Temperature dependence of strength and fatigue of fused silica fiber in the range 77 to 473 K

M. John Matthewson,* Charles R. Kurkjian, Christopher D. Haines, Navin Venugopal
Photonic Component Reliability Group, Department of Ceramic & Materials Engineering,
Rutgers University, Piscataway, NJ 08844

ABSTRACT

The strength of optical fiber at low temperature is an important parameter since it approximates the inert strength, *i.e.* the starting strength of the material before degradation by fatigue. Published data suggest that the fatigue may abruptly slow below some temperature. However, published data are limited to strength *vs* temperature or fatigue in liquid nitrogen. We report strength ***and*** fatigue data for both bare (stripped) and metal coated fused silica optical fiber at temperatures down to 77 K. While fatigue slows as the temperature is reduced (*i.e.* the stress corrosion parameter increases with falling temperature) fatigue is still measurable at 77 K. This is the case even for hermetic metal coated fiber with extremely low water activity at the glass surface. The results confirm that fused silica exhibits "intrinsic" fatigue, *i.e.* fatigue in the absence of moisture.

Keywords: Optical fiber, strength, fatigue, stress corrosion, temperature, intrinsic fatigue.

1. INTRODUCTION

When the strength of fused silica optical fiber is measured under ambient conditions, moisture causes fatigue so that the measured strength is the initial, inert or starting strength of the material (*i.e.* the strength that would be measured in the absence of moisture) minus the loss in strength due to subcritical stress corrosion cracking during testing. The inert strength is important since it is a key parameter in models for mechanical reliability, plus it is needed to properly assess the measured strength – a weak specimen may be weak either because it contained a large flaw or because it fatigued rapidly. The most convenient way to estimate the inert strength is to measure the strength in liquid nitrogen, at which temperature the fatigue process (a chemical reaction between environmental moisture and strained bonds in the region of stress amplifying surface defects) is greatly slowed. However, in early work on fused silica fiber strength, Proctor *et al.*[1] measured the strength of bare, hand-drawn fibers in tension and found that the strength in boiling helium (4.2 K) is higher than in boiling nitrogen (77 K) indicating that perhaps fatigue is not stopped at 77 K.[2,3] Assuming that the fatigue process is a thermally activated chemical reaction, one expects the strength to roughly follow Arrhenius behavior.[4] Fig. 1 shows an Arrhenius plot of the data of Proctor *et al.*[1] (triangles). Apart from the point with the arrow which corresponds to a temperature of 4.2 K, the data roughly form a straight line suggesting that fatigue still proceeds at low temperature. Duncan *et al.*[5] measured the strength of silicone coated optical fibers in two-point bending. Their data are also shown in Fig. 1 (squares). Their data also form a straight line on the Arrhenius plot, except that the measurement in boiling nitrogen is much lower than projected from the higher temperature data. This suggests a break in the behavior around 125 K ($1/T = 8 \times 10^{-3}$ K^{-1}); below this temperature fatigue is very much slower. However, this result may be an artifact of the silicone coating. At sufficiently low temperature the coating will embrittle and so could cause premature failure of the glass. Fortunati and Matthewson[6] measured the strength of bare (*i.e.* acid stripped) fibers in two-point bending. Their experiments had poor temperature control so that measurements could not be averaged at a particular temperature. Their results (individual tests, circles in Fig. 1) show considerable scatter but do not show any evidence of a break in the linear behavior down to 77 K. Nonlinear behavior of strength on the Arrhenius plot has been found for a borosilicate glass,[5] a soda-lime silica glass[7] and E-glass.[8]

* mjohnm@fracture.rutgers.edu; phone 1-732-445-5933; fax 1-732-445-3258; http://www.rci.rutgers.edu/~mjohnm/mypage; Department of Ceramic & Materials Engineering, Rutgers University, 607 Taylor Road, Piscataway, NJ, USA 08854-8065

Reliability of Optical Fiber Components, Devices, Systems, and Networks, Hans G. Limberger, M. John Matthewson, Editors, Proceedings of SPIE Vol. 4940 (2003) © 2003 SPIE · 0277-786X/03/$15.00

