

# Acid Stripping of Fused Silica Optical Fibers Without Strength Degradation

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**Abstract**—Glass optical fibers are almost always coated with a polymer immediately after drawing to protect them from subsequent handling damage. When studying the strength and fatigue properties of the fibers, it is useful to be able to remove this coating in order to directly observe the fatigue properties of the glass in immediate contact with the environment. Fused silica optical fibers are frequently stripped by immersion in hot ( $\sim 200^\circ\text{C}$ ) concentrated sulfuric acid. Two recent papers have claimed that hot acid stripping significantly degrades the strength and increases the width of the strength distribution. However, there is a large literature that implies that, at least for most coating systems, acid stripping does not degrade the strength provided sufficient care is taken to protect the bare fiber surface during stripping and subsequent testing. This paper *explicitly* proves this result, showing that careful complete stripping has little or no effect on the strength of fiber tested in both tension and bending. It is also shown that the immersion time in the hot acid has no noticeable effect on the strength. Experimental protocols are described that minimize the likelihood of accidental damage to the fiber during stripping.

## I. INTRODUCTION

**O**PTICAL fibers are almost always coated with a polymer layer during the draw process. A liquid prepolymer is applied soon after the fiber emerges from the draw furnace and is typically cured using UV radiation. The polymer is therefore applied before the fiber makes mechanical contact with any solid object so that the condition of the fiber surface is protected from mechanical damage. Typical fibers for communications applications are constructed of 125  $\mu\text{m}$  diameter fused silica coated with an epoxy-acrylate or similar polymer layer of outer diameter  $\sim 250 \mu\text{m}$ . This coating provides substantial mechanical protection and the fiber can experience severe abuse during subsequent manufacturing, cabling, connectorizing, installation, and repair operations, without strength degradation. The coating therefore protects the surface of the fiber and maintains its as-drawn condition. This is particularly important in the case of fused silica fibers that have “pristine” flaw-free surfaces [1]. Without the coating,

the fiber strength is rapidly degraded with subsequent handling and can disintegrate during normal handling.

The polymer coating may need to be removed from the fiber for a variety of reasons. Connections to an optical fiber usually require precise positioning of the fiber end in order to avoid optical loss. Because the polymer has poor dimensional stability due to its low hardness and stiffness, the coating is usually removed before, for example, inserting the fiber into the connector assembly. Fusion splicing the fibers also requires prior removal of the coating. Of more relevance to this work, it is often desirable to remove the coating for the purpose of research on the strength and fatigue behavior of the fiber. There are three important classes of experiment that require removal of the coating.

- 1) Glass fibers, and fused silica optical fibers in particular, suffer stress corrosion cracking (fatigue) and strength degradation in the presence of environmental moisture. Understanding of this phenomenon is a key component in assuring the reliability of fiber optic based systems and is an area of active research. Polymer coatings are not hermetic, do not exclude moisture from the fiber surface and so do not prevent fatigue. However, they do act as a diffusion barrier; for example, if a typical coated silica fiber is removed from storage in ambient atmosphere and placed in liquid water it takes on the order of a day for the strength to equilibrate at room temperature [2], [3] and minutes at  $90^\circ$  [4]. In addition, the coating can influence the fatigue by changing the local environment at the fiber surface [4] or may profoundly influence the rate of strength degradation under zero applied stress (aging) [5]. Also, the coating can act as a carrier for colloidal particles that can slow the fatigue process [6]–[9]. In order to understand the strength and fatigue behavior of coated fiber, it is important to understand the behavior of bare fiber so that the direct interaction of the glass with the environment can be explored. It is therefore necessary to have a technique for removing the coating prior to testing without degrading the fiber strength.
- 2) It is often necessary to remove the coating when measuring the inert or initial strength of the fiber which is an important parameter needed for lifetime prediction models. It is extremely difficult to remove all moisture from the test environment and therefore inert strength is most conveniently measured at liquid nitrogen temperatures at which the water activity is negligible. However, coatings are normally brittle at low temperatures and can

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cause premature failure of the fiber thus necessitating their removal before testing.

- 3) It is now well established that the development of roughness on the surface of the fiber due to etching by environmental water can dominate the strength degradation under some circumstances (particularly in liquid water environments at high temperature or at long duration). Investigation of this phenomenon involves aging the fiber under zero stress, followed by removal of the coating so that the surface roughness can be examined using such high resolution techniques as scanning tunneling microscopy [10] or atomic force microscopy [5].

#### A. Stripping Techniques

A requirement of any stripping technique is that it should remove the coating without degrading the fiber strength and without leaving surface residues and, if possible, it should not affect the surface chemistry of the glass. The coating is typically removed in the field for splicing and connectorizing using mechanical strippers, perhaps after softening with a suitable solvent. Residues, often left on the fiber surface, must be removed by solvents and rubbing which leads to direct mechanical damage of the fiber surface either during stripping or subsequent cleaning. An alternative technique is to swell the coating using a solvent such as methylene chloride, so that the coating loses its mechanical integrity and can easily be removed without touching the fiber. However, this technique can also leave residues that are difficult to remove without damaging the fiber. The final technique, which is the subject of this paper, involves stripping the fiber by immersing it in hot concentrated sulfuric acid at a temperature of usually between 180 and 220 °C (a technique first described by Simpkins and Krause [11]).

