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Fracture statistics of ceramic laminates strengthened by compressive residual stresses

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Abstract

Ceramic multilayer composites have been developed in recent years to enhance toughness and reliability of ceramics. It has been demonstrated by theoretical as well as experimental means, that surface compressive stresses protect the composite against the negative action of surface flaws.

The behaviour of an alumina–alumina/zirconia laminate having significant compressive residual stresses at its alumina surface is investigated. Compared to alumina specimens its strength is increased by the amplitude of the residual compressive surface stress, which is also a lower threshold value for strength. The consequences of that behaviour for the fracture statistics and reliability are discussed.

Keywords: Strength; Fracture; Al_2O_3; ZrO_2; Laminate

1. Introduction

Ceramic materials suffer from brittle fracture, which – in general – starts from small flaws, which are distributed in the material or on its surface.[1,2] The strength of a specimen is defined by the major flaw, i.e. by its shape, size, orientation and its position in the specimen.[3,4] In general it is assumed that flaws behave similarly to cracks.[1–6] Thus a fracture mechanical failure criterion can be defined, which correlates the strength of the specimen with the size of the crack. For example, following the well known Griffith/Irwin criterion the strength (σ_f) is inverse proportional to the square root of the size (a) of the crack[1–6]: $\sigma_f \propto 1/\sqrt{a}$. Since the size and position of flaws are statistically distributed the strength of ceramic specimens shows a large scatter and design with ceramic materials has to be performed with statistical means. Strength tests show that the probability of failure, F, increases with the applied load and the size of the specimen.[2,5,6] This behaviour is well described by the two-parameter Weibull distribution function, which, in its simplest form and for volume flaws distributed in a specimen of volume V and loaded in a uniaxial and homogeneous tensile stress state with amplitude, σ, is given by[7,8]:

$$F(\sigma, V) = 1 - \exp\left[-\frac{V}{V_0}\left(\frac{\sigma}{\sigma_0}\right)^m\right] \qquad (1)$$

The Weibull modulus m depends on the distribution of the flaw sizes.[9,10] It describes the scatter of the strength data: the smaller is m, the larger is the scatter. V_0 is an arbitrary normalising volume. The characteristic strength σ_0 is the stress at which – for specimen of volume $V = V_0$ – the probability of failure is 63% ($F = 63\%$). σ_0 and V_0 are not independent. The two independent parameters are m and $V_0\sigma_0^m$. Even for very small tensile stresses being only a little above zero some probability of fracture exists.

All specimens tested in this study had a nominally identical size and were tested under nominally identical conditions. In the following, the normalising volume in Eq. (1) is set equal to the specimens' volume ($V = V_0$). If surface flaws are important an analogous Weibull distribution for surface flaws can be found and the analogous simplification will be used.

The probability of failure depends sensibly on the parameters in the Weibull distribution, which must therefore be determined with the highest possible precision. Yet, the experimental determination of a Weibull distribution is expensive and laborious.

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Standards advise that at least the strength of 30 specimens has to be measured.[11] The investigated interval of fracture probabilities depends on the number of tested data (roughly speaking it ranges from $1/2N$ to $(2N-1)/2N$, with N being the number of broken specimens).[2,6] With only 30 specimens the lowest "measured" fracture probability is $(1/60) \approx 1.67\%$. In common laboratory practice even a smaller database is often used. Therefore there is a great need for data extrapolations in the mechanical design process, where high reliabilities (R, note that $R = 1 - F$) of ceramic components are required. However, there remains some risk of failure even in the case of a very low loading. This highlights the desire for more reliable and less brittle materials.

Nature shows us that large improvements in toughness and reliability can be achieved by a layered architecture of materials.[12,13] Even using constituents having poor mechanical properties, the strength and toughness of natural materials with their hierarchical architecture can be impressive. An attempt to copy nature is to make ceramic materials and components having a layered architecture. The idea is to deflect, bifurcate or stop cracks at the boundaries between the layers.[14] This should result in an increased fracture toughness and reliability of the laminates compared to monoliths.

