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ZERO-STRESS AGING BEHAVIOR OF OPTICAL FIBERS WITH VARIOUS PROTECTIVE COATINGS

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ABSTRACT

Optical fibers with various coatings are subjected to zero-stress soaking in water at room temperature and at 80°C. Change in fiber strength over time is tracked using two-point bend strength testing. The aging behavior of the fibers with different coatings is compared and the results are discussed.

INTRODUCTION

Long-term predictions of the mechanical reliability of optical fiber are based on the initial fiber strength and the degradation of that strength over a fiber's operational lifetime. Strength degradation, in turn, is determined by how a fiber responds to the effects of fatigue and aging, which are two distinct mechanisms responsible for fiber strength degradation. They can be accelerated by environmental factors, especially in applications demanding high operational temperatures or harsh chemical conditions. Therefore, it is important to understand the effect of fatigue and aging since it adds uncertainty in estimating fiber lifetime.

Fatigue, also known as stress corrosion, is defined as moisture-assisted sub-critical crack growth under stress. Sub-critical crack growth refers to the slow growth of the most severe micro-cracks that exist on a glass fiber surface. The size of the most severe micro-cracks dictates the actual mechanical strength of the fiber. The mechanism of the crack growth may be understood to occur when the reaction of water with silica results in the breaking of the strained silicon-oxygen bond at the crack tip, followed by formation of a weak hydrogen bond, as indicated below:



where “ $_$ ” represents three Si-O bonds and “ \cdots ” a hydrogen bond. Under stress (tensile or bending), sub-critical crack growth will slowly proceed to a delayed failure. The crack growth velocity, \dot{c} , defined as the change in crack length as a function of time, is characterized by the power-law relationship:

$$\dot{c} = AK_I^n \quad (1)$$

where A is a constant dependent upon the environment, K_I is the stress intensity factor, and n is the fatigue resistant factor, also known as stress corrosion resistance factor. The stress intensity factor K_I is defined as a function of the applied stress and the glass crack:

$$K_I = Y\sigma_a\sqrt{c} \quad (2)$$

where: Y = crack shape parameter
 σ_a = applied stress
 c = crack depth (or length)

When the applied stress σ_a reaches the failure stress σ_f , then K_I exceeds the critical stress intensity factor K_{IC} , which is a constant ($K_{IC} \approx 0.79 \text{ MPa}\cdot\text{m}^{1/2}$) and failure occurs. Since the stress intensity factor K_I as expressed in equation (1) is always less than one, a higher n value has a strong effect on impeding the effect of fatigue. Most standard optical fibers have an n value of around 20, while hermetically coated fibers have a fatigue factor, n , greater than

100. The n value can be viewed as a measure of how well the glass surface is isolated from ambient moisture, hence the hermeticity of the fiber against the chemical reaction occurring at the crack tips with water molecules, precluding the onset of fatigue.

The n value of silica optical fibers can be conveniently used to estimate the mechanical lifetime of the fiber, e.g. in the approximation below:¹

$$t_f = \left(\frac{\sigma_p}{\sigma_a} \right)^n \quad (3)$$

where: t_f = time to failure (seconds)
 σ_p = proofstress
 σ_a = application stress
 n = fatigue factor

As the stress corrosion resistance factor increases, crack growth leading to failure at K_{IC} is impeded such that 1.) the time to failure under standard application stresses is greatly extended or 2.) a higher application stress may be employed over the conventional lifetime of the fiber. This latter point has important implications for fibers used in high-stress applications and/or corrosive environments that can lead to accelerated strength degradation and failure.

