

# Fe-Coated Optical Fiber for Distributed Corrosion Monitoring in Soil and Aqueous Environments

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## Abstract

Natural gas pipeline corrosion represents a substantial cost and safety concern during normal operations. The effective and real-time monitoring of corrosion is important to detect and mitigate pipeline risks before corrosion-related catastrophic events happen. Here, we describe the use of iron (Fe)-coated optical fiber sensors (OFS) for distributed corrosion monitoring where Fe acts as a corrosion proxy. By using an optical backscattering reflectometer (OBR), corrosion was monitored based on the increase in the backscattered intensity amplitude of the light being passed as Fe underwent corrosion. The Fe-coated OFSs were prepared with a film thickness between 25–225 nanometers (nm) by an electroless plating approach and corroded by a carbon dioxide (CO<sub>2</sub>)-saturated acidic electrolyte. The corrosion rate (CR) was approximately 2.5 millimeters (mm)/year for Fe with a film thickness of 30 nm and increased with film thickness. Backscattering-based CRs were supported by visible light transmission measurements, which have been previously demonstrated. Additionally, a correlation between the intermittent transmission and residual Fe film thickness during the corrosion of Fe was established. The corrosion was measurable out to > 100 meters (m), which is a significant improvement over our previous work, which showed corrosion sensing at < 10 m. The corrosion sensor was also tested in soil at > 1 foot depth. Fe-coated OFS was loaded into a Draka cable, which provides mechanical support during fiber deployment and installation into either acidified soil, top-soil, or sandy soil (50/50). The corrosion was determined to be faster in acidified soil relative to top-soil and sandy soil when using this method.

**Keywords:** Optical fiber, natural gas, humidity, distributed sensing, corrosion

## Introduction

Natural gas production, processing, and transmission pipeline materials are mainly composed of iron (Fe) or steel, which are prone to corrosion under operating conditions.<sup>1-4</sup> Corrosion is a thermodynamically favored electrochemical process, which is initiated and sustained due to the presence of condensed water, carbon dioxide (CO<sub>2</sub>), hydrogen sulfide (H<sub>2</sub>S), and dissolved salts.<sup>5-9</sup> The United States (U.S.) encounters billions of dollars (approximately \$6 billion) loss annually due to corrosion.<sup>10, 11</sup> In certain instances, catastrophic events have also been reported due to pipeline failure caused primarily by corrosion.<sup>10, 12</sup> Therefore, it is very important to have real-time monitoring of corrosion in these infrastructures, so preventive measures would be applied before the actual corrosion-related events happen.

The common conventional methods of monitoring corrosion include electrochemical sensor probes, corrosion coupons, electrical resistance probes, ultrasonic testing sensors, magnetic flux leakage, pipeline inspection gauges, and electromagnetic sensing.<sup>4</sup> The majority of these techniques suffer from issues, such as not being useful for real-time monitoring, low sensitivity, being limited to surface detection, and low cost effectiveness.<sup>4, 11, 13</sup> Because of this, the optical fiber sensing (OFS) platform is being progressed more recently due to its advantages, such as its small size, light weight, flexibility, improved safety in the presence of inflammable gases, inherent immunity to electromagnetic interference, and long-range and distributed sensing capabilities<sup>4, 11, 14</sup> Furthermore, the OFSs are easy to deploy and install along the long-distance natural gas transmission pipelines.<sup>15</sup> Therefore, there is growing interest in the use of OFSs for pipeline monitoring in the U.S. and globally.

