Kinetics of degradation during fatigue and aging of fused silica optical fiber

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ABSTRACT

Fused silica optical fiber tested in aggressive environments can exhibit a "knee" in both the zero-stress aging and the fatigue under stress; degradation proceeds at an accelerated rate beyond the knee. This behavior leads to shorter lifetimes than predicted from short term data and to strength degradation even in the absence of an applied stress which can result in handleability problems. While the first observation of this behavior was for a humid environment, later work only reported the knee in liquid aqueous environments. This paper reports the observation of a pronounced fatigue and aging knee for a fiber tested in 85°C, 85% relative humidity, clearly indicating this phenomenon can occur in more benign environments. Surface roughness measurements using atomic force microscopy also show an abrupt increase in roughness indicating that, for this fiber at least, the development of surface roughness before the knee can not be used as a precursor for predicting the position of the knee.

Keywords: optical fiber, strength, fatigue, fatigue knee, aging, reliability, environmental effects, cracks, roughness, AFM.

1. INTRODUCTION

High strength fused silica optical fiber normally shows well behaved "power law" fatigue in which the time to failure plotted as a function of static applied stress on a double logarithmic plot exhibits approximately straight line behavior. However, under some conditions, deviations from linearity are observed at longer times to failure, lower applied stresses, that result in enhanced degradation and failure times substantially shorter than would be predicted by extrapolation from short term data. This is the so-called fatigue transition or "knee". The phenomenon was first observed by Wang and Zupko[1] for one type of fiber tested in 32.6°C, 90% relative humidity, who ascribed it to some unspecified coating effect. Their data (Fig. 1) show a typical value for the fatigue parameter of $n \sim 20$ before the knee, but a value of around 7 beyond the knee. Krause[2,3] saw similar behavior for both coated and bare (*i.e.* stripped of its polymer coating) fiber tested in 90°C water. These results indicate that while the polymer coating can influence the onset and severity of the fatigue knee, the phenomenon is associated with the glass since is occurs even in the absence of a coating. Krause[3] observed that the strength of the fiber can degrade upon soaking in 90°C water even in the absence of an applied stress. This observation is of practical significance since the static fatigue knee is normally observed at stress levels much higher than would be observed in a fiber in the field. However, degradation of long lengths of fiber under zero stress can result in so much strength loss that handling the fiber for subsequent repair or reconfiguration operations can become impossible. Matthewson and Kurkjian[4] observed that the abrupt strength loss during zero stress aging occurs at a similar time as the fatigue knee indicating that both result from the same phenomena, namely the formation of surface etch pits that act as a new source of stress concentrators. This pit model was originally proposed by Kurkjian *et al.*[5] but has only recently been verified by direct observation of etch pits by Yuce and coworkers. Robinson and Yuce[6] used scanning tunneling microscopy to correlate the residual strength of aged fibers with the amplitude of surface roughness of fiber that had been coated with gold after aging to make the surface conducting.

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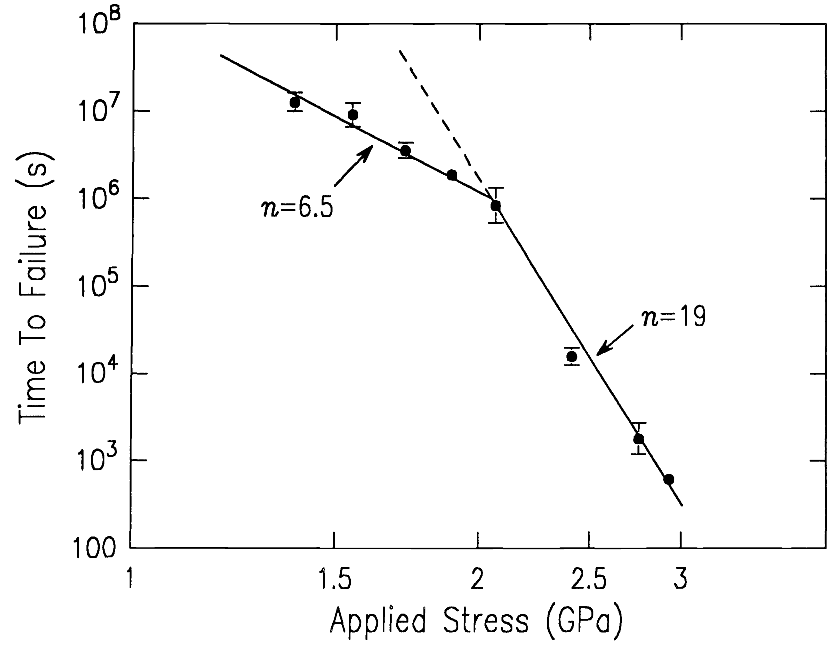

Fig. 1. Tensile static fatigue data for laser drawn TO-8 clad fiber tested in 32.6°C 90% relative humidity (after Wang and Zupko[1]).

