TEST REPORT
Test Date: March 8-9, 2016

PEEK

Coated Fiber Evaluation

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ABSTRACT

Zeus Industrial Products partnered with Luna, a leader in fiber optics technology and testing, to design a test regime to evaluate a polyether ether ketone (PEEK) coating for optical fiber. The PEEK was applied using an enhanced coating process developed by Zeus. Testing was done to assess the effects of PEEK coating on signal attenuation which may result from compression stresses and other distortions induced to the fiber due to shrinkage characteristics of the coating. PEEK, known for its ability to provide abrasion, radiation, and temperature resistance when used as a coating, was applied to a standard single-mode Nufern 155 μm polyimide R1550B-P fiber to yield a final outside diameter of 400 μm. Testing was done to compare transmission loss between the PEEK coated optical fiber and an uncoated reference fiber in three test configurations: sub-zero thermal cycling from ambient temperature to -10 °C; elevated thermal cycling from ambient temperature to 240 °C; and testing when inducing small bend radii within the test section of the optical fiber at ambient temperature and 150 °C. For the sub-zero and elevated temperature testing, the results were analyzed for a 250 m coiled section of optical fiber, we found that at ambient temperature the reference fiber displayed 1.14 dB/km attenuation compared to 1.34 dB/km of the PEEK coated fiber. At -10 °C, these fibers exhibited no difference in attenuation. At temperature above ambient, a maximum average attenuation was observed of 1.38 dB/km between the two test fibers. The reference fiber displayed 0.28 dB/km less attenuation than the PEEK coated fiber at temperatures below 150 °C. The PEEK coated fiber, however, displayed improved attenuation at temperatures above 150 °C up to a maximum of 0.37 dB/km less attenuation compared to the reference fiber. At 150 °C, the PEEK coated and reference fibers exhibited similar attenuation. For the small bend radii testing, there was no difference observed in the attenuation between the PEEK coated and reference fibers at 50 mm or 30 mm bend radii at any of the temperatures tested. Notably, at the smallest bend radius tested, 10 mm, the PEEK coated fiber exhibited 1.22 dB/km attenuation which was 0.37 dB/km less than the reference fiber when measured at 150 °C. Taken together, these results show that aside from providing additional protection from physical damage such as abrasion and heat, the Zeus PEEK coated fiber performed at least as well as the uncoated fiber. In some cases, the PEEK coated fiber performed better than the uncoated fiber such as when bent in small bend radii at higher temperatures. The Zeus PEEK coated fiber also did not appear to suffer from compression stresses due to fluctuating temperatures. Thus, PEEK coated optical fibers provide added durability while maintaining a high level of functionality.

INTRODUCTION

PEEK is a well-characterized organic thermoplastic material routinely used in engineering applications due to its physical and mechanical robustness and chemical resistance. PEEK is commonly applied to optical fiber to increase the fiber's durability in harsh environments throughout the medical, aerospace, energy, and civil engineering industries. Significant challenges have been encountered when applying PEEK coating to optical fiber, however, including physical stresses to the fiber as the coating cools below its glass transition temperature. Optical fibers are susceptible to these distortions including compression, bends, and other deformations which negatively affect fiber transmission resulting in signal attenuation.

To evaluate the utility and performance of PEEK coated optical fibers, Zeus conducted preliminary testing comparing PEEK coated fibers and uncoated fiber. Using Zeus' optical time-domain reflectometer (OTDR), initial testing indicated that PEEK coated fiber retained their attenuation characteristics compared to uncoated fiber. To confirm these findings, Zeus collaborated with Luna, an industry leader with unique capabilities in fiber optic technology and testing. Zeus PEEK coated fiber were evaluated using Luna's optical backscatter reflectometer model 4600 (OBR 4600). This instrument uses optical frequency domain reflectometry (OFDR) to determine the optical characteristics of the fiber. The heightened capabilities of the OBR 4600 enable detection of defects not routinely apparent with a typical OTDR. Testing of the PEEK coated fibers with Luna's OBR 4600 revealed deficiencies in the fiber which were not observed during previous testing, particularly for areas containing fiber Bragg gratings. Compressive effects of the PEEK caused arrays to show a deformed grating peak which was not evident with the earlier Zeus OTDR. To address these concerns, Zeus developed an optimized PEEK coating process focused on achieving thermal stability with coated optical fiber.

