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Fatigue limit in fused silica

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Abstract

The threshold stress intensity factor for sub-critical crack growth in fused silica was measured using the interrupted static fatigue (ISF) test and from observations of indentation crack growth under load. Good agreement was obtained between the two techniques with values of 0.31–0.34 MPa$\sqrt{}$m and 0.30±0.02 MPa$\sqrt{}$m, respectively. The relative advantages of the two experimental approaches are discussed.

Keywords: Crack growth; Fatigue; Glasses; Indentation; SiO_2

1. Introduction

Fused silica glass is an essential material in several modern technological applications. The optical fibres used in telecommunications or the dielectric present in many microelectronic devices are two such examples. In these various applications, the silica is subjected to mechanical loading that can be static or cyclic. These stresses can lead to the premature failure of the component, especially when loading occurs in a corrosive environment. Since even humid air represents a corrosive environment for silica, this is a common situation. The phenomenon which determines the premature failure of a component under the contemporaneous action of load and of a corrosion process is known as fatigue and is common for most ceramics and glasses.[1–3] The design of devices or structural components with such materials is usually performed by the assumption of a minimum lifetime which, in turn, determines the maximum applied load. If higher loads are applied, failure will occur in shorter times. An important aspect of this process is that failure occurs in a subtle manner, in the sense that the sub-critical growth of the critical defect only becomes evident in the last instants of the fracture process, without any prior warning.

In some cases, the fatigue phenomenon has been shown to possess a lower bound corresponding to a minimum load below which sub-critical growth of defects does not occur.[3–18] This lower limit has been quantified in terms of a stress intensity factor, K_{th}, called the threshold stress intensity factor or the fatigue limit, below which, the crack velocity is zero. Values of K_{th} have been proposed for several glasses in humid environments and for some ceramics.[5–19] Unfortunately, in spite of the technological importance of silica glass, no fatigue limit data has been clearly identified or measured for this material. Recently, Ritter et al. measured crack arrest in DCDC specimens at about 0.4 MPa$\sqrt{}$m.[15] Similar values were calculated by Cook on the basis of indentation–strength measurements.[15] Michalske and Fuller[20] and Lawn et al.[21] have suggested that crack healing can occur around 0.10–0.13 MPa$\sqrt{}$m for silica glass in humid air and presumably this occurs below the fatigue limit. Studies have also been performed for the analysis of the fatigue behaviour of silica glass, though some discrepancies between proposed fatigue laws are evident. Sub-critical crack growth kinetics have been established for both bulk fused silica[3,5,8,15,16,22–28] and silica optical fibres[29–36] in humid environments. For example, Muraoka and Abé[36] have measured crack velocities in optical fibres as low as 10^{-13} m/s without detecting a threshold. These authors showed their data fitted an exponential law based on chemical kinetics better than the ubiquitous empirical power law. This is in agreement with earlier observations by Michalske et al., who employed this idea to explain the discrepancies observed between the fatigue

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behaviour of bulk silica and high-strength silica fibres.[34]

In recent papers, Sglavo and Green proposed two different methodologies for the determination of K_{th} in glasses and ceramics.[13,14,18,37] In the first approach, the fatigue limit is determined from the strength of samples subjected to an interrupted static fatigue (ISF) test. In the second, K_{th} is calculated from the length of median cracks generated during the loading phase of Vickers indentation tests. In the current study, these two techniques are applied to fused silica samples with the aim of the determination of K_{th} in a water environment. For the indentation approach in the current study, the crack geometry was distinctly different from that observed in other silicate glasses[38,39] and this necessitated a modification of the data analysis. The results obtained from the study are also used to compare the relative advantages of the two approaches.

