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Optical Fibers with Polyimide Coatings for Medical Applications

Andrei A. Stolov, Brian E. Slyman, Debra A. Simoff,
Adam S. Hokansson, R. Steve Allen
and John P. Earnhardt

OFS, Specialty Photonics Division
55 Darling Drive, Avon, CT 06001
www.SpecialtyPhotonics.com

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Abstract

Key properties of polyimide-coated optical fibers, unaged and exposed to various harsh environments, were investigated. The main intent was to model extreme conditions that can be encountered in medical applications of the fibers. A fiber designed by OFS showed good strength and was able to withstand exposure to extreme heat and humidity, multiple autoclave cycles, extended water soak and immersion in organic solvents. Similar fibers offered by other suppliers displayed shortcomings in some of the tested properties.

Keywords: Optical fiber, polyimide coating, medical applications, autoclave, harsh environment

1. Introduction

Fiber optics is successfully used in various areas of medicine, including urology, general surgery, ophthalmology, cardiology, endoscopy, dentistry and medical sensing [1 – 4]. One highly useful characteristic of optical fibers is their ability to enter the tiny passageways and hard-to-reach areas of the human body. Hence most medical applications require optical fibers to be relatively small and flexible so as to negotiate the complex curved anatomy of central spaces of vessels in-vivo: such as arteries, veins, gastrointestinal tracts, bronchi, and urinary tracts.

The majority of medical optical fibers are silica-based, i.e., their core and sometimes the cladding are made of pure or doped silica. Such fibers display significantly higher strength, reliability and optical performance in comparison with plastic optical fibers.

In addition to the core and the cladding, silica optical fibers always contain a polymer coating. Such coatings provide mechanical protection to the silica fiber surface and make fiber handling possible during fiber optic cabling, termination and deployment.

Many specific applications require fibers designed with unique optical, thermal and/or mechanical characteristics. All these properties strongly depend on the type of the utilized coating. Polyimides represent a class of materials successfully used for this purpose. Cured polyimides comprise numerous benefits, including high strength and hardness, low coefficient of friction, good chemical resistance and outstanding thermal stability. Selected polyimide grades are proven to be biocompatible. Another key feature of polyimides is that they can be applied and used reliably with small coating thickness (typically 10 – 15 μm), which is beneficial for size reduction and enhancing flexibility of optical fibers.

Notwithstanding the advantages of polyimides in general, there are various grades of polyimides available on the market, each having unique physical and chemical characteristics. Thus, mechanical, thermal and optical properties of polyimide-coated fibers can vary depending upon the grade of material used for the coating. Furthermore, the coating application and the cure conditions may also influence the fiber properties. Therefore, the polyimide chemistry and manufacturing process must be optimized to achieve the best performance of the fiber.

In this work we investigate the key properties of polyimide-coated optical fibers designed for medical use. A representative OFS fiber is compared with similar fibers obtained from other suppliers. The two non OFS fibers are from leading polyimide-coated optical fiber manufactures. All three fibers are readily available in the marketplace and are assumed to have a fully qualified product pedigree. Primary attention is given to mechanical and environmental tests of the fibers that are driven by their potential use in medicine.

2. Fiber Design

The fibers selected for this study had 200 μm silica glass cores, 220 μm glass claddings and a numerical aperture (NA) of 0.22. The selection was based on the wide medical applicability of such fibers. An OFS fiber (denoted hereafter as **Fiber A**) and two fibers offered by other suppliers (denoted hereafter as **Fiber B** and **Fiber C**) were investigated. The polyimide coating thicknesses were in the range 10 – 15 μm, bringing the coating diameters to 240 – 250 μm.

3. Results and Discussion

3.1 Fiber Strength and Fatigue Parameters

Mechanical strength is one of the most important characteristics of optical fibers. To assess the fiber strength, two approaches were utilized. Two-point bend tests were performed at a strain rate of 4%/min using a Fiber Sigma 2 Point™ Bend Apparatus [5]. In addition, tensile strength tests were performed using an MTS Sintech 5/G tensile bench at strain rates of 4%/min. In the latter case, the fiber specimen lengths were 50 cm. All strength testing was performed at controlled humidity and temperature in accordance with a Telcordia GR-20 condition (RH = 50 ± 5%, T = 23 ± 2°C), even though polyimide-coated specialty optical fibers are not technically covered by GR-20 [6]. The samples were held at least 12 hrs at this condition before the testing, which is also required by the GR-20 standard [6].