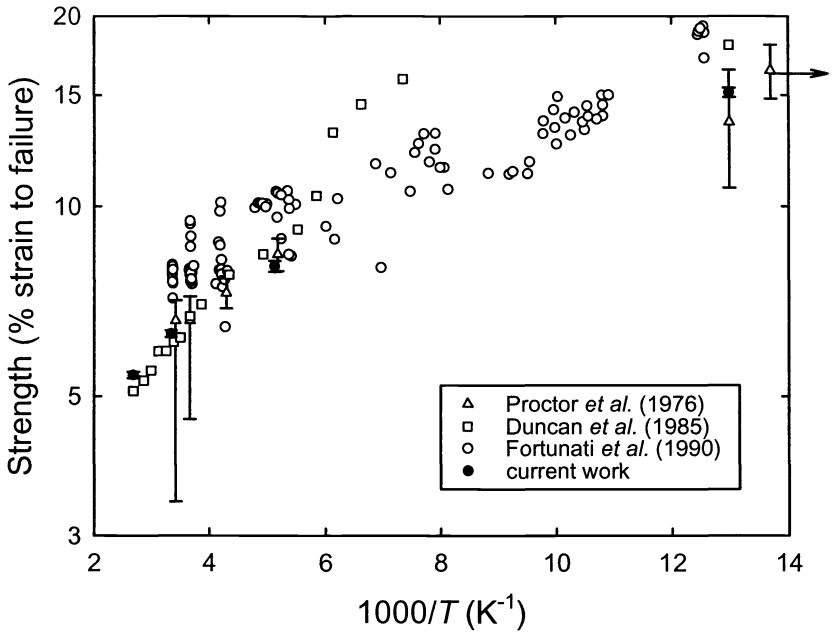

Figure 1. Arrhenius plot of strength (characterized by the strain to failure) *vs.* reciprocal temperature. The data are from several studies but all measurements are in environments essentially saturated with moisture.

These studies have not elucidated the low temperature fatigue behavior in detail. In particular, it is not clear if the behavior is simple Arrhenius or whether there is a change of mechanism at low temperature leading to a reduced rate of fatigue. Interpretation of the low temperature results is complicated since as the strength approaches the inert strength, the apparent stress corrosion parameter increases because the dynamic fatigue curve levels off at very high loading rates. This can cause the strength as a function of temperature also to level off at low temperature, even though the fatigue process is still active.

This paper presents experimental results on both bare and metal coated fibers which address some of these issues. We present results for the strength of bare fibers as a function of temperature. In addition, results for the strength ***and*** fatigue of metal hermetic coated fiber are presented which elucidate the fatigue behavior at low temperature and low water activity.

2. EXPERIMENTAL

The strength and fatigue measurements were all made using two-point bending.[9] In this method a fiber is held bent between two faceplates; one stationary and the other attached to a stepper motor driven translation stage. This is the simplest method for testing over a broad range of temperature since the faceplates can be inserted into the test environment without having to expose the rest of the apparatus to that environment. However, changes in temperature of the faceplates, their mounts and the components of the translation stage can cause variations in the relative position of the faceplates. The strengths measured at low temperature are very high and so the faceplates separation at failure is small meaning that any variation in faceplate alignment can cause significant error. To overcome this, the faceplates were brought into contact after every fiber was broken while still in the test environment in order to correct for any drift in the zero position. During the course of an experiment the faceplate zero separation position varied by up to 20 μm making this zero correction procedure essential for obtaining accurate results.

Bare fiber was prepared for testing by immersing UV-acrylate coated fiber in methylene chloride. The polymer swells and is easily removed by hand leaving a clean surface. The bare fiber is then carefully balanced between polished faceplates in order to avoid scratching the fiber surface on the sides of the grooves that are used to hold coated fiber.[9]

The temperature of the test environments was maintained using a substance at its equilibrium transformation temperature, namely boiling water at 373 K, subliming CO_2 in acetone at 195 K and boiling nitrogen at 77 K. Measurements at room temperature used a temperature controlled water bath. Measurements on metal coated fiber at 473 K used a temperature controlled oil bath. At all these temperatures except 473 K, the water has essentially unit activity because measurements were in liquid water above 273 K, while below 273 K the test environments were below the dew point of the laboratory air and so can be considered saturated with moisture.