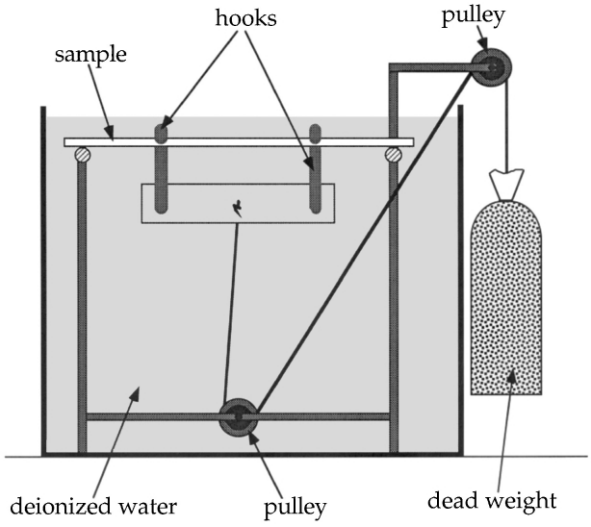

Fig. 1. Schematic of the apparatus used for ISF tests.

2. Experimental procedure

Silica glass (99.7 wt% pure) in the form of bars and rods was used in this work. Two different procedures were used to determine the fatigue limit of silica glass in a water environment. In the first approach, specimens were subjected to interrupted static fatigue in deionized water and the strength of the surviving samples was compared to that measured in an inert environment. In the alternative approach, Vickers indentations were introduced into glass surfaces in the presence of water. The cracks so formed were then compared to those produced in the inert environment.

2.1. Interrupted static fatigue tests

Silica glass in the form of rods, nominal diameter of 2 mm and length of about 90 mm, were used. The strength was measured by four point bending tests both in deionized water and in silicone oil. This latter liquid was considered to be the inert environment, preventing the access of water to the crack tip.[13,17,39] In this latter case, samples were dried at 120°C for 2 h before being completely immersed and fractured in a silicone oil bath. Analogously, a deionized water bath was used for the tests as the active environment. Bending tests were conducted with inner and outer span equal to 40 and 80 mm, respectively, using a displacement rate of 20 mm/min. For the strength testing, 30 specimens were broken in both environments.

Interrupted static fatigue (ISF) tests were conducted in deionized water using the apparatus shown schematically in Fig. 1. Both the frame and the pulleys were fabricated from stainless steel. The apparatus consisted of a series of frames like the one shown in Fig. 1. These frames were joined side by side in order to allow 15 samples to be tested simultaneously. The dead weight was applied such that the stress on the specimens during the constant stress phase of the ISF test was equal to 85, 94, 100 and 105 MPa. The load was maintained for times ranging from 1 h to 50 days. Fifteen to 30 samples were tested at each load or time condition. The samples that survived the ISF test were then broken in deionized water using a displacement rate of 20 mm/min.

2.2. Indentation tests

Silica plates with a nominal thickness of 3 mm were used for the indentation tests. Bars of 4–5 mm in width were machined from the as-received sheets. The edges of the bars were polished with diamond paste in order to remove macroscopic defects. Vickers indentations were introduced on the surface of the bars. Care was taken to place the indentation in the middle of the bar and to align one of the indenter diagonals perpendicular to the sample edges. The indenter movement was controlled such that the maximum load was reached in about 10 s. Indentations were performed in both silicone oil and deionized water. In the former case, samples were dried according to the procedure previously described and a drop of oil was immediately placed on the surface of the bar at the location where the indentation was to be introduced. The indenter was then maintained on the surface of the sample for 15 s at the maximum load. Maximum loads ranging from 1.96 to 39.2 N were used. Indentations in water were obtained after placing a drop of the water on the surface of the bar. In this case, the load was maintained for times ranging from 15 s to 8 days. For the longest times, additional water drops were added to the indentation site during the test. After the removal of the indenter, samples were cleaned and broken manually using the indentation crack as the critical

defect. The fracture surface was then observed using an optical microscope.

