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Liquid nitrogen strengths of coated optical glass fibres

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The strengths of plastic-coated glass fibres have been measured at liquid nitrogen temperatures using a bending technique. The method yields data on the strengths of coated optical fibres in the absence of stress corrosion. Pristine strengths corresponding to a breaking strain of 21% have been measured for silica fibre and 12% for sodium borosilicate compound glass fibre, corrected to 50 cm gauge length. The low temperature strength was found to be directly related to the tensile strength measured at room temperature, and the relationship was valid for a variety of glass compositions with differing amounts of surface damage.

1. Introduction

Methods for producing high-strength glass fibres in long-gauge lengths are well established [1, 2]. At present the techniques used for measuring the strength of such fibres can be broadly put into two categories: namely, tensile tests on fibres of about 1 m gauge length [3, 4], and proof-testing at low stress levels on long lengths of fibre [5, 6]. Dynamic and static fatigue measurements have been used to determine long-term strengths of glass fibres, but all of these tests are, in general, made at room temperature and are therefore subject to the effects of stress corrosion. Stress corrosion has been postulated to account for the finite lifetime of stressed fibres in certain environments (static fatigue) and also for the dependence of fibre strength upon the rate of strain used in the measurement (dynamic fatigue) [7]. In essence, atmospheric water reacts with the glass and, enhanced by stress in the fibre, ruptures the glass structure. A fibre must be stressed over a finite time interval in order to obtain a measurement of its strength and so all measurements made in the presence of atmospheric-water give lower values than the intrinsic strength. Stress corrosion is, however, temperature dependent and at liquid nitrogen temperatures is negligible. Hence measurements made at − 196° C can yield values close to the intrinsic glass cohesive strength [8]. Measurements made on non-pristine fibre can also give a direct indication of the extent of surface damage.

Optical glass fibres are frequently given protective plastic coatings to preserve their strength and it is the strengths of the coated fibres which are currently of most interest. It is, however, difficult to measure the tensile fracture stresses of coated fibres at − 196° C since, at this temperature, the coating becomes hard and brittle, and contributes significantly to the total measured stress. If, however, a strain is induced by bending, and if this strain can be measured directly in the fibre, then the presence of an outer hard coating will have no effect.

We have used a bending technique which measures the liquid nitrogen strengths of coated glass fibres by straining them in a bending mode between two parallel plates. The plates and fibre can then be immersed in liquid nitrogen and closed together until the fibre fractures, giving a measurement of fibre strength in the absence of stress corrosion.

2. Technique

2.1. Theory

The strain on the outer surface of a glass fibre due to a bend of radius R is given by:

$$\tau = \frac{r}{R}, \tag{1}$$

where τ is the induced bending strain and r the radius of the fibre.

The radius of curvature of a bend induced by

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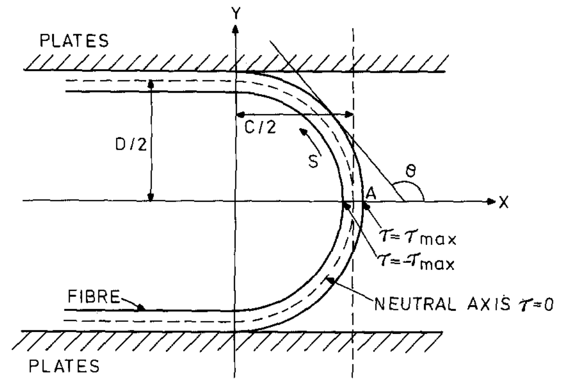

Figure 1 Induced curvature of glass fibre bent between two parallel plates. x and y are the axes, θ is the angle between the tangent to the fibre and the x-axis, $C/2$ is the distance between the origin and the point A, and s is the arc length.

