ZERODUR® Glass Ceramics for High Stress Applications

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ABSTRACT

Recently SCHOTT has shown in a series of investigations the suitability of the zero expansion glass ceramic material ZERODUR® for applications like mirrors and support structures of complicated design used at high mechanical loads. Examples are vibrations during rocket launches, bonded elements to support single mirrors or mirrors of a large array, or controlled deformations for optical image correction, i.e. adaptive mirrors.
Additional measurements have been performed on the behavior of ZERODUR® with respect to the etching process, which is capable of increasing strength significantly. It has been determined, which minimum layer thickness has to be removed in order to achieve the strength increase reliably.
New data for the strength of the material variant ZERODUR K20® prepared with a diamond grain tool D151 are available and compared with the data of ZERODUR® specimens prepared in the same way. Data for the stress corrosion coefficient n of ZERODUR® for dry and normal humid environment have been measured already in the 1980s. It has been remeasured with the alternative double cleavage drilled compression (DCDC) method.

Keywords: ZERODUR®, ZERODUR K20®, bending strength, mechanical stress, high loads, Weibull distribution, failure probability, stress corrosion

1. INTRODUCTION

In the last two years SCHOTT performed a series of measurements on the strength of ZERODUR®[1,2]. The main goals were to improve the statistical significance of the characteristic data by using larger sets of specimens per measurement run, which will be called a sample in the following, and to demonstrate that ZERODUR® can endure much higher tensile stress loads than 10 MPa[3]. This value had commonly been used in the past as a very conservative safe design value. We investigated the strength of frequently used surface conditions ground with bonded diamond grains with size distributions specified as D64 and D151 and surfaces subsequently etched for further strength enhancement.

Since long times, surfaces ground with fine grain tools like D64 or etched surfaces have been used for high strength applications of ZERODUR® components.

- D64 is used for surfaces to be processed on by polishing or for non-optical surfaces where somewhat higher strengths are deemed to be necessary.
- Etching is applied
  - for the relief of grinding induced stresses, as can occur with light-weighting by core grinding in the corners of blind holes with small angles,
  - for additional light-weighting with higher material removal from back structure ribs by longer etching processes[4]
  - or to achieve highest possible strength for critical ZERODUR® components.

This publication adds new data about etched D151 surfaces, strength data for the ZERODUR® variant material ZERODUR K20® ground with D151 grain and new data for the stress corrosion coefficient information, obtained with the double cleavage drilled compression (DCDC) method.

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2. EXPERIMENTS

2.1 Investigated surface condition

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[5].

D151 stands for a range of grain sizes limited by two sieves with mesh width of 125 and 150 µm according to the international standard ISO 6106[6]. This corresponds to US mesh number 100/120. Such tools are widely used for efficient grinding and represent a very common surface condition.

For finer grinding tools with D64 grains are often used. It specifies a range of 53 to 63 µm (US mesh number 230/270).

Etching has been done according to the procedure, which is commonly used at SCHOTT and described by R. Jedamzik[7]. Each sample has been etched in batches of 12 specimens. With measurements of the specimens' thickness changes, the variation of the thickness of the layers removed from one side were determined to better than 2 µm (1 standard deviation) within an etching batch and better than 5 µm (1 s) within a total sample.

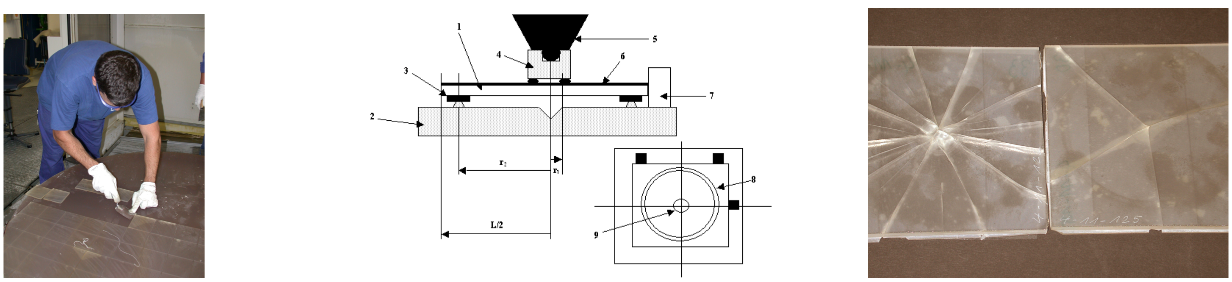

Fig. 1.: Left: Taking single ZERODUR® specimens, Middle: Ring-on-Ring test setup, Right: Broken specimens

2.2 Specimen preparation

As in the preceding measurement series the 148 D151 ground and etched specimens with size 100 mm x 100 mm x 6 mm have been produced from a 1.5 m diameter ZERODUR® disk assuring that the surface under investigation is in a representative condition without residual cracks from preceding processes. The procedure is described in more detail in Hartmann et al[1]. The 60 specimens for the ZERODUR K20® have been cut from a single block.

2.3 Test setup

The breakage tests have been performed according to the ring on ring method described in the European standard EN 1288-5[8] with an R 45 adapter (Fig. 1). The square specimen lies on the support ring of radius 45 mm and is bent by a force applied through the load ring with radius 9 mm. A more detailed description is given by Hartmann et al.[2]. The load increase rate applied is 2 MPa/s for all samples. Breakage origins have been recorded. Only specimens, which broke within or at the load ring have been used, according to the requirements of European standard EN 1288-5.