3. Results

3.1. Interrupted static fatigue tests

The strength distributions of the glass rods measured in silicone oil and deionized water are reported in Fig. 2. The strength data are shown as a Weibull plot in which the failure probability, F, is defined as:

$$F = \frac{j}{N+1} \tag{1}$$

where j is the rank in the ascending ordered strength distribution and N the total number of samples (30). The Weibull modulus values in silicone oil and deionized water are 6.0±1.2 and 6.9±1.3, respectively.

The strength of the specimens that survived the ISF tests was also reported as a Weibull plot. Fig. 3 shows a comparison of the strengths with and without the static hold for one of the test conditions. For the construction of the Weibull plot, the complete set of specimens initially subjected to the interrupted static fatigue test was considered and a strength of zero was assigned to samples which fail during the hold period of the ISF test.[13,14,17] In a previous paper, Sglavo and Green performed a theoretical analysis of the ISF test and proposed a procedure for the determination of the fatigue limit from the strength distribution of samples subjected to interrupted static fatigue.[14,37] The potential K_{th} is evaluated as the stress intensity factor, K_W, that is applied at the start of the static hold to the weakest specimen that survives this phase of the test and is given by[13,37]

$$K_W = K_C\left(\frac{\sigma}{S_0}\right) \tag{2}$$

where σ is the hold stress, K_C is the fracture toughness (0.75 MPa$\sqrt{m}$)[3] and S_0 is the inert strength for sample with the same failure probability. This calculation is performed for increasing duration in the ISF test and when K_W becomes independent of time, it can be considered as the fatigue limit.[13,14,37]

The results of this computing procedure are shown in Fig. 4. The data corresponding to applied hold stress of 85 MPa were not considered in the present discussion because only two, three and one specimens failed during the ISF tests for times of 10, 20 and 50 days, respectively. For shorter times no specimens failed during the stress hold. It is considered that such situations give rise to large errors in the K_W calculation.[37] It is important to point out that K_W evaluation is theoretically unaffected by the particular choice of failure probability [Eq. (1)] since the characteristic inert strength depends only on the rank in the ordered distribution.[37] Calculation

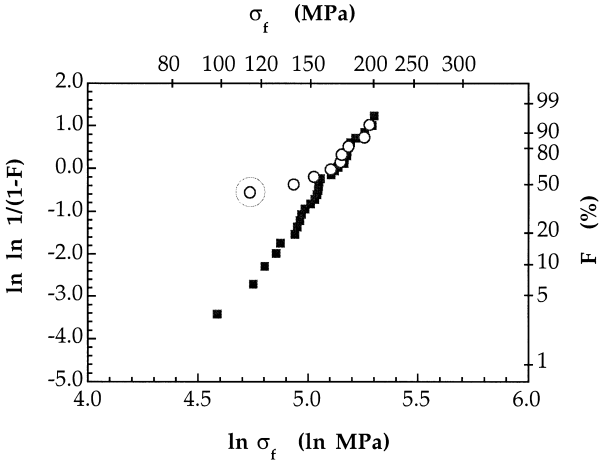

Fig. 3. Weibull plot of the strength measured in deionized water for samples subjected to ISF test with duration of 5 days and applied stress of 94 MPa. The weakest survivor is highlighted by a circle. The initial strength distribution (solid symbols) is shown for comparison.

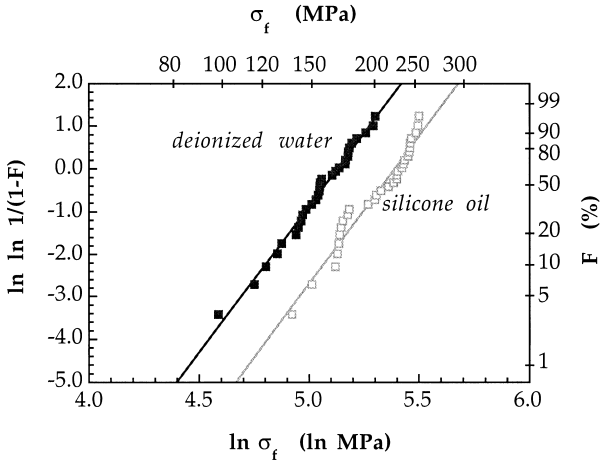

Fig. 2. Weibull plot of the strength measured in deionized water and silicone oil.