Yuce *et al.*[7] found similar results using atomic force microscopy (AFM) which obviated the necessity of applying a gold coating. Yuce *et al.*[8] determined the aging and surface roughening behavior of two different fibers in both high humidity and liquid aqueous environments and found that while the degradation kinetics varied considerably for the four different fiber/environment combinations, a plot of residual strength versus surface roughness produced a single line strongly indicating the causal relationship between the two. Inniss *et al.*[9] measured both the residual strength of fiber and the shape of etch pits produced by HF vapor and verified the theoretical relationship between the shape of a blunt crack and the associated stress concentration.

The existence of the fatigue and aging knees has now been observed by many research groups and is widely accepted. The position and abruptness of the knee shows considerable variation for different fibers and is thought to be sensitive the properties of the polymer coating such as its adhesion.[10] However, in almost all cases, with the notable exception of the first observation by Wang and Zupko,[1] the fatigue knee has only been observed in aggressive (*i.e.* hot or high pH) ***liquid*** aqueous environments. The significance of the fatigue knee might therefore be questioned since in most applications the optical fiber is not immersed in water but only experiences humidity. While there have been some indications that strength degradation can occur in humidity,[8,11–13] the existence of a fatigue knee in humid environments has not been unequivocally established; the purpose of this article is so to do.

2. EXPERIMENTAL

Both zero stress aging and static fatigue experiments have been performed on a fused silica optical fiber (125 μm diameter) coated with a commercially available dual acrylate polymer coating system (250 μm outer diameter). Static fatigue was performed in two-point bending[14] in which specimens were bent double and inserted into precision bore glass tubes of various internal diameters. The stress experienced by the fibers is determined by the tube diameter. The specimens were then immersed either into a bath of 85°C deionized water or in a chamber with a controlled environment of 85°C, 85% relative humidity air. In these experiments the environments were controlled to within ±1°C and ±3% relative humidity respectively. Fiber breaks were detected using an acoustic transducer and the log-normal average of the time to failure of 21 specimens has been calculated for each experimental condition.

Zero stress aging was performed by immersing lengths of fiber for various times in the test environments described above. The residual strength of the aged specimens was determined by breaking them in two-point bending[15] at a constant faceplate velocity of 10 μm.s^{-1}; testing was performed in controlled ambient environment (25±1 °C, 50±3% relative humidity) and all specimens were equilibrated for at least 24 hours in this environment prior to testing. The average strength of 21 specimens was determined for each experimental condition.

Some specimens that had been aged under zero stress were prepared for AFM analysis by stripping the polymer coating in hot sulfuric acid; this technique for removing the coating is the most reliable for exposing the strength controlling surface of the fiber.[16] The surface roughness of the fiber was characterized by determining the peak to valley height of a 1.5 μm square AFM scan and the average for 7 individual specimens for each experimental condition is reported.

3. RESULTS

3.1 Static Fatigue

Fig. 2 shows the two-point bending static fatigue results for deionized water and 85% relative humidity environments. Distinct knees are observed for both environments. Before the knee the data are linear giving well defined power-law fatigue with stress corrosion parameters, n, of 14.9±0.8 and 17.7±0.7 for the liquid and vapor environments respectively. Beyond the knee the data are also well described by a power law with effective values of n of 1.1±0.2 and 1.5±0.1 for the liquid and vapor environments respectively; the time to failure is relatively insensitive to the applied stress in this region. The knees are still apparent if these data are replotted on semi-logarithmic axes indicating that they are not an artifact of the assumed form of the degradation kinetics model, be it a power or exponential form.

