

# Metallic Coating Enabled Optical Fiber Sensor for Distributed Corrosion Monitoring

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# Disclaimer

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# Outline

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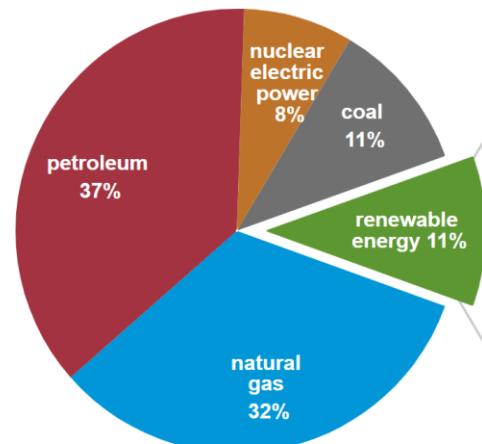


- **Research background**
- **Experimental setup**
- **Results and discussion**
- **Conclusions**
- **Disclaimer**

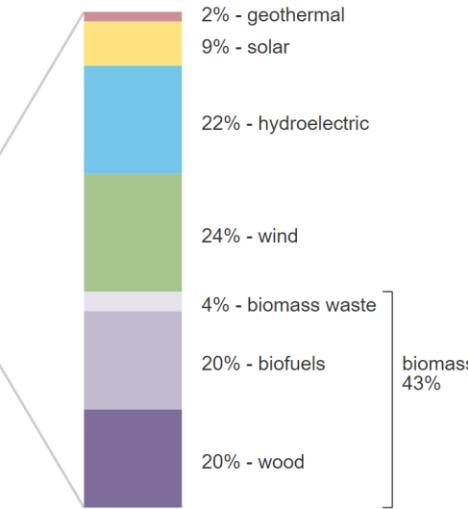
# Corrosion in Oil and Gas Infrastructure

## U.S. primary energy consumption by energy source, 2019

total = 100.2 quadrillion  
British thermal units (Btu)



total = 11.4 quadrillion Btu

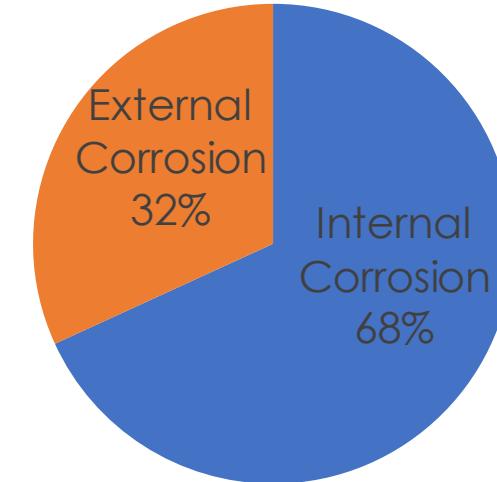


Note: Sum of components may not equal 100% because of independent rounding.  
Source: U.S. Energy Information Administration, *Monthly Energy Review*, Table 1.3 and 10.1, April 2020, preliminary data



- \$1.4 billion annually: direct corrosion costs in domestic oil and gas exploration and production in the U.S.
- \$589 million: surface piping and facility costs
- \$463 million: downhole tubing expenses
- \$320 million: capital expenditures related to corrosion.
- **25-30% can be saved** with optimal corrosion management.

## 2010-2016 Transmission Pipelines



Corroded production casing pipe sample from downhole

(University of North Dakota Energy & Environmental Research Center)

Koch, G. H., Brongers, Michiel, P. H., Thompson, N. G., Virmani, Y. P. and Payer, J. H., "Corrosion costs and preventive strategies in the United States" (2002).

# Corrosion is an Electrochemical Process Causing Mass Loss and Structural Deterioration

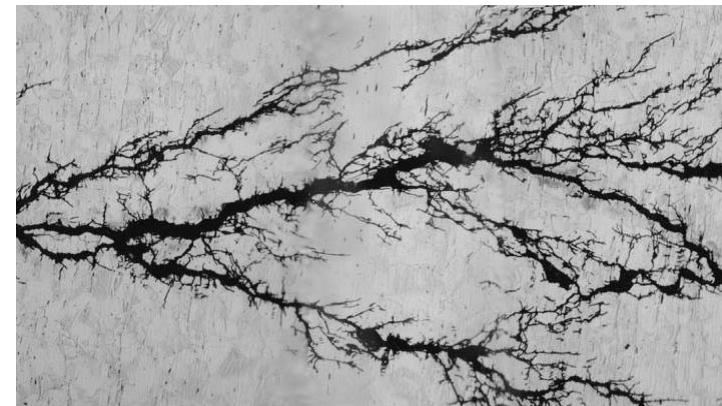
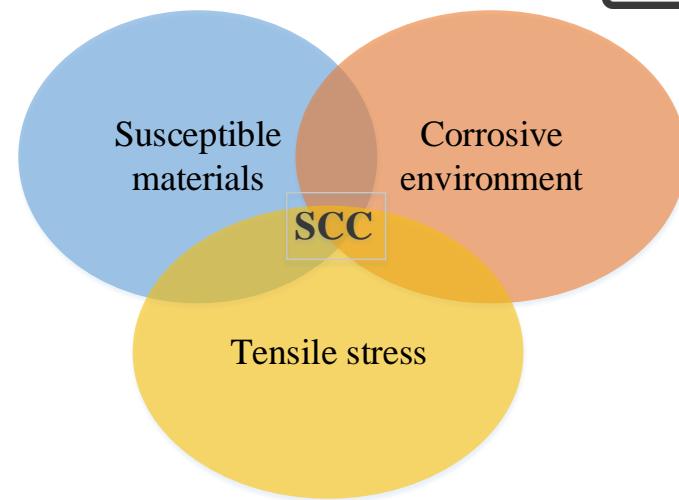
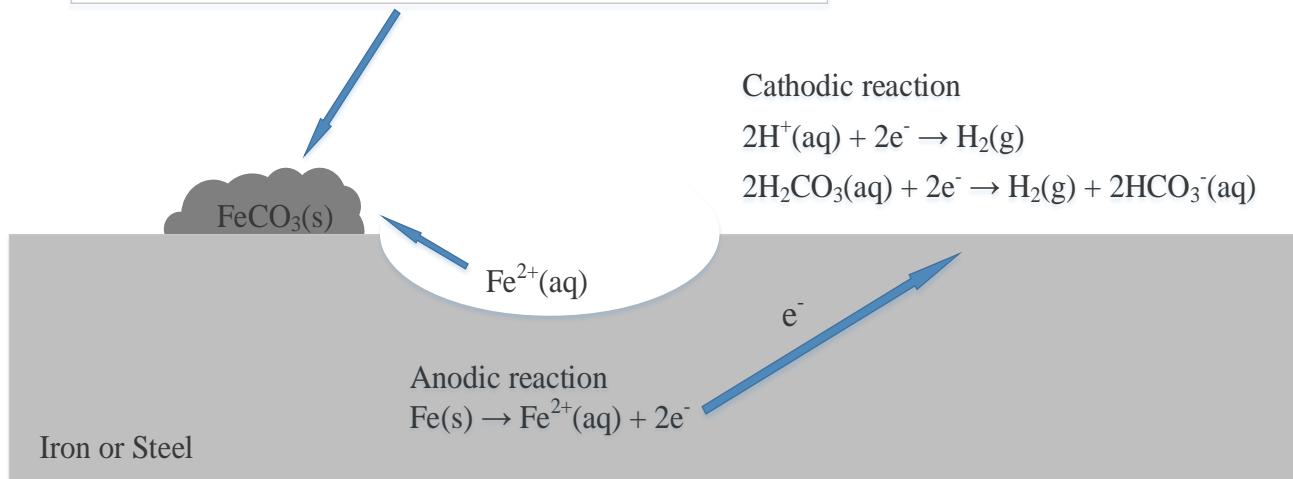
Dissolution of  $\text{CO}_2$ :  $\text{CO}_2(\text{g}) \leftrightarrow \text{CO}_2(\text{aq})$

Hydration of  $\text{CO}_2$ :  $\text{CO}_2(\text{aq}) + \text{H}_2\text{O}(\text{l}) \leftrightarrow \text{H}_2\text{CO}_3(\text{aq})$   
~ 0.2% of  $\text{CO}_2(\text{aq})$  and quite slow

