

# Acrylate-Based Specialty Optical Fiber Coatings for Harsh Environments

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## Abstract

Applications of optical fibers at high temperatures and in harsh environments demand coatings sustainable under those conditions. The goal of this work was to evaluate thermal and environmental stability of a series of commercially available and in-house formulated acrylate-based coatings. Dual and single-coat fiber designs were tested. The individual formulations included non-urethane and urethane acrylates, silicone acrylates and an organic-inorganic hybrid acrylate. The “as drawn” fibers were tested for attenuation, mechanical reliability, microbend sensitivity, delamination resistance and coating stripability. Thermogravimetric analysis (TGA) was used for estimating the use temperature of the coatings. In addition, the fibers were exposed to harsh environments, including dry aging in air (150°C) and in nitrogen (250°C), autoclaving, immersion in high pressure/temperature water (up to 200°C and 2000 psi) and immersion in a hot cable gel (160°C). Based on the obtained results, the best formulations were ranked for suitability in different types of fiber application.

**Keywords:** Optical fiber; thermal stability; coating; acrylate; silicone; TGA; degradation; low temperature; attenuation.

## 1. Introduction

There are several requirements for polymer coatings used on optical fibers. The main role of a coating is to mechanically protect the optical waveguide from glass surface contact and breaks, ensuring long term mechanical reliability [1]. Next, an ideal coating should cushion microbends to minimize optical losses. Also, to terminate the fiber easily, the coating should be removable with a suitable technique, most desirably with a mechanical strip tool. Applications of optical fibers at harsh conditions complicate requirements for the coating performance [2-8]. The list of harsh conditions may include high and low temperature ranges, high humidity, high pressure, aggressive chemicals, mechanical interactions or combinations of the above. During use, a high quality fiber must have low optical attenuation in combination with high mechanical reliability. The goal of this work was to evaluate thermal and environmental stability of a series of commercially available and in-house formulated acrylate-based coatings. Dual and single-coat designs were tested. The individual formulations included non-urethane and urethane acrylates, silicone acrylates and an organic-inorganic hybrid acrylate. We fabricated graded-index 50/125/250  $\mu\text{m}$  fibers with those coatings. The “as drawn” fibers were tested for attenuation, mechanical reliability, microbend sensitivity, delamination resistance and coating stripability. Thermogravimetric analysis (TGA) was used for estimating the use temperature of the coatings. In addition, the fibers were exposed to harsh environments, including dry aging in air (150°C) and in nitrogen (250°C), autoclaving, immersion in high pressure/temperature water (up to 200°C and 2000 psi) and immersion in a hot cable

gel (160°C). Based on the obtained results, the best formulations were ranked for suitability in different types of fiber application.

## 2. Coating material selection

A series of acrylate-based materials was trialed as primary (“P”), secondary (“S”) coating and monocoat (“M”) types. Table 1 displays the list of the coatings. Most of the trialed formulations use polyether and/or polyester backbones. The exceptions are: P8 and P9 use polybutadiene-based oligomers (PBD); Si1 and Si2 use silicone-based oligomers, and Hyb uses a silsesquioxane backbone.

Table 1. List of the coating materials

Coating ID	Description	$E'_{23^\circ\text{C}}$ (MPa)	$E'_{\text{eq}}$ (MPa)	$T_g$ (°C)
P1	U	1.1	1.1	-36
P2	U	1.0	1.0	-18
P3	U	9.3	7.7	9
P4	U	1.4	1.4	-19
P5	U	15.5	6.5	19
P6	P2+10%CR, U	8.0	5.7	-4
P7	P2+20%CR, U	46.8	16.9	0
P8	PBD-based, U	12.5	6.7	1.5
P9	PBD-based, N	4.1	3.6	-40
Si1	Silicone-acrylate, N	3.5	2.7	-99
Si2	Si1+AP, N	3.5	2.7	-99
S1	U	1420	35.6	67
S2	U	1220	42.9	65
S3	N	760	13.2	37
S4	U	1350	69	57
S5	U	1590	1.0	61
M1	U	1300	1.1	61
M2	N	730	13.7	37
M3	M2+AP, N	760	13.7	78
M4	N	2240	43.8	126
Hyb	Hybrid, N	550	131	47

Some of the compositions contain urethane (marked “U” in Table 1). Formulations containing no urethane are marked “N”. Compositions P6 and P7 were made by adding 10 and 20% of a crosslinker (CR) to formulation P2. Compositions Si2 and M3 were

obtained by adding 1% of an adhesion promoter (AP) to Si1 and M2, respectively. Table 1 also shows the data on storage modulus at 23°C ( $E'_{23^\circ\text{C}}$ ), equilibrium storage modulus ( $E'_{\text{eq}}$ ) and the glass transition temperature ( $T_g$ ) obtained using Dynamic Mechanical Analysis (DMA) at 1 rad/sec.