TEST CONFIGURATION

Testing was designed to evaluate whether decibel (dB) loss (attenuation) was induced to a standard single-mode Nufern 155 µm polyimide R1550B-P fiber suited for Brillouin based distributed temperature and strain sensing following the application of PEEK coating. The PEEK coating was applied to the optical fiber to produce a final outside diameter (OD) of 400 µm. Luna and Zeus engineers performed three modes of testing comparing uncoated and Zeus PEEK coated fiber: low temperature (sub-zero) thermal cycling from ambient temperature to -10 °C; high temperature thermal cycling from ambient temperature to 240 °C; and elevated temperature testing when inducing small bend radii within a test section of the optical fiber. For each test mode, loss in dB over a 250 m test section of the fiber was measured using Luna's OBR 4600.

The Luna OBR 4600 was attached to a 150 m delay line to act as a jumper between the instrument and the test location. A 410 m length of the reference fiber and a 455 m length of PEEK coated fiber were coiled in approximately 0.4 m diameter loops and placed first into a freezer for sub-zero testing and later in a furnace for high-temperature testing. Located with each fiber were two K-type thermocouples to record the temperature inside of the furnace (and thus of the fiber). Each fiber was terminated by crushing the end and coiling the fiber into 5-6 mm diameter loops which were held with Kapton® tape. The tape was used to reduce back-reflection from the end of the fiber to levels that ensured the lowest noise floor and best Rayleigh backscatter signal-to-noise ratio. At each temperature interval each fiber was scanned and the data recorded. These data were evaluated using the OBR software to measure dB loss within the fiber along the 250 m section beginning 50 m from the end of the delay line. The insertion loss (IL) integration width (the region over which the Rayleigh scatter is averaged) was set at 20 m. The reference fiber and the PEEK coated fiber were each tested separately. Reference data was collected before testing for each fiber (Figure 1). [A loss event was observed at 150 m for the PEEK coated sample due to connector loss between the 150 m jumper and the PEEK fiber sample. This loss, however, played no role in subsequent fiber testing (Figures 1 and 2)].

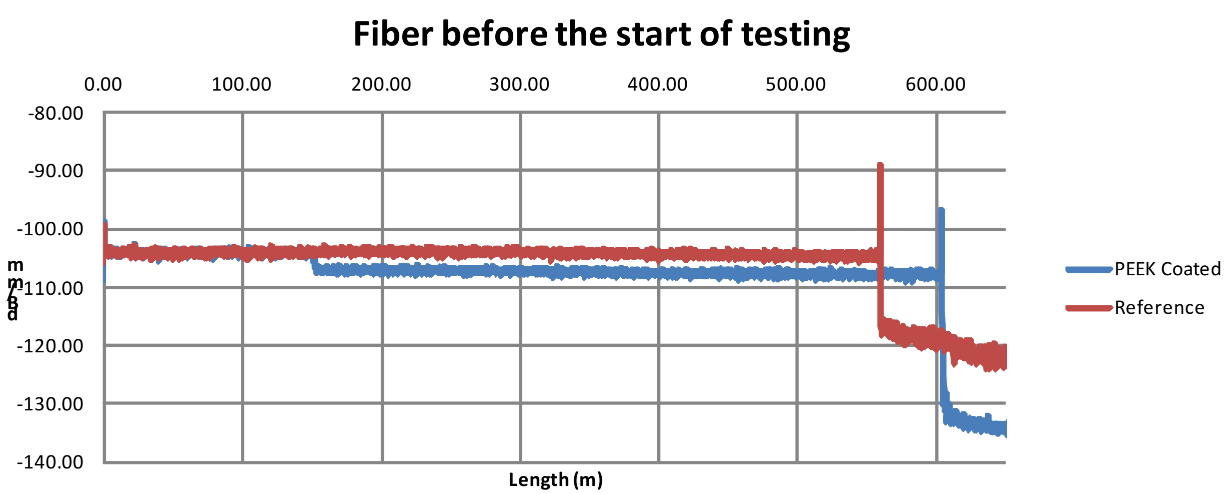

Figure 1: Reference data for the PEEK coated (blue) and reference fiber (red) prior to temperature and bend radii testing.

