Strength Degradation of Silica Fibers by Acetone Immersion

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ABSTRACT

A single-layer UV-curable polyacrylate-coated telecommunications grade fused silica fiber was found to have a significant reduction in two-point bending strength after immersion in acetone. The two-point bending and tensile strengths of this fiber as a function of immersion time in acetone were determined, and this strength loss was not seen for 0.5-m gauge length tensile specimens. SEM and optical fractography was performed on the weak specimens, and the cause of the strength reduction is proposed to arise from particles smaller than 3 μm in the coating. These particles could cause surface flaws by sliding contact damage incurred during relative motion between the coating and the glass. This sliding could occur either while flexing the fiber in preparation for a bending strength measurement or due to coating elongation. While it is not clear which mechanism is operating, both are consistent with the observation that degradation is only observed for bending strength measurements.

Keywords: Optical fiber, strength, coatings, fractography, reliability, fiber Bragg gratings

1. INTRODUCTION

Removal of the polymer coating from optical fibers is required for a variety of reasons including splicing, connectorizing or writing Bragg gratings.[1] Acetone or other organic solvents are frequently used to remove coating residue either by dipping or by wiping with a wetted cloth. While strength degradation is expected if the process involves mechanical contact with the bare glass surface, degradation due to exposure to the acetone is not expected. We report here results of a preliminary study which shows, for the first time, that soaking in acetone can indeed lead to substantial strength loss under some circumstances.

It is well established that the strength of fused silica optical fiber is controlled mechanically by the presence of extrinsic surface defects and chemically by the stress-enhanced chemical reaction at the crack tip. The micromechanics model used to describe the mechanical effect of these surface flaws is the Irwin extension of the Griffith relation, *i.e.*, failure occurs at a failure stress, σ_f:

$$\sigma_f = \frac{K_{IC}}{Y\sqrt{c}} \tag{1}$$

where Y is a geometrical factor derived from the shape of the flaw, c is the crack length or flaw size, and K_{IC} is the critical stress intensity factor and is commonly referred to as the fracture toughness of the material. The subcritical crack growth model is used to describe the effect of the stress-enhanced chemical attack at the crack tip from environmental moisture. In this model, a crack grows slowly under low applied stress until K_I exceeds the critical value and failure occurs. The mathematical form often used to represent the slow crack growth velocity, $\dot{c}$, is:

$$\dot{c} = AK_I^n, \tag{2}$$

where A is a constant depending on the environment, and n is the stress corrosion susceptibility parameter which is often treated as a material constant. Subcritical crack growth results in a time dependence of the mechanical strength, *i.e.*, fatigue. Michalske and Freiman[2] propose a molecular mechanism for fatigue which predicts that species other than water can

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enhance slow crack growth if they have features similar to water, *i.e.*, a proton donor site near a lone pair orbital. Because acetone has no proton donor site, it is not expected to chemically influence the strength of fused silica fiber.

With more and more information being transmitted down a single fiber, a need for higher reliability exists due to the increased loss of information from just a single fiber failure. For this reason, understanding the root cause of the strength loss of optical fibers after soaking in acetone is of practical importance.

2. EXPERIMENTAL

The strength of a single-layer UV-curable polyacrylate-coated, telecommunications grade, 125-μm diameter fused silica fiber (250-μm diameter coated) was measured in two-point bending and tension. The bending measurements were made at a constant faceplate velocity of 1000 μm/s. Figure 1 shows a schematic of the experimental procedures used to prepare specimens for two-point bending.

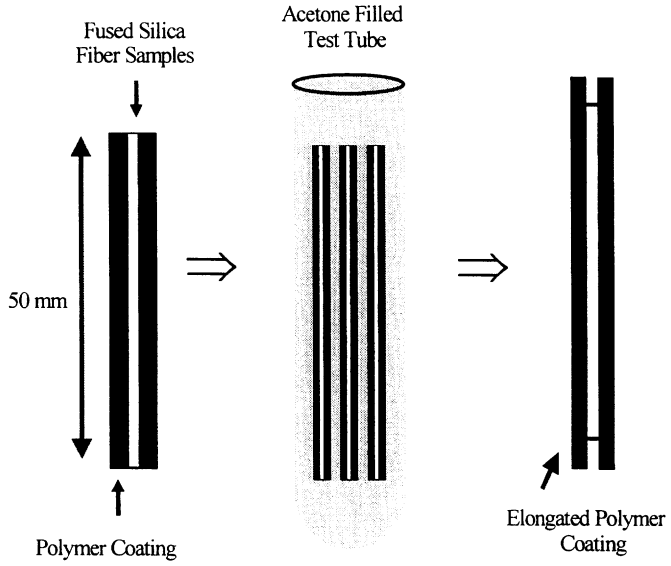

Figure 1. Schematic of sample preparation for bending measurements.

