valid for these versions. \*Originally measured as SFL6 which has a composition very similar to N-SF6. All data are representative for surfaces only without any additional larger scratches. All n-values are calculated from formula 14 except for Duran® and float glass where measured values were taken from given references.

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### 7. Bending strength of ZERODUR®

The bending strength of the low expansion glass ceramic ZERODUR® has been studied in much more detail than that of optical glasses because mirrors and frames made from this material are more commonly subjected to mechanical loads. Mirrors of 8 meter diameter and less than 200 mm thickness are much more delicate than 4 m mirrors with 600 mm thickness, which represented the beginning of large ZERODUR® mirrors. Lightweight mirrors for space telescopes must survive launch vibrations, wobbling lightweight secondary mirrors have to endure vibrations through their entire lifetime, support frames for reticles and wafers in microlithography devices must endure strong accelerations in the stepping and scanning processes optimized for shortest exposure times. Data sets obtained in the 1980s – 1990s mostly consisted of not more than 20 specimens each. This is not enough to support calculations of failure probability as low as one per mille and below with sufficient credibility. Therefore new measurements have been performed since 2007 with the aim of obtaining sets with much higher specimen numbers. This has been achieved

by producing the specimens together in one process from large disks or plates rendering 100 to 150 pieces per set [5, 6, 16, 17, 18, 19, 20].

Figure 10 shows these data sets together with two older ones, which already consisted of much more than 20 specimens. All of them represent surfaces ground with bonded diamond grains or lapped with SiC grains and are fitted with a three parameter Weibull distribution. The D151, D25, SiC 320 and SiC600 sets fit almost perfectly to the three parameter Weibull distribution, the D64 set drops even steeper at the lower end and the D151 ZERODUR® K20 set is equally well represented by a two parameter Weibull distribution. This fully ceramized version of ZERODUR® shows about ten percent higher strength than ZERODUR® because of the much larger microcrystals, which occupy ca. 99 % of the material's volume.

It is remarkable that the sets of D25 and SiC600 lie very close to each other. Both are characterized by maximum grain sizes around 20 µm.

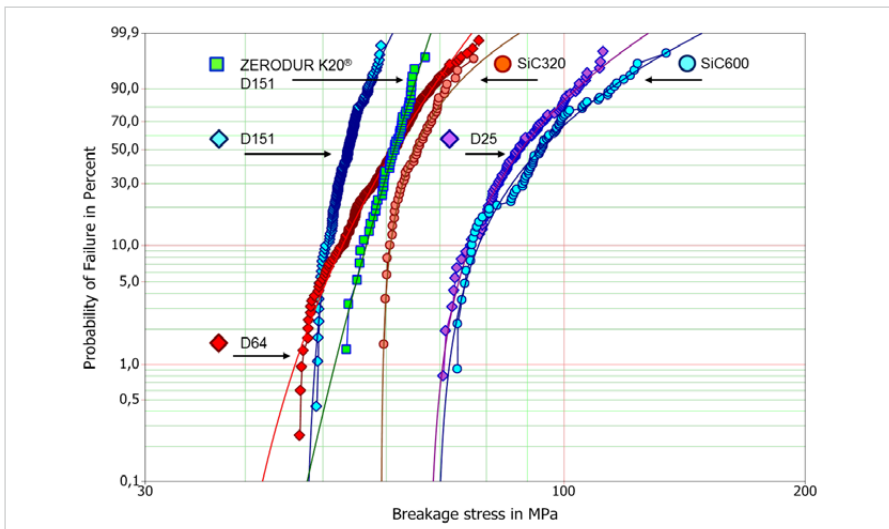


Figure 10: Breakage stress data sets for ZERODUR® with specimens number of about 50 and more. Surfaces are ground with bonded diamond grains (D) or lapped with SiC grains.

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Figure 11 shows data sets for ZERODUR® prepared for achieving higher strength [21, 22]. For comparison the D151 ground data set is also given. A set of specimens etched to remove a shallow layer of 34 µm lies almost at the same position but with a slightly more inclined slope. In the following layer thickness etched off is denoted in microns with a leading “E” as for example D151E34.

