Heat-Resistant Thin Optical Fiber for Sensing in High-Temperature Environments

Kazuyuki SOHMA* and Tomoyuki HATTORI

We have developed a new heat-resistant optical fiber coated with ultraviolet (UV)-curable silicone resins. Its diameter (250 μm) is thinner than that of conventional heat-resistant optical fiber coated with thermosetting silicone resins and a poly(tetrafluoroethylene-co-perfluoropropylvinylether) (PFA) outer sheath. While showing excellent heat resistance at 200˚C, it has microbending resistance and dynamic fatigue properties superior to those of conventional heat-resistant optical fiber. These features enable this new optical fiber to be used for high density cabling and optical fiber sensing in high-temperature environments up to 200˚C.

Keywords: ultraviolet-curable silicone resin, heat-resistant optical fiber, optical fiber sensing

1. Introduction

The importance of optical fiber sensing has been recognized and it has been adopted for various applications in recent years(1). Optical fiber sensing enables measurement of various physical parameters, such as temperature, strain, displacement, vibration, and pressure. In these applications, temperature measurement plays a significant role in maintenance, safety and production, and is utilized in such uses as fire detection, power cable temperature control, and the monitoring of pipelines, oil wells and gas wells(2). For these purposes, optical fibers are used over a long period in high-temperature environments, and accordingly must be coated with heat-resistant materials. The optical fibers are often inserted into a metal tube in cable form for installation at the temperature measurement site. An example of utilizing such heat-resistant optical fibers and a metal tube cable for optical fiber sensing is shown in **Fig. 1**. In the figure, one of the two heat-resistant optical fibers is for temperature distribution measurement along the length of the cable; the other optical fiber is for signal transmission with optical sensors for multipoint measurements of pressures or strains, etc(1). Techniques used to measure temperature distribution include the detection of Raman scattering light(2)-(5) and of Brillouin scattering light(6).

A thinner cable is desirable to reduce the space used by the laid cable and to maintain good handling characteristics. On the other hand, high density cabling (increased number of optical fibers in the metal tube) and use at temperatures approaching 200˚C may be desired. In these cases, thin optical fibers with 200˚C heat resistance are required.

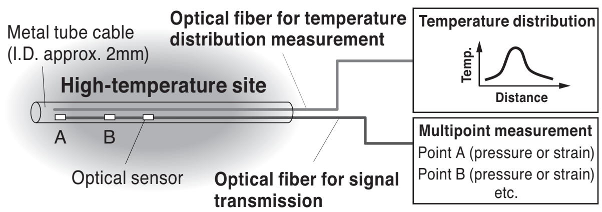


Fig. 1. Optical fiber sensing in high-temperature environment using heat-resistant optical fibers

Table 1 shows two kinds of typical heat-resistant optical fiber currently on the market and a conventional optical fiber, while **Table 2** shows the symbols used in this paper. One heat-resistant optical fiber is PIF, which has a coating of polyimide (PI) resin; the other, TSSF, has two coating layers of thermosetting silicone (TSS) resins and an outer sheath of poly(tetrafluoroethylene-co-perfluoropropylvinylether) (PFA)(7). In **Table 1**, the conventional optical fiber coated with ultraviolet-curable urethane acrylate (UV-UA) resins, referred to below as simply UAF, is also shown for refer-

Table 1. Structures and characteristics of two typical heat-resistant optical fibers and one conventional optical fiber

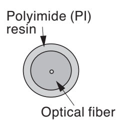
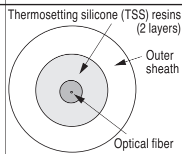
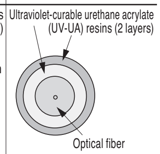
	Polyimide resin coated optical fiber (PIF)	Thermosetting silicone resin coated optical fiber (TSSF)	Ultraviolet-curable urethane acrylate resin coated optical fiber (UAF)
Structure			
Upper temperature limit (typical) [°C]	300	200	85
Outer diameter [μm]	155-162	400-700	250
High density cabling (increased number of optical fibers)	available	not available	available
Handling characteristics	poor	good	good
Lateral pressure resistance	very low	high	high
Productivity	very low (low line speed)	low (requires extrusion process)	high

