Optical fibers with dual coatings for high-temperature applications

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ABSTRACT

We describe a new optical fiber coating, comprising layers of UV-curable silicone and high-temperature acrylate, with and without hermetic carbon. Optical and mechanical properties of graded index 50/125 μm multimode fibers drawn with the new coating are examined. The new coatings display superior thermal stability in comparison with conventional dual acrylate coatings.

Keywords: Optical fiber, thermal stability, carbon coating, silicone coating, high-temperature acrylate

1. INTRODUCTION

Thermal stability is an important property of optical fibers used in specialty applications, including oil wells, aerospace, medical, military and others. Most conventional optical fibers have evolved to use UV-curable dual acrylate coatings, which have a nominal upper use temperature of ~85°C. For higher-temperature applications, thermally cured coatings such as RTV silicones or polyimides are commonly used. Although more heat resistant, thermally cured coatings can be difficult to strip for fiber termination. Also, their raw materials are more costly and their manufacturing processes are more complicated, involving solvent removal or control of limited pot life. They have slower cure rates, requiring lower fiber draw speeds, which increases cost. An ideal coating for specialty optical fibers should comprise advantages of conventional dual acrylate coatings with higher thermal stability.

Recently, we developed a dual-acrylate coating system with improved high-temperature properties.[1] In the present study we explore a different dual coating system, containing a UV-curable silicone primary and a high-temperature acrylate secondary coating. This coating is applied over a hermetic carbon layer, which is included for improving the fiber reliability and for preventing diffusion of hydrogen into the fiber core.

2. FIBER DESIGN

The fiber design was selected based on potential use of the new coated fibers for Distributed Temperature Sensing. We selected the following base fiber: a phosphorous-free 50 μm multimode, graded-index core with 125 μm silica cladding. Various dual coating systems were tested as follows: "regular" dual acrylate (**RA**), a dual acrylate with improved heat resistance (**HTA**), a dual system with a UV-curable silicone primary coating and a high-temperature acrylate secondary coating (**SiHTA**). The primary and secondary coating diameters were 190 and 250 μm, respectively. When the coating system included an additional 400 Å carbon layer, these systems were denoted, respectively as **CRA**, **CHTA**, and **CSiHTA**.

For comparison, we also collected fibers with monocoats. Regular acrylate secondary and high-temperature acrylate secondary coatings were used as the monocoats. Those fibers are referred to as **RA-Sec** and **HTA-Sec**, respectively. The coating diameter for those fibers was 250 μm.

3. OPTICAL AND MECHANICAL PROPERTIES OF "AS-DRAWN" FIBERS

Spectral attenuation, numerical aperture and bandwidth were determined for the as-drawn (unconditioned) fibers in accordance with appropriate FOTPs. The fiber strength was characterized using a two-point bend tester at a strain rate of 4%/min. Table 1 compares the obtained results. No significant differences are observed between the optical parameters of the fibers. As expected,[2] an introduction of the carbon coating reduces the median fiber strength. However, introducing the carbon coating improves the fatigue properties of the fiber. Thus, the determined *n*-value is above 100 for the carbon-coated fibers under study, while without the carbon, the *n*-values were in the range 20 – 35. We also see that the fiber strength is not affected by the type of the primary coating.

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Table 1. Optical and mechanical parameters of 50/125/250 μm GI MM fibers with different coatings

Property	Target spec	RA	CRA	HTA	CSiHTA
Attenuation @ 850 nm (dB/km)	≤ 3.0	2.3	2.3	2.4	2.4
Attenuation @ 1300 nm (dB/km)	≤ 1.5	0.50	0.45	0.70	0.60
Numerical aperture	0.200 ± 0.015	0.20	0.20	0.20	0.20
Bandwidth @ 1300 nm (MHz-km)	≥400	750	930	580	1400
Fiber strength (2-point bend, median, GPa)	>3.8	5.9	3.9	5.8	3.8
Weibull slope (2-point bend)	-	19	10	14	36

4. STRIP FORCE TESTING

Coating strip force was measured using an MTS Sintech 5/G tensile bench fitted with a strip tool (0.013″ guide tube, 0.0063″ 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 target strip force value was in the range 1 – 9 N. It can be seen that the silicone/ acrylate dual coat can be stripped off of the fiber with ease. For comparison, removal of monocoats requires a much higher mechanical force. In those cases, chemical removal of the coating is preferable.