The fatigue data were analyzed using a computer program that can fit an arbitrary kinetics model for fatigue to experimental data without making any unnecessary assumptions.[10] In particular, when the crack growth equations are integrated from zero time to the failure time, the resulting equations are usually simplified by making the approximation that the measured strength of the fiber is significantly smaller than the inert strength. This assumption will not be valid at low temperatures and for this reason the computer program does not make this approximation.

3. RESULTS

3.1 Bare Fiber

Fig. 1 shows the results for the strength measurements on bare fiber (solid circles) compared to the data from the earlier work.[1,5,6] Although the data are not very extensive, they are more accurate then the earlier work (the error bars are 95% confidence intervals). As for some of the earlier work, the boiling nitrogen measurement is perhaps lower than would be extrapolated from the higher temperature measurements. As discussed above, this could be a change in mechanism or it could simply be that the strength is leveling off, not because of slowing fatigue, but because the strength is approaching the inert strength. To determine which of these mechanisms is operative, the simplest approach would be to measure the fatigue at each test temperature. However, bare fibers must be broken one at a time so working at low temperature is extremely tedious and time consuming. Making fatigue measurements on bare fiber was therefore beyond the scope of this study. Polymer coated fiber can not be used since embrittlement at low temperatures causes premature failure of the glass.

Glaesemann and Helfinstine[11] measured the strength over a range of temperatures down to ~100 K of fiber that had been deliberately weakened by abrasion. Their results are shown in Fig. 2 together with all the data for high strength fiber from Fig. 1. The general trends of the data have similar slopes suggesting that the temperature dependence of strength is the same

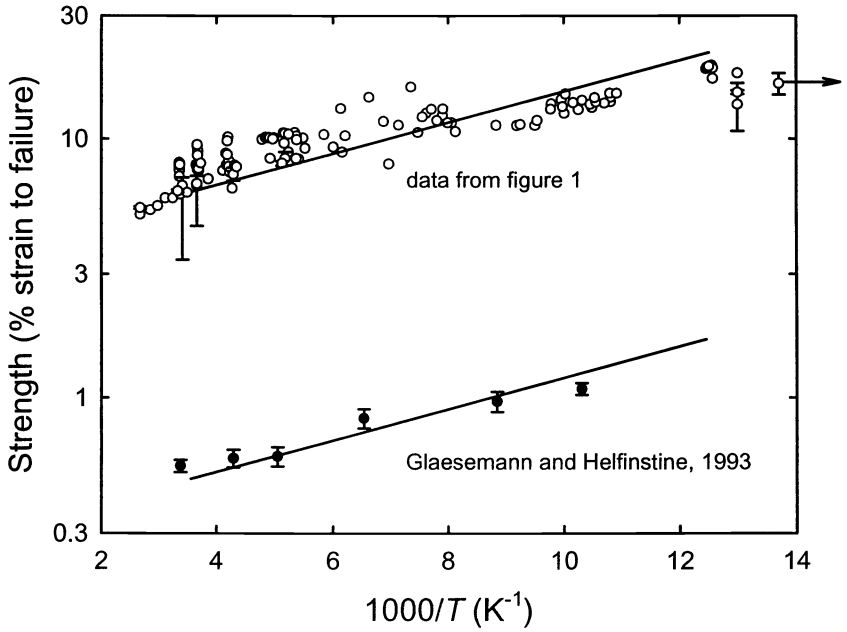

Figure 2. Data of Fig. 1 compared to results of Glaesemann and Helfinstine[11] for deliberately weakened fiber.

for both weak and high strength fiber. If more detailed measurements show that this scaling similarity holds in detail, it would make it straightforward to incorporate the effect of temperature into reliability models.

