Effect of Secondary Coating on the Fatigue and Aging of Fused Silica Fibers

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ABSTRACT

The mechanical reliability of fused silica glass fibers is significantly influenced by the properties of their polymer coatings. The primary coating, which is in contact with the fiber surface, is expected to control the chemistry there, but the secondary coating does have a considerable effect on the strength and aging behavior of the fiber. This observation is confirmed by data obtained for ten fused silica fibers, having the same primary coatings but ten different secondary coatings. These fibers were aged at 85°C in both de-ionized water and 85% relative humidity for up to 6 weeks and the residual strength as a function of aging time was measured. Dynamic fatigue measurements were carried out on as-received and aged fibers using two-point bending. The results show that the secondary coating has a notable effect on the aging behavior and the coating strip force, but does not greatly influence the dynamic fatigue parameter.

Keywords: Optical fiber, fused silica, coating, zero-stress aging, dynamic fatigue, two-point bending, strip force.

1. INTRODUCTION

Low loss fused silica optical fibers are an excellent medium for the transmission of data. These fibers are made from silica and hence are susceptible to surface flaws which can lead to catastrophic failure.[1] Coatings are used to protect the fiber from abrasion and other flaw inducing mechanisms. These coatings may be polymers, carbon or metals. But for most applications, dual layers of polymer are used. The primary or inner coating has a low modulus to reduce microbending optical losses, while the secondary or outer coating has a higher modulus. The coating acts as a barrier and impedes the transport of the outside environment to the silica surface and reaction products away.[2] Polymer coatings are readily permeable to environmental agents such as water. Hermetic coatings, such as metals have been effective in preventing water from getting in, but can induce microbending losses.[3]

Many researchers have investigated the properties of polymer coatings and their effect on the mechanical reliability of the fiber. Properties like adhesion of the primary to the surface,[4] water permeability, water vapor transport,[5] coating thickness, wetting angles, coating composition,[6,7] ink materials on polymer coatings *etc*,[8] all affect the strength and fatigue of the fiber. In most of the prior work, both the primary and secondary polymer coatings have been looked at as a complete entity.

The primary coating is in contact with the surface of the fiber. Hence, it is normally assumed that it controls the chemical environment at the fiber surface, and consequently controls the fatigue and strength characteristics, which are sensitive to the adhesion properties of the primary.[9] The primary coating though, is not solely responsible for controlling these phenomena. To investigate whether the secondary coating also has any significant effect on the properties of the fiber, fibers having the same primary coating but various secondary coatings were used in this study. From our results, it clear that the secondary coating also plays an important part in mechanical reliability. In this study, the thickness of the secondary was kept constant, and only the nature of the coating was changed to see the impact of this change on the strength and fatigue of the fiber.

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2. EXPERIMENTAL PROCEDURES

Ten dual-coated fibers were drawn at Kwangju Institute of Science and Technology (KJIST), each of them having the same urethane-acrylate primary but different secondary coatings. All the fibers had a glass diameter of ~ 125 μm and an overall diameter of ~ 250 μm. We selected three representative samples from the results. These fibers were designated X, Y and Z. The remaining ten samples exhibited behavior similar to these three. A commercial fiber, C, was also tested to compare with the results of the sample fibers. This fiber had both primary and secondary coatings which were different from the experimental fibers studied in this work. Measurements were made of the strength, dynamic fatigue and coating strip force of the fibers in both as-received condition and after aging.

The zero-stress aging was carried out in environments of 85 ± 0.5^{o}C de-ionized water and at 85 ± 0.5^{o}C/$85 \pm 2\%$ relative humidity (RH) air to accelerate the aging. The fibers were aged for times of 0 hrs (as-received), 3 hrs, 8 hrs, 168 hrs (7 days), 720 hrs (30 days), and 1008 hrs (42 days) and the residual strengths for fifteen samples each were made in two-point bending at a constant faceplate velocity of 1000 μm/s. Dynamic fatigue measurements were made in two point bending on as received fibers and fibers aged for 30 days in 85°C/85% R.H. and in 85°C DI water. Faceplate velocities of 3000, 1000, 200, 40 and 5 μm/s were used. We also used the liquid environment to further accelerate the aging to help distinguish between fibers which did not degrade in humidity.

Coating strip force measurements were conducted on the fibers according to FOTP-178 [110]. Strip force was measured for fibers in the as-received condition and after aging for 30 days in both environments.