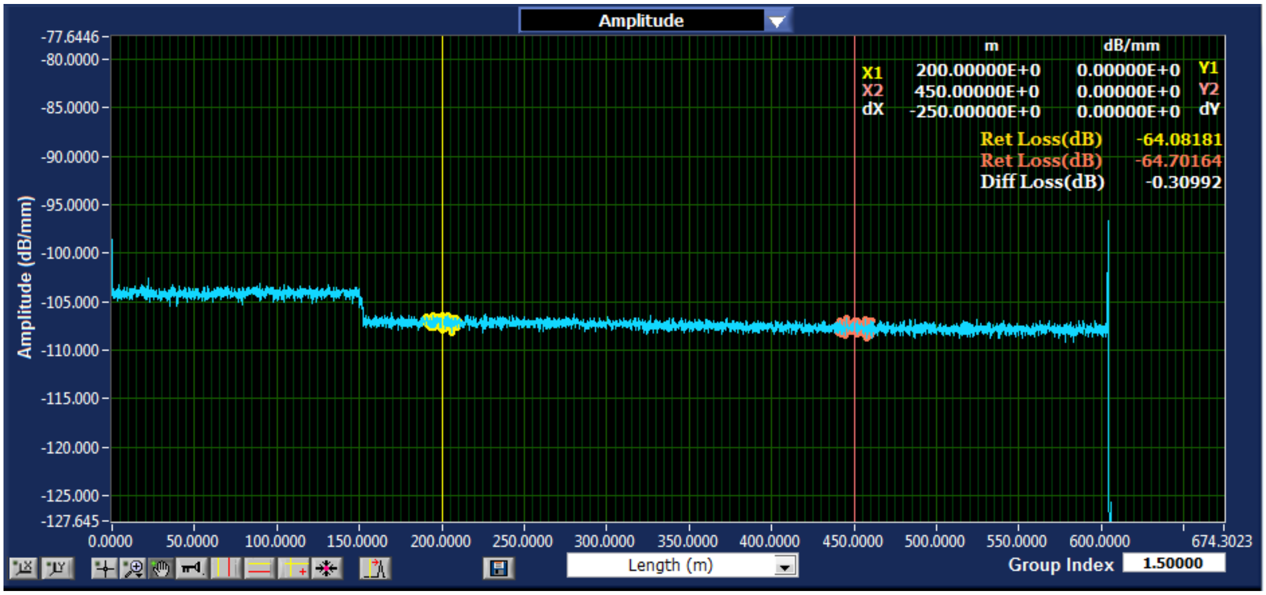

Figure 2: Luna OBR 4600 screen capture depicting the loss measurement at 150m for the PEEK coated fiber before temperature and bend radii testing.

Using Luna's OBR 4600, polarization states for both fibers were measured. The evolution of the polarization states as a function of fiber length appeared to be from a coiled section of fiber as detected by the OBR receiver (Figure 3 and 4). S and P polarization states from fringes showed a period of approximately few meters; this period was consistent with the birefringence beat length induced by coiling the fiber. No inconsistencies regarding differences between the reference and PEEK coated fibers were apparent. This absence of difference observed between the PEEK and reference fibers suggests that minimal, if any, residual strain had been induced by the Zeus PEEK coating process.

