Journal of the European Ceramic Society **12** (1993) 487–491

A Fracture Statistics Estimate of the Fatigue Limit of a Borosilicate Glass

Kunio Hayashi

Department of Chemistry and Materials Technology, Kyoto Institute of Technology, Matsugasaki, Sakyo-ku, Kyoto, 606 Japan

Timothy E. Easler

Dow Corning Corp., 3901 South Saginaw Road, Michigan 48686-0995, USA

&

Richard C. Bradt

Mackay School of Mines, University of Nevada, Reno, Nevada 89587-0136, USA

(Received 8 January 1993; revised version received 3 June 1993; accepted 15 June 1993)

Abstract

The fatigue limit of a borosilicate glass was experimentally determined by imposing a series of holding-stresses at intermediate stress levels during conventional direct-to-failure strength tests. This technique results in a distinct change in the low strength, large flaw size portion of the strength distribution of the survivors. The break-point for this change in the strength distribution was used to estimate the room temperature fatigue or stress corrosion limit (K_{IO}) of the glass in distilled water. A K_{IO} of 0·21 MPa m$^{1/2}$ was estimated and is compared with other estimates of the fatigue limit in other and similar glasses.

Die Ermüdungsgrenze von Borsilikatglas wurde experimentell durch eine Serie von Haltespannungen im mittleren Spannungsbereich während konventioneller Versagenstests bestimmt. Diese Technik ergibt einen klaren Umschlag in der Festigkeitsverteilung nicht gebrochener Proben mit großen Defekten und geringer Festigkeit. Der Umschlagspunkt in der Festigkeitsverteilung wurde zur Abschätzung der Ermüdungsgrenze bei Raumtemperatur oder der Spannungskorrosion (K_{IO}) des Glases in destilliertem Wasser verwendet. Ein K_{IO}-Wert von 0·21 MPa m$^{1/2}$ wurde berechnet und mit anderen Abschätzungen der Ermüdungsgrenze in anderen oder ähnlichen Gläsern verglichen.

On a determiné expérimentalement la limite de fatigue d'un verre de borosilicate en imposant, lors de tests conventionnels de rupture directe, des paliers de contrainte pour des valeurs intermédiaires de la contrainte. Cette technique modifie clairement la distribution en résistance des échantillons qui ont survécu, plus précisément la partie de cette distribution concernant les échantillons de faible résistance, contenant des défauts de grande taille. Le point où se produit ce changement dans la distribution des résistances a été utilisé pour estimer la limite de fatigue à température ambiante, on limite en corrosion K_{IO} du verre dans de l'eau distillée. Le calcul donne un K_{IO} de 0·21 MPa m$^{1/2}$, valeur que l'on compare à d'autres estimations de la limite de fatigue, dans d'autres verres ou des verres similaires.

1 Introduction

The time-dependent, stress-dependent, environment-dependent failure of glasses at stresses well below the rapidly measured fracture stress is an established fact.[1–6] It has been generally recognized that this fatigue or weakening process is related to stress corrosion processes which lead to an increased severity of the flaws that are present in the material until one of the flaws achieves a critical condition and catastrophic failure occurs.

Engineering design of structures with materials susceptible to this phenomenon usually proceeds either through the estimation of an acceptable stress level to assure a minimum time to failure for the anticipated campaign of the component, or through

Journal of the European Ceramic Society 0955-2219/93/$6.00

the application of the design criterion of an allowable stress that is below the fatigue limit, K_{IO}, that stress intensity level below which fatigue processes do not occur.[7] From an engineering design viewpoint, the existence of a fatigue or stress corrosion limit is widely recognized as being highly desirable for it allows the calculation of that stress level below which dealyed failure will not occur. However, the direct experimental determination of the fatigue limit has proven to be a rather formidable challenge. This paper reports an experimental estimate of the fatigue limit of a commercial borosilicate glass at room temperature in distilled water. A technique based on changes in the strength distribution which is related to the experiments pioneered by Wilkins and Dutton at 673 K is applied.[8]

2 Experimental

The borosilicate glass used in this study was a commercial material. The chemical composition of the glass was shown in Table 1. It was chosen because of its ready availability in cane form which is suitable for strength measurements, and also because its fracture toughness at room temperature is essentially the same as at the temperature of liquid nitrogen, as has been reported by Wiederhorn.[9] Cane over 1 m in length and 4 mm in diameter were cut to 80 mm rod specimens then annealed in air for 15 min at 843 K and slowly cooled to relieve the residual stresses present in the as-received glass. After this initial annealing, the specimens were abraded by ball milling with −240 mesh SiC powder for 15 min to develop a more uniform flaw distribution. The specimens were then annealed again to remove any residual stresses which may have developed at the flaws introduced by the abrasion process. Polaroscopic examination of the rods after this preparation procedure revealed that the specimens were free of residual stresses.