Dissociation of  $\text{H}_2\text{CO}_3(\text{aq})$ :

$\text{H}_2\text{CO}_3(\text{aq}) \leftrightarrow \text{H}^+(\text{aq}) + \text{HCO}_3^-(\text{aq})$ ,  $pK_1=6.35$  at 25 °C

$\text{HCO}_3^-(\text{aq}) \leftrightarrow \text{H}^+(\text{aq}) + \text{CO}_3^{2-}(\text{aq})$ ,  $pK_2=10.33$  at 25 °C

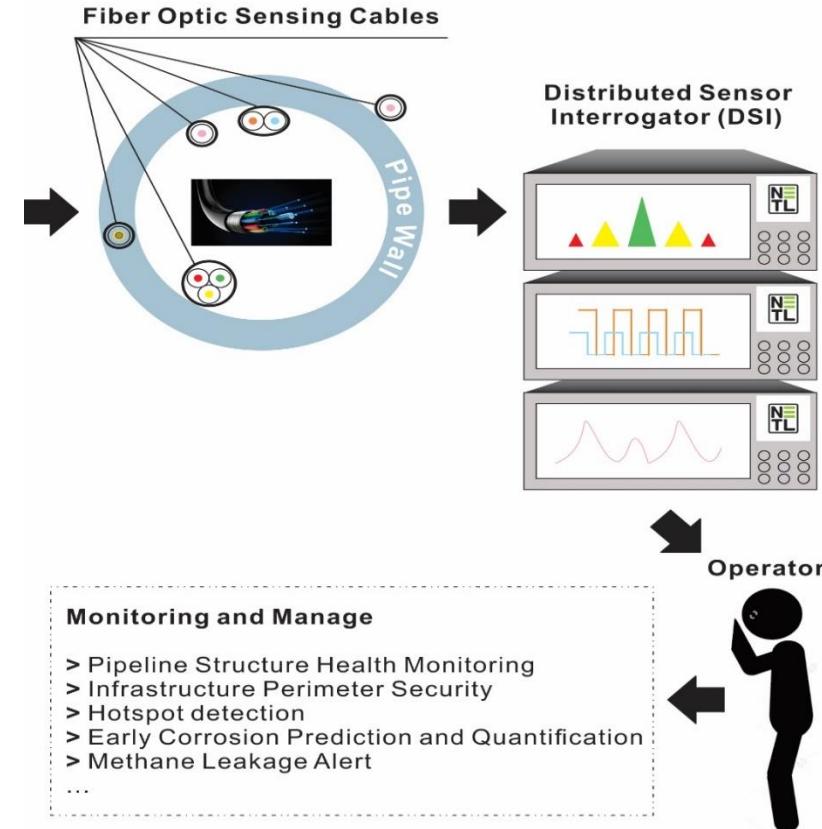
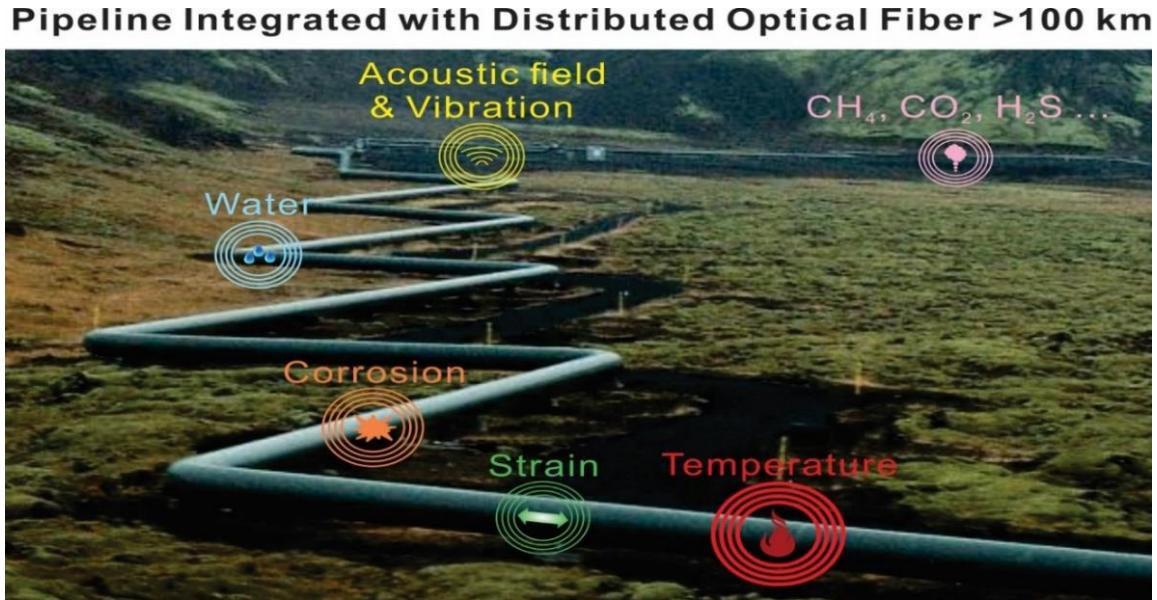


<https://corrosion.ksc.nasa.gov/stresscor.htm>

Jones, D. A., Principles and Prevention of Corrosion, P 237.

Han, J., Nesić, S., Yang, Y., Brown, B., *Electrochimica Acta*, 2011, 5396-5404.

# Optical Fiber Sensor Platform for Natural Gas Pipelines



- Early Corrosion Onset Detection
- Methane Leak Detection

**A Multi-Parameter, Distributed Optical Fiber Sensor (OFS) Platform Enabling Reliability & Flexibility**  
**Target Metrics = >100km Interrogation, <1m Spatial Resolution**

# Distributed OFS Sensing Using Optical Backscatter Reflectometer (OBR)



OBR measures the Rayleigh backscattering from the fiber-under-test (FUT).

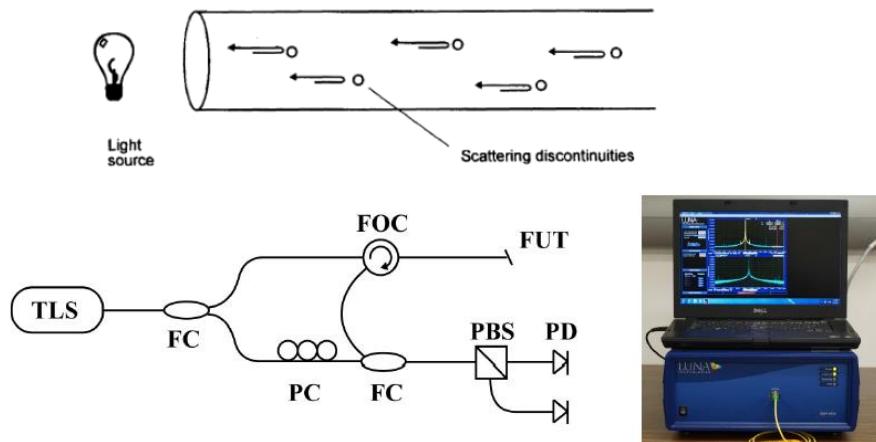
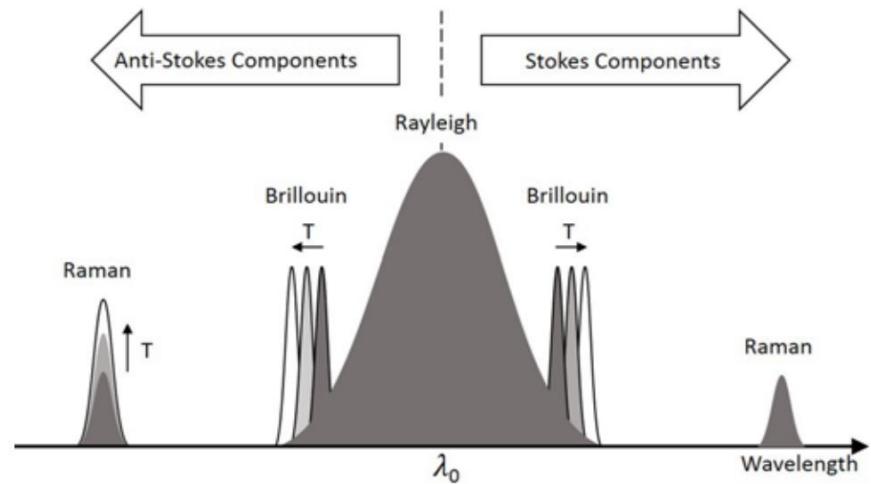


Figure 1 – LUNA OBR 4600 distributed interrogator LEFT: internal components (TLS: tunable laser source, FC: fiber coupler, FOC: fiber optic circulator, PC: polarization controller, PBS: polarizing beamsplitter, PD: photodiode); RIGHT: photo of instrument.