#### B. Previous Results

Stripping the polymer coating by hot acid is a technique that is widely used for research purposes when studying fused silica fiber but is also used commercially when splices of consistent high strength are required. Clearly, such an aggressive environment might change the surface chemistry of the glass, particularly if there are relatively reactive species on the surface. This is of concern for fibers aged for prolonged periods that may have a hydrated surface layer of silica gel that would be removed by the acid. Rondinella and Matthewson [12] found that surface roughening due to prolonged aging might not be apparent using atomic force microscopy if the fiber is stripped mechanically after swelling with methylene chloride; there then being no correlation between surface roughness and residual strength. In contrast, roughness measurements made after hot acid stripping (or after dipping mechanically stripped fiber in hot acid) do closely correlate with the residual strength. They found that the strength of fiber aged and then stripped (or indeed fiber aged bare and then subjected to stripping procedures) was independent of the stripping technique, be it mechanical or with methylene chloride or hot acid. This work provides evidence that first, the

surface layer formed during aging does not contribute to the mechanical strength and second, hot acid does not adversely affect the strength of aged fiber.

However, the concerns about changes in surface chemistry are beyond the scope of this paper and are a subject of ongoing research. What is considered here is how acid stripping affects the strength of pristine fiber and whether it necessarily introduces flaws into the surface or extends existing ones. This is of concern because two recent publications assert that acid stripping does substantially degrade fused silica fiber strength while increasing the scatter [13], [14]. Choi [13] stripped a 10 mm length of fiber by immersion in room temperature concentrated sulfuric acid for 50 min and found that afterwards the tensile strength had fallen by a factor of approximately two and the Weibull modulus had fallen from  $\sim 50$  to 3 or less, signifying a substantial increase in the strength scatter. He also found similar behavior for fiber stripped by immersing in 70 °C concentrated acid for 4 min. The weakening was attributed to “changes in the flaw morphology.” These results were found in both a fatiguing environment (25 °C distilled water) and in so-called “inert” conditions (25 °C dry nitrogen gas). However, it should be noted that it is well known that dry nitrogen does not approximate inert conditions for this high strength material—the strength of pristine fiber measured in this environment,  $\sim 8$  GPa, is substantially less than the accepted inert strength of  $\sim 12$  GPa measured both in liquid nitrogen [15] and for fiber drawn and broken under ultrahigh vacuum [16]. Fibers weakened by Vickers indentation have also been found to be considerably stronger in liquid nitrogen [17] than in dry nitrogen [18], [19] so that dry nitrogen is not a suitable inert atmosphere even for weak fiber.

Baikova *et al.* [14] report similar results to those of Choi [13]. They measured the strength of fibers stripped either by 200 °C sulfuric acid or by swelling in room temperature acetone. Most of the measurements were made using a miniaturized three-point bend apparatus [20] although tensile measurements showed similar trends. The stripped fiber exhibited a broad strength distribution with the strongest fibers having strengths similar to coated fiber and with the weakest having a strength some 3.5 times lower. Significantly, they observed that these results were similar to those obtained on fiber that was drawn without a coating and tested without touching, also using the three-point bend apparatus [21]. Baikova *et al.* [14] suggested that this effect is due to atmospheric moisture locally attacking regions under structural stress on the exposed surface of uncoated or stripped fiber to produce microcracks. The implication of this is that the coating therefore protects the glass surface from such attack. However, polymer coatings are not hermetic and coated fiber exhibits similar fatigue to bare fiber.

While these two papers [13], [14] do conclusively prove that the fiber strength *can* be degraded by stripping and testing (despite much effort to avoid it), at least for the fiber/coating systems they used, they do not prove that it *must* be degraded. In fact, there are several papers concerned with bare fiber strength that do not report strength degradation.

In the earliest work on fused silica fibers, Proctor Whitney and Johnson [22] measured the strength of bare hand-drawn

