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Dynamic fatigue of indented, soda-lime glass as a function of temperature

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Dynamic fatigue data as a function of temperature for indented-annealed and indented-aged soda-lime glass was analysed using an exponential crack velocity equation based on stress corrosion theory. Agreement in the crack velocity parameters as derived from the two sets of data indicates that the residual contact stress of the indented-aged samples was properly accounted for. However, the predicted macroscopic crack velocity curves based on the indentation fatigue data did not agree with those based on double cantilever beam measurements and possible reasons are discussed.

1. Introduction

Time-dependent strength (failure) behaviour of glasses is generally believed to be controlled by crack velocities in region I (low velocities) where crack growth is controlled by reaction kinetics at the crack tip [1]. Knowledge of the exact analytical form of the velocity–stress intensity dependency in this region is important when extrapolating short-term data to long lifetimes. The theory that has demonstrated the best capability for modelling stress-corrosion-governed crack growth is that due originally by Charles and Hillig [2] and later put into fracture mechanics terms by Wiederhorn [3–5]. Wiederhorn assumed that crack velocity is governed by the rate of chemical reaction at the crack tip and that atomically sharp cracks grow with a constant tip radius. He then derived that crack velocity (v) is related to the stress intensity factor (K_I) by:

$$v = a \exp(-E^*/RT) \exp(nK_I/K_c RT) \quad (1)$$

where E^* is an empirical measure of the zero stress activation energy of the stress corrosion reaction, R is the gas constant, T is the absolute temperature, K_c is the critical stress intensity factor, and a and n are constants. Wiederhorn determined the constants a, n, and E^* for a variety of glasses in water using double cantilever beam specimens.

Lawn and co-workers [6–8] have shown that the macroscopic crack velocity data of Wiederhorn [4], expressed in terms of a power-law function, could be used to predict the indentation flaw-strength behaviour of soda-lime and borosilicate glasses in water at room temperature under dynamic fatigue conditions (constant stressing rate). The purpose of the present study is to characterize the dynamic fatigue behaviour of indented soda-lime glass as a function of temperature in water. The results will determine whether the temperature dependence of fatigue for soda-lime glass in water is correctly represented by Equation 1. The indentation-strength technique has the advantages of reproducibility being relatively high as well as the results having direct relevance to the microhardness of naturally occurring flaws that can be subject to residual driving forces originating from their formation.

2. Experimental procedure

Test specimens were cut from sheets of soda-lime glass† and had nominal dimensions 57 mm

†Pittsburgh Plate Glass, Pittsburgh, Pennsylvania, USA.

square by 3 mm thick. Each specimen was indented in air at the centre of one face with a Vickers microhardness indenter using a standard load of 10 N for 10 sec. Each specimen was inspected microscopically to ensure that a well-defined radial crack pattern was formed. The specimens were then divided into two groups. One group was aged in distilled water for 12 h to normalize the effects of ageing before and during strength testing. The other group was annealed at 520° C for 24 h to relieve the post-indentation residual stress. After annealing the samples were then aged for 12 h in water before strength testing.

The dynamic fatigue behaviour of the samples was measured in distilled water at 5, 25, 55, and 85° C. For a given test temperature strength was determined with the indent in biaxial tension using a ring-on-ring test fixture (i.d. = 12.6 mm and o.d. = 29.8 mm) in conjunction with a universal testing machine‡ at four stressing rates ranging from 0.03 to 43 MPa $\sec^{-1}$. The appropriate equation to calculate fracture strength is given by Shetty *et al.* [9]. The temperature of the water was maintained $\pm$ 1° C by a constant temperature circulator bath.§ Fifteen specimens were tested at each temperature/crosshead speed condition.