Table 2. Symbols used in this paper

Type of resin	Symbol	Type of optical fiber	Symbol
Polyimide resin	PI resin	PI resin coated optical fiber	PIF
Thermosetting silicone resin	TSS resin	TSS resin coated optical fiber (with PFA outer sheath)	TSSF
Poly(tetrafluoroethylene-co-perfluoropropylvinylether)	PFA		
Ultraviolet-curable urethane acrylate resin	UV-UA resin	Conventional optical fiber (coated with UV-UA resin)	UAF
Ultraviolet-curable silicone resin	UVS resin	UVS resin coated optical fiber	UVSF

ence. The PIF has a very low lateral pressure resistance, which can result in increased attenuation due to high density cabling. It also has poor handling characteristics because of its small outer diameter and very low productivity. The TSSF has a large outer diameter, and therefore, it is not readily used for high density cabling. The conventional optical fiber has low heat resistance, i.e. a low upper temperature limit for operation, and therefore, is not suitable for use in high-temperature environments. In other words, it is difficult for these three optical fibers to satisfy all the requirements raised above and important characteristics for practical use.

The purpose of the development reported here is to create a heat-resistant optical fiber that satisfies the five requirements of 1) sufficient heat resistance to allow long-term use at temperatures up to 200˚C, 2) small outer diameter to allow high density cabling, 3) high lateral pressure resistance to allow high density cabling without increased attenuation, 4) good handling characteristics, and 5) high productivity. To achieve these requirements, we have developed a heat-resistant optical fiber that has the same diameter as conventional optical fiber (250 µm) and the same or better heat resistance as TSSF at 200˚C, as well as other excellent characteristics by applying newly developed ultraviolet-curable silicone (UVS) resins for coating materials.

2. UVS Resin Design and Properties

2-1 Resin design

The UVS resins that we have developed comprise mainly component A, component B and a photo-initiator. Components A and B greatly affect the heat resistance, surface tackiness and Young's modulus. Some restrictions also exist for optical fiber coating materials. For example, when a tacky resin is applied as a secondary coating (outer layer) material, the manufacturability of the metal tube cable decreases, and therefore a low tackiness is desired for the coating material. It is also necessary to apply resins with a higher Young's modulus as the secondary coating rather than the primary coating (inner layer). The characteristic referred to here as "heat resistance" was evaluated by forming the resin as a sheet coating on a glass substrate, subjecting it to a temperature of 200˚C for 60 days, then observing any cracking. The resin sheet was formed by spin coating followed by irradiation with UV light at 1000 mJ/cm^2 under a nitrogen atmosphere and heating for 10 min at 120˚C in a thermostatic oven. The sheet thickness was about 30 µm. The surface tackiness was evaluated by a touch test done prior to the thermal degradation process described above. The measurement method for the Young's modulus is explained in section 2.2.

According to **Table 3**, composition F-3, which contains a relatively large amount of component B, has lower heat resistance, but no surface tackiness. Composition F-1, which contains a large proportion of component A, has higher heat resistance but also higher surface tackiness. From these findings, we can recognize that there is a trade-off between the two characteristics. Adjusting the composition of the two components also changes the Young's modulus; a relatively large amount of component A tends to decrease the Young's modulus. Consequently, compositions F-1 and F-2, which have high heat resistance, were adopted as coating materials. F-2, which has low surface tackiness and a large Young's modulus, was selected as the secondary coating material, and F-1, which has a low Young's modulus, was selected as the primary coating.

Table 3. UVS resin compositions and their characteristics

		F-1	F-2	F-3
Composition	Component A	more	middle	less
	Component B	less	middle	more
Heat resistance (200˚C)		high	high	low
Surface tackiness		high	very low	none
Young's modulus (relative value)		1	2.7	5.2

The relative Young's modulus of UVS and TSS resins, normalized by the Young's modulus of the TSS resin for primary coating, are compared in **Table 4**. The relative Young's modulus of the UVS resin for the primary coating was 19, which is very large compared with that of the TSS resin for primary coating. Although this is a cause of concern for degradation of UVSF's lateral pressure resistance, we confirmed that this is not a problem, as will be described later in this paper.