3.2 Metal hermetic coated fiber

Metal coated fiber is of particular interest in this study. Firstly, we find here that it can be successfully broken at low temperature without the coating damaging the glass. Secondly, because the coating is hermetic and excludes moisture the

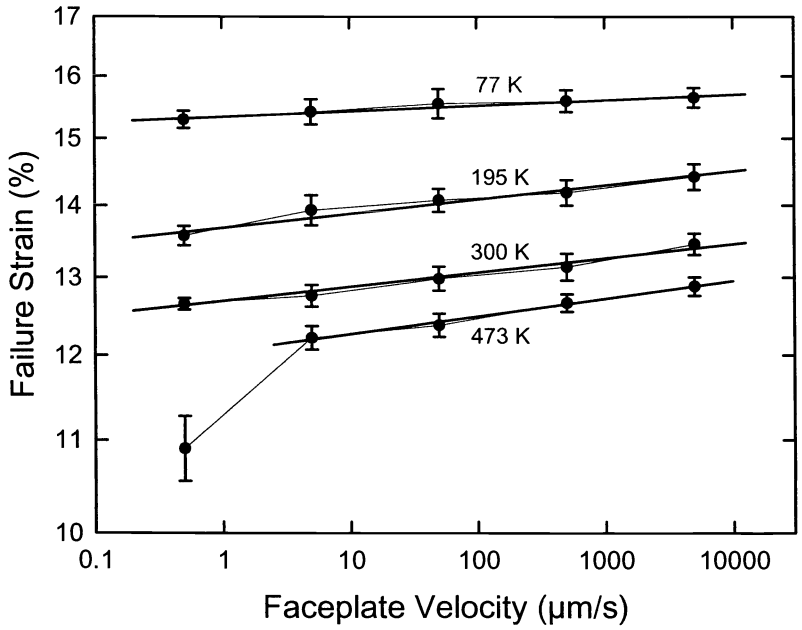

Figure 3. Results for the dynamic fatigue in two-point bending of tin coated fiber.

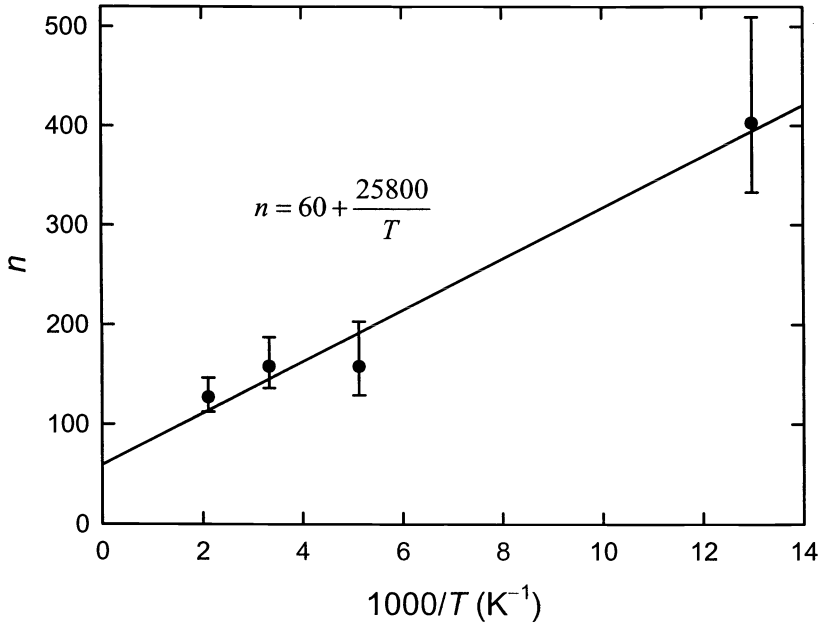

Figure 4. Power law stress corrosion parameter, n, calculated from the data of Fig. 3 (omitting the 0.5 μm/s, 473 K anomalous measurement).

strength of the fiber is already close to the inert value. Finally, being coated, the fibers do not interfere with each other when they break so that several may be broken simultaneously using multi-grooved faceplates. This makes collecting data in statistically significant quantities relatively easy.