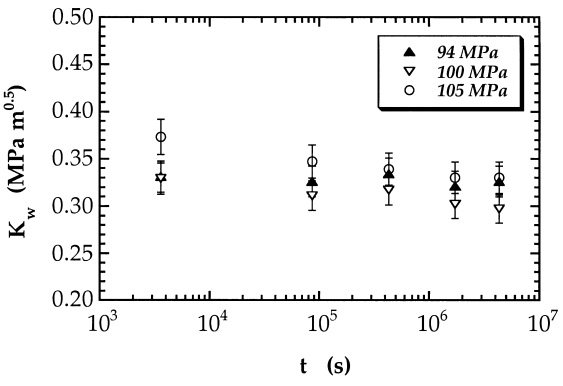

Fig. 4. K_W data as function of dwell time for the constant stress.

performed by using $F=(j-0.5)/N$ furnished results equal to those shown in Figs. 2–4, except for the Weibull moduli, which gave results equal to 7.6±1.6 and 7.7±1.6 in water and silicone oil, respectively. Typical uncertainty in K is ±5% and this was used to calculate the error bars shown in Fig. 4. The K_W data in Fig. 4, that are threshold estimates, clearly approach a limiting value in the range of 0.31–0.34 MPa$\sqrt{m}$.

3.2. Indentation tests

The sub-surface pattern of deformation and fracture produced by Vickers indentation in fused silica is shown in Fig. 5. The main crack is conical and it initiates outside the plastic zone. This observation is in agreement with previous studies in which silica was defined as an "anomalous" glass.[38,40] For these glasses, plastic deformation is associated with densification processes occurring within the plastic zone, which "blunts" the contact. This behaviour promotes formation of cone cracks instead of the median–radial cracks normally associated with sharp indentations.

The size of cone crack formed in water increased with the duration of the indentation test. In order to determine the possibility of a fatigue limit, the growth of the cone cracks was determined as a function of indentation time. Fig. 6 shows the length of the cone crack, l, as a function of the duration of the maximum indentation load. For both indentation loads, 9.8 and 39.2 N, the crack length increases as testing time increases at least up to ≈100 ks. For longer indentation times crack arrest appears as the fatigue limit is reached, most noticeably for the indentation force of 39.2 N. For quantitative evaluation of the fatigue limit, the stress intensity factor applied to the cone crack during the indentation test must be analytically defined. Fig. 7 shows the plot of log P vs log l data determined from indentations performed in silicone oil. A straight line with slope equal to 1.5 fits the experimental data. Therefore, the stress intensity factor applied to the cone crack can be defined as:

$$K=\xi\frac{P}{l^{1.5}} \tag{3}$$

where the factor ξ can be evaluated by linear regression of the data in Fig. 7 and the fracture toughness of silica glass. The value obtained by this procedure is $\xi=0.046$. The relation expressed by Eq. (3) is similar to the stress intensity factor proposed for Hertzian cone cracks and median–radial cracks produced by Vickers indentation.[3]

The factor ξ defines the intensity of the stress field around the indentation site upon loading and, therefore, can be considered as invariant as the crack length increases during a constant load test. On the basis of Eq. (3), the stress intensity factor applied to the cone cracks reported in Fig. 6 was evaluated. The results are shown in Fig. 8. Similar values of K are obtained at the same testing time for the two indentation loads. This represents a further confirmation of Eq. (3). For time durations longer than 100 ks an invariant stress intensity factor is reached. This allows one to define a fatigue limit of approximately 0.29–0.31 MPa$\sqrt{m}$ for the fused silica glass used in this work. More precisely, if the complete set of crack lengths recorded at $t\geqslant 2$ days is considered, the threshold stress intensity factor is determined to be