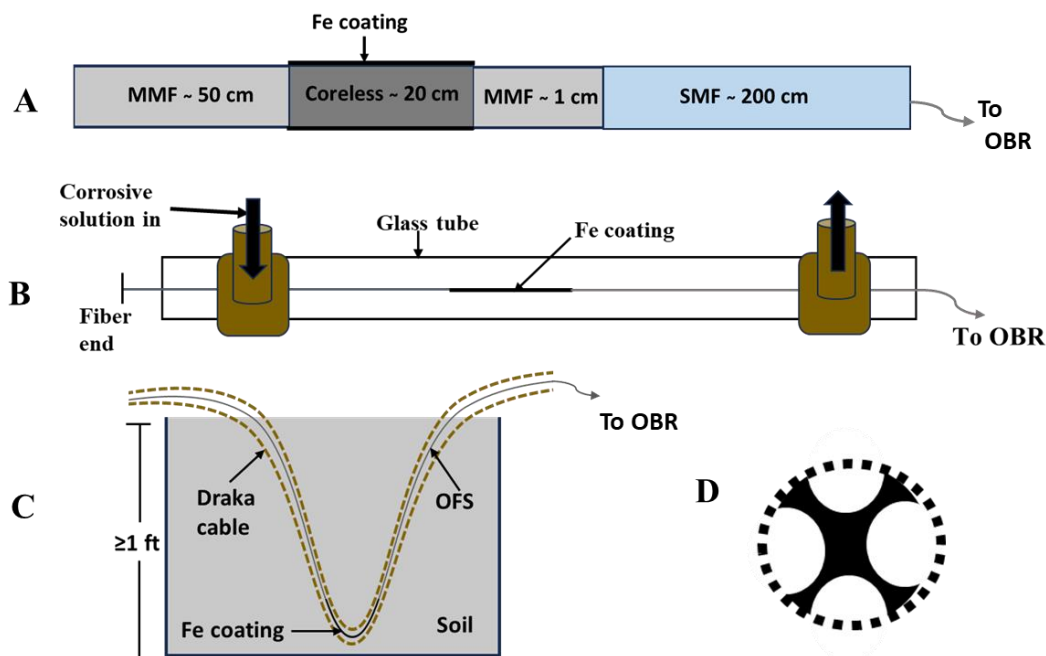
OFS have been modified differently depending upon the sensing purpose. For example, some groups have used optical fibers with a variety of sensing materials, such as polymers, hydrogels, metal oxides, and carbon nanotubes.<sup>5, 11</sup> A distributed humidity and gas sensor has been developed by employing a polyacrylate coated, single mode fiber (SMF) by our group.<sup>16</sup> The polymer material has the ability to absorb moisture, which undergoes swelling and can produce strain change and hence enables humidity and gas sensing.<sup>11, 17</sup> Accordingly, the fully distributed sensing of humidity is carried out based on the quantification of humidity-induced strain changes.<sup>16-18</sup> Besides humidity, the Fe coated on to the coreless fiber section of the OFS has been demonstrated to be useful for monitoring corrosion under high pressure natural gas pipeline relevant conditions, ambient air, or in concrete structures.<sup>18, 19</sup> Because there is no core-cladding separation in coreless fiber, laser light leaks out of the glass boundary while passing through it and generates an evanescent field. Removal of the jacket of the coreless fiber, followed by coating of a light absorbing material (e.g., Fe, Fe carbide (FeC)), absorbs light from the coreless fiber section. Conversely, the corrosion of the deposited Fe/FeC results in a gradual lowering of extent of the light absorption and hence increases the amplitude of the backscattered light being passed. This type of fiber design and mechanism of corrosion monitoring using Fe as corrosion proxy has been successfully demonstrated to serve as a corrosion sensor.<sup>5</sup> A special advantage of these sensors is they simultaneously allow monitoring of humidity and corrosion with location information along the entire length of OFS alongside the pipe.<sup>5, 17, 18</sup>

Most of the literary works indicate that the use of OFS for corrosion monitoring has been carried out in ambient air, under high pressure conditions, or in concrete structures. However, there is little or no work that describes corrosion monitoring in aquatic environments or in soil. Monitoring corrosion under these environments is of especial importance to mimic the corrosion monitored in wellbore or buried pipeline conditions. In this work, the previously developed approach of distributed corrosion monitoring has been extended for corrosion monitoring in a liquid and in soil. The corrosion was monitored by using a thin Fe film coated onto the coreless fiber section of the OFS, which acted as a corrosion proxy. An increase in the backscattered intensity amplitude of the light being passed indicated corrosion, which was measured by optical backscattering reflectometer (OBR). The corrosion rate (CR) was measured based on the rate of change in the light intensity amplitude until attaining steady state, as Fe with a specific film thickness underwent complete corrosion. CRs predicted by the OBR measurement were then supported by the results obtained by the transmission mode of measurement. Regarding the corrosion monitoring in soil, the fragile-natured OFSs were reinforced by a supporting cable to prevent the sensor from mechanical damage or disturbance during fiber deployment and installation.