Compared with the phenomenon of fiber fatigue behavior, aging is less well understood, especially for fibers with different coatings. Aging is defined as a process of fiber strength degradation in the absence of stress. In this case, crack growth is probably not driven by stress, but by the increase of the surface roughness as a result of dissolution of silica on the surface of the fiber by water corrosion. Research has led to understanding how coating conditions of a fiber will affect water (or other active species) corrosion; this research has been variously split into the following avenues:

1. Absorption and migration of water to the glass surface^{2,3}
2. Adhesion and/or delamination of the primary coating layer^{4,5}
3. Chemistry/pH at glass/coating interface^{6,7,8}

Diffusion and corrosion are both thermally-activated processes and the correlation between soaking temperatures and aging has been well documented.^{9,10} Fiber samples aged in water often show an aging “knee” characterized by an abrupt drop in strength after a given time (Figure 1). This accelerated drop is believed to be related to a change in the strength-limiting basis _ from intrinsic flaws on the fiber surface to surface roughness caused by dissolution of silica on the surface of the glass. When the most severe flaw is caused by inhomogeneous dissolution of the silica surface, aging becomes a predominant mechanism.

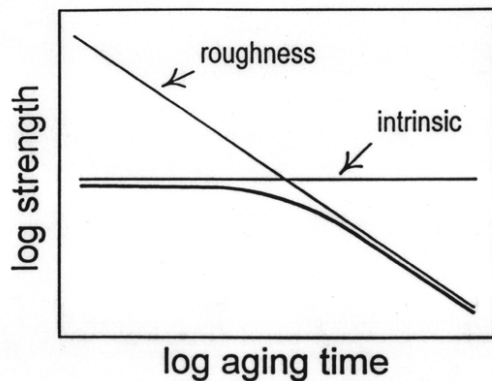


Figure 1. Aging behavior with a “knee”

Most of the coating investigations cited have focused exclusively on acrylates, while studies of specialty coating types often compared fiber samples obtained from different vendors, thus ignoring processing conditions that may affect both glass and coating behavior – differences that may be accentuated with aging, especially in accelerated testing conditions. As the application of optical fibers moves from telecommunications into more demanding fields such as oil well sensors,¹¹ medical devices, or in-flight data links, understanding the effects of aging and fatigue on fiber reliability becomes more important to ensure long-term mechanical reliability. In this paper, we report and discuss the results of our experiments on aging of fibers with acrylate, polyimide, carbon-acrylate, and carbon-polyimide coatings soaked in water at room temperature and 80°C. This is a part of on-going research with extended scope, aiming to understand the behavior of fibers with various coatings under various conditions.

EXPERIMENTAL

Coating systems used in this study:

- Acrylate
- Polyimide
- Carbon-acrylate
- Carbon-polyimide

To minimize variations in strength that may arise from different glass sizes or drawing conditions, all fiber was drawn from the same preform; the furnace temperature, draw speed, draw tension, and fiber size were all held constant. All samples were pulled from the same draw furnace. Because of the slow draw process for thermally cured coating, the draw speed for all samples was less than 1 meter per second. The glass size was held at 125 μm for all samples.

The acrylate fiber sample is coated with a secondary acrylate coating only (i.e., not dual-coated) for direct comparison of the chemical effect of this coating versus other homogenous coatings. The acrylate coatings were applied and cured with ultraviolet (UV) radiation. The polyimide coatings were cured with thermal ovens. The carbon samples are prepared by pyrolysis of a hydrocarbon gas on the surface of the fiber, resulting in a very thin layer chemically bonded to the surface of the glass.¹² The geometries of the fiber samples are summarized as follows:

<u>Coating type</u>	<u>Coating thickness</u>
Acrylate	37 μm
Polyimide	7 μm
Carbon-acrylate	$\approx 400 \text{ \AA} + 37 \mu\text{m}$
Carbon-polyimide	$\approx 400 \text{ \AA} + 7 \mu\text{m}$

All samples were proof-tested at 100 kpsi and equilibrated in ambient environment before baseline strength testing, then cut into approximately 30 10-inch samples. The samples were soaked with zero-stress under the two conditions below:

- Distilled water at room temperature
- Distilled water at 80°C

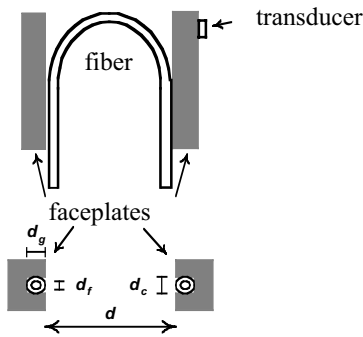


Figure 2. Schematic of a two-point bend testing

Two-point bend testing uses two moving faceplates to hold the fiber sample within two grooves and applies a bending stress to the sample at a controlled strain rate. Figure 2 is a schematic of a two-point bend tester. As these two faceplates move closer towards each other, the bend radius of the fiber becomes smaller, corresponding to a greater strain. Therefore the outer surface of the fiber experiences an increasingly greater tension. When the K_I value exceeds the K_{IC} value due to the tension increase, fracture occurs and a transducer picks up the sound of the break. The computer that controls the tester records the distance between the two faceplates, d . The breaking strain and stress then can be readily calculated.