### 3. Fiber design

The coatings were applied on optical fiber having a 50  $\mu\text{m}$  graded-index (GI) core and 125  $\mu\text{m}$  diameter silica cladding. The primary and secondary coating diameters were  $190\pm 5$  and  $250\pm 10$   $\mu\text{m}$ , respectively. The numerical aperture of the fibers was 0.2. Fibers with this particular design are known to be strongly bend sensitive, which helps in ranking coating effects on the fiber attenuation. In addition GI 50/125  $\mu\text{m}$  fibers are commonly used in downhole applications [9] where some of the harsh conditions can be encountered. Sixteen fibers were drawn at OFS. The coatings discussed in the previous section were applied on the fibers in accordance with Table 2.

**Table 2. List of drawn fibers**

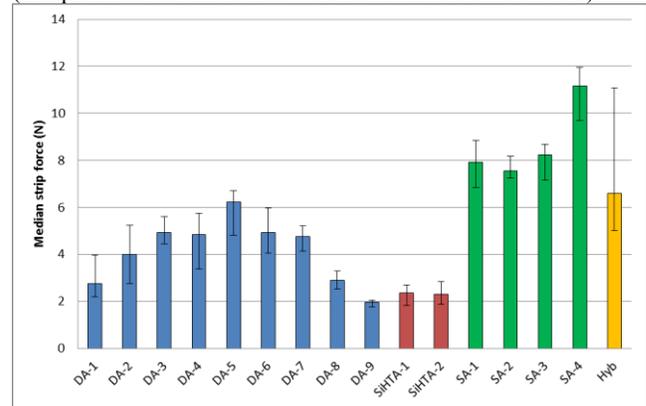
Fiber ID	Single or dual coat	Primary coating	Secondary coating
DA-1	Dual	P1	S1
DA-2	Dual	P2	S2
DA-3	Dual	P3	S3
DA-4	Dual	P4	M2
DA-5	Dual	P5	S5
DA-6	Dual	P6	M2
DA-7	Dual	P7	M2
DA-8	Dual	P8	S4
DA-9	Dual	P9	S4
SiHTA-1	Dual	Si1	S4
SiHTA-2	Dual	Si2	S4
SA-1	Single	M1	
SA-2	Single	M2	
SA-3	Single	M3	
SA-4	Single	M4	
Hyb	Single	Hybrid	

## 4. Results

### 4.1 Coating stripability

Coating strip force was measured at room temperature using an MTS Sintech 5/G tensile bench fitted with a Microstrip tool (338  $\mu\text{m}$  guide tube, 161  $\mu\text{m}$  strip blade) at a pulling speed of 500 mm/min and a strip length of 30 mm. The results are summarized in Figure 1. The error bars represent the range of the measured values. The target strip force value was in the range 1 – 9 N. We see in general that single-layer coatings require higher strip force.

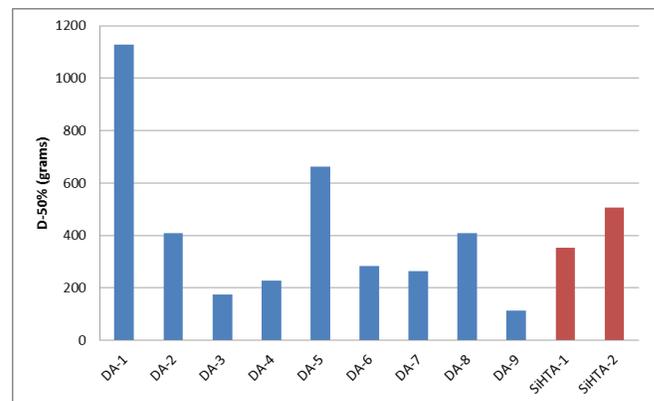
Also, one of the coatings exhibits a strip force above 9 N. For this particular fiber it is difficult to strip the coating without damaging the rest of the fiber. Another important observation is that the strip force seems not be affected by the adhesion promoter (compare SiHTA-1 with SiHTA-2 and also SA-1 with SA-2).