fibers in tension and under ambient conditions. Apart from one weak fiber with a strength of  $\sim 2$  GPa, the mean strength of 59 specimens was 6 GPa with a Weibull modulus of  $\sim 20$ . Much of the scatter in these results was due to uncertainty in the fiber radii. They found that such high strengths could only be achieved by very careful handling. Krause [23] compared the tensile static fatigue behavior of coated and hot acid stripped fibers in  $90^\circ\text{C}$  water and found indistinguishable behavior before the fatigue "transition" or "knee" (time to failure  $\lesssim 10^4$  s) and did not report unusual scatter in the stripped fiber data. Under similar test conditions Krause [24] found that for a particular applied stress and polymer coating material, all fibers failed in the coated section rather than in the central stripped section indicating that the bare fiber was stronger than the coated fiber in this case. Therefore there could have been no significant strength degradation due to stripping. Krause and Kurkjian [25] found that the strength (both mean and dispersion) of fusion splices was essentially the same as the unspliced fiber; the spliced ends had been stripped by 30 s immersion in hot acid [26]. This prior work was conducted in tension with gage lengths on the order of 10 mm. Matthewson et al. reported a two-point bending technique that could be used to measure bare fiber strength (dynamic fatigue) [4] and static fatigue [27]. Two-point bending experiments on hot acid stripped fiber have not shown strength degradation or excessive increase in scatter [3], [4], [6], [8], [10], [28]–[31]. Rondinella and Matthewson [3] examined the effect of loading mode in two-point bending (constant faceplate velocity, constant strain rate and constant stress rate) and found that the fatigue parameters did not depend on the loading mode, but additionally, they found only small differences between stripped and coated fiber. This implies that in short term tests (typically failure in 1 to  $10^4$  s) under mild conditions (pH 7 water at room temperature) the presence or absence of the coating has little effect on the fatigue as well as the strength, contrary to statements of Choi [13].

It is interesting to note that in both the previous studies showing degradation the strongest stripped fibers had similar strengths to the coated fibers; Choi's data showed a noticeable high strength mode with a high Weibull modulus for 10–20% of the population. Therefore, some specimens survived the stripping and subsequent testing without degradation.

The thesis that typical polymer coated fiber can be stripped by immersion in hot acid without strength degradation is effectively proved in the literature reviewed above. However, no explicit discussion of the topic has appeared in the literature. This omission is the stimulus for this paper, together with the desire to refute contradicting claims and to establish the validity of carefully performed experiments on bare fiber. This paper will explicitly establish that hot acid stripping can be performed without strength degradation, whereas the prior work has only shown this implicitly.

## II. EXPERIMENTAL

Three fibers have been used in this study. The first, designated fiber A, was drawn from a 16 mm diameter rod

of Suprasil F<sup>1</sup> fused silica. The fiber was drawn down to a  $125.1 \pm 0.1$   $\mu\text{m}$  diameter and was coated in-line with a double layer of UV-curable epoxy acrylate to an outside diameter of  $258.0 \pm 0.6$   $\mu\text{m}$ . The primary "soft" coating had a lower elastic modulus than the "hard" outer secondary layer. The coating configuration and material are typical of those used for commercial fiber. The fiber was not proof tested but more than 50 0.5 m gage length tensile specimens showed no low strengths; extrinsic defects are therefore too rare to perturb the results. The second specimen, designated fiber B, was similar to the first except that it had only a single epoxy-acrylate coating and had been proof tested to 2% strain. The third specimen, designated fiber C, was provided by V. P. Pukh (A. F. Ioffe Physic-Technical Institute, Russian Academy of Sciences, St. Petersburg, Russia) and was the same as the fiber used in the study by Baikova et al. [14]. The dimensions of this fiber are similar to the other two specimens. All three fibers were drawn at different establishments.

The stripped fibers were prepared by immersing half of a loop of fiber (typical radius 15 mm) in a dish of concentrated sulfuric acid ( $>95$  wt%  $\text{H}_2\text{SO}_4$ ) at  $190 \pm 10^\circ\text{C}$  for approximately 10 s. Most coatings effervesce when placed in the acid and give a faint audible "pop" when dissolved. A useful "rule of thumb" is to immerse the fiber for at least two to three times longer than the time it takes to produce the "pop" giving a total immersion time of  $\sim 20$  s. The temperature of the acid was tailored to the particular coating used. If the acid is too cool the coating is incompletely stripped. On the other hand,  $200^\circ\text{C}$  can be too hot for some coatings which can leave a carbonaceous layer on the fiber [32]; this is avoided by maintaining the acid at a few tens of kelvins cooler. Occasional specimens were examined using an optical microscope to ensure that their surface was clean and that the stripping had been successful. After stripping, the fiber was rinsed for approximately 5 s each in deionized water and then acetone. Clean reagents were used throughout (it is advisable to replace all reagents at least daily). In particular, if a fiber failed in one of the reagents due to accidental contact with the side of the container, fresh reagent was used to avoid contact between subsequent specimens and debris. It is important to replace the acid periodically since it absorbs atmospheric moisture while cool; though not reported, in earlier work [4], failure of fibers during stripping under high stress was much less likely in fresh acid suggesting fatigue in wet acid. However, for these experiments fresh acid was used for each test condition and so it was never exposed to atmosphere for more than a few hours. After stripping, the fibers were tested immediately. Care was taken throughout to avoid contact between the bare fiber surface and any solid object; this included avoiding placing the fibers in draughts that might cause collisions with airborne particles. However, bare fibers can be retained without strength loss for several days when stored vertically in a dust free environment.

