

## ADVANCES IN DESIGN AND DEVELOPMENT OF OPTICAL FIBERS FOR HARSH ENVIRONMENTS

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### ABSTRACT

Optical fiber for harsh environments presents unique design challenges, particularly in coatings required to maintain the properties of the fiber. In a typical harsh environment application, the optical fibers are exposed to water, hydrogen or other harmful chemicals at elevated temperatures, e.g.  $> 200$  °C. The fibers are commonly coated with a thin layer of carbon and further coated with thermosetting polyimide materials that can withstand significantly higher temperature than the common UV cured acrylate coatings. In this paper, we describe our recent efforts in understanding the impact of the environment on the fiber performance and report the advances in improving fiber and coating design to minimize the impact. Along with the discussion of the familiar fiber failure mechanisms, we also show the existence of a unique failure mechanism, i. e. the degradation of coating properties that leads to optical transmission loss.

### BACKGROUND

#### Optical Fiber for Harsh Environments

Optical fibers have been proven advantageous over traditional copper based data communication media in many aspects such as high transmission capacity, immunity to electromagnetic interference, and size. More recently, optical fibers have also been successfully used in distributed sensing both as sensor and signal transmission link<sup>1,2</sup>. Applications in harsh environments, where water, hydrogen, and other harmful chemicals are commonly present often under high pressures and temperatures, or where the fibers are under high stress, have prompted the development of optical fibers with specialty designs. Typical applications include monitoring of oil wells, undersea data communications, geophysical exploration, power cable temperature monitoring, fire and leak detection, and geothermal well sensing. To meet these particular challenges, optical fibers with specialty coatings have been developed. A typical optical fiber of this kind consists of a silica glass lightguide, a thin carbon coating, and a polyimide coating. The carbon layer functions as a barrier to water and

hydrogen diffusion, improving the fiber's resistance to fatigue and hydrogen induced optical loss. The polyimide coating provides additional protection for the carbon-coated fiber against mechanical damage, especially at elevated temperatures.

### **Failure Mechanisms**

For successful application in harsh environments, it is imperative for an optical fiber to retain its characteristics, primarily mechanical strength and waveguide properties. An optical fiber can experience delayed fracture due to interaction with water and suffer reduced transmission as a result of macro-bend, micro-bend, and hydrogen diffusion. The effects of these deleterious processes on fiber are intensified under harsh conditions. There exist other failure mechanisms unique to the harsh conditions, such as degradation of the coatings caused by water or corrosive chemicals. Understanding of the failure mechanisms is therefore critical to optimize fiber design for the applications.

It is well known that the presence of surface flaws or cracks, induced mechanically or chemically, reduces the strength of silica optical fibers<sup>3</sup>. These flaws act as local stress concentrators under strain, resulting in a much lower tensile strength than the theoretical value of silica glass. Furthermore, the reaction of these flaws with moisture causes delayed fracture of the fiber or fatigue. Water molecules break the strained chemical bonds at the crack tip, forming two hydroxyl groups for each Si-O bond and leading to the crack growth<sup>3</sup>. This impairs the long-term reliability of the fiber. To minimize the impact of water, coatings that are impervious to water molecules, e. g. carbon

coatings were developed. Carbon coatings lower fiber tensile strength, but significantly increase the stress corrosion factor, the  $n$  parameter, which is a measure of the fiber's resistance to fatigue. The significant increase in  $n$  parameter allows the carbon-coated fiber to be reliably used under higher stress than that acceptable for non-carbon coated fiber.