**Figure 1. Median coating strip force. Error bars show the range of the observed values**

### 4.2 Delamination resistance

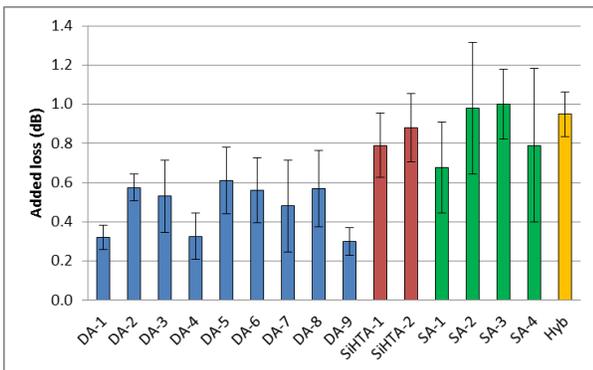
The delamination resistance is one of the features that characterize robustness of dual coatings. The delamination resistance was determined according to the method developed previously [10]. In this method, an optical fiber is placed on a glass slide and indented by a 6.35 mm diameter steel rod, aligned perpendicularly to the fiber. At high applied load, the indentation may cause a delamination between the primary coating and the glass cladding. The rod is connected to a free-floating plate, which is loaded with a desired test weight. The rod is lowered at speed of 1.7 mm/sec onto the fiber, rests for 6 seconds on the fiber and is then raised off of the sample. For each tested fiber specimen, 30 sites are impacted at equivalent distances of 6.35 mm from each other. Then the fiber is placed in an index matching liquid (glycerin), and possible delaminations are examined under a microscope. The probabilities of delamination, i.e., the ratios of the number of damaged sites to the total sites, are plotted versus load in grams. These data are then fit with Weibull functions, and the load for 50% delamination, D-50%, is calculated. The obtained D-50% values are shown in Figure 2.



**Figure 2. Delamination resistance (D-50%) values for dual-coated fibers**

### 4.3 Microbend sensitivity

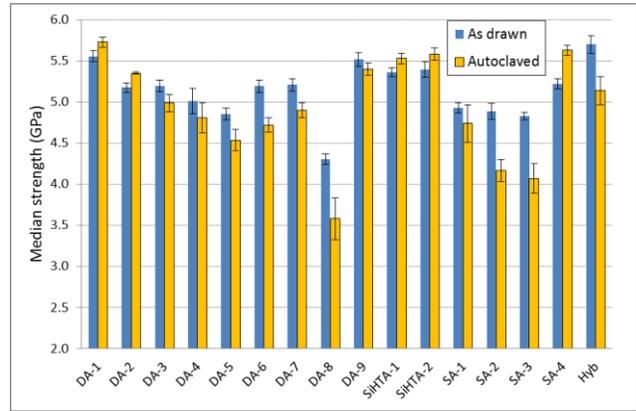
Microbends of optical fibers are known to induce additional optical loss. It is widely known that effects of microbends can be minimized by using a dual coating with soft primary and hard secondary layers [11]. The main purpose of the primary coating is to cushion possible mechanical perturbations and this way it reduces microbends in the fiber. It is therefore assumed that without using a soft primary coating (as for instance in single-coated fibers), the fiber should be susceptible to significant microbend-induced losses. Different approaches to assess the microbend sensitivity are known [12]. In this work, we implemented “Method C,” described in IEC/TR 62221 Technical Report [12]. In this method, a fiber is deployed as a loop with a diameter of ~98 mm on a rubber pad having a Shore A hardness of 75. The sample is covered from above by a plain metal woven mesh with a mesh number of 70. A flat plate is positioned above the mesh, which allows imposing different loads onto the fiber. The transmitted optical signal (at 850 and 1300 nm) was measured during the fiber loading. The fibers ends were cleaved and fixed inside ST connectors. Mode conditioning was performed either by transmitting light through a >1 km spool of the fiber under test (from the launch end) or by wrapping the fiber under test 5 times around a 2.54 cm mandrel. Thus the mode distribution in tested pieces of the fiber in both approaches was expected to be close to the equilibrium modal distribution. Figure 3 displays the results obtained using 2.5 kg load on each fiber. As expected, we observe lower microbend-induced losses for dual coatings. For coatings with our particular silicone primary and for single coats, the effect is ~1.5 – 2 times stronger than for most of the dual-acrylate coatings.