Backscattering enables distributed temperature, strain, and acoustic sensing (DTS, DSS, DAS)

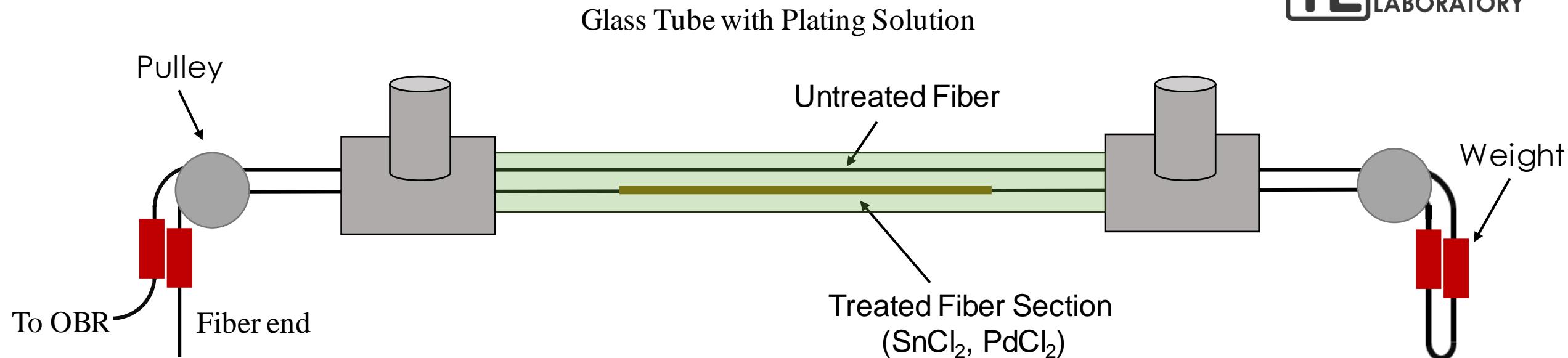


OBR can measure spatial profiles of either temperature or strain changes

$$\text{Temperature Change} = \Delta T = -\frac{\bar{\lambda}}{cK_T} \Delta\nu \quad \text{or} \quad \text{Strain} = \varepsilon = -\frac{\bar{\lambda}}{cK_\varepsilon} \Delta\nu$$

where  $\bar{\lambda}$  is the center wavelength of the scan;  $c$  is the speed of light;  $K_T$  and  $K_\varepsilon$  are the temperature and strain calibration constants, respectively;  $\Delta\nu$  = spectral shift.

# Experimental Setup



**Schematic diagram of the experimental setup for distributed strain measurements in a glass tube during electroless plating or metallic film dissolution**

- Both the treated (sensitized and activated) section and a untreated fiber section as the control were inserted into a glass tube.
- The difference between the treated and untreated sections can provide insights into the strain changes solely caused by plating of metallic coatings (Ni or Fe), and the effects of temperature and water-induced strains would be compensated using the untreated control section.

# Optical Fiber Preparation and Electroless Plating



## ➤ Optical fiber cleaning

Chemical	Conc.	Operation
Acetone	100%	Sonicate <5min
Dry in Air		

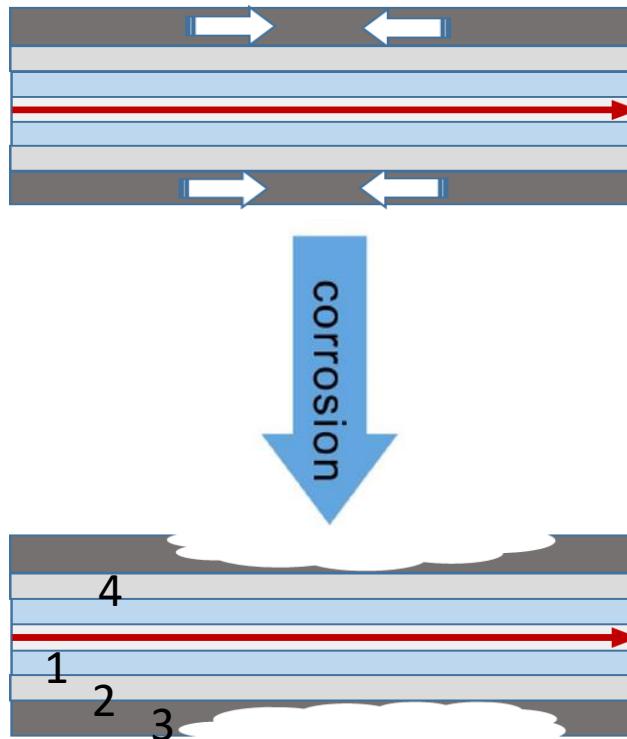
## ➤ Sensitization and Activation (Twice)

Chemical	Amount	Operation
SnCl <sub>2</sub>	22.6g/L	30 seconds at 50 °C, rinse in DI water
HCl, conc.	10mL/L	
PdCl <sub>2</sub>	0.795g/L	30 seconds at 50 °C, rinse in DI water
HCl, conc.	5mL/L	

## ➤ Electroless plating solutions and conditions

Electroless plating soln.	Ni (High pH)	Ni (Low pH)	Fe
Metal source	19 g/L NiSO <sub>4</sub> ·6H <sub>2</sub> O	19 g/L NiSO <sub>4</sub> ·6H <sub>2</sub> O	11 g/L FeSO <sub>4</sub> ·7H <sub>2</sub> O
Complexing agent	8 g/L C <sub>6</sub> H <sub>5</sub> Na <sub>3</sub> O <sub>7</sub> ·2H <sub>2</sub> O	8 g/L C <sub>6</sub> H <sub>5</sub> Na <sub>3</sub> O <sub>7</sub> ·2H <sub>2</sub> O	57 g/L C <sub>6</sub> H <sub>5</sub> Na <sub>3</sub> O <sub>7</sub> ·2H <sub>2</sub> O
Reducing agent	15 g/L NaH <sub>2</sub> PO <sub>2</sub> ·H <sub>2</sub> O	15 g/L NaH <sub>2</sub> PO <sub>2</sub> ·H <sub>2</sub> O	3.0 g/L NaBH <sub>4</sub>
pH stabilizer	6.5 g/L NH <sub>4</sub> Cl	6.5 g/L NH <sub>4</sub> Cl	10 g/L H <sub>3</sub> BO <sub>3</sub>
pH	10 @ 21.5 °C	5.8 @ 21.5 °C	10 @ 21.5 °C
Solution temperature	~50 °C	~50 °C	21.5 °C

