Optical Fiber Sensor Technologies For Subsurface Hydrogen Storage Monitoring

NATIONAL ENERGY TECHNOLOGY LABORATORY

SHASTA
Subsurface Hydrogen Assessment, Storage, and Technology Acceleration

Presenter: Ruishu F. Wright, Ph.D.
Research and Innovation Center

2022 DOE Hydrogen Program Annual Merit Review and Peer Evaluation Meeting (AMR)
June 6–8, 2022

Solutions for Today | Options for Tomorrow

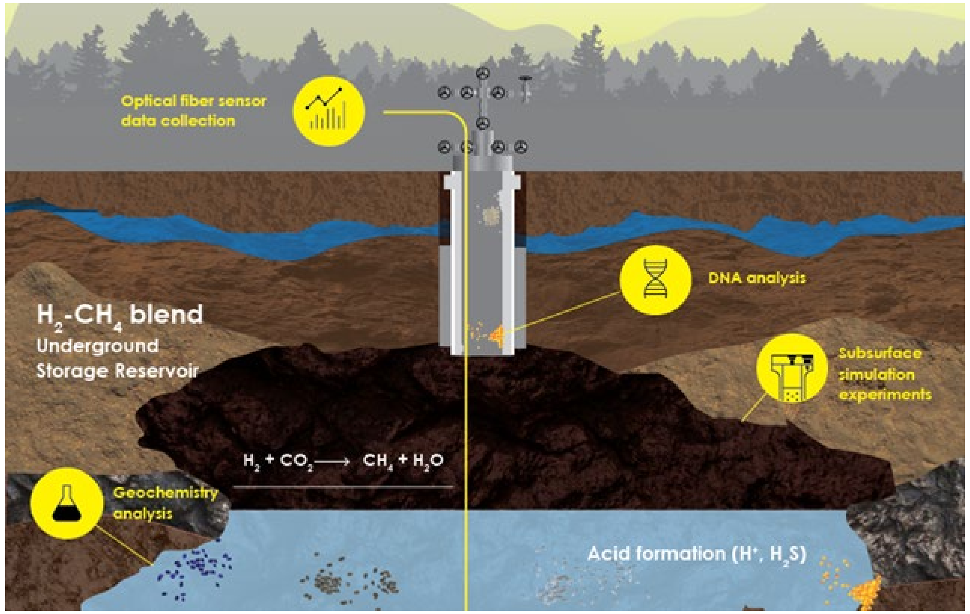
U.S. DEPARTMENT OF ENERGY

Outline

Scope: Enabling Sensor Technologies within *Subsurface Hydrogen Assessment, Storage, and Technology Acceleration (SHASTA) Project*

- **Project Objectives**
- **Approach**
- **Previous work at NETL**
- **Progress**
- **Accomplishments**
- **Future plans**
- **Acknowledgement**

