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High-Performance Hermetic Optical Fiber for Downhole Applications

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Abstract

Optical fibers have been used in the petroleum industry for many years; their applications range from real-time temperature and pressure measurements, data transmission, to imaging applications. In this paper, we demonstrate significantly improved reliability of recently developed hermetic, high operating temperature optical fiber. The study includes extensive testing of this fiber under controlled conditions; and a comparison with previous studies and other non-hermetic fibers.

The hermetic optical fiber of this study is coated with a thin layer of carbon, which provides excellent resistance to hydrogen permeation, water diffusion, and corrosive chemicals attack. Our results show high resistance of the fiber to hydrogen, crucial in the harsh conditions of downhole well completion.

To simulate the harsh environment, a test chamber has been built capable of exposing the fiber to temperatures up to 200°C and pure hydrogen pressures up to 120atm. Attenuation spectra from 0.8 to 1.6 μ are recorded before and after exposure. At 150°C and 100atm of pure hydrogen exposure, the induced attenuation at 1.24 μ is typically less than 0.4dB/km/100hours. From these spectra, we extract the permeation time constant τ_f as a function of temperature and thereby obtain the activation energy for hydrogen permeation through the carbon layer. The use of τ_f is extremely useful to describe carbon coating resistance to hydrogen ingress. Moreover, this parameter allows us to separate the characteristics of the carbon coating from the reaction characteristics of the glass with the ingressing molecules.

In addition to its high resistance to ingressing molecules, the hermetic fiber of this study also offers enhanced mechanical

fatigue resistance determined by both static and dynamic fatigue tests. This improved fatigue resistance is indicated by the high stress corrosion parameter N value ≈ 150 .

Introduction

Optical Fibers, with some form of protective coating on the glass surface, have been used in downhole applications for a number of years by the Oil, Gas and Energy Industry¹. In these applications the optical fiber is employed either as a distributed or point sensor, or as a data communication highway in geophysical and geothermal wells. Typical downhole applications can be up to ~7 kilometers deep and the use of optical fibers to either retrieve data at high bandwidths, not available via copper transmission lines, or to obtain detailed distributed temperature or pressure information along the length of the well bore is essential for the optimization of oil production in “Intelligent Wells”. While the benefits of using optical technology over traditional electronic techniques have been demonstrated, the widespread use of optical fiber in downhole applications is just beginning to occur.

Although many fiberoptic-based measurement techniques have been demonstrated in downhole applications, one of the most commonly used and widely deployed in oil wells is the Distributed Temperature Sensing (DTS) which uses Raman backscattering in glass fibers. This technique provides a continuous temperature profile along the length of a fiberoptic cable. The principle of DTS operation is based on Optical Time Domain Reflectometry (OTDR) using the intensity of Raman backscattering as the temperature detection feature. In this technique, the launch-to-detection time defines the position along the sensing fiber, and the reflected Anti-Stokes Raman signal intensity defines the temperature level. The Raman Anti-Stokes band is used for temperature sensing as it is very sensitive to temperature variations. The Stokes band being less sensitive to temperature is typically used for loss calibration. The optical fiber of this study has been successfully deployed and used in DTS systems.

There are two major impediments to the widespread use of optical fiber technology in a downhole environment. First, it is relatively new, compared to what has been used for the majority of the last century, e.g. electronics; and there is the usual concern associated with introducing new technology in a well-established, highly cost and reliability conscious industry. Secondly, the characteristics of the fiber regarding

handling (deployment, connectorization, splicing etc.) and performance are not fully understood; of the former one can say that significant efforts need to be made to standardize handling issues currently faced by well completion groups involved in such deployment, however, this does not appear to be a fundamental operational barrier that cannot be overcome. On the other hand, the performance characteristics of optical fibers in downhole environment are still subject to widespread misinterpretation and misunderstanding.

Although there are other secondary characteristics of interest to the downhole users such as the performance of the fiber polymeric coatings at temperatures in excess of 100°C, the main focus of this paper is to quantify the performance characteristics of optical fibers in regards to two of the most important aspects:

- ✓ Hydrogen ingress for optical performance
- ✓ Mechanical strength for deployment and handling

To evaluate hydrogen ingress in optical fibers, we use a characteristic time constant called “permeation time constant” to quantify the amount of hydrogen that diffuses through the protective coating applied to the glass surface of the fiber, as well, we characterize the temperature dependence of this parameter, which allows the prediction of hydrogen ingress characteristics of the protective coating at any temperature.