First, the fiber was cut into 50-mm long samples which were randomized and stored at a controlled 50% relative humidity and 25°C for at least 24 hours to ensure a uniform initial strength. Then the samples were completely immersed in acetone in a test tube, so that acetone could penetrate to the coating/glass interface by diffusing either through the coating and/or along the interface from the ends. Specimens were soaked in acetone for various periods extending up to two months, and then they were broken inside a beaker filled with acetone. After each strength measurement, the fiber ends of the weak failures were saved for fracture surface evaluation.

For the tensile testing, the fiber was cut into ~ 2-m long samples which were again stored in an environmental chamber at 50% relative humidity and 25°C for at least 24 hours before testing. Inside the environmental chamber, the center section of each fiber was immersed in acetone while the remainder of the fiber was subjected to the surrounding environment, as shown in figure 2. Therefore, acetone was not able to reach the coating/glass interface from the ends. Each fiber was extracted from the acetone immersion after certain soaking times and then tested immediately in air. The lengths of fiber were gripped by winding on two rubber-coated capstans with a gauge length of 0.5 m, as shown in figure 2. The fiber was loaded by moving the capstans apart at a constant speed of 200 mm/min and the failure load was determined using a load cell attached to one of the capstans.

In addition to the bending and tensile strength measurements, the coating stripping force was measured as a function of acetone immersion time. Figure 3 shows a schematic of the stripping force experimental procedures. One end of lengths of fiber was soaked in acetone for various times. Half of the samples were then dried for 24 hours in the 50% relative humidity and 25°C environmental chamber before stripping, while the other half were stripped immediately after removal from the

acetone. Strip force measurements were made using a standard two-bladed stripping tool mounted in a universal tensile tester at a speed of 500 mm/min as specified in a standard test procedure.[3]

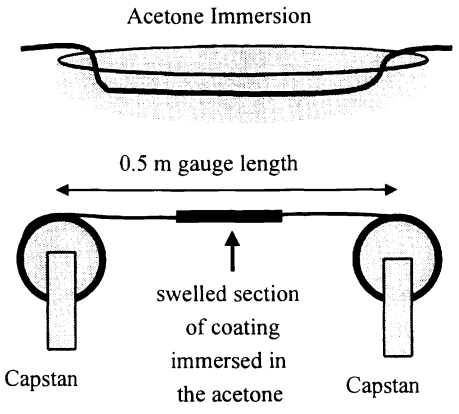

Figure 2. Schematic of tensile experimental set-up.

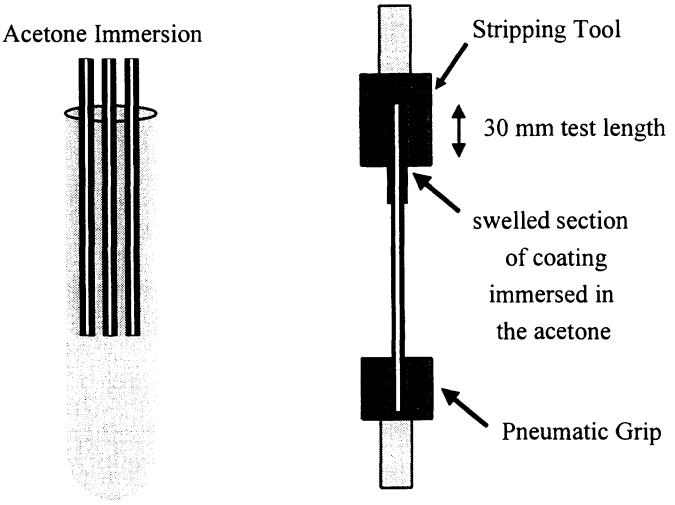

Figure 3. Schematic of stripping force experimental set-up.