3. RESULTS AND DISCUSSION

3.1 Tests performed and observations

Together with the new measurements in total more than 850 specimens are tested now. 595 of them could be evaluated, since they fulfilled the condition of breakage within or at the load ring. More than 200 out of the 850 broke outside of the load ring; this comes by far mostly from the etched specimens, which is typical in such tests. For such samples the probability of failure outside the loading ring is higher. In fact, the strength and the dispersion are so high that the high load within the loading ring is most of the times not sufficient to break the specimen, and the effect of the larger area outside the loading ring prevails. This explains the large amount of specimens to be discarded.

Other specimens have been used for calibration of the test setup and for plausibility tests.

Since important observations can be seen best in direct comparison the new data are given together with those, which have been published previously. In order to ease comparisons the sample numbers as used in Hartmann et al.[1] have been maintained. The characteristic values of all samples are given in table 1, see below.

3.2 Ground surface condition

Figure 2 shows all samples, which are representative for ground surfaces: ZERODUR® ground with D151 and D64 and ZERODUR K20® ground with D151. Sample # 1 comes from the strength measurements done on the occasion of the AXAF / CHANDRA project of NASA, where SCHOTT delivered 24 ZERODUR® hollow cylinders in 1989 to 1991. It was combined from 9 subsets of D64 ground specimens all measured with 2 MPa/s load increase rate. Sample # 2 consisting also of D64 ground specimens has been measured in 2008. We think that the reproducibility of the strength distributions of D64 surfaces is remarkably good. Samples # 3 and # 4 both consist of D151 ground specimens taken from two different disks of the same cast blank. The results are in such perfect agreement, that both samples will be treated as one combined sample in the following, namely as sample # 5. Sample # 11 is the new measurement of ZERODUR K20® D151 ground.

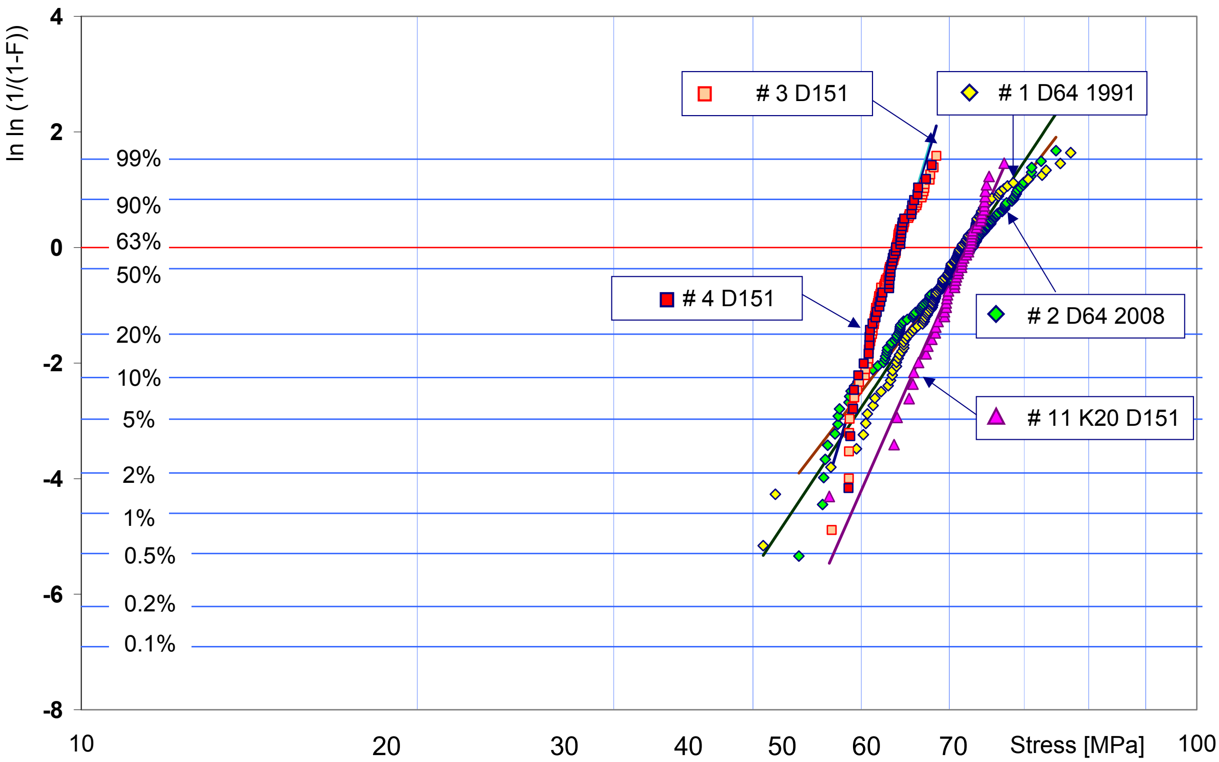

Fig. 2.: Weibull plot: Failure probability per breakage stress given in percent for all ZERODUR® samples with ground surface condition. Sample # 1: D64 data obtained in 1989 to 1991 with the AXAF / CHANDRA project, sample # 2 D64 data obtained in 2008, sample # 3 and # 4 D151 and sample # 11 ZERODUR K20® D151

As expected for ground surfaces all distributions are narrow with steep slopes. The samples follow the Weibull distribution satisfactorily with some obvious deviations mostly in the failure probability ranges above 90% and below 5%. There is a tendency that in both ranges the strength values are somewhat higher than expected on the basis of the Weibull distribution.