In this paper, the mechanical strength of the fiber was studied using the traditional “stress corrosion parameter”, n -value, to characterize the fatigue resistance of the fiber. This parameter along with Weibull² mean strength (σ_0) describes the failure probability of the fiber under different stressed conditions².

Theory

Permeation Time Constant “ τ_f ”

The traditional method of providing protection to optical fibers in such harsh environments has been the application of an impervious coating on the surface of the glass. Many different coatings have been tried, e.g. metals³ ceramics^{4,5} and carbon^{6,7}. Metals tend to form polycrystalline structures where the grain boundaries act as short circuit diffusion paths and can themselves get rapidly corroded in harsh environments; additionally even soft metals such as tin and aluminum tend to induce micro-bending losses.

Ceramic coatings such as silicon nitride or silicon carbide have also been used, and have demonstrated to be effective in providing resistance to water at elevated temperatures and pressures, however for these coatings, generally the mean strength is significantly below the value for standard polymer coated glass fibers, and as in metals, due to the high modulus of these materials the coatings tend to give rise to significant micro-bending losses. Hence, neither ceramic nor metal coatings can fulfill the requirements for strength, hermeticity and bend loss all in the same fiber. In addition, producing long continuous lengths of these fiber types has proven problematic due to processing complexity.

Carbon coatings on the other hand appear to be the most

attractive in providing solutions to all the above mentioned problems for temperatures in excess of 100°C; at these temperatures e.g., saturation lifetimes with respect to hydrogen ingress are on the order of years; micro-bending is minimal and mean strength while on average still below the optimal values obtained for polymer coated fibers, can be slightly improved with improved process/roughness control during the deposition of carbon. Carbon coated optical fibers have been manufactured in the past⁷; and the carbon permeability to hydrogen extensively studied⁸. The time dependence of the concentration of hydrogen in the glass fiber is given by Le Maire et al.⁸

$$\Delta\alpha_{1.24}(t,T)/\{\Delta\alpha_{1.24}(\text{inf},T) \times P_{H_2}\} = [1 - \exp\{-(t-\tau_i)/\tau_f\}] \quad (1)$$

Where $\Delta\alpha_{1.24}(t,T)$ is the change in attenuation (at 1.24 μm) after the fiber has been exposed to a hydrogen environment for time t at temperature T ; $\Delta\alpha_{1.24}(\infty,T)$ is the change in attenuation when equilibrium has been reached between the fiber and the environment; and P_{H_2} is the hydrogen pressure. τ_i and τ_f are the time constants for initial lag (before any increase in attenuation is observed) and the rate at which the attenuation increases at any given temperature T . Also for relatively thin coatings, τ_i is generally much less than τ_f . For relatively short times, equation (1) becomes

$$\Delta\alpha_{1.24}(t,T)/\{\Delta\alpha_{1.24}(\infty,T) \times P_{H_2}\} = [t/\tau_f] \quad (2)$$

A plot of the Left Hand Side of equation (2) vs. time “ t ” should yield a line with slope $1/\tau_f$. τ_f and τ_i contain all the information of interest regarding the permeation characteristics of the carbon coating. Larger values of τ_f imply a more resistant coating, and maximizing its magnitude has been the goal of many researchers⁸. Most studies to date that have tried to optimize the value of τ_f have focused on the carbon deposition conditions and the selection of the carbon precursor^{9,10}.

One of the key properties of the permeation time constant is that it describes only the hydrogen permeation characteristics of the protective coating on the surface of the glass fiber, independently of the properties of the glass. This allows us to separate out the resistance to H_2 of the protective layer from that of the glass. Additionally, this information allows the design optimization of the glass and carbon layer independently.

Strength/Fatigue Characteristics of Fibers

The fatigue characteristics of optical fibers are described by the theory of propagating micro-cracks, first developed by Griffiths¹¹. The Griffiths micro-crack is an elliptically shaped crack of depth χ and a tip radius ρ , the stress intensity factor is defined as:

$$K = Y \chi^{1/2} \sigma \quad (3)$$

Where σ is the stress, in megapascals applied to the fiber and χ is the crack depth in millimeters. The dimensionless constant Y depends on the geometry of the flaw, for surface flaws (most critical for optical fibers) $Y=(\pi^{1/2})$. The maximum value of K (K_c : critical stress intensity factor) for silica glasses is in the range 0.6-0.9M-N/m^{3/2}. Equation (3) defines the maximum crack depth for a given applied stress, before fracture occurs.