SHASTA
Subsurface Hydrogen Assessment, Storage, and Technology Acceleration



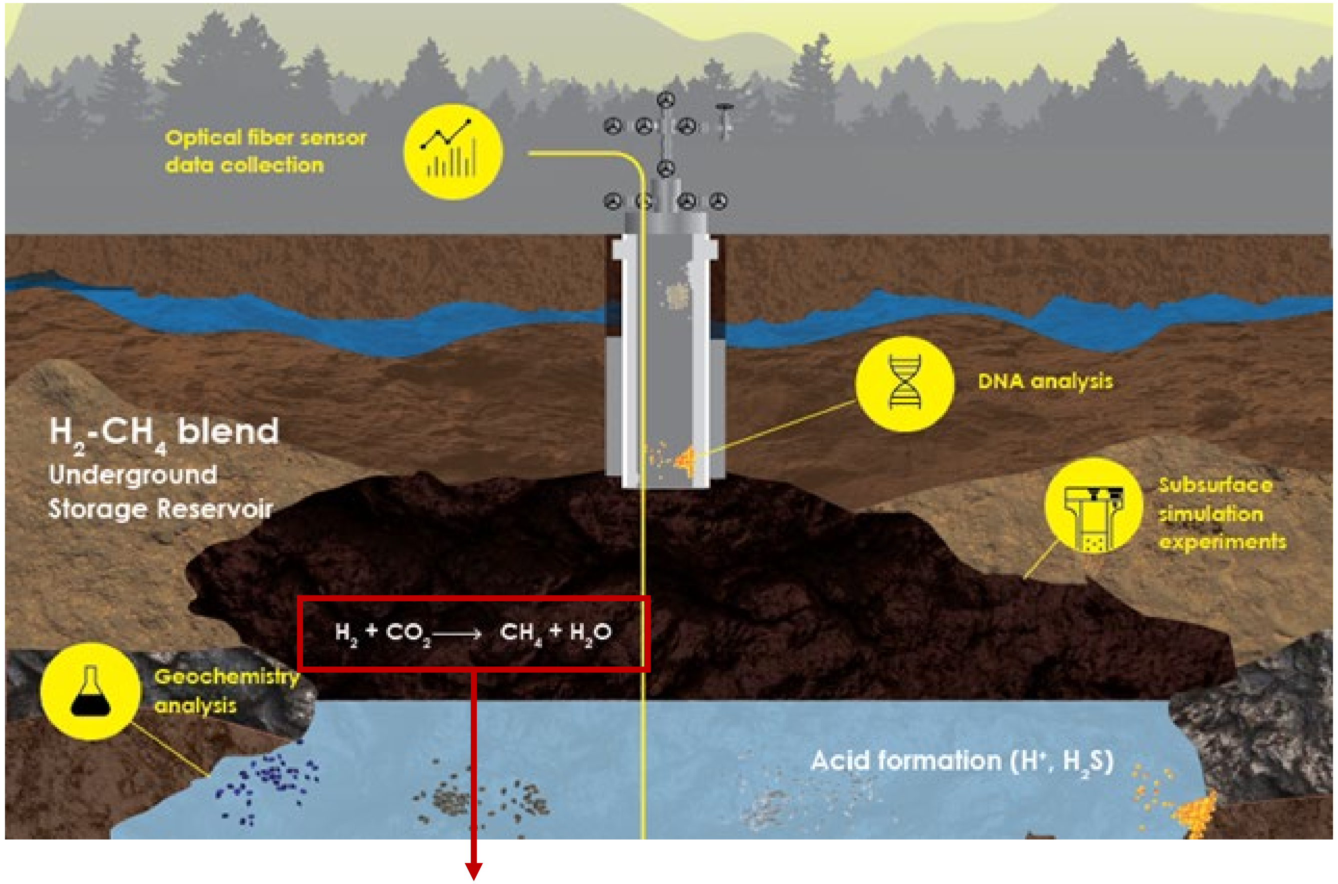
Project Period: 04/2021-04/2024

Project Objectives

- In-situ optical fiber sensors for real-time monitoring of **<u>hydrogen, methane, and chemical parameters</u>** at subsurface hydrogen storage conditions

Impact on Subsurface Hydrogen Storage

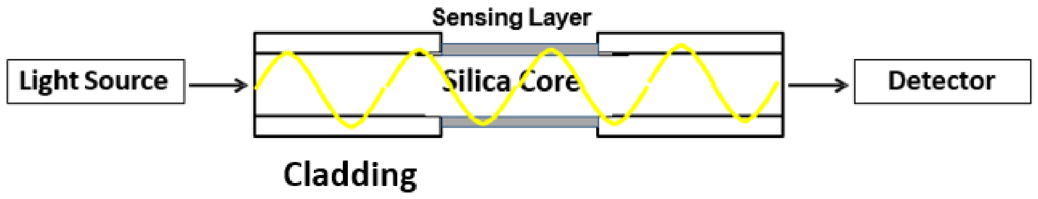
- Determine microbiological H_2 consumption/depletion and pH change
- Identify well integrity risks



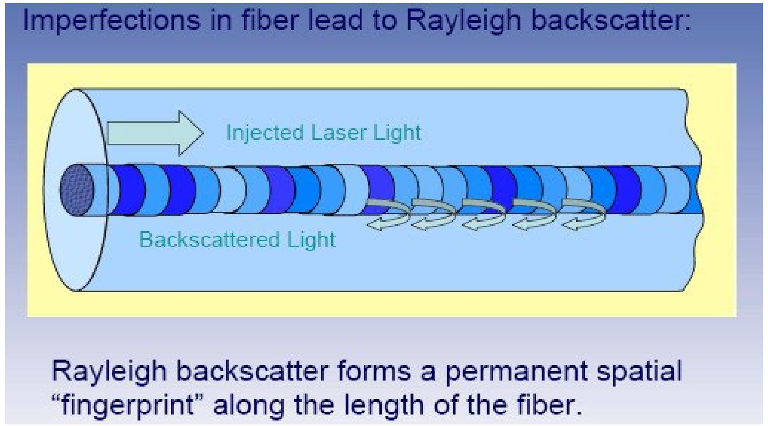
- Microbial conversion of hydrogen to methane in subsurface wells
- Need for real-time monitoring of gas composition and geochemical conditions.

