

v-K-data for silica from interrupted lifetime measurements

V. Sglavo¹, T. Fett², K.G. Schell², M. J. Hoffmann², S. M. Wiederhorn³

KIT SCIENTIFIC WORKING PAPERS 144



KIT – Universität des Landes Baden-Württemberg und nationales Forschungszentrum in der Helmholtz-Gemeinschaft

1) Department of Industrial Engineering, University of Trento, Trento, Ital.

2) Karlsruhe Institute of Technology, Institute for Applied Materials, Karlsruhe, Germany

3) National Institute of Standards and Technology, Gaithersburg, MD, USA

Impressum

Karlsruher Institut für Technologie (KIT) www.kit.edu



This document is licensed under the Creative Commons Attribution – Share Alike 4.0 International License (CC BY-SA 4.0): <u>https://creativecommons.org/licenses/by-sa/4.0/deed.en</u>

2020

ISSN: 2194-1629

Abstract

Different methods were applied so far in order to determine subcritical crack growth for silica. Mostly, fracture mechanics standard tests with *macro cracks* were used for this purpose. In this report, we evaluated the subcritical crack growth curves from interrupted lifetime tests on silica bending specimens containing small *natural flaws*. The resulting *V*-K-curve showed crack growth rates down to 10^{-14} m/s indicating a threshold for subcritical crack growth at

*K*_{th}**≅0.31** MPa√m

In the plot of $v=f(K/K_{Ic})$ slight material differences could be eliminated and suitable agreement with macro-crack results by Wiederhorn and Bolz [1] on DCB-specimens and Michalske et al. [2] on DCDC-specimens could be stated.

Contents

1	Introduction	1
2	Evaluation of lifetimes from ISF-tests by Sglavo and Green	1
3	Threshold stress intensity factor	5
	References	7

1. Introduction

Different types of test specimens were used in the past for the measurement of subcritical crack growth in silica. Crack-growth data by Wiederhorn and Bolz [1] were measured with the Double-Cantilever Beam (DCB) method and Michalske *et al.* [2] used the Double cleavage drilled compression (DCDC) specimen. Minimum crack-growth rates of 10^{-11} - 10^{-10} m/s could be reached. Lower rates are reachable by lifetime methods. In the present note, data obtained by Sglavo and Green [3] on interrupted lifetime tests will be evaluated with respect of the *V*-*K* curve. For this purpose, we use the procedure given by Fett and Munz [4]. The *V*-*K* of subcritical crack growth curves may be described by the straight-line relation [1]

$$\mathbf{V}(K) = \mathbf{V}_0 \exp[bK] \tag{1}$$

For the initial *K* value for a crack of length $a=a_0$, i.e. at the beginning of crack growth, we make use of the relation

$$\frac{K_i}{K_{\rm lc}} = \frac{\sigma}{\sigma_c} \tag{2}$$

(K_{Ic} =fracture toughness, σ_c =inert strength). The representation of the inert strengths by a Weibull distribution reads

$$F = 1 - \exp\left[-\left(\frac{\sigma_c}{\sigma_0}\right)^m\right]$$
(3)

with the failure probability F and the two parameters σ_0 and m which can be determined by using the Maximum-Likelihood method [5]. The crack-growth rate results in [4]:

$$\mathbf{V}(K_{\rm i}) = -\frac{2}{t_{\rm f,1}\sigma_{\rm l}^2} \left(\frac{K_{\rm i}}{Y}\right)^2 \frac{\mathrm{d}[\log(K_{\rm i})]}{\mathrm{d}[\log(t_{\rm f,1})]} = -\frac{2}{t_{\rm f}\sigma_{\rm c}^2} \left(\frac{K_{\rm Ic}}{Y}\right)^2 \frac{\mathrm{d}[\log(K_{\rm i})]}{\mathrm{d}[\log(t_{\rm f,1})]}$$
(4)

2. Evaluation of lifetimes from ISF-tests by Sglavo and Green [3]

Figure 1 shows the strength in silicone oil interpreted as the inert strength σ_c as the open squares. It was found by testing 45 specimens: $\sigma_0=211$ MPa, m=8.2.

Then several test series of each 29 specimens were loaded for dwell times of $d_w = 1h$, 1d, 5d, 20d and 50d under constant bending stresses of $\sigma_{dw} = 94$, 100 and 105 MPa in water. The circles in Fig. 1 show the residual strengths for specimens that survived a dwell time of $t_{dw} = 5$ days under $\sigma_{dw} = 105$ MPa bending stress. This type of tests is called "interrupted static fatigue test (ISF-test)".

The failure probabilities for the specimens failed *before* 5 days, $t_f < t_{dw}$ are indicated by the crosses near the ordinate (lowest 9 spontaneous fractures not plotted). The highest failure probability of the tests with failure before 5 days is illustrated by the bold cross. Its failure probability is denoted as F_1 . The failure probability corresponding to the first survival is denoted as F_2 . The inert strengths related to the two failure probabilities are $\sigma_{c,1}$ and $\sigma_{c,2}$, respectively. They can be obtained from the inert strength series at the same failure probabilities as is symbolized in Fig. 1 by the blue lines for $(F_1, \sigma_{c,1})$ and the red ones for $(F_2, \sigma_{c,2})$.