## Experimental

### Design of Optical Fiber Corrosion Sensor

Commercially available coreless fiber is mostly composed of approximately 125 micrometer ( $\mu\text{m}$ ) silicate glass and 250  $\mu\text{m}$  polymer jacket in diameter. For these experiments, Fe was coated onto the coreless fiber section by an electroless approach by following the similar protocol, as in the several publications by our group previously.<sup>12, 18</sup> Fe coating with variable coating thickness was carried out by simply immersing the activated coreless fiber section into the Fe plating bath for 1, 2, 3, 4, and 5 minutes. The thickness of the Fe coating was then measured by scanning electron microscopy (SEM) images. As shown in Figure 1(A), the Fe coated fiber section (20 centimeters (cm)) was then spliced to approximately 50 cm of multimode fiber (MMF) on one end and 1 cm of MMF on another end. The shorter MMF end was then connected to an SMF. The OFS sensor was then loaded inside a water-tight glass tube provided with an inlet and outlet of  $\text{CO}_2$  saturated aqueous 3.5% sodium chloride (NaCl) plus hydrogen chloride (HCl) with pH 3.2, as shown in Figure 1(B). The OFS sensor was finally connected to a LUNA 4600 OBR through the SMF end.



**Figure 1:** Schematics of the experimental setup for corrosion monitoring in this work. (A) Design of OFS consisting of Fe-coated coreless fiber section spliced together with MMF on either end. (B) Fe-coated OFS installed inside a glass tube for corrosion experiments in liquid. (C) Fe-coated OFS was first loaded onto a Draka cable and then buried in soil. (D) Schematics of the cross-sectional view of the Draka cable used in Figure 1(C). In each case, the SMF connected to the Fe-coated fiber is finally connected to an OBR, which provides a spatial profile of light intensity amplitude along the fiber section chosen.

Results about corrosion monitoring, as measured by the OBR, were supported by the results obtained by corrosion experiments in transmission mode. For this, approximately 10 cm of the Fe-coated coreless fiber section was spliced to approximately 75 cm MMF on either end. The resultant

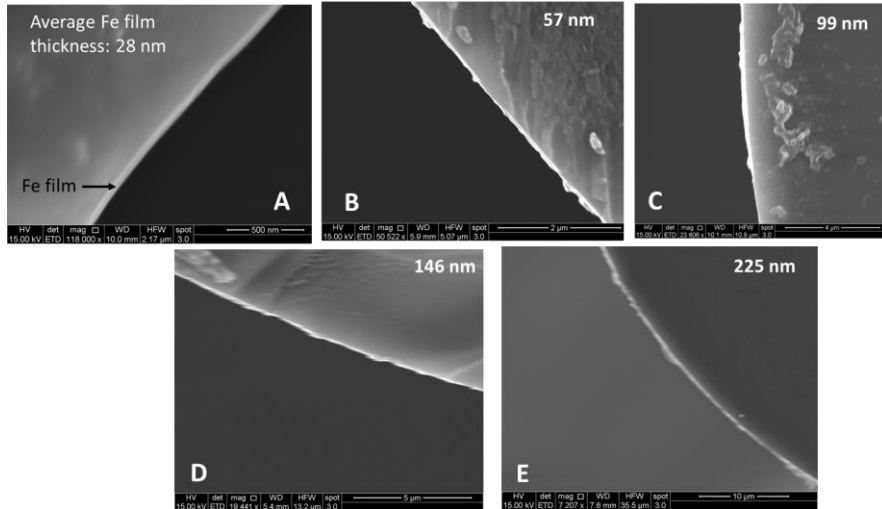
fiber was then loaded onto a glass tube provided with a flow of a corroding liquid in and out in a similar way as in Figure 1(B). The intensity of the laser light being passed through this fiber during the corrosion of Fe was then monitored by using the Ocean Optics Jaz spectrometer.

The similar optical fiber design was used for corrosion experiments in soil with the fiber buried to about 1 foot depth. Figure 1(C) illustrates the design of the OFS for corrosion monitoring in soil. The fragile OFS sensor was first loaded onto a Draka cable and then installed into the soil. The Draka cable provides necessary mechanical support to the Fe-coated OFS, preventing it from physical disturbance or damage while burying into the soil, as well as during corrosion monitoring. The three types of soil that were used for this experiment were top-soil, sandy soil (50/50), and soil acidified with calcium sulfate ( $\text{CaSO}_4$ ).