Table 4. Relative Young's modulus of coating materials for UVSF (UVS resins) and TSSF (TSS resins)

	UVS resin	TSS resin
Primary	19	1
Secondary	52	5

2-2 Conditions for fabricating the evaluation samples

Together with the UVS resins that we have developed, we evaluated the properties of TSS resins and UV-UA resins. The properties evaluated were thermogravimetric analysis (TGA) and the effects of exposure at 200˚C on the Young's modulus, breaking strength, and the breaking elongation. The sample resin sheets used for evaluation were fabricated by spin coating onto a substrate and then curing each resin under different conditions. For curing the UVS resins and the UV-UA resins, we used a conveyor-type UV irradiator to expose the resins to UV light at 500 mJ/cm^2 under a nitrogen atmosphere. Only the UVS resins were subjected to heat treatment at 120˚C for 30 min after the curing. The TSS resins were cured by heat treatment for 30 min at 150˚C in a thermostatic oven. The spin coating conditions were adjusted to produce a sheet of about 100 µm in thickness. Fabricated resin sheets were peeled off from the substrates and used as evaluation samples.

To measure the Young's modulus, breaking strength, and breaking elongation, we formed the resin sheets into JIS No. 2 dumbbell shaped samples.

2-3 Results of resin property evaluation

The TGA results for the fabricated resin sheets are presented in **Fig. 2** for the primary coating materials, and in **Fig. 3** for the secondary coating materials. We measured them at the temperature increase rate of 10˚C/min in air. The developed UVS resins show weight loss rates lower than the UV-UA resins and about the same as the TSS resins. The 5% and 10% weight loss temperatures obtained from the TGA results are summarized in **Table 5**. The UVS resins have 5% and 10% weight loss temperatures higher than UV-UA resins and about the same as TSS resins.

Table 5. 5% and 10% weight loss temperatures obtained from TGA

	UVS resin		TSS resin		UV-UA resin	
	P	S	P	S	P	S
5% weight loss temperature [˚C]	330	326	323	374	275	278
10% weight loss temperature [˚C]	400	377	362	428	300	301

Figure 4 presents the results for the resin properties of the Young's modulus, breaking strength, and breaking elongation after 200˚C degradation testing. The results presented are for the 14th day for the UVS resins and TSS resins and the 7th day for the UV-UA resins. Although

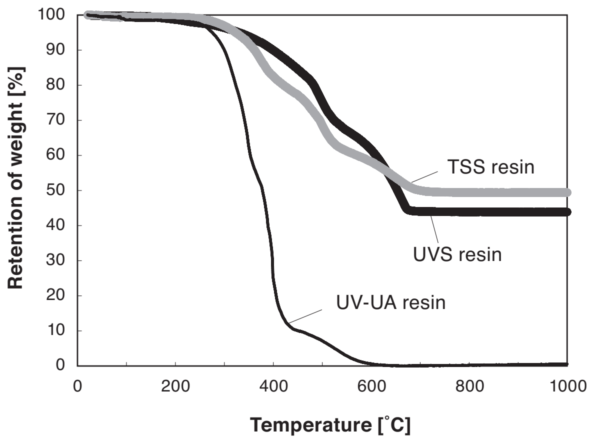


Fig. 2. TGA results of UVS, TSS and UV-UA resins for primary coating (measured at the temperature increase rate of 10°C /min in air)