Hydrogen-induced optical transmission loss is another common failure mechanism of optical fibers when used in hydrogen or water containing environments, such as undersea optical cables<sup>4</sup>. Mobile hydrogen molecules, generated as a product of the corrosion process of metal components, or released from polymer materials or water, diffuse into the core of the optical fiber, inducing optical loss. The optical loss is characterized by its wavelength dependence. Molecular hydrogen in GeO<sub>2</sub>-doped silica optical fibers shows several absorption bands in the spectral range from 1.08  $\mu\text{m}$  to 2.42  $\mu\text{m}$ , with the well known bands in the range from 1.08  $\mu\text{m}$  to 1.24  $\mu\text{m}$ <sup>5</sup>. Another important characteristic of these absorption bands is their reversibility. They disappear when the hydrogen molecules are allowed to leave the fiber core<sup>5</sup>. The OH groups as a product of the reaction between molecular hydrogen and the glass matrix exhibit several absorption bands including one at near 1.38  $\mu\text{m}$ . Different from the reversibility of the bands related to molecular hydrogen, the OH related bands are stable because the hydrogen in OH is strongly bonded to the glass matrix.

In addition to the hydrogen, water diffused into the fiber under high pressure has been observed to induce a fiber dimension change and a change in fiber index of refraction<sup>6</sup>. This change may be

related to the water-induced structural relaxation of highly quenched glass matrix of the optical fiber. Carbon coating on the fiber surface has shown to be effective in preventing the fiber property change<sup>6</sup>.

Microbend loss is referred to the optical transmission loss in optical fibers caused by an external perturbation of the guiding condition of the fibers. Typical perturbation includes the variation in coating thickness, and residual stresses due to coefficient of thermal expansion (CTE) mismatch between the coating and the glass. The loss exhibits much less wavelength dependence than macrobend loss, which is the loss observed when a fiber is bent to a radius of several centimeters. For standard telecommunication fibers, the microbend loss of the fiber is overcome by selecting a low modulus primary coating and a high modulus outer secondary coating. The harder outer coating provides high mechanical protection while the softer primary coatings serves as a cushion to absorb the external perturbation.

### **Design Considerations**

It becomes clear from the preceding discussion of failure mechanisms that the success of the application of optical fibers in harsh environments relies, to a great extent, on the coatings and their survivability and reliability. Due to its suited properties and relative ease of processing, carbon is perhaps the most common material used for coating as barriers to water and hydrogen diffusion. Polymeric coatings are required to provide additional protection for the carbon-coated fiber against mechanical damage. Thermosetting polyimide coating is usually selected for its wider working temperature range than that of

the common UV cured acrylate coating. However, in general the properties of polymeric materials are more sensitive to temperature and the environment, as compared to the inorganic silica glass and carbon. Therefore, the stability of the polymeric materials under harsh conditions dictates the reliability of the fibers, since the degradation of the properties of the polymeric coatings can lead to the degradation of the mechanical and optical properties of the fiber. Furthermore, waveguide designs may also need to be optimized to minimize the impact of the degradation of the polymeric coatings. Other characteristics of the coatings may be of particular importance in some applications, such as chemical durability, water resistance, and surface texture of the coatings. In the present work, we will provide experimental results on the degradation of polyimide coatings and its impact on the fiber performance and discuss the degradation as an important failure mechanism, particularly for the fibers used in harsh environments.

## **EXPERIMENTAL**

### **Samples**

Optical fibers used in the experiments were graded index multimode optical fibers coated with carbon and polyimide coatings, manufactured at OFS (formerly SpecTran). Table 1. Lists the type of the fibers used in the accelerated tests, their geometry, optical properties, type of polyimide coatings, and the type of water treatment.

More specifically, Fiber 1 was a standard fiber and Fiber 2 was a similar fiber, but with a type B polyimide coating. Fiber 3 was a same fiber as the Fiber 1, but was treated only in low-pressure water.

Fiber 4 was essentially a Fiber 1 with a larger core size and a larger NA (Numerical Aperture). Type B polyimide has a higher elastic modulus and higher water resistance than Type A polyimide.

### **Mechanical Test**

Fiber samples were tested for tensile strength according to the standard procedures described in FOTP 28, or TIA/EIA-455-28C. Briefly, the samples were tested using a gage length of 0.5 m at a strain rate of 4%/min. The stress corrosion factors were measured and derived using the procedure in the same TIA/EIA standard. A 0.5 m gage length, and strain rates of 25%, 2.5%, 0.25%, and 0.025% were used.