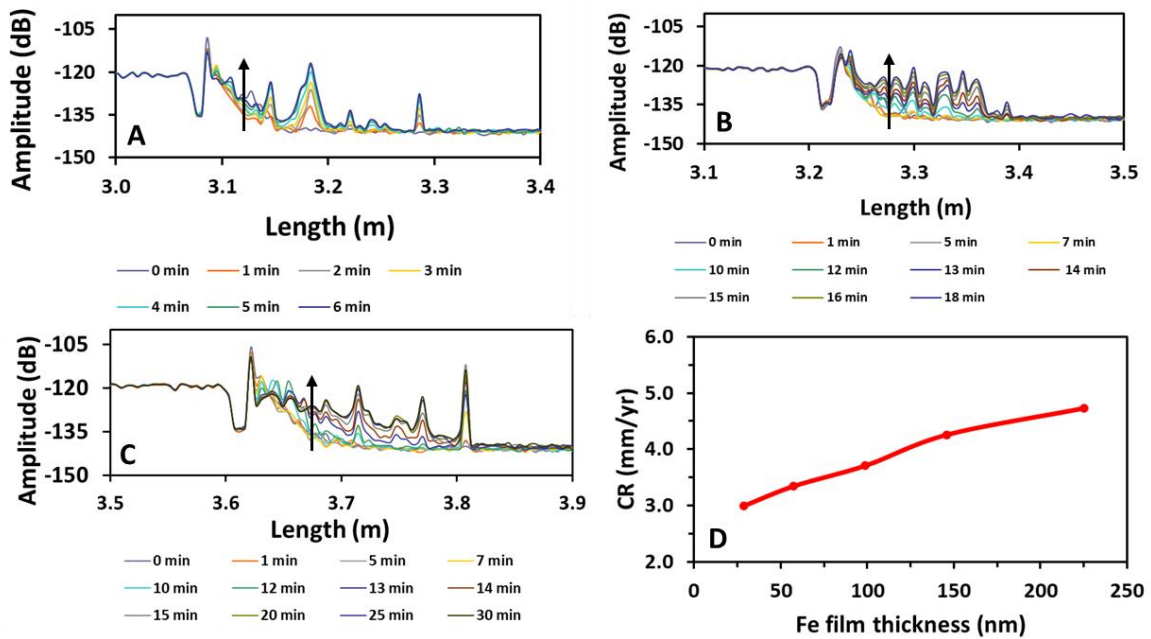
## Results and Discussion

### Corrosion Monitoring in Aqueous Phase

Metallic Fe-coated OFSs are useful for distributed corrosion monitoring under high pressure or in ambient air as reported in some of our previous publications.<sup>12, 17</sup> Coating of Fe onto the OFS section was carried out by an electroless approach simply by immersing the unjacketed portion of the coreless fiber section into a basic  $\text{Fe}^{2+}$  solution using sodium borohydride ( $\text{NaBH}_4$ ) as the reducing agent and trisodium citrate as the stabilizing agent. Accordingly, the Fe films with an average thickness of 28, 57, 99, 146, and 225 nanometers (nm) were developed onto the unjacketed portion of the coreless fiber section. The Fe film thickness was estimated from the SEM images of the finely cut cross-section of the Fe-coated fiber section sputtered with 20 nm gold palladium (AuPd) before imaging, as shown in Figure 2(A–E). The coreless fiber was then spliced together with the MMF on either end and finally connected to an OBR. The capability of the OFS for corrosion monitoring under high pressure or in ambient air was extended for studying corrosion in the aqueous phase. The Fe-coated OFS was first loaded into a glass tube designed with a flow of corrosive solution. After the installation of the OFS into the glass tube, corrosive solution was flowed into it, and an OBR trace was taken immediately. Upon continuous exposure of the fiber to the corrosive solution, an increase in amplitude of backscattered intensity of the light being passed along the Fe-coated fiber section was observed, indicative of corrosion. The backscattered intensity amplitude further increased with time and eventually reached a stable value with a negligible amplitude change after that point. Figure 3(A–C) shows changes in the backscattered light intensity amplitude with time until the saturation state is reached once complete corrosion of Fe takes place. A thicker Fe coating required a longer time to reach a steady state as compared to the thinner one, indicating a strong dependency of the corrosion time with Fe coating thickness. The corrosion time was measured as the time required to attain stable backscattered light intensity amplitude when the Fe coating was completely removed. Using the information about corrosion time and the Fe film thickness, the average CR was estimated. It was found that the CR increased with the Fe film thickness, as shown in Figure 3(D). An increase in CR with an increase in Fe film thickness could be explained in terms of the variable nature of surface morphology of Fe at different film thickness. Because the surface acquires more roughness at higher film thickness during electroless coating, the CR is found to increase with the surface roughness due to increased area of exposure. However, we do not have a detailed analysis of surface roughness and surface area or its correlation to the CR at this point.