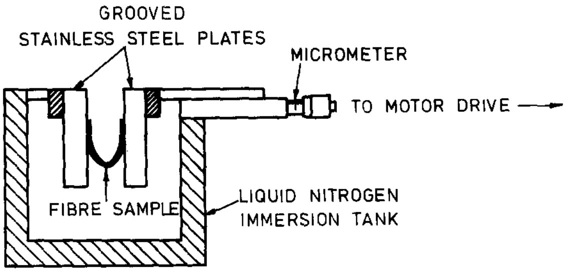

Figure 2 Bending technique for measuring fibre strength at liquid nitrogen temperatures.

placing the fibre between two parallel planes (Fig. 1) can be readily solved using thin beam theory. Assuming that the curvature of the fibre is proportional to the bending moment, so that (using the parameters defined in Fig. 1)

$$\frac{d\theta}{ds} = k^2 x, \tag{2}$$

where k is a constant, it can be shown that the minimum radius of curvature R which occurs at point A, is given by

$$R = C/4 = 0.42D, \tag{3}$$

Hence from Equations 1 and 3

$$\tau_f = r/0.42D, \tag{4}$$

where τ_f is the breaking strain and therefore τ_f can be measured from D, the modified distance between the plates when the fibre fractures. The gauge length can be estimated by considering the strain distribution over the fibre surface and is a function of the fibre breaking strain, varying between about 0.3 and 0.9 mm for the range of fibres tested.

2.2. Apparatus

A schematic diagram of the apparatus is shown in Fig. 2. The fibre was bent through 180° between two grooved stainless steel plates. The plates were attached to a motorized micrometer cross-head, and closed together with a uniform velocity. In order to make a measurement, a 5 cm length of fibre was inserted between the plates and the lower part of the assembly immersed in liquid nitrogen. After waiting a few seconds for the fibre to reach the required temperature the plates were uniformly closed together until the fibre fractured. At liquid nitrogen temperatures the breaking strains involved were fairly high and a significant amount of stored energy was released on fracture. Consequently a break was distinctly audible – giving an indication of when the fracture occurred. Moreover the sound wave generated in the plates could be used to trigger the motor using a conventional acoustic pick-up. The separation D, on failure was measured and adjusted to account for the groove depth in the plates and also the thickness of coating on the fibre. The breaking strain was then given by Equation 4.

2.3. Fibre

Most of the fibre used for measurements was prepared from sodium borosilicate glasses using the Double Crucible technique [9]. A typical diameter was 125 μm with a 50 μm graded-index core, and the optical losses of most fibres were below 5 dB km^{-1}. All fibre was protected with a 70 μm coating of silicone resin applied "in-line" with the fibre drawing [10]. The fibre was wound onto specially prepared drums which were designed to release any drawing tension from the fibre during storage.

3. Results

3.1. Strength probability distributions

The results of strength measurements made on typical sodium borosilicate optical fibre, coated in-line with silicone resin, are shown in Fig. 3. The cumulative per cent failure (Weibull) is plotted along the ordinate against fibre strength (per cent elongation) along the abscissa. Curve a shows measurements made with an Instron tensile tester on 50 cm gauge length samples in ambient con-

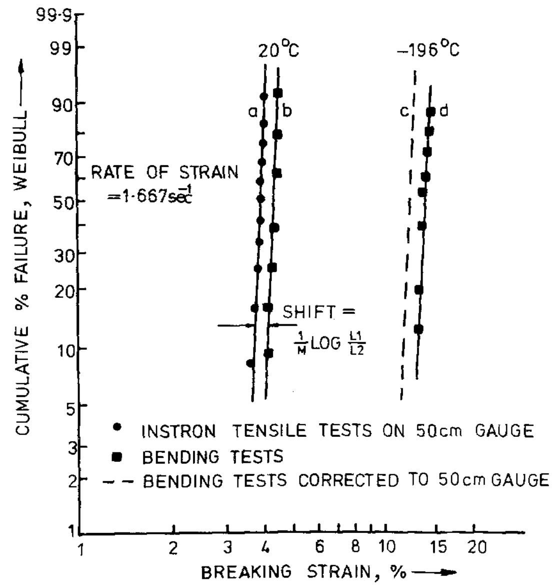

Figure 3 Weibull probability plots of fibre strength showing effects of temperature and gauge length.

ditions at 20° C. The rate of strain was 1.67 × 10^{-3} sec^{-1} and the strength at the 50% Weibull probability level was 4%. The high slope of the Weibull plot is characteristic of protected sodium borosilicate glass fibres and reflects the uniformity of strengths.