Two concepts were followed up in the past: First, composites with weak interfaces were developed, showing an increased fracture energy of the system in comparison to monolithic reference materials. Early work on SiC/graphite laminates showed an outstanding apparent fracture toughness values up to 18 MPa $\sqrt{m}$ in comparison to 3.6 MPa $\sqrt{m}$ corresponding to monolithic SiC compounds. The mechanism responsible for such enhancement is based on the capability of the graphite layers in guiding the propagating crack along the interface, and thus suppressing catastrophic failure as it would be expected in brittle monolithic materials.[15,16] Secondly, composites with strong interfaces were developed in which significant residual stresses have an important influence on the propagation of the cracks.[17–20] Designs with outer layers under residual tension show a pop-in of cracks even under gentle loads but the cracks stop in general within the second layer, which is compressed by the residual stresses.[21] The strength of these laminates is modest but the scatter of strength is low.[22] In laminates having the outer layers under residual compression an increase of strength[23] and fracture toughness with crack extension (R-curve behaviour) can be observed in the compressed surface layer.[24] For longer cracks, the R-curve can decrease again.[24,25] In fact the shape of the R-curve can be designed to a large extend by the layer architecture. On the basis of this concept fracture toughness values well above 10 MPa $\sqrt{m}$ seem to be possible.[21,25–28]

In this paper the strength of an alumina–alumina/zirconia laminate is investigated. The bending strength of the laminate is measured and the data are analysed using Weibull statistics. The existence of a lower limit on strength is discussed and the consequences for design are explained.

2. Experimental procedure and results

Two types of specimen were investigated: laminates (L) consisting of layers made from alumina (a) and from an alumina/zirconia mixture (az, 60 vol% alumina + 40 vol% Y-TZP) with the stacking sequence 2a/az/a/az/a/az/2a and alumina specimens (A) with a stacking sequence 10a. The thickness of the individual layers in the sintered plates was approx. 170 μm for a-layers and approximately 220 μm for the az-layers, respectively. Laminated plates as well as alumina plates were produced via the same processing route at ISTEC-CNR in Faenza, Italy. Green tapes were laminated, pressed and sintered with the same sintering profile for laminates and alumina specimens respectively. Special care was taken that the surface layer of the composite was made of the same alumina ceramic as the in the case of the alumina specimen. Details on the processing and the involved materials can be found elsewhere.[29,30]

Due to the thermal expansion mismatch residual stresses are persistent in the L-laminate after sintering. They are compressive in the a-layers and tensile in the az-layers. Their magnitude depends on the elastic properties of the materials (Young's modulus, Poisson's ratio), the CTEs, the temperature below which no stress relaxation takes place during sintering (T_{sf}) and the layer thicknesses. They were evaluated by analytical estimations,[31] 3D-FEM calculations[30,32] and with an indentation technique[33] employing values for the material properties that were independently determined for each constituent material.[30] The compressive stress in the a-layers of the L-specimens is around −134 to −160 MPa, the tensile stress in the az-layers is approximately 207–246 MPa. The most questionable quantity in these analyses is the temperature T_{sf} which has been estimated to be 1160–1200 °C.[32,34]

27 bend bars from the alumina A and 21 bend bars from the laminate L (1.7 mm × 2.6 mm × 28 mm) were diamond machined from plates in such a way that the prospective tensile and compressive sides were kept in the as-sintered state. 4-point bend tests were carried out using 20 and 10 mm spans on a Zwick Z010 universal testing machine with 2 mm/min crosshead speed. The results of the strength tests on both batches are shown in Fig. 1 where the probability of failure is plotted versus the strength in a Weibull plot. It is obvious that the laminate specimens (△) have a superior strength compared to the alumina (◯). The parameters of the Weibull distributions as evaluated using the maximum likelihood method[11] for L and A can be found in Table 1. The given spans given with the numbers refer to the 90% confidence intervals.