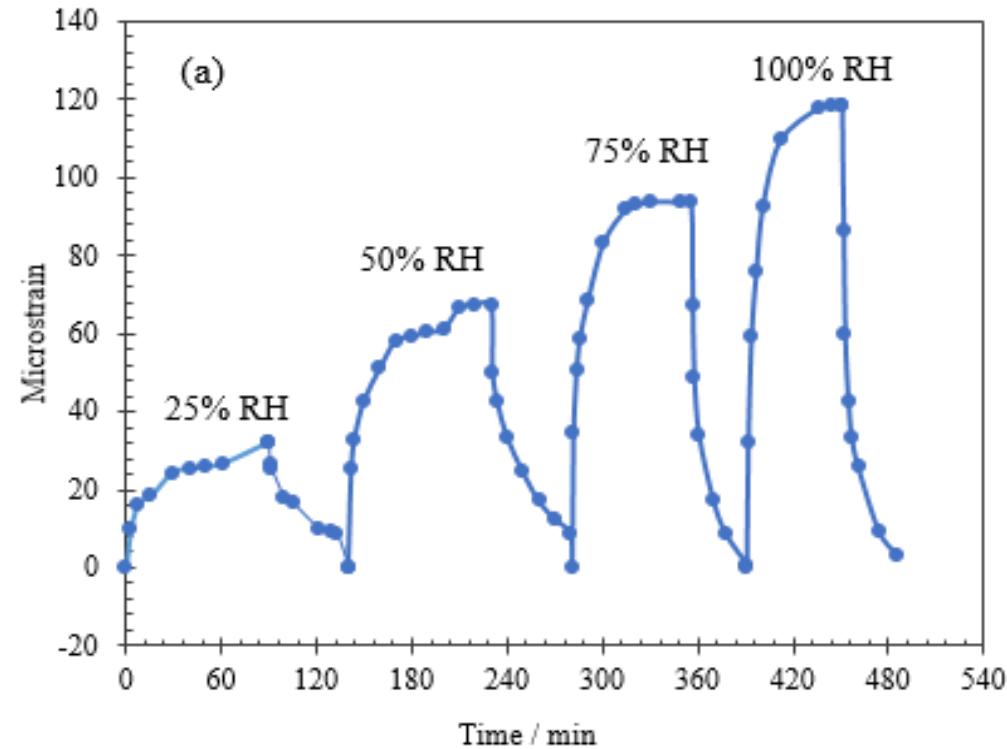
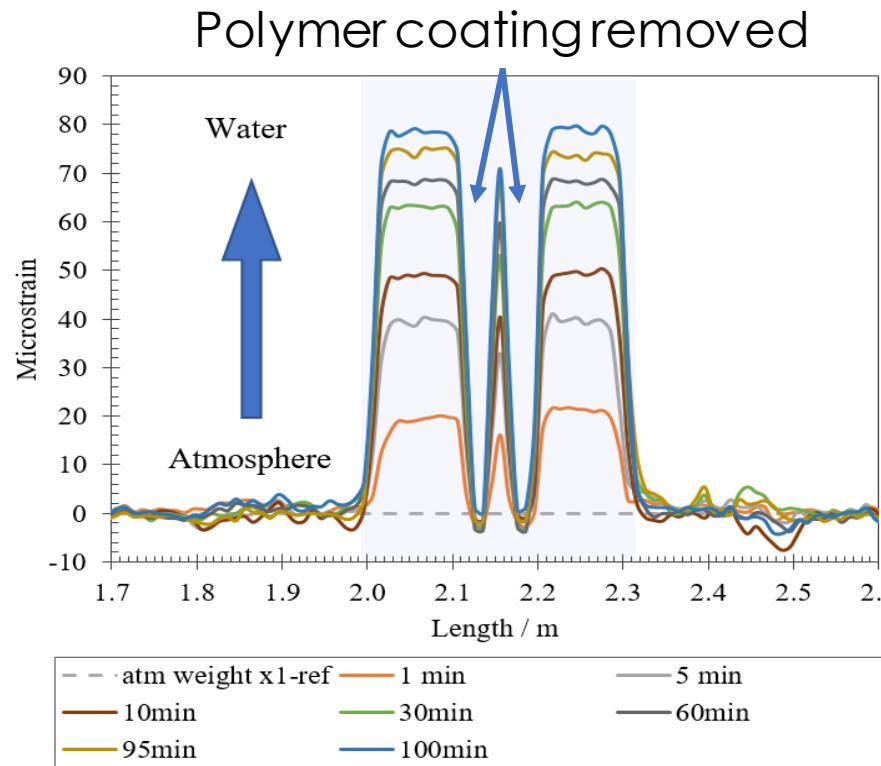
# Corrosion Sensing Principle based on Strain Changes on Optical Fibers



- Inherent internal stress in the electrolessly plated metallic films can induce strains on the fiber.
- As the metallic film gets corroded, the internal stress will be released, causing changes in strains which can be measured using OBR.

- 1: single-mode fiber core;
- 2: cladding;
- 3: polymer jacket;
- 4: coated metallic film

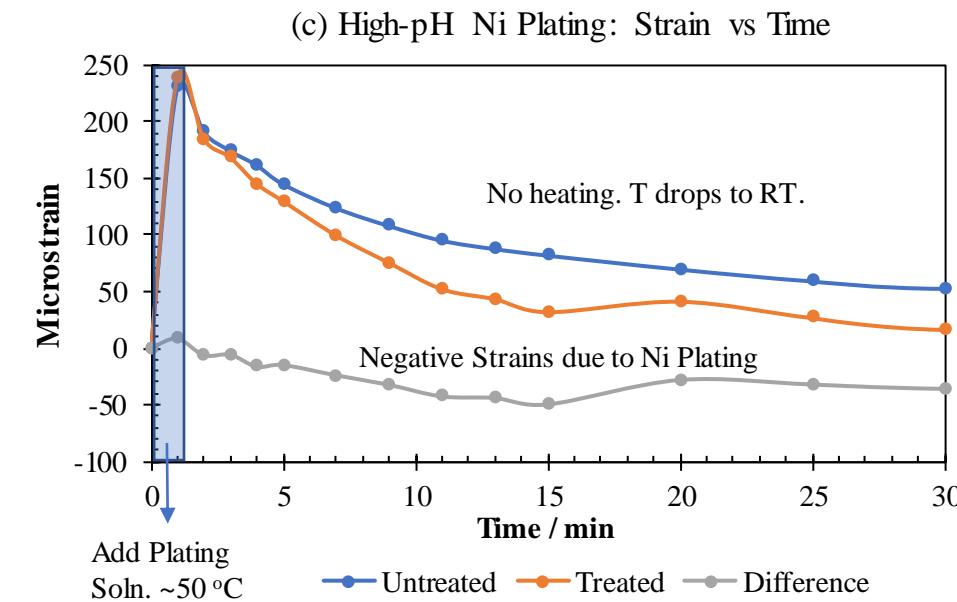
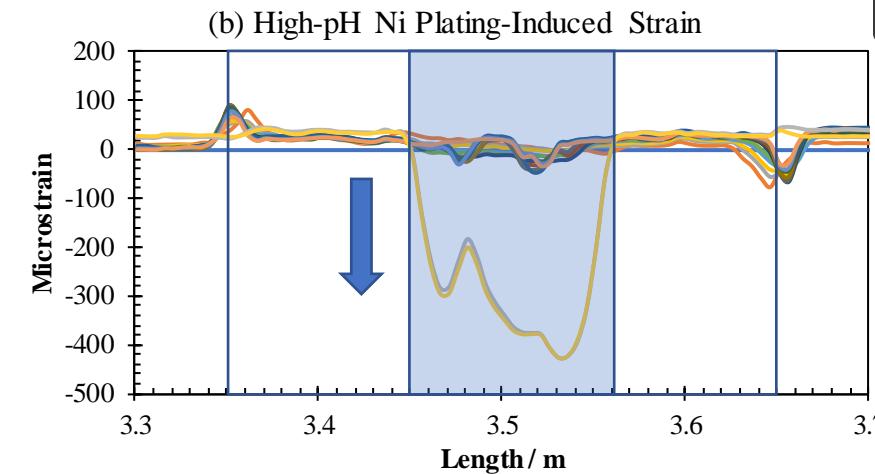
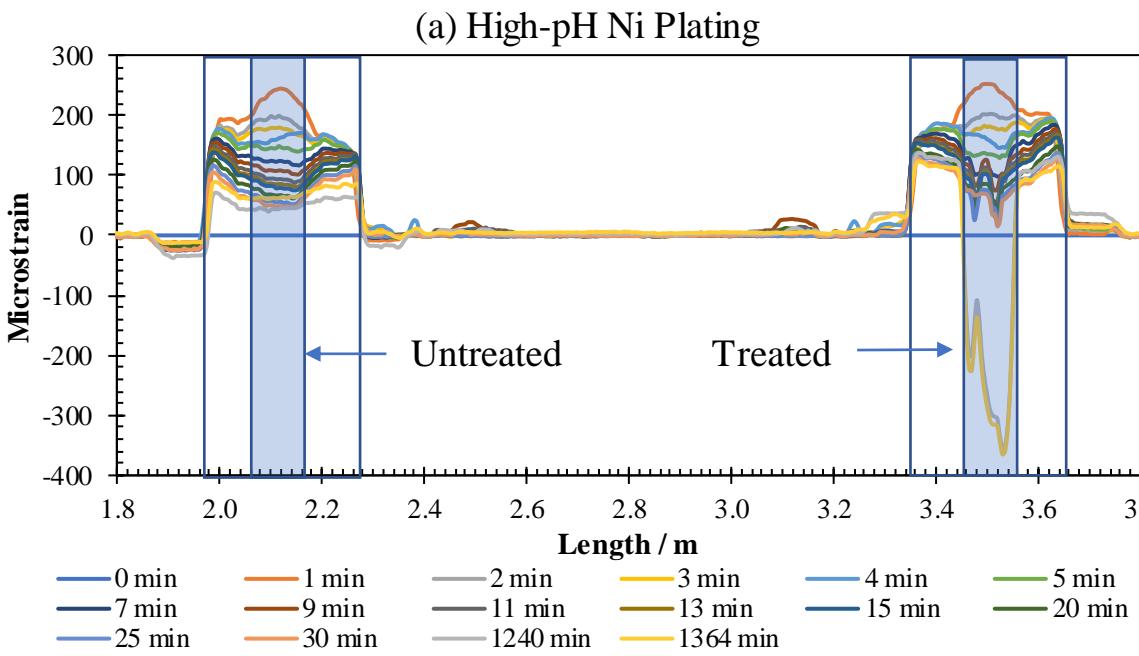
# Water and Humidity Sensors From Previous Work