3.2 Zero Stress Aging

Fig. 3 shows the residual strength of the fiber after aging for various times in the two environments. In both cases the strength initially shows little degradation until an abrupt loss of strength occurs. The times at which the aging knees are observed are similar to the times of the fatigue knees shown in Fig. 2 but the aging knees occur somewhat earlier. In practice it is generally considered that a minimum strength of about 2 GPa is

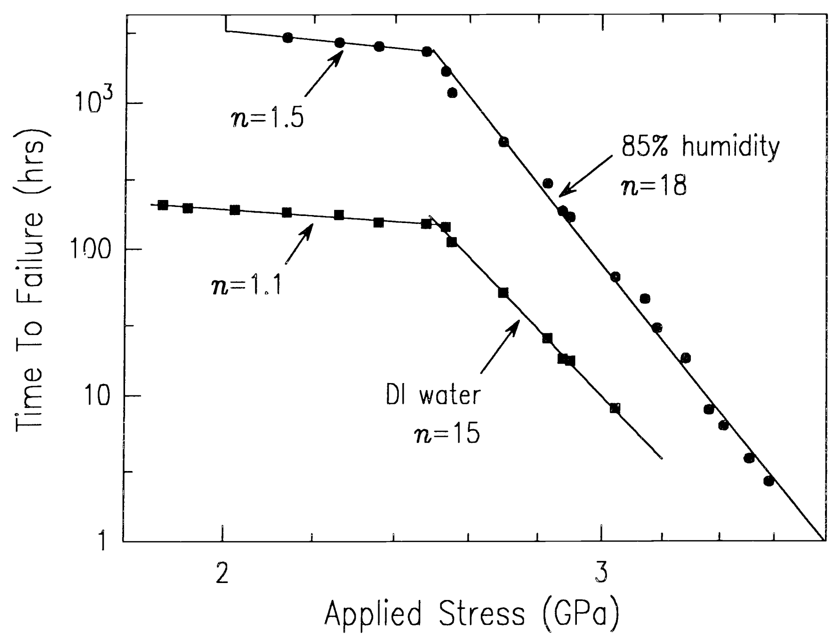

Fig. 2. Two-point static fatigue of fiber in 85°C (■) deionized water and (●) 85% relative humidity.

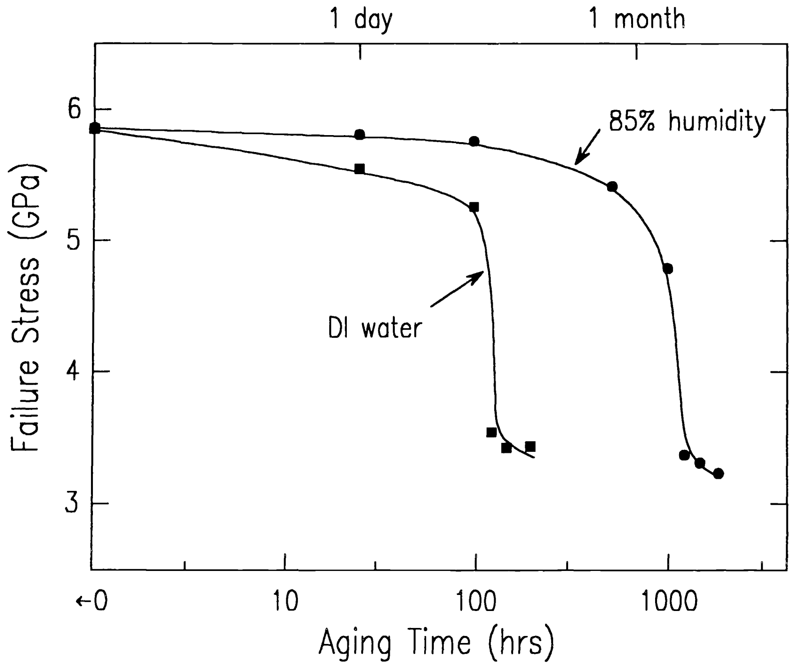

Fig. 3. Residual strength, measured in two-point bending in 23°C, 50% relative humidity, as a function of aging time in 85°C (■) deionized water and (●) 85% relative humidity.