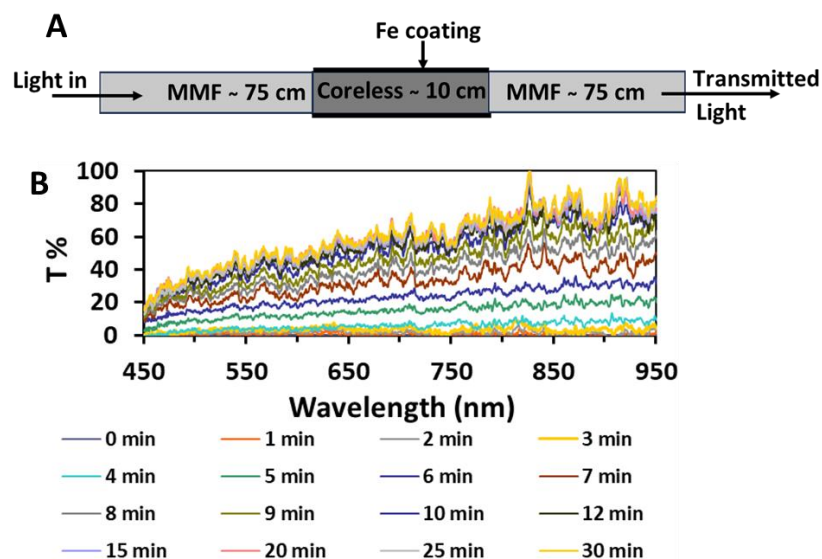
**Figure 2:** Representative SEM images of the cross-section of the Fe-coated coreless fiber section prepared by the electroless coating approach. A variable Fe film coating was obtained by immersing the fiber section into the coating solution for different times: (A) 1 minute, (B) 2 minutes, (C) 3 minutes, (D) 4 minutes, and (E) 5 minutes. The corresponding average Fe film thickness in each case is provided.



**Figure 3:** OBR traces showing changes in backscattered light intensity amplitude with time caused by corrosion of Fe coated onto the coreless fiber section with variable thickness (A: 28 nm, B: 146 nm, C: 225 nm) and CR as a function of Fe film thickness (D). Corrosion was studied in CO<sub>2</sub> saturated 3.5% NaCl plus HCl, pH 3.2 solution using OBR.

In order to confirm the results obtained by amplitude-based corrosion monitoring of Fe in aqueous phase, transmission-based corrosion monitoring was performed by following the approach

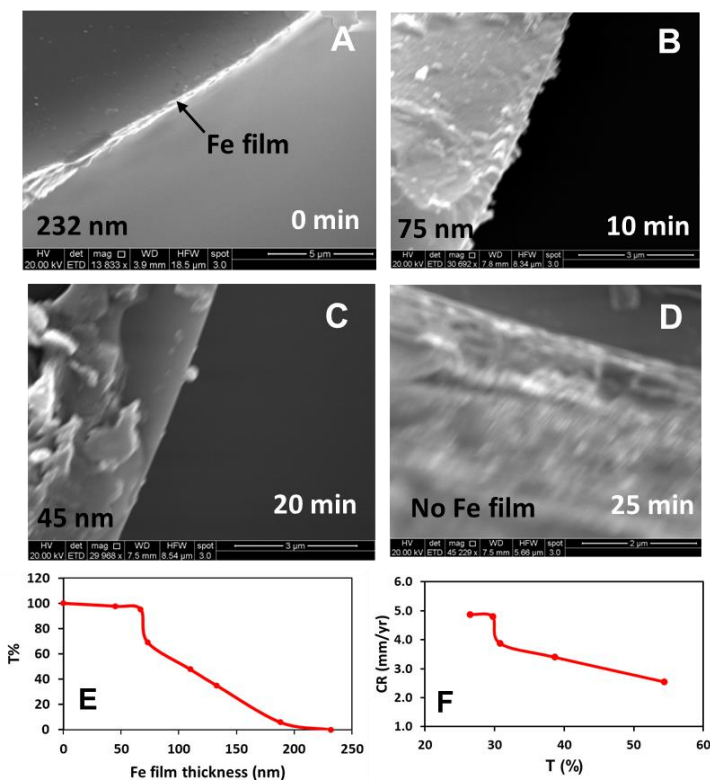
as reported in the literature.<sup>20</sup> As discussed in the experimental section, transmission based corrosion monitoring was carried out by analyzing the intensity of transmitted light passing through the Fe-coated optical fiber, as shown in Figure 4(A), where the Fe-coated coreless fiber section was spliced together with the MMF on either end. Similar to the case of backscattering of light, metallic Fe absorbed visible light (450–950 nm) when coated onto the coreless fiber section and lowered the intensity of the light being transmitted through it. The intensity of the transmitted light during corrosion of Fe exposed to the corrosive solution was analyzed by using an Ocean Optics Jaz spectrometer. As the thin Fe film underwent corrosion, the amplitude of transmitted light gradually increased and attained saturation state when corrosion was completed. Figure 4(B) shows a variation of the normalized intensity of the transmitted light as a function of time. The CR was measured by measuring the rate of change of the transmitted light. The CR measured by the transmission mode was found to be similar to the one measured by the OBR mode under the same corrosive environment. The transmission mode of monitoring corrosion provided information about the total transmission of light at a specific Fe film thickness, which would then be correlated to the CR.