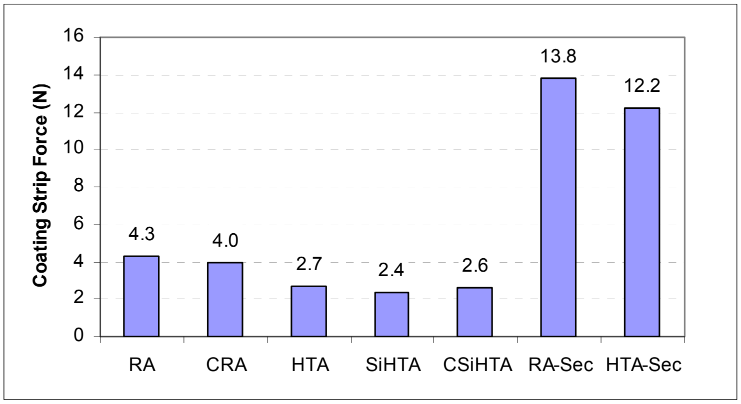

Figure 1. Coating strip force data

5. ASSEMBLY ROBUSTNESS

To evaluate robustness of the fiber/connector assemblies, the following experiments were performed. The fiber coating was stripped off, and the carbon-coated cladding was wiped with alcohol. Then, epoxy/polish SMA connectors were attached to the fibers. For consistency, the bond length was controlled to be the same for all the specimens. Only carbon-coated fibers were studied. For each fiber type, 5 assemblies were made with connectors on both sides. For CRA fiber, the obtained retention force magnitudes fell in the range 26 – 30 N, while for CSiHTA fibers, they were in the range 16 – 27 N. The determined values are comparable to each other and significantly higher than the target specification minimum of 6.7 N (1.5 lbs).

6. THERMAL STABILITY

Described in detail elsewhere,[3] the concept of “thermal stability” implies the upper use temperature at which a certain fiber can be used during a certain (pre-determined) time. Or, vice versa, it shows how long the fiber can be used at a certain (pre-determined) temperature before it fails. At elevated temperatures, different failure modes of the fiber can be observed, and depending on the fiber application, certain modes may be more important than the others. Thermal stability of different coating systems can be roughly ranked using an approach developed previously.[3] The approach begins with defining the failure criterion, which fully depends on specifics of the fiber application. Next a thermogravimetric analysis (TGA) of the coated fiber is conducted and the fiber failure is correlated with the coating weight loss. Then, the appropriate time-temperature range for the fiber application can be estimated by extrapolations of the TGA data.

Figure 2 shows the failure weight loss-time-temperature diagram obtained for SiHTA coating in air. The “lifetime” is shown as a function of two variables: failure weight loss and use temperature. Thus, if we assume that the fiber fails at 10% coating weight loss, such fiber can sustain 20 years at 125°C. It can also be used at higher temperatures, but for shorter times. For example, this fiber will sustain 1 year at 154°C and 1 month at 182°C, again based on the 10% weight loss criterion. If the failure criterion is different, say 50% weight loss, then the use temperatures will shift to higher magnitudes: 20 years at 148°C, 1 year at 183°C and 1 month at 217°C. With a diagram such as shown in Figure 2, one can easily estimate the highest operation temperature (when the failure criterion and the use time are known) or the “lifetime” of the fiber at the known use temperature.