Curve b shows results made on the same fibre using the bending technique described, but made in ambient conditions and at the same temperature and strain rate as the Instron tests above. The shift of strength to 4.6% at the 50% Weibull level can be almost entirely accounted for by the gauge length variation and is given by

$$\Delta s = \frac{1}{m}\log\frac{L_1}{L_2}$$

where Δs is the strength shift in log % elongation, m the slope of the Weibull plot and L_1 and L_2 are the respective gauge lengths of samples (50 cm, 0.5 mm).

Although the gauge lengths tested using the bending technique (L_2) depend on the breaking strain as already described, the variation is small compared with the Instron gauge length L_1. Hence the variation in Δs is only small and so an average value of 0.5 mm was substituted for L_2 in all cases.

Measurements made on fibre immersed in liquid nitrogen using the bending technique again gave an almost vertical Weibull plot at 14.0% breaking strain (Curve d) which, when corrected to 50 cm gauge length, became 12.1% (Curve c). This allows direct comparison between measurements made at 20° C and − 196° C, on 50 cm samples. This value was also obtained on similar measurements made on uncoated fibre which had been carefully handled to avoid micro-damage. The reduction in strength from 12.1% to 4% breaking strain is due to the effects of stress corrosion occurring during the measurement.

The value of 12.1% breaking strain closely agrees with fast fracture measurements made by others on pristine uncoated fibres of sodium borosilicate glasses, and is close to the theoretical glass cohesive strength [11].

3.2. Degradation under zero stress

Glass fibres can degrade as a result of surface attack from water, even when stored under zero stress, and glass compositions which maintain their initial high strength over long periods are being sought. With a view to this, certain fibres were stored in distilled water at 20° C and their strengths measured as a function of time. The fibres were again coated in-line with fibre drawing, using 70 μm of silicone resin. To make a measurement, the fibre was removed from the water bath and measured while still wet. (Drying for several hours in dry nitrogen did not alter the strength). Fig. 4 shows the results from a reasonably durable composition whose strength decreased steadily with time, from a value of 4.2% breaking strain to

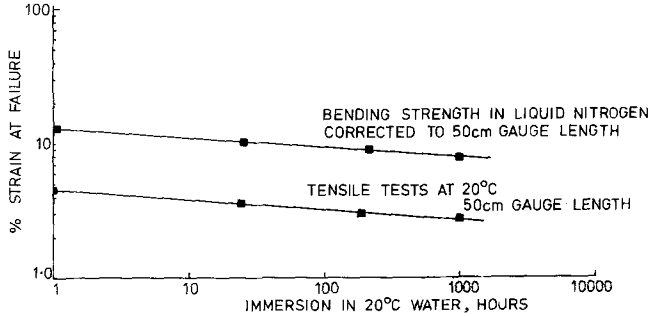

Figure 4 Strength degradation under zero stress of a reasonably durable sodium borosilicate fibre, stored in distilled water at 20° C.

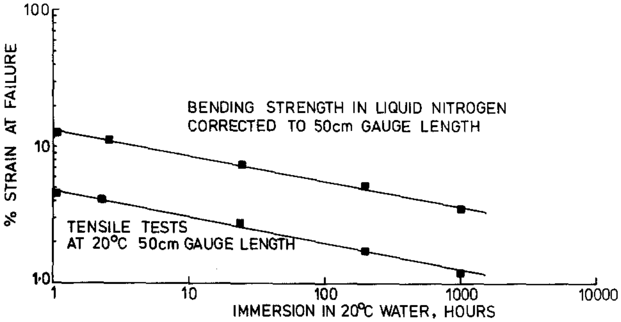

Figure 5 Strength degradation under zero stress of a less durable sodium borosilicate fibre.

2.9% after 1000 h immersion. The results shown in Fig. 5 are for a less durable cladding composition. In both cases the strength obeyed the relationship:

$$\tau_f = \alpha t_w^{\beta}, \tag{5}$$

where τ_f is the breaking strain at 20° C, t_w the immersion time in distilled water and α, β are constants.