Inert strength of 15 each of the indented-aged and indented-annealed specimens was measured by placing a drop of mineral oil over the indent. A strip of plastic was placed over the oil and stress was applied at a fast stressing rate (80 MPa $\sec^{-1}$). After failure the specimens were examined under an optical microscope to obtain the corresponding radial crack size at fracture. It was assumed that of the two mutually perpendicular radial cracks, the one which did not cause failure must have been very close to instability and accordingly provides a measure of the crack size at failure in an inert environment.

3. Results and discussion

Trantina [10] numerically integrated Equation 1 for residual stress-free cracks and found that fracture strength (σ_f) could be expressed as a function of stressing rate ($\dot{\sigma}_a$) by:

$$\sigma_f/\dot{\sigma}_a = B \exp(-n\sigma_f/\sigma_m RT) \quad (2)$$

where σ_m is the inert strength and $B = 2K_c^2/AY^2\Omega\sigma_m^2$, is the critical stress intensity factor, Y is a dimensionless parameter related to the crack loading geometry (approximately equal to $\pi^{1/2}$), Ω is a crack shape parameter ($\Omega = 4/\pi^2$ for half-penny shaped cracks), and $A = a \exp(-E^*/RT)$. Rearranging Equation 2 and taking logarithms gives:

$$\sigma_f = \alpha + \beta T + \gamma T \ln(\sigma_f/\dot{\sigma}_a) \quad (3)$$

where $\alpha = \sigma_m E^*/n$, $\beta = (\sigma_m R/n) \ln(2K_c^2/\sigma_m^2 Y^2 \Omega a)$, $\gamma = -\sigma_m R/n$. Therefore, a linear regression analysis of the dynamic fatigue data with $\bar{\sigma}_f$ as the dependent variable ($\bar{\sigma}_f$ is the median fracture strength) and T and $T \ln(\bar{\sigma}_f/\dot{\sigma}_a)$ as the independent variables will yield the constants α, β, and γ from which the crack velocity parameters n, E^*, and a can be determined knowing the median inert strength ($\bar{\sigma}_m$) and K_c.

Ritter *et al.* [11] numerically integrated Equation 1 for indentation flaws with a residual contact stress and expressed their results in direct analogy with Equation 2:

$$(\sigma_f/\dot{\sigma}_a) = B' \exp(-n'\sigma_f/\sigma_m RT) \quad (4)$$

where B' and n' are the parameters corresponding to the residual stress-sensitive condition. These parameters are related to the crack velocity constants via the transformation equations:

$$n' = 0.84\, n \quad (5a)$$

$$\ln B' = 2.74 - \frac{0.18n}{RT} + \frac{E^*}{RT} + \ln(C_m/a) \quad (5b)$$

where C_m is the crack size corresponding to the instability condition in an inert environment. Substituting Equation 5 into the logarithm of Equation 4 and rearranging:

$$\sigma_f = \alpha' + \beta' T + \gamma' T \ln(\sigma_f/\dot{\sigma}_a) \quad (6)$$

where

$$\alpha' = \frac{1.19E^*\sigma_m}{n} - 0.21\,\sigma_m$$

$$\beta' = \frac{\sigma_m R}{n}[3.26 + 1.19 \ln(C_m/a)]$$

$$\gamma' = -\frac{1.19\,\sigma_m R}{n}$$

Thus, similar to the stress-free condition a trivariant regression of Equation 6 with $\bar{\sigma}_f$ as the

‡Instron Corp, Canton, Massachusetts, USA.

§Fisher Scientific Co, Medford, Massachusetts, USA.