- The intrinsic hygroscopic polymer jacket coating of SMF-28Ultra serves as the water sensing layer due to expansion/swelling from water absorption.
- Reversible humidity sensing and linear correlation between humidity levels and strain along the fiber at room temperature.

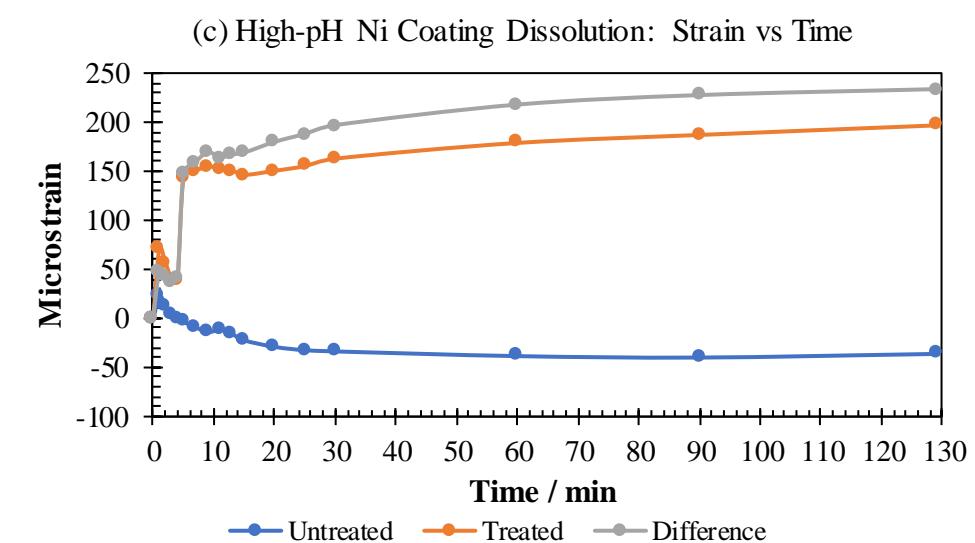
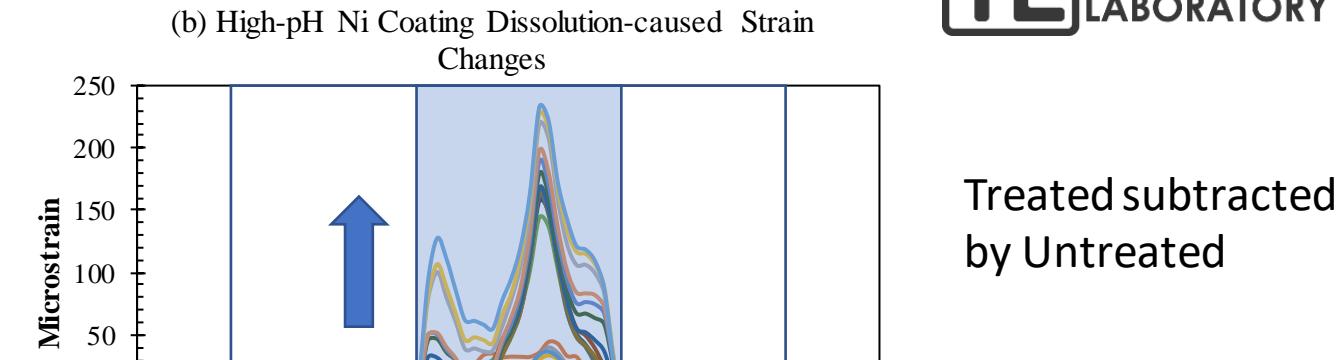
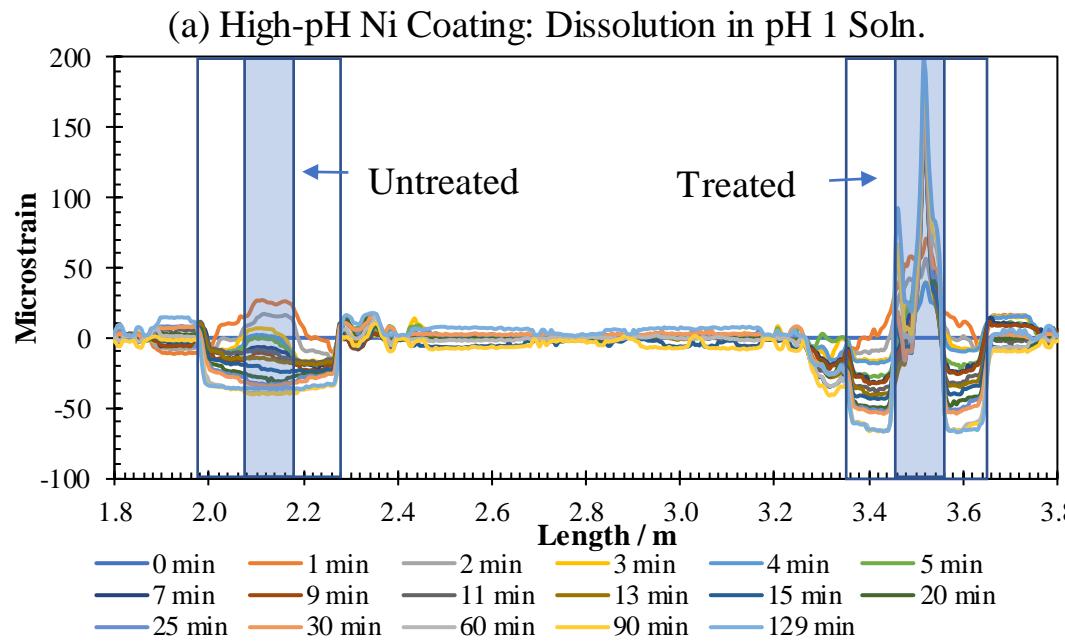
R F. Wright, et al. "Fully distributed optical fiber sensor for water and humidity monitoring," Proc. SPIE 11000, Fiber Optic Sensors and Applications XVI, 1100007 (14 May 2019)

# High-pH Ni Plating Induced Negative Strains



- Rectangles highlight the solution region inside the tube.
- Gray areas highlight the treated and untreated sections.
- Untreated section:** strain increased due to ~50 °C Ni plating solution and water-induced swelling; then decreased as the solution cooled down (no heating).
- Treated section:** strain decreased comparatively due to Ni plating. 30 min: -36  $\mu\epsilon$ ; 22 hr: -430  $\mu\epsilon$ .