The set of specimens ground with D151 and etched afterwards removing 83 µm layer thickness lies much higher. The width of its distribution is much broader ranging from 124.7 MPa to 405.8 MPa (D151E83: 38.3 – 71.0 MPa). This is typical for all high strength data sets as can be seen from the other distributions in the diagram. Most of the sets with etched specimens originally comprised about

150 pieces. With high strength distributions it is also common that many specimens break outside the load ring and therefore are discarded from evaluation.

Etching does not improve strength in any case. Obviously a minimum layer thickness must be removed to achieve much higher breakage stresses. Etching deeper does not really improve strength further. The distributions become broader with many specimens breaking at stresses even higher than 300 MPa. However, the weakest specimens remain in the 130 to 170 MPa range for all data sets. Interestingly the distribution of optically polished specimens lies in the same range as the sets with the minimum sufficient thickness removed: D64E73 and D151E83.

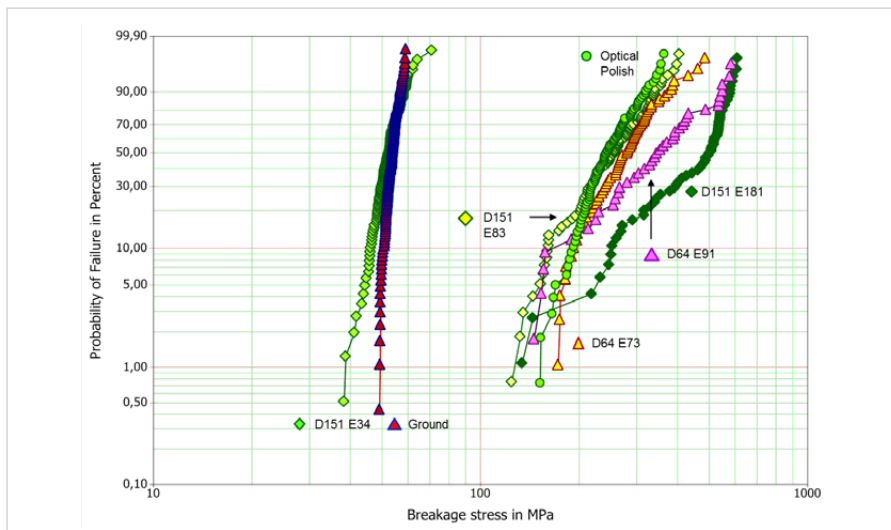


Figure 11: Breakage stress data sets for ZERODUR® first ground and then etched surfaces and for optically polished surfaces. The first part of the set denominations gives the tool grain of the pregrinding step and the second part the layer thickness etched off in micrometer. All etching was done using the SCHOTT standard acid recipe [22]. For comparison the set of only D151 ground ZERODUR® is also plotted.

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Table 2 gives the Weibull parameters of all ZERODUR® data sets including the older ones.