Fig. 3 shows that dynamic fatigue data for tin coated fiber taken at five different faceplate velocities spanning four decades in rate with 15 strength measurements made per rate. All the data show well-defined fatigue behavior, except for the slowest rate at 473 K. This temperature is close to the melting point of the tin coating, 505 K, so it is suspected that the coating crept over the course of the slowest experiments and this somehow damaged to fiber surface, or perhaps the tin grains separated to expose the fiber surface to moisture. Fig. 4 shows the power law fatigue parameter, n, as a function of reciprocal temperature, calculated from the data of Fig. 3 using the computer program described above. As might be expected, the values for n are considerably higher than the accepted value of ~20 for fiber in ambient conditions. In particular, the room temperature measurement is consistent with the n measured in static fatigue for a similar tin coated fiber.[12] For high values of n the strength is relatively insensitive to loading rate; as a result the 95% confidence error bars for n are large, with an error of about $\pm$ 25%.[13] If fatigue were completely suppressed, the value of n would be effectively infinity, or $1/n$ would be indistinguishable from zero within experimental error. However, even at 77 K, the fatigue is clearly measurable and n is not infinite.

What is causing fatigue under these very dry and cold conditions? It is possible that small amounts of moisture trapped under the metal coating during its application could be causing the fatigue.[14] However, we have found similar results for fiber coated with aluminum at a significantly higher temperature which reduces the amount of moisture that can be trapped, plus any such moisture would react with the aluminum so becoming chemically fixed. Therefore, it is likely that the fatigue observed here is occurring in the absence of moisture. Zhurkhov[2] proposes a "thermofluctuation" model that predicts that strained bonds will fatigue on their own due to random thermal fluctuations occasionally taking the bonds past their breaking point. The activation energies involved are large so that the process is extremely sensitive to the stress, *i.e.* n is large.[12] However, the value of n should vary with temperature. The generalized chemical kinetics model for fatigue developed elsewhere in this volume in the paper by Matthewson predicts that n should vary with temperature according to:

$$n = \frac{n_H}{RT} + \frac{n_S}{R}, \tag{1}$$

where n_H and n_S are parameters that quantify how the activation barrier for fatigue is reduced through enthalpic and entropic effects, respectively. This model does not have to assume the presence of an attacking environmental species, but only assumes some chemical process that is thermally activated with an activation barrier which is reduced by the local stress. It is therefore equally applicable to fatigue in the absence of moisture, *i.e.* so-called "intrinsic" fatigue.[2,3,12,15-18] It should be noted that Matthewson derives Eq. 1 as a general result for any reasonable form of the crack growth kinetics; this same behavior is expected whether a power law or an exponential form is assumed. Analysis of the data in Fig. 3 using exponential forms yields values for n which are again approximately linear with reciprocal temperature. Whichever kinetics form is used, the term in n_S in Eq. 1 is significant which means entropic effects are important. Entropic contributions are more important if exponential forms are assumed. The importance of activation entropy has also been demonstrated for fatigue in the presence of moisture.[19]

4. CONCLUSIONS

Data for the strength of bare fiber as a function of temperature have not unequivocally shown a change in fatigue mechanism at low temperatures. Dynamic fatigue measurements on metal coated fibers show measurable fatigue down to 77 K. The results are consistent with a chemical kinetics model for fatigue, even though there is no little or no moisture available. The results therefore suggest that "intrinsic" fatigue is taking place which does not require the presence of an attacking environmental species. This means that strength is a time dependent quantity even in boiling nitrogen, though the time dependence is weak and is probably of little practical significance. These results confirm that the true "inert strength" can only be encountered at absolute zero temperature.

ACHNOWLEDGMENTS

We thank Drs. S. L. Semjonov and V. A. Bogatyrjov of the General Physics Institute, Moscow, Russia, for supplying the metal coated fiber used in this study.