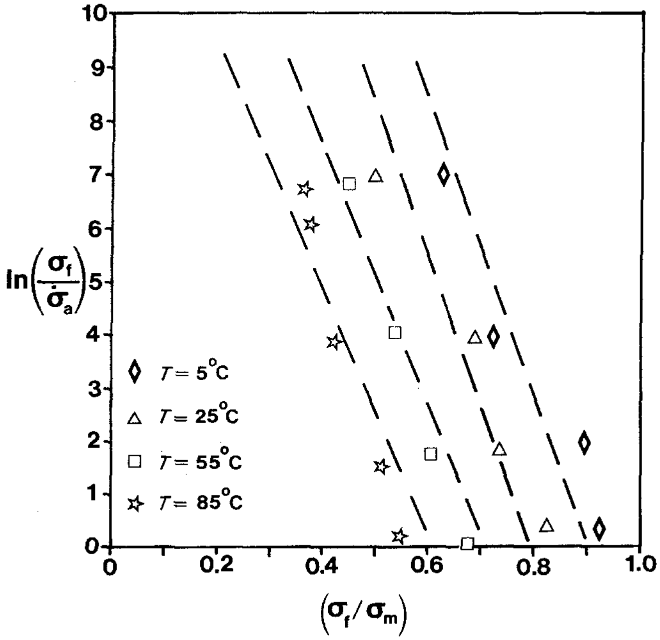

Figure 1 Dynamic fatigue results for indented-annealed soda-lime glass in water.

dependent variable and T and $T \ln (\bar{\sigma}_f/\dot{\sigma}_a)$ as the independent variables will yield the crack velocity parameters knowing the values for $\bar{\sigma}_m$ and C_m.

A summary of the dynamic fatigue data as a function of temperature is given in Figs. 1 and 2 for the indented-annealed and indented-aged samples. The lines in the figures are the regression lines based on Equations 3 and 4, respectively. The median inert strength for the indented-annealed specimens was 73.4 ± 7.7 MPa and for the indented-aged specimens 59.1 ± 7.5 MPa. Median values for C_m were determined by direct observation to be $0.9 \pm 0.1 \times 10^{-4}$ m for the indented-annealed samples and $3.0 \pm 0.9 \times 10^{-4}$ m for the indented-aged samples. Note that these results confirm the condition on the solution of Equation 4 that the initial crack size for the as-indented is less than that at instability under inert conditions. Analysis of the data in Figs. 1 and 2 (using the appopriate values for $\bar{\sigma}_m$ and C_m and taking $K_c = 0.75\,\mathrm{MPa\,m^{-1/2}}$) gives

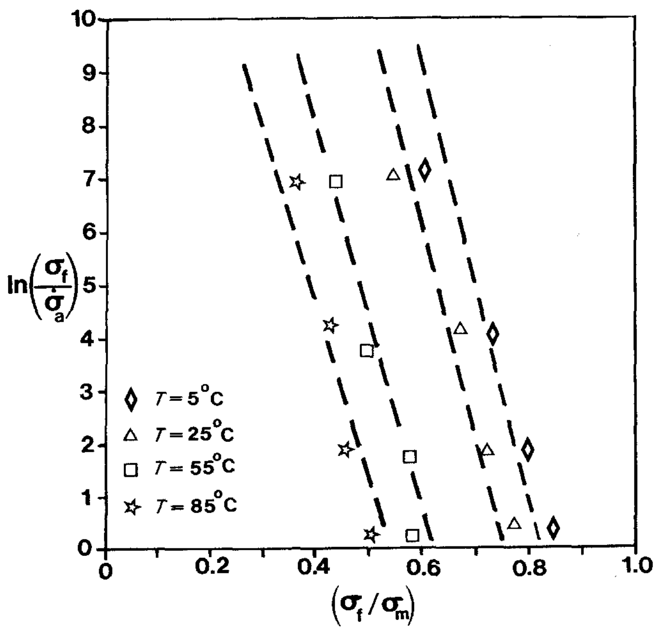

Figure 2 Dynamic fatigue results for indented-aged soda-lime glass in water.