Sample	N	N <sub>v</sub>	2P $\sigma_c$	2P $\lambda$	3P $\beta$	3P $\eta$	3P $\sigma_T$	Min	Max
D15A	20	19	132.1	11.1	–	–	–	100.8	155.3
D25	91	86	93.2	11.5	2.16	24.5	67.7	70.7	112.0
D35	20	20	80.2	16.7	–	–	–	67.2	90.5
D64	281	278	63.4	12.7	4.93	28.1	35.1	46.8	78.4
D151	157	157	54.8	30.1	3.04	7.32	47.3	49.1	59.2
D251	20	20	49.8	11.2	–	–	–	36.5	55.0
SiC600	78	75	99.7	9.4	1.91	28.8	69.3	73.7	134.3
SiC320	50	46	67.1	22.0	1.42	7.00	59.4	59.9	76.0
SiC230	20	20	58.7	16.4	6.22	24.9	33.7	48.2	66.0
SiC100	12	12	55.1	19.2	–	–	–	47.2	58.4
D151 E Std 34	148	134	54.4	13.4	6.15	28.0	26.4	38.3	71.0
D151 E Std 83	148	91	280.1	4.55	2.81	199.2	79.9	124.7	405.8
D151 E Std Mod 140	271	96	163.2	4.26	1.65	85.0	73.8	82.6	299.4
D151 E Std 181	148	63	499.3	3.65	n.a.	n.a.	n.a.	134.2	610.6
D151 E Polish 485	50	15	722.7	1.80	n.a.	n.a.	n.a.	117.0	994.2
D64 E Std 73	149	65	304.3	5.28	2.25	161.4	138.5	172.9	486.5
D64 E Std 91	109	39	394.6	3.15	2.55	348.9	45.5	145.9	584.7
Optical Polish	94	93	267.8	6.5	2.45	130.7	134.6	152.3	364.4
D151 ZERODUR® K20	53	52	62.4	23.7	11.89	33.1	29.3	53.5	67.2
SiC 600 at 77K	–	–	192.7	10.7	–	–	–	–	–
Glassy SiC 320	20	20	63.1	17.7	–	–	–	51.92	68.63

- N Total number of specimens
- N<sub>v</sub> Number of valid specimens regularly broken
- 2P  $\sigma_c$  Characteristic Strength in MPa
- 2P  $\lambda$  Weibull slope parameter
- 3P  $\beta$  3P Weibull shape parameter
- 3P  $\eta$  3P Weibull scale parameter in MPa
- 3P  $\sigma_T$  3P Weibull location parameter (threshold stress) in MPa
- Min Minimum value in MPa
- Max Maximum value in MPa

**Table 2:** Weibull parameters of ZERODUR® samples with different surface preparation. If possible the three parameter Weibull distribution was fitted. Small differences between total and valid number of specimens result from the elimination of outliers. Large differences typical for high strength etched sets come from the removal of specimens broken outside the load ring. E Std means etched with SCHOTT standard acid recipe, E Std mod stands for a modified standard recipe, E Polish for an etching process used for technical glass surface polishing with a very thick layer removed.

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### 8. Strength change with time

#### 8.1 Stress intensity factor and fracture toughness

A brittle material will break if two conditions are fulfilled at the same time. First there must be tensile stress of sufficient height at the surface where secondly a flaw with a minimum size is present. The simultaneous occurrence and the values of both quantities are characterized by the stress intensity factor  $K$ . In glass and glass ceramics only one directional mode of  $K$  is present – the crack opening mode or tensile mode. There are no shear modes. Traditionally opening mode stress intensity factor is denoted as  $K_I$  (pronounce: K one) and calculated according to Formula 11

$$K_I = \sigma \sqrt{af} \quad (11)$$

with  $\sigma$  being the stress at the crack tip,  $a$  the crack length and  $f$  a geometrical factor that is dependent on the shape of the crack [1, 2, 4]. For glass and glass ceramics it is very close to 2. Fracture will occur if  $K_I$  reaches a critical value  $K_{IC}$ .

$$K_I \geq K_{IC} \quad (12)$$

$K_{IC}$  is called the fracture toughness for fracture mode I. It is a material constant. For glasses without additional strengthening by thermal or chemical pre-stressing the value is typically smaller than  $1 \text{ MPa m}^{1/2}$ . Table 3 gives fracture toughness values of some glasses:

Glass	$K_{IC}$ [MPa m <sup>1/2</sup> ]	Glass	$K_{IC}$ [MPa m <sup>1/2</sup> ]	Glass	$K_{IC}$ [MPa m <sup>1/2</sup> ]
FK6*	0.64	N-BAK4	0.98	F2	0.70
FK52*	0.51	N-SK16	0.89	SF1	0.77
SCHOTT N-BK7®	1.08	BAF4*	0.86	SF6	0.74
K5	0.86	LAK10*	1.08	SF58*	0.56
N-K50*	0.77	LASF12*	1.04	Duran 50	0.85
N-ZK7	1.03	LLF1	0.70	ZERODUR®	0.91 / 1.12 / 1.39

Table 3: Fracture toughness of some glasses. All glass types where the change to lead and arsenic free versions needed only very small changes in composition are denoted with the leading N in order to indicate that the strength values are also valid for these versions. The star\* denotes obsolete glass types given for reference. For ZERODUR®  $K_{IC}$  was determined with three different methods [23, 24].