Coated and stripped specimens were tested both in uniaxial tension and two-point bending. The tensile specimens were gripped by wrapping the fiber ends around 50 mm diameter rubber coated capstans, with a 300 mm gage length between

<sup>1</sup>Heraeus-Amersil Inc., Duluth, GA.

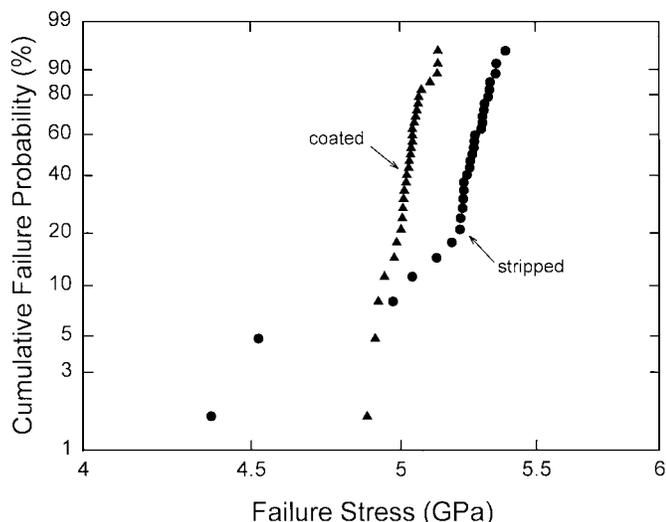


Fig. 1. Weibull plot of the two-point bending strengths of fiber A coated ( $\blacktriangle$ ) and stripped ( $\bullet$ ). Estimates of statistical parameters are given in Table I.

the capstans. Except for the occasional weak specimen (to be described) the fibers shattered at failure, and it was not possible to determine the position of the fracture origin from the remnants. Both coated and stripped fibers tested in two-point bending were held between polished faceplates (see schematic in [3]). Polished rather than grooved faceplates were used in order to avoid damaging the bare fibers [33].

All results in this work were obtained in ambient laboratory atmosphere that did not have controlled temperature and humidity. Fibers for each individual test condition were therefore tested over as short a time period as possible in order to avoid large changes in the environment during the testing. In addition, coated and bare fiber measurements were interleaved (i.e., one coated fiber was broken, then one bare, one coated, etc.) in order to remove any systematic differences between the coated and stripped strength due to residual fluctuations in the environment. The temperature and humidity were noted when each specimen broke and their standard deviations were typically  $\pm 0.2^\circ\text{C}$  and  $\pm 1\%$  RH which has negligible influence on the results. Thirty specimens were used for each test condition unless otherwise stated.

### III. RESULTS

#### A. Two-Point Bending Strength Results

Figs. 1 and 2 show Weibull probability plots of the two-point bending strengths of both coated and stripped specimens of fiber A (Fig. 1) and fiber B (Fig. 2). Tables I and II give the results of statistical analyzes of the data; the Weibull modulus,  $m$ , was calculated using an unbiased maximum likelihood estimator technique [34] and all errors represent a 95% confidence interval. These tables also give the test temperature and humidity.

For both fibers, the coated specimens exhibit unimodal behavior while the stripped specimens show bimodal behavior with a “low” strength mode containing five and two specimens. The “low” strength modes are quite narrow and the

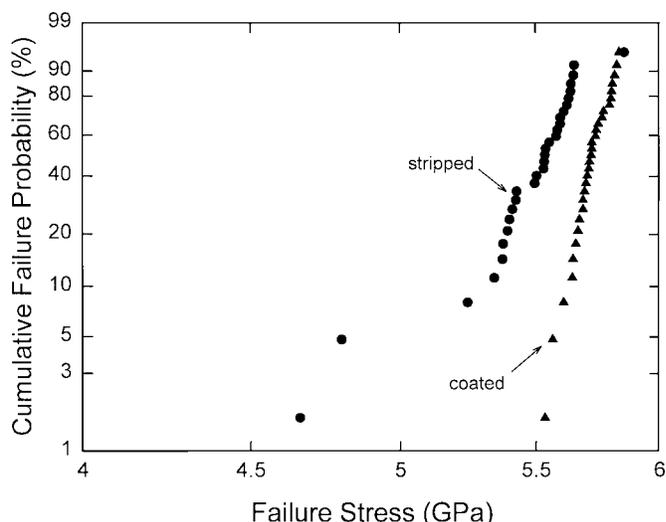


Fig. 2. Weibull plot of the two-point bending strengths of fiber B coated ( $\blacktriangle$ ) and stripped ( $\bullet$ ). Estimates of statistical parameters are given in Table II.