Fracture surfaces were investigated by stereomicroscopy and SEM. In the laminate as well as in the alumina specimens fracture origins were large alumina grains at the surface of the specimens (see Fig. 2). Probably these grains have grown by abnormal grain growth.[35] Differences in the microstructure between L- and A-specimens could not be found in the outer alumina layer.

3. Discussion

In both types of specimens fracture started at surface defects. For both types of specimens the surface region consists of alumina processed in an identical way and having the same microstructure. In both cases the fracture origins were large grains at the surface. The only significant difference between L-

Table 1
Parameters of the Weibull distributions discussed in this paper

	Characteristic strength [MPa]	Weibull modulus	Threshold stress [MPa]
Evaluation of individual batches A and L, two-parameter Weibull curve fitting			
Alumina (batch A)	492 ± 17	10.4 ± 2.6	0
Laminate (batch L)	650 ± 15	18.1 ± 5.1	0
Evaluation of a combined batch A + L, three-parameter Weibull curve fitting			
Alumina (batches A + L)	492 ± 11	11.5 ± 2.2	0
Laminate (batch L)	492 ± 11	11.5 ± 2.2	158 ± 32

The upper and lower limits refer to estimates of the 90% confidence intervals.

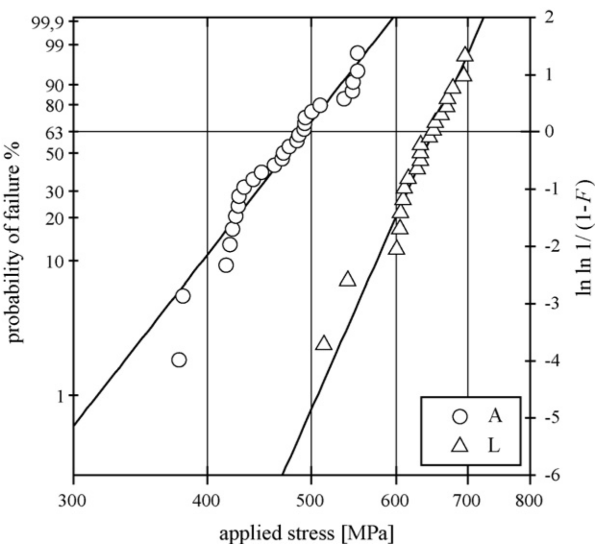

Fig. 1. Probability of failure versus applied rupture stress for alumina specimens (○) and specimens of the alumina–alumina/zirconia laminate (△) in a Weibull plot. The lines represent best fits of a two-parameter Weibull distribution to the data.

and A-specimens at the position of the fracture origin was the occurrence of compressive residual stresses in the surface region of the laminate specimens. In the following, the significant difference in the mechanical behaviour between the laminates and the monoliths (see Fig. 1) is put down to these stresses.

The total stress (σ_t) is the sum of the applied stress (σ_a) and the residual stress (σ_{res}): $\sigma_t = \sigma_a + \sigma_{res}$. In the monolithic ceramic the residual stress is zero. In the laminate it is compressive and almost constant in the surface region, where fracture initiates.[30] It is assumed in the following that for the laminates, which fail

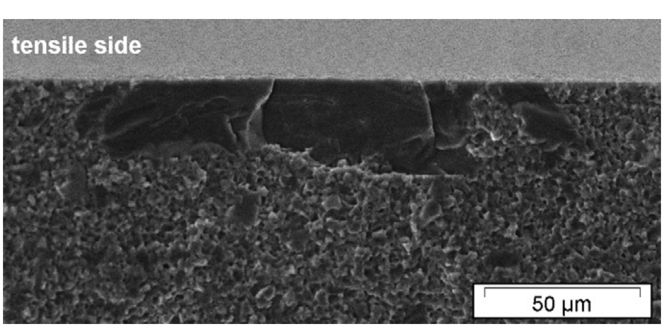

Fig. 2. A large alumina grain acting as fracture origin in an alumina specimen. The same type of defects was responsible for failure in the laminates.