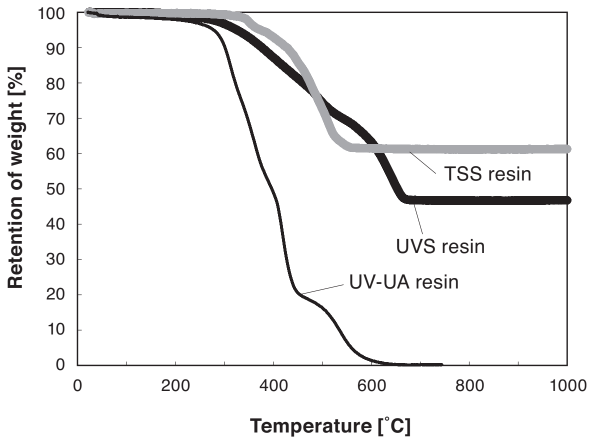


Fig. 3. TGA results of UVS, TSS and UV-UA resins for secondary coating (measured at the temperature increase rate of 10°C /min in air)

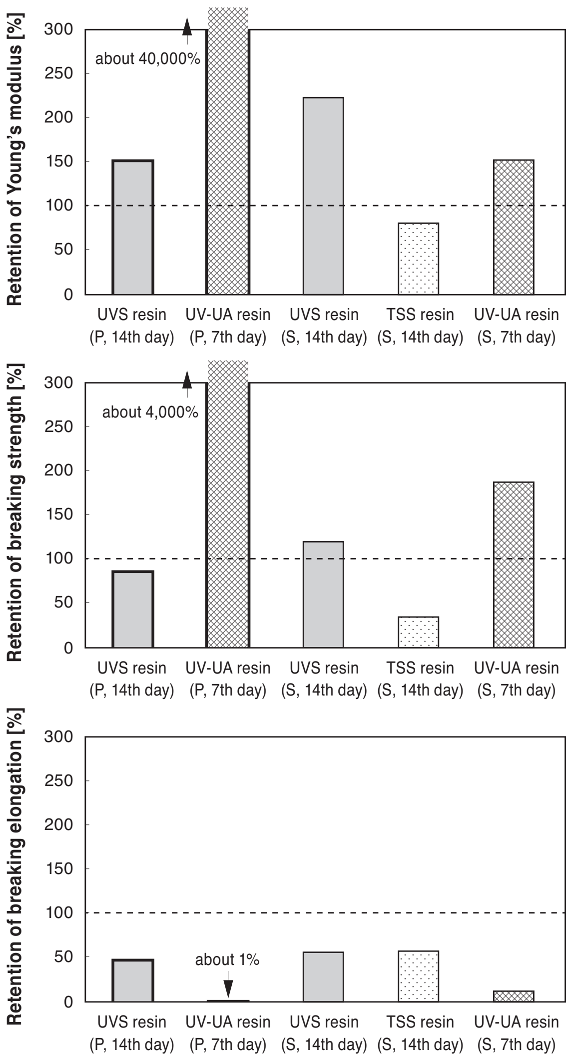


Fig. 4. Change in properties of coating materials due to 200°C degradation. (a) Young's modulus, (b) breaking strength, (c) breaking elongation. (P: primary, S: secondary)

some changes were exhibited in the properties of the UVS resins, the retention of all properties remained close to those for the TSS resins. The most dramatic decline was shown in the primary coating material of UV-UA resins. The Young's modulus and the breaking strength increased greatly, and the retention of breaking elongation decreased greatly to about 1%. The reason for these dramatic changes is considered to be that the UV-UA resins have urethane bonds and other organic compounds with low heat resistance, resulting in rapid oxidation or thermal decomposition at 200˚C.

The measurement results for the resin properties presented above show that our UVS resins have a much higher heat resistance than the UV-UA resins, and about the same level as the TSS resins.