To establish the necessary baseline data for subsequent fatigue limit estimates, series of 50 specimens each of these rods were broken in four-point bending, one-quarter point loading over a 57·2 mm span at a crosshead speed of 1·27 mm/min by using the apparatus as shown in Fig. 1. One series of 50 specimens was broken under liquid nitrogen at 77 K and the other series of 50 specimens under distilled water at 296 K. Those results were presented on the familiar Weibull coordinates. To estimate

Table 1. Chemical composition (wt%) of borosilicate glass used

SiO_2	B_2O_3	Al_2O_3	Na_2O	K_2O	MgO	CaO
80·8	12·0	2·0	4·2	0·6	0·2	0·2

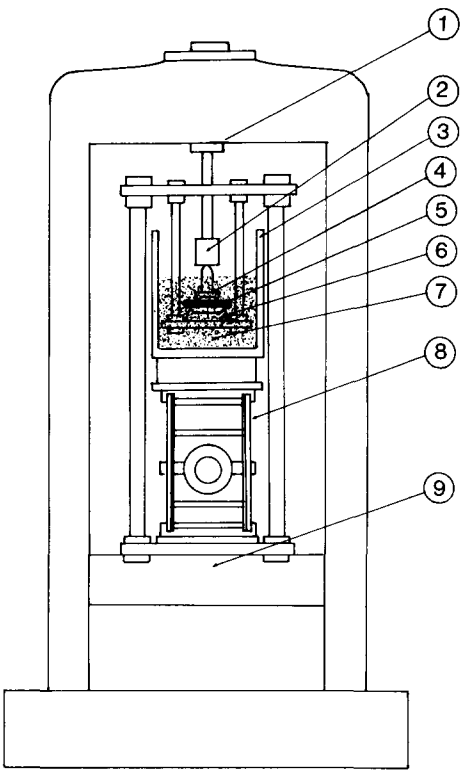

Fig. 1. Schamatic showing of apparatus for measuring four-point bending strength and holding at various stress levels of specimens in liquid nitrogen and in distilled water. 1, Load cell; 2, universal joint; 3, container; 4, upper edge; 5, specimen; 6, lower edge; 7, liquid nitrogen or distilled water; 8, jack; 9, crosshead arm.

the fatigue limit of this glass at room temperature, 296 K, in distilled water, a series of interrupted strength measurements involving a low-level holding stress were completed. Instead of directly loading to failure in the distilled water, the loading was interrupted and the stress was held at a constant low level (24–60 MPa) for 10 min. Loading was then continued to failure and the strength distributions were also presented on the familiar Weibull coordinates. Using a technique based on changes in the strength distribution, a K_{IO} of the glass at room temperature in distilled water was estimated.

3 Results and Discussion

Figure 2 illustrates the strength results obtained under liquid nitrogen and in distilled water, a form which was chosen to accent the low strength portion of the strength distribution. It is evident that the distilled water results are at much lower strength levels. The average strengths are 180·6 and 91·4 MPa, respectively. The Weibull moduli, m, of 5·7 and 5·5 are very similar. This suggests that comparable amounts of slow crack growth occurred for all the strength levels. Applying Gupta's equation[10] obtained from strain-rate dependence of strength and an iteration process using the data in Fig. 2 yields the stress corrosion susceptibility coefficient, N, of 22·0