from small flaws in the surface layer, the Weibull distribution of Eq. (1) can be modified to account for the action of the residual stress by setting $\sigma = \sigma_t$:

$$F(\sigma) = 1 - \exp\left[-\left(\frac{\sigma_a + \sigma_{res}}{\sigma_0}\right)^m\right]. \quad (2)$$

The results of the fracture experiments can be explained on the basis of this idea. The characteristic strength (in terms of applied stress) of the laminate (650 ± 15 MPa) is significantly higher than that of the A-specimens (492 ± 17 MPa). Following the above argument the difference (−158 ± 32 MPa) is caused by the compressive residual stress in the first layer of the laminate. This fits well to the values for the residual compressive stress mentioned above.

It is assumed that in terms of total stress, the strength distributions of both materials are identical, since they are based on the same flaw population. In other words the data sets A and L are samples out of the same parent distribution. Since evaluation uncertainties arising from the sampling procedure are smaller for a large than for a small number of tests in a sample a common data evaluation will result in more precise Weibull parameters. Fig. 3 shows the Weibull statistics of a set of tests which consists of the data of batch A and L. For the residual stress of the data in batch L the value $\sigma_{res} = -158$ MPa

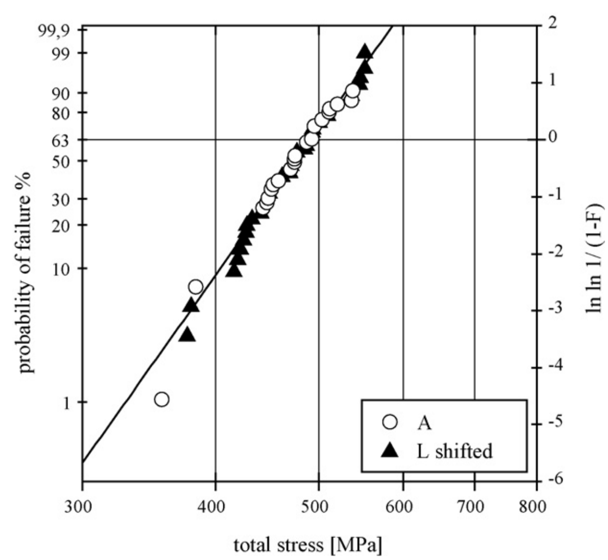

Fig. 3. Same data as in Fig. 1 plotted versus the sum of applied and residual stress: $\sigma_t = \sigma_a + \sigma_{res}$. The line represents a common evaluation of both batches.

is used. The data fit very well to a Weibull distribution with $m = 11.5 \pm 2.2$ and $\sigma_0 = 492 \pm 11$ MPa. It should be recognised that these parameters are determined more accurately, than if they would have been determined for each set of data separately (remark: a more detailed discussion on the relationship between sample and parent distribution has been published).[6,36] A summary of the determined parameters is given in Table 1.

If the strength data of the sets A and L are evaluated using the conventional procedure based on Eq. (1) the Weibull moduli are 10.4 and 18.1 respectively (Fig. 1). The apparent increase of the modulus of batch L is a consequence of the application of inappropriate fracture statistics. The right hand curve in Fig. 1 results from adding a constant value (158 MPa) to the data of the left hand curve. In a logarithmic scale a constant added to a small number causes a wider shift of the datum than if it is added to a high number. Of course this shift transforms a straight line into a curve but this is masked by the scatter of the data. Therefore a straight line can be confidently fitted to the data but this line must have a higher slope (the distribution has a higher "Weibull modulus") than the original distribution.