3. RESULTS & DISCUSSION

Several dual-coated fibers and one single-coated fiber were tested in bending to determine if they showed any weakening from acetone soaking. In general, these fibers initially exhibited a slight strength increase with a short exposure time (~1 min) in acetone, presumably due to dilution (combined with hydrogen bonding) of environmental moisture by the acetone. However, only the single layer UV- curable polyacrylate-coated fiber showed weakening and a broadening of the strength distribution after approximately 10 min in acetone. This strength loss was investigated in more detail. The two-point bending and tensile strengths of this fiber as a function of immersion time in acetone are shown in figure 4. All error bars in this work represent a 95% confidence interval for the estimate of the mean strength. The tensile strengths appear to be initially weaker than the bending strengths as a result of the different loading rates – the loading rate in tension corresponds to a nominal strain rate of 40%/min, while the constant faceplate velocity of 1000 μm/s used in bending corresponds to a strain rate of approximately 300%/min at failure. The scatter in the bending strengths increases after ~10 min and the Weibull plots in figure 5 show that this is caused by the presence of a low strength mode which increases in importance with soak time. The high strength mode is still present even after long soak times and its strength is not affected by the soaking.

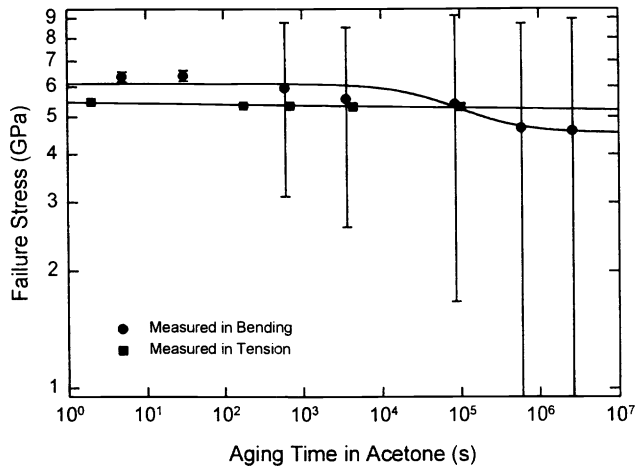

Figure 4. Two-point bending and tensile strength as a function of soak time in acetone.

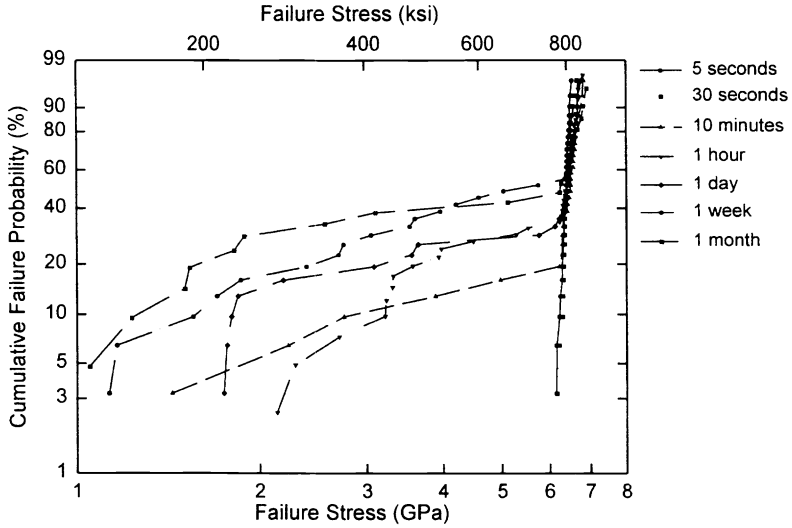

Figure 5. Weibull plot of the bending strengths of fibers soaked for various times in acetone.

A graph of the overall Weibull modulus (assuming the data follow a single strength distribution) as a function of acetone immersion time for the bending measurements is shown in figure 6. Estimates of the Weibull moduli and their 95% confidence intervals were obtained using the unbiased maximum likelihood estimator technique described by Thoman, Antle and Bain.[4] The distribution dramatically broadens after just 10 min of acetone immersion. The bimodal distribution for the bending specimens was examined in more detail, as shown in figure 7, in which the high and low strength modes are analyzed separately. It is also clear that the strong mode is effectively pristine while the weak mode is very weak. The weakest specimens were so weak that they were almost unhandleable. A few fibers broke prior to loading in the two-point bending apparatus with their coatings still intact. All weak mode fracture surfaces were saved for later evaluation with optical and scanning electron microscopy. The Weibull modulus for the weak specimens is approximately 5.