In general, under stressed conditions sub-critical crack growth will take place resulting in eventual failure, the rate of crack growth is determined empirically to follow the power law relationship:

$$d\chi/dt = AK^n \quad (4)$$

Where A is a material constant and n is the stress corrosion factor. For standard telecommunications grade fibers, n is approximately 20. Since the value of K is always less than one, large values of n imply highly fatigue resistant fiber, with significantly enhanced lifetimes in moisture laden atmospheres. As we will see below, this occurs for example in carbon coated fibers, which provide a significant barrier to any moisture from permeating to the surface of the glass fiber.

Experimental Description

Fiber Processing

The optical fiber of this study was manufactured from a high purity glass preform using the draw tower method. Thin layers (a few hundred Angstroms thick) of carbon were deposited via the Chemical Vapor Deposition (CVD) process on the surface of the freshly drawn glass, and then the fiber was overcoated with a polyimide protective material to allow for high temperature testing. The quality of the deposited carbon was monitored and exhibits great consistency over very long draw runs; this consistency was also confirmed by sampling different sections of the drawn fiber, and measuring the thickness of the carbon using a high resolution SEM and its roughness using AFM. The process parameters for the carbon deposition were adjusted to provide optimal hermeticity and consistency of the coating.

High Pressure Hydrogen Vessel

In order to simulate extremely harsh environments, a high pressure, high temperature, hydrogen setup was constructed allowing on-line measurements of hydrogen induced loss in the optical fiber. The H₂ setup is illustrated in Fig. 1. The apparatus consists of a stainless steel chamber with high pressure optical fiber feed-throughs, a white light source, and an Optical Spectrum Analyzer (OSA). The data was collected electronically in real time in intervals of several minutes. The chamber is designed to be pressurized up to 2000psi (of H₂) and heated to temperatures as high as 200°C.

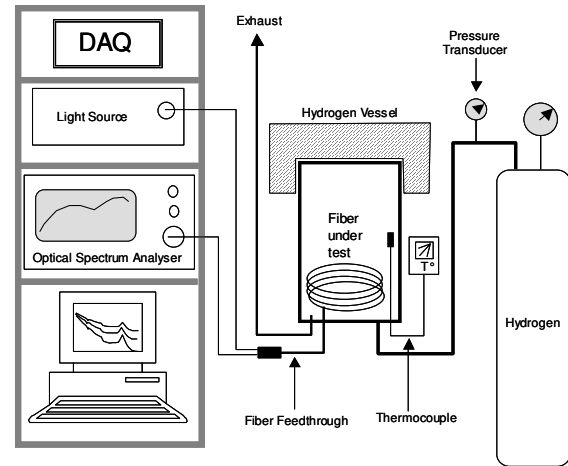


Figure 1: Real-Time Hydrogen Ingression Test Setup

Dynamic Fatigue Test (DFT)

Dynamic fatigue tests were performed on long lengths of fiber (14meters gauge length). In comparison with the two-point bend test, the tensile test for dynamic fatigue analysis is more representative for actual application. The DFT experiments were performed at a number of different strain rates ranging from 2.5MPa/s to 881MPa/s. The DFT setup used in this study is shown in Figure 2.



Figure 2: Long-Length Tensile Tester

High Pressure / High Temperature Water Test

Figure 3 describes the set-up used for performing high pressure, high temperature water tests on the hermetic optical fiber. Typically the fiber sample is wrapped on a 1/4" stainless steel mandrel with minimum tension. The approximate stress on the fiber due to bending on the mandrel is 200kpsi. The fiber is entirely immersed in water and the chamber is then pressurized with compressed air at 1000psi. The chamber is then rapidly heated to 150°C, and typically held at this temperature for 24 hours.