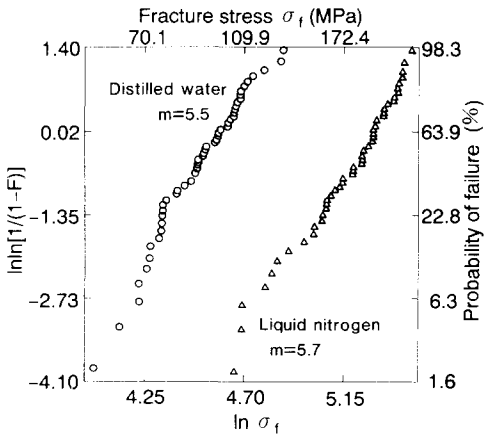

Fig. 2. The strength distributions in liquid nitrogen at 77 K and in distilled water at 296 K.

for this glass. It represents the susceptibility to stress corrosion and is similar to the values summarized for a number of similar borosilicate glasses by Freiman[11] and by Zdaniewski *et al.*[12]

The results are summarized in Fig. 3 for holding stresses of 24, 36, 48, and 60 MPa along with the direct-to-fracture strength distribution previously depicted in Fig. 2. Although the stress scale has been expanded and there exists some statistical scatter along the stress axis, the high-strength portions of the distributions with the holding stresses are parallel. It is, however, the abrupt breaks and strength decreases of the low-strength specimens which suggest the existence of a K_{IO}. This strength decrease of low-strength specimens subjected to holding-stresses is analogous to the results of Baker and Preston[13] who demonstrated that 'understressing' results in premature failure in static fatigue tests of glass.

The 24 MPa holding-stress strength distribution remained essentially unchanged compared to that for direct strength measurements; however, the distributions determined after holding stresses of 36, 48, and 60 MPa were applied to all exhibited distinct breaks at the low strength tail compared to the direct-to-fracture distributions. In the latter two of these, two and eight specimens respectively actually failed during the 10 min holding-stress period; thus those failed specimens which would have had low probability points are not included in Fig. 3. As the holding stress was increased, the number of low-strength specimens exhibiting distinct strength decreases increased: 6, 10, and 14 for the latter three. The number of specimens failing during the holding-stress period also increased from 0 to 2 to 12. Obviously, a more extended series of systematic experiments at different hold times similar to the study of Michalske[14] on crack aging could provide data for additional analysis.

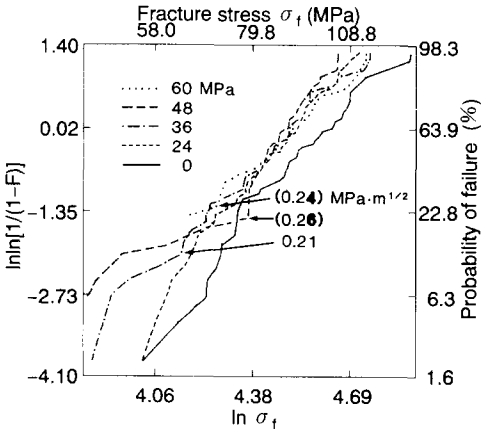

Fig. 3. The strength distributions for testing in distilled water at 296 K and for holding at various stress levels (σ_a). Distribution breaks and estimated K_{IO} values are included.

From these results it is possible to estimate the K_{IO} for this glass at 296 K in distilled water. During the strength measurements at 296 K in distilled water reported in Fig. 2, the flaws are initially at zero stress intensity prior to loading, but the stress intensity increases during the test. When K_{IO} is reached, slow crack growth commences and proceeds during the remainder of the test until the critical stress intensity factor, K_{IC}, is achieved and failure occurs. If, however, an intermediate holding stress at a level yielding a stress intensity factor, K_I, which is greater than K_{IO} is imposed during the test, then flaw extension in addition to that which occurs during the direct loading-to-failure strength measurement may be expected to occur. Upon further direct loading to the fracture stress, the strength will be less than if no intermediate holding stress were imposed. However, if the holding-stress intensity level is below K_{IO}, no additional flaw growth or extension is to be expected. This latter example is characteristic of the 24 MPa stress level in Fig. 3. The other three intermediate holding stresses of 36, 48, and 60 MPa all exhibit the characteristic of exceeding the K_{IO} with the holding stresses for specimens were weakened and some actually failed.