For specimen *i* of the *N* ranked strength data, the failure probability *F* was computed in [3] can be computed via F=i/(N+1), and eq.(3) solved with respect to σ_c . This procedure is useful in all cases where a sufficient straight-line behaviour in the Weibull diagram is observed. This is the case for the squares in Fig. 1.

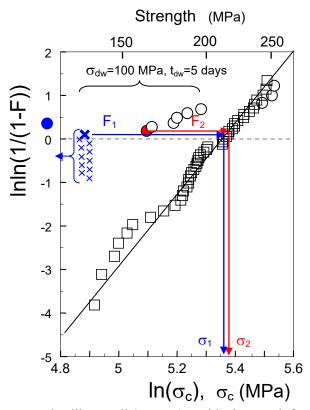


Fig. 1 Strength measurements in silicone oil (squares), residual strength for samples subjected to ISF test in deionized water with duration of t_{dw} =5 days and applied stress of σ_{dw} =105 MPa (circles). Crosses indicate the specimens that failed during the test duration (bold cross: "strongest" specimen that failed before 5 days).

In the case of noticeable deviations from a Weibull distribution (bi- or multimodal distributions or materials with pronounced R-curve) it is recommended to interpolate

linearly the individual inert strength data directly below and above the interesting failure probability.

The results of Fig. 1 can be used to determine the v-K-curve of subcritical crack growth at very low K-values. For this purpose, we use eq.(4) in the from

$$\mathbf{V}(K_w) = -\frac{2}{t_{\rm dw}\sigma_{\rm c,f}^2} \left(\frac{K_{\rm Ic}}{Y}\right)^2 \frac{\mathrm{d}[\log(\sigma_{\rm dw}/\sigma_{\rm c,f})]}{\mathrm{d}[\log(t_{\rm dw}\sigma_{\rm dw}^2)]}$$
(4a)

with $Y \cong 1.3$ and the fracture toughness $K_{Ic}=0.75$ MPa \sqrt{m} . The inert strength value $\sigma_{c,f}$ is the strength of a specimen that would fail directly at the dwell time $t_f = t_{dw}$. The related stress intensity factor, K_{dw} , at the start of the static hold, is simply given by

$$\frac{K_{dw}}{K_{lc}} = \frac{\sigma_{dw}}{\sigma_{c,f}}$$
(2a)

In the evaluation of (4a) we need that inert strength value, $\sigma_{c,f}$ that would cause failure at the lifetime $t_f = t_{dw}$ under the applied load σ_{dw} . This strength is limited by

$$\sigma_{c,1} \leq \sigma_{c,f} \leq \sigma_{c,2} \tag{5}$$

Since the strength data $\sigma_{c,1}$ and $\sigma_{c,2}$ are very close to each other, an approximate attempt is the use of the average of the two limits in (5), i.e. application of

$$\sigma_{c,f} \cong \frac{1}{2}(\sigma_{c,1} + \sigma_{c,2}) \tag{6}$$

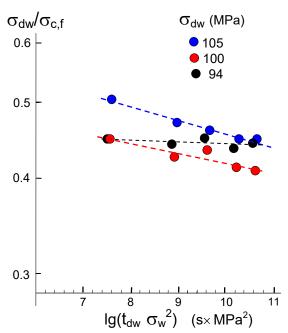


Fig. 2 Auxiliary plot used for the determination of the derivative term in eqs.(4, 4a).

For the determination of the logarithmic derivative in eq.(4a) it is of advantage to plot the data in the form of Fig. 2. The derivative can be obtained from the straight lines in this representation. The slopes are -0.00266 for $\sigma_{dw}=94$ MPa, -0.0179 for 100 MPa, and -0.0136 for 105 MPa. In the case of a power-law description assumed for subcritical crack growth, $V=A\times K^n$, the slopes would be equal to 1/(n-2). This would result in very high *n*-values of n=378 for 94 MPa, n=75 for 100 MPa, and n=58 for 105 MPa compared to $n\approx40$ as usual for silica. This discrepancy is typical for threshold behaviour.

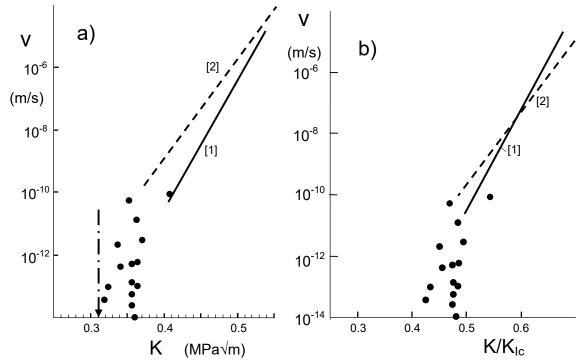


Fig. 3 Subcritical crack growth measurements on silica by Wiederhorn and Bolz [1] (DCB) and Michalske et al. [2] (DCDC), represented by the lines, solid circles: data obtained from the ISF-procedure by Sglavo and Green [3].