TABLE I Summary of the crack velocity constants for soda-lime glass in water

Condition	n (kJ mol^{-1})	E^* (kJ mol^{-1})	ln a (m sec^{-1})
Indented-annealed	96.9 (± 15.3)	181.7 (± 20.8)	35.1 (± 1.0)
Indented-aged	82.7 (± 9.0)	147.5 (± 18.1)	25.3 (± 4.5)
DCB*	82.4 (± 3.0)	108.8 (± 4.6)	10.3 (± 0.5)

*Data from [4].

the crack velocity parameters in Table I. Also included in Table I for comparison are the values obtained by Wiederhorn [4] from his double cantilever beam specimens. It is important to note that the room temperature (25° C) dynamic fatigue results of this study on indented-annealed and indented-aged samples agrees well with the results obtained by Marshall and Lawn [6, 11].

From Table I it is seen that there is relatively good agreement between the values obtained for the stress intensity parameter n for indented-annealed, indented-aged, and double cantilever beam specimens. However, values for the pre-exponential factor a and the activation energy E^* appear to be different for the three sets of data. A Monte Carlo computer simulation [12] was performed to determine whether the stress corrosion constants obtained from the results of these dynamic fatigue tests on indented samples are statistically different. The computer simulation results showed that the standard deviations based on statistical reproducibility are reasonably close to those based on the experimental data (Table I). Thus, the differences between the parameters E^* and a for the indented samples do not seem to be very significant; however, if they are indeed different it is probably related to the effect of residual stress on failure.

A graphic comparison of the results is given in Fig. 3 where crack velocity curves are drawn based on Equation 1 using the crack velocity parameters in Table I. It is apparent that the reconstructed crack velocity curves based on the dynamic fatigue data for the indented-annealed and indented-aged samples are not very different. Thus, these predicted crack velocity curves are not very sensitive to the differences observed in the parameters E^* and a. This is because the positioning of the predicted curves is dependent on the product term "$a \exp(-E^*/RT)$" and the differences observed between the parameters E^* and a for the indented-annealed and indented-aged samples counterbalance one another so that the product term is approximately the same for the two glasses.

On the other hand, it is evident from Fig. 3 that the indented glass fagitue data does not correlate well with the macroscopic crack velocity data as determined from double cantilever beam specimens. This apparent inability to reconstruct the macroscopic crack velocity curves may be related to interaction effects between the radial and lateral crack systems. It has been shown that these effects are more important in the measurement of inert strength, where the radial and lateral cracks are comparable in size, than in the determination of fatigue strength where radial cracks grow to a size considerably larger than the lateral cracks [6]. For the indented-annealed samples in this study the initial ratio of radial to lateral crack size was about 1.64 and for the indented-aged specimens 1.15; whereas, under fatigue conditions lateral cracks did not grow and the radial cracks propagated subcritically to a size generally over twice their initial value depending on the stressing rate and temperature. For the indented-annealed samples it can be seen from Equation 3 that the parameters a and n depend on the inert strength and for the indented-aged samples n and E^* depend on σ_m and a depends on C_m (see Equation 6). For a 20% decrease in inert strength the major effect on the predicted crack velocity curves is that they will be shifted horizontally to the right about $0.1\,K_I/K_c$ (and vice versa for a 20% increase in inert strength). Thus, although this causes better agreement between the macroscopic and predicted crack velocity curves at 85° C, agreement becomes worse at 5° C.

It is important to realize that we have depended on fatigue strength data to reconstruct the macroscopic $V(K_I)$ behaviour and to convert these data we assumed the validity of Equation 1. However, other exponential crack velocity expressions have been proposed based on various theories [1, 13] but these alternative expressions give rise to considerably more