At the 36 MPa holding stress, the six specimens with a probability of fracture, P_f, below 11·8% exhibited obvious strength decreases. While those above a P_f of 11·8% did not change noticeably from the original distribution. One would then attribute the strength decreases at P_f below 11·8% to loading at a stress intensity level above K_{IO} during the intermediate holding-stress period and the lack of any change above a P_f of 11·8% to being below K_{IO}. This interpretation is reasonable for the low-strength specimens at a P_f below 11·8% have the most severe flaws, while those above 11·8% are less severe. According to

$$K_I = \sigma_f c^{1/2} Y \qquad (1)$$

the larger flaw size specimens, those at the low strength tail of the distribution, would be expected to experience the highest stress intensities during the holding-stress period. It is then possible to estimate the K_{IO} of this glass from the results, using the break

from the trend established by the distribution without an intermediate holding stress.

Wiederhorn's results indicate that the K_{IC} of this glass is essentially the same at 77 K in liquid nitrogen as at room temperature in a moist environment. However, the precise cause of fatigue in strength specimens remains controversial for in eqn (1), the flaw dimension c can increase and the geometry parameter Y can also change. A direct correspondence of fracture mechanics and strength studies would suggest that it is the flaw dimension term which dominates; however, Doremus[15] argues that crack tip sharpening is the chief cause of weakening. Michalske[14] suggests a combination of crack tip sharpening followed by flaw growth. This controversial point is not addressed in this paper. Only an estimate of K_{IO} from the data in Fig. 3 is considered.

From the liquid nitrogen data in Fig. 2, it is possible to estimate the initial value of the $(c^{1/2}Y)$ term of eqn (1) for the appropriate failure probability prior to any fatigue processes. Then applying the static holding stresses it is possible to estimate the K_{IO} values using the flaw sizes corresponding to the breaks in the distributions. Substituting the maximum value of strength σ_f (=230 MPa) in the liquid nitrogen data in Fig. 2 and the $K_{IC} = 0{\cdot}77\ \text{MPa m}^{1/2}$ of this glass[9] for σ_f and K_{IC} in eqn (1) yields the $(c^{1/2}Y)$ of $3{\cdot}34 \times 10^{-3}\ \text{m}^{1/2}$. Subsequently, substituting the $(c^{1/2}Y)$ values calculated and the strengths at the breaks in the distributions in Fig. 3 for σ_f in eqn (1) yields K_{IO} values of 0·21, 0·26 and 0·24 MPa m$^{1/2}$ for this glass at the increasing holding-stress levels of 36, 48 and 60 MPa, respectively. In Fig. 3, the latter two are put in parentheses for it is believed that they are too high an estimate of the K_{IO}. The reason 0·21 MPa m$^{1/2}$ is the preferred estimate of the fatigue or stress corrosion limit is simply that it is the lowest of the three, although the other two are also comparable to other literature values. The slight differences may also be due to the intrinsic character of the flaws. Although 0·21 MPa m$^{1/2}$ is the best estimate of K_{IO} from these results, it is evident that additional experiments based on a greater number of specimens in each statistical set, more closely spaced intermediate holding stresses, and also a series of longer holding times are desirable in order to gain a more precise estimate of K_{IO}.

It is appropriate to compare this estimate of K_{IO} to other fatigue limit measurements or estimates in the literature. In his recent review, Doremus[16] suggests that the fatigue limit may be about 20% of the liquid nitrogen strength, but that it is expected to vary with the chemical durability of the specific glass. This 20% level is in general agreement with Shand's classical study[17] and also with Mould and Southwick's[18] concept of a universal fatigue curve. If ratios are taken applying eqn (1), then an estimate of K_{IO} of about 20% of K_{IC} is reasonable. These are in general agreement with Wilkins and Dutton[8] who summarize (K_{IO}/K_{IC}) ratios for various glasses and report results ranging from about 0·15 to 0·40. There exists reliable fracture mechanics measurements, on soda-lime-silica glasses, which can be compared with the K_{IO}/K_{IC} ratio of $(0{\cdot}21/0{\cdot}77) = 0{\cdot}27$ observed for the borosilicate glass in this study. Recently Simmons and Freiman[19] have observed an apparent K_{IO} of about 0·27 MPa m$^{1/2}$ for a soda-lime-silica glass in water at room temperature. This compares favorably with Wiederhorn and Bolz's[20] steepening of their $(K_I$–$V)$ curves at about 0·25 MPa m$^{1/2}$, although they do not report actual crack arrest. Matsui *et al.*[21] report similar results of a K_{IO} of about 0·29 MPa m$^{1/2}$, while Michalske's results[14] suggest that about 0·225 MPa m$^{1/2}$ may be an appropriate K_{IO}. The fatigue results of Pavelchek and Doremus[22] suggest that soda-lime-silica glass exhibits a fatigue limit at about 0·23 of K_{IC}.