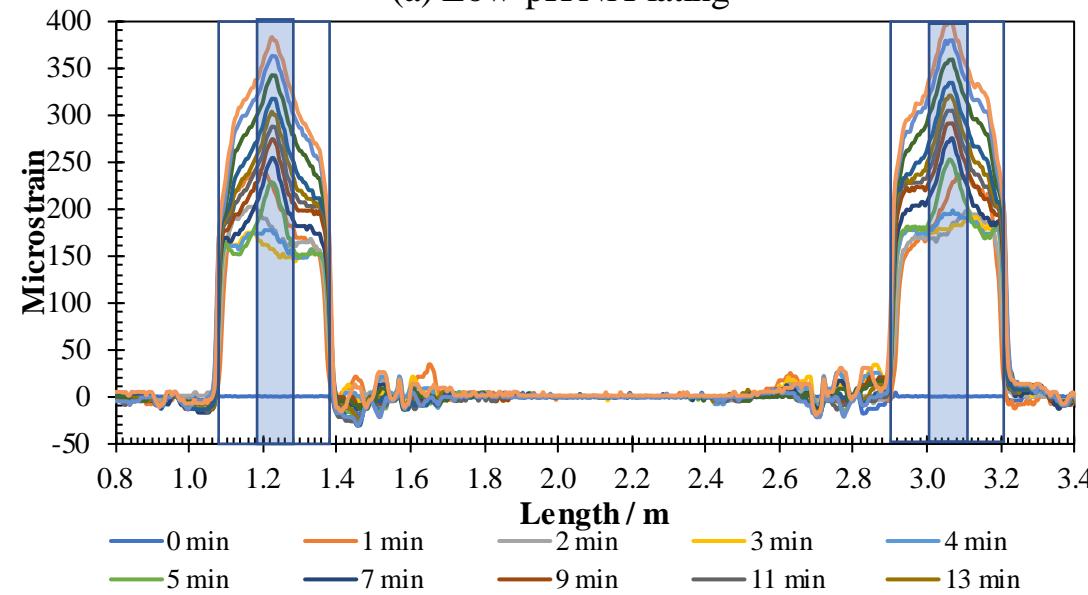
# High-pH Ni Coating: Dissolution Induced Positive Strains



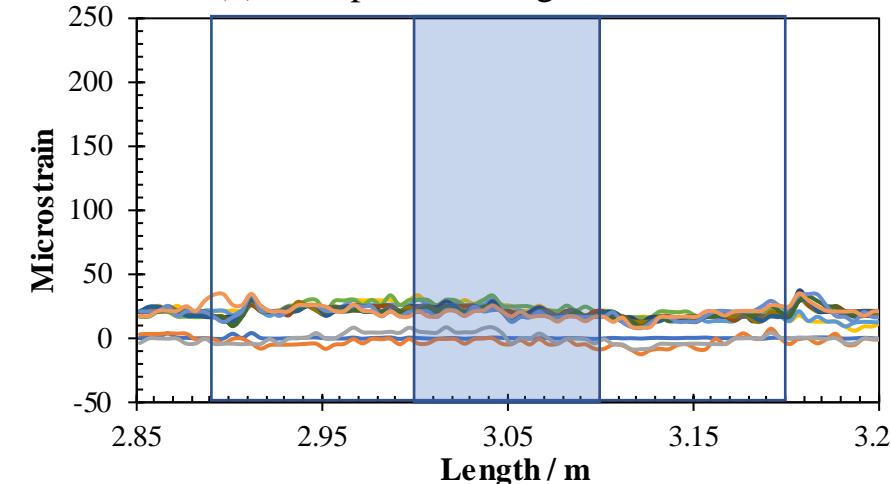
- After plating, high-pH Ni coating was exposed to pH 1 HCl soln.
- Untreated section:** at room temperature (RT), strain showed a minimal decrease to  $-36 \mu\epsilon$ .
- Treated section:** strain increased up to  $233 \mu\epsilon$  due to Ni dissolution. Two preferable dissolution locations, corresponding to Ni deposition strains.

# Low-pH Ni Plating Induced Minimal Strains

(a) Low-pH Ni Plating

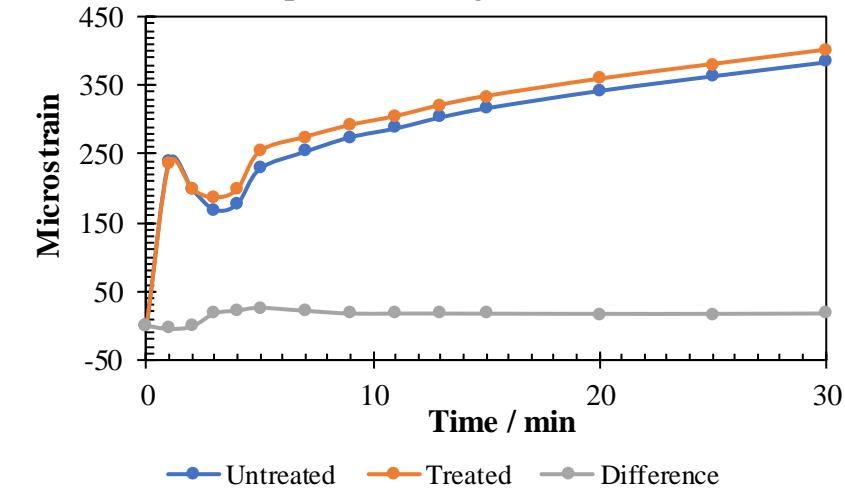


(b) Low-pH Ni Plating-Induced Strain



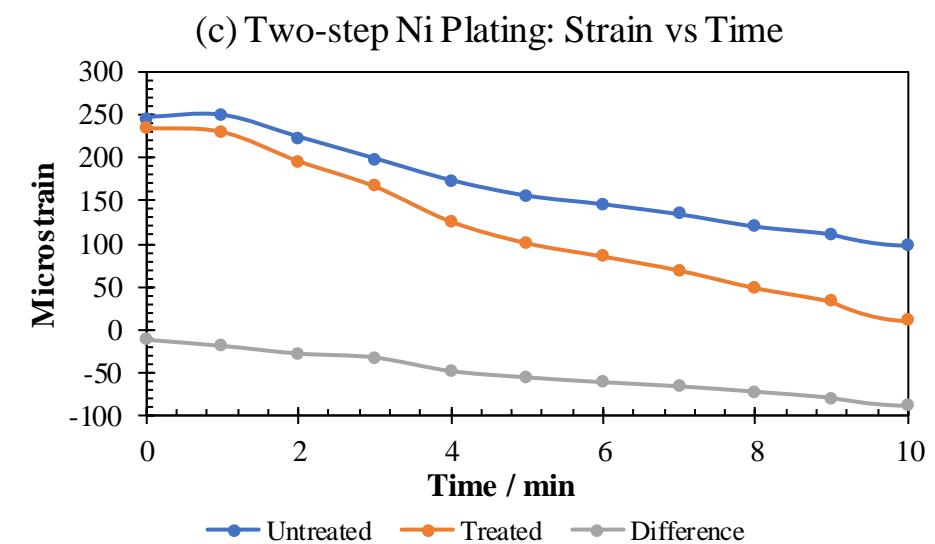
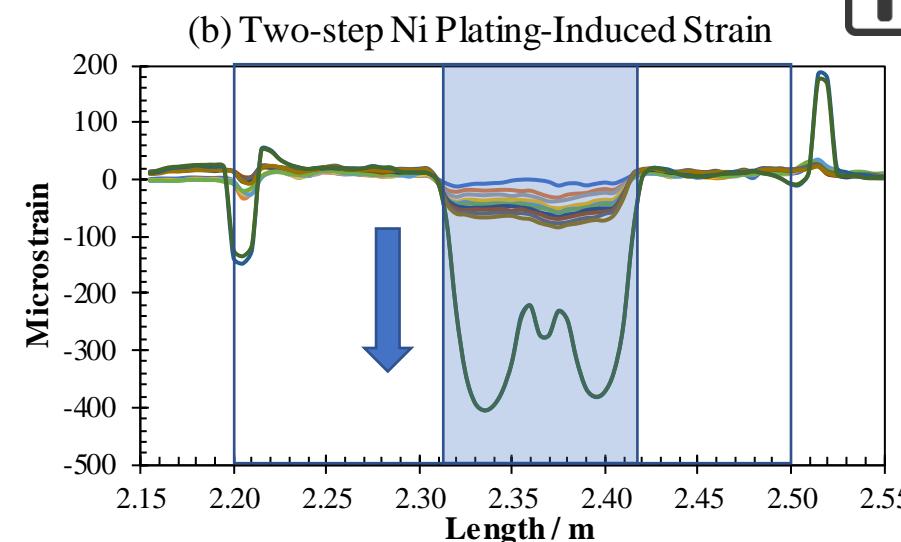
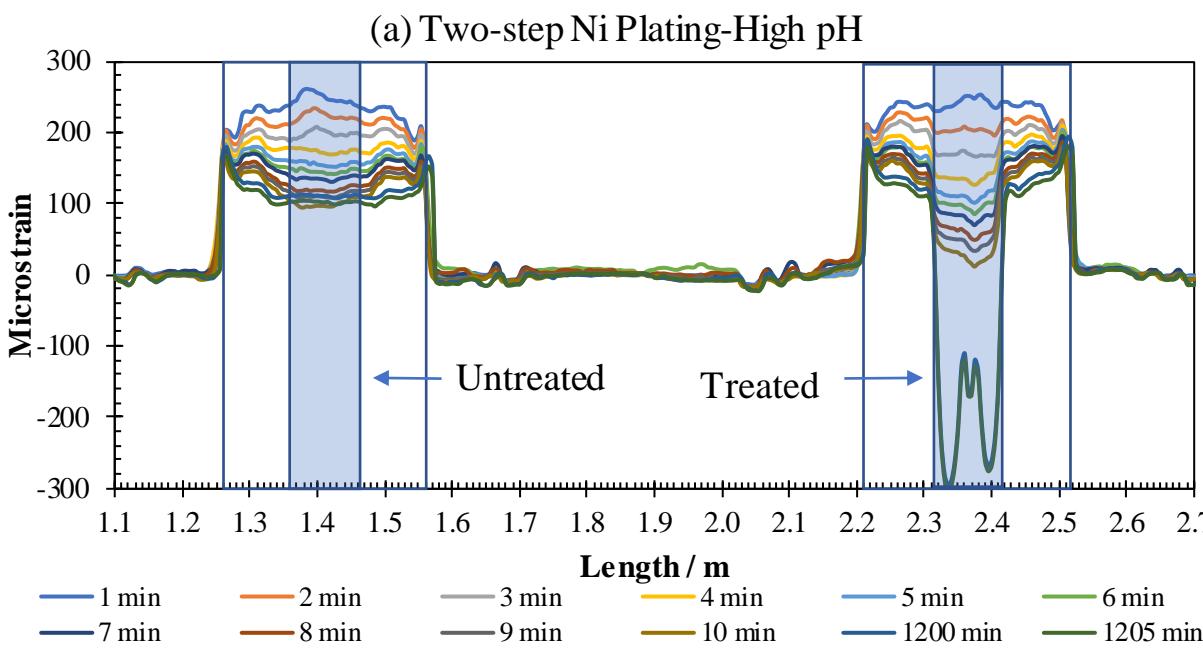
Treated subtracted  
by Untreated