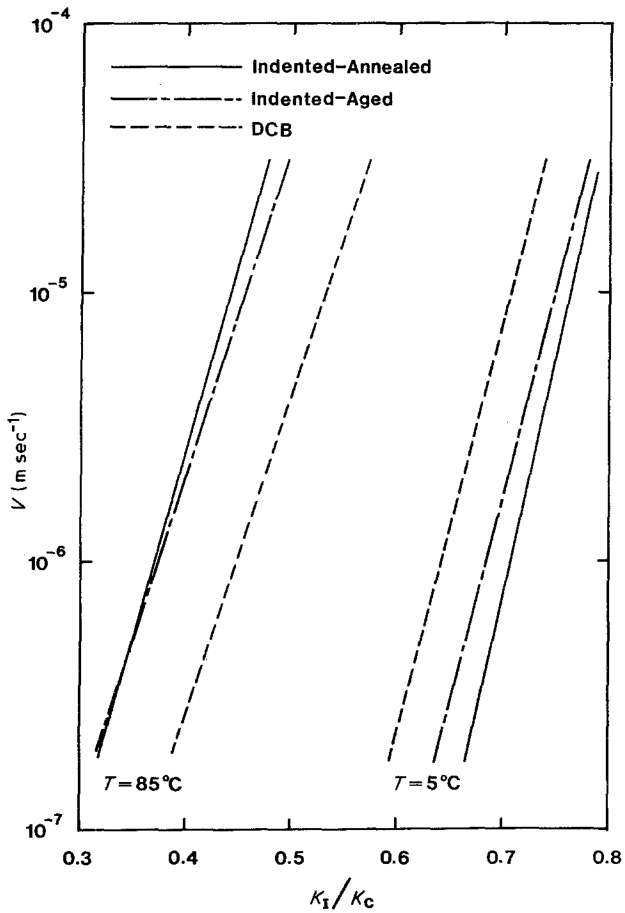

Figure 3 Crack velocity curves of soda-lime glass in water based on Equation 1 and using the crack velocity parameters in Table I.

mathematical complexity and a corresponding greater uncertainty. It is clear that further research is necessary to understand the inability to reconstruct that V (K_I) behaviour from fatigue data on indented samples.

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References

1. S. W. FREIMAN, Fracture Mechanics of Glass, in "Glass Science and Technology", Vol. 5, edited by D. R. Uhlmann and N. J. Kriedl (Academic Press, New York, 1980) pp. 21–78.
2. R. J. CHARLES and W. B. HILLIG, The Kinetics of Glass Failure by Stress Corrosion, in "Symposium on Mechanical Strength of Glass and Ways of Improving It", Florence, Italy, 25–29 September (1961) (Union Scientifique Continentale de Verre, Charleroi, Belgium, 1962) pp. 511–27.
3. S. M. WIEDERHORN and L. H. BOLZ, *J. Amer. Ceram. Soc.* **53** (1970) 543.
4. S. W. WIEDERHORN, Mechanics of Subcritical Crack Growth in Glass, in "Fracture Mechanics of Ceramics", Vol. 4, edited by R. C. Bradt, D. P. H. Hasselman, and F. F. Lange (Plenum Press, New York, 1978) pp. 549–80.
5. S. M. WIEDERHORN, E. R. FULLER Jr and R. THOMSON, *Metal Sci.* **14** (1980) 450.
6. D. B. MARSHALL and B. R. LAWN, *J. Amer. Ceram. Soc.* **63** (1980) 532.
7. B. R. LAWN, D. B. MARSHALL, G. R. ANSTIS and T. P. DABBS, *J. Mater. Sci.* **17** (1982) 1108.
8. T. P. DABBS, B. R. LAWN, and P. L. KELLY, *Phys. Chem. Glasses* **23** (1982) 58.
9. D. K. SHETTY, A. R. ROSENFELD, P. McGUIDE, G. K. BANSAL and W. H. DUCKWORTH, *Amer. Ceram. Soc. Bull.* **59** (1980) 1193.
10. G. G. TRANTINA, *J. Amer. Ceram. Soc.* **62** (1979) 377.
11. J. E. RITTER, M. VICEDOMINE, K. BREDER and K. JAKUS, *ibid.* **67** (1984) 198.
12. J. E. RITTER Jr, N. BANDYOPADHYAY and K. JAKUS, *Amer. Ceram. Soc. Bull.* **60** (1981) 798.
13. K. JAKUS, J. E. RITTER Jr and J. M. SULLIVAN, *J. Amer. Ceram. Soc.* **64** (1981) 372.

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