TABLE I

SUMMARY OF RESULTS OBTAINED FOR STRIPPED AND COATED FIBER A, TESTED IN TENSION AND TWO-POINT BENDING. THE NUMBER OF SPECIMENS ANALYZED IS GIVEN BY  $N$  WHERE IT IS LESS THAN 30, THE  $N$  STRONGEST SPECIMENS ARE ANALYZED. RANGES OF VALUES REPRESENT 95% CONFIDENCE INTERVAL

Technique	Test Condition	$N$	$m$	Mean Strength GPa	Stress rate MPa.s <sup>-1</sup>	Temp. °C	Humid. %
2-Point Bending	Coated	30	88 [65–117]	5.030 $\pm$ 0.022	69	22.0	35
	Stripped	30	48 [36–64]	5.199 $\pm$ 0.082			
	Stripped subset	28	79 [58–107]	5.253 $\pm$ 0.034			
Tension	Coated	30	143 [106–190]	4.943 $\pm$ 0.014	62 $\pm$ 4	22.4	35
	Stripped (25 mm)	30	109 [81–145]	4.923 $\pm$ 0.018			

TABLE II

SUMMARY OF RESULTS OBTAINED FOR A FIBER B. RANGES OF VALUES REPRESENT 95% CONFIDENCE INTERVAL

Technique	Test Condition	$N$	$m$	Mean Strength GPa	Stress rate MPa.s <sup>-1</sup>	Temp. °C	Humid. %
2-Point Bending	Coated	30	87 [65–116]	5.716 $\pm$ 0.028	69	23.1	43
	Stripped	30	33 [10–44]	5.464 $\pm$ 0.086			
	Stripped subset	28	41 [30–55]	5.527 $\pm$ 0.048			
Tension	Coated	30	76 [57–101]	5.752 $\pm$ 0.030	62 $\pm$ 4	24.4	8
	Stripped	30	28 [21–37]	5.314 $\pm$ 0.158			
	Stripped subset	28	47 [34–63]	5.406 $\pm$ 0.048			

weakest specimens are 10–20% weaker than the strongest. This compares sharply with the data in [13] and [14] where the weakest fibers are several hundred percent weaker than the strongest. The weak mode in bending is due to occasional slight misalignment of the fiber in the polished faceplates. If the fiber is not quite perpendicular to the faceplates when it is inserted, the misalignment is exaggerated as the faceplates come together and should the fiber slip, it is scratched and fails. Slippage can be observed for coated fibers tested with polished faceplates since they twist out of the apparatus without breaking (but is less likely because of higher friction); the slippage can not be detected for bare fiber because of immediate failure. While such misalignment causes an increase in the scatter in the results, it is trivial compared to that observed by Choi [13] and Baikova *et al.* [14] and it does not usually affect the mean strength; Tables I and II show that ignoring the two weakest specimens for each fiber substantially

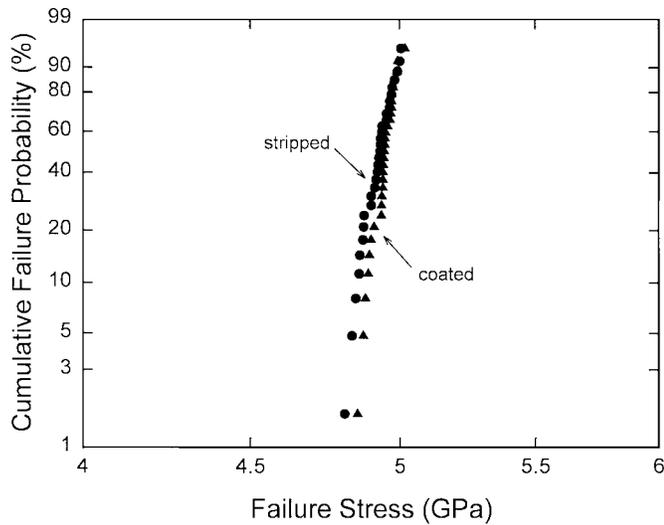


Fig. 3. Weibull plot of the tensile strengths of fiber A coated ( $\blacktriangle$ ) and stripped ( $\bullet$ ). Estimates of statistical parameters are given in Table I.

increases the Weibull modulus until it is similar to that of the coated fiber, but has no significant effect on the mean strength.

The coated and stripped strengths in Figs. 1 and 2 differ somewhat. This is to be expected since, while the coating does not exclude moisture, it can alter the local environment at the fiber surface by changing the water activity (effective humidity) or pH. The data for fiber B (Fig. 2) are typical in that the stripped strength is a little lower than the coated strength. In contrast, fiber A is somewhat stronger when bare so that this coating presumably increases the water activity or pH locally at the fiber surface. Such behavior, while unusual, has been observed before by Krause [24]. In both cases, stripping produces  $\sim 5\%$  change in strength and, once a couple of outliers are excluded, also produces little broadening of the strength distribution. Once again, it is noted that these changes are minor compared with the several-fold weakening and broadening observed by Choi [13] and Baikova *et al.* [14]. There is no evidence that the slight weakening observed in Fig. 2 is anything other than simply due to the environment change that accompanies stripping.