Twenty measurements were included in two-point bend testing for each fiber type. The obtained results are shown in **Figure 3.1.1**, with the numerical data summarized in **Table 3.1.1**. The glass fiber strength can be described well by Weibull statistics [7]. This approach uses two parameters for characterizing the strength: the median strength value and the Weibull slope. The latter parameter is a measure of the variability in the strength and is inversely proportional to the standard deviation. A broad distribution of strength (and hence a low Weibull slope) may indicate an out-of-control process of fiber manufacturing.

From **Table 3.1.1** we see that Fiber A displayed the highest strength. Both Fibers A and C broke over narrow ranges of fracture stress, resulting in high Weibull slopes. In contrast, Fiber B displayed two "outlier" points, indicating possible strength problems. Thus, the Weibull slope for Fiber B is much lower than for the other two fibers.

In the two-point bend approach, the tested length of the fiber is very short (~ 1 mm). In theory, the shorter the tested fiber is, the narrower the measured strength distribution will be. On longer pieces of fiber, larger flaws at the glass cladding surface can be revealed with higher probability, which may lower the apparent fiber strength. Thus, comparison of strength data that are collected from both shorter and longer fiber lengths may give an insight to the flaw distribution in the fibers.

The tensile strength data were collected using a 0.5-meter gauge length with ten measurements initially performed for each fiber type. For Fiber B, two "outlier" breaks were observed within the planned 10 measurements, and thereafter, two more strength measurements were performed for this fiber. The obtained results are shown in **Figure 3.1.2** and **Table 3.1.1.**

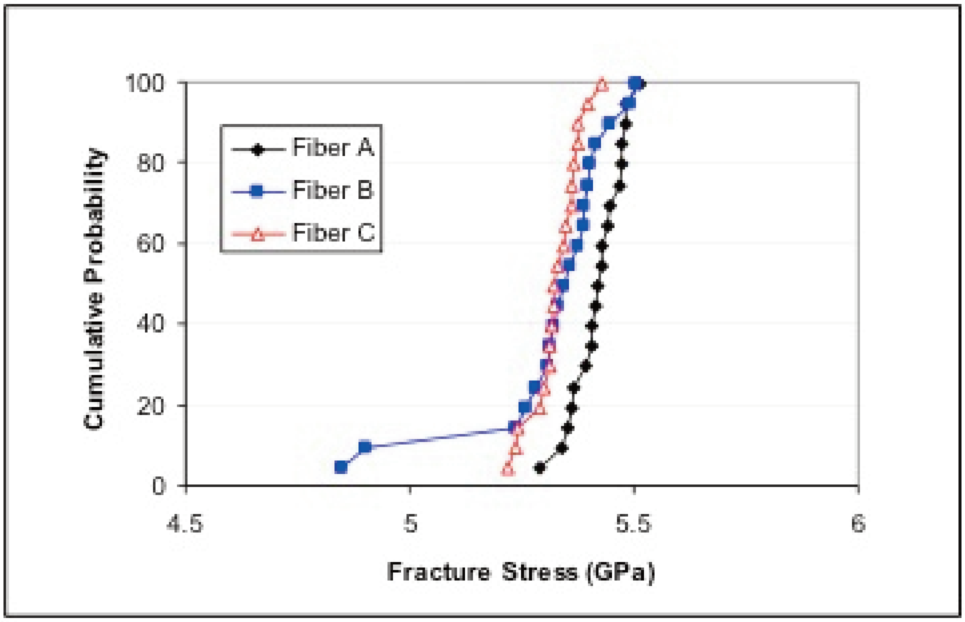

Figure 3.1.1. Strength of "as-drawn" fibers determined via the two-point bend approach at 4%/min strain rate

Table 3.1.1. Strength and Fatigue Parameters of "As Drawn" Fibers

Test Approach	Parameter	Fiber A	Fiber B	Fiber C
Two-Point Bend	Median Strength (GPa)	5.43	5.35	5.33
	Weibull Slope	111	33	116
	n_d value	25	24	20
Tensile, 0.5 m guage	Median Strength (GPa)	4.86	3.80	4.65
	Weibull Slope	104	11	118