### **Thermal Test**

Samples were tested for thermal stability of optical transmission according to the procedures described in FOTP 3, or TIA-455-3A. Specifically, in thermal soaking test, samples were heated and held at 300 °C in air for 50 hours while the transmission of the fiber was continuously monitored at 850 nm. In the thermal cycling test, the temperature varies between -65 °C and 175 °C while the fiber transmission was also continuously monitored.

### **Accelerated Test**

This test is to examine the impact of harsh environments on fiber optical transmission, particularly under high temperatures in the presence of water in a laboratory environment. The fiber samples were soaked in water at ambient temperature and at high pressure (>100 atm.) for 2 hours and then at ambient pressure for more than 20 hours. The reference sample, Fiber 3,

was soaked only at ambient pressure for the same period of time. The samples were removed from water and subsequently heated in nitrogen in a loose coil to temperatures ranging from 200 °C to 300 °C. The optical transmission of the samples was continuously monitored at 1.064 μm using an OTDR.

## **RESULTS AND DISCUSSION**

### **Tensile Strength and Stress Corrosion Factor**

Table 2 summarizes the tensile strength and stress corrosion factor for our standard carbon/polyimide coated fibers (New) and polyimide coated fibers (Non-Carbon). The properties of our fibers (Old) made before recent improvement of the carbon deposition process are also listed. The carbon/polyimide coated fiber showed a lower median strength than that of the polyimide coated fiber, as expected. However, the tensile strength was at least 10% greater than that previously reported<sup>7</sup>. More importantly, the average value for the stress corrosion factor,  $n$ , obtained from dynamic fatigue data is >200 while the value of  $n$  for polyimide coated fibers is about 20. The previously reported value for  $n$  of carbon/polyimide coated fiber was > 100. This improved strength along with the  $n$  parameter is an indication of better carbon coating quality and higher deposition efficiency of our modified process. For more detailed discussion, see the reference<sup>8</sup>.

Fig.1 compares the tensile strength of a typical carbon/polyimide coated fiber, same as the Fiber 1, and a typical polyimide coated fiber.

## **Surface Morphology**

Scanning Electron Microscopy (SEM) was used to examine the effects of temperature and environment on the physical integrity of the polyimide coatings. Fig. 2 and Fig. 3 are the SEM micrographs of two fibers before and after the high-pressure water soaking and heat treatment in air at 300 °C for 2 days. The fiber samples shown in Fig. 2 are the same as the Fiber 1 and the fibers in Fig. 3 are the same type of fibers with an extra cured polyimide coating. Notice the fiber diameter is 155  $\mu\text{m}$ .

After the treatment, the standard coating, Fig. 2 (b), has shown signs of surface degradation while the extra cured coating, Fig. 3 (b), remained unchanged. From the comparison above, the polyimide coating with the extra cure seems to have higher resistance to water and high temperature than the standard coating.

## **Thermal Cycling Date**

The purpose of the test is to examine the thermal stability of the polyimide coating and its effect on the optical transmission of the fiber. In the thermal soaking test, a standard polyimide coated, graded index multimode optical fiber showed an induced loss decrease of 0.6 dB/km at 300 °C, but no significant permanent induced optical loss when cooled down to room temperature, indicating reasonable thermal stability of the polyimide coating.

Fig. 4 shows a typical optical loss change in the same type of fiber as a function of temperature in a typical thermal cycling test. The initial drop in loss from 0.4 dB/km to 0 dB/km and the lack of response in loss to the temperature

change in the first 10 hours are perhaps due to some experimental error and thus should be ignored. The induced loss decreases as the temperature increases. This is mainly caused by the reduction in compressive residual stress on glass fiber surface. There were also small peaks when temperature was held at the high temperature located between the large peaks in the loss curve. These peaks may be related to the stress change due to property changes of the polyimide coating. After the test, a permanent induced-loss of less than 0.4dB/km was observed. To summarize, the variations in temperature have more severe impact on the coating and fiber than the high temperature soaking.