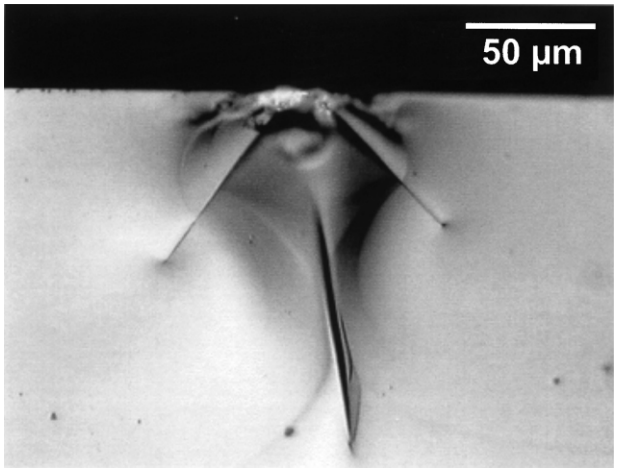

Fig. 5. Typical cone crack obtained by Vickers indentation in silica glass ($P=9.8$ N, $t=120$ s).

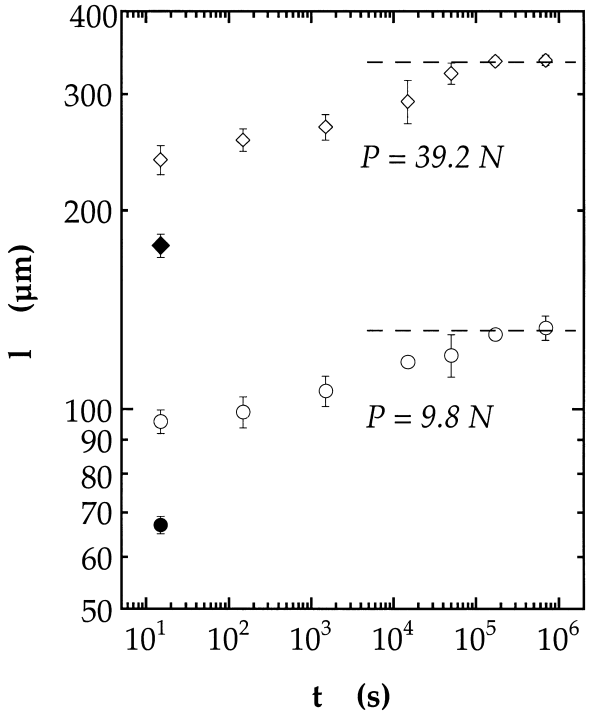

Fig. 6. Cone crack length as a function of loading time for indentation loads of 9.8 and 39.2 N. Solid symbols represent crack length obtained in silicone oil.

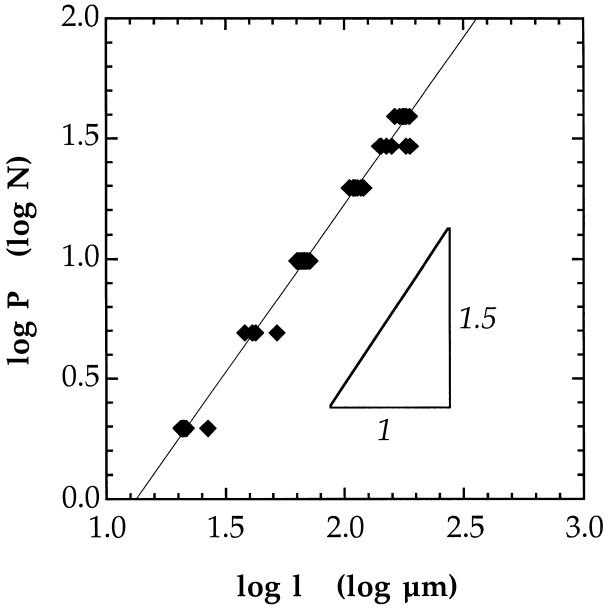

Fig. 7. Plot of log P as a function of log l from indentation tests performed in silicone oil.