Two-point bend testing¹³ was chosen because its gage length is so small, usually less than 10 mm at break, that it allows focusing on the intrinsic strength of the fiber rather than the extrinsic or “weakest link” strength found with tensile testing in a much larger sample. To ensure the reliability of the data, measurement is repeated several times.

Fiber samples were removed from soaking tanks at pre-determined time intervals and tested with two-point bend testing at a strain rate of 4%/minute. The median strength is reported with ranges for all samples.

RESULTS AND DISCUSSION

Initial strengths for the four types are given in Table 1.

	Median strength (kpsi)	Range (kpsi)
Acrylate	926	109
Carbon-acrylate	589	30
Polyimide	1004	40
Carbon-Polyimide	597	35

Table 1. Summary of initial strength values for the samples

The change of strength as a function of time is shown for acrylate coated fibers soaked in water at room temperature and 80°C in Figure 3. In general, the fiber strength decreases with soaking time. It seems that there exist two distinctive phases in the strength-time curves. For samples soaked in room temperature water, the strength decreases quickly over time and seems to reach a plateau at ~90% of its initial strength after ~28 days. For the fiber samples soaked in 80°C water, the initial decrease lasted almost 70 days until the strength reached ~50% of its initial strength and then the strength in essence remained steady over time. This may be explained by saturation of the water that migrated to the glass-coating interface with dissolved silica. Conceivably, the activity of water to dissolve silica decreases as it dissolves more silica. It is clear that the higher the temperature, the greater the strength reduction. The fiber soaked in water at 80°C showed a significant drop in strength in one day (to 700 kpsi from 900 kpsi), while the fiber strength remained the same at room temperature even after two days. It is also clear that the range of the data, expressed by the error bars, increases with the soaking temperature (~40 kpsi for the room

temperature data and 100 to 200 kpsi for the 80°C data. This indicates that the distribution of flaw sizes on the fiber surface become greater, leading to greater uncertainty in lifetime prediction.