REFERENCES

1. B. A. Proctor, I. Whitney and J. W. Johnson, "The strength of fused silica," *Proc. Roy. Soc. Lond.* **297A** 534-557 (1967).
2. S. N. Zhurkov, "Kinetic concept of the strength of solids," *Int. J. Fracture Mech.* **1** [3] 311-323 (1965).
3. G. M. Bartenev, *The Structure and Properties of Inorganic Glasses,* Wolters-Noorhoff Publishers, Groningen, Netherlands (1970).
4. P. K. Gupta, "Examination of the tensile strength of E-glass fiber in the context of slow crack growth" in "Fracture Mechanics in Ceramics," eds. R. C. Bradt, D. P. H. Hasselman, F. F. Lange and A. G. Evans, Plenum Press, New York, NY, (1982).
5. W. J. Duncan, P. W. France and S. P. Craig, "The effect of environment on the strength of optical fiber" in "Strength of inorganic glass," ed. C. R. Kurkjian, pp. 309-328, Plenum Press, New York, (1985).
6. B. Fortunati and M. J. Matthewson, "Temperature dependence of optical fiber strength," abstract in *Am. Ceram. Soc. Bull.,* **69** [3] 511 (1990).
7. F. M. Ernsberger, "Tensile and compressive strength of pristine glasses by an oblate bubble technique," *Phys. Chem. Glasses* **10** [6] 240-245 (1969).
8. N. M. Cameron, "The effect of environment and temperature on the strength of E-glass fibres. Part I. High vacuum and low temperature," *Glass Tech.* **9** [1] 14-21 (1968).
9. M. J. Matthewson, C. R. Kurkjian and S. T. Gulati, "Strength measurement of optical fibers by bending," *J. Am. Ceram. Soc.* **69** [11] 815-821 (1986).
10. M. J. Matthewson, "Fiber lifetime predictions," *Proc. Soc. Photo-Opt. Instrum. Eng.* **1580** 130-141 (1991).
11. G. S. Glaesemann and J. D. Helfinstine, "Measuring the inert strength of large flaws in optical fiber," *Proc. Soc. Photo-Opt. Instrum. Eng.* **2074** 95-107 (1993).
12. V. A. Bogatyrjov, M. M. Bubnov, E. M. Dianov, S. D. Rumyantsev and S. L. Semjonov, "Mechanical reliability of polymer-coated and hermetically coated optical fibers based on proof testing," *Opt. Eng.* **30** [6] 690-699 (1991).
13. J. E. Ritter, Jr., N. Bandyopadhyay and K. Jakus, "Statistical reproducibility of the dynamic and static fatigue experiments," *Ceram. Bull.* **60** [8] 798-806 (1981).
14. M. M. Bubnov, E. M. Dianov and S. L. Semjonov, "Influence of residual moisture on the strength of metal coated optical fibers," *Mat. Res. Soc. Symp. Proc.* **244** 97101 (1992).
15. V. S. Kusenko, V. A. Petrov, A. V. Savitsii, V. V. Baltizmanski and V. S. Khotimchenko, "Strength and durability of defect-free optical fibers from the standpoint of the kinetic theory of the fracture," *Sov. Phys. Dokl.* **32** [2] (1989).
16. C. R. Kurkjian and D. Inniss, "Strength and fatigue of silica" in "Proc. 7th Int. Conf. on Phys. of Non-Cryst. Solids," eds. L. D. Pye, W. C. LaCourse and H. J. Stevens, pp. 649-653, Taylor & Francis, London, (1992).
17. C. R. Kurkjian, P. G. Simpkins and D. Inniss, "Strength, degradation, and coating of silica lightguides," *J. Am. Ceram. Soc.* **76** [5] 1106-1112 (1993).
18. C. R. Kurkjian, P. K. Gupta, R. K. Brow and N. Lower, "The Intrinsic strength and fatigue of oxide glasses," *J. Non-Cryst. Solids* **316** 114-124 (2003).
19. Y. S. Shiue and M. J. Matthewson, "Stress dependent activation entropy for dynamic fatigue of pristine silica optical fibers," *J. Appl. Phys.* **89** [9] 4787-4793 (2001).