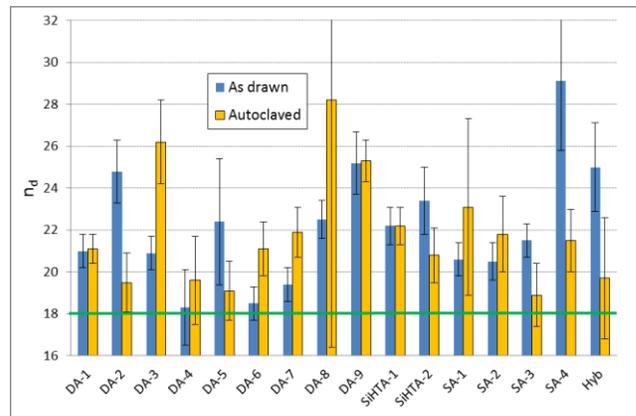
**Figure 3. Microbend-induced loss at 2.5 kg load force on the fibers measured at 1300 nm. The error bars correspond to standard deviations**

### 4.4 Fiber strength and fatigue

Mechanical strength of the fibers was determined using a 2-point bend technique (Fiber Sigma Instruments) at a strain rate of 4%/min. For evaluating the dynamic stress corrosion parameter,  $n_d$ , the 2-point bend data were collected at strain rates of 0.08, 0.57, 4 and 28%/min. The fibers were preconditioned at 23±2°C/50±5%RH for at least 24 hours before the testing. Figures 4 and 5 display the obtained data. Most fibers display strengths close to or above 5 GPa, which is typical for glass optical fibers. For one of the fibers (coating DA-8), the observed strength was below 4.5 GPa, which might be related to the primary coating. The industry standard for  $n_d$ -value is  $n \geq 18$ . There are two coatings with critically low  $n_d$ -values: DA-4 and DA-6.



**Figure 4. Median strength of as drawn and autoclaved fibers. The error bars show the ranges of the values**

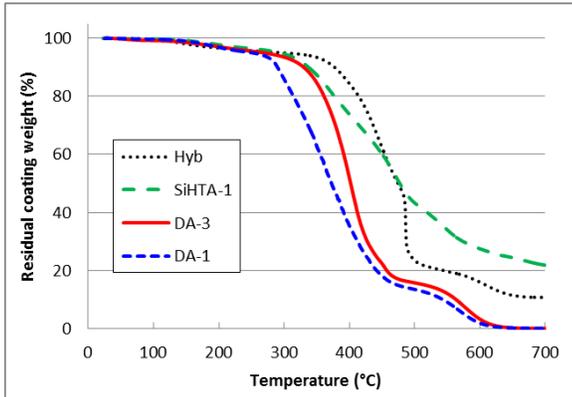


**Figure 5. Stress corrosion parameters determined for the as drawn and autoclaved fibers. The error bars show the 95% confidence intervals**

### 4.5 Thermal stability via TGA

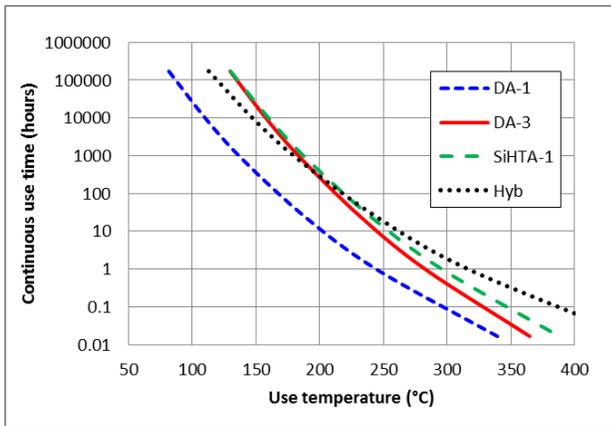
The term “thermal stability” may have a different meaning depending on the application of a particular polymer, coating or a product comprising those materials. For an end user of a specialty optical fiber, it is necessary to know the highest temperature at which the fiber can function for a given duration of time, or equivalently, how the fiber’s performance changes as a function of temperature and time. There are three essential elements in defining thermal stability of a specialty optical fiber. They are: fiber performance, use temperature, and duration or lifetime. For all practical purposes, we may define the thermal stability of specialty optical fiber as the highest temperature at which the optical fiber retains its acceptable performance level for a specific duration, e.g., 20 years. The acceptance criteria for fiber performance must be defined based on the application of particular fibers. Since thermal decomposition of the coating eventually leads to a failure of the fiber, it is often possible to characterize the thermal stability of the fiber through its coating decomposition. Following our previous approach [13], we assume (somewhat arbitrarily) that the coating “fails” when it loses 25% of its initial weight due to thermal decomposition. The time for a coating to lose 25% of its initial weight can be

determined from thermogravimetric analysis (TGA) [13]. In this work, weight loss vs. temperature was analyzed using a TA Instruments Model 2950 thermogravimetric analyzer at heating rates in the range 0.5 – 20°C/min in air. The samples were short pieces of coated optical fibers. Figure 6 displays TGA curves obtained for a series of the studied coatings. The weight of the glass is subtracted from these data. Above 700°C, most of the acrylate coatings are fully burned away, however some residue remains after similar heating of the Hyb and SiHTA coatings.