Approach: Optical Fiber Sensors

Sensing Principle : Evanescent Wave Sensors



Distributed Sensing

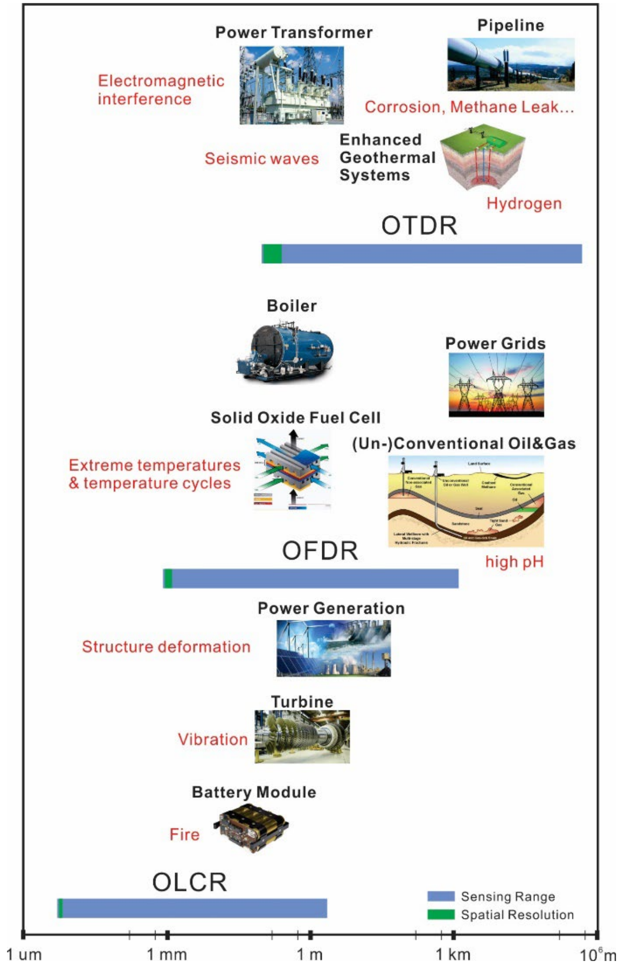


Advantages of Optical Fiber Sensors (OFS)

- **Improved safety** in the presence of flammable gases compared to electrical based sensors
- **Stable** in subsurface harsh environments
- Small size and flexibility
- Long reach, light weight
- Can be **functionalized** for targeted parameters through functional materials
- Compatible with **distributed or multi-parameter** interrogation.

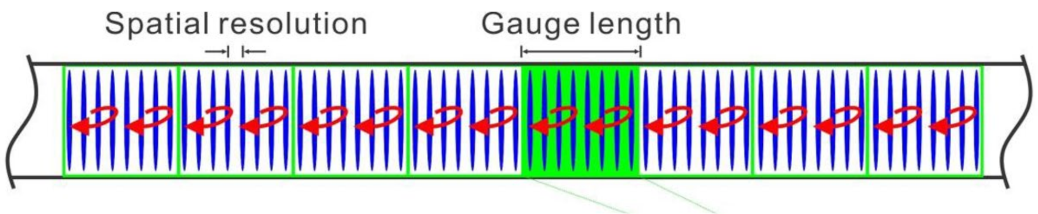
Need functional sensitive materials that enable H_2, CH_4, and geochemical sensing (e.g. pH and corrosion), which are compatible with high pressure high temperature and humid conditions in subsurface

NETL Capability in Distributed Optical Fiber Interrogator Development



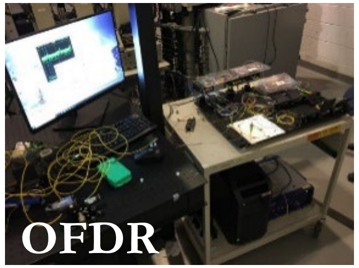
Ref: Lu et al, Appl. Phys. Rev. 6, 041302 (2019)

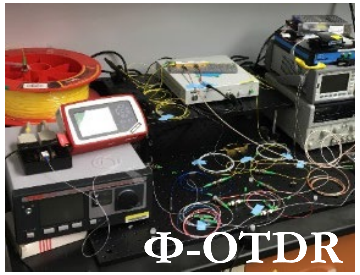
In-House NETL Distributed Optical Fiber Sensor Interrogators

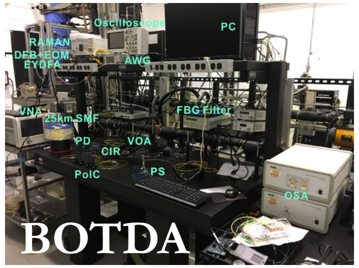


Technology	Sensing Range	Spatial Resolution	Measurement Time	Fiber Type	Sensing Performance
Coherent Rayleigh OFDR	m – km	mm – cm	seconds	SMF	Temperature, strain, vibration, chemical sensing
Coherent Rayleigh OTDR	km	m	seconds	SMF	Acoustic wave, vibration
Brillouin OTDR/BOTDA	> 100 km	cm – m	minutes	SMF	Temperature, strain,

Multiple Distributed Optical Fiber Sensing Platforms Have Been Developed to Enable Structural Health Monitoring of Natural Gas Pipeline, particularly for Corrosion Onset and Gas Leak Detection.