## **Loss-Temperature-Time Curves**

Figs. 5 and 6 show the optical loss change of Fibers 1, 2, 3, and 4 during the accelerated tests. See Table 1 for the description of the fiber samples and the related water treatment.

From the curves, at least three significant observations can be made.

1. In all 4 samples, loss gradually increased under constant temperature and it became more severe when temperature was higher. Fiber 1 showed an enormous increase at 275°C.
2. Loss in Fiber 2 with Type B polyimide coating decreased at each step increase in temperature. In Fibers 1, 3, and 4, the loss decrease was not significant until the temperature reaches 275°C. It was much more prominent for Fiber 1 when temperature is increased from 275°C to 300°C.

3. Loss in Fibers 2, 3, and 4 remained relatively low while loss in Fiber 1 became unacceptable.

### **Loss Increases**

As shown by the curve for Fiber 1 in Fig. 5, the increase in optical loss became drastically higher when the fiber was heated to and held at 275°C, reaching 70 dB/km from less than 10 dB/km in about 24 hours. Optical loss in Fiber 2 which has a more moisture resistant coating, however, stayed below 10 dB/km under 275°C during the same period and started to increase when the temperature increased to 300°C. The curve for Fiber 3 shown in Fig. 6 was from a same fiber, but was soaked in water only at the ambient pressure. The curve showed no increase in loss even at 300°C, as does the untreated standard fiber of same type heated in air in the thermal soaking test. This indicates that soaking in water at low pressure has no significant effect on the coating properties. In contrast, the comparison of this curve with Fiber 1 shows clearly the effect of high-pressure water treatment. Since the fiber samples were treated in water and heated at elevated temperatures, the hydrogen or water diffusion directly into the core of the fibers will be naturally considered as a possible cause of the loss increase. To determine the nature of the induced transmission loss, which was observed also in several similar fibers, attenuation spectra of the samples were taken after the test. The spectra showed a broad, featureless background attenuation increase over a wide spectral range, with no distinguishable bands associated with hydrogen or OH groups, suggesting that the loss increase is not caused by direct diffusion of hydrogen or water molecules into the core of the waveguide. There-

fore, we may eliminate the diffusion of hydrogen induced loss as a loss mechanism in this case. A more probable cause of the loss increase is microbend-related loss. As is well known, microbend loss can be induced by residual stresses developed due to the significant CTE mismatch between the glass and the polyimide. The CTE for silica glass,  $\alpha = 0.5 \times 10^{-6}/^{\circ}\text{C}$  while that of typical polyimide fully cured will be at least  $3 \times 10^{-6}/^{\circ}\text{C}$ . As a result, tension and compression are likely to develop in the coating and on the glass fiber surface respectively when the fiber is cooled down to room temperature from the temperature at which the polyimide is solidified during fiber draw. However, one would expect no change in optical transmission at a constant temperature if the coating properties remain unchanged. Thus, the loss increase with time at given temperature may only be explained to be caused by the slow build-up of residual stress as a consequence of the degradation of coating properties. Water-induced post curing of the polyimide coating is a most probable type of degradation at elevated temperature. Conceivably, the polyimide coating applied in-line during the fiber draw may be less cured and undergo more post curing during the subsequent heating, since the initial curing process is limited in time by the draw speed. Post curing of polyimide continues to convert the polyamic acid into a fully aromatic, insoluble polyimide and drive off the residual solvent. Consequently, post curing of the polyimide reduces the dimension of the polyimide coating and causes the elastic modulus to approach a larger value corresponding to the fully cured polyimide. At same time, the CTE decrease with increased cross-linking as post curing proceeds. Significant amount of resid-

ual stress can result from these changes in coating properties, thus leading to an increase in microbend loss.