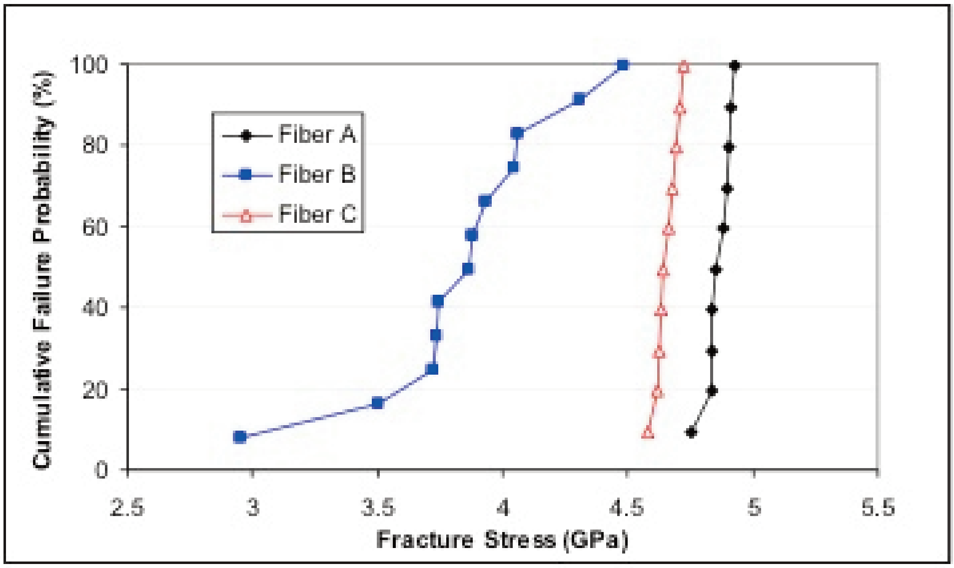

Figure 3.1.2. Strength of "as-drawn" fibers determined via the tensile test approach (0.5 m gauge) at 4%/min strain rate

There is a good correlation between the two methods for the data collected for Fibers A and C, although the strength distributions are shifted to lower values for all of the fibers. The relative weakness of Fiber B is magnified and more obvious using the longer sample lengths. This must be due to the presence of larger or more numerous flaws in this fiber, in comparison with the other two fibers.

The strength values obtained in our work fall in the range 5.3 – 5.4 GPa (~780 kpsi) per the two-point bend testing and 3.8 – 4.9 GPa (550 – 710 kpsi) per the tensile testing. These magnitudes, although reasonable, are at the lower side of the distribution of data documented for polyimide-coated optical fibers [8 – 11]. Explanatory factors may include core and cladding dimensions, glass composition, coating thickness and quality, strain rate and humidity during testing.

It is known that the strength of silica optical fibers under stress is time-dependent due to crack growth that is enhanced by moisture. The degradation of fiber over time is known as fatigue and is characterized by the stress corrosion parameter (nd). Higher values of nd correspond to lower rates of crack growth, i.e. to higher mechanical reliability of the optical fiber [12].

In our study, the stress corrosion parameter was measured for each fiber at strain rates of 0.08, 0.57, 4 and 28%/min using the two-point bend technique. Ten measurements were taken per each strain rate. The obtained results are shown in **Figure 3.1.3**. From the plots, dynamic stress corrosion parameters were determined (**Table 3.1.1**). Fibers A and B show similar high nd values (~24), while nd for Fiber C it is lower (~20). In the publications quoted above, the following magnitudes of nd were obtained: 38 [8], 23 [9], 24 [10], 22 [11]. Thus, our data agree well with the documented ones.

The differences in strength can, in principle, derive from factors including the glass layer, the coating layer, or variables related to the draw process or draw environment. Whatsoever, Fiber A shows the best performance in strength and stress corrosion resistance within this set. Fiber B exhibits a problem with its strength uniformity (low Weibull slope, larger or more numerous flaws present). At the same time Fiber C displays the lowest nd value, indicating somewhat poorer stress corrosion behavior among the studied fibers.