**Figure 4:** Design of experiment for monitoring corrosion of Fe in transmission mode (A) and normalized transmission (T%) of laser light in the visible range (450–950 nm) with time (B).

The transmission mode of corrosion measurement was further extended to analyze the residual film thickness of Fe taking place during corrosion. Because Fe-coated OFS could not be intermittently taken out for SEM measurement during transmission measurement, a parallel Fe-coated optical fiber sample was immersed in the same corrosive solution. SEM analysis of a piece of Fe-coated optical fiber sample was then carried out by sampling it out of corroding solution at different instants of time. By analyzing the SEM images of the Fe-coated fiber surface undergoing corrosion, it was possible to determine the instantaneous Fe film thickness and its correlation to transmission. Figure 5(A–D) shows representative SEM images of the cross-section of the Fe-coated fiber section during transmission measurement. The average residual Fe film thickness in each case decreased with time. At the same time, an increase in transmission of the light was observed, showing strong correlation between the transmission of light and the Fe film thickness. Figure 5E shows the variation of the transmission of light at a wavelength of 600 nm with a change in Fe film thickness. Clearly,

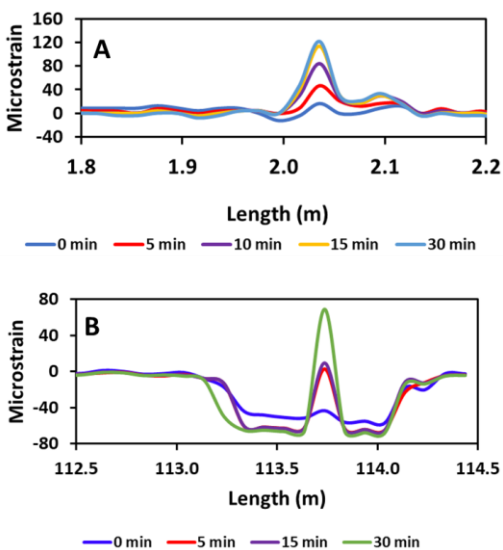
transmission decreases with increasing Fe film thickness with a sharp decrease in transmission (%) being observed at approximately 70 nm thickness of Fe film. This shows a close agreement with the Fresnel's theoretical calculation, showing the variation of the transmission of light with the Fe film thickness with a sharp decrease in the transmission at approximately 50–60 nm Fe film thickness.<sup>20, 21</sup> Based on the transmission data and the Fe film thickness measured at different instants of time during corrosion, the corrosion rate of Fe is predicted directly from the transmission data (Figure 5F). However, due to saturation of transmission below approximately 60 nm Fe film thickness and negligible transmission at and above around 200 nm Fe film thickness, the CR could not be predicted throughout the transmission range of 0-100%. Nevertheless, by simply knowing the transmission data, corrosion rate of Fe could be predicted based on this approach.