Previous work at NETL: Optical Fiber Hydrogen Sensor

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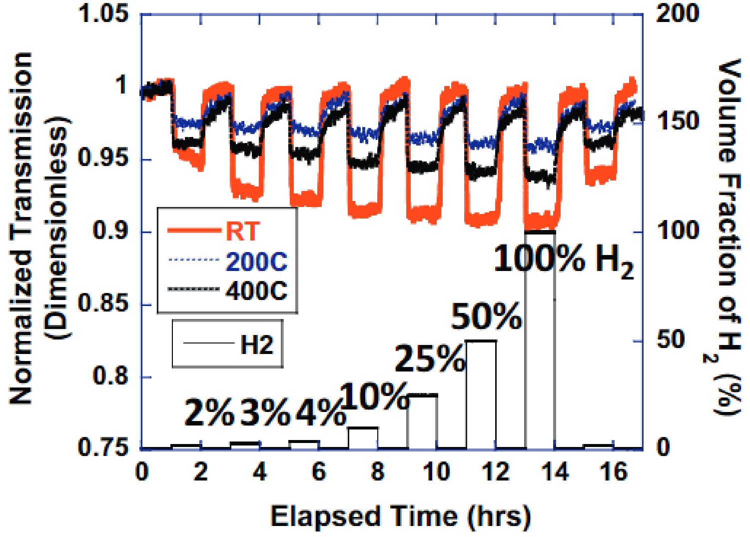
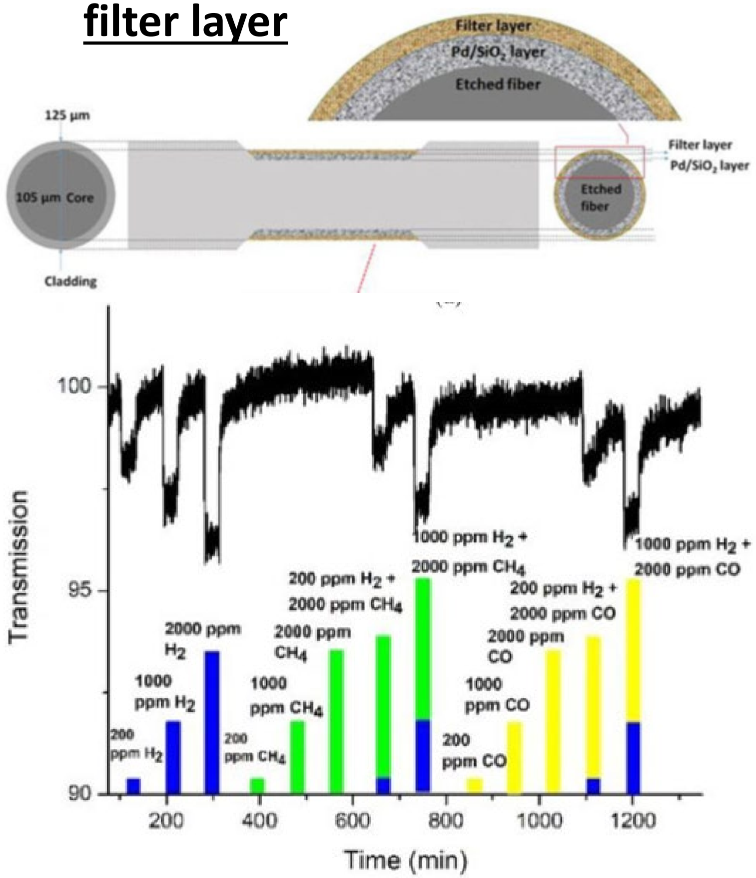
Pd Nanoparticle-Incorporated SiO_2 Thin Film coated Optical Fiber Sensor