(c) Low-pH Ni Plating: Strain vs Time



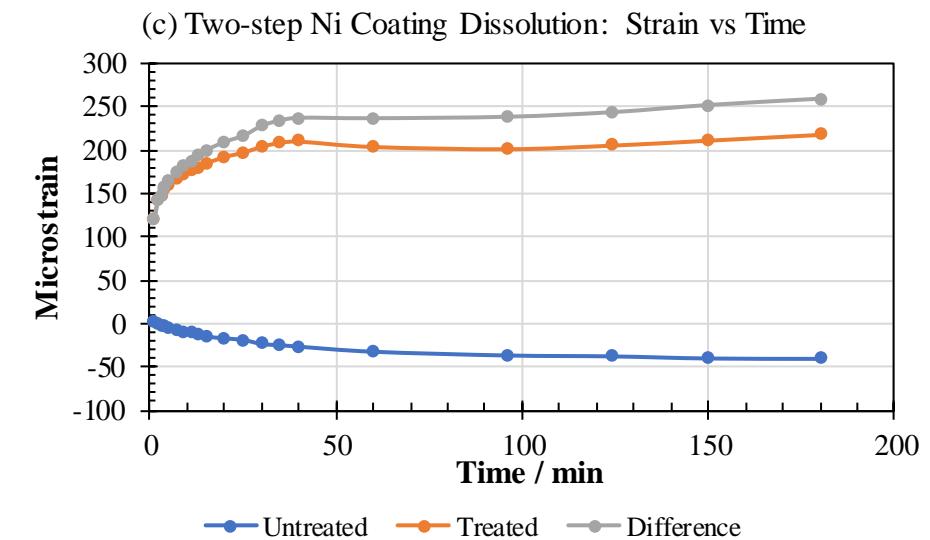
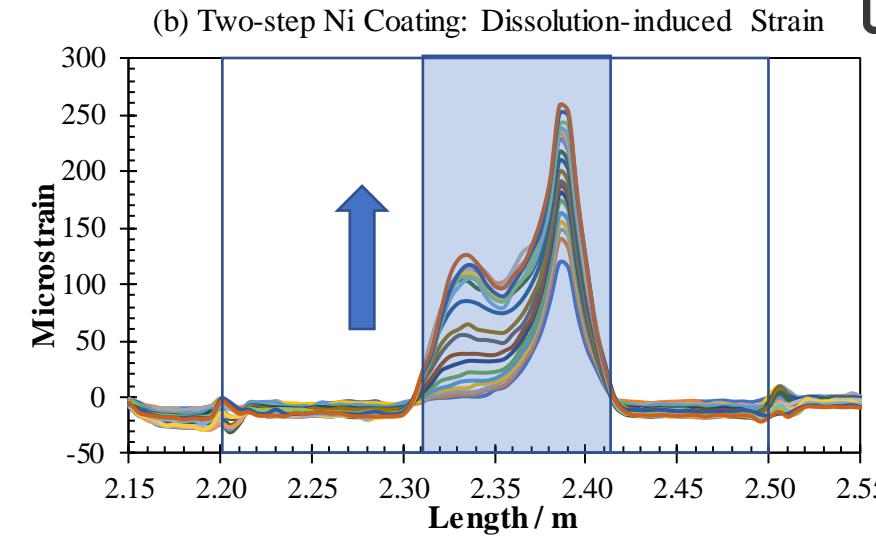
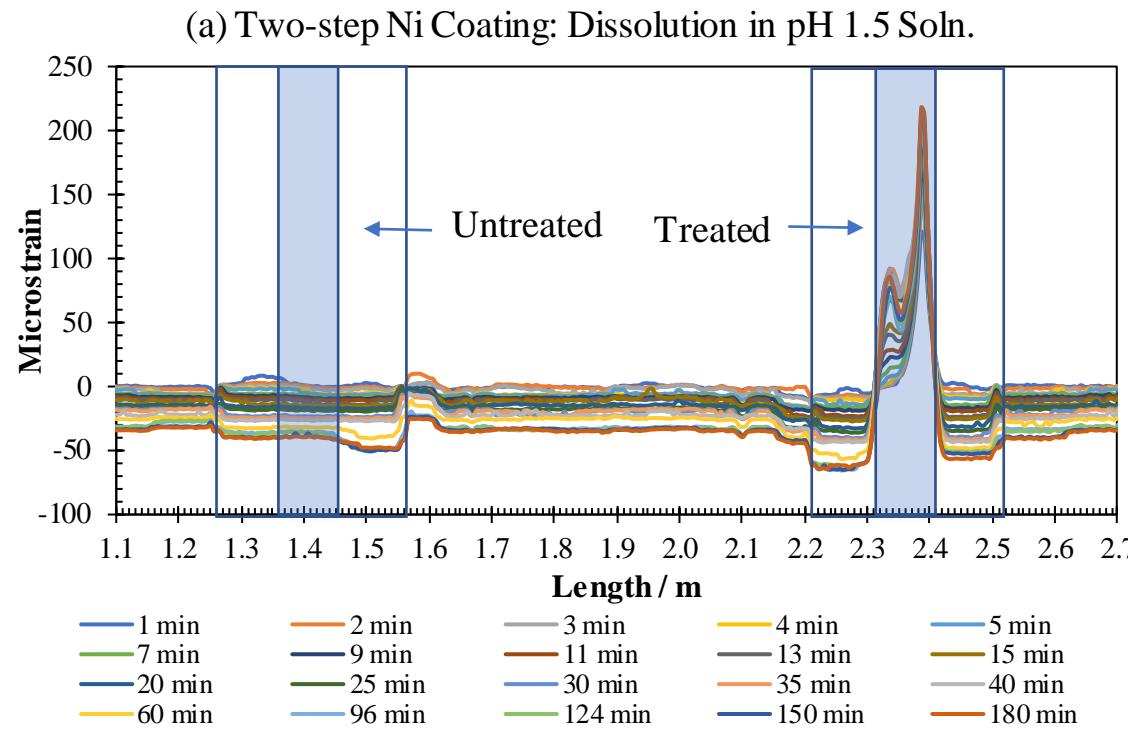
- **Untreated section:** strain increased due to  $\sim 50$  °C Ni plating solution and water-induced swelling.
- **Treated section:** strain increased similarly to the untreated section, leading to minimal strains due to Ni plating.