### **Loss Decreases**

Transmission loss decreases were observed for all 4 fibers when the temperature was abruptly increased. This is consistent with the loss behavior observed in Fig. 4. A large dip in transmission loss was observed for the Fiber 1 when the temperature was quickly increased from 275°C to 300°C. This observation was confirmed by similar dips observed in several similar fibers. This may be explained by the microbend loss related to the residual stress in the fibers. As expected, there is a partial release of the residual stress in the coating induced by the dimension increase of the coating,  $\Delta L/L$ , according to its CTE,  $\alpha$ , and the temperature difference,  $\Delta T$ ,  $\Delta L/L = \alpha \Delta T$ , upon the increase in temperature. A decrease in elastic modulus as temperature increases may also contribute to the partial stress release. The dips with slightly larger amplitude observed in Fiber 2 at each step increase in temperature may be explained by the fact that only the Fiber 2 has a polyimide coating, derived from a polyimide, Type B, having an elastic modulus much higher than that of the coating Type A. Fibers with high elastic modulus coatings are more sensitive to microbend.

### **Microbend Loss**

Thus far we have eliminated the possibility of hydrogen/water diffusion as a cause for the induced losses. We have also postulated that the loss increases with time are caused by microbend due to residual stress build-up generated by the continuous dimensional reduction of the polyimide coating resulted from

the water-induced post curing at high temperatures. To verify that microbend is the cause of the loss, Fiber 4, which is a high NA, carbon/polyimide coated fiber, was tested and its loss-temperature-time curve is presented as Fiber 4 in Fig. 6. It is clearly noticed that Fiber 4, which underwent the same treatment as for Fiber 1, showed no significant loss increase at temperatures up to 300°C. This improvement is not surprising if microbend is indeed the major contributing cause of the loss. An empirical expression for microbend loss in a multimode optical fiber is given by<sup>9</sup>:

$$M_L \propto \left[ \frac{a^4}{b^6 \Delta^3} \right] \left[ \frac{E}{E_F} \right]^{3/2}$$

where  $M_L$  is the microbend loss for multimode fiber,  $a$ , core radius,  $b$ , fiber diameter,  $E_F$ , elastic modulus of the fiber,  $E$ , elastic modulus of the surrounding medium,  $\Delta$ , index difference between core and cladding. According to this expression, to minimize the microbend loss, one can either increase the fiber diameter and fiber NA, or reduce core size or choose a softer primary coating. If Fiber 1 is replaced by Fiber 4, the model predicts a reduction in microbend loss greater than 60%, assuming that this model is also applicable for graded index fibers. Although the reduction shown by the comparison is much greater for some unknown causes, this comparison has strongly suggested that the loss and the loss increase are indeed microbend related. This model also indicates that the softer polyimide coating can reduce microbend loss. However, the stability of the physical dimension and of the elastic modulus of the coating becomes more critical when the coating is continuously being cured.

## **Role of Water**

From the aforementioned comparison, the large loss increase in Fiber 1 is clearly correlated with the water present in the polyimide coatings. In the absence of water, the standard can retain their optical transmission at temperatures  $> 300^{\circ}\text{C}$  in air. Fiber 1 probably contains the largest amount of water while Fiber 2 contains less because the new polyimide coating is much more moisture resistant. This observation confirms the association of the loss with the presence of water. Fiber 3 contains perhaps the least amount of water, since it was treated only in low-pressure water. Water in the polyimide may act as plasticizer at low temperature, lowering the elastic modulus of the coating. However, at higher temperature, water triggers some accelerated process, causing the loss to increase drastically with time.

It can be hypothesized that water present in the polyimide coating of an optical fiber enhances the post curing of the coating at high temperature, resulting in a coating dimensional change, creating higher compressive residual stress that leads to optical loss increase in the optical fiber. Water molecules may diffuse into the polyimide coating under high pressure and become bonded to the unreacted acid groups of the polyimide via weak bonding such as hydrogen bond. The presence of a significant amount of water in the polyimide may impede the reaction of post curing and also soften the material at low temperature. During subsequent heating, however, when the temperature is sufficiently high, the bonded water molecules are dissociated from the acid groups, evaporated, and probably driven out of the coating through the pores,

making the acid groups available for the reaction of post curing. The residual solvent molecules that are released during curing dissolve in the water and are driven out of the system with water. This may also contribute to the acceleration of the curing process.