A simple application of formulas 11 and 12, however, is not possible. In practice the length of a flaw cannot be determined precisely enough because it must be observed in its extension for cleavages down to atomic dimensions. On the other hand  $\sigma$  is not just the long range surface tensile stress. Local conditions such as the presence of other flaws in direct vicinity or eigenstress in isolated flaws makes things much more complicated [4]. Moreover for long term applications one must avoid the critical stress concentration factor considerably anyway.

#### 8.2 Strength change with time – Fatigue

The underlying process of brittle materials' fatigue is the growth of existing micro cracks. Such growth happens only if tensile stress surpasses a threshold value at the crack tips. So if there is no load on the glass or glass ceramic item there will be also no fatigue.

Over a wide range of many orders of magnitude crack growth velocity follows the Paris law (Formula 13). The exponent  $n$  to the stress intensity factor  $K_I$  is called stress corrosion constant. It depends on the environmental medium with humidity being the most important influence with glass and glass ceramics.

$$\frac{da}{dt} = AK_I^n \quad (13)$$

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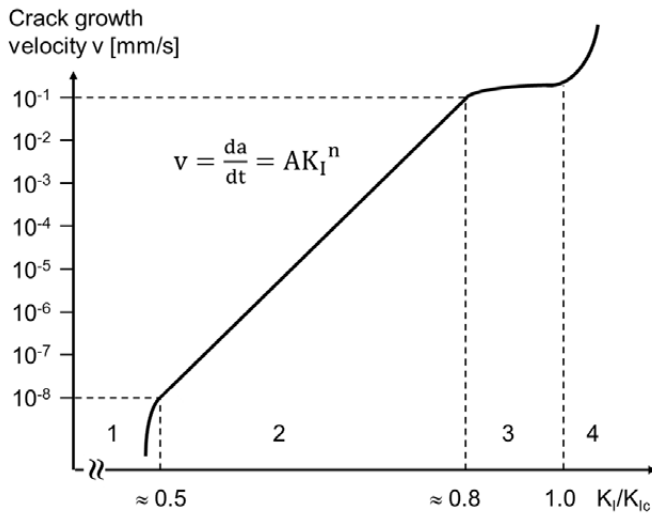


Figure 12: Micro crack growth velocity plotted against the stress intensity factor related to the fracture toughness, i. e. the stress intensity factor, where fast unstoppable breakage starts.

Figure 12 shows micro crack growth depending on the stress intensity factor  $K_I$  related to the fracture toughness  $K_{Ic}$ , i. e. the stress intensity factor, where fast unstoppable breakage starts. In region 1 crack growth if existing at all is so slow, that it can be neglected. In region 2 crack growth velocity follows Paris' law with the slope being given by the exponent  $n$  which is called stress corrosion constant. It depends on the environmental medium and its reaction rate with the glass breaking the atomic bonds at the crack tip. In region 3 the crack propagates faster than the environmental medium can follow. Therefore the medium cannot enhance crack growth any further. In region 4 the critical stress intensity factor for breakage is surpassed and the crack will grow with high speed leading to macroscopic breakage.

Water is an unfavorable environmental medium since it supports the breakage of stressed Si-O-Si bonds and since it is ubiquitous in form of vapor. Dry air and vacuum are preferable environments from fatigue point of view [25, 26].

### 8.3 Stress corrosion constant $n$

There are several methods for determining the stress corrosion coefficient  $n$ . With the double torsion beam method a single crack is directly observed while growing with increasing stress. The double cleavage drilled compression method (DCDC) measures crack growth starting from a hole drilled into the specimen. The third method uses the ring-on-ring test setup as described above. Several sets of specimens are broken each with different stress load increase rates. From the shift of the sets' medians with stress load increase rates one can calculate the stress corrosion constant  $n$ .