H_2 Sensing from RT to 400 °C



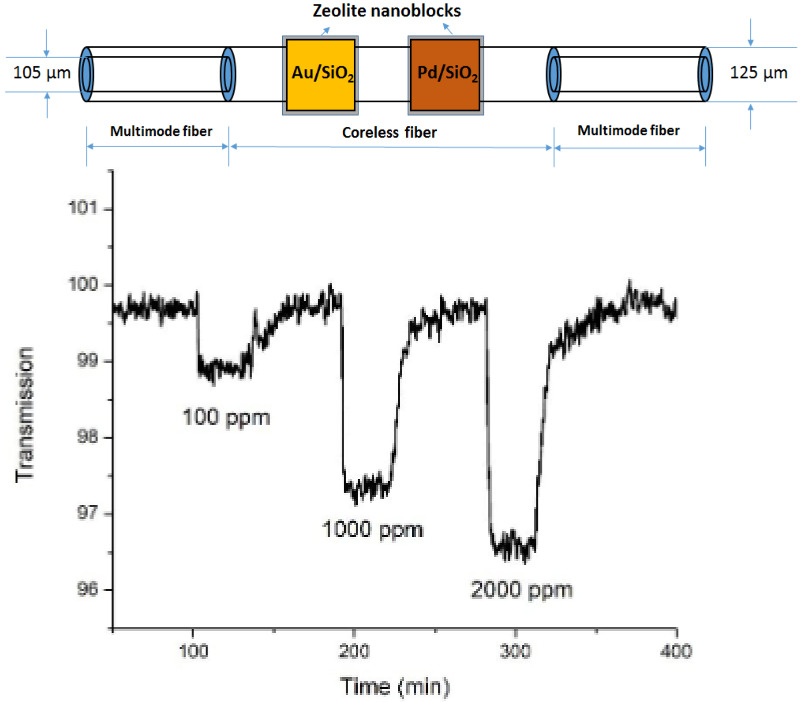
Ref: Ohodnicki et al, Sensors and Actuators B 214 (2015)159–168.

Selective H_2 Sensing with nano-filter layer



Ref: Sun et al, IEEE Sensors Letters, Vol. 1, No. 5, October 2017.

Multi-parameter sensing of H_2 and Temperature



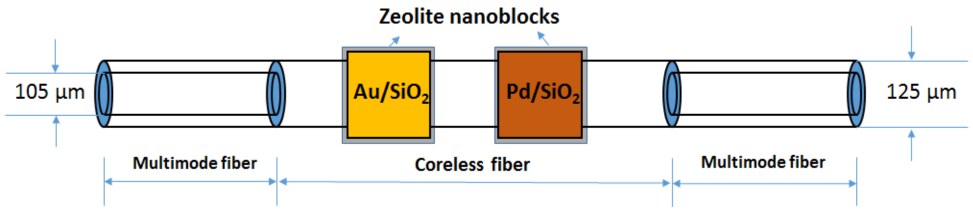
Ref: Sun et al, Proc. SPIE 10654, Fiber Optic Sensors and Applications XV, 1065405 (14May 2018);

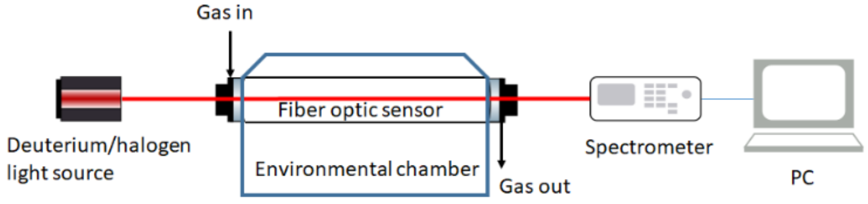
U.S. Patent No: 10,345,279
Issued: 07/09/2019
Title: Palladium and Platinum-based Nanoparticle Functional Sensor Layers and Integration with Engineered Filter Layers for Selective H_2 Sensing