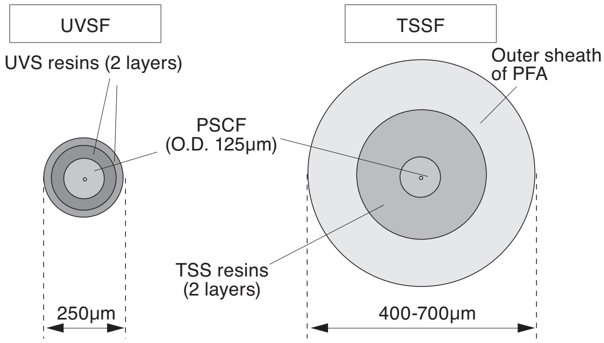


Fig. 5. Structures of UVSF and TSSF

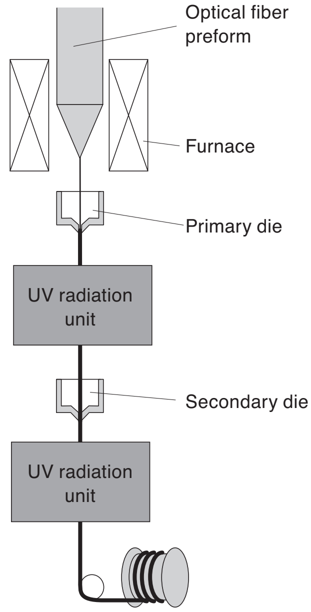


Fig. 6. Optical fiber drawing machine

3. Prototype Optical Fibers and Evaluation Conditions

We fabricated a prototype optical fiber using the UVS resins for primary and secondary coatings described in the previous section. The structures of UVSF and TSSF are illustrated in **Fig. 5**. The outer diameter of UVSF is 250 μm, the same as that of the conventional optical fiber. We have thus reduced the cross-sectional area to 12-38% of that of the TSSF for high density cabling while maintaining good handling characteristics.

The prototype UVSF is a single-mode optical fiber with a pure silica core and a fluorine-doped silica cladding [pure silica core optical fiber (PSCF)], which features lower attenuation and higher resistance to hydrogen and radiation than optical fibers with germanium-doped silica cores. It is therefore well-suited to optical fiber sensing in both harsh and high-temperature environments. The prototype UVSF was fabricated with an optical fiber drawing machine that incorporates a tandem-die coating system (**Fig. 6**). First, the optical fiber preform is melted in furnaces and drawn to a diameter of 125 μm. Next, the optical fiber is coated in two layers with the newly developed UVS resins. The coated optical fiber is then wound onto a bobbin. The UVSF was wound onto a metal bobbin and set in a thermostatic oven at 120˚C for 30 min. The TSSF is subjected to a PFA sheathing process after the drawing process, but the extrusion process for PFA outer sheath was eliminated for UVSF to improve productivity.

We tested the prototype UVSF described above together with PIF and TSSF as conventional heat-resistant optical fibers as well as the conventional optical fiber, UAF (all of them were PSCF). The four types of tests conducted and their standard specifications are listed in **Table 6**. The lateral pressure resistance test is based on the International Electrotechnical Commission (IEC) 60793-1-C3B and is performed by measuring the attenuation at a wavelength of 1.55 μm as the sample optical fiber is wound under the prescribed tension onto a bobbin with a metal mesh. After that, the same optical fiber is evaluated in a loose coil. The lateral pressure resistance is evaluated from the difference in the attenuation values measured under these two conditions.

The dynamic fatigue property test was conducted according to IEC 60793-1-B7A. The dynamic fatigue coefficient (n_D) was obtained from the median values of optical fiber tensile strength measured at four strain rates with a gauge length of 500 mm and N=15 for each rate.

Table 6. Tests conducted and associated specifications

Test items	Specifications
Lateral pressure resistance	IEC 60793-1-C3B
Dynamic fatigue property	IEC 60793-1-B7A
Attenuation (OTDR)	IEC 60793-1-C1C
Optical fiber tensile strength	IEC 60793-1-B2A

The attenuation was measured after degradation by inserting a loosely coiled optical fiber into a thermostatic oven heated to 200˚C. This was done in an atmosphere of air, no particular control of the humidity in and around the oven. The ends of the optical fiber were led out of the oven before the measurements and the attenuation was measured at a wavelength of 1.55 μm by an optical time-domain reflectometer (OTDR) when the specified number of degradation days had passed (IEC 60793-1-C1C). At the time of measurement, the temperature of the optical fiber was maintained at 200˚C. The attenuation change was taken to be the difference between the values measured at each number of degradation days and the value measured at room temperature before the degradation process had begun. The temporal change in that value was then studied.