As a means for estimating  $n$  if measurement results are not available one can use the empirically derived formula after R.R. Tummala [15, 27]:

$$n = 38 - 2.6 \cdot CTE \quad (14)$$

with CTE being the coefficient of thermal expansion. Table 1 gives such estimation values for optical glasses since there have been no direct measurements of  $n$  for these glasses. For glass types with extreme CTE (FK52 and IRG24)  $n$  values according to this formula make no sense and therefore are omitted.

Table 4 comprises available stress corrosion constant values for ZERODUR®. With the latest determination performed in 2014 differences observed in the past have been removed considerably. Especially with normal room humidity the results of three independent measurement campaigns now match perfectly. The results from 2014 are based on variation of the stress load rate by 4 orders of magnitude and confirm those derived in 2010 with a variation of less than 2 orders of magnitude and those of the DCDC method. Therefore the value of 31 is well established. With dry environment fatigue is very low [16, 20, 24]. This is underpinned by the very high value of 79 for  $n$  for extremely dry nitrogen gas [28].

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## TIE-33 Bending strength of optical glass and ZERODUR®

Humidity	Method				
	Ground samples ring-on-ring setup load rate variation	Ground samples ring-on-ring setup load rate variation	Double cleavage drilled compression DCDC	Single crack propagation observation	Crack indentation
Dry	79.1 (Dry N <sub>2</sub> )	–	50 ± 3 (3%)	–	–
Normal	31.1 (50%)	29.3 +3.6/–2.9 (50%)	31 ± 3 (37%)	51.7 (50%)	59.3 (50%)
Humid	–	–	–	–	30.7 (100%)
Year	2014	2010	2008	1989	1990

Table 4: Stress corrosion constant n of the glass ceramic ZERODUR®. Relative humidity is given in parentheses.

### 9. Design strength for ZERODUR® ground surfaces including fatigue

In order to calculate the total crack growth under tensile stress  $\sigma$  for a given loading time  $t_b$  it is necessary to integrate the Paris law [1, 18, 29]

$$da = A(\sigma\sqrt{af})^n dt \quad (15)$$

$$\int_{t_i}^{t_B} dt = \int_{a_i}^{a_B} \frac{1}{A(\sigma\sqrt{af})^n} da \quad (16)$$

Such integrations for the special cases of constant stress and of constantly increasing stress, if combined, render useful formulas for either allowable stress  $\sigma_{B,c}$  for a specified lifetime or minimum lifetime  $t_{B,c}$  for a specified stress.

$$\sigma_{B,c} = \left[ \frac{\sigma_{B,r}^{n+1}}{t_{B,c}} \frac{1}{(n+1)\dot{\sigma}_r} \right]^{\frac{1}{n}} \quad (17)$$

$$t_{B,c} = \frac{\sigma_{B,r}^{n+1}}{\sigma_{B,c}^n} \frac{1}{(n+1)\dot{\sigma}_r} \quad (18)$$

With

- $\sigma_{B,r}$  – breakage stress measured with constant stress increase experiment
- $\dot{\sigma}_r$  – experimental stress increase rate
- n – stress corrosion coefficient of the given material environment combination

Inserting the 3P Weibull threshold stress  $\sigma_T$  as measured breakage stress value  $\sigma_{B,r}$  in these formulas allows calculating the design stress or design lifetime including the fatigue effect. These formulas do not necessarily hold for isolated cracks in otherwise polished surfaces due to eigenstress in such cracks. However, for ground and lapped surfaces such eigenstress is expected to be different because of the many neighbouring micro cracks. The formulas should work best with stress corrosion coefficient determined with ground surfaces, especially the different loading rates method. The breakage stress values and stress increase rate can be determined with sufficient accuracy. Inserting  $\sigma_T$  for  $\sigma_{B,r}$  and stress corrosion constant  $n = 31$  for normal humidity and 79 for dry environment (vacuum) results in the following diagrams in Figures 13 and 14 for the minimum lifetime under constant tensile stress.