The D64 samples show higher strength values only for failure probabilities higher than 10%, which sheds some doubt on the practice in the past to grind surfaces expected to endure elevated stress with finer grains. This rule may not be false for other grains size ratios, but the observed results do not support it for D64 and D151. The well known and proven grinding and lapping sequence preceding optical polishing of ZERODUR® using always-finer grains is not touched by this result. It has been shown in practice, that it leads to strengths of similar values as etched surfaces.

The D151 ground sample of ZERODUR K20® is described well by the Weibull distribution and lies higher than that of normal ZERODUR® but not that much higher as expected from the higher crystal phase content (ZERODUR®: 70 – 78 % of 30 – 50 nm sized crystals and ZERODUR K20®: > 90% of 1 – 2 µm crystals).

3.3 All D64 samples, only ground and ground plus subsequently etched

Figure 3 shows all samples, which have been prepared with D64 grinding. Samples # 1 and # 2 are the same as in Fig. 2. They are shown in comparison with the D64 first ground and then etched samples. Sample # 6 and # 7 have been ground with D64 in the same way as sample # 2 and then etched taking off a layer with thickness 91 µm and 73 µm respectively.

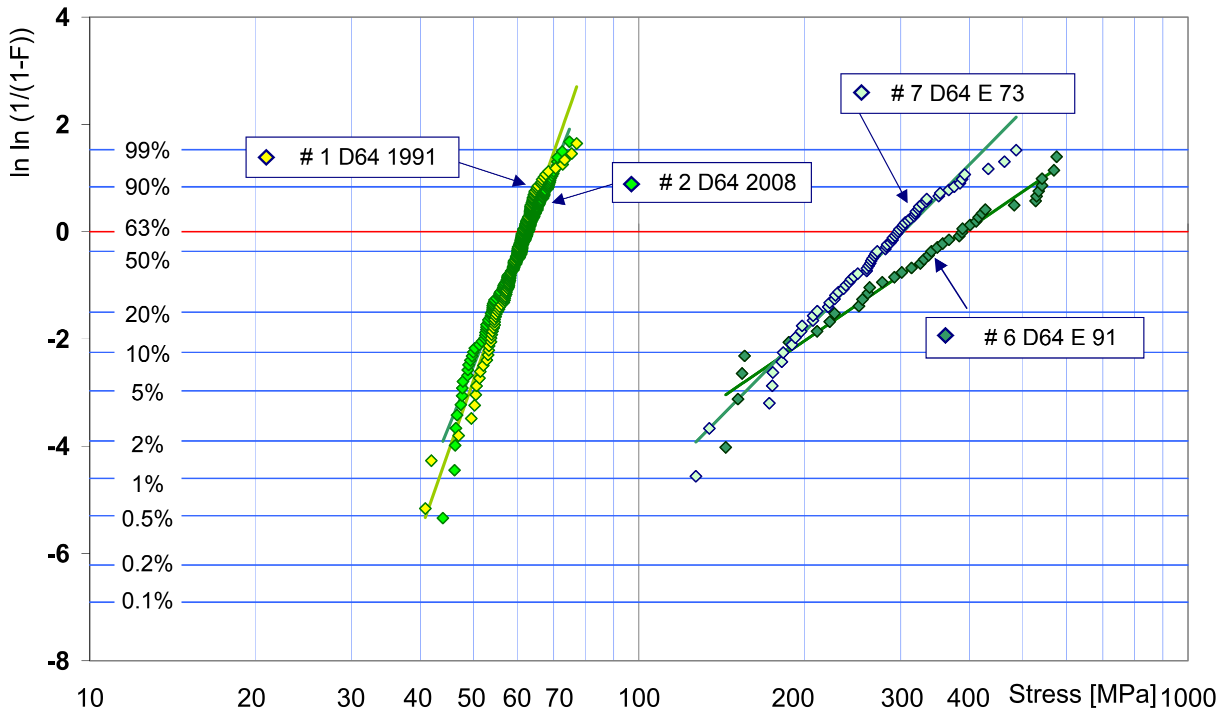

Fig. 3.: All ZERODUR® samples with D64 ground surface condition: Sample # 1 and # 2 as in Fig 4, samples # 6 and # 7 subsequently etched 91µm and 73 µm deep.

Both distributions of the etched samples lie much higher than that for the only ground D64 surface samples #1 and # 2 and are broader as expected. Sample #7 shows similar tendencies like the ground samples to deviate from the Weibull distribution to slightly higher values at the extreme parts of the sample. It has a smaller characteristic strength of 303 MPa and a steeper slope being reflected in the higher value for the Weibull modulus of 4.5 with respect to sample # 6 (characteristic strength: 391 MPa / slope: 3.0). Sample # 6 is fairly well represented by the Weibull distribution. The smallest breakage value observed is 127 MPa. From a SCHOTT internal report it is known that the maximum subsurface cracks of D64 ground surfaces have depths of about 60 µm. The layer thickness taken off with sample # 7 lies at 1.2 times this value, whereas that of sample # 6 is about a factor of 1.5 higher. The etch depth to maximum crack depth ratio, will be called the relative etch depth in the following. Etching deeper obviously leads only to a higher characteristic strength (63% failure probability value) but not to an improvement in the lower strength region of the distribution.