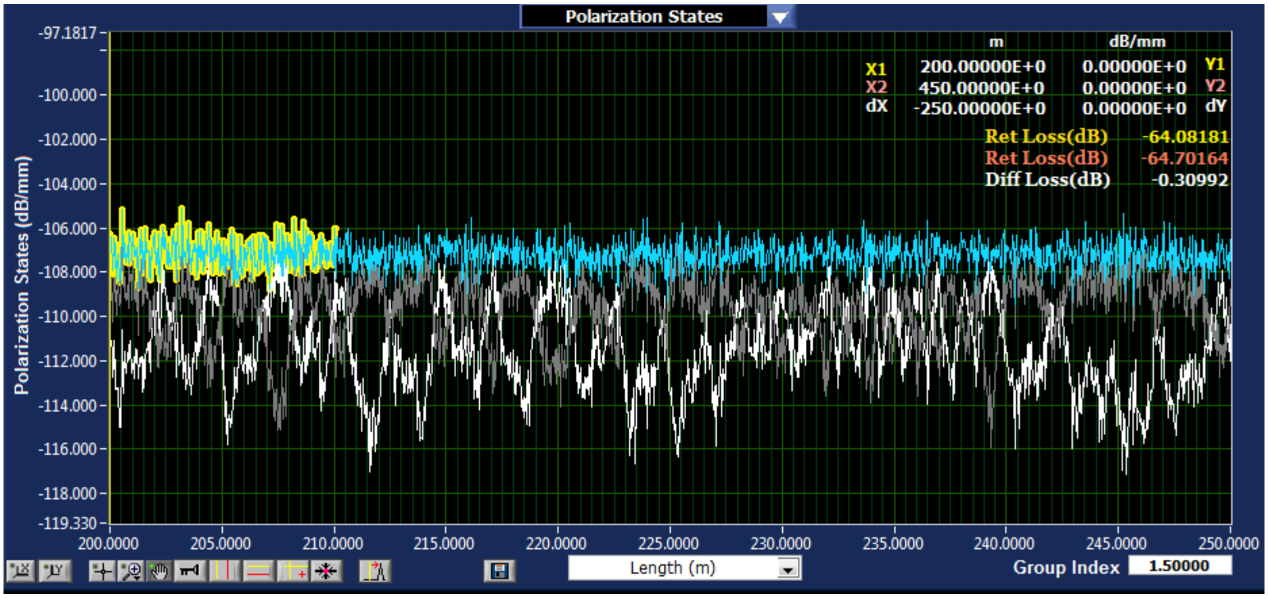

Figure 3: Polarization states for the first 50 m of the measurement region of the PEEK coated fiber prior to the start of temperature and bend radii.

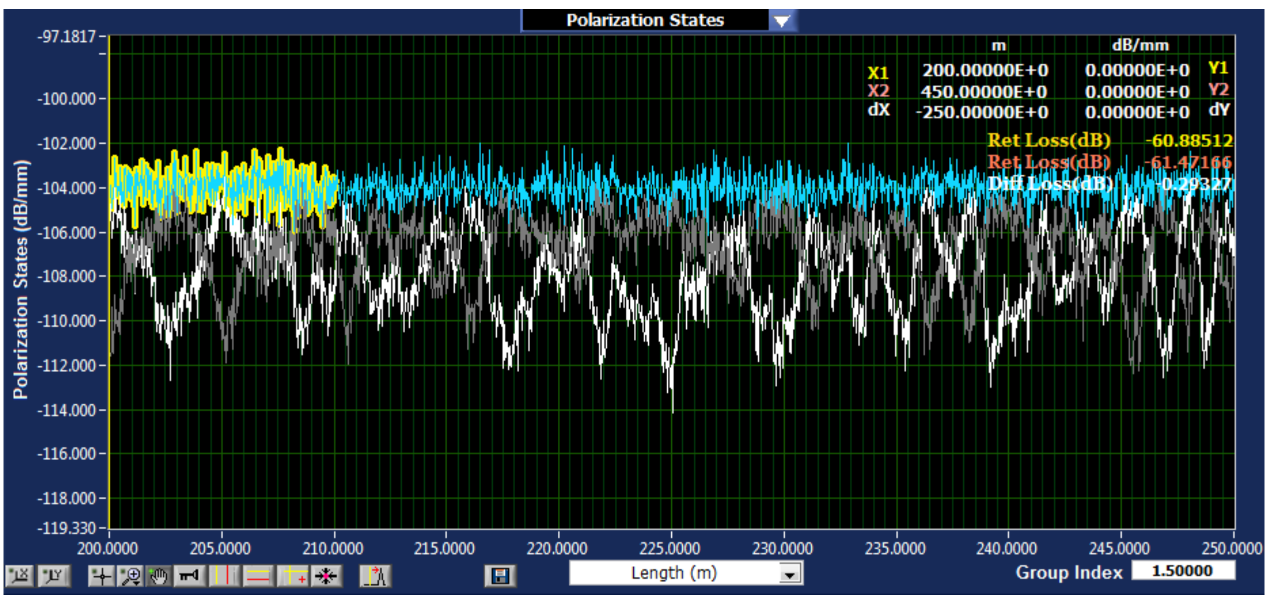

Figure 4: Polarization states for the first 50 m of the measurement region of the uncoated reference fiber prior to the start of temperature and bend radii testing.

TEST RESULTS

This section summarizes the data obtained for the sub-zero testing, elevated thermal cycle, and bend radius testing.

Sub-zero test results

The first series of tests involved placing the optical fiber coils into a sub-zero freezer and cycling the temperature between ambient temperature (~24 °C) and -10 °C (Figure 5). The K-type thermocouples in the freezer testing compartment were used to determine when temperatures had stabilized prior to testing. Final temperatures were taken as the average of the two thermocouple measurements. Each fiber optic cable was then tested for dB loss along the cable using the Luna OBR 4600 during the temperature cycling (Table 1).

Figure 5: Testing scheme for sub-zero temperature measurements showing placement of fiber optic coils in the sub-zero freezer.

Table 1: Loss results obtained during low temperature testing using Luna OBR 4600.