**Figure 6. TGA curves obtained for selected fiber coatings at 20°C/min in air environment**

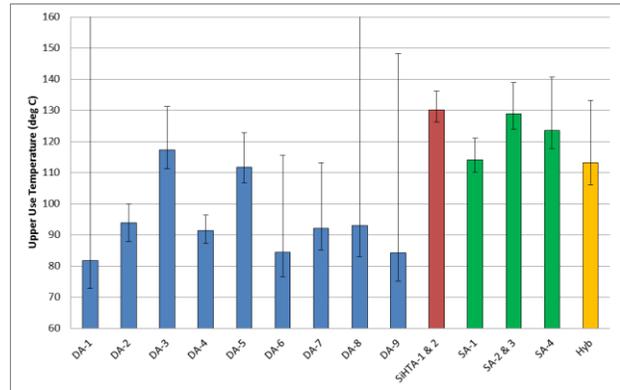
The experimental data for 25% wt. loss can further be fit using the Arrhenius equation and extrapolated to other time-temperature conditions. Examples of the extrapolated curves are given in Figure 7.



**Figure 7. 25% wt. loss data extrapolation for selected fiber coatings in air environment**

Figure 8 summarizes the extrapolated data for all the coatings. It can be seen that many dual coatings “fail,” i.e., lose 25% of their initial weight, within 20 years of continuous use at temperatures of about 85°C. This agrees well with the known temperature rating for telecom grade acrylate coatings and tends to justify the selected failure criterion. Using the same failure criterion for all the coatings, it can be seen that there is a group of coatings with definitely higher thermal stability: DA-3, DA-5, SiHTA (1 and 2) and all of the mono-coats. The observed maximum upper use temperature did not exceed 130°C in any case. Although we studied just a limited group of coating formulations, it appears that 130°C is about the maximum temperature that can be achieved with acrylate-type materials for

continuous (20 year) use, at least using the 25% weight loss criterion. Unexpectedly, the hybrid formulation did not show a significant advantage, while SiHTA exhibits the highest thermal stability within the series.



**Figure 8. 20-year upper use temperatures for different coatings in air assuming 25% wt. loss criterion**

#### 4.6 Dry aging in nitrogen environment

In this test, the fibers were aged 1 day in a nitrogen environment at 250°C and a pressure of 1500 psi (10.3 MPa). After the aging, the fibers were removed from the vessel and inspected for stickiness, odor and discoloration. There were two coatings that became semi-liquid upon the aging: DA-1 and DA-2, which should be considered as the worst behavior. Two more coatings, DA-8 and DA-9 became strongly “sticky.” The rest of the coatings developed only minor tackiness and some discoloration upon the aging.

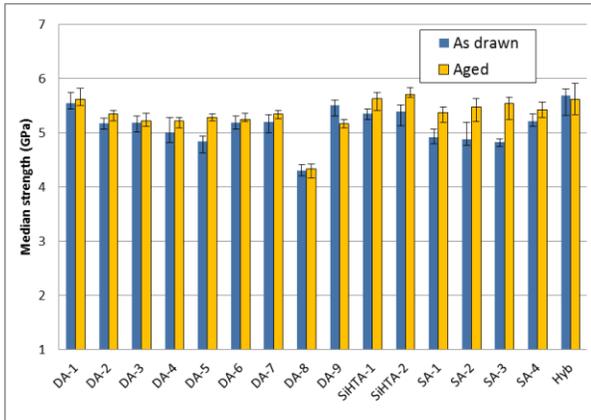
#### 4.7 Dry aging in air environment

We performed a dry aging experiment for short (~10 cm) fiber pieces that were placed in sample tubes (a single specimen per tube). The sample tubes were inserted into an oven preset to 150°C with no humidity control. The fiber images and coating diameter data were collected in 1, 7, 14, 21 and 28 days after the start of aging. Figure 9 displays some of the fiber images after 28 aging days. For all coatings we observed 5 – 10% diameter shrinkage and some discoloration. One of the coatings (Hyb) cracked spirally upon the aging. Visually, SiHTA-1 and SiHTA-2 coatings look the least affected by the aging; in particular they showed the least yellowing.