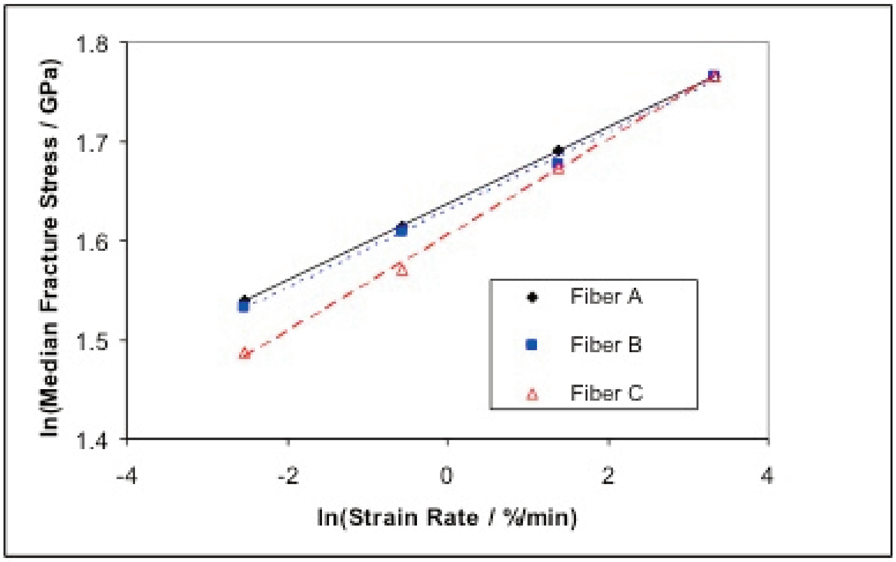

Figure 3.1.3. Stress corrosion data for "as-drawn" fibers determined via the two-point bend approach at 4%/min strain rate

3.2 Coating Behavior at Elevated Temperatures

There are two major concerns regarding the use of polyimide-coated fibers at elevated temperatures. First, at elevated temperatures (e.g., during delivery of high laser power), the fibers should not release any unwanted chemicals into the environment. Second, in some applications the fibers must be overjacketed by a plastic, and the process involves passing the fiber through a hot extruder. In the latter process, the fiber coating must sustain a short term heating. Depending on the plastic being used and the extrusion conditions, the upper temperature of the process may be as high as 400°C.

To model possible adverse effects of the high temperatures, we performed the following study. The fibers (short pieces) were placed into a thermal oven preheated to 430°C. The fibers were arranged vertically in a specially designed fixture so that they did not make contact with anything other than the hot air. The exposure time was 30 seconds.

Thereafter, the fibers were inspected under a microscope to reveal possible defects.

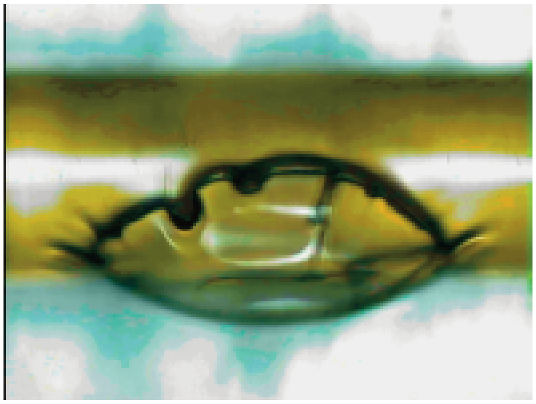

Figure 3.2.1. Microscopic image of Fiber C after a 30 sec exposure to 430°C

Following this challenge, Fibers A and B exhibited no change in their appearance. At the same condition, the coating on Fiber C developed bubbles and lost its integrity (**Figure 3.2.1**). The bubble formation indicates that coating chemicals were partly released into the atmosphere. Such release is intuitively unfavorable for medical applications and may create problems upon cabling by plastic materials.

3.3 Effects of Autoclaving

Since autoclaving is historically a common method of sterilization, medical grade optical fibers typically must withstand this exposure to steam. Given that the same fiber is sometimes reused several times, it may also be important for the fiber to survive multiple autoclaving cycles.

For autoclaving experiments, we used a Napco E Series Test Chamber, Model 8100-TD. Each sample was conditioned in the autoclave using the following procedure:

- The samples were placed into the autoclave (which had been filled with the prescribed amount of water).
- The autoclave was turned on and reached maximum temperature and pressure of 121 – 122°C and 0.22 MPa (32 psi) in about 20 minutes.
- The samples were left in the autoclave for 20 minutes (after proper temperature and pressure had been reached).
- The autoclave was shut off and the pressure was allowed to decrease.
- Once the temperature had decreased to a safe value, the door was opened.