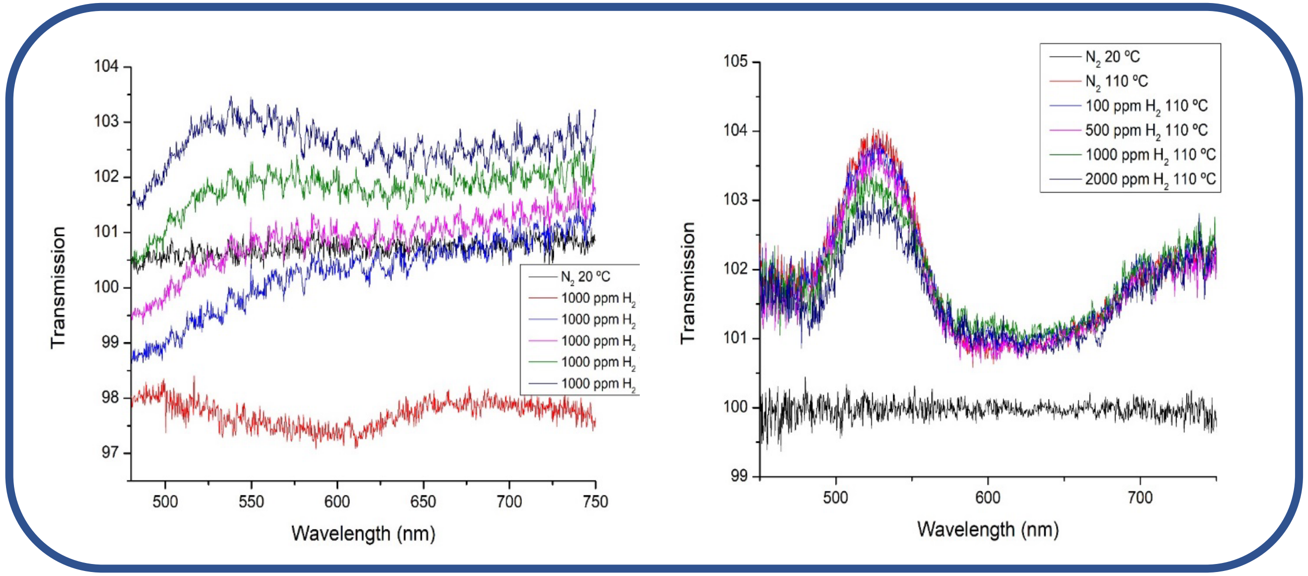
U.S. Patent No: 11,268,984
Issued: 03/08/2022
Title: Low-Cost Fiber Optic Sensor Array for Simultaneous Detection of Multiple Parameters

Simultaneous Detection of H_2 and Temperature

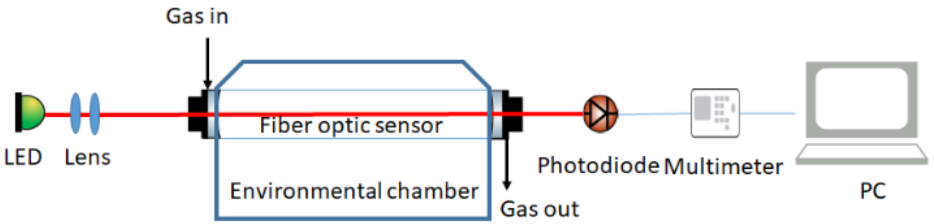
Functionalized Optical Fiber Sensor for H_2 and T



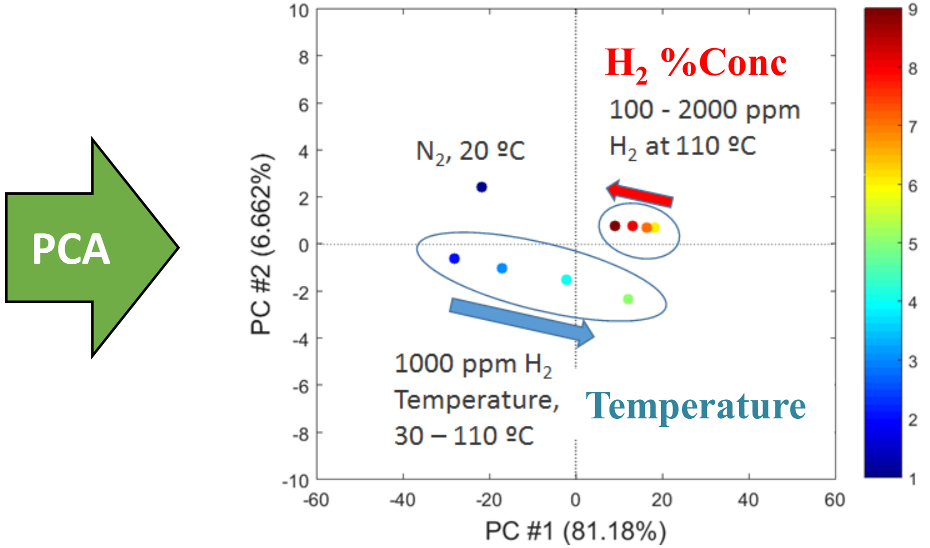




Low-cost Optical Fiber Sensor Design



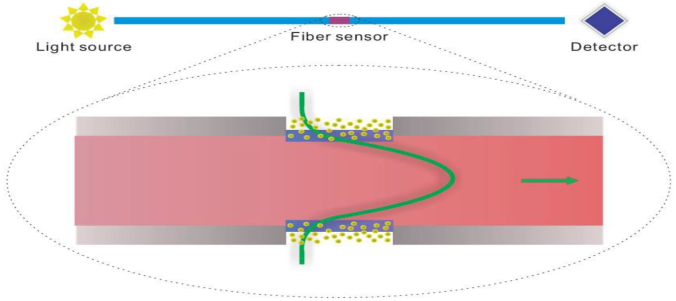
Simultaneous detection of H_2 and T was achieved through Au-Pd nanoparticles incorporated SiO_2 thin films via Principal Component Analysis (PCA).