For comparison, the strengths at liquid nitrogen temperatures, corrected to the same 50 cm gauge length, were also measured as a function of prior immersion time in water and these points are also plotted in Figs. 4 and 5. Low temperature measurements exclude the effects of stress corrosion and hence give a realistic value of the extent of surface damage arising from aqueous corrosion. Clearly surface flaws grew steadily with time and probably consisted of small corrosion sites. One further point of interest is the close relationship between the two sets of measurements made on both fibres which is emphasized by the near parallel lines in both Figs. 4 and 5. This is discussed further in the next section.

3.3. Comparison of strengths at 20° C with those at − 196° C

In order to examine more closely the relationship between values of strength measured at 20° C and values at − 196° C, several different fibres were tested using the two techniques. The results are displayed in Fig. 6. Each point on the curve represents the value of strength measured from each technique for 50 cm gauge lengths. Each value was taken from the 50% level on near-vertical Weibull probability distributions, and therefore represents the results from at least ten individual measurements.

The measurements were made on fibres of several compositions which had been stored in a variety of environments for different lengths of time. It was thus possible to measure fibres with a range of breaking strains from 0.5 to 4.2%. Pristine sodium borosilicate fibre had a breaking strain of 12% when measured at − 196° C which compared with a value of 21% measured on silica fibre also at − 196° C. This value for silica closely agrees with measurements of tensile strength made by Proctor *et al.* [12].

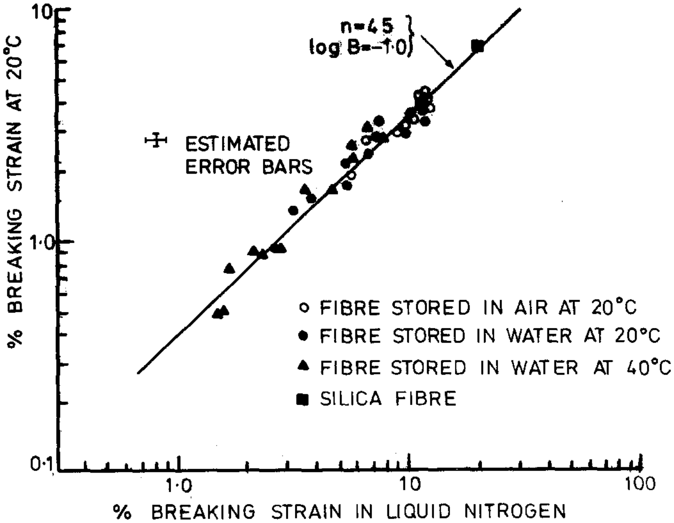

Figure 6 Comparison of strength at liquid nitrogen temperatures to that at 20° C on a range of glass fibres. Measurements at 20° C were made on 50 cm gauge length samples, and measurements at − 196° C on 0.5 mm gauge lengths but corrected to 50 cm for comparison.

The results clearly show that despite the differences in glass composition, in extent of surface corrosion and in the environment under which the fibre was stored, there is a close relationship between the strength measured at liquid nitrogen temperatures to that measured in normal ambient conditions. Moreover the ratio γ between the two measurements appeared to be almost constant at a value of about 2.8, in a large variety of fibre samples. The straight line in Fig. 6 is a theoretical fit to the experimental points discussed in the following section.

4. Discussion

Following the model for stress corrosion described by Charles [13] and extended into fracture mechanics by Evans and Weiderhorn [5] the basic relation of fracture theory is given by

$$K_{\mathrm{I}} = \sigma Y a^{1/2},$$

where K_{I} is the stress intensity factor, σ the applied stress, a the crack depth and Y the geometrical factor for flaw shape. Using the usual assumptions that

$$\frac{\mathrm{d}a}{\mathrm{d}t} = AK_{\mathrm{I}}^{n},$$

where A is the material constant, and n the stress corrosion susceptibility; and that $K_{\mathrm{IC}} \gg K_{\mathrm{I(initial)}}$ (where K_{IC} is the material constant (critical stress intensity required for fracture) and $K_{\mathrm{I(initial)}}$ the initial stress intensity factor), it has been shown that the dynamic strength of glass is related to its strength measured in an inert atmosphere (liquid nitrogen) by [14]