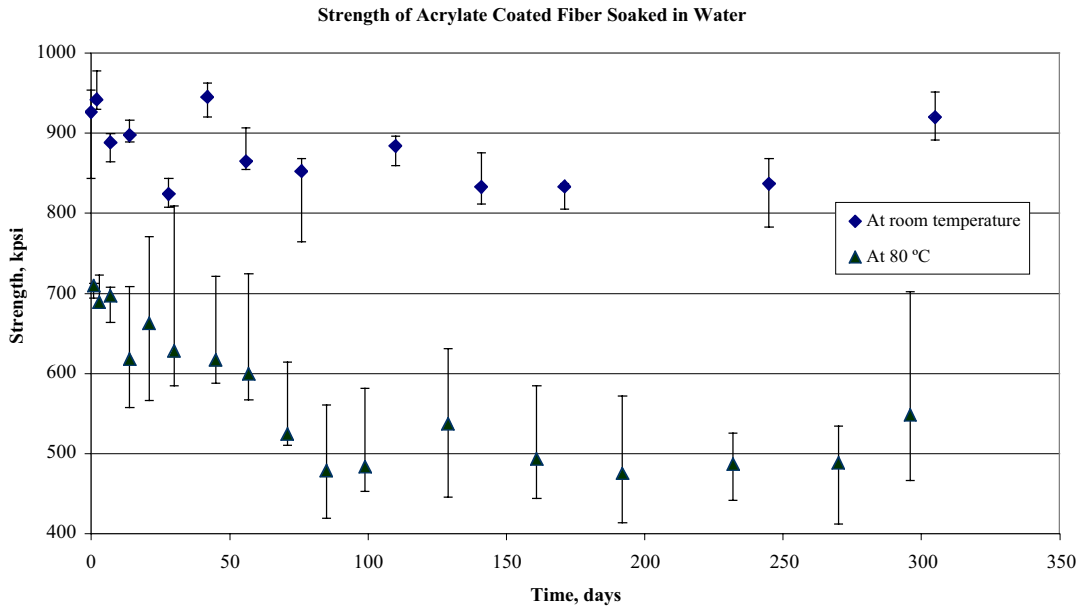


Figure 3. Strength of acrylate coated fibers soaked in water at room temperature and 80°C

Figure 4 displays the data for fiber strength as a function of time for carbon-acrylate coated fibers soaked in water at room temperature and 80°C. The fibers remained essentially unchanged in strength even in 80°C water over long period of time. The apparent difference in behavior between the curves shown in Figures 3 and 4 can be positively attributed to the carbon coating. Carbon coating is known to provide resistance to fatigue, as it is hermetic to water. This observation allow us to conclude that the carbon coating on silica optical fiber is also an anti-aging layer for soaking in water even at 80°C for a prolonged period of time. Under the protection of the carbon coating, the fibers remained strong and the distribution of flaw size remained unchanged.

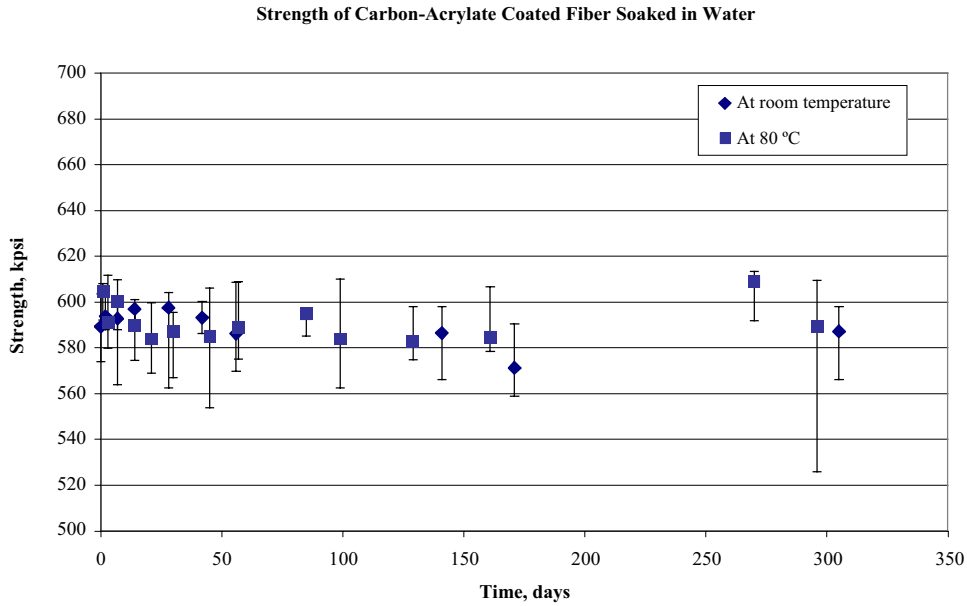


Figure 4. Strength of carbon-acrylate coated fibers soaked in water at room temperature and 80°C

Strength degradation during aging for polyimide-coated fibers is plotted in Figure 5. The fiber strength for fibers soaked at room temperature kept its initial value for almost 14 days. It then decreased continuously and reached ~90% of its initial value. It is not clear whether the strength reduction will continue. The 80°C data show a constant strength value for the first 7 days within the detection limits of the two-point bend testing and then decrease until reaching a lowest value of ~70% of the initial value at around 230 days before stabilizing. After 230 days, the strength appeared to show a slight increase; a similar increase is also suggested in Figure 3. It is possible for the strength of the fiber during aging to recover if we assume crack tip blunting that could occur during the dissolution of silica in water. It certainly requires more investigation to understand the mechanism. The flaw size distribution becomes greater for the 80°C data, as indicated by the large range (~100 kpsi).