- The excess water was drained, the reservoir was topped off, and the cycle was repeated.

Each cycle required about 40 – 45 minutes. The samples were subjected to 10, 20, 30, 40 and 50 cycles. In most medical applications, the maximum number of autoclave cycles does not exceed 20. Thus, in our study we strongly exceeded the "most extreme" practical condition. The conditioned fibers were then examined under a microscope and thereafter tested for their strength.

After all 50 cycles, we saw no significant changes in the appearance of Fibers A and B. In contrast, after as few as 10 cycles, Fiber C began showing "bright spots" when using polarized light microscopy. Those bright spots indicate intrinsic stress localized within the coating. Moreover, development of micro-cracks was revealed in the coating for Fiber C (**Figure 3.3.1**). The "bright spots" in polarized light were localized around those cracks.

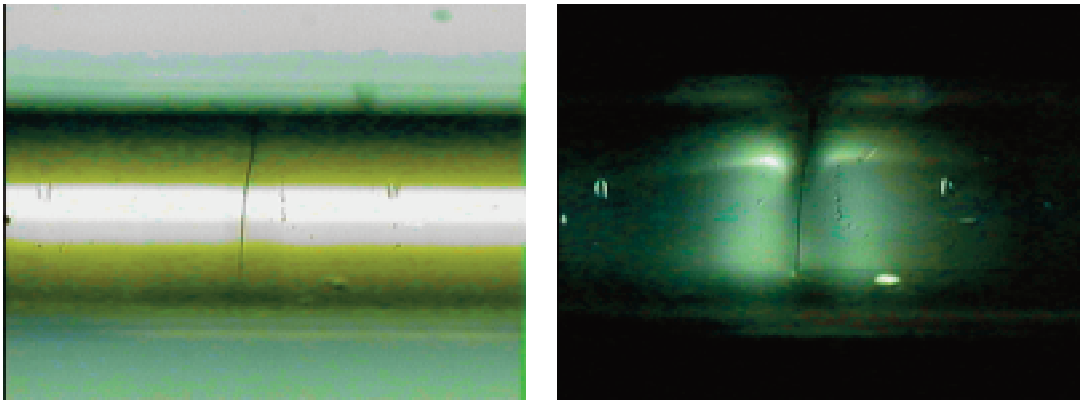

Figure 3.3.1. Microscopic images of Fiber C after 10 autoclave cycles using non-polarized (left) and polarized (right) imaging techniques

The fiber strength after 10, 20, 30, 40 and 50 autoclave cycles was determined by two-point bend testing. Sixteen specimens were tested for each fiber type and number of cycles. The results are summarized in **Table 3.3.1**. **Figure 3.3.2** displays the autoclave effects on the median fiber strength. Fibers A and B withstood the autoclave cycles successfully, while Fiber C became progressively weaker during the test. In addition to the median strength, we determined the nd values for the 50-times autoclaved fibers.

Fiber Type	Unaged	10 Cycles	20 Cycles	30 Cycles	40 Cycles	50 Cycles
Fiber A	5.43	5.44	5.38	5.37	5.38	5.33
Fiber B	5.35	5.56	5.50	5.43	5.46	5.50
Fiber C	5.33	4.16	4.66	4.58	3.40	3.45