Ideally, the effect of differences in the environment between stripped and coated fiber would be eliminated if the inert strength, in the absence of fatigue, were measured. However, liquid nitrogen at 77 K is the only truly inert environment that is readily useable. Unfortunately, the coating embrittles at low temperature so the technique is unsuitable for coated specimens.

### B. Tensile Strength Results

Figs. 3 and 4 show the results of tensile testing of fibers A and B, respectively. The tensile results for Fiber B (Fig. 4) show similar trends to the two-point bending results (Fig. 2); apart from one weak specimen, presumably caused by mishandling, the stripped fibers are  $\sim 5\%$  weaker than the coated specimens and the width of the distributions are similar. Again, there is no evidence that stripping does anything other than slightly change the environment at the fiber surface.

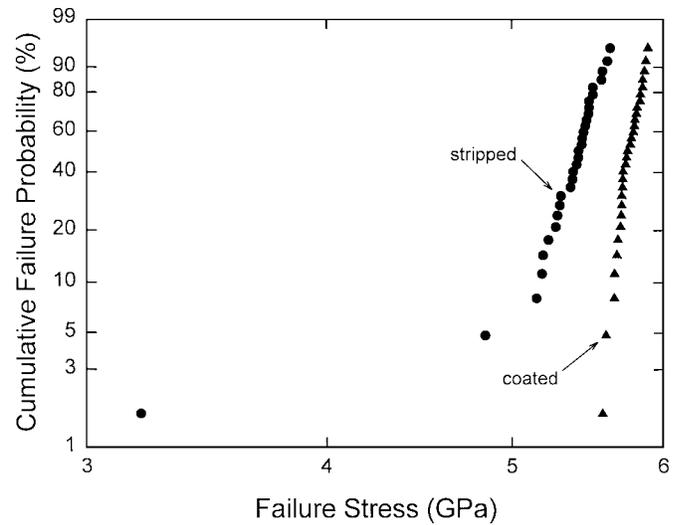


Fig. 4. Weibull plot of the tensile strengths of fiber B coated ( $\blacktriangle$ ) and stripped ( $\bullet$ ). Estimates of statistical parameters are given in Table II.

TABLE III  
SUMMARY OF RESULTS OBTAINED FOR A FIBER C. RANGES  
OF VALUES REPRESENT A 95% CONFIDENCE INTERVAL

Technique	Test Condition	$N$	$m$	Mean Strength GPa	Stress rate MPa.s <sup>-1</sup>	Temp. °C	Humid. %
2-Point	Coated	20	32 [22–46]	$5.691 \pm 0.074$	69	22.6	54
Bending	Stripped	20	49 [34–70]	$5.324 \pm 0.064$			
Tension	Stripped	6	65 [31–136]	$5.026 \pm 0.046$	$\sim 30$	22.6	54

The tensile strength distributions for coated and stripped Fiber A (Fig. 3) are virtually identical. The two-point bending results show that this fiber is slightly stronger when stripped. Since bare fiber can not be gripped, the “stripped” tensile specimens are actually coated with only a 25 mm stripped central section in the 300 mm gauge length. Therefore, given the two-point bending results, we expected the stripped specimens to break in the coated sections. This is not observable since most of the gauge length shatters at failure and the fracture origin can not be identified. As a result, both stripped and coated specimens will have the same apparent strength—the coated strength. Therefore, these results are entirely consistent with the two-point bending results. Again, there is no evidence that stripping produces any strength degradation.

### C. Results for the Fiber of Baikova *et al.*

The results for fiber C, the same fiber used by Baikova *et al.* [14], are summarized in Table III and Fig. 5. Because of the limited quantity of fiber available to us, only 20 coated and bare specimens were tested in two-point bending. The stripped fiber exhibited no weak specimens and had a somewhat narrower strength distribution than the coated specimens. As is commonly observed, the coated fiber is a little stronger, probably due to it reducing the activity of water at the fiber surface.

There was insufficient fiber to perform tensile testing using capstan grips which require  $\sim 1.5$  m per specimen. Instead, 75 mm length specimens were gripped over a 25 mm length at each end using rubber coated pneumatic grips. There was only

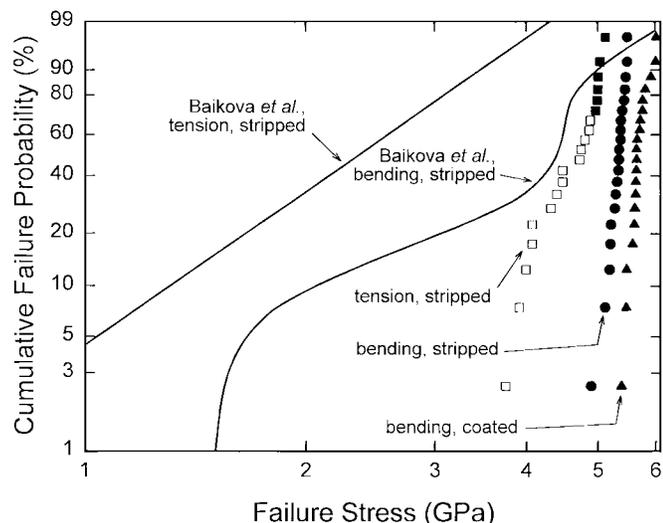


Fig. 5. Weibull plot of the bending strengths of fiber C coated ( $\blacktriangle$ ) and stripped ( $\bullet$ ) and the tensile strengths of failed specimens ( $\blacksquare$ ) and censored specimens ( $\square$ , maximum stress before slippage at the grips). Estimates of statistical parameters are given in Table III.