# Two-Step Ni Plating: High-pH Ni Plating Induced Negative Strains



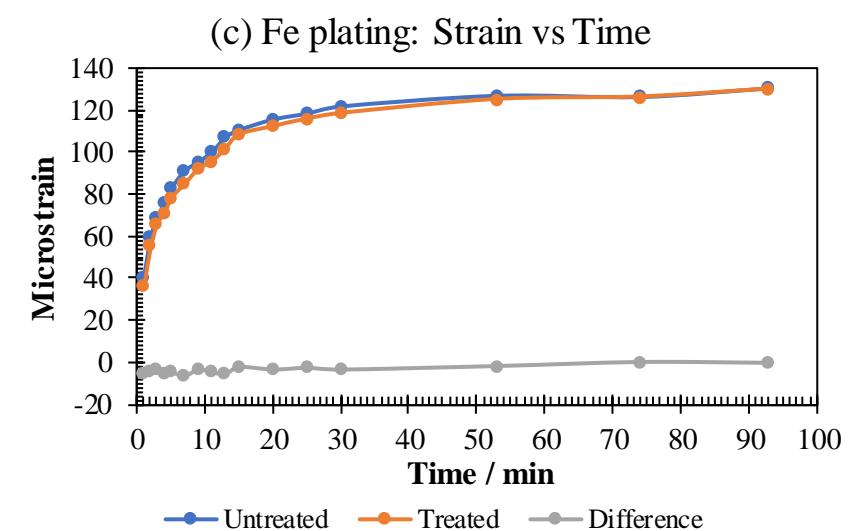
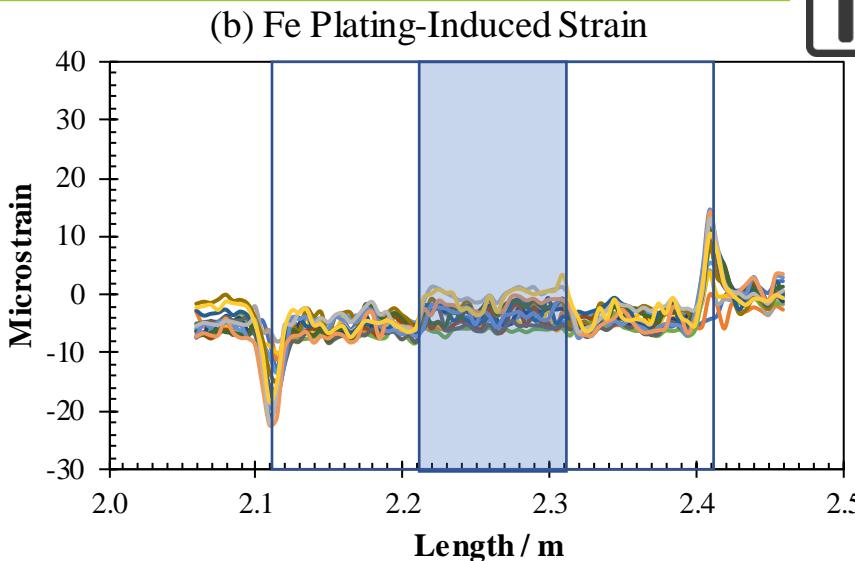
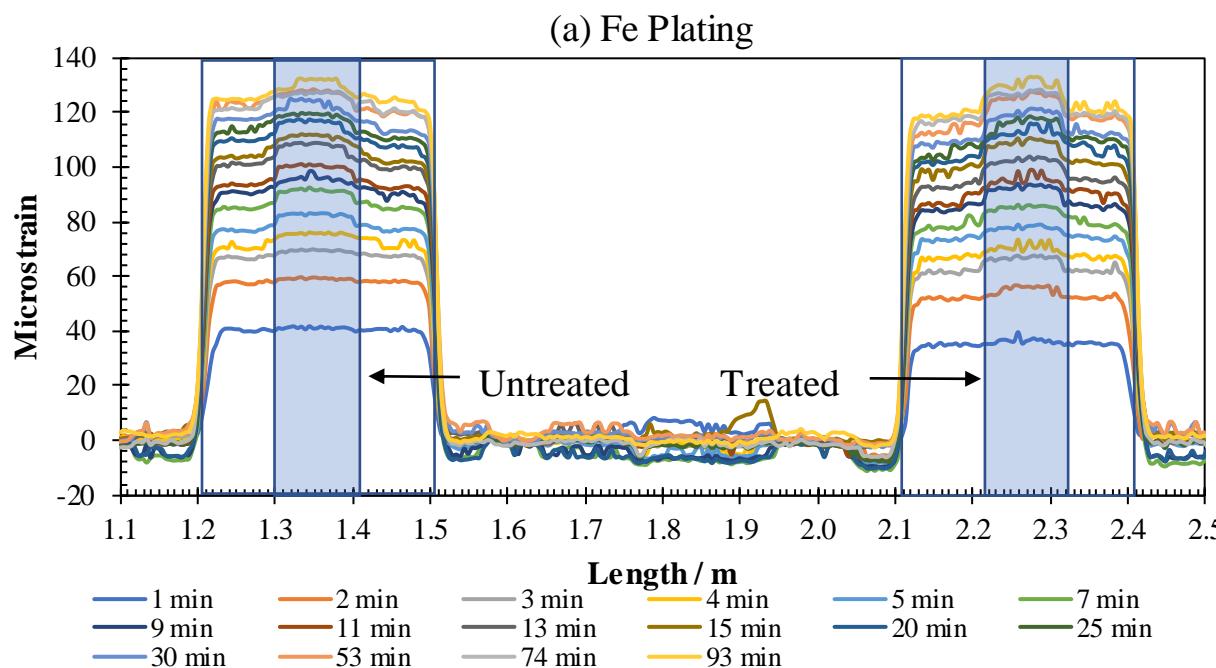
- Two-step Ni plating: Low-pH plating solution for 6 min to deposit the initial layer; then high-pH plating solution with a faster plating rate.
- **Untreated section:** strain increased due to  $\sim 50$  °C Ni plating solution and water-induced swelling; then decreased as the solution cooled down (no heating).
- **Treated section:** strain decreased comparatively due to high-pH Ni plating. 10 min:  $-88 \mu\epsilon$ ; 20 hr:  $-407 \mu\epsilon$ .

# Two-Step Ni Coating: Dissolution Induced Positive Strains



- After plating, two-step Ni coating was exposed to pH 1.5 HCl soln.
- Untreated section:** at RT, strain showed minimal changes.
- Treated section:** strain increased comparatively up to  $\sim 260 \mu\epsilon$  due to Ni dissolution. Two preferable dissolution locations, corresponding to Ni deposition strains.

# Fe Plating Induced Negligible Strains



- **Untreated section:** at RT, strain increased due to water-induced swelling.
- **Treated section:** strain increased similarly to the untreated section, leading to minimal strains (near-zero) due to Fe plating.

# Conclusions



- A distributed optical fiber corrosion sensor was demonstrated based on strain changes on the optical fiber, interrogated using an OBR.
- Electroless plating leads to inherent internal stress in the metallic coating, and induces strains on the fiber. As the metallic film gets corroded, the internal stress will be released, causing changes in strains, which can be used as an indicator for dissolution/mass loss.
- The difference between the treated and untreated sections can provide insights into the strain changes solely caused by plating of metallic coatings (Ni or Fe), and the effects of temperature and water-induced strains would be compensated using the untreated control section.
- High-pH Ni plating had a faster deposition rate and induced negative strains, and the dissolution induced positive strains in the corresponding coating locations.
- Low-pH Ni plating and Fe plating had a relative slow and smooth coating, which induced minimal strains.
- Two-step Ni plating includes low-pH plating to deposit an initial layer and then high-pH plating with a faster deposition rate. The high-pH Ni plating induced negative strains.

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