Table 3.3.1. Median Fiber Strength (GPa) vs. Number of Autoclave Cycles

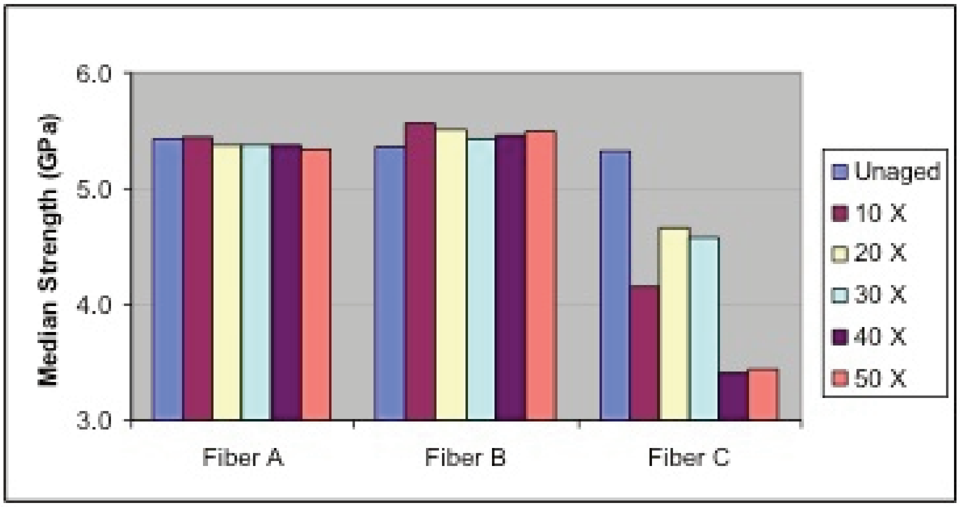

Figure 3.3.2. Median strength of autoclaved fibers as a function of the number of cycles

As above, two-point bend testing was performed at 0.08, 0.57, 4 and 28 %/min strain rates. The number of tested specimens was 5, 5, 16 and 10 for the aforementioned strain rates, respectively. The obtained data are shown in **Figure 3.3.3**. The determined nd values are 27, 24 and 16 for Fibers A, B and C, respectively. Thus the obtained stress corrosion parameters show a similar trend as we saw for the unaged fibers (**Figure 3.1.3**). A significant difference is that the autoclaved Fiber C exhibited much lower strength at all studied strain rates.

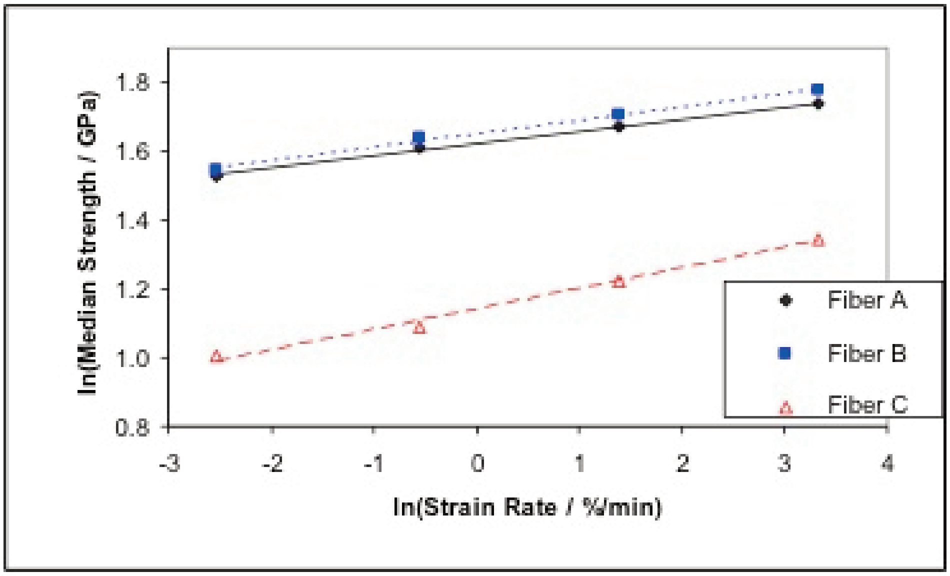

Figure 3.3.3. Stress corrosion data for 50-times autoclaved fibers

Thus, Fibers A and B withstood autoclaving (up to 50 cycles) with no significant problems while Fiber C exhibited significant (up to 35%) strength loss under the same conditions.

3.4 Effects of Immersion in Water and Organic Solvents

In their end use or during assembly into a component or device, optical fibers may be exposed to various solvents or water. It is important that the fibers sustain their strength upon such interactions. Sensitivity of fibers to organic solvents and water was studied in the following way. Short pieces (6 cm) of each fiber were prepared. The fiber ends were cautiously cleaved using a hand-held diamond scribe tool. Those specimens were placed into sample tubes (10 pieces of each fiber per tube) and the tubes were filled with a selected liquid. The tubes were sealed and kept at ambient temperature for 190 hrs (~8 days). Then the liquids were removed from the tubes, and the specimens were dried for approximately 1 hr. Immediately thereafter, the fiber ends were inspected under a microscope (with a goal to detect any coating swell-off). Then the fibers were stored for one day at 23°C/50% RH and their strength was tested via the two-point bend technique (4%/min strain rate).