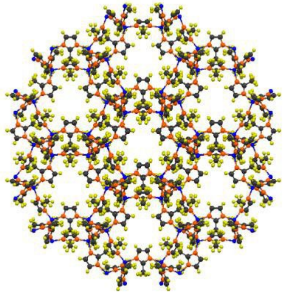
Ref: Sun et al, Proc. SPIE 10654, Fiber Optic Sensors and Applications XV, 1065405 (14May 2018);

Previous work at NETL: Optical Fiber Methane Sensing

Functional Sensing Layer Integrated Fiber Optic



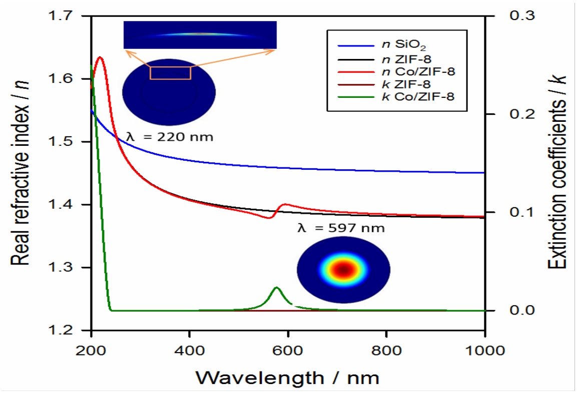
Porous Metal Organic Framework (MOF)



Micro-porous Gas Permeable Polymers

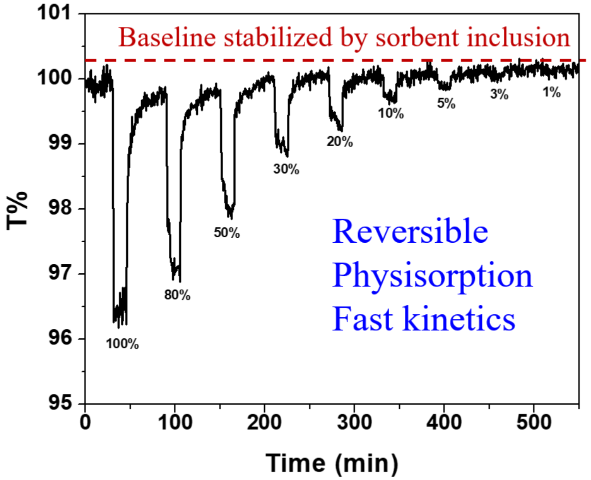
Evanescent Wave Absorption Based Sensors

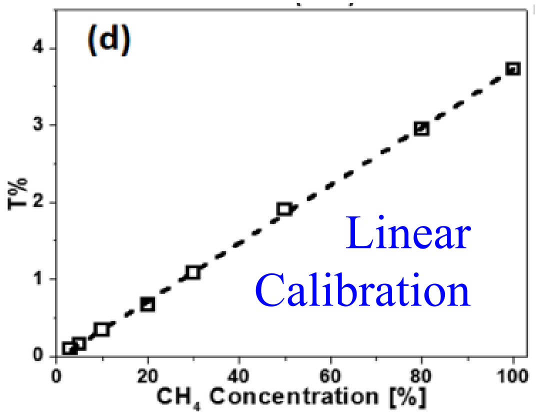
$$I_T(\lambda) = I_0 \exp[-\gamma\alpha(\lambda)CL]$$



Gas adsorption in the sensor coating causes $RI_{(coating)} > RI_{(fiber)}$, inducing optical power changes.

Ref: Kim et al, ACS Sensors, 2018, 3, 386-394.





Light Intensity Based Methane Sensing Technology. Integration of Fiber Optic Sensors with Engineered Porous Sensing Layers by Design.