**Figure 5:** Representative SEM images of the cross-section of Fe-coated OFS measured at different instants during corrosion of Fe in CO<sub>2</sub> saturated aqueous 3.5% NaCl plus HCl solution with pH 3.2 (A) 0 minutes, (B) 10 minutes, (C) 20 minutes, and (D) 25 minutes. Transmission (T%) of light along the Fe-coated OFS at wavelengths of 600 nm as a function of Fe film thickness (E) and corrosion rate predicted at different transmission values (F).

Amplitude-based corrosion monitoring employs analysis of backscattered light when passed through the fiber sensor. An accurate analysis of backscattered intensity amplitude greatly suffers from signal loss and splice loss when the sensing distance from the source is extended significantly. One of the approaches we developed in this scenario involved the use of strain-based corrosion sensing. Our group previously employed nickel (Ni)-coated SMF, which demonstrated significant strain change during corrosion in an acidic environment.<sup>22</sup> A decrease in microstrain was observed during the electroless plating of Ni onto the stretched SMF, while a positive microstrain was observed during the

corrosion of the plated Ni. Here, a follow-up of this work was carried out by first depositing Ni onto a stretched SMF by an electroless approach inside a glass tube. Exposure of the Ni-coated SMF to 3 M HCl caused an increase in microstrain at about 2.0–2.1 m fiber length, indicating corrosion, as shown in Figure 6(A). This clearly shows a relaxation of the Ni-coated SMF occurred once Ni was corroded. The corrosion sensing range was then extended to above 100 m, shown in Figure 6(B). Also, in this case, a positive microstrain was observed as compared to the uncoated fiber section once the Ni coating was corroded. However, the pattern of microstrain change in the long-distance sensing range was slightly different from the one monitored for the short distance sensing range. We will continue working for more detailed analysis of corrosion monitoring for the long-distance corrosion sensing using this strain-based approach.

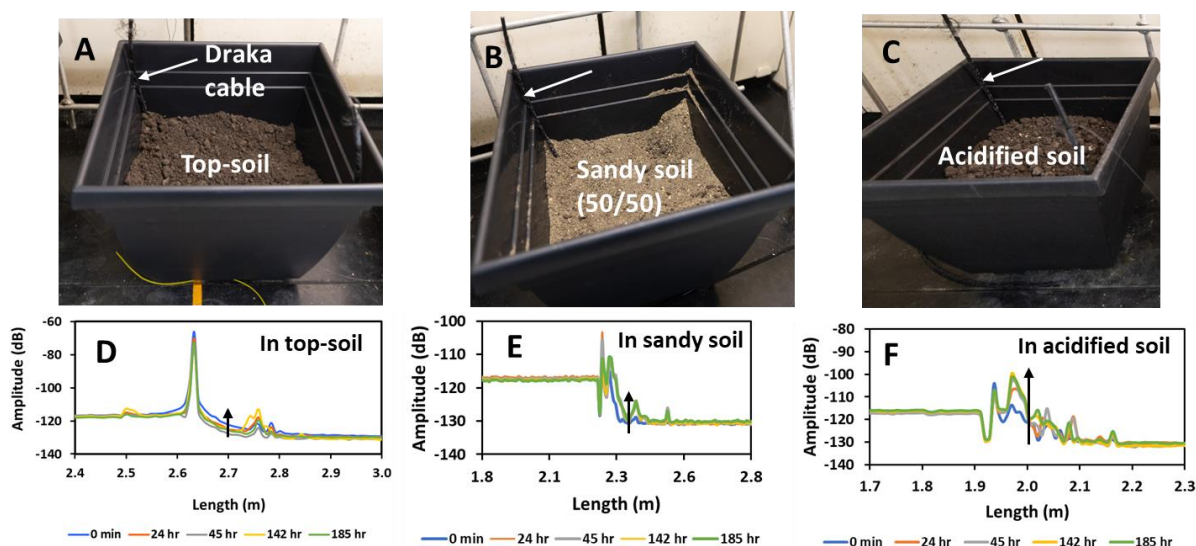


**Figure 6:** Microstrain changes of the Ni-coated SMF when exposed to the 3M HCl as a function of time for  $< 3\text{ m}$  (A) and  $> 100\text{ m}$  (B). The Ni coating on to the stretched SMF was carried out inside the same glass tube designed with a controlled flow of a liquid and where the corrosion of the deposited Ni was also studied.