3.4 All D151 samples, only ground and ground plus subsequently etched

Figure 4 shows all samples which have been prepared with D151 grinding. Sample # 5 is the one combined from samples # 3 and # 4 of Fig. 2. They are shown in comparison with the D151 first ground and then etched samples. Sample # 8 and # 9 have been ground with D151 in the same way as sample # 5 and then etched taking off a layer with thickness 181 µm and 34 µm respectively. Sample # 10 is the new result obtained from specimens with 83 µm etched off. Sample # 11 is the new measurement of ZERODUR K20® D151 ground.

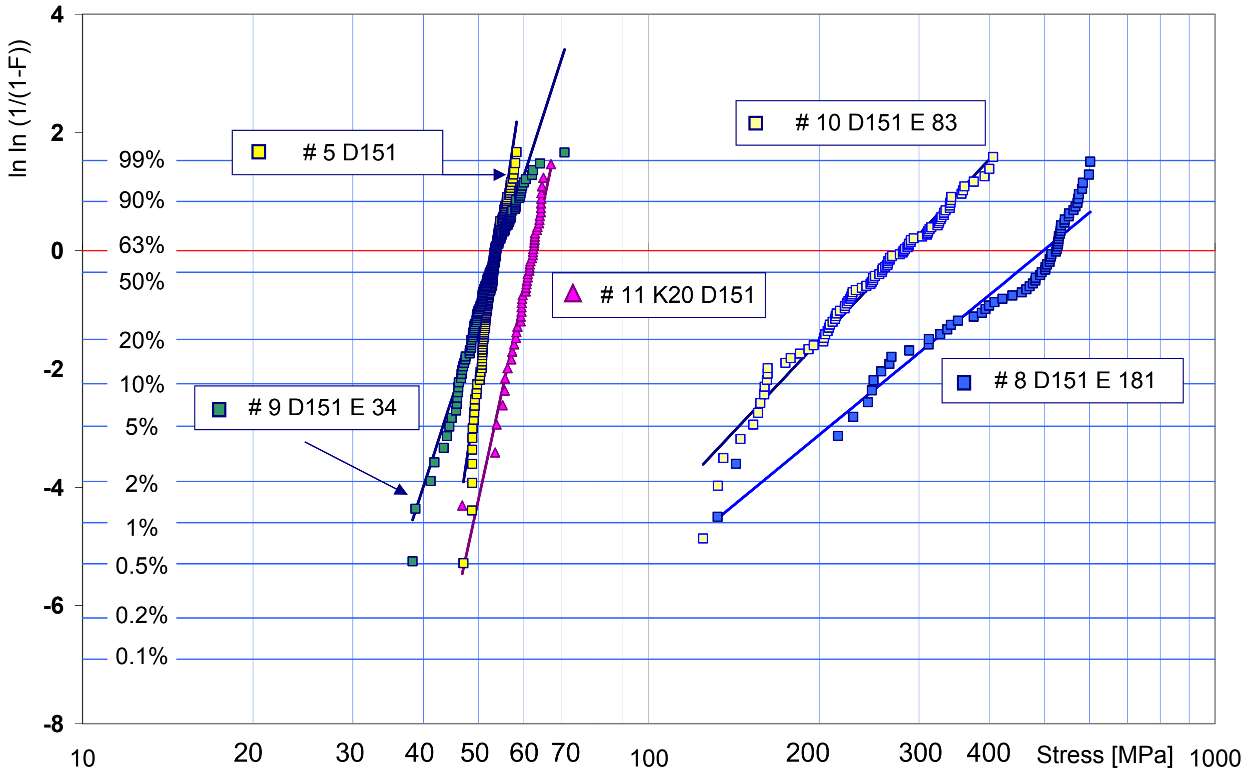

Fig. 4.: All ZERODUR® samples with D151 ground surface condition: Sample # 5 only ground, samples # 8, # 9 and # 10 subsequently etched 181µm, 34 µm and 83 µm deep. Sample # 11 ZERODUR K20® D151 only ground

The samples #8 and # 9 show deviations from the Weibull distribution, sample # 9 in the same way as some of the preceding samples: Slightly higher values at its extreme parts. Sample # 8 shows different deviations not seen in other samples and two specimens at the lower end which could be valued as outliers.

The data situation for D151 surfaces last year showed a gap, since only a sample with deeply etched specimens was available (# 8 with 181 µm, corresponding to 1.5 times of the maximum crack depth 120 µm observed for D151 ZERODUR® surfaces) and sample # 9 with 34 µm or about 0.3 times the maximum crack depth respectively.

The new measured sample # 10 was adjusted to lie in between at a layer thickness somewhat smaller than the maximum crack depth in order to get information about the value of the minimum thickness to be etched off for high strength results. The 83 µm etch depth of sample # 10 corresponding to about 0.7 times the maximum crack depth is obviously sufficient to let strength jump to the high values region. Just like for the D64 ground and then etched samples of Fig. 3 one observes that the characteristic strength of the deeper etched sample lies higher than that of the less deep etched one and the lower end values approach each other at the same minimum value of about 125 MPa just like the D 64 ground and etched surfaces.