**Figure 9. Images of DA-2, SiHTA-1 and Hyb fibers after 28 days aging at 150°C in air**

The fiber strength of the aged fibers measured via two-point bend approach shows no strength degradation within the series, aside from possibly small decrease for fiber DA-9 and Hyb (Figure 10). In most cases the fiber strength increased upon dry heat aging.



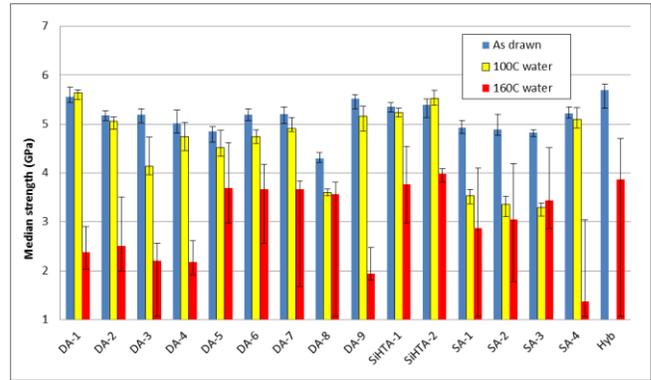
**Figure 10. Median strength of fibers before and after dry aging in air at 150°C for 28 days. The error bars show the range of the obtained values**

#### 4.8 Effects of Autoclaving

The autoclaving was applied in saturated steam at 132°C, 30 psi (0.2 MPa). Three cycles were performed, with 1 hour spent at the highest temperature-pressure condition. Thereafter, the fiber strength and  $n_f$  values were determined via 2-point bend testing as described above. Figures 4 and 5 display the data obtained before and after the autoclaving. There is a group of fibers where a significant strength reduction was observed: DA-8, SA-2, SA-3 and Hyb. For another group of the coatings, we find the  $n_f$ -value after autoclaving overlapping 18 with error bars: DA-2, DA-4, DA-5, DA-8, SA-3 and Hyb. The rest of the fibers sustained this harsh condition without any issues.

#### 4.9 Aging in high-temperature/pressure water

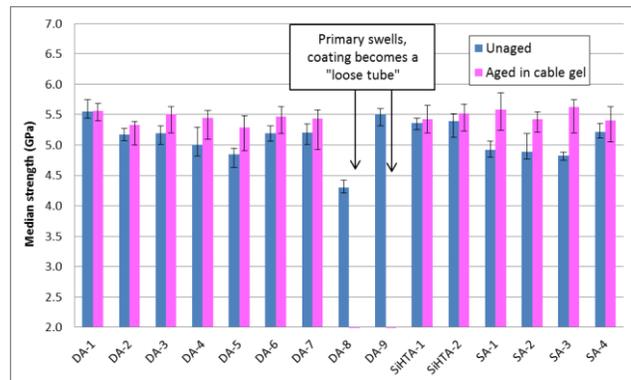
In addition to the above autoclave procedure, we exposed our fibers to even more challenging conditions. The fibers were immersed in high-temperature/high-pressure water using a stainless steel pressurized vessel. The pressure of 2000 psi (13.8 MPa) was applied using nitrogen gas from a cylinder. In separate experiments, the aging temperatures were 100, 160 and 200°C and the aging time was 7 days. The specimens were fibers coiled to ~5 cm diameter. After each exposure, the specimens were dried, preconditioned at 50% RH, and 2-point bend tested as described above. Figure 11 displays the obtained results. The fiber strength behavior after 100°C/2000 psi water aging is similar to the one observed after the above autoclave condition (132°C/30 psi). Although a significant strength reduction was observed for some of the fibers (DA-3, SA-1, SA-2 and SA-3), their ultimate strength exceeds 3 MPA and the fracture stress distribution is tight. After 160°C exposure, most of the fibers become really weak so that they break at handling. Still there is a group of fibers that survived this harsh condition exhibiting the minimum fracture stress above 3 GPa: DA-5, SiHTA-1, SiHTA-2 and SA-3. After 200°C (not shown in Figure 11), all fibers became very weak.