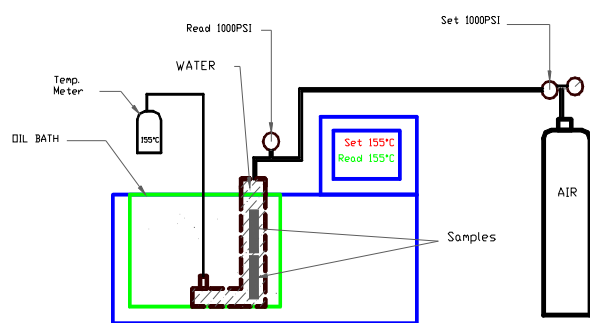


Figure 3: High P / High T Water Test Setup

Results and Analysis

Hydrogen Ingression

Figure 4 shows a spectral scan taken from two sets of a Graded Index Multimode 50/125 Fibers (MM50/125). Sample 1 was a standard MM50/125 polyimide-coated fiber (without carbon). Sample 2 has a hermetic carbon coating on the glass surface. It is evident from Figure 3, that the carbon coated fiber exhibits very small or no change in spectral attenuation characteristics, whereas Sample 1 has become “dark” over most of the spectral range of interest. Clearly the carbon coated fiber impedes the permeation of hydrogen into the glass fiber.

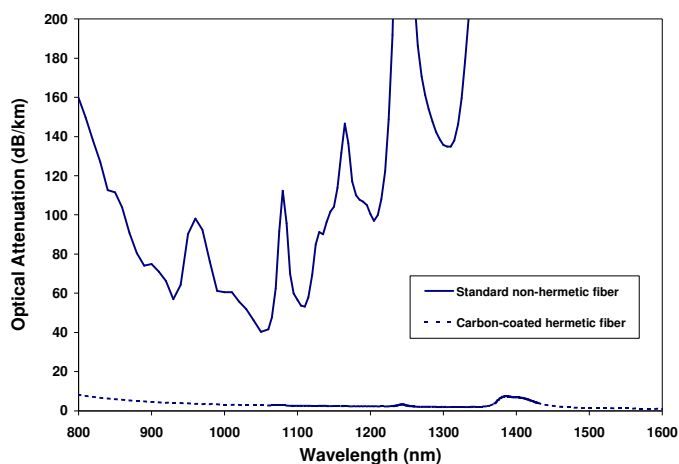


Figure 4: Optical Spectra of a standard MM50/125 fiber compared to a carbon-coated hermetic fiber after being tested at 200°C and 1500psi of H_2 for 17 hours

Figure 5 shows a spectral scan of carbon-coated single mode fiber before and after being exposed to hydrogen at 1500psi and 150°C. There is a small change in the spectrum at 1.24μm, characteristic of molecular hydrogen, and a slight increase in the magnitude of the Ge-Si:OH peak ~1.39μm. The growth of the molecular hydrogen peak is actually used to determine the value of τ_F . In the particular case illustrated by Fig. 5, at 150°C, τ_F is extrapolated and its value was calculated to be 1.5×10^5 hours. Using the on-line monitoring

system, one is able to visualize clearly the time dependence of the growth of different parts of the spectrum, Fig. 6 shows an on-line scan of the time dependent growth of attenuation at 1.24μm. A series of experiments performed at extracting τ_F at different temperatures give rise to different $\tau_F(T)$'s; these are then plotted as a function of $1/T$ on a logarithmic scale to extract the activation energy for τ_F . The plot is shown in Figure 7. This plot enables us to extract the diffusion characteristics of the hydrogen, through the protective coating at any temperature and pressure.

As emphasized above, larger values of τ_F imply higher resistance to hydrogen ingress. A comparison of τ_F available from previously published studies⁸ indicates that the values of the present study are significantly greater than those reported in prior studies by almost an order of magnitude. It is important to note that τ_F may be increased significantly by increasing the thickness of the carbon coating – however, this is often accompanied by a corresponding significant decrease in the fiber mean strength and Weibull fatigue characteristics. The value of τ_F quoted here, therefore corresponds to fibers optimized with respect to both hermetic and mechanical performance.

While the ability to predict the amount of hydrogen that has diffused through the carbon coating is very valuable, Figure 4 clearly shows that after hydrogen ingress, the spectrum exhibits multiple strongly absorbing windows, some of which are the result of the reaction of hydrogen with the silica matrix and with the dopants in the core of the fiber. Additionally, as is evident from Fig. 4, different regions of the spectrum are affected differently by the in-diffusing hydrogen. Therefore, in order to predict the lifetime of the fiber, one needs to determine the maximum acceptable loss at any specific wavelength, and then determine the kinetics of absorption growth for a given temperature and hydrogen pressure. By fitting the growth behavior to the appropriate functional form, it is possible to extrapolate to long-term behavior of the fiber. While this approach is empirical, it provides guidance on the functional form based on assumptions regarding the kinetics of reaction of H with Si-O and Ge-O sites in the glass. The on-line monitoring capability provides invaluable and extensive data that allowed us to make these predictions with a high degree of confidence.