Fig. 4 clearly shows that a minimum thickness has to be etched off in order to achieve the significantly higher strength, which one usually attributes to etched surfaces in general. From practical applications we know that this thinking was extended too far. Just a little bit of etching using a weak acid does not produce any strength increase at all. This can be seen from sample # 9, which has been treated with the same strong acid compound as all the other etched samples but which achieved only 34 µm due to the intentionally shorter interaction time. So one can conclude using weak acids or applying stronger acids for only short time does not have any strength increasing effect. Care must be taken, that a layer of minimum thickness of at least 80 µm or better 100 µm to be sure has been etched off for D151 ground items.

3.5 All samples together

Figure 5 shows all samples of the preceding diagrams together.

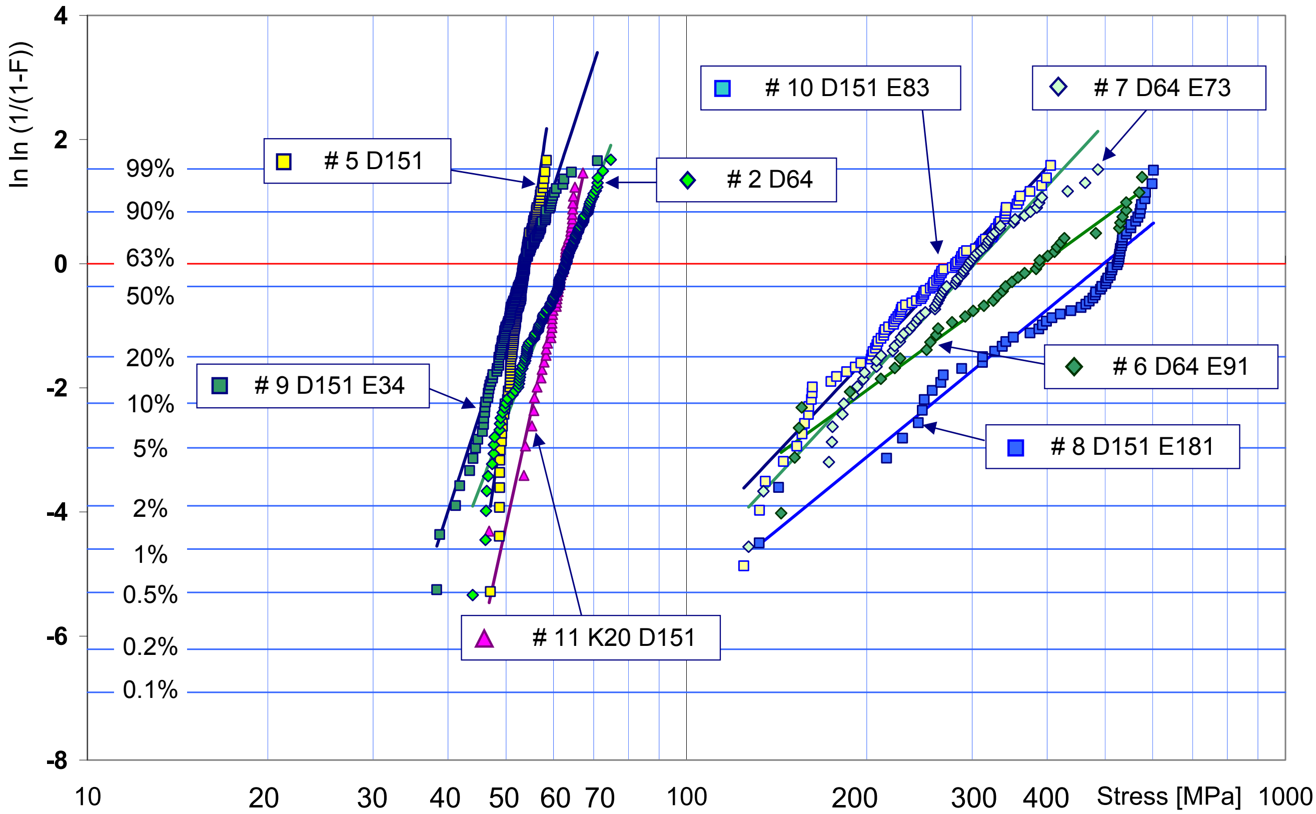

Fig. 5.: All samples of ZERODUR® and ZERODUR K20® with D64 or D151 ground plus etched surface conditions

Fig 5. shows that the strength values for all ground samples lie in the range of about 40 to 70 MPa whereas the samples etched with the necessary minimum layer thickness extend from about 125 to 600 MPa. All four high strength samples meet with their lower end at about 125 to 150 MPa.

An at first sight surprising result comes from samples #6 and # 8, if one considers that for both samples the layer thickness etched off is about 1.5 times higher than the maximum crack depths for the differently ground surfaces (ca. 60 µm for D64 and ca. 120 µm for D151). The distributions lie almost parallel to each other, but the one where etching started from the coarser grain ground surface ends up with strength values about 100 MPa higher (except from the two outliers at the lower end). The D151 etched sample # 10 with a relative etch depth of 0.7 lies very close to the D64 etched sample # 7, which has the significantly higher value of 1.2.