Based on their extensive use in medical and industrial areas, three liquids were selected for the immersion tests: acetone, isopropyl alcohol (IPA) and water.

A careful inspection of the fiber ends after the exposures showed absence of any residual swell-off effects for any of the fiber/solvent systems.

Table 3.4.1 summarizes the obtained strength data and **Figure 3.4.1** illustrates the same data. Here, the error bars correspond to the standard deviations of the measurements. Fibers A and C proved to be insensitive to the solvents, while Fiber B weakened after exposure to acetone.

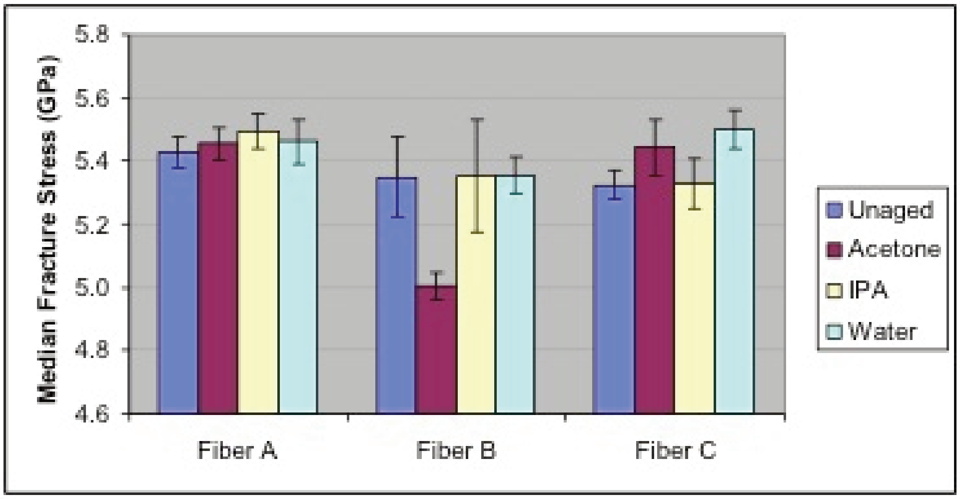

Figure 3.4.1. Solvent effects on median fiber strength

Fiber Type	As Drawn	Acetone	IPA	Water
Fiber A	5.42	5.46	5.49	5.46
Fiber B	5.35	5.00	5.35	5.36
Fiber C	5.33	5.44	5.33	5.50

Table 3.4.1. Solvent Effects on Median Fiber Strength (GPa)

3.5 Acid Strip of the Coating

In all fiber applications, at least one fiber end must be terminated in such a way as to enable coupling to a light source. Often this requires removal of the polymer coating from the end of the optical fiber [13]. For polyimide coatings, acid (chemical) stripping is recommended. Normally, hot (140°C) sulfuric acid is utilized for that purpose. The basic procedure includes several steps of immersing in the acid, rinsing and drying the specimens.

An objective of our study was to see whether this procedure is equally applicable to the fibers provided by different suppliers. If the coating would not be fully removed by the acid, we would expect to see some residue at the cladding surface. Also, it was of interest to follow the kinetics of the coating removal. For this, we varied the time of immersion of the fiber ends into the acid (1 and 2 minutes in total). Three specimens of each fiber type were tested for each immersion time condition. The obtained fiber specimens were inspected under microscope and by FTIR.

After the 1-minute immersion in the acid, we found that the coating was fully removed from Fibers A and C. In contrast, a significant layer of residue was found for Fiber B. FTIR analyses confirmed that the residual layer was due to unreacted polyimide. It follows that the coating of Fiber B has the slowest removal rate with sulfuric acid.

After the 2-minute immersion in the acid, the coating was fully removed from all three fibers. Thus, notwithstanding the reaction kinetics, the coatings were adequately strippable by sulfuric acid for all studied fibers.

3.6 Effects of Long-Term Thermal and Humid Aging

The goal of this portion of our study was to observe possible effects of relatively long-time thermal and humidity aging on the fiber strength. In the thermal aging study, the fibers were kept at 175°C for 30 days. In the humid aging study, the fibers were conditioned at 85% RH/85°C, also for 30 days.
The strength of the aged fibers was determined via two-point bend testing. Sixteen specimens were tested for each fiber/aging condition. **Figure 3.5.1** displays the obtained strength diagrams. The error bars correspond to the standard deviations. Definitely, Fiber A survived both aging conditions successfully. In contrast, Fibers B and C exhibited some weakening upon the aging.