For example, information extracted from this study predicts the lifetime of our carbon-coated optical fiber to be greater than 7 years in a continuous operation at 125°C in 1atm of hydrogen.

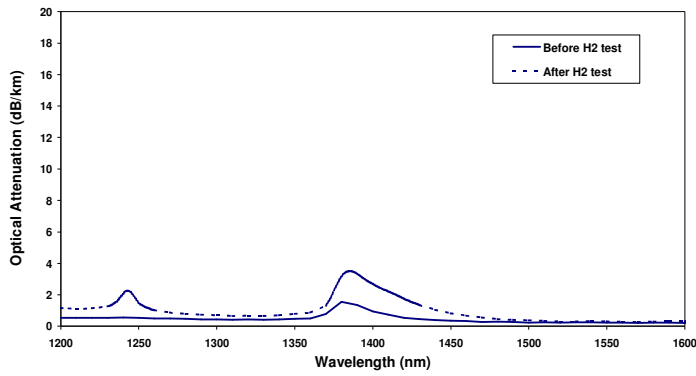


Figure 5: Attenuation Spectrum for Single-Mode fiber before and after hydrogen test

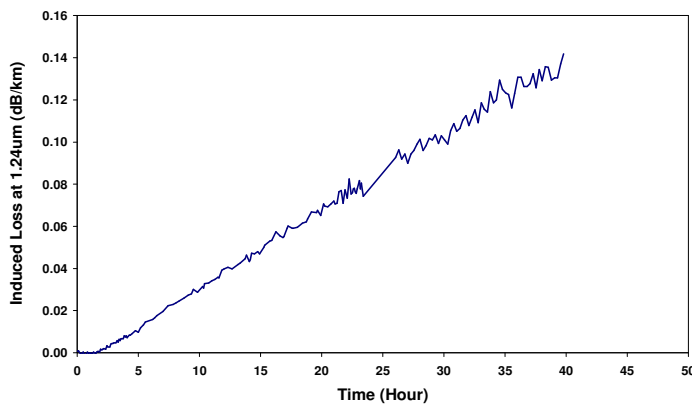


Figure 6: Induced loss at 1.24μm as a function of time for a carbon-coated single-mode fiber tested at 150°C and 1500psi of H₂ pressure

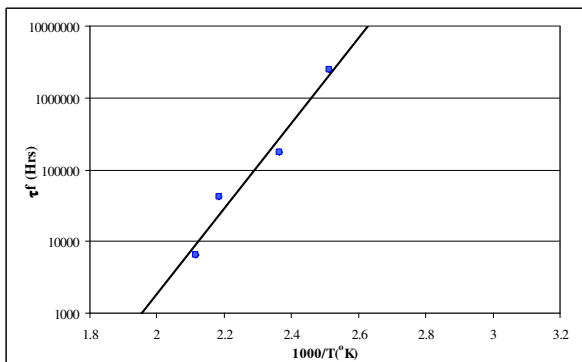


Figure 7: Temperature Dependence of Permeation Time Constant

The value of carbon coatings in protecting optical fibers against molecular hydrogen ingress is therefore clearly evident from the present findings.

Fiber Strength

Weibull plots for the carbon-coated fiber are shown in Figure 8. The gauge length for this fiber was 14 meters, this allowed us to better sample the tails of the fiber strength distribution. It is very interesting to note that the Weibull plots of fibers pulled at different strain rates are literally superimposed to

each other, which is not the case for non-hermetic fibers, where these individual curves are distinctly separated. This result confirms our assumption that carbon coated fibers of this study provide a very hermetic barrier to moisture, thus the enhanced crack propagation that takes place due to moisture ingress, regardless of strain rates, no longer occurs with the carbon-coated fibers.

