# Application of optical reflectometer for monitoring corrosion process

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**Abstract**—This work presents a corrosion sensor based on an optical time domain reflectometer. The first sensor with a bare tip was used to measure the corrosion process of silica glass fiber. Another sensor with a deposited silver layer was used to monitor nitric acid's corrosion process. In both cases, reflectance at the fiber's end decreased with immersion time. Thus we can describe the corrosion stage by the level of Fresnel reflectance. The maximum sensitivities of the analyzed sensors were as follows: 0.7 dB/min (3% HF solution) and 0.15 dB/h (5% $HNO_3$ solution). Results showed that the corrosion process in all cases wasn't entirely linear, and all reactions began almost instantly after immersing sensors in tested corrosive environments.

Optical fiber technology is essential to modern telecommunications, providing high-speed connections. The main advantage of this technology is high bandwidth, low transmission losses, and the possibility of multiplex information [1–2]. However, some researchers focused on developing fiber-optic chemical sensors (FOCS) as an alternative to electronic sensors. After many years this technology became well developed and popular due to its reasonable costs, small size, accuracy, and capability of performing measurements at inaccessible sites [3].

A great example of the beginnings of FOCS are studies by M.A. Butler focused on the fiber-optics hydrogen sensors with deposited palladium film [4–5]. He paves the way toward new solutions by presenting a simple intensity-based sensor with a palladium layer on tip of the fiber and another interferometric based on the Mach-Zehnder interferometer (MZI). Except two mentioned before, we can classify one more type of sensor - fiber grating-based sensor [6]. Over time, researchers presented accurate and simple solutions like methane sensors based only on optical time-domain reflectometry (OTDR) [7]. This work presented a considerable advantage of optical fiber sensors – the possibility of making a distributed network of sensors on a large area. It's worth of mention that this solution doesn't need any additional sensing layer. The only thing needed is proper laser wavelength matching absorption spectra of methane. Chemical reactions like corrosion are also worth monitoring. Pipelines or oil rigs are often exposed to corrosion environments like saltwater. Long distances between measure points make the commercially available instruments limited solutions in these conditions [8]. Some electronic corrosion sensors were presented in the past [9–10], but they aren't capable of sensing in mentioned conditions.

In this paper, we proposed and demonstrated an optical fiber corrosion sensor capable of sensing long distances using OTDR. Our solution allows us to measure silica glass and silver corrosion in harsh conditions and can be used for other materials such as aluminum or iron.

Two versions of sensors were prepared to measure corrosion in acid environments. Both of them use the same commercially available SMF28 optical fibers. The first optrode was fabricated by stripping all the coatings at the end of the fiber and cut with a fiber cleaver. This sensor allowed us to measure the corrosion process of silica glass in three different concentrations of water solutions of hydrofluoric acid. The second sensor was prepared to measure corrosion of the silver layer deposited at the end of the fiber (Fig. 1). The first steps of fabrication were the same (stripping, cutting); after that, silver, the layer was deposited using the Tollens test. The process of depositing silver can be divided into three steps. The first step was preparing three water solutions: 0.042 g $AgNO_3$ in 2 ml of water, 0.028 g of KOH in 1.4ml of water, and 0.028 g glucose in 0.4 ml of water. In the next step, KOH and $AgNO_3$ solutions were mixed turning into a grey fluid. After that ammonia was added to the test tube and mixed well to clarify the obtained solution. Finally, glucose was added to the solution, and fiber tips were immersed in depositing a silver layer on optical fiber. The sensor prepared in this way was exposed to 1% and 5% nitric acid ($HNO_3$) solutions, allowing monitoring of the silver corrosion process.

Fig. 1. Photo of the silver layer deposited on end face SMF28 optical fiber with the use of Tollens test.

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The measuring system consists of OTDR (Noyes OFL280 FlexTester), 5 km of SMF28 connected with another 60 m of single-mode fiber with prepared optrode immersed in a corrosion environment (Fig. 2). Corrosion of silica glass was measured for one hour for three HF solutions. For the second sensor with the silver, layer measurements took almost 241 hours for 5% $HNO_3$ and over 900 hours for 1% solution.

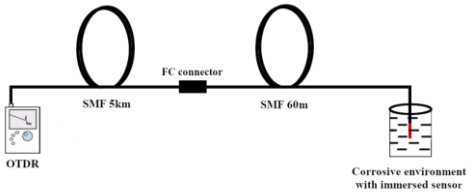


Fig. 2. Measuring setup.

First, we measured the corrosion process in silica glass fiber in laboratory conditions. As mentioned before, as an etching reagent, we used three hydrofluoric acid solutions. The reaction of silicate glass with HF solution can be described as follows [11]:

$$SiO_2 + 4HF \rightarrow SiF_4 + 2H_2O \quad (1)$$

We used OTDR with a laser operating at a wavelength of 1550nm, with a 30ns pulse duration and averaging time of the 30s. Reflectance at the end of optical fiber was measured with 2-minute steps. Figure 3 shows results for three different solutions of HF. As we can see, the reaction between $SiO_2$ and HF begins instantly for all measured solutions, and reflectance slowly starts dropping off from around 14 dB to 1 dB. Only for 1% solution reflectance didn't decrease to a minimum level, and we can notice that is much slower than 2% and 3%.