A few observations were made in comparing the effects of the acetone soaking of bending and tensile specimens. The single-layer acrylate coating rapidly lost adhesion, swelled, and stretched with approximately a 7% elongation after longer aging times for 50-mm long bending samples. In contrast, the tensile specimens swelled, but did not elongate since both ends were held securely by the coating that was not immersed in the acetone.

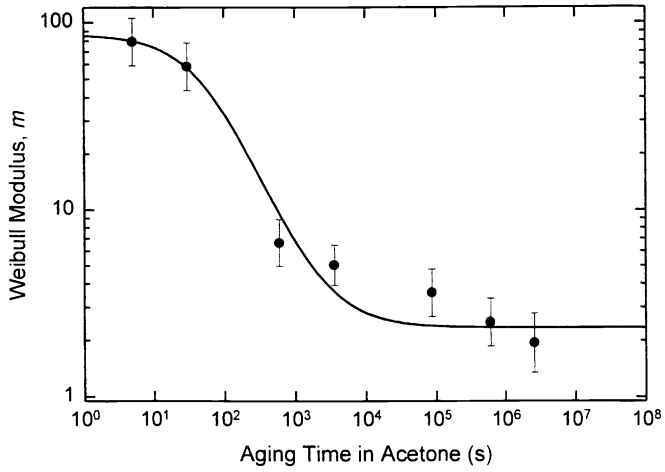

Figure 6. Weibull modulus, *m*, of fiber measured in bending as a function of aging time in acetone.

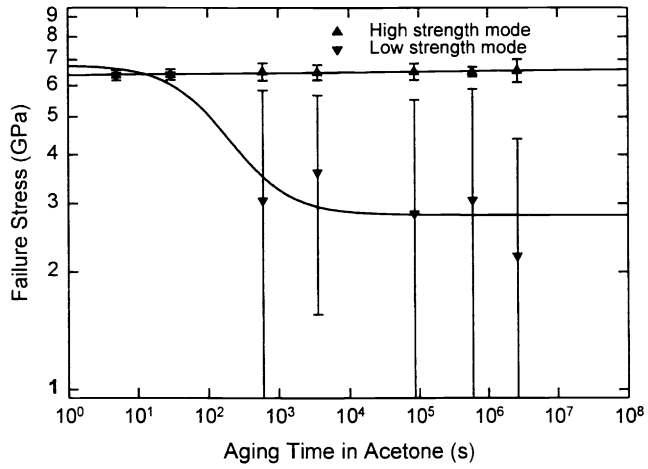

Figure 7. Bimodal residual strength of fiber measured in bending as a function of aging time in acetone.

The loss of adhesion with aging was examined in more detail by measuring the stripping force, as shown in figure 8. The results indicate that the single-layer acrylate-coated fiber appeared to lose adhesion in about 5 minutes when tested immediately after aging. However, after drying for one hour in ambient, the coating strip force was only 50% of that of the unaged samples. These differences are most probably the result of a lower coefficient of friction between the coating and the fiber surface when tested wet and the lower elastic modulus of the coating when saturated with acetone. After drying, the fiber remained elongated and the coating became rigid and thus the mechanical properties of the coating at these soak times is altered. Also, an additional observation after drying for one hour was the presence of dark and light circumferential stripes under the coating. Upon closer optical examination of this phenomenon, it was determined that these stripes were the effect of separation of the coating from the surfaces, *i.e.*, bands of coating in contact and out of contact with the fiber surface (or more correctly, Newton's interference rings caused by the varying separation distance). After a day of drying in ambient, the striped pattern disappeared completely.

Fracture surfaces of the weakest samples broken in bending were examined by optical microscopy (OM) and scanning electron microscopy (SEM). Due to the low depth of focus in OM, it was very difficult to locate any significant fracture surface markings. Each of the fracture surfaces was prepared for SEM by treating in an ultrasonic bath of distilled water to remove any debris and then gold coated to eliminate surface charging effects.