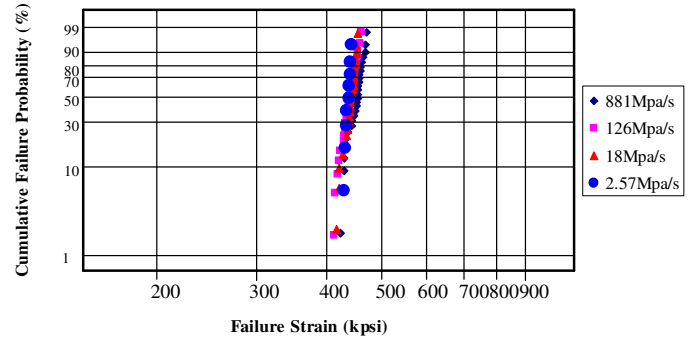


Figure 8: Weibull Plots for Carbon-Coated Fiber (14m gauge)

Stress corrosion parameters extracted from the above experiment indicate very large n -values ≥ 240 . Typical non carbon coated fiber exhibit n -values ~ 20 . Hence the reliability of carbon coated fibers is increased significantly, especially in high moisture environments. This is further exemplified in the high pressure water exposure experiments described in the following section.

High Pressure/High Temperature Water Exposure

As described previously, four meter lengths of both, carbon coated and non-carbon coated fiber were wound on multiple 1/4" stainless steel mandrels. These mandrels were then immersed completely in a vessel filled with de-ionized water, the vessel was pressurized to ~ 900 psi, and the temperature then ramped up to 150°C (resulting in a final pressure of approximately 1000psi). The system was stabilized at these temperature and pressure for 24 hours. After 24 hours the system was shut down and returned to room temperature and atmospheric pressure. The results of this experiment are strikingly contrasted in the photographs of Fig. 9(a) and 9(b). The carbon-coated fibers, in Fig. 9(b), are clearly very resistant to moisture ingress and fatigue. Whereas the non-carbon coated fibers shown in Fig. 9(a), have all fractured, under the identical conditions (150°C, 1000psi of water). This test was repeated on many samples from different fiber draw runs and confirmed the same behavior of the tested fibers.

Based on this result, it is clear that the use of carbon-coated optical fibers in high humidity and elevated temperature environments provide a major reliability advantage in applications such as steam injected oil wells.

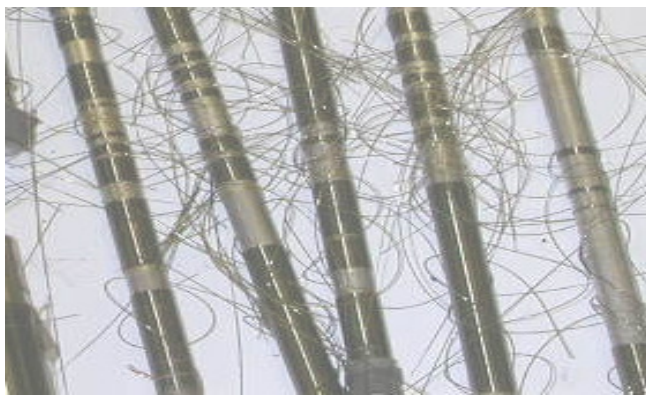


Figure 9a: Non Carbon-Coated Fiber (after water test)



Figure 9b: Carbon-Coated Fiber (after water test)

Conclusion

We have studied and presented in this work two key aspects for the users of optical fibers in harsh environments: resistance to hydrogen ingress and mechanical reliability. While both of these aspects have been addressed in the past by a variety of investigators, the purpose has always been on the reliability of fibers in telecommunications applications such as submarine cables, where the hydrogen partial pressures and temperatures are more clement. In our study the focus has been on developing a robust optical fiber that withstands downhole harsh environment, where temperatures, pressures and corrosive chemicals render standard optical fibers unusable.

We have outlined some of the analytical methods and experimental tools, such as in-situ hydrogen ingress monitoring capability; tools that we believe are absolutely necessary to fully understand and predict the behavior of optical fibers in high temperature high pressure hydrogen containing environments.

To this end, we have described our advanced capabilities in producing a highly hermetic optical fiber and in testing it under simulated downhole conditions. Our monitoring capability of loss changes at elevated temperatures and hydrogen pressures for extended periods as well as our mechanical testing, demonstrated that our carbon-coating provides significantly extended lifetime at temperatures up to 150°C. A very large portion if not the majority of oil wells

operate at temperatures below 150°C, which makes the fiber of this study very suitable for deployment in a wide range of wells both in logging and permanent installations.

We have also demonstrated a significant improvement in the long term mechanical properties of the fiber exemplified by a stress corrosion parameter greater than 150. This further helps the optical fiber users in the deployment and long term operation where fiber failure is usually a concern.

Acknowledgements

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