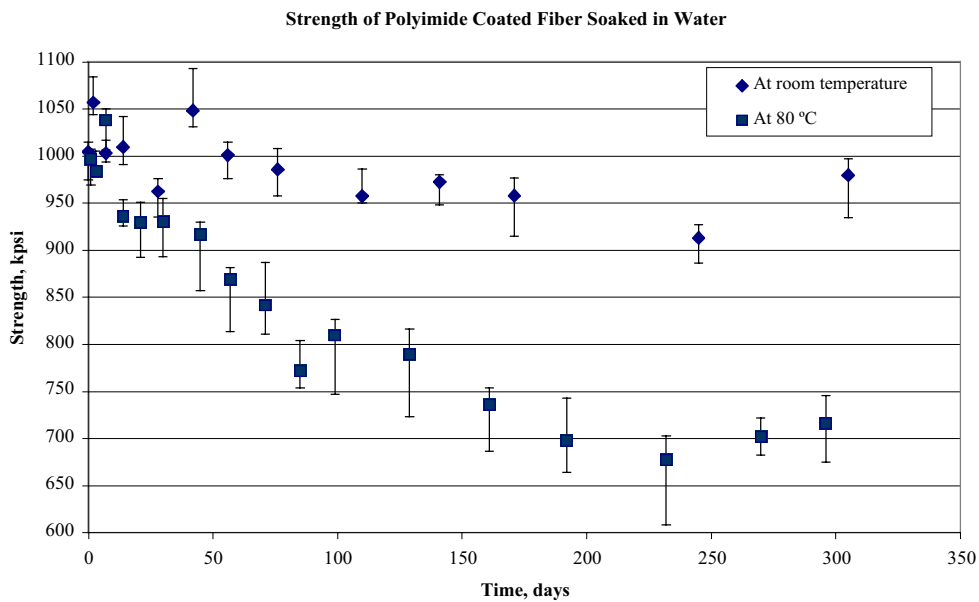


Figure 5. Strength of polyimide coated fibers soaked in water at room temperature and 80°C

Figure 6 shows the corresponding data for carbon-polyimide coated fibers soaked in water at room temperature and at 80°C. Comparing Figures 6 and 4, it seems that once there is carbon coating, the behavior of fiber will be the same whether there is an acrylate coating or a polyimide coating, and whether the fiber is soaked in room temperature water or heated to 80°C. Once again, the protection that the carbon layer provides against aging is evident.

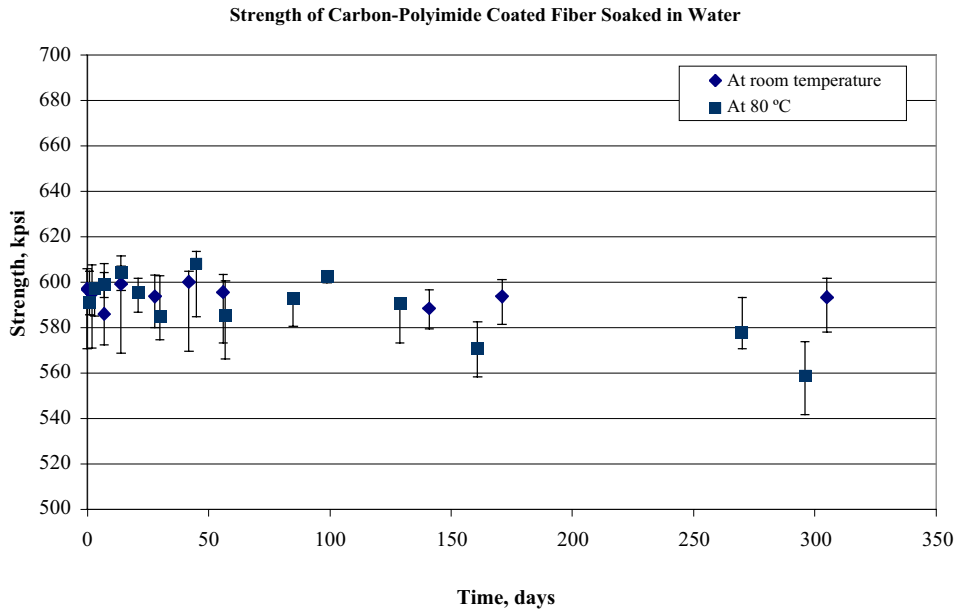


Figure 6. Strength of carbon-polyimide coated fibers soaked in water at room temperature and 80°C.