Set point (Temp.)	Reference Fiber			PEEK Coated Fiber		
	Measurement (Temp.)	Scan 1 (dB loss)	Scan 2 (dB loss)	Measurement (Temp.)	Scan 1 (dB loss)	Scan 2 (dB loss)
ºC	ºC	dB/km	dB/km	ºC	dB/km	dB/km
Ambient	24.1	1.20	1.17	24.1	1.23	1.24
-10	-10.6	1.11	1.08	-9.7	1.19	1.23
Ambient	24.1	1.21	1.20	24.1	1.27	1.25
-10	-11.1	1.25	1.24	-10.1	1.13	1.11
Ambient	22.9	1.04	1.02	22.9	1.51	1.53

The average ambient temperature for the sub-zero (low temperature) cycling was 23.7 °C, and the average low temperature was -10.4 °C (Table 2). At ambient temperature, the average loss of the PEEK coated fiber was 1.34 dB/km; this loss, however, was only marginally higher than the average reference fiber loss which was 1.14 dB/km. At -10 °C, the PEEK coated and reference fibers both displayed equal loss of 1.17 dB/km. These results suggest that the PEEK coating applied to the optical fiber does not alter the attenuation characteristics inherent in the fiber.

Table 2: Summary of sub-zero / low temperature cycling test results from Table 1.

Average Temperature (Ref and PEEK ambient temps. were identical)	Average loss		Difference (PEEK – Ref)
	Reference Fiber	PEEK Coated Fiber	
ºC	dB/km	dB/km	dB/km
23.7	1.14	1.34	0.20
-10.4	1.17	1.17	0.00

Elevated Temperature Testing

Elevated temperature analyses were done on the reference and PEEK coated optical fibers to measure dB loss over an extended temperature range (~217 C°). The test fibers were placed in a furnace with the two K-type thermocouples as previously described for the sub-zero fiber testing (Figure 6). The thermocouples identified a temperature gradient within the furnace compartment which was typically < 10 °C. Test temperatures were then cycled between ambient temperature (~27 °C) and 240 °C. Transmission loss of the fibers was then measured at ambient temperature, 100 °C, 150 °C, 200 °C, and 250 °C. Loss measurements were done both on the increasing and decreasing temperature slopes of each cycle using the Luna OBR 4600 and were analyzed similarly to the sub-zero testing (Tables 3 and 4).

Figure 5: Testing scheme for elevated temperature cycling measurements showing placement of fiber optic coils in the furnace.

Following the elevated temperature cycling and testing, both the reference and PEEK coated fibers showed an average loss of <1.5 dB/km for every temperature tested (Tables 3 and 4; Figure 7). At temperatures nearer to ambient, the reference fiber displayed < 0.3 db/km loss than the PEEK coated fiber. Both fibers, however, exhibited very similar increased loss characteristics (~1.35 dB/km) at 150 °C despite this temperature being near the glass transition temperature of the PEEK material. The increased attenuation observed in the reference fiber at 150 °C was thus not expected and is unexplained at this time. At temperatures above 150 °C, however, attenuation in the PEEK coated fiber appeared to decrease (from 1.38 dB/km to 1.01 dB/km) as the fiber was heated compared to the reference fiber. These attenuation differences observed between the two fibers, while measureable, are nevertheless considered very low. Thus, these data suggest that Zeus PEEK coating has minimal effect on dB attenuation for the coated optical fiber.

Table 3: Loss results obtained during elevated temperature cycling using Luna OBR 4600.

Set point (Temp.)	Reference Fiber			PEEK Coated Fiber		
	Measurement (Temp.)	Scan 1 (dB loss)	Scan 2 (dB loss)	**Measurement (Temp.)**	Scan 1 (dB loss)	Scan 2 (dB loss)
ºC	ºC	dB/km	dB/km	ºC	dB/km	dB/km
Ambient	22.9	1.04	1.02	22.9	1.51	1.53
100	100.5	1.02	0.98	101.2	1.43	1.43
150	153.5	1.30	1.33	152.8	1.72	1.68
200	204.3	1.02	0.78	204.8	0.79	0.82
240	244.9	1.51	1.53	244.2	0.91	0.89
Ambient	31.5	1.19	1.18	32.1	1.23	1.23
100	100.9	1.06	1.04	101	1.20	1.15
150	148.4	1.30	1.30	146.8	1.27	1.25
200	204.1	1.40	1.43	207.8	1.20	1.19
240	246.1	1.25	1.24	245.8	1.12	1.12
150	154.5	1.38	1.32	154.2	1.20	1.18
Ambient	28.5	1.00	1.01	27.2	1.34	1.29