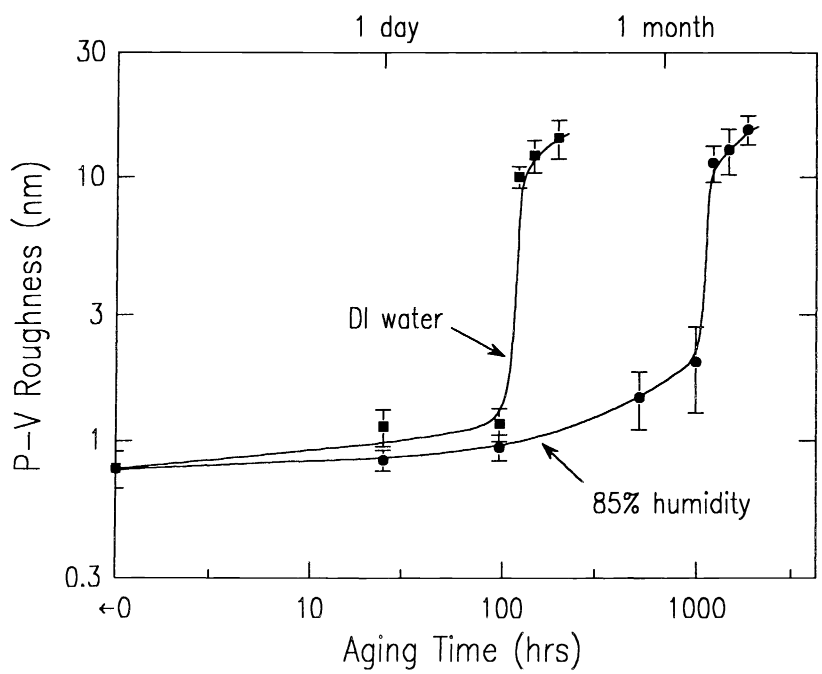

Fig. 4. Peak to valley roughness (measured from 1.5 μm square AFM scans) as a function of aging time under zero stress in 85°C (■) deionized water and (●) 85% relative humidity.

required in order to be able to successfully strip the fiber for connectorizing operations. This fiber approaches this limit in both environments.

3.3 Surface Roughness

Fig. 4 shows the roughness of the fiber surface measured using atomic force microscopy (AFM) as a function of the aging time in the two environments. The data are qualitatively similar to the aging data; there is initially little change in the roughness until an abrupt knee is encountered at which the amplitude of the roughness increases by an order of magnitude. Comparison of Figs. 3 and 4 shows that the knees in both strength and roughness occur at almost identical times, supporting the conclusions of earlier work that they are causally related.[8] It should be noted that the abrupt changes in both strength and roughness are still apparent whether the aging time axis is linear or logarithmic.

4. DISCUSSION

The above results clearly show that the abrupt changes in fatigue and aging behavior that have been widely observed for liquid aqueous environments can, at least for some fiber/coating systems, also be observed in vapor environments. This is of considerable practical significance since in most applications the contact of the fiber and liquid water can be avoided, while contact with the vapor can not. It should be noted that the polymer coating in this fiber has otherwise unremarkable properties. It should further be noted that these results are by no means unique; we have observed other fiber/coating systems exhibiting knee behavior at longer times and others showing knee behavior at times substantially shorter than reported here. These results should therefore be considered when designing optical fiber systems; especially those that might not sufficiently protect the fiber from the environment.

Since the fatigue and aging knees can severely impact fiber reliability, it is desirable to be able to predict in advance whether or when such behavior is expected, rather than to simply demonstrate it, as has been done here. Matthewson[17] proposed a model for the behavior in which the strength of fiber is controlled by two separate and independent flaw populations; the first being intrinsic defects at or near the glass surface such as structural defects; the second being surface roughness that forms on prolonged exposure to corrosive environments. The overall behavior is controlled by the weaker of the two defect populations; the fatigue and aging knees occur when surface roughening becomes strength controlling. This model makes certain predictions, including that the aging knee should occur a little earlier in time than the fatigue knee and this is indeed observed in Figs. 2 and 3 as well as in previous work.[4,18] Zero stress aging behavior is usually characterized by little or no degradation in the early stages followed by abrupt and serious degradation at some later time, as shown in Fig. 3. Matthewson's model[17] suggests that during this initial "incubation" stage the surface roughness should be steadily increasing until is becomes strength controlling at the knee. Monitoring the roughness before the knee should then enable prediction of the position of the knee. However, Fig. 4 clearly shows that, at least in this case, the roughness does not develop gradually, but rather, develops suddenly at the knee. Roughness can not be used as a knee predictor in this case, but some phenomenon must be proceeding gradually during the incubation period. The adhesion of the coating is thought to be an important controlling factor for the knee.[10] One possibility is that good adhesion of the coating initially effectively passivates the surface of the glass. However, the adhesion could gradually degrade until the surface is exposed to corrosion by the water. Adhesion loss of the coating was visible in the optical microscope for the fiber aged for periods corresponding to the position of the aging knee. Liquid ligaments, presumably water, were visible in the coating delaminations produced in fiber aged in the humid environment. It is therefore possible that for this particular fiber/coating system the coating adhesion could serve as a predictor for the knee. Correlation of coating adhesion with the aging behavior is a subject of ongoing research.