$$\sigma_{\mathrm{f}}^{n+1} = B(n+1)\dot{\sigma}\sigma_{\mathrm{i}}^{n-2}$$

where σ_{f} is the strength at 20° C, σ_{i} the inert strength (− 196° C) and $\dot{\sigma}$ the rate of stress, with

$$B = \frac{2}{(n-2)AY^2K_{\mathrm{IC}}^{n-2}}.$$

In terms of breaking strain (τ) we can re-write this as

$$\tau_{\mathrm{f}}^{n+1} = \frac{B(n+1)\dot{\tau}\tau_{\mathrm{i}}^{n-2}}{E^2},$$

where E is Young's Modulus, or

$$\log \tau_{\mathrm{f}} = \left(\frac{n-2}{n+1}\right)\log \tau_{\mathrm{i}} + \frac{1}{(n+1)}\log \dot{\tau} + \frac{1}{(n+1)}\log\left(\frac{B(n+1)}{E^2}\right). \qquad (6)$$

Also the ratio γ is given by

$$\gamma = \frac{\tau_{\mathrm{i}}}{\tau_{\mathrm{f}}} = \tau_{\mathrm{f}}^{3/(n-2)}\left(\frac{E^2}{B(n+1)\dot{\tau}}\right)^{1/(n-2)}. \qquad (7)$$

Substituting numerical values into Equation 6 provides a theoretical curve to fit to Fig. 6. Measured values of n, for the range of samples tested, varied between 30 and 60 and so an average value of 45 was used. Clearly the correct value of n should be used in each case to obtain an exact solution but since Equation 6 is largely insensitive to large changes in values of n, the value of 45 is sufficiently accurate. A value of -1.0 for $\log B$ was used following the work of Kalish and Tariyal [15]. In all cases $\dot{\tau}$ was $1.667 \times 10^{-3}\ \mathrm{s}^{-1}$ and a value of $7 \times 10^{10}\ \mathrm{N/m^2}$ was used for E. Substituting these values into Equation 6 gives

$$\log \tau_{\mathrm{f}} = 0.934 \log \tau_{\mathrm{i}} - 0.518. \qquad (8)$$

This is shown plotted in Fig. 6, and is in reasonable agreement with the experimental points. For clarity Fig. 6, is plotted in terms of per cent elongation although Equation 6 is written in terms of real strain.

The ratio γ is also given by

$$\gamma = \frac{\tau_{\mathrm{i}}}{\tau_{\mathrm{f}}} = 3.578\,\tau_{\mathrm{f}}^{0.071}, \qquad (9)$$

and for the range of τ_{f} measured (1–4%) then γ is approximately constant at 2.8. This accounts adequately for the near parallel lines in Figs. 4 and 5 since, although τ_{f} decreases with time, the ratio γ between strength at 20° C to strength at − 196° C only changes from 2.8 to 2.7.

The errors involved are too large for Equation 6 to be used in reverse in order to estimate the value of n for the fibre tested. However, they are sufficiently accurate for us to conclude that the reduction in strength from liquid nitrogen temperatures to normal room environments can be adequately accounted for, using conventional stress corrosion theory, and furthermore that the stress corrosion is independent of the initial flaw size.

5. Conclusions

By bending glass fibres between two parallel plates it was possible to measure the strengths of samples immersed in liquid nitrogen. The breaking strain was given simply by $\tau_{\mathrm{f}} = r/0.42D$, but only small gauge lengths were tested. Sodium borosilicate fibres had maximum breaking strains of around

12% compared to 21% for silica fibre close to the theoretical glass cohesive strengths for these compositions.

The strength at −196° C was found to be closely related to the strength measured using a conventional Instron-type machine in ambient conditions, according to

$$\log \tau_f = 0.927 \log \tau_i - 0.518,$$

and the ratio between the two values is given by

$$\gamma = \frac{\tau_i}{\tau_f} = 3.58\,\tau_f^{0.071},$$

which is approximately constant at 2.8. The ratio between the two values was explained using conventional stress corrosion theory, and was found to be valid over a large range of initial flaw sizes.

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