In summary, room temperature estimates of soda-lime glass suggest that the K_{IO} is between about 0·20 and 0·30 MPa m$^{1/2}$ or at a (K_{IO}/K_{IC}) ratio approximately between 0·25 and 0·40. However, strength measurements suggest that a lower limit of 0·20 may be more appropriate.

Borosilicate glass K_{IO} estimates are not so numerous. The steepening of the borosilicate $(K_I$–$V)$ curve of Wiederhorn and Bolz[20] occurs at about 0·32 MPa m$^{1/2}$. But Simmons and Freiman[19] do not show any $(K_I$–$V)$ steepening or any crack arrest in their borosilicate glass in water at 0·37 MPa m$^{1/2}$ and Ritter and Manthuruthil[2] did not observe any apparent fatigue limit at a ratio of about 0·3 in their static fatigue tests of a borosilicate glass. The 0·21 MPa m$^{1/2}$ value of K_{IO} estimated in this study is lower than any of these. It compares favorably with the lower limit of the measured (K_{IO}/K_{IC}) ratios reported for the soda-lime glass, about 0·27.

Although this statistical estimate of the K_{IO} is in general agreement with the values reported or implied in other studies, in this study there is no indication of strengthening of the glass as reported by Wilkins and Dutton[8] in their statistical study and also by Pranatis[23] in his dynamic fatigue report of 'coaxing' in this same commercial borosilicate glass, as well as the reports of the aging of abraded glass.[24] Michalske's observation[14] of time dependent increases of the K_I values necessary to restart arrested cracks is directly related to the same phenomena. The simplest explanation for this difference is that the particular experimental conditions applied in this study are not very favorable to the observation of strengthening. Wilkins and Dutton[8] did their experiment at 673 K where crack tip flow processes

are much more likely to occur, Pranatis' experiments[23] and the aging studies both were for times much greater than 10 min. The results of Michalske[14] confirm that much longer times are necessary to observe strengthening phenomena when stress remains on the specimens. Thus, the lack of any direct evidence for strengthening phenomena in this study do not contradict its observation in the other studies.

4 Conclusions

An experimental estimate of the fatigue limit, K_{IO}, of a commercial borosilicate glass at room temperature in distilled water was performed using a technique based on changes in the strength distribution. To establish the necessary baseline data for subsequent K_{IO} estimates, one series of 50 specimens was broken under liquid nitrogen at 77 K and the other series of 50 specimens under distilled water at 296 K and those results were presented on the familiar Weibull coordinates. The average strengths were 180·6 and 91·4 MPa, respectively, while the Weibull moduli of 5·7 and 5·5 were very similar.

To estimate the K_{IO} of this glass at room temperature, 296 K, in distilled water, the loading was interrupted and the stress was held at a constant low level ranging from 24 to 60 MPa for 10 min. Loading was then continued to failure. The strength distributions were also presented on the familiar Weibull coordinates. The 24 MPa holding-stress strength distribution remained essentially unchanged compared to that for direct strength measurements. On the other hand, the distributions determined after holding stresses of 36, 48, and 60 MPa all exhibited distinct breaks at the low strength tail compared to the direct-to-failure distributions. The break-point of this change in the strength distribution was used to estimate the room temperature K_{IO} of the glass in distilled water. A K_{IO} of 0·21 MPa $m^{1/2}$ was calculated and this value was comparable with other estimates of the fatigue limit in other glass studies.