The fibers were preconditioned overnight at 25°C/50% R.H. before the residual strength, dynamic fatigue and strip force measurements were all made. For the strength and fatigue measurements of fibers aged in humidity, the fibers were tested in air. The fibers aged in water were tested in water at 25 ± 1^{o}C *i.e.* while still wet. Fifteen samples were tested for each strength determination.

The rationale behind using two-point bending for strength measurement is that this testing method avoids the occasional weak "manufacturing" defects in the fiber induced while the fiber is being drawn. This will focus our study on the influence of the secondary coating by avoiding occasional extrinsic weak defects which might obscure the results.

3. RESULTS AND DISCUSSION

3.1 ZERO-STRESS AGING IN WATER

Figure 1 shows the strength of the representative fibers as a function of aging time in 85^{o}C de-ionized water. The error bars on the data points in the graph represent a 95% confidence interval. The error bars of the fiber C are too small to be seen. The as-received strengths of the fibers clearly are not the same. The fibers X, Y and Z had initial mean strengths of 6.1 GPa, 6.3 GPa and 5.6 GPa, respectively. The reason for the differences is not clear given that the preforms and the primary coatings of X, Y and Z are nominally the same. However the fibers were approximately four months old when tested and it is possible that components in the secondary coating could have migrated to the fiber surface and changed the local environment (*e.g.* pH).

In some earlier work, there has been a distinction among the different types of knees observed.[121] Sometimes for different specimens, the emergence of the knee is abrupt and sometimes it is gentle, *i.e.* the knee might start off at the same time, but the rate of strength degradation after the knee might be quite different. Sometimes the strength appears to level off at long times while other specimens continue to degrade.[6] The data in figure 1 show these qualitatively different behaviors which have been seen in the past. Fiber Y shows the behavior most often seen in the literature; the initial strength is more or less retained until some time at which a rather pronounced and steep knee is seen. In this case, the strength reduced by around 40% after 30 days and by around 60% after 42 days of aging. Fibers X and Z, on the other hand show more gentle slopes beyond the knee region.

Fiber Z lost only around 20% of its strength after aging for 42 days. The commercial fiber C shows no knee out to 42 days. It is assumed that a knee will appear at some later time however, and so this fiber will probably fit into one of the

above categories. Fiber X shows a suggestion of a very important type of behavior. The strength appears to have leveled off at long times; that is, there appears to be an aging limit. It is clearly very important both practically and scientifically to know if a limiting strength is reached at long times.

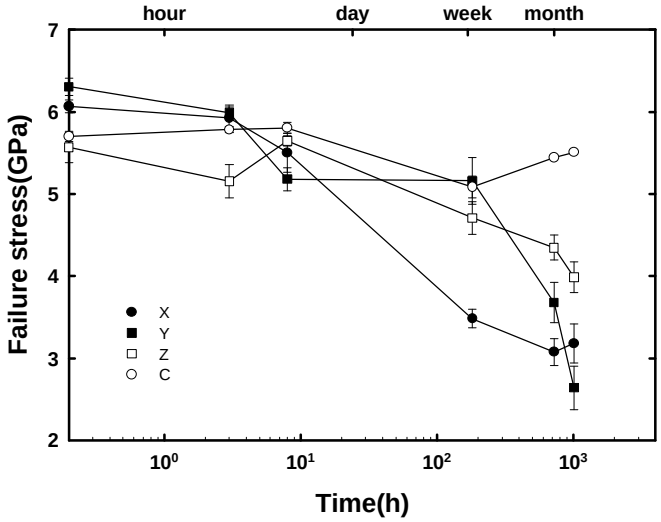

Figure 1 Residual strength after aging *versus* aging times for fibers aged in 85°C water.

The reduction in strength of a fiber is now phenomenologically understood. The water diffuses through the coating in the order of minutes[132] and after a period of time, in some cases, delaminates the coating from the fiber.[143] These water molecules, now on the surface of the silica, cause inhomogeneous dissolution of silica, thus roughening the fiber surface.[154] The dissolution of silica is inhomogeneous due to the intrinsic inhomogeneity of the silica glass structure and also perhaps due to inhomogeneities in the coating adhesion. The rough spots act as stress concentrators, causing failure of the fiber at lower stress.