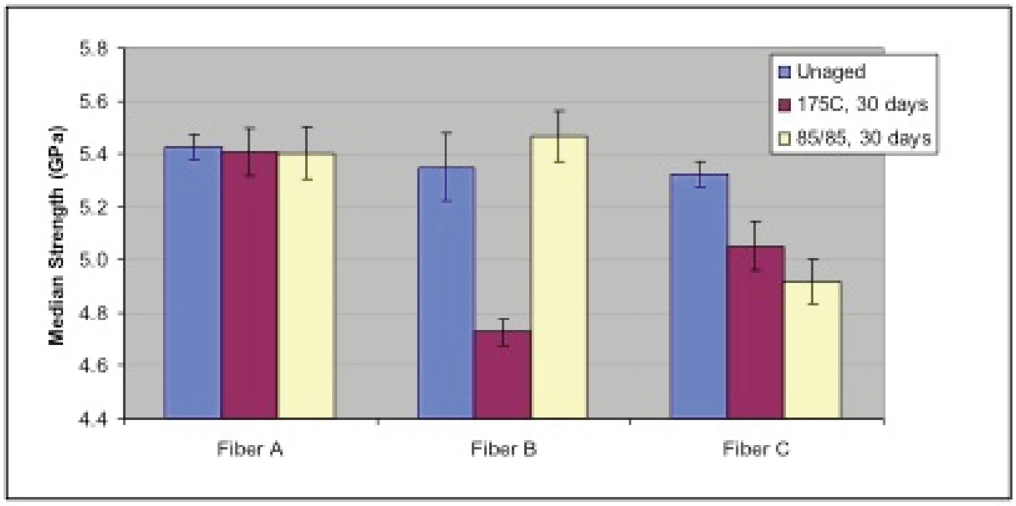

Figure 3.5.1. Aging effects on fiber strength

4. Conclusive Remarks

Table 4.1 summarizes the most important results obtained for the trialed fibers.

Property	Fiber A	Fiber B	Fiber C
Meridian Strength, unaged, tensile test (GPa)	4.86	3.80	4.65
Weibull slope, unaged, tensile test	104	11	118
Median strength, unaged, 2-pt bend (GPa)	5.43	5.35	5.33
Weibull slope, unaged, 2-pt bend	111	33	116
n_d, unaged, 2-point bend	25	24	20
“Bubbling” upon rapid heating to 430°C	No	No	Yes
Strength loss, autoclave (50 cycles)	No loss	No loss	35%
n_d, autoclave (50 cycles)	27	24	16
Strength loss in water (8 days, 23°C)	No loss	No loss	No loss
Strength loss in acetone (8 days, 23°C)	No loss	No loss	No loss
Strength loss in IPA (8 days, 23°C)	No loss	No loss	No loss
Acid strip	OK	Slower	OK
Strength loss, 30 days @ 175°C	No loss	12%	6%
Strength loss, 30 days @ 85% RH/85°C	No loss	No loss	8%

Table 4.1. Data Summary for Polyimide-Coated Fibers

It follows that the fibers provided by different vendors exhibit significant differences in their properties. While the OFS Fiber (A) survived all of the harsh conditions that were trialed, the performance of the other two fibers was lesser in some respects. The observed differences should be attributed to the coating material selection and the fiber manufacturing process, which were optimized for Fiber A.

Once the fiber and coating design are optimized, polyimide-coated fibers can successfully withstand harsh environments, including multiple autoclaving, exposure to organic solvents and extreme thermal and humid aging. All the listed properties are crucial for medical applications of the fiber. At the same time, the polyimide coating can be effectively removed for fiber termination by immersion in sulfuric acid.
Finally, if the fiber is intended for use inside a cable, it must withstand rapid heating to high temperatures (up to 400°C) inside an extruder. The OFS Fiber (A) proved to survive such condition successfully.

Disclaimer

Some of the results herein are based on the latest techniques of accelerated testing. Although as such, they may provide an indication of the useful service performance of materials, they do not constitute or imply any warrantee on behalf of OFS. No warrantees other than OFS' normal contractual warrantees should be inferred.

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