References

1. Adams, R. & McMillan, P. W., Static fatigue in glass. *J. Mater. Soc.*, **12** (1977) 643–57.
2. Ritter, J. E. Jr & Manthuruthil, J., Static fatigue of silicate glasses. *Glass Technol.*, **14** (1973) 60–4.
3. Holland, A. J. Jr & Turner, W. E. S., Effect of sustained loading on breaking strength of sheet glass. *J. Soc. Glass Technol.*, **24** (1940) 46–57.
4. Ritter, J. E. Jr & LaPorte, R. P., Effect of test environment on stress-corrosion susceptibility of glass. *J. Am. Ceram. Soc.*, **58** (1975) 265–7.
5. Wiederhorn, S. M. & Johnson, H., Effect of electrolyte pH on crack propagation in glass. *J. Am. Ceram. Soc.*, **56** (1973) 192–7.
6. Freiman, S. W., Effect of alcohols on crack propagation in glass. *J. Am. Ceram. Soc.*, **57** (1974) 350–3.
7. Wiederhorn, S. W., Evans, A. G. & Roberts, D. E., A fracture mechanics study of the skylab windows. In *Fracture Mechanics of Ceramics* (Vol. 2), ed. R. C. Bradt, D. P. H. Hasselman & F. F. Lange. Plenum, New York, 1974, pp. 829–41.
8. Wilkins, B. J. S. & Dutton, R., Static fatigue limit with particular reference to glass. *J. Am. Ceram. Soc.*, **59** (1976) 108–12.
9. Wiederhorn, S. M., Fracture surface energy of glass. *J. Am. Ceram. Soc.*, **52** (1969) 99–105.
10. Gupta, P. K., Examination of the tensile strength of E-glass fiber in the context of slow crack growth. In *Fracture Mechanics of Ceramics* (Vol. 5), ed. R. C. Bradt, A. G. Evans, D. P. H. Hasselman & F. F. Lange. Plenum, New York, 1983, pp. 219–303.
11. Freiman, S. W., Fracture mechanics of glass. In *Glass Science and Technology* (Vol. 5), ed. D. R. Uhlmann & N. J. Kriedl. Academic Press, London, 1980, pp. 21–78.
12. Zdaniewski, W. A., Easler, T. E. & Bradt, R. C., Gamma radiation effects on the strength of a borosilicate glass. *J. Am. Ceram. Soc.*, **66** (1983) 311–13.
13. Baker, T. C. & Preston, F. W., Fatigue of glass under static loads. *J. Appl. Phys.*, **17** (1946) 170–8.
14. Michalske, T. A., The stress corrosion limit: Its measurement and implications. In *Fracture Mechanics of Ceramics* (Vol. 5), ed. R. C. Bradt, D. P. H. Hasselman, A. G. Evans & F. F. Lange. Plenum, 1983, pp. 277–89.
15. Doremus, R. H., Modification of the Hilling–Charles theory for static fatigue of glass. *Engng Fract. Mech.*, **13** (1980) 945–53.
16. Doremus, R. H., Fracture and fatigue of glass. In *Treat on Mat. Soc. and Tech.* (Vol. 22, Glass III), ed. R. H. Doremus & M. Tomazawa. Academic Press, London, 1982, pp. 169–239.
17. Shand, E. B., Experimental study of fracture of glass: I. The fracture process. *J. Am. Ceram. Soc.*, **37** (1954) 52–60.
18. Mould, R. E. & Southwick, R. D., Strength and static fatigue of abraded glass under controlled ambient conditions: II, Effect of various abrasions and the universal fatigue curve. *J. Am. Ceram. Soc.*, **42** (1959) 582–92.
19. Simmons, C. J. & Freiman, S. W., Effect of corrosion processes on subcritical crack growth in glass. *J. Am. Ceram. Soc.*, **64** (1981) 683–6.
20. Wiederhorn, S. M. & Bolz, L. H., Stress corrosion and static fatigue of glass. *J. Am. Ceram. Soc.*, **53** (1970) 543–8.
21. Matsui, M., Soma, T. & Oda, I., Subcritical crack growth in electrical porcelains. In *Fract. Mech. of Ceramics* (Vol. IV), ed. R. C. Bradt, D. P. H. Hasselman & F. F. Lange. Plenum, New York, 1978, pp. 711–24.
22. Pavelchek, E. K. & Doremus, R. H., Static fatigue in glass—A reappraisal. *J. Non-cryst. Sol.*, **20** (1976) 305–21.
23. Pranatis, A. L., Coaxing effect during the dynamic fatigue of glass. *J. Am. Ceram. Soc.*, **52** (1969) 340–1.
24. Mould, R. E., Strength and static fatigue of abraded glass under controlled ambient conditions: III, Ageing of fresh abrasions. *J. Am. Ceram. Soc.*, **43** (1960) 160–7.