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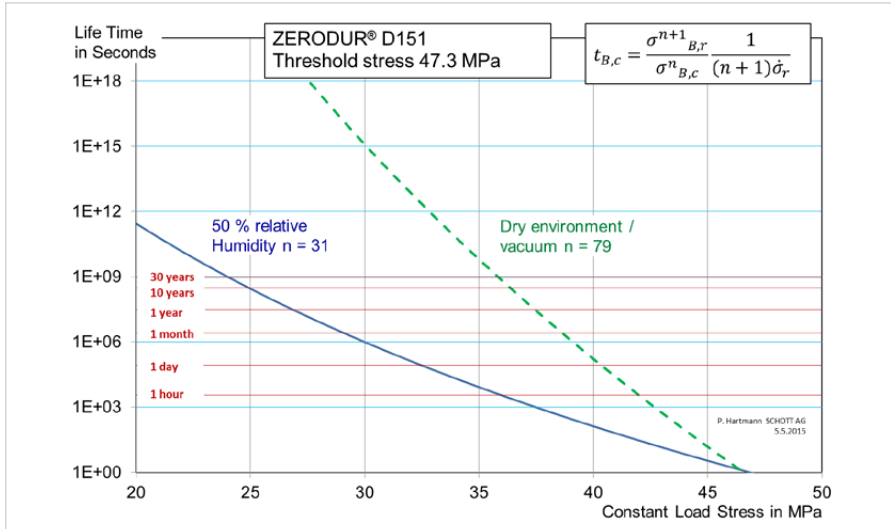


Figure 13: ZERODUR® D151 ground: Lifetime as a function of constant tensile stress load.

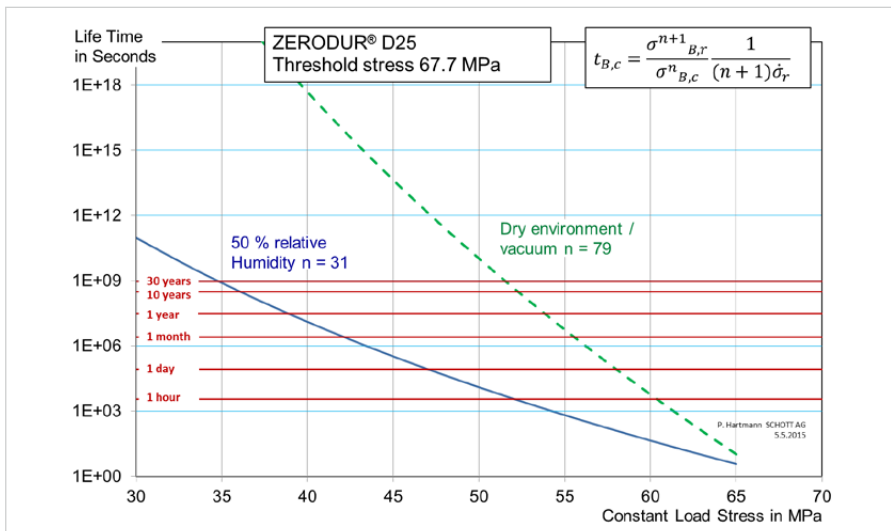


Figure 14: ZERODUR® D25 ground: Lifetime as a function of constant tensile stress load.

