Coating additives for enhanced mechanical reliability of fused silica optical fibers: effect on mechanical and optical performance

Y. S. Shiue[*a], M. J. Matthewson[*a], P. R. Stupak[**b] and M. J. O'Connor[b]

[a]Department of Ceramic and Materials Engineering, Rutgers University, Piscataway, NJ 08854-8065
[b]SpecTran Corporation, Sturbridge, MA 01566

ABSTRACT

It is now well known that fused silica optical fiber can suffer from enhanced strength degradation after prolonged exposure to aggressive environments. This is caused by corrosion of the glass surface by moisture leading to roughening, strength loss, and, potentially, problems with handleability. It has been found that addition of nanosized silica particles to the polymer coating can improve the long term mechanical reliability by slowing corrosion and delaying the onset of strength loss. However, previous studies have shown that addition of these particles can lead to unacceptably high added optical loss, when measured using the "basketweave" test. In this work, it is shown that the added loss caused by coating additives can be reduced by improving the mixing and dispersion of the silica powders in the polymer. It is further shown that well dispersed powders still substantially improve the long term fatigue and aging behavior. This clearly shows that coating additives can improve the mechanical reliability without significantly degrading the optical performance.

Keywords: optical fiber, static fatigue, fatigue knee, coating additives, optical loss

1. INTRODUCTION

Polymer coatings are used to protect optical fibers from damage caused by exposure to handling. While the commonly used UV-curable acrylate coatings provide protection from mechanical damage, they are permeable to water and thus can not stop ambient moisture reacting with the glass surface. This reaction causes strength degradation, either with or without an applied stress; *i.e.*, fatigue[1,2] and zero stress aging.[3,4] Although the fatigue and zero stress aging behavior are not properties of the coating (they occur even for bare fiber[3]), the severity of the fatigue knee and the zero stress aging are strongly influenced by different types of coating.[2,5] Rondinella *et al.* have shown that adding nano-sized silica powders to the coating of a single coated optical fiber can improve its long term mechanical reliability.[6] They found that the coating additive not only delays the onset of the fatigue knee but also reduces the strength degradation due to zero stress aging. A more recent study showed that adding the silica powder only to the secondary coating of a dual coated fiber can also improve the long term mechanical reliability.[7] However, the same study also found that large microbending losses were induced by incorporating the additive in the secondary coating.[7]

The earlier work suggested that the silica powder was poorly dispersed in the prepolymer and that large, micron-sized agglomerates, were causing enhanced loss. It is the purpose of this work to improve the dispersion of the additive in the liquid prepolymer, in an attempt to reduce the microbending loss while maintaining good mechanical performance. In this work, the behavior of the optical fibers with secondary coatings containing 3 wt% silica powder is investigated. Various pretreatments were applied to the powder before addition to the liquid prepolymer, with the aim of improving the powder dispersion. The microbending loss of these fibers was measured, and the long term mechanical behavior was studied by performing both static fatigue and zero stress aging tests. These results are compared to the behavior of a reference fiber with no coating additive.

* Correspondence: Email: yshiue@rci.rutgers.edu; mjohnm@fracture.rutgers.edu
** Now at Alcatel, France

2. EXPERIMENTAL PROCEDURE

Specimens of coatings containing silica additive were prepared by mixing 3 wt% of silica powder in a commercially available secondary coating prepolymer. A commercial fumed silica powder†, with a nominal diameter of 20 nm, and a specific surface area of 200 ± 2.5 m^2/g, was used in this work. Four kinds of secondary coatings were prepared: (1) a reference with no coating additive; (2) a specimen with "wet" powder; (3) another with "dry" powder; (4) and one with powder which was pretreated with a dispersant. Silica powder stored in ambient laboratory environment is considered "wet" due to the presence of adsorbed moisture. The "dry" powder was obtained by heating it to ~120°C in an oven for two days. The powder with the dispersant was prepared by adding a commercially available dispersant to the "dry" powder. The silica powders were mixed into the liquid prepolymer by machine stirring for 3 hours.