**Figure 11. Median strength of fibers before and after aging in high-pressure water. The error bars show the range of the obtained values**

#### 4.10 Aging in Cable Gel

As another test, we immersed the fibers in a cable gel (Infogel LAHX 6015/146 D/A) and aged them at 160°C for 7 days at 2000 psi (13.8 MPa). After the exposure, the fibers were wiped with dry paper, preconditioned at 50% RH, and their strength was then measured using the 2-point bend technique. The obtained results are displayed in Figure 12. Most of the fibers survived this exposure with no strength degradation and even appeared to become stronger (as in Figure 10). The two fibers that had issues are those with the PBD-based primary coatings (DA-8 and DA-9). Those coatings strongly swell in the gel, becoming loose and easily removable from the glass.

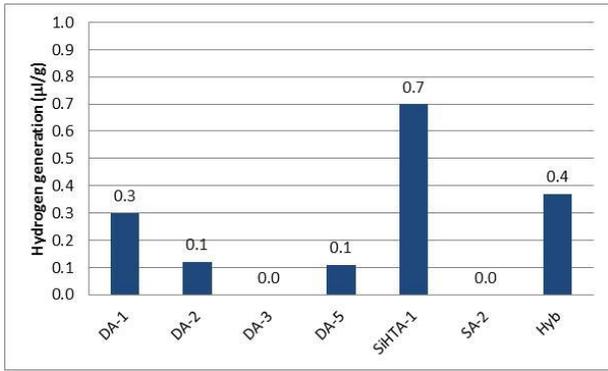


**Figure 12. Median strength of fibers before and after aging in cable gel at 160°C (2000 psi, 7 days). The error bars show the range of the obtained values**

#### 4.11 Hydrogen evolution

The hydrogen generation was measured for selected fibers only, using gas chromatography. The fibers were preconditioned by heating in 150 mm Hg oxygen for 24 hours at 100°C. The obtained results ( $\mu\text{l}$  per gram of the coating) are shown in Figure 13. Target values were  $\leq 10 \mu\text{l/g}$ , and preferably  $\leq 1 \mu\text{l/g}$ . The observed values are below 0.7  $\mu\text{l/g}$  and there were two coatings (DA-3, SA-2) where the generated hydrogen was below the detection limit of the equipment. It should be noted that the hydrogen generation from the silicone-acrylate coating studied in

this work ( $\sim 0.7 \mu\text{l/g}$ ) is much lower than typical values found for RTV vinyl-hydrise silicone coatings ( $\sim 100 \mu\text{l/g}$ ).

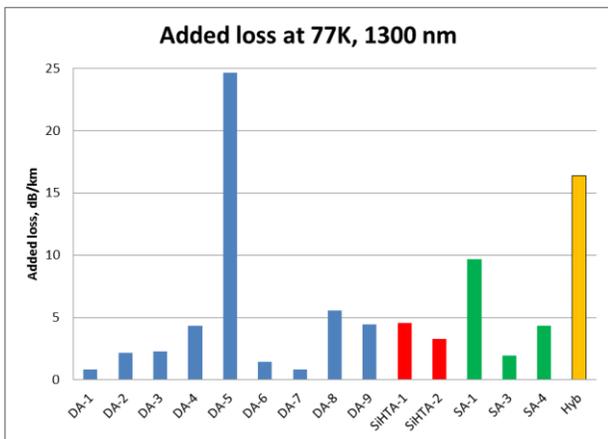


**Figure 13. Hydrogen generation produced by different fiber coatings**

#### 4.12 Attenuation at low temperatures

Another important feature of an optical fiber is the attenuation that can develop when the fiber is used at low temperatures. In most cases, such attenuation increase is due to coating shrinkage that leads to microbends in the fiber. A quick way for testing such properties is immersing the fibers in liquid nitrogen ( $-196^\circ\text{C}$ ) with real-time transmission monitoring [14]. We used an AFL OLS1 dual optical light source operating at 850 and 1300 nm and an AFL Optical power meter OPM4. For mode conditioning, at the launch end the studied fibers were wrapped 5 times around a 2.54 mm mandrel. The fiber coils (100 meters) were placed into a pan and the optical signal was zeroed. Then liquid nitrogen was poured into the pan and the added optical loss was determined. After taking the measurements at  $-196^\circ\text{C}$ , the liquid nitrogen was removed from the pan and the fibers were warmed to room temperature. For all the fibers, no residual loss was observed after stabilization at room temperature.