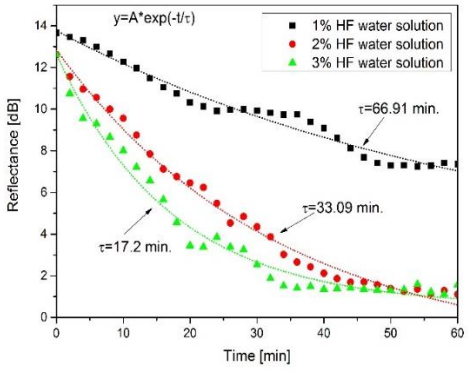


Fig. 3. The reflectance of OTDR event at the end of fiber vs. immersion time.

In all analyzed cases, sensor response wasn't entirely linear. If we look at the exponential fitting (Fig. 3), we notice that increasing HF concentration lowers the τ value. The highest value is equal to 66.91 min we have obtained for the 1% solution. We also noticed that lowering HF concentration by 1% increases our τ value twice. For example, for 3% we have τ=17.2 min, and for 2% we have an example for 3%, we have τ=17.2 min, and for 2% we have τ=33.09 min. Figure 4 presents sensitivity curves for three measured solutions of hydrofluoric acid. As we can see, we have almost linear characteristics for a 1% HF solution but with the lowest sensitivity. The highest sensitivity we achieved in the case of 3% solution for the first 15 minutes of reaction. If we look at the results for the 2% HF water solution, we notice that after the first 15 minutes of the corrosion process, we have the highest sensitivity until the end of the 3 tested sensors. In all cases, the sensitivity of sensors drops with the corrosion process.

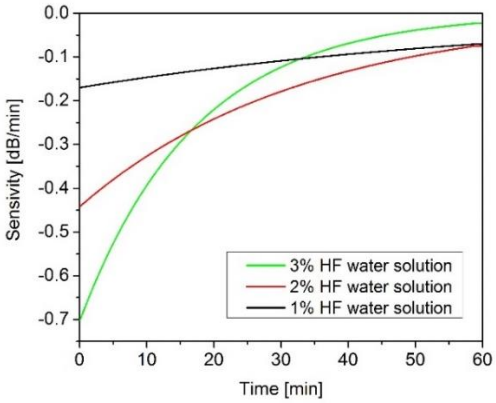


Fig. 4. Sensitivity curves for measurements in HF.

In the case of the second sensor with a deposited silver layer, we used the same OTDR settings. Figure 5 shows sensor response for 1% and 5% $HNO_3$ solution. The reaction between silver and $HNO_3$ can be described as [12]:

$$3Ag + 4HNO_3 \rightarrow 3AgNO_3 + 2H_2O + NO \quad (3.2)$$

As we can see our starting point is around 23 dB and drops to around 9 dB at the end of the corrosion process for 5% solution (for 1% solution we didn't achieve the full corrosion process). In the case of 5% solution our reflectance after the whole process dropped to around 9 dB. We can see that after the whole corrosion process reflectance at the end of the fiber in this case holds at a higher level than during measurements in HF (Fig. 3). During the test in 1% solution, our reflectance dropped only by around 1.5 dB. When we look at Fig. 5 we can notice that our τ value increased twice when we decreased nitric acid concentration. Also, these values are much higher than for measurements in HF.

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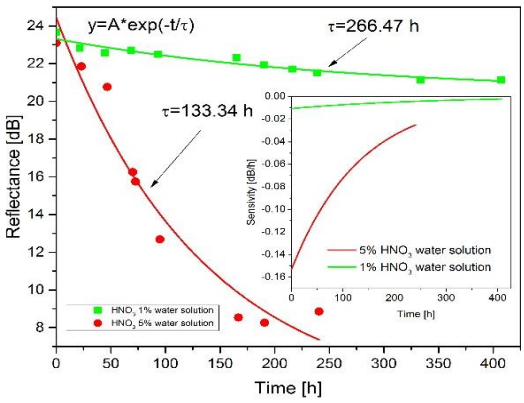


Fig. 5. The reflectance of sensor vs. Immersion time for 5% and 1% $HNO_3$ water solutions.

When we look at sensitivity curves (Fig. 5) we notice that increasing the volume of $HNO_3$ in water causes increased system sensitivity. However, higher concentration also causes a faster decrease of this value over time.

We demonstrated commercially available OTDR for corrosion monitoring applications. Two optrodes were tested. The first one was optical fiber cut at the end, while the second one was optical fiber with a deposited silver mirror. For tests in hydrofluoric acid, only 1% solution didn't reach the whole corrosion time under one hour, and all three reactions were nonlinear. During tests in nitric acid 5% solution reached full corrosion time, while 1% of solution dropped reflectance only by around 1.5dB. Increasing acid concentration causes a faster decrease in sensitivity over time. Highest τ value we obtained during tests in 1% $HNO_3$, and the lowest in 3% HF. After 5km of fiber, the signal is strong enough to provide reliable measurement data and can be used even on higher distances. It's hard to state which one of the two presented sensors is better. These solutions are dedicated to different corrosion environments. However, the advantage of the second sensor is that its sensitivity can be adjusted by changing the thickness of the silver layer

Presented setup can find applications in harsh corrosive environments by depositing layer matching material of measuring element for example pipeline, or linchpin of an oil rig. Moreover, there are possibilities to extend the system into a network of sensors over a significant distance using fiber splitters. By using optical fibers, it's possible to minimalize the risk of ignition to zero, which is a considerable advantage over commercially available electronic sensors.

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