There is obviously a dependence of the strength gain on the roughness or microcrack population resulting from the different grinding grain sizes. For surfaces ground with the coarser grain D151 we observe higher relative strength gains compared to the D64 surfaces with the same relative etch depth.

The expectation before the investigation probably would have been different. The established thinking was D64 surfaces are stronger than D151 surfaces, etching is better in any case, so D64 with etching must be the best. Which is not true if this statement presupposes the same relative etch depth. Comparing the D64 / D151 strengths both with 100 µm etched off the common thinking probably becomes true again. The D64 ground 100 µm etched surface should lie a little bit higher than sample # 6, the D151 ground 100 µm etched surface is expected to lie also somewhat higher than sample # 10 but still lower than the distribution of the D64 ground 100 µm etched surface. However, the difference does not really matter in practice.

Tab. 1.: Strength data of all samples of ZERODUR® and ZERODUR K20® with surface conditions, load increase rate - Statistical data for the Weibull distribution and for the Normal distribution (explanations next page)

Sample No, Surface Condition Load rate	NiL	NaL	NoL	d_{etch} [μm]	d_{etch}/ d_{cmax}	Weibull σ_c [MPa]	Weibull m	Normal σ [MPa]	Normal s
# 1 D64 ground 2 MPa/s Data of 1991	122	-	-	-	-	62.3	12.6	59.8	5.64
# 2 D64 ground, 2 MPa/s	131	15	0	-	-	62.8	11.0	60.0	6.58
# 3 D151ground, 2 MPa/s	80	13	0	-	-	54.1	27.4	53.0	2.34
# 4 D151ground, 2 MPa/s	35	10	0	-	-	54.0	28.4	53.0	2.22
# 5 D151ground, 2 MPa/s (Set # 3 + # 4)	115	23	0	-	-	54.1	28.2	53.0	2.29
# 6 D64 ground & etched 2 MPa/s	14	25	70	91	1.5	391.4	3.04	349.3	125.5
# 7 D64 ground & etched 2 MPa/s	49	18	82	73	1.2	303.1	4.51	276.9	72.7
# 8 D151 ground & etched 2 MPa/s	42	21	48	181	1.5	497.2	3.42	443.7	123.6
# 9 D151 ground & etched 2 MPa/s	108	26	14	34	0.3	54.5	12.9	52.4	5.0
# 10 D151 ground & etched 2 MPa/s	91	3	54	83	0.7	281.8	4.4	257.1	67.2
# 11 D151ground ZERODUR K20® 2 MPa/s	52	0	8	-	-	62.4	19.0	60.7	3.67

NiL	Number of specimens broken within the load ring
NaL	Number of specimens broken at the load ring
NoL	Number of specimens broken outside of the load ring
d_{etch}	Layer thickness etched off from one surface
d_{cmax}	Maximum crack depth of diamond grain ground ZERODUR® surface (D64 ca. 60 μm, D151 ca. 120 μm)
σ_c	Weibull Distribution Characteristic strength
m	Weibull modulus
σ	Normal distribution mean strength
s	Normal distribution standard deviation

4. EXTRAPOLATION TO LOWER FAILURE PROBABILITY

Even though the specimen numbers per sample have been significantly enlarged, the lowest failure probability reached with measured data lies between 0.5 % for ground samples and 1 - 2 % for the etched samples. Such failure probabilities are usually not acceptable for highly valuable and unique objects like large astronomical mirrors or satellite optics. So the question arises, how to find design strength values for use in practical purposes. The frequently used formula (1) to calculate the factor of safety contribution f_p for a given design failure probability F_V and Weibull modulus m as given e.g. in Exner[8] or in SCHOTT TIE 33[9] presupposes the validity of the Weibull distribution and hence the extrapolation to lower failure probabilities than the measured ones.

$$f_p = \frac{1}{\left(\ln \frac{1}{1-F_V}\right)^{1/m}} \tag{1}$$

For ground surfaces this seems to be justified down to 0.1 % failure probability. At least it is a conservative approach not too far from reality. The recently measured samples for D64 and D151 show deviations in lower single digit failure probability range from the Weibull distribution to higher strength values as expected from extrapolation. From the physical point of view this deviation is not surprising, since there is a real maximum in crack depths, which is not described in the Weibull distribution in its purely mathematical extrapolation.