The results of the computations outlined in this Report are included in Fig. 3. The crack velocities obtained with eq.(4a) are introduced in Fig. 3 as the solid circles. The dashed line is a trend line. Figure 3a shows as the curves subcritical crack growth results from literature for which the stress intensity factors were available in form of handbook solutions. From this plot, a fairly good agreement with the data by Wiederhorn and Bolz [1] and Michalske et al. [2] can be stated at about $v=10^{-10}$ m/s. The minimum crack-growth rates are at about 10^{-14} m/s. They indicate a threshold for subcritical crack growth of $K_{th} \cong 0.31$ MPa \sqrt{m} .

In order to get results independent of the slightly different K_{Ic} -values reported in literature (K_{Ic} =0.72-0.80 MPa $\sqrt{\text{m}}$ for data in Fig. 3), the abscissa in Fig. 3b is given in normalized form by K/K_{Ic} .

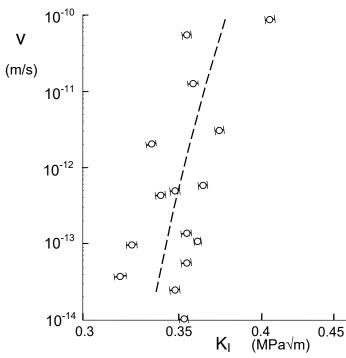


Fig. 4 v-K-data from ISF data [3] together with the error bars resulting by the approximation of eq.(6).

The possible errors ranges due to the approximation by eq.(6) are shown in Fig. 4. Whereas the circles are again determined by using $\sigma_{c,f}$ in eq.(4a), the right end of the error bars was computed by use of $\sigma_{c,1}$ and the left end by use of $\sigma_{c,2}$. The slope of the error bars comes from the fact that the deviating strengths affect the *K*-values and the crack rates *v* simultaneously.

3. Threshold stress intensity factor

The threshold value obtained in this study is close to the result in [3] based on the same lifetime data measured in liquid water (i.e. under relative humidity of 100%), where the threshold value was evaluated as the stress intensity factor that is applied in the lifetime tests at the start of the static load to the weakest specimen that survives this phase of the test. It has to be expected that the threshold is dependent on temperature. Therefore, the two results are introduced in Fig. 5 (solid circles) where the threshold stress intensity factor is plotted versus reciprocal absolute temperature. The open circles represent the measurements of the static fatigue limit by Aaldenberg

and Lezzi [6] in lab air at a relative humidity of 40-60 % for cracks slowing down and arresting. The straight line is an interpolation that shows the general temperature trend of increasing threshold with increasing temperature.

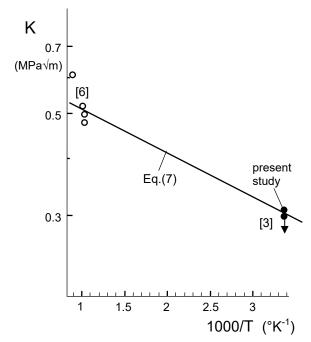


Fig. 5 Threshold of subcritical crack growth, solid circles: data obtained from the ISF-procedure by Sglavo and Green [3] and the present study (100% relative humidity), open circles: crack arrest measurements by Aaldenberg and Lezzi [6] (40-60% relative humidity).

For a more realistic comparison of the measurements, it would be necessary to determine the threshold stress intensity factors at the same water vapour pressure. In this context, it has to be noted that for a common relative humidity of about 50%, the results from Sglavo and Green [3] and from this study must shift slightly downwards as indicated by the arrow in Fig. 5. Ignoring this humidity effect the threshold stress intensity factor as a function of temperature θ may be described by an Arrhenius dependency

$$K_{th} \cong K_{th,0} \exp\left[\frac{-Q}{RT}\right]$$
(7)

with the absolute temperature $T=273^{\circ}+\theta$ and the parameters $K_{\text{th},0}=0.638$ MPa $\sqrt{\text{m}}$ and Q=1.83 kJ/mol (line in Fig. 5).

References

1 S.M. Wiederhorn and L.H. Bolz, Stress Corrosion and Static Fatigue of Glass, J. Am. Ceram. Soc. **53**(1970) 543-548.

2 Michalske, T.A., Smith, W.L., Bunker, B.C., Fatigue mechanisms in high-strength silicaglass fibers, J. Am. Ceram. Soc., **74**(1991), 1993-96.

3 V.M. Sglavo and D.J. Green, "Fatigue limit in fused silica," J. Eur. Ceram. Soc. **21** (2001) 561-567.

4 Fett, T., Munz, D., Determination of v- K_I -curves by a modified evaluation of lifetime measurements in static bending tests, Comm. Am. Ceram. Soc. **68** (1985), C213–C215.

5 Thoman, D.R., Bain, L.J., Antle, C.E. (1969): Inferences on the parameters of the Weibull distribution, Technometrics 11, 445.

6 J. S. Aaldenberg, P. J. Lezzi, Measurement of the silica glass fatigue limit, J. Am. Ceram. Soc., **103**(2020), 3097-3103.

KIT Scientific Working Papers ISSN 2194-1629 **WWW.kit.edu**