## Corrosion Monitoring in Soil

The corrosion monitoring in soil needed additional effort as compared to the experiments performed in air or in a liquid environment, as soil causes stronger physical blockage while deploying and installing the OFS into it. Due to the delicate nature of the OFS, especially at the splice point and along the Fe-coated fiber section, the OFS should be protected in a way to prevent it from being mechanically disturbed or easily broken during the installation process. Concurrently, the protecting substance should allow easy exposure of the Fe-coated fiber section to the soil, enabling the successful corrosion monitoring. Out of the several commercially available products, the Draka cable was found to be a suitable one, which would provide enough mechanical support on one hand and allow easy access of the Fe-coated OFS to the soil. Similar to the case of corrosion monitoring in a gas or a liquid, the corrosion was studied by using a commercially available, coreless fiber spliced together with the MMF on either end. Metallic Fe with a film thickness of approximately 230 nm was coated onto the

unjacketed coreless fiber section by the electroless deposition approach. As discussed earlier, Fe-coated OFS was then loaded on to a Draka cable, see Figure 7(A–C). Due to technical difficulty and safety concerns, we created the artificial soil environment in the laboratory for the corrosion experiments rather than real field work. Three types of soil were chosen for this study: (1) top-soil, (2) sandy soil (top-soil and sandy soil mixed in a ratio of 1:1), and (3) acidified soil (top-soil acidified with  $\text{CaSO}_4$ ). The soil was enriched with moisture content, dissolved ions, and low pH conditions, which are conducting environments for Fe corrosion to happen. Upon burial into the soil, a change in backscattered intensity amplitude of the light being passed along the Fe-coated fiber section in each case was monitored by an OBR. The corrosion time was measured as the time required to attain saturation of the backscattered light intensity amplitude when the Fe coating was completely removed. Figure 7(D–F) shows changes in the amplitude of backscattered light intensity with time for Fe-coated OFS buried in soil. It was observed that there was negligible change in backscattered intensity amplitude of the light in the case of top-soil when the experiment was performed for up to 185 hours, shown in Figure 7(D). Relative to this, there was a moderate increase in backscattered light intensity amplitude in the case of the sandy soil, shown in Figure 7(E), while it was the highest in the case of acidified soil, shown in Figure 7(F) within the same time frame as in the top-soil. The moderate CR in the case of sandy soil and the highest CR in the case of acidified soil are most likely caused by increased moisture content and lowered pH conditions in these soil types as compared to those in top-soil. Overall, we successfully deployed and installed the OFS in soil and monitored corrosion under different soil conditions.



**Figure 7:** Fe-coated OFS deployment and installation in a set of the three types of soil selected for the corrosion experiments: (A) top-soil, (B) sandy soil, and (C) acidified soil. In each case, OFS was first loaded onto a Draka cable (as indicated by the white arrow) before burying into the soil. Changes in backscattered intensity amplitude of the light being passed as a function of time for the Fe-coated OFS section buried in the three types of soil (D–F).

## **Conclusions**

A simple, fully distributed OFS has been designed and employed for corrosion monitoring in both soil and aqueous environments. This sensor exhibits the advantages of distributed sensing, simplicity, ease of operation, and real-time monitoring. A thin Fe film has been used as corrosion proxy for these experiments where coating of the Fe film is carried out by the electroless approach. The visible light absorbing property of Fe allows it to be used as a corrosion proxy. An increase in backscattered intensity of the light being passed when Fe is exposed to a corrosive environment indicates corrosion of Fe and was measured by OBR. The CR was monitored by monitoring the rate at which the backscattered light intensity amplitude attained a stable state due to completion of corrosion. The time required to attain the stable state was different for different Fe film thickness in the range 25–225 nm. The CR was found to increase with the Fe film thickness, possibly due to increasing Fe surface roughness at higher film thickness. Transmission measurement carried out for corrosion monitoring supported the results obtained by OBR measurements. Additionally, a good correlation between the transmission of light at different Fe film thickness during corrosion of Fe was also established. The corrosion was also monitored in soil where a commercially available Draka cable was found to be useful for the safe deployment and installation of the fiber into the soil, protecting it from mechanical damage or disturbance. Corrosion of Fe was compared among the three types of soil where the CR was fastest in acidified soil as compared to that in top-soil and sandy soil. These approaches and findings will provide valuable information about use of OFS for real-time and distributed corrosion monitoring under harsh environmental conditions in natural gas transmission pipelines and related infrastructures.

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