As discussed above, the aging characteristics of the fibers presented are substantially different. The primary/secondary coating acts as a barrier to water getting to the surface of the fiber, but it has been proved that the moisture penetrates the coating on a time scale of 10^2 to 10^3 s at room temperature.[132] The strengths after aging for these time scales do not differ much. It is therefore most unlikely that the diffusion of moisture is controlling the strength degradation. It is clear that the transportation of some chemical species (*e.g.* large ions) to the glass surface, or the transport of dissolution products away of the surface controls the strength of the fiber. The different behaviors observed here are clearly due to the changes in the secondary coating, as this is the only difference between the fibers. Since the secondary coating has a higher elastic modulus than the primary, it is reasonable to expect it has a lower permeability to large species and so can indirectly control the environment at the fiber surface. As a result it is anticipated that the secondary can strongly influence aging as is indeed shown here.

3.2 ZERO-STRESS AGING IN HUMIDITY

Figure 2 shows the residual strength of the fibers as a function of aging time in the 85% RH/85°C environment. The error bars on the data points in the graph are too small to be seen and represent a 95% confidence interval. The fibers show no significant strength degradation compared to the fibers aged in water. The same was found for the other seven experimental fibers whose results are not shown here. The lack of degradation means that all these fibers comply with the common industry standard requirements. Even compared to the standard sample, C, the fibers do not show very different behavior. Again, the as-received strengths show some variation.

In earlier work it was found that the aging knee occurred approximately 10 times further out in time in 85% RH/85°C compared with 85°C water.[165] Interestingly, fiber X shows a factor of at least 100 given the knee in note occurs at ~ 10 hrs in water but more than 42 days in humidity.

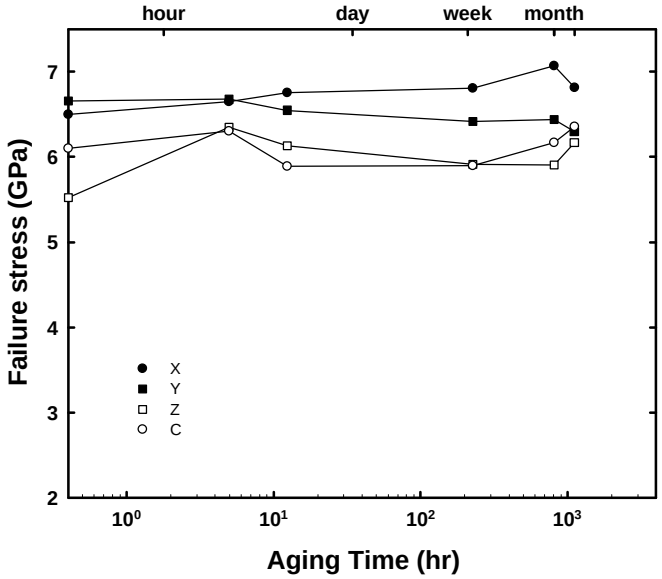

Figure 2 Residual strength after aging *versus* aging time for fibers aged in 85°C/85% RH.

3.3 DYNAMIC FATIGUE

The values of the stress corrosion parameter, *n*, for various test environments are shown in figure 3. The error bars represent a 95% confidence interval. Given the confidence intervals, the dynamic fatigue parameters of the fibers are generally not different from each other.

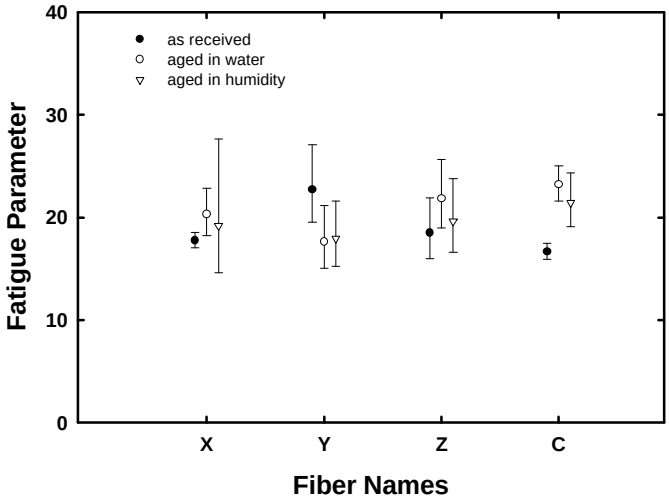

Figure 3 Stress corrosion parameters ($n \pm$ 95% confidence intervals) as received and after aging in 85°C and 85% humidity

In particular, the values after aging do not differ, in contrast to the strength measurements. The data suggest that the secondary coating is not affecting the dynamic fatigue of the fiber, even after aging. Table 1 presents the Weibull moduli of the strength measurements used to determine the fatigue parameters shown in figure 3. The values are typically around 40 for as-received and humidity aged fibers but the values are significantly lower for the water aged fibers.