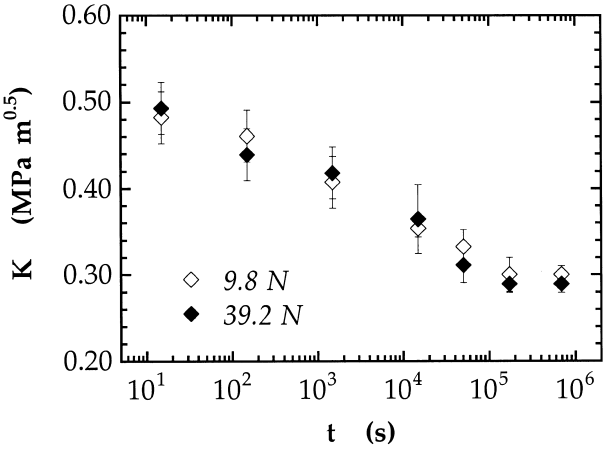

Fig. 8. Stress intensity factor applied to indentation cone cracks as a function of time at maximum load.

0.30±0.02 MPa$\sqrt{}$m. In order to point out the validity of the results obtained by indentation, a statistical test was performed on K data gathered at times equal to 50.4 ks (14 h), 172.8 ks (2 days) and 691.2 ks (8 days). The aim was to demonstrate that K data corresponding to the two highest duration represent the same number and are different from K results obtained at $t=14$ h. Typical statistical approaches based on hypothesis testing were used.[41] For both indentation loads, K values at $t=172.8$ ks and 691.2 ks represent the same number (0.30 MPa$\sqrt{}$m) with a probability of a Type II error lower than 20% for a difference of 0.03 MPa$\sqrt{}$m from the average. Conversely, values calculated at $t=50.4$ ks are larger than K data at $t=691.2$ ks at a 0.5% level of significance (error Type I). Therefore, one can claim with a confidence higher than 80% that crack arrest has been detected during indentation tests performed with duration equal to or in excess of 691.2 ks.

Additional information on the fatigue behaviour of the glass can be gathered from the data in Fig. 6. Crack length values recorded at times less than 100 ks correspond to the sub-critical growth of cracks under a specific stress field. The sub-critical crack velocity can be expressed as:

$$\frac{dl}{dt} = v_0\left(\frac{K}{K_C}\right)^n \tag{4}$$

where v_0 and n are parameters which depend on the material and the environment. Eq. (4) can be combined with Eq. (3) to obtain a relation between the crack length and indentation time, t, as:[39]

$$l \cong l_0\left(hv_0\frac{t}{l_0}\right)^{1/h} \tag{5}$$

where l_0 is the cone length in silicone oil and $h=1.5\ n+1$. Eq. (5) can be easily transformed into a linear relation:

$$\log l = \log\left[l_0\left(\frac{hv_0}{l_0}\right)^{1/h}\right] + \frac{1}{h}\log t. \tag{6}$$

The experimental data shown in Fig. 6 for $t<100$ ks were expressed in terms of log l vs log t and linear regression was used to determine n and v_0. Values equal to 19.7±2.9 and 3.9±1.4 mm/s were obtained for n and v_0, respectively, from crack length data obtained with $P=39.2$ N. Correspondingly, values equal to 22.0±3.4 and 26.9±12.6 mm/s were calculated for $P=9.8$ N.