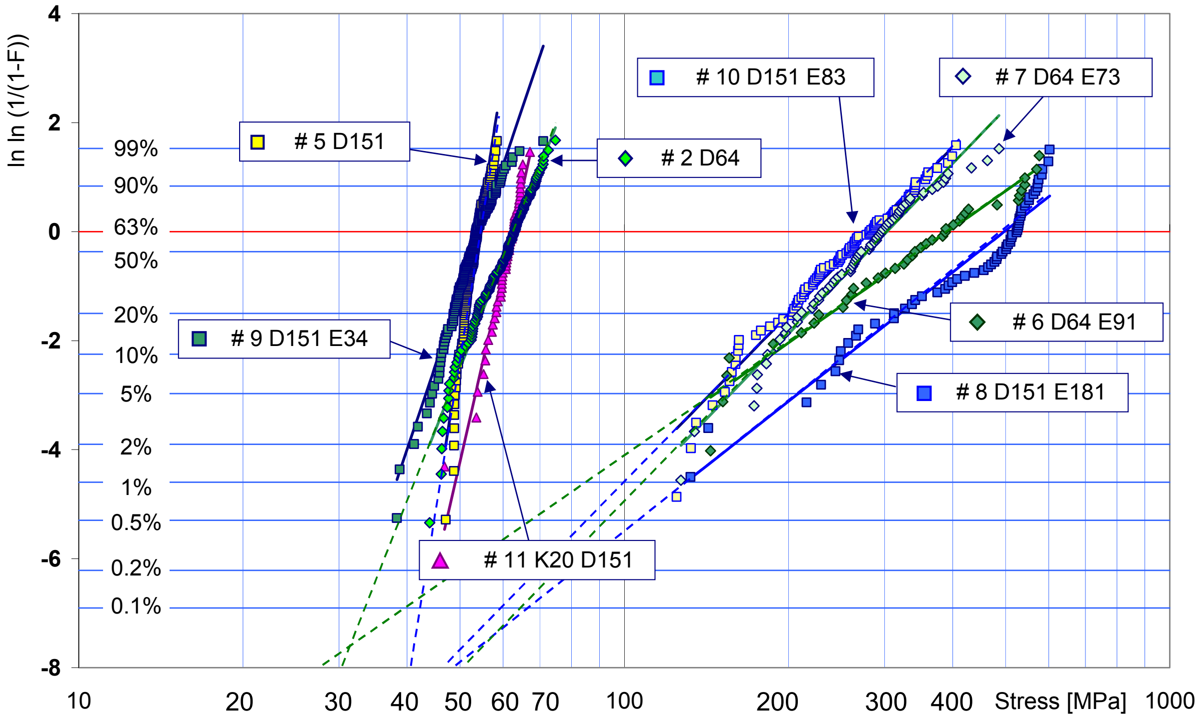

Fig. 6.: All samples of ZERODUR® and ZERODUR K20® with D64 and D151 ground and ground plus etched surface condition with extrapolated distributions

The invalidity of the extrapolation is even more obvious for the etched samples. Their distributions would meet those of the ground samples in the failure probability ranges of 0.1% to about 0.01%. This again bases only on mathematics, a physical reason cannot be seen. On the contrary there are hints that also for etched surfaces there seems to be a maximum crack depth. Maybe they should better be called micro flaws, since the remaining structures weakening the surface with respect to its much higher intrinsic strength are not known. The first hint is that all etched surfaces have minimum strength values above 125 MPa. The other is that also their distributions tend to bend down at their lower ends. The data points within the bending are still in accordance with the Weibull distribution and they are admittedly only few but the trend is still noticeable to our point of view. One would like to have much better statistics here, but this means also much higher effort. Many hundreds of specimens would have to be broken.

Additional to the justification problem of the distribution shape the increasing number of specimens breaking outside of the load ring indicates that a second distribution arises, a probability distribution for the occurrence of a flaw per area. Reasons for this assumption is the the well established fact, that within a ground surface there are many microcracks within the test setup's loaded area of 2.5 cm^2. So the statistical breakage behaviour will be dominated by a weakest link distribution. The results for etched surfaces show that not only the depth of a flaw is important but also its presence within a given area.

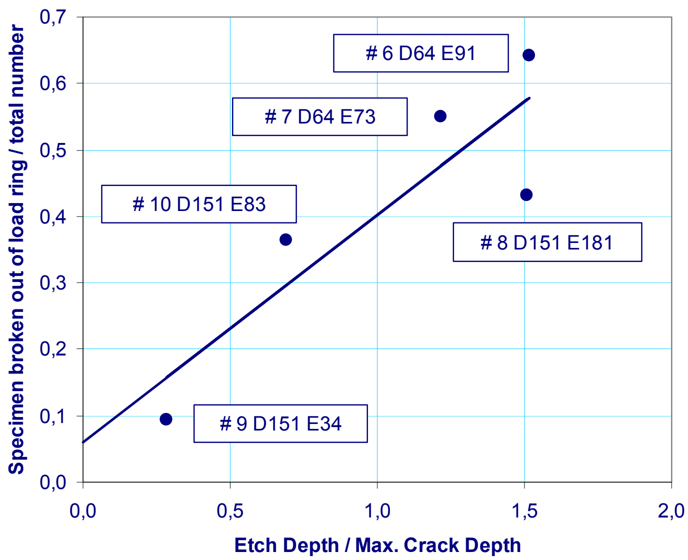

Fig. 7: Ratios of specimens broken out of load ring versus relative etch depth for all ZERODUR® etched samples

Fig. 7 shows the strong increase of the ratio of the number of specimens broken outside the load ring per total number with the increasing ratio etch depth to maximum crack depth.

So especially for the etched surfaces the design strength value for low failure probability like the quite commonly specified 0.1% still is not answered unambiguously. There is reason to believe, that there is a minimum strength slightly above 100 MPa. This is supported by the observation of minimum strength values of about 125 MPa for the lowest strength specimens out of a total set of 263 specimens, if one takes all sufficiently etched samples together. However, this is only plausible but not really a data supported proof.