enough fiber to examine the stripped strength. The central  $\sim 25$  mm test section was acid stripped over a 10–15 mm length. The as-received coating surface slipped in the pneumatic grips but all of 5 specimens tested survived stresses ranging from 2.2 to 3.5 GPa. Higher stresses could be applied by briefly dipping the coating in acetone. Of 20 specimens treated in this way, 14 survived stresses ranging from 3.8 to 4.9 GPa while six were successfully loaded to failure at stresses exceeding 4.9 GPa. The statistics for these six failure are given in Table III. The loading rate at failure was difficult to characterize and was very variable due to slippage, but was  $\sim 30 \text{ MPa}\cdot\text{s}^{-1}$ . These tensile results are not ideal because of slippage and because of poor alignment of the load train brought about by the small test length. However, the results, even when failure did not occur, do specify a useful lower bound on strength.

Fig. 5 shows a Weibull plot of both bending and tensile results. The tensile data include both the failed specimens (solid squares) and the maximum stress achieved by those that slipped (open squares). This plot is therefore censored—the true strength distribution should lie to the right of these data. Also shown in Fig. 5 are sketched lines indicating the results obtained by Baikova *et al.* [14]. All our data lie well to the right of their tensile data; our results for the same fiber do not support their observation of severe degradation upon acid stripping.

#### D. Effect of Radius Fluctuations

Table I shows that the strength in two-point bending is somewhat higher than that in tension (fiber B, Table II, shows the converse behavior because of the low humidity for the tensile experiments). This is partly due to the higher stress rate used in bending but also partly due to the difference in effective tested length which is only a few tens of microns in bending [33]. Although there is some overlap of the 95% confidence intervals, the Weibull modulus for the coated fiber in tension is substantially higher than for the coated fiber in

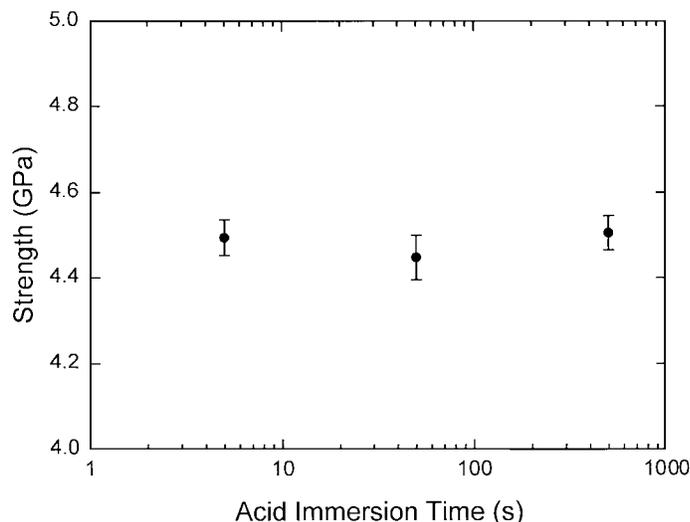


Fig. 6. Strength measured in two-point bending of fiber A, stripped in hot acid, as a function of acid immersion time.

bending ( $\sim 140$  c.f.  $\sim 90$ ). Kurkjian and Paek [1] showed that the scatter in the strength of pristine fiber is comparable to the scatter in the fiber radius. This means that the intrinsic strength is essentially single valued and that the scatter in the apparent strength arises from radius fluctuations. The strength scatter is different for different testing techniques due to their different radius dependencies [27]. A more detailed analysis of radius fluctuations shows that the scatter in two-point bending strength can be either higher or lower than that in tension, depending on whether the length scale (along the fiber length) of the radius fluctuations is shorter or longer than the effective test length in bending. No unique correlation between the Weibull moduli measured in tension and bending is therefore expected.

#### E. Effect of Acid Immersion Time

The strength of stripped fiber has been determined for fiber A as a function of immersion time in the hot acid. Fig. 6 shows the results for 5, 50, and 500 s immersion times. The strengths were determined using two-point bending with the fiber immersed in 23 °C pH 7 buffer solution and ten specimens per point were used. The strength does not depend on the immersion time over a broad range of times and prolonged stripping does not degrade the strength. Immersion times can therefore be made long enough to ensure complete removal of the coating material without having to be unduly concerned about fatigue occurring during the stripping process.

