Strength degradation of lightguide fibres in room temperature water

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A fused silica optical fibre which shows 25% strength degradation in 2.3 days at 100°C shows similar degradation after 9 years at 25 °C. This is predictable using an activation energy of 90 $kJ.mol^{-1}$.

Introduction: Many studies have been made of the aging and fatigue of polymer-coated silica fibres (and silica-based lightguides) [1]. These have normally been conducted at elevated temperatures (60–100°C) in order that degradation may be observed in reasonable times. The hope is that extrapolations can be made to ambient or other field conditions. Chandan and Kalish [2] made fatigue studies between 40 and 80°C and found that the time of appearance of the so-called fatigue "knee" (the point at which severe strength degradation begins) increased as the temperature decreased, but equally important, the rate of degradation appeared to become less. They predicted that the knee would disappear at about room temperature. Their lack of detailed data caused Matthewson and Kurkjian [3] to question their conclusions. In addition, recently reported results from two field trials [4,5] suggest that room temperature degradation must be seriously considered. In this study, we have carried out aging tests in room temperature distilled water for times >10 years, and measured the time dependence of the strength determined in two-point bending.

Experimental procedure: 125μm diameter fibres were drawn from silica rods (from Heraeus Amersil, USA) in a zirconia induction furnace and coated in-line with a UV-curable urethane-acrylate polymer giving an overall diameter of ~250μm. They were aged under zero stress in room temperature distilled water (uncirculated and uncontrolled, but ~25°C, ~4000cc). After the prescribed aging time, the residual strength of the fibres was determined at room temperature in the same distilled water, either coated or made bare by stripping in hot sulfuric acid. The strength measurements were made in two-point bending [6, 7] using a faceplate velocity of $1000\mu m.sec^{-1}$.

Results: Fig. 1 shows strength data from this room temperature study, together with the results of our previous study on the same fibre at 100°C [3]. Although neither of the curves is very well-defined in detail, their general shape is clear. They can both be described (on a semi-log plot) by an initial region in which there is very little change in strength, followed by an abrupt transition (the 'knee') after which rather severe degradation takes place. Similar shaped curves can be drawn through the two sets of data. The shift of the curves on the log t scale is then a function of the activation energy of the process responsible for the aging. A model, first proposed by Kurkjian *et al.* [8] and later found to explain

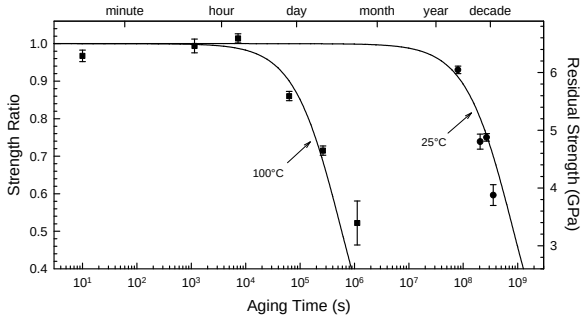

Fig. 1 *Residual strength of fused silica optical fibre after aging under zero stress in distilled water*

ℓ at 100°C
◆ at 25°C

other aging and fatigue results, suggested that inhomogeneous solution of the glass surface can occur due to the existence of density (or free energy) fluctuations intrinsic to glass. These fluctuations give rise to variable solubility and dissolution rates which lead to the development of roughness or 'pits'. These pits (which have since been imaged with an atomic force microscope [9]) act as stress concentrators and are responsible for the reduction in strength of the glass.

The data have been fitted to an empirical model first proposed by France *et al.* [10]

$$\frac{\sigma}{\sigma_0} = (1+\alpha t)^{-\beta} \qquad (1)$$

where σ is the strength after aging for time t and σ_0 is the strength at $t = 0$. Temperature effects can be incorporated into this model by assuming that the time to achieve a given strength degradation (a given value or σ/σ_0) is inverse Arrhenius i.e. that α is Arrhenius [11]:

$$\alpha = \alpha_0 \exp\left(-E_a/RT\right) \qquad (2)$$

where E_a is the activation energy and R and T have their usual meaning. A weighted fit gives values of $E_a = 90 \pm 7$kJ.mol^{-1}, $\beta = 0.7 \pm 1.9$ and $\alpha_0 = (9 \pm 48) \times 10^6$ s^{-1} (95% confidence). The large errors in β and α_0 result from the shape (onset and slope) of the knee that these parameters describe being poorly defined by the data. However, the shift with temperature is well defined giving a reasonably accurate value for E_a. Fitting other functional forms for the degradation (eqn. 1) produced effectively the same value for the activation energy. This activation energy is essentially the value normally found for many SiO_2 + H_2O reactions [12], in agreement with the model proposed for the solution of silica in water. In the present case of polymer-coated fibre, it is presumably necessary for delamination of the coating from the fibre to occur, at least locally, before solution can take place. Thus it is suggested that delamination is the first step in the corrosion process. It is then to be expected that good adhesion will result in a shift of the onset of corrosion (*i.e.*, the 'knee'), to longer times. However, the adhesion will not affect the apparent activation energy for degradation, since the adhesion is usually improved using silane promoters. Therefore adhesion loss will be controlled by the breaking of Si-O-Si bonds, just as is the dissolution.

As far as we are aware, the only other data for aging fused silica fibres at room temperature in water are those published by Griffioen [11]. His data in the range of 30 to 60°C fit eqn. 1 and eqn. 2 reasonably well and give an activation energy of 83 ± 12 kJ.mol^{-1}. However, his 20°C data do not fit the trend; the reason for this is unclear, but may be an artefact of the use of a plastic vessel for 20°C experiments and metal vessels for the higher temperatures, this detail being confirmed by Griffioen.

While it is disturbing from a practical point of view that corrosion has been observed at room temperature in silica at times less than the design life of optical fibre cables. It is clear that degradation is caused by water and is inhibited if water is excluded from the fibre surface [13]. In addition, these studies were carried out on fibres with an early coating type (*ca.* 1983) and are perhaps not representative of the behavior of fibres produced commercially today. Today's coatings have greatly improved adhesion, and because of this, the knee in hot water occurs at much longer times. For instance, in the coating studied here, a 25% reduction in strength occurs in ~2.3 days at 100°C but in many more modern coatings, this time is substantially longer. Ritter *et al.* [14] observed aging knees in 80°C water at up to 1.7 years for some more modern coatings; using the experimentally determined activation energy of 90kJ.mol^{-1}, a time of ~500 years is calculated for a loss in strength to be observed at room temperature. Degrada-tion might not be considered severe until 50% or so of the strength is lost; this typically takes an order of magnitude longer than 25% degradation. Therefore, degradation at room temperature may not be an issue for the reliability of fused silica fibres with modern coatings.

Summary: The strength of fused silica fibres is reduced after relatively long times in room temperature water. The observed degradation is predictable from higher temperature studies on the same fibre by using an activation energy of 90kJ.mol^{-1}. Increases in the glass/polymer adhesion increases the time for observable corrosion in liquid water at elevated temperatures and thus would be expected to have a similar effect in less aggressive (i.e. lower temperature) environments.

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