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## TIE-33 Bending strength of optical glass and ZERODUR®

### 10. Some recommendations for optimizing and maintaining the strength of glass items

#### 10.1 Risk considerations

- Acceptable failure probability varies considerably. A broken household drinking glass will just be thrown away and replaced. Functionally relevant glass items of satellites and safety relevant glass windows of space vehicles represent the opposite case, where failure is no option. Most technical applications lie somewhere in between. Before designing a mechanically loaded glass item some thoughts should be made about possible risks during the different stages of the item's life-time production, storage, assembly, operation and disposal:
  - the value of the item
  - the possibility to replace it
  - possible consequences of damages and breakage to facilities and to human beings
  - the possibility to repair it
- In case of very low acceptable failure probability there are additional methods to improve safety e.g.:
  - Failure tolerant design
  - Proof test of glass components

#### 10.2 Design of a glass item

- Avoid stress concentration elements such as sharp corners and edges. If possible apply radii of several millimeters at such elements.
- If bore holes are needed the distance between the edges of two neighboring holes should be at least half of the glass part's thickness.
- Highly loaded areas should be polished or etched. Start this from surface conditions containing micro cracks not deeper than the thickness to be polished or etched off.  
If necessary an additional fine grain grinding step should be introduced removing a layer at least three times thicker than the maximum crack depth of the preceding grinding process. Strength improving etching requires the application of a strong acid capable of removing a layer more than 80 µm thick. Insufficient etching will not increase strength at all. Thermal and chemical pre-stressing as applied for technical glass items for increasing strength is no option for optical

glasses and glass-ceramics, since important properties are changed such as refractive index, homogeneity and birefringence.

#### 10.3 Design of frames or support structures

- Locate higher tensile stress loaded surfaces and weaker parts of the item (ground surfaces) where they are not accessible in application e.g. under covers or within frames.
- Avoid point like forces on the glass item. Support forces should be distributed over a large area. Use compliant materials between metal fixtures and glass surfaces with sufficient thickness to compensate flatness deviations of the metal fixtures. During fixing of frames apply forces step by step and evenly distributed avoiding bending forces due to wedge angles.
- Do not use glues for joining or fixing without careful considerations. Bonding of brittle materials is generally possible but has to be done carefully as a rule as a dedicated development. Especially epoxy resins can cause breakage due to the high stresses they develop while hardening. Even fixing of small parts exerting no forces at first sight can lead to chipping off.
- If thermal shocks are to be expected in application of glasses with normal coefficient of thermal expansion provide measures to damp such shocks before they reach the glass items e.g. by layers of thermally insulating materials.

#### 10.4 Maintain strength

- If glass items need to be stored before use with mechanical loading this is no problem as long as they are not subject to continuous loads. If such loads coming from bonding or frames are high enough that strength fatigue cannot be excluded it will be useful to pack them hermetically in a plastic bag e.g. applying a drying agent in the package.

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- If cleaning is necessary procedures should use liquids in abundance and soft cloths. Avoid rubbing. Check the glass part for scratches. Scratches reduce the strength significantly!
- In contrast to metals vibration loads do not lead to stronger fatigue than constant loads of the same height as the peak vibration stresses. Micro cracks can grow only in the high tensile stress parts of the cycles.

### 10.5 Repair

- Glass items might be damaged by edges chipping off of cracks caused by impacts. Such pieces may be repaired restoring strength if replacement is too much effort. Dressing out a chip or flaw is possible with a grinding tool. Its diamond grains should be equal or smaller than those of the tool by which the original surface had been prepared. Dressing out cracks must be done carefully to avoid enlarging the crack due to the applied working force. Remaining sharp edges should be chamfered.

## 10. Appendix: Grain sizes of commonly used diamond tools and of silicon carbide lapping agents

	Designation	Mean size [ $\mu\text{m}$ ]	Max. size [ $\mu\text{m}$ ]	ASTM equiv.
<b>Bonded diamond grains acc. ISO 6106</b>	D251	231	250	60/70
	D151	138	150	100/120
	D107	98	106	140/170
Micro grit sizes are not standardized but supplier dependent	D64	58	63	230/270
	D35 micro grit	36	40	–
	D25 micro grit	30	40	–
	D15A micro grit	12.5	15	–
<b>Loose silicon-carbide grains acc. FEPA Std. 42-D-1984</b>	SiC 100	129	150	–
	SiC 230	53	84	–
	SiC 320	29	49	–
	SiC 500	12.8	25	–
	SiC 600	9	19	–

Table 5: Grain sizes of grinding tools [31, 32].

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