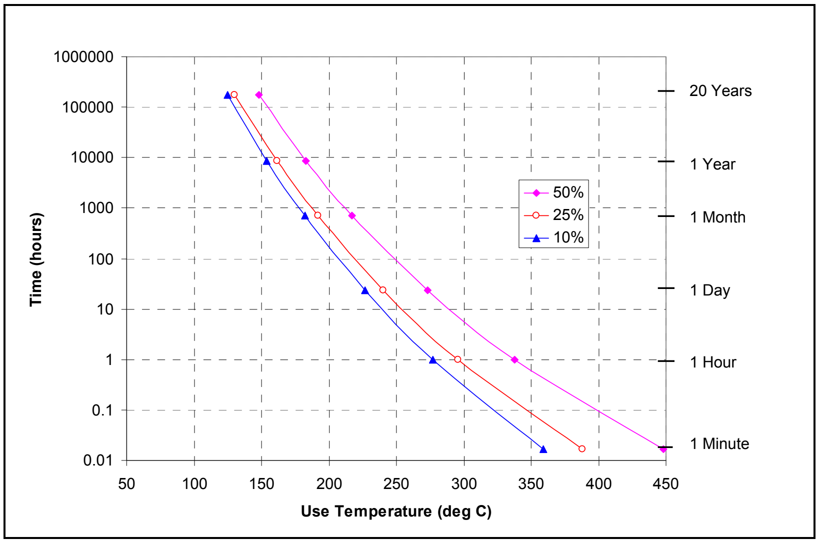

Figure 2. Weight loss-time-temperature diagrams for SiHTA coating in air

Figure 3 compares the upper use temperatures of different fiber coatings in air, obtained assuming 25% wt. loss criterion. It can be seen that SiHTA coating displays superior thermal stability in comparison with acrylate coatings, including the monocoats. SiHTA provides up to 50°C improvement in the use temperature in comparison with regular dual acrylate coatings (including those studied previously).[1] Thus on a thermal stability scale, SiHTA appears intermediate between regular acrylates and polyimide (the latter is also represented in Figure 3).

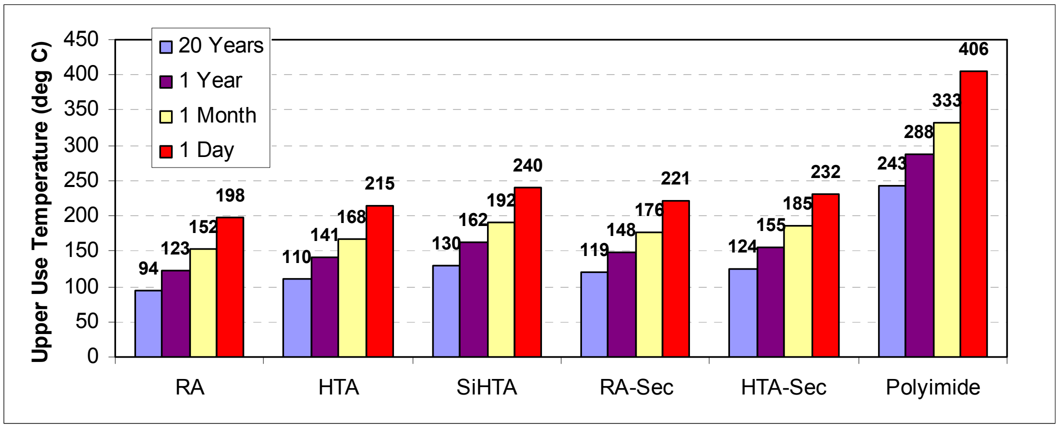

Figure 3. Upper use temperatures for different coatings in air, based on 25% weight loss criterion

7. THERMAL AGING AND CYCLING

To test optical and mechanical stability of the fibers, we applied two accelerated aging conditions: 48 hours at 200°C in air, and 48 hours at 250°C in air. In addition, a thermal cycling experiment was performed. The temperature was cycled five times between -65°C and +175°C with 90 min dwelling at the highest and lowest temperatures. Table 2 shows that the 200°C thermal aging followed by the thermal cycling does not affect the ultimate fiber attenuation.