Table 1 Weibull moduli of the strength measurements used to generate the fatigue parameter in figure 3.

Fiber name	Speed	Weibull Modulus			Fiber name	Speed	Weibull Modulus		
		As received	Water aged	Humidity aged			As received	Water aged	Humidity aged
X	3000	33	10	43	*Z*	3000	18	19	14
	1000	47	9	41		1000	17	14	13
	200	59	6	45		200	18	16	14
	40	55	7	20		40	15	13	14
	5	81	15	39		5	25	22	24
Y	3000	45	9	29	*C*	3000	85	19	46
	1000	64	8	27		1000	31	16	48
	200	62	7	32		200	28	24	29
	40	46	8	25		40	103	18	57
	5	52	10	42		5	55	25	54

3.4 STRIP FORCE

The strip force measured for the fibers is shown in figure 4. The error bars represent a 95% confidence interval.

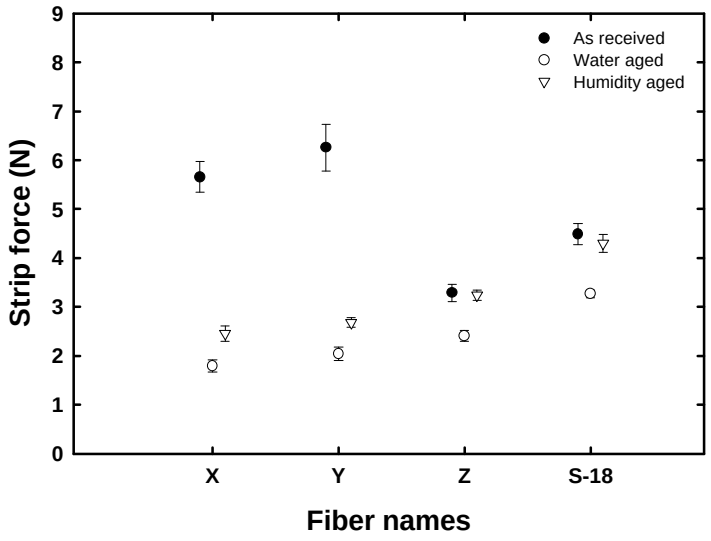

Figure 5 Coating strip forces of the fibers

The strip force of the fibers as-received is greater than that of the fibers aged in humidity for 30 days, which in turn is greater than that of the fibers aged in water for 30 days. This is expected since aging degrades the coating adhesion; the more aggressive environment to a greater extent.

The strip force is a complicated measure of a combination of the coating/glass adhesion and the viscoelastic properties of the coating. An interesting point to note is that the trend of the strip force after aging in 85^0C water for 30 days is similar to the trend in the residual strengths of these fibers aged under the same conditions. As strip force depends on the viscoelastic properties of the coatings, the secondary coating properties do affect the strip force results as can be seen from these data.

The observation that the strength after a given aging treatment correlates with the strip force, might be explained by the suggestion that the secondary coating changes the chemical environment at the glass surface and so affects the adhesion, which then in turn affects the aging *e.g.* the secondary causes rapid adhesion loss, rapid aging occurs and both the strip force and strength will degrade in concert.

5. CONCLUSIONS

The strength, dynamic fatigue and coating strip force of fibers having the same primary coating but different secondary coatings have been measured both before and after aging. The zero-stress aging behavior of the fiber in water brings out significant differences between the fibers. In contrast, the dynamic fatigue parameters were not very different for these fibers. The strip force values are also different for two reasons: secondary could change the chemical environment at the glass surface and the strip force is controlled to some extent by the viscoelastic properties of the secondary.

The results of this investigation clearly show that the secondary coating can have a strong influence on the strength and aging behavior of silica fibers, even though the secondary coating is not in direct contact with the glass surface. The results are consistent with the suggestion that the diffusion of some chemical species other than water, into or out of the coating, is controlling the strength degradation during aging.

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