5. STRESS CORROSION COEFFICIENT OF ZERODUR®

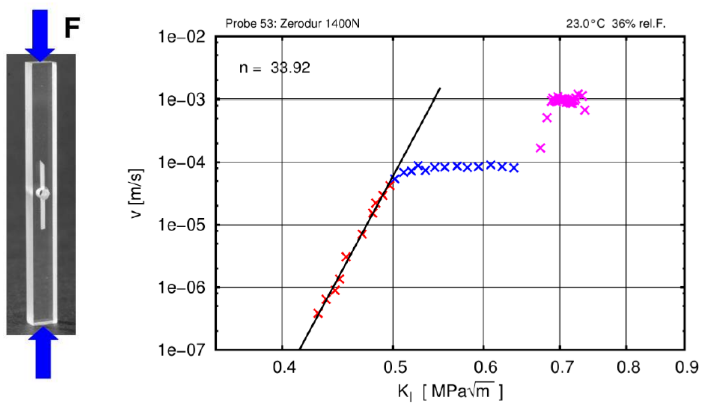

Fig. 8: Left: ZERODUR® DCDC sample 50 x 5 x 3 mm^3 with diameter 1.7 mm borehole, right: ZERODUR® crack growth velocity versus stress intensity factor for 37% humidity representative measurement

The stress corrosion coefficient n of ZERODUR®[9] has been measured with the double cleavage drilled compression (DCDC) method originally introduced in 1974 by Janssen[11]. It uses samples with a central bore hole as the origin of cracks, see figure 8. The force applied to the end faces makes the cracks grow, which allows the growth velocity to be measured. The advantage of the method is, that the growth velocity decreases with increasing crack length, which comes from the decreasing stress concentration factor K_I. Two sets of several samples have been measured, one for normal room humidity and one for dry environment conditions. Figure 8 shows one of the measurements of the growth velocity v versus the stress intensity factor K_I done at normal room humidity.

The results are:

n (25 – 27°C, 3% relative humidity) = 50 ± 3

n (23 – 25°C, 37% relative humidity) = 31 ± 3

The result for 3% humidity can be used for vacuum applications like stress loads during space missions. It should be noted that the 37% relative humidity value for n is effective in normal environment only if significant tensile stress is present. The stresses at the surfaces of glass and glass ceramic items due to their own weight of are usually far below the minimum K_I value, where cracks start to grow.

The normal humidity value differs from those being quoted in literature[9] in the past, which have been measured with different methods. We see need for further clarification here.

6. CONCLUSION

Increasing numbers of cutting-edge technologies intend to use ZERODUR® mirrors or structures capable to withstand high mechanical loads. They have to carry their own weight or additional loads caused by high and rapidly varying deformations or accelerations. These are e.g. segments of a large primary mirror, which have to be fixed to support structures and to be deformed with push-pull elements usually attached to the segment via bonding. Here it is essential to know which tensile strength ZERODUR® can endure. Satellite missions use support structures of complicated design subjected to high mechanical loads, like vibrations during rocket launch. Microlithography wafer steppers uses frames, which are highly accelerated many thousand times during their operational lifetime.
This makes the strength of a ZERODUR® structure a key design parameter.

This paper has presented additional results of investigations of D64 and D151 ground and etched surfaces with special emphasis to enlarging the statistic database for the strength evaluation of ZERODUR®. Large samples of ZERODUR® specimens produced with a common grinding process have been tested up to failure using standard double ring tests according to DIN EN 1288-5.

The main results are:

- There is a good reproducibility of the strength distribution of ground surface samples even after long time.
- The fine ground D64 surface shows no relevant strength increase with respect to the D151 surface condition at lower failure probability.
- ZERODUR K20® shows 15 % higher strength than normal ZERODUR®, when both are ground with a D151.
- In order to achieve a significant strength increase via etching care must be taken to remove a minimum layer thickness. For D64 ground surfaces 73 µm (factor 1.2 with respect to the maximum crack depth) is proven to be sufficient, for D151 it is 83 µm (factor 0.7). Taking off about 100 µm is sufficient in any of the investigated cases.
- Insufficient etching not achieving the minimum necessary layer thickness does not lead to strength increase.
- If related to the same relative etch depth the strength of etched samples depend on the preceding grinding process whether D151 or D64 has been used. The sample ground with the coarser D151 grain tool exhibits higher strength values after etching than that which first had been ground with the finer D64 grain tool and then subsequently etched.
- Deviations from the Weibull distributions have been observed for only ground and ground plus etched samples, which give some indication for the existence of minimum strength values as one would expect from the physical point of view, considering that there are cracks or flaws in the surfaces with a maximum depth.
- Extrapolation of the Weibull distribution down to low failure probability like 0.1% seems to be justified to a certain extent for ground surfaces but not adequate for etched surfaces.
- New data for the stress corrosion coefficient have been measured confirming the observation of high values for ZERODUR® even though values from literature have not been met satisfactorily. Here some work still has to be done.

All results can be related to practical cases (for detailed discussion see e.g. Nattermann et al.[12]) only under the precondition that holds for all such surfaces: They must free from local damages, which are not representative for the investigated surface conditions, like deep scratches, flaws or macroscopic cracks. Such damages will locally reduce the component's strength significantly. Therefore it is highly recommendable to perform a final visual surface and edge inspection at the earliest possible stage and then to secure all exposed surfaces by covers or simply by inaccessibility.

7. ACKNOWLEDGEMENTS

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