In the optical fiber tensile strength tests, the optical fiber samples were degraded in a thermostatic oven at 200˚C. After the specified number of days of degradation, the samples were removed from the oven and measured at room temperature according to IEC 60793-1-B2A. The optical fiber tensile strength was taken to be the median values measured at the gauge length of 500 mm, strain rate of 25 mm/min, and N=15.

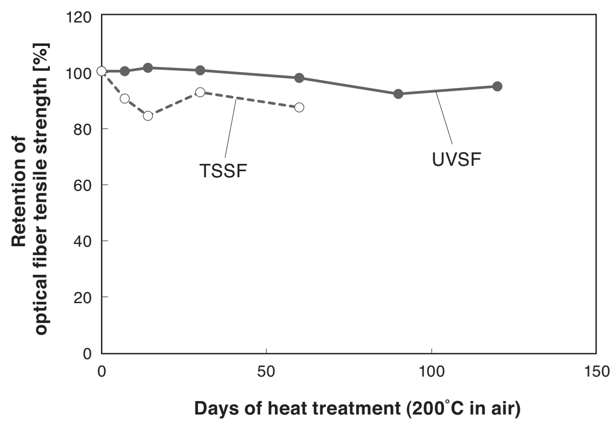


Fig. 7. Change in optical fiber tensile strength due to 200˚C degradation

4. Results of Prototype Optical Fiber Evaluation

The results of the dynamic fatigue property and lateral pressure resistance tests for the UVSF and the other optical fibers are presented in **Table 7**. The dynamic fatigue coefficient (n_D) for the UVSF was 25, the same as or better than the results for the other optical fibers.

Table 7. Results of dynamic fatigue property and lateral pressure resistance tests

Type of optical fiber	UVSF	PIF	TSSF	UAF
Dynamic fatigue coefficient (n_D)	25	24	23	21
Lateral pressure resistance	high	very low	high	high

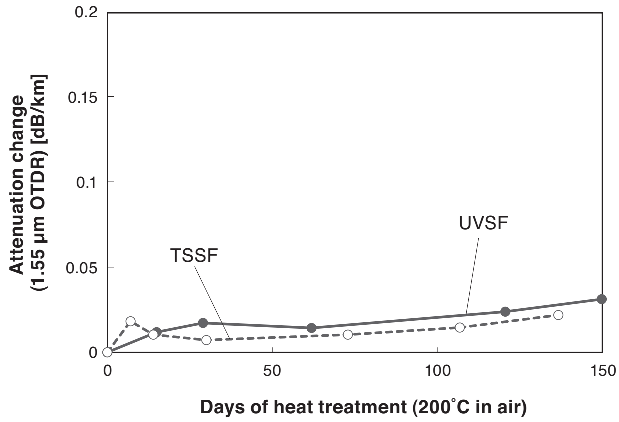


Fig. 8. Attenuation change due to 200˚C degradation

On the other hand, the results of the lateral pressure resistance tests showed that a large attenuation occurs when the PIF is wound onto the bobbin with metal mesh, indicating low lateral pressure resistance. Possible reasons for this result are that the thin PI resin coating (15 to 20 μm) has a small buffering effect against lateral pressure, or that microbending usually occurs because of the low flexural rigidity of the optical fiber. The UVSF has about the same characteristics as the other optical fibers and exhibits better lateral pressure resistance than the PIF.

Figure 7 presents the retentions of tensile strength for the optical fibers degraded at 200˚C in air, showing measurements for the UVSF and TSSF. The UVSF retained 90% or more of its tensile strength even after 120 days, exhibiting very good results. The initial tensile strength was 59 N and the minimum retention after 120 days was 92%, with a tensile strength of 55 N. For the TSSF, on the other hand, although the initial tensile strength was the same as the UVSF, 59N, the value after 60 days of degradation was 50 N, for a minimum retention of 84%, thus exhibiting a decrease due to degradation.

The attenuation change due to degradation at 200˚C in air is shown in **Fig. 8**. For both types of optical fiber, the attenuation changes increased with the number of degradation days. The increase was 0.03 dB/km after 150 days for the UVSF and 0.02 dB/km after 140 days for the TSSF, thus the attenuation was negligible for practical use.