Table 2. Optical attenuation of aged and thermally cycled fibers, measured at room temperature (dB/km)

Fiber/coating	Wavelength	As-drawn	Aged (200°C, 48 hrs)	Aged (200°C, 48 hrs) + thermally cycled
CRA	850 nm	2.3	2.4	2.3
	1300 nm	0.45	0.54	0.49
CSiHTA	850 nm	2.3	2.3	2.3
	1300 nm	0.49	0.48	0.47

The fiber strength, determined before and after aging, is shown in Figure 4. We observe no strength degradation for the fibers with silicone primary coating.

In addition, the fibers were soaked 46 hours in 85°C water. Specimens were about 1 meter long, and both fiber ends were kept outside the water bath. No strength degradation was observed for any of the studied fibers.

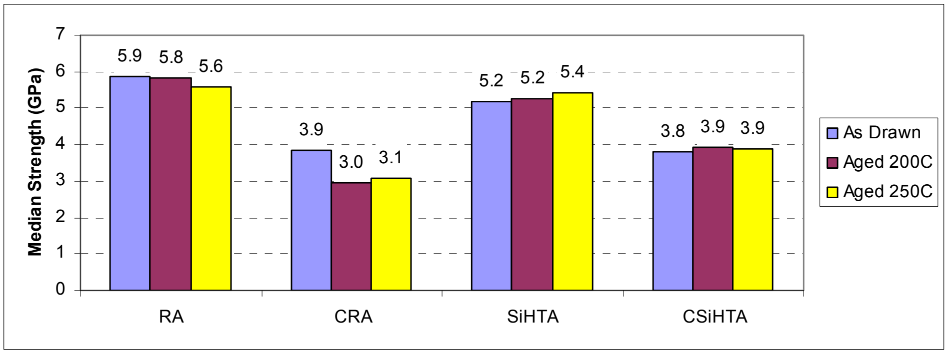

Figure 4. Strength of the aged and unaged fibers (2-point bent test data)

Fiber assembly performance can degrade significantly due to thermally induced shrinkage of the coating. The axial coating shrinkage developed upon thermal aging was studied in the following way. One-meter fiber specimens were cut by sharp razor blades. The initial pistoning of the glass vs. the coating was measured with a microscope. The specimens were placed in an aluminum tray and aged 48 hours at 200°C. Then, the pistoning of the glass vs. the coating was determined again and the developed shrinkage was calculated. For each fiber type, five specimens were tested from both sides. The obtained average shrinkage magnitudes (Figure 5) are expressed in percentages with respect to the specimen length (1 m). We see that the silicone/acrylate developed significantly lower shrinkage, which is an advantage of this coating system.

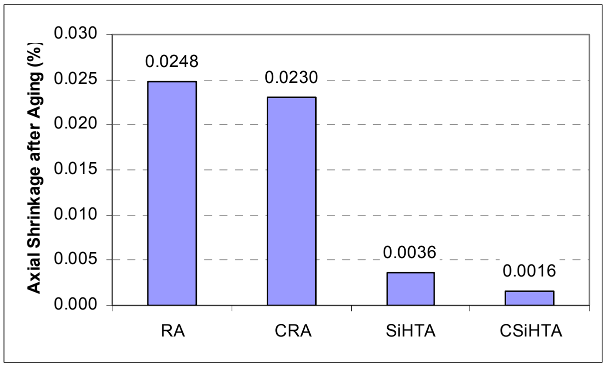

Figure 5. Axial shrinkage of the coatings induced by 48 hrs aging at 200°C.

8. CONCLUSIONS

The developed silicone/high-temperature acrylate coating displays superior thermal stability and lower thermally induced axial shrinkage in comparison with regular acrylate coatings. Other optical and mechanical properties are not sacrificed with the new coating. The new coatings can be used atop a hermetic carbon layer, to support the needs of fibers used in Distributed Temperature Sensing.

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