A further consequence of the use of inappropriate statistics is that the value of the "apparent" Weibull modulus is not well defined. It becomes residual stress dependent. Near the threshold stress it tends to infinity. It also becomes dependent on the number of test pieces in the sample. Since – for a large number of tests – more specimens have a strength value near the threshold stress than for a small number of tests, the apparent modulus of a large group of tests is higher than that of a small group.

The appropriate fracture statistics for the laminate (L) is the three-parameter Weibull distribution, where the residual stress determines the threshold stress σ_u: $\sigma_u = -\sigma_{res}$. In the case of the alumina specimens the residual stress is zero and the three-parameter distribution is equal to the two-parameter distribution. But as discussed above the data of both batches (A and L) can be evaluated together, which makes the database wider and the fit more reliable. The corresponding Weibull parameters are also shown in Table 1 and the strength distributions are plotted in Fig. 4. The shaded area in the top right corner corresponds to the parameter field of Fig. 1. The left line is the distribution of batch A shown on the left hand side in Fig. 1. The improvements in strength caused by the layered architecture can clearly be recognised. The full curve shows the trend of the fracture statistics (three-parameter Weibull) for the laminate.

In the experimentally assessed parameter range (shaded area) big differences between the three-parameter and the simple two-parameter statistics do not occur. In this range fracture probabilities are high. But in mechanical design low fracture probabilities (high reliabilities) are required. In this range relevant differences between both statistics occur. To give an example for a reliability of 99.999999% (failure probability $F = 10^{-8}$) the "design" stress for the alumina specimens is 99 MPa (point (◯) in Fig. 4). This stress can be significantly increased by the layer architecture. Following the (inappropriate) two-parameter extrapolation it results in 235 MPa (△) and the (appropriate) three-parameter extrapolation yields 257 MPa (▲). In terms of reliabilities (failure probabilities) for a given design stress the differences may even be more pronounced. At

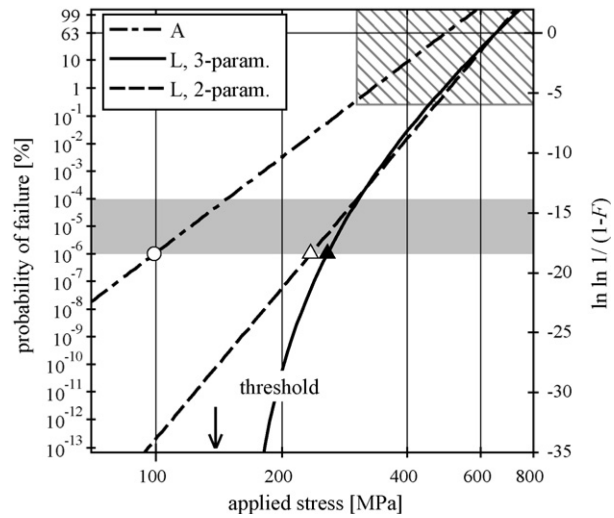

Fig. 4. Extrapolation of strength data. In technical applications very high reliabilities are often claimed. Although the difference between the two-parameter and the three-parameter distribution is small in the experimental accessible parameter range (shaded area) the tolerable design stresses can be quite different. Shown is also the Weibull distribution of alumina specimens.

a design stress of 250 MPa the failure probability is $4 \times 10^{-2}\%$ in the case of batch A and of $3 \times 10^{-6}\%$ and $4 \times 10^{-7}\%$ in the case of batch L and for the conventional and advanced evaluation procedure, respectively. It should also be noticed that the laminate has a threshold strength and this threshold is only accounted for in the three-parameter distribution. At the threshold stress of 158 MPa, the failure probability of batch A is still $2 \times 10^{-4}\%$. For batch L and using the inappropriate conventional evaluation procedure it is $8 \times 10^{-10}\%$. If the appropriate three-parameter distribution is used, it is exactly zero.