## IV. DISCUSSION

Stripping the polymer coating from a fused silica fiber by immersion in hot concentrated sulfuric acid does not degrade the fiber strength. This has been shown implicitly in previous work as well as explicitly here. This is the case for both the strength in two-point bending (with an effective tested length of a few tens of microns) and in tension with stripped lengths of a few tens of mm. Considerable care and cleanliness of reagents is required to avoid the occasional weak specimen,

but such specimens become obvious in a Weibull plot of the results and so can be safely identified and ignored. Provided the strength distribution has a well defined high strength mode with a high Weibull modulus (greater than 40 or so), a large number of weak specimens will usually indicate poor handling or strength testing technique rather than degradation by the stripping process itself.

We have never observed strength degradation by acid stripping either in this or in previous work. However, this does not preclude the possibility that degradation can occur for particular coating/fiber systems by some as-yet unidentified mechanism. For example, for some specific coating formulations, we have observed substantial strength loss after soaking in room temperature acetone for a few hours, though the same fiber does strip in acid without degradation. Choi [13] was able to strip his fiber by soaking in 25 °C acid for 50 min or 70 °C acid for 4 min. We have never encountered a coating that can be stripped so quickly under these conditions so it is possible that his coating, while untypical, did produce the degradation by some unknown chemical means.

Baikova *et al.* [14] use both tension and three-point bending to obtain their results. It should be noted that their bending results always show broad and low strength distributions for bare fiber (whether stripped or never coated) and narrow distributions for coated fiber. They measured the strength of 125  $\mu\text{m}$  diameter fiber using a miniaturized three-point bend apparatus with supports separated by 1100  $\mu\text{m}$ ; such dimensions require large forces and large deflections of up to 45° [20] to break high strength fibers. It is therefore possible that the fiber is damaged by contact with the supports (described as “knife edges”) and that this damaged region is then pushed between the supports into a region of tension causing premature failure. The results of Baikova *et al.* [14] would be explained if this effect occurred for bare fiber but not for coated fiber which is protected from mechanical damage at the supports by the coating. It is therefore necessary to establish the usefulness of the miniaturized three-point bending technique for high strength bare fiber. However, these arguments do not completely explain their results since they also observed low tensile strengths for stripped fiber. Their results in tension and bending on stripped fiber show broad distributions ( $m \sim 2$ ) with the tensile data being some 50% weaker. This represents a discrepancy since the tensile data would be expected to be substantially weaker. Given a 50 mm tensile test length (the length of the stripped section), and assuming a 1 mm effective bending test length (though this would be expected to be much smaller [35]), a simple Weibull analysis would predict that the tensile strength would be seven times smaller than the bending strength for  $m = 2$ . Their data therefore lack internal consistency. (Note that the arguments made above about radius fluctuations are not relevant since we are concerned here with degraded, not pristine fiber.)

Baikova *et al.* [14] report results from an earlier study [21] for fiber that had never been coated or exposed to the stripping environment. These specimens show similar degradation to the stripped specimens. However, the earlier work of Proctor *et al.* [22] clearly shows that uncoated as-drawn fibers can exhibit a narrow distribution of pristine strength. Therefore,

the bare specimens in [21] must have been unintentionally damaged during manufacture, subsequent handling or testing. This suggests that the experimental protocols of Baikova *et al.* do not preserve the pristine strength of bare fiber. This is supported by the results obtained here for the same fiber, which show no degradation in either bending or tension.

In all cases, the data of Choi [13] and Baikova *et al.* [14] exhibit narrow high strength distributions for coated fiber and broad low strength distributions for bare fiber, whether stripped or never coated. While their data might indeed be valid for some particular fibers, they do not establish that their techniques are capable of measuring pristine strengths in bare fiber. We strongly suggest that stripping, handling and testing techniques be validated by testing fiber that is known to not degrade during acid stripping, before any claims are made about post-stripping strength.

## V. CONCLUSION

It is demonstrated, both explicitly here and implicitly in earlier literature, that stripping the acrylate polymer coating from a fused silica optical fiber by briefly dipping in  $\sim 200$  °C concentrated sulfuric acid does not degrade the strength of the fiber, at least for the large number of coating formulations studied so far. This contradicts two earlier studies of this topic which appeared to show substantial strength degradation and distribution broadening upon acid stripping [13], [14]. However, in the earlier work it was not demonstrated that the experimental techniques used were capable of preserving pristine strength in bare fiber. While the earlier results may have been due to unusual chemical effects, they most probably result from unintentional damage being introduced into the fibers.

Stripping the polymer buffer from an optical fiber is a technologically important process since it is needed, for example, when connectorizing or splicing fiber. This paper clearly demonstrates that hot acid stripping is one technique (though perhaps not the practically most convenient) for removing the coating that does not degrade the fiber, provided the process is conducted with sufficient care.

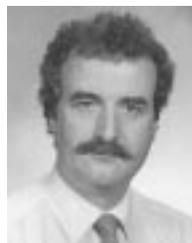
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