Table 4: Summary of elevated temperature testing results. (*Ambient temperature: 27.4-6 °C*)

Average Temperature			Average Loss		Difference
Reference	PEEK	Ref and PEEK combined	Reference Fiber	PEEK Fiber	(PEEK – Ref)
ºC	ºC	ºC	dB/km	dB/km	dB/km
27.6	27.4	27.5	1.07	1.36	0.28
100.7	101.1	100.9	1.02	1.30	0.28
152.1	151.3	151.7	1.32	1.38	0.06
204.2	206.3	205.3	1.16	1.00	-0.16
245.5	245.0	245.3	1.38	1.01	-0.37

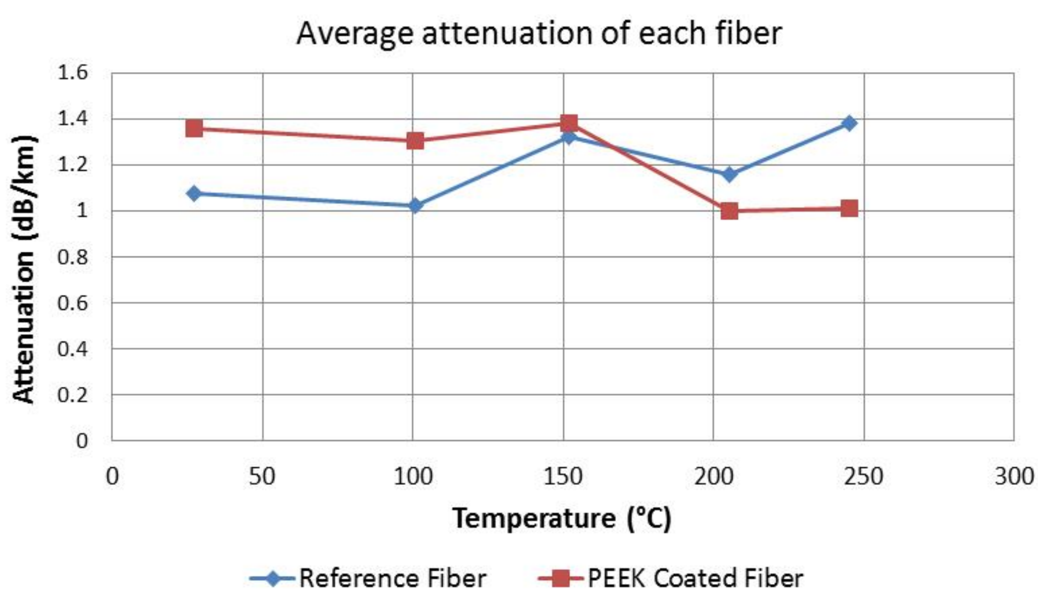

Figure 7: Graphical representation of the attenuation data from Table 4 for the reference and PEEK coated fiber optics during elevated temperature cycling. Temperatures are the average of the PEEK and reference fiber temperature measurements combined.

Bend Radius Testing

A final test was conducted to quantify dB loss attributed to introducing a bend of a known radius into the fiber. For this test, a fixture was designed and built to hold the fiber at three different radii by wrapping it around a cylinder. The PEEK coated fiber or the reference R1550B-P fiber was placed on the test fixture and gripped on one side using a spring loaded clamp with a silicone pad (Figure 8). The clamp pad prevented the fiber from being strained or broken by the clamping pressure. The fiber was then wrapped 1.5 times around one of three radii of the cylinder of the fixture and back to the clamp where it was held securely (Figure 8). The cylinder radii (and thus the fiber test radii) were 50 mm, 30 mm, and 10 mm. Attenuation for these measurements was done over a 10 m section of fiber. The location of the spring clamp on the fixture was chosen by artificially introducing attenuation to the fiber by bending it while measuring attenuation observed in the fiber using the Luna OBR 4600 (Figure 9). The location at which the attenuation occurred was clearly observed and recorded.