Four lengths of 125 μm diameter multimode silica optical fiber, with 200/250 μm primary/secondary coating diameters, were drawn with the same primary coating, but with the different secondary coatings described above. The microbending loss of the fibers was measured in ambient environment, using a "basketweave test" technique,[8] after 0 and 24 hours after winding on the cylinder. The microbending loss was determined from the difference between the OTDR attenuation of the fiber on the basketweave cylinder and the one meter circumference measurement spool. Two-point bend static fatigue[9] was conducted in 90°C pH 7 buffer solution at various applied stresses; 20 specimens were broken at each test condition. The zero stress aging experiments were conducted in 90°C pH 7 buffer, with aging times varying from 0 to 100 days. The residual strength was then measured in 25°C pH 7 buffer using the dynamic two-point bend technique,[10,11] at a faceplate speed of 1000 μm/s.[12] 15 specimens were broken at each condition.

3. RESULTS AND DISCUSSION

Figure 1 shows the time to failure of the four fibers as a function of applied stress in two-point bending. The fiber with no additive fails sooner than the other fibers. The fatigue knee also appears in a shorter time for this reference fiber than the

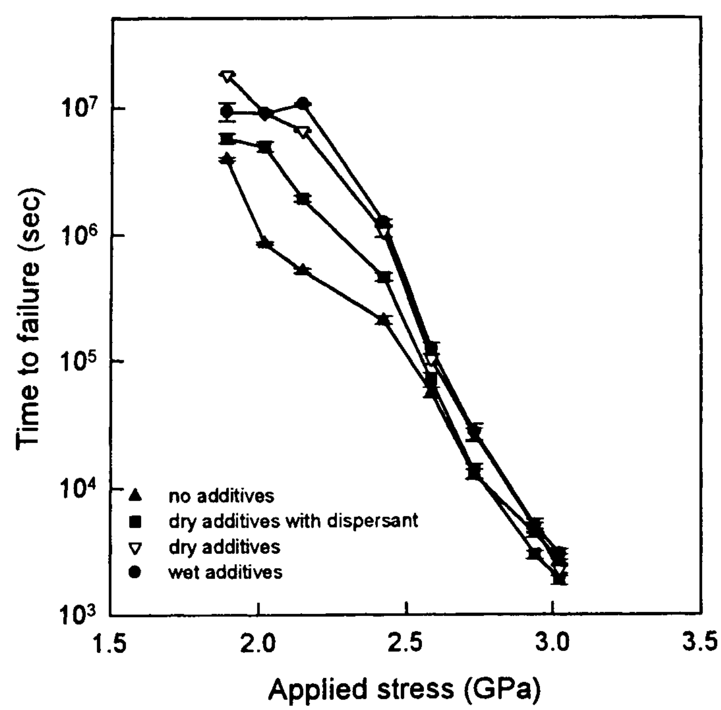

Figure 1. Static fatigue data of fibers with various coating additives.

† Cab-O-Sil M5 fumed silica, Cabot Corp., Tuscola, IL 61953, USA

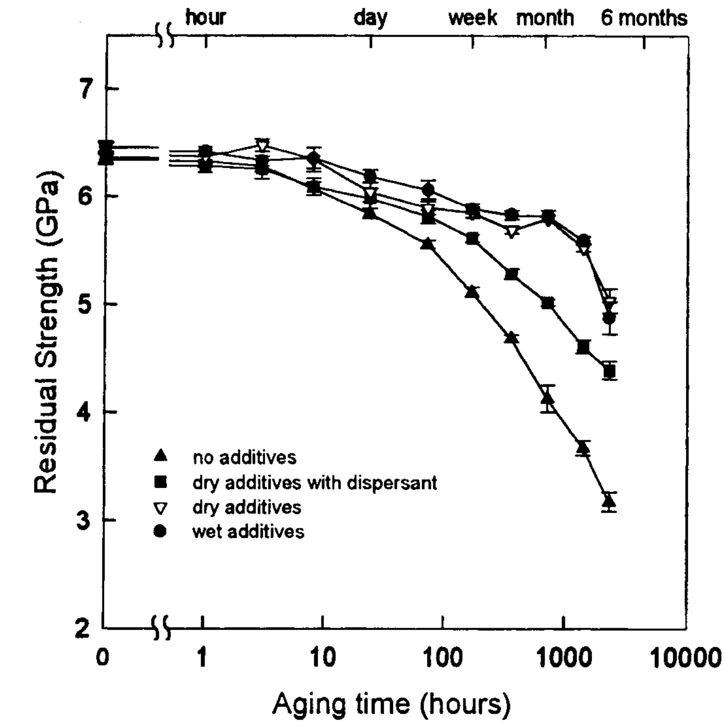

Figure 2. Residual strength of the fibers as a function of zero stress aging time.

ones with the additives. Fibers with "dry" and "wet" additives show similar behavior under high applied stress (> 2.5 GPa). In the low stress region, there is some difference, though more data are required to determine the significance of the difference. The fiber containing additive with dispersant, although showing better long term performance than the reference fiber, is clearly not as effective as the specimen with no dispersant.