A 10 m loose coil of UVSF was also produced and subjected to high-temperature degradation for 120 days at 200˚C. The surface of the optical fiber was observed with a microscope. Although some discoloration was observed, there was no cracking, peeling or other abnormalities in the coating.

The results presented above show that the newly developed UVSF has the same or better heat resistance as the TSSF.

5. Result of Metal Tube Cable Fabrication

A metal tube cable was fabricated by inserting four optical fibers into a metal tube with an inner diameter of 2 mm. The structure of the cable is shown in **Fig. 9**.

The attenuation measured with an OTDR showed that

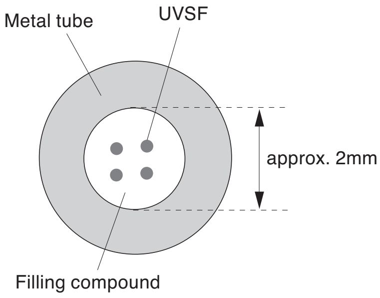


Fig. 9. Fabricated metal tube cable with four UVSFs

there was no difference in attenuation between the metal tube cable and the loosely coiled UVSF. From these results, we conclude that no attenuation due to lateral pressure arises as a result of fabricating the metal tube cable.

6. Conclusions

We have developed UVS resins as coating materials for heat-resistant optical fiber and used them for a prototype optical fiber with a coating diameter of 250 um. This diameter is small enough to enable high density metal tube cabling while maintaining good handling characteristics. The coating has a two-layer structure that provides the optical fiber with the same high lateral pressure resistance as UAF has, yet exhibits no increased attenuation from high density metal tube cabling. The dynamic fatigue property is also as good as UAF, PIF, and TSSF. This new optical fiber also improves productivity by eliminating the PFA sheathing process used in the production of TSSF.

Measurements of optical fiber tensile strength and attenuation changes made after the new optical fiber was subjected to a degradation treatment at 200˚C in air confirmed that those characteristics are at least as good as those of the TSSF and that the heat resistance is superior.

From the results presented here, we conclude that this new heat-resistant optical fiber is effective in high density metal tube cabling and is well-suited to optical fiber sensing under high-temperatures up to 200˚C.

References

(1) Y. Fujii and H. Nakamura, "Optical Fiber Sensing," Plant Engineer, Oct. 2009, pp. 52-57.
(2) H. Kato, "Optical Fiber Sensing—Heat sensing and applications," Plant Engineer, Nov. 2009, pp. 52-57.
(3) G.W. Bibby and J.N. Ross, "Temperature distribution measurement using Raman ratio thermometer," SPIE vol. 566 Fiber Optic and Laser Sensors III, 1985, pp. 249-256.
(4) A.H. Hartog and A.P. Leach, "Distributed temperature sensing in solid-core fibers," Electronics Letters, vol. 21, No. 23, 1985, pp. 1061-1062.
(5) G.P. Lees, A.P. Leach and T.P. Newson, "1.64 μm pulsed source for a distributed optical fibre Raman temperature sensor," Electronics Letters, vol. 32, No. 19, 1996, pp. 1809-1810.
(6) E. Sasaoka, Y. Yamamoto, T. Hayashi, and I. Sakabe, "Brillouin Scattering Optical Fiber Temperature Sensor (TermoGazer™)," SEI Technical Review, No. 170, 2007, pp. 14-18.
(7) M. Shimizu, T. Hattori, K. Tsuneishi, R. Rubino, and D. Norton, Metal-free heat-resistant optical fiber for fiber optic sensing systems in oil wells," Proc. 48th IWCS, 1999, pp. 708-713.

Contributors (The lead author is indicated by an asterisk (*).)

K. SOHMA*

• Assistant Manager
Optical Communications R&D Laboratories
He has been engaged in the development of special optical fibers and the applications since joining Sumitomo Electric in 2001.

T. HATTORI
• Department Manager
Optical Communications R&D Laboratories