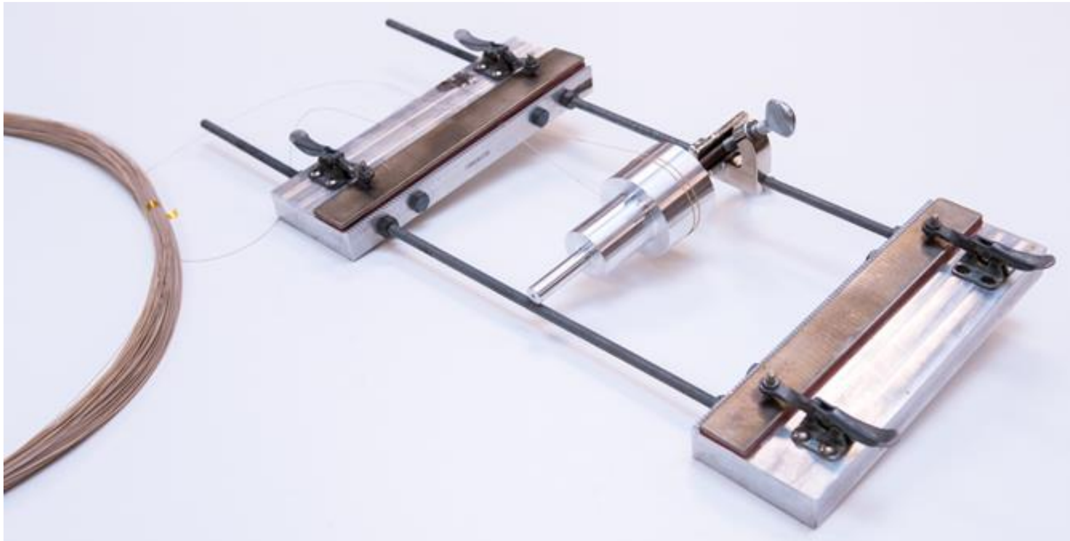

Figure 8: Test fixture used for bend radii testing. The Multiple radii test cylinder is in the middle of the fixture, and the silicone padded spring-loaded clamps are on the ends.

After placement of the fiber in the test fixture, attenuation was measured at near ambient conditions and at 150 ºC following placement of the fixture in the furnace. Attenuation measurements were taken when heated steady state temperature had been reached. The test fixture was then removed from the furnace, and the fiber was unwrapped from the cylinder and allowed to return to ambient temperature. Attenuation was measured again once the unwrapped fiber had reached ambient temperature. This procedure was repeated for each of the three bend radii and on both the PEEK coated fiber and the R1550B-P control fiber. Bend radius test data were analyzed similarly to the sub-zero and elevated temperature test sets. A 20 m integration width was again used for each measurement point along the fiber. The measurement points were spaced 30 m apart on the fiber and equally spaced on either side of the bend.

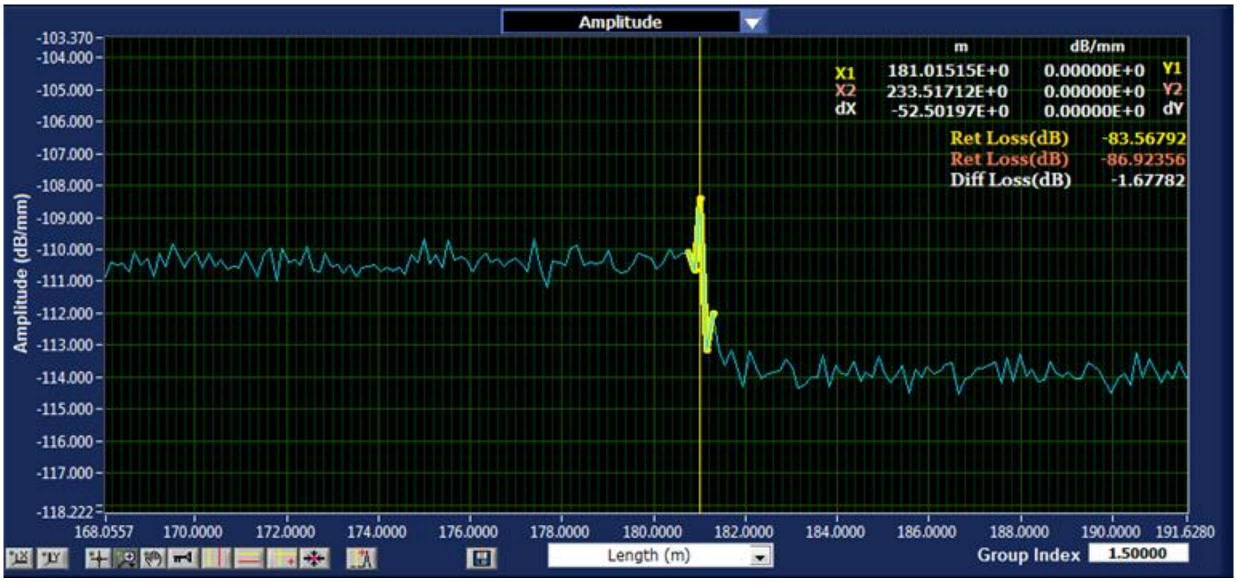

Figure 9: Representative image showing the determination of the positioning of the spring clamp on the fiber by artificially inducing attenuation by bending (PEEK coated fiber shown).