Let us now discuss one mathematical aspect related to the evaluation of the data. It is – in general – expected, that a threshold stress can be recognised in a conventional Weibull plot (probability of failure versus applied stress; see for example Fig. 1). But in the right hand curve of Fig. 1 no indication of a threshold exists.[37–39] This observation raises immediately the query of what is the reason for that. This behaviour is caused by three reasons. First, the characteristic strength of the data set is much higher than the threshold stress. The characteristic strength defines also the maximum of the relative frequency of Weibull distributed strength data, i.e. most of the experiments have a strength value near the characteristic strength, which is far from that part of the distribution, where the influence of the threshold is significant. Second, the number of tests is very small (21 tested specimens). For a small number of tests (each group of tests with less than a few thousand data will be small) the behaviour of the investigated sample can be quite different to that of the parent distribution (examples related to Weibull distributions have already been discussed).[6,36] Again it is most probable that most strength data are not far from the characteristic value. Outliers, which define the shape of the distribution, occur only very seldom in batches containing only a few test pieces. Third, the scatter of the data is relatively large. This makes possible differences between sample and parent populations even more pronounced. All three aspects are important and all together prevent the recognition of a threshold by simple analysis of strength data in the discussed case.

An interesting consequence of this behaviour is that the fitting procedure for three-parameter Weibull distributions gives unstable results in many cases. For example the omission of a single data point may have significant influence on the determined threshold stress. In our analysis the threshold stress has not been fitted to the data, it has been set equal to the residual stress in the outer layer of the laminate. Only if the threshold stress is well defined, a stable fitting of the other two parameters is possible on the basis of a small number of tests.

Finally, it should be realised that this simple analysis is only valid if the stress field can be considered to be almost constant over the extension of the crack, i.e. if the crack size is small compared to the thickness of the first compressed layer. In other cases a more complicated analysis based on the analysis of the stress intensity factors of cracks would be necessary.[6,40,41] The critical (Griffith) crack size can be determined from strength data[2–6] σ_f via $a_c = (1/\pi)(K_c/Y\sigma_f)^2$. The fracture toughness in the first alumina layer is $K_c = 3.8\,\text{MPa}\sqrt{m}$.[33] The geometry factor of a surface crack is approximately $Y = 1$. With these assumptions the critical crack sizes for batches A and L range from about 15–35 μm. This analysis fits to the fractographic evidence, Fig. 2.

4. Conclusions

General conclusions related to the behaviour and fracture statistics of laminates strengthened by compressive stresses and more special conclusions on the behaviour of the investigated laminate can be drawn from this work. In general it holds that:

- The strength of ceramics can significantly be increased by a layered architecture of the specimens if the outer layer of the specimen has compressive residual stresses.
- The residual compressive stress causes a lower bound (threshold) for the strength, therefore a high amplitude of the compressive stress is beneficial.
- The threshold is masked by the scatter of the strength data. Therefore it can hardly be recognised in a conventional Weibull diagram.
- For this type of laminate the appropriate fracture statistical approach is the three-parameter Weibull method. The two-parameter Weibull approach is not appropriate and should not be used, since its application can cause inappropriate extrapolations. Compared to the Weibull modulus of the outer layer material (were fracture initiates) the inappropriate "two-parameter Weibull modulus" of the laminate is increased. It even depends on the number of tests undertaken. For extrapolations to very high reliabilities the inappropriate two-parameter Weibull distribution gives conservative results.

In the case of the investigated alumina–alumina/zirconia laminate:

- Fracture always initiated (in the alumina specimens as well as in the laminate specimens) at abnormally large alumina grains, which occurred at the as-sintered surfaces. Since fracture originated due to the same flaw population in both batches, the fracture statistics of both batches is identical, if the total stress (applied stress plus residual stress) at the position of the flaws is correctly taken into account.
- The layered architecture causes compressive residual stresses in the outer alumina layer, which cause an increase in strength of approximately 158 MPa. This also causes a lower bound of the strength (threshold stress).

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