Figure 2 shows the residual strength of the fibers after zero stress aging. The fiber with no additive degrades significantly faster than the others. After 100 days, its strength approaches 3 GPa and, therefore, could exhibit some handleability problems. The fiber with "wet" and "dry" additives show approximately the same residual strength, both perform better than the other two fibers. The fiber containing additive with dispersant shows intermediate behavior. These results are consistent with the static fatigue results: the rate of strength degradation is slowed by the silica additive, but the effect is smaller if dispersant is used.

It was observed that "wet" silica powder contains 0.64 wt% of water, which was removed after heating in the oven. The fiber with "dry" additive does not show significantly higher strength than the one with "wet" additive, indicating the small amount of water in the powder is not critical for controlling the strength degradation of the fiber. However, more information is needed to confirm the different behavior past the "fatigue knee" for these two fibers, as shown in figure 1. The additive with dispersant is less effective. This might be because the particles are more strongly bonded to the polymer and are, therefore, less reactive with water. However, while the role of the dispersant is not clear, these results are similar to an earlier study on single coated fibers.[13]

Table 1 Added optical loss as a function of silica additive, additive treatment, and basketweave spool residence time (dB/km).

Fiber	No additive	Dry additive (3%)	Wet additive (3%)	Additive with dispersant (3%)
0 hr	~0.1	0.8	0.3	0.5
24 hr	<0.1	0.5	0.1	0.25

Table 1 shows the added room temperature microbending loss obtained immediately after spooling, and after a residence time on the basketweave spool of 24hr. Both the initial added attenuation increases (t = 0 hr) and attenuation decay as a function of time (t = 24 hr) characterize the microbending performance. The time dependent microbending attenuation decrease, after basketweave spooling, was thought to be a result of relaxation of the winding tension by viscoelastic deformation of the polymer coating.[8] Earlier results of room-temperature basketweave microbending loss, obtained from fiber with hand stirred coating mixtures, are shown in table 2.[7] Clearly, the added losses shown in table 1 are lower than the previous results for 3% silica additive, indicating the optical performance can be improved simply by enhancing the stirring technique. However, the fiber with coating additive still has the relatively higher added loss compared with the reference fiber. Different additive states have different effects on the added losses, suggesting it is possible to change the treatment of the powders to optimize the optical performance. Further investigation on this issue is needed.

Table 2 Added optical loss as a function of amount of silica additive and basketweave spool residence time (dB/km). (ref. 7)

Fiber	No additive	1% additive	3% additive	6% additive
0.1 hr	0.1	0.8	1.25	6.3
26 hr	0.05	0.43	0.73	5.3

4. CONCLUSIONS

The long term mechanical reliability and the microbending losses of four silica optical fibers, which have 3 wt% silica powders in the secondary coating, have been investigated. The secondary coatings were prepared in the laboratory by adding the silica powders into liquid acrylate coating and the mixture was machine stirred. Different treatments were applied to the powders before they were added to the coating. The static fatigue and zero stress aging behavior of the fibers were studied using two-point bend techniques, and microbending losses were measured by a "basketweave test".

The static fatigue results show that all the coatings with the silica additive improve the long term mechanical reliability by prolonging the time to failure. In contrast to the coating with "dry" or "wet" powders, the coating with dispersant has a smaller effect on slowing the fatigue rate. Zero stress aging studies show the coating additives reduce the rate of strength degradation. Similar to the static fatigue results, the coating with dispersant has a smaller effect on preserving the strength of the fiber.

In comparison with the earlier results, the added microbending losses presented in this work are substantially reduced, indicating it is possible to minimize the microbending loss by enhancing the coating mixing procedures. The silica powders, with different pretreatments, have different effects on the microbending losses, suggesting the behavior can be enhanced by optimizing treatment of the additive.

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