The reference fiber did not show attenuation at ambient conditions following thermal cycling to 150 °C at 50 mm and 10 mm bend radii (Table 5). These results were expected, and thus attenuation for the 30 mm radius for the reference fiber at ambient temperature was not recorded. The reference fiber and the PEEK coated fiber likewise did not show attenuation at elevated temperature when inducing a 50 mm or 30 mm radii bend in them. Most notable was that for the 10 mm radius bend at 150 °C, the reference fiber exhibited an attenuation of 1.6 dB while the PEEK coated fiber showed 1.2 dB loss, representing 25% less attenuation than the reference fiber. This improved attenuation characteristics of the Zeus PEEK coated fiber may be explained by the increased fiber thickness after the application of the PEEK coating. This resulting thickness would place the light guiding core of the fiber within a larger radius than that of the uncoated fiber thereby mitigating attenuation.

Table 5: Attenuation during elevated temperature cycling at multiple fiber bend radii. Measurements were for a 10 m fiber test length containing the bend*. (*Unwrapped fiber; ND = not determined.).*

Radius	Temperature	Reference Fiber		Temperature	PEEK Coated Fiber		Difference
		Scan 1	Scan 2		Scan 1	Scan 2	
mm	ºC	dB	dB	ºC	dB	dB	dB
50	24.1	0.01	*ND*	22.4	0.04	0.04	0.03
50	143.9	0.02	0.03	154.2	0.04	0.04	0.01
30	*ambient*	*ND*	*ND*	33.6	0.03	0.04	*ND*
30	136.0	0.03	0.00	149.2	0.04	0.03	0.02
10	146.8	1.60	1.59	150.5	1.23	1.22	-0.37
*0	24.1	0.01	*ND*	22.4	0.02	0.02	0.01

CONCLUSION

Zeus Industrial Products and Luna collaborated to carry out testing to assess the effects of a new optimized PEEK coating process developed by Zeus for fiber optic applications. Zeus' primary project aims centered on deriving a PEEK coating process that was thermally stable towards the optical fiber and enable the coated fiber to retain its desirable attributes such as efficiency in signal transmission. To this end, Zeus and Luna developed a series of test methods to evaluate Zeus' PEEK coated fiber comparing attenuation in coated and uncoated fibers. Testing incorporated a series of thermal cycling techniques exploring both high and low temperatures and their effects upon the fibers as well as upon Zeus' PEEK coating process. Testing revealed that at sub-freezing temperatures (~ -10 °C), Zeus' PEEK coated optical fiber and the reference fiber exhibited identical attenuation over their 250 m test lengths. At ambient temperatures, the PEEK coated fiber exhibited only slightly increased attenuation (~0.2 dB/km) compared to the reference fiber. This difference in attenuation between the PEEK coated and reference fibers, however, decreased as temperatures increased to 150 °C where their attenuation difference was equal.

Notably, at temperatures above 150 °C, the reference fiber showed increased *(albeit slight)* attenuation compared to the Zeus PEEK coated fiber.

Following the temperature cycling tests, Zeus and Luna investigated the effects of inducing small bend radii to the PEEK coated fibers and compared attenuation in the PEEK coated and uncoated reference fibers. These tests were also carried out at elevated temperatures. This testing showed that at 50 mm and 30 mm bend radii, at ambient and at 150 °C, the Zeus' PEEK coated fiber and the uncoated reference fiber exhibited identical attenuation over the 10 m fiber test lengths. Significantly, however, for the smallest bend radii tested, 10 mm, at 150 °C the uncoated reference fiber showed greater attenuation than the PEEK coated fiber.

Taken together, these findings suggest that Zeus' PEEK coating process did not introduce physical stresses or deformations such as microbends to the fiber. Furthermore, these data indicate that the coating is thermally stable once it has been applied and is thus not susceptible to shrinkage, particularly where large temperature swings may occur. Zeus PEEK coated fiber performed at least as well as uncoated similar fiber while still imparting improved resistance to heat, chemicals, and radiation for which PEEK is typically known. Under other conditions, however, Zeus PEEK coated fibers may exceed the performance of uncoated fibers.