## **Minimizing the Impact**

As has been shown, the thermal stability of polyimide coating properties in the presence of water is key to ensure successful applications. The need to prevent water from entering the polyimide coating becomes obvious. One can either avoid high-pressure water, or select more water-resistant coatings in case high-pressure water is expected. Polyimide coatings that allow a lesser amount of post curing may also increase the property stability. Polyimide primary coatings with lower elastic modulus and good thermal stability are also preferred to cushion the perturbations. As predicted by the microbend model and proven by the Fiber 4 result, fibers with higher NA, are much less sensitive to the perturbation. High Ge concentration in the core of the fiber required by higher NA can in some cases be beneficial, such as Distributed Temperature Sensing, which uses Raman back scattering for interrogation.

## **SUMMARY**

We have reported recent advances in our efforts to improve design and performance of the fibers for harsh environment applications, specifically in fiber mechanical strength, coating durability, and the stability of optical transmission. We have successfully identified the degradation of the polyimide coatings as a primary cause of the optical transmission deterioration of the fibers

at elevated temperatures in the presence of water. The degradation is suggested to be caused by the water-induced post curing of the polyimide coatings. Residual compressive stress is being developed in the fiber as the polyimide coating cures, inducing optical transmission loss in the fiber, or microbend loss. Improvements on fiber design and performance based on the model of microbend loss have proven the nature of the loss mechanism. Several recommendations as to minimize the impact of the coating property change have been made based on the experiment results. To prevent water from entering the polyimide coatings, avoid the exposure of the fibers to water at high pressure, and select polyimide coatings with high water resistance. High NA provides the fibers low susceptibility to microbend loss, and then allows more flexibility in coating selection. Work towards the more definitive verification of the loss failure mechanism is still in progress.

## ACKNOWLEDGEMENT

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Table. 1 Fiber sample properties and treatment

Fiber	1	2	3	4
Core Size, $\mu\text{m}$	50	50	50	62.5
Clad Size, $\mu\text{m}$	125	125	125	125
Fiber Size, $\mu\text{m}$	155	155	155	155
NA	0.2	0.2	0.2	0.275
Carbon	Yes	Yes	Yes	Yes
Polyimide Type	A	B	A	A
Water Soaking Pressure, atm	> 100	> 100	1	> 100

Table 2. Fiber mechanical properties

Fiber	Median Fracture Stress, Gpa	Stress Corrosion Factor, n
Old	3.5	>100
New	4	>200
Non-Carbon	>5	>20

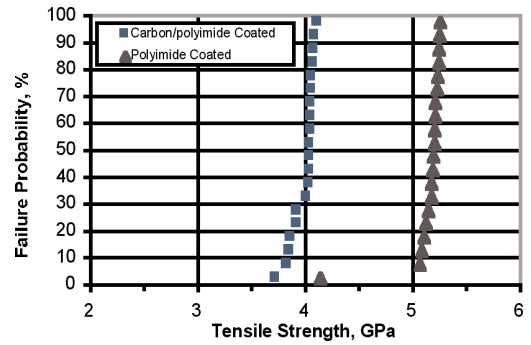


Fig. 1 Strength comparison of the carbon/polyimide coated fiber and the polyimide coated fiber.