If we present all four data sets for fibers soaked in water at 80°C in a semi-log plot, we can observe the aging “knee” for both acrylate and polyimide coated fibers, represented by the diamonds and triangles in Figure 7. The knee signifies the onset of a different mechanism for fiber strength degradation, presumably surface damage due to inhomogeneous dissolution of silica by water. Another feature is the much greater reduction in fiber strength that is observed for acrylate-coated fiber than for polyimide-coated fibers. This probably indicates that polyimide coating is more effective in protecting the fiber from aging, although the coating thickness is only $\sim 7 \mu\text{m}$ while that of the acrylate coating is $\sim 37 \mu\text{m}$. Data for carbon-coated fibers remained essentially independent of soaking time.

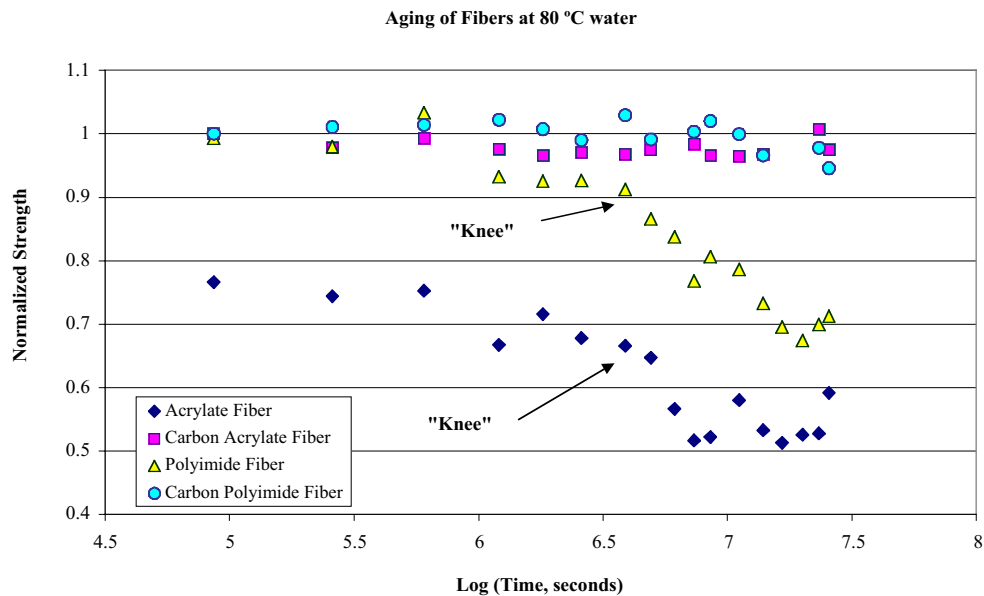


Figure 7. Aging curves at 80°C showing “knee”

CONCLUSIONS

Strength degradation of optical fibers due to aging has been studied using optical fibers coated with acrylate, polyimide, carbon-acrylate, and carbon-polyimide coatings, soaked in water at room temperature and 80°C. Fibers coated with acrylate and polyimide coatings show a reduction in strength after soaking in water. The magnitude of the reduction depends strongly on the water temperature. The uncertainty, expressed in error bars, has very similar dependence on soaking time and soaking temperature. An aging knee is observed in the curves for both acrylate- and polyimide-coated fibers. The strength of carbon-acrylate and carbon-polyimide coated fibers, however, seems to be unaffected by the soaking temperature, and essentially shows no strength degradation during the soaking times. The carbon layer, although only 400 Å thick, effectively stops water migration and thus isolates the silica surface from water. In that sense, the carbon coating acts as an anti-aging layer as well as a fatigue resistance layer.

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