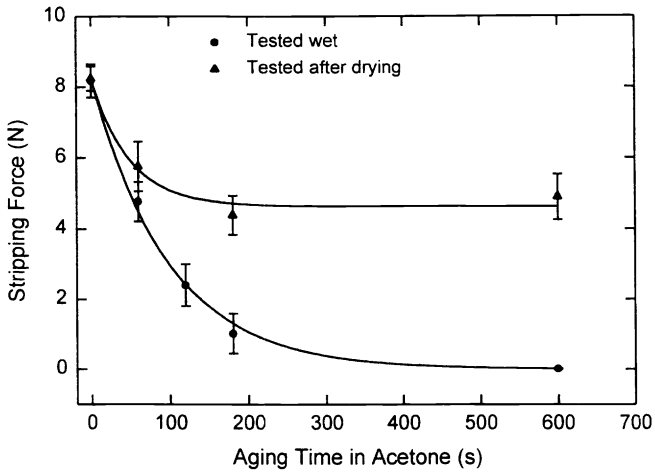

Figure 8. Stripping force of fiber as a function of immersion time acetone.

For many specimens the original fracture origin could not be unequivocally located due to shattering. The origin could be identified for the very weakest failures and the mirror / mist / hackle regions could be observed. However, examination at magnifications of up to 11,000× did not provide any information about the source of failure. In particular, no abrasion damage was visible on the fiber surface, though this might be limited by the resolution of the SEM used.

The source of the weak defects caused by acetone soaking is not immediately obvious. Acetone is not expected to cause weakening by fatigue, and, in any event, such fatigue weakening would not produce the very low strengths observed and would not result in a bimodal strength distribution. The strength distributions of figure 5 are typical of specimens that have been exposed to handling where some have suffered abrasion damage. The coating is several orders of magnitude softer than silica and would not itself cause abrasion damage. The most plausible explanation for the behavior is that small hard particles in the coating are abrading the fiber surface after adhesion has been lost due to the acetone soak. Adhesion loss allows the coating to slide over the fiber surface during swelling and bending. In the tensile tests, no relative motion between the glass and coating occurs explaining why strength degradation did not occur for those tests. The importance of adhesion loss is suggested by the observation that the time scale of the weakening corresponds to the time scale of the reduction in strip force.

The liquid prepolymer coating materials generally used for coating high strength fibers are filtered through ~3 μm filters with current trends reaching submicron filtration sizes.[5] Huff and coworkers[5] incorporated alumina particles of various sizes into fiber coatings and measured the residual strength of the fiber after it was passed through a proof test machine. The proof tester presses the fiber so that any particles next to the glass surface indent the surface. Huff *et al.* concluded that particles below 3 μm in size do not lead to failures during proof testing to 1.4 GPa. However, it is well known that in sliding contact the tensile stresses induced in the surface can be very much larger than the stresses produced by normal contact[6] leading to enhanced abrasion damage.[7] Therefore, we can anticipate that particles below 3 μm in the coating could cause substantial weakening from sliding contact damage incurred during the elongation of the fiber coating or during bending of the fiber.

The damage produced by the particles will depend on the stiffness of the polymer in which they are embedded. Telecommunications fiber typically has a double coating with an inner layer whose elastic modulus is very much lower than that of the single-coated fiber used in this study. Contact damage will be reduced for the compliant coating since it will not be able to support sufficient load to allow the particle to damage the glass. This would explain the observation that weakening in acetone was not observed for dual coated fibers.

4. CONCLUSIONS

A single layer UV-curable polyacrylate-coated, telecommunications grade, 125-μm diameter fused silica fiber was discovered to exhibit substantial strength reduction in two-point bending after immersion in acetone. The cause of the strength reduction is proposed to arise from particles in the coating below 3 μm in size which could cause surface flaws by sliding contact damage incurred during relative motion between the coating and the glass. This sliding could occur either while flexing the fiber in preparation for a bending strength measurement or due to coating elongation. While it is not clear

whether flexing or elongation causes the damage, both are consistent with the observation that degradation is only observed for bending strength measurements and not for tensile strength measurements.

This strength degradation is of clear practical significance since optical fibers can be exposed to organic solvents under a variety of circumstances. The weakening that has been observed to occur has not been reported before and was not expected. Our results are preliminary in nature and more work is required to properly understand this phenomenon. In particular, the proposed degradation mechanism suggests that any chemical species that is highly soluble in the coating (and hence causes swelling) and causes adhesion loss could potentially cause damage. While not quantified, we did observe similar strength loss for the single-coated fiber when exposed to methylene chloride. Future work will include screening other chemicals for this behavior. While modern coating configurations, with a low-modulus primary coating, are not expected to be susceptible to this phenomenon, standard testing procedures might need to be developed to characterize the coating performance in this regard.

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