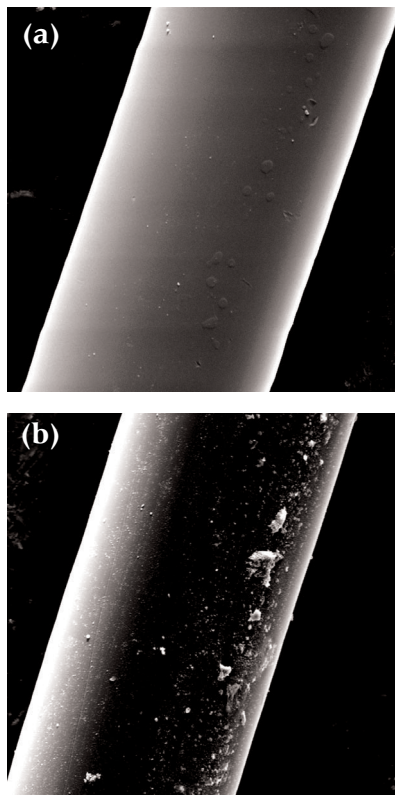


Fig. 2 SEM micrographs of the surface of the polyimide coating on the standard carbon/polyimide coated optical fibers before (a) and after (b) the high pressure water soaking and heating in air at 300°C for 2 days.

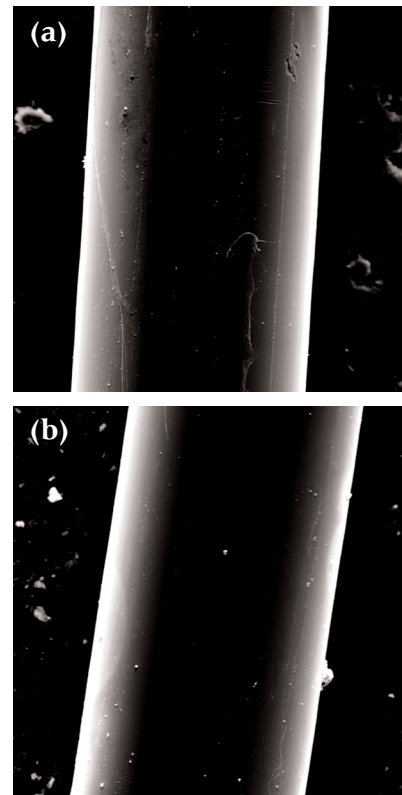


Fig. 3 SEM micrographs of the surface of the polyimide coating with the additional curing on carbon/polyimide coated optical fibers before (a) and after (b) the high pressure water soaking and heating in air at 300°C for 2 days.

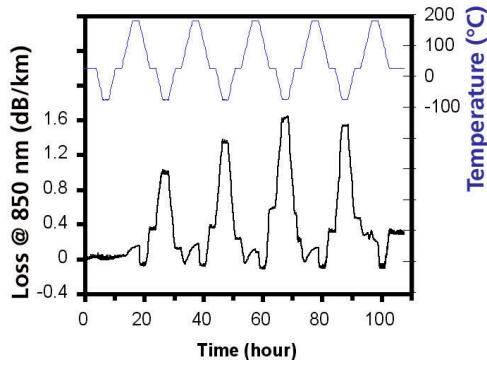


Fig.4 Induced optical loss of a standard polyimide coated, graded index, multi-mode optical fiber, the bottom curve, as a function of temperature change, top curve.



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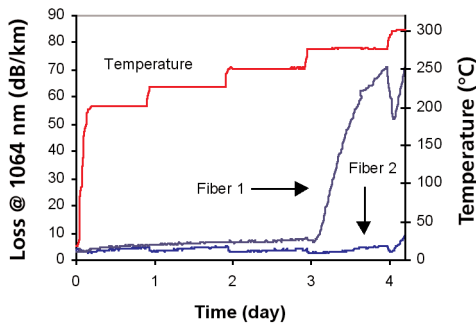


Fig. 5 Loss-temperature-time curves for Fibers 1 & 2.

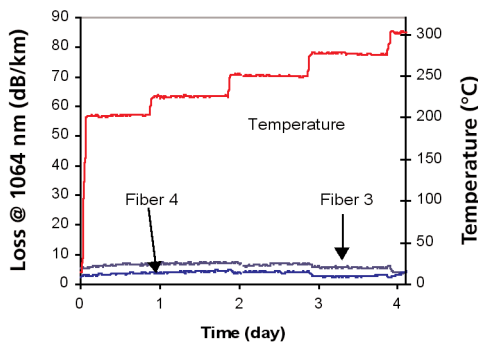


Fig. 6 Loss-temperature-time curves for Fibers 3 & 4.

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