Figure 14 displays the obtained results. Three coatings display the highest attenuation at low temperatures: DA-5, SA-1 and Hyb. For a few fibers (DA-1, DA-6 and DA7), the observed added loss is very low.



**Figure 14. Added optical loss for fibers immersed in liquid nitrogen at  $-196^\circ\text{C}$**

## 5. Discussion

Different applications and types of optical fibers dictate different requirements for their coatings. Based on the above study and knowing the environmental conditions for the end use of the fiber, it is now possible to select an optimum coating for it. In Table 3 we tried to summarize the deficiencies of different coatings observed in the study. It can be seen that none of the coatings displayed the best performance in all the testing. Telecom-grade coatings DA-1 and DA-2 were found to be the best at room and lower temperatures, however they are the first to fail at harsh conditions. In contrast, the coatings that perform well at elevated temperatures (DA-3, DA-5, SiHTA-1, SiHTA-2, SA-2, SA-3 and SA-4) are less competitive at ambient conditions.

**Table 3. Major deficiencies of the trialed coatings**

Fiber ID	Room & low temperatures	High temperatures
DA-1	-	Degrades in $\text{N}_2$ and air
DA-2	-	Degrades in $\text{N}_2$ and air
DA-3	Easy to delaminate	Degrades in $\text{N}_2$
DA-4	Low $n_d$ -value	Degrades in air
DA-5	Low-T loss	-
DA-6	Low $n_d$ -value	Degrades in air
DA-7	Low $n_d$ -value	Degrades in air
DA-8	Low fiber strength	Swells in gel, degrades in $\text{N}_2$ & air
DA-9	Easy to delaminate, low-T loss	Swells in gel, degrades in $\text{N}_2$ & air
SiHTA-1	Microbend sensitive	-
SiHTA-2	Microbend sensitive	-
SA-1	Low-T loss	Strength loss at high %RH
SA-2	Microbend sensitive	Strength loss at high %RH
SA-3	Microbend sensitive	Strength loss at high %RH
SA-4	Hard to strip, microbend sensitive	-
Hyb	Low-T loss, microbend sensitive	Coating cracks

Telecom-grade optical fibers utilize a dual coating system, the primary component being more easily degraded than the secondary one. Therefore the thermal stability of the whole fiber is limited mostly by thermal stability of its primary coating. The latter can be improved by either increasing the degree of crosslinking [15] or by utilizing a material with an entirely different chemistry. The higher degree of crosslinking strategy is employed in monocoats, which can be interpreted as “dual coats with identical primary and secondary layers.” Alternatively, silicone can be utilized as an entirely different material for the primary coating. Indeed, per our TGA lifetime criterion, the coatings with the highest thermal stability are SiHTA-1 and SA-2 (and their versions with added adhesion

promoter, SiHTA-2 and SA-3). Note that SA-2 monocoat does not contain urethane, which is known to be a relatively weak link at high temperatures. A clear advantage of dual coating systems is lower microbend sensitivity. In addition, the use of silicone makes the fiber more stable to moisture. Thus, in a combination of properties, SiHTA-1 (or SiHTA-2) coating outperforms SA-2 (or SA-3).

The upper use temperature for those coatings is about 130°C, assuming 20-year continuous use and 25% weight loss failure criterion (Figure 8). This is ~45°C higher than similar values obtained for a “telecom-grade” coating, DA-1. It looks that for acrylate-based formulations we can hardly expect improvements better than 45°C. Obviously, for shorter times, the same coatings can be used at higher temperatures, in accordance with Figure 7.

Another important factor is the environment in which the fiber is being used. In this work we aged fibers in air, nitrogen, steam and under high pressure in hot water or hot cable gel. Each new environment provides a different challenge to the coating system and reveals their positive and negative features. As we see from the data, the coatings showing the best performance in air atmosphere are not necessarily the best in humid and/or water environments and vice versa.

## 6. Conclusions

This study proves once again that telecom-grade optical fiber coatings are optimized for use at temperatures around 23 - 85°C or lower but tend to fail at elevated temperatures and/or other harsh conditions. Silicone-acrylate dual coatings SiHTA-1 & SiHTA-2 display the best performance overall at elevated temperatures and exhibit high resistance to water. Second in performance are single coats SA-2 and SA-3 that sustain well the high temperatures but are inferior in moisture resistance. Per our TGA lifetime criterion, there is a limitation for the upper use temperature of acrylate-based coatings: even the best coating cannot be continuously (20Y) used above 130°C. At shorter use times however, the coated fibers can survive much higher temperatures.

## 7. Acknowledgments

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