The recognition of surface corrosion as the mechanism for the aging and fatigue knees has opened new areas of research. Suppression of the corrosion can have a beneficial impact on the long term reliability of fiber in harsh environments. One approach that has been used is to incorporate colloidal (~20 nm diameter) silica particles in the polymer coating. These particles are more soluble than the fiber because of their high surface curvature and so preferentially dissolve in environmental moisture making that moisture less aggressive at the

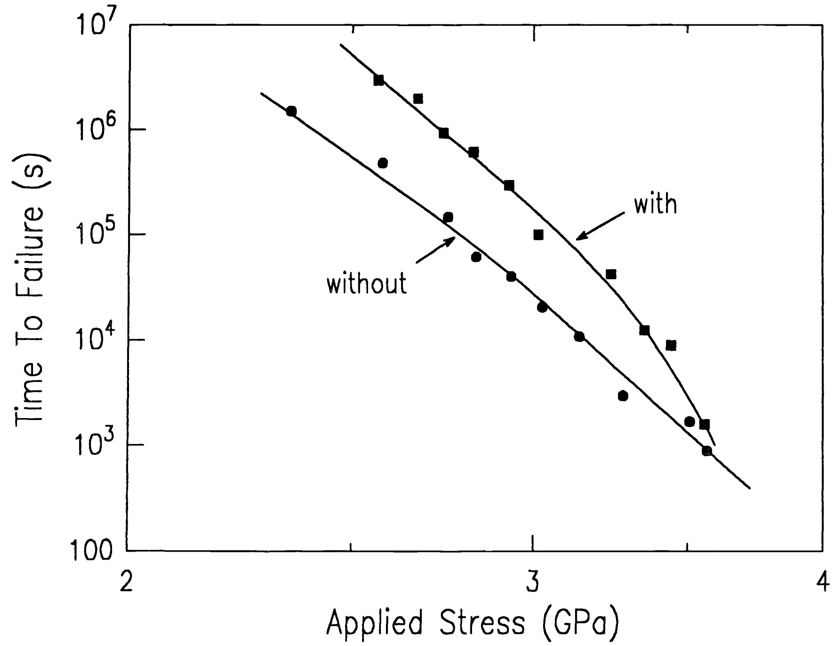

Fig. 5. Static fatigue data measured in two-point bending and a 85°C, 85% relative humidity environment for fiber both with (■) and without (●) ~0.7 wt% of colloidal silica incorporated in the polymer coating.

fiber surface.[19] The addition of silica has been found to delay the fatigue knee in time by orders of magnitude.[20,13] Fig. 5 shows results for fiber containing ~0.7 wt% of silica particles in the polymer coating. Previous results on the same fiber found that the failure times beyond the knee were extended by factors of up to 30 when tested in pH 7 buffer solution.[19,21] In 85°C, 85% relative humidity the coating additive is observed to still be effective, prolonging the lifetime by factors of up to ten. These results show that while the knee is an important reliability consideration, it can be controlled to some extent by suitable coating additives.

5. CONCLUSIONS

This work clearly demonstrates that, at least in some fiber/coating systems, a fatigue knee can be observed and strength degradation can occur in vapor, as well as liquid environments. While the degradation rate is typically an order of magnitude slower than in liquid, the eventual strength degradation in vapor can be just as severe. This shows that the degradation, originally thought to be only relevant to the harshest conditions, is an issue in a much wider range of applications and environments. However, it is demonstrated that the degradation can be controlled to some extent by incorporating colloidal silica particles in the polymer buffer coating. This technique promises to be a useful and relatively inexpensive approach to improving fiber reliability.

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