It is worth comparing the fatigue parameters obtained here with previous results. Two distinct ranges of n data can be found in the literature for silica glass. Values of n ranging from 14 to 26 have been obtained by static or dynamic fatigue tests on silica fibres in humid environments.[29,31,32,34,36] Conversely, direct measurements of the large crack velocities in bulk silica specimens furnished n values between 36 and 44. Dabbs and Lawn determined values of n equal to 25 and 40 by dynamic fatigue tests on silica fibres in which sub-threshold and post-threshold indentation cracks, respectively, had been produced.[32] The effect of the indentation residual stress field, which is much stronger for sub-threshold cracks, was used to account for the reduction of n from 40 to 25. An alternative explanation for the different values of n was advanced by Muraoka and Abé.[36] These researchers measured the sub-critical growth of indentation cracks in silica fibres by direct observation and calculated values of n around 23 and 40 for K intervals 0.3 to 0.45 and 0.3 to 0.6 MPa$\sqrt{}$m, respectively.[36] This

proposed dependence of n on the sub-critical crack growth velocity or on K is in agreement with previous results by Michalske et al.[34] Similarly, Cook has proposed that the values of n may be lower when the lower part of the $v(K)$ curve is explored.[40] On this basis, one would expect the n values in the indentation tests to be in the lower range since the measurements were made at low crack velocities, in agreement with the current data for the n values.

The calculated values for v_0 reported earlier are different for the two indentation loads. This should not be too surprising. In most papers dealing with fatigue, v_0 values are not reported and the fatigue behaviour is described only by the n parameter. The calculation of v_0 uses an evaluation of a power (e^x or 10^x) where small differences in the argument can develop into large variations in the final result. The difference in the v_0 values is mainly a reflection of the difference in the n values.

4. Discussion

There was good agreement between the two techniques used for the determination of the fatigue limit of silica glass in the water environment. Interrupted static fatigue tests furnished values of K_{th} around 0.31–0.34 MPa$\sqrt{}$m while a value of 0.30±0.02 MPa$\sqrt{}$m was obtained by the indentation procedure. At this point, it is useful to discuss the results shown in Figs. 4 and 8 in more detail.

The ISF tests were performed using quite long times (up to 50 days) and a clear trend (Fig. 4) in K_W towards a range from 0.31 to 0.34 MPa$\sqrt{}$m was identified. For hold stresses 94 MPa or greater, the threshold is reached at $t\sim100$ ks. The choice for the constant stress in the ISF test plays an important role in this test. For the smallest sampling errors, a similar number of samples need to fail in the stress hold compared to those that survive and this only occurs for the higher hold stresses. The main disadvantage of the ISF test is the large number of specimens that are needed, especially when there is large strength variability. The theoretical analysis of Sglavo and Green[37] showed that a hold time of 20 ks would be necessary for a specimen (of average strength) to fail without the presence of a threshold when subjected to $K=0.30$ MPa$\sqrt{}$m at the start of the hold period. This suggests that the hold times used in the ISF were long enough to detect the threshold if this were its value.

The indentation data clearly has the advantage of involving less experimental scatter and involves using a small number of specimens. It is, however, clear from Fig. 4 that testing times in excess of a 10^5–10^6 s are needed for fused silica. The indentation method is a simple technique whose results are easily analysed and give less experimental scatter. In addition, the sub-critical growth of the cracks can be directly observed. Unfortunately, this technique can be applied only to materials in which indentation cracks can be easily observed. The interrupted static fatigue test presents the advantage of being applicable to any material and at high temperature. The difficulty is that it can involve testing a large number of specimens.

5. Conclusions

Two techniques were used to determine whether a fatigue limit occurs in fused silica. In the first approach the interrupted static fatigue test was used. In this technique, determining the strength of the weakest sample that survives the constant stress phase of the test identifies the threshold. A value for the threshold in the range 0.31–0.34 MPa$\sqrt{}$m was obtained. In the second approach, the growth of cone cracks under indentation load was used to determine the sub-critical crack growth parameters and the threshold value. For this latter technique, a threshold value of 0.30±0.02 MPa$\sqrt{}$m was obtained. Of the two techniques, the indentation approach involved less experimental scatter, fewer specimens and direct observation of crack growth. Unfortunately, this technique, cannot be readily applied to all materials, especially at high temperature and it was found to require longer testing times.

Acknowledgements

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