Previous work at NETL: Distributed pH Sensing

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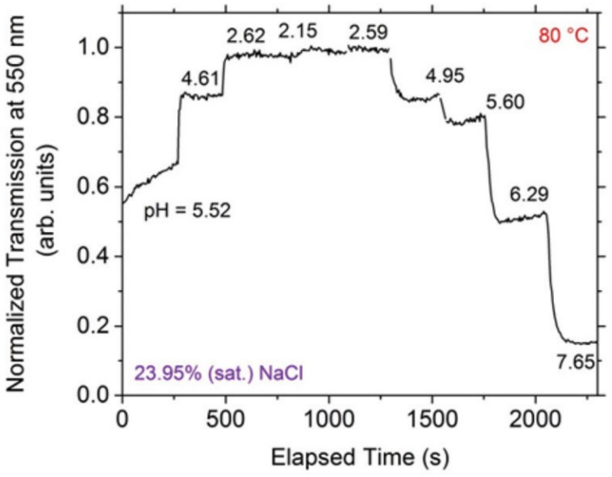
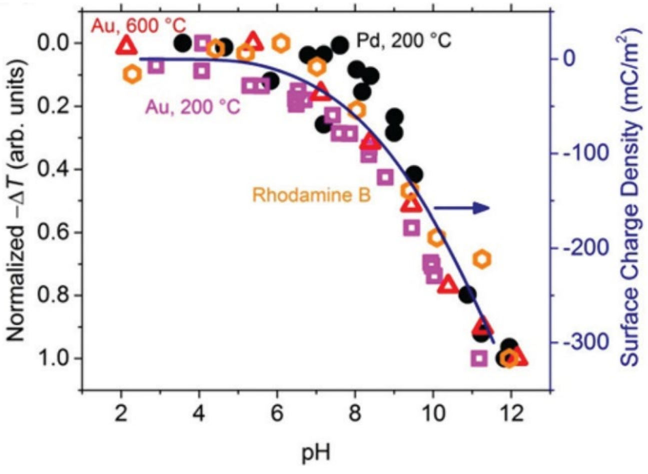
Au nanoparticles incorporated SiO_2 thin film

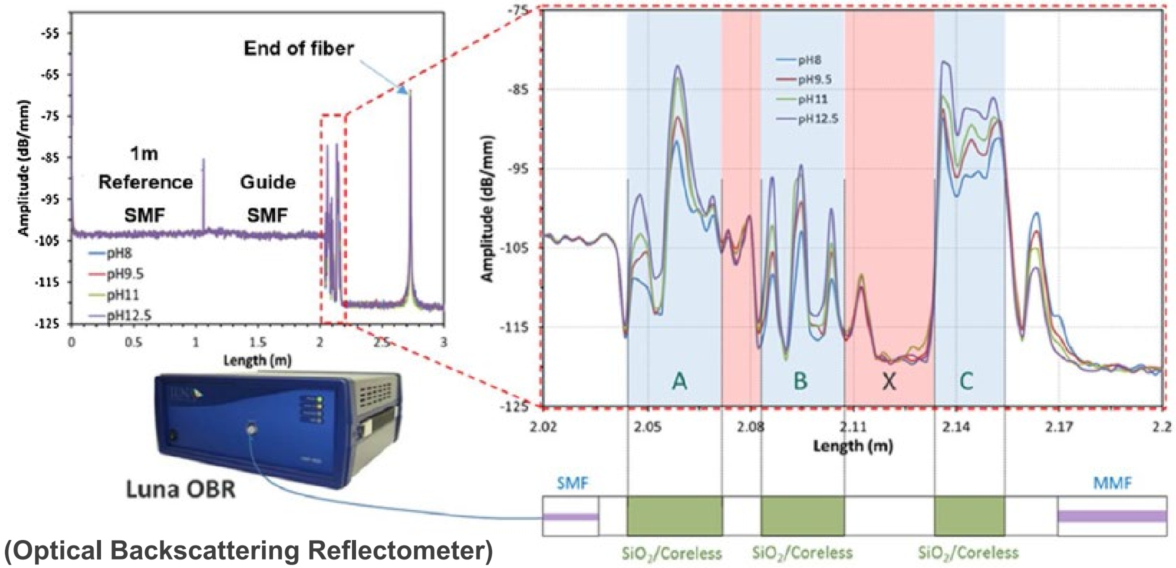


Optically active materials incorporated SiO_2 thin film



Ref: Wang et al, Nanoscale,2015,7,2527–2535

Distributed pH Sensing



(Optical Backscattering Reflectometer)

Ref: Lu et al, Sensors and Actuators B: Chemical, Volume 340, 1 August 2021, 129853

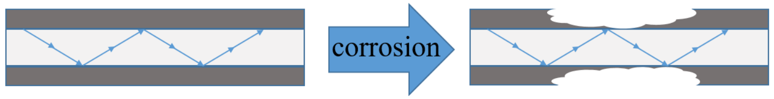
pH sensitivity was demonstrated with optically active materials-incorporated SiO_2 thin film using the optical fiber sensors.

Spatially distributed pH sensing with location information was demonstrated using the optical backscatter reflectometer (OBR) at room temperature. Only the functionalized SiO_2 coated segments have the pH sensitivity.

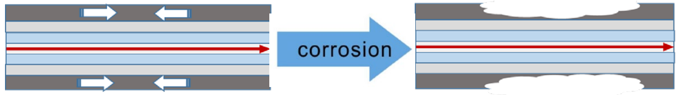
Previous work at NETL: Early-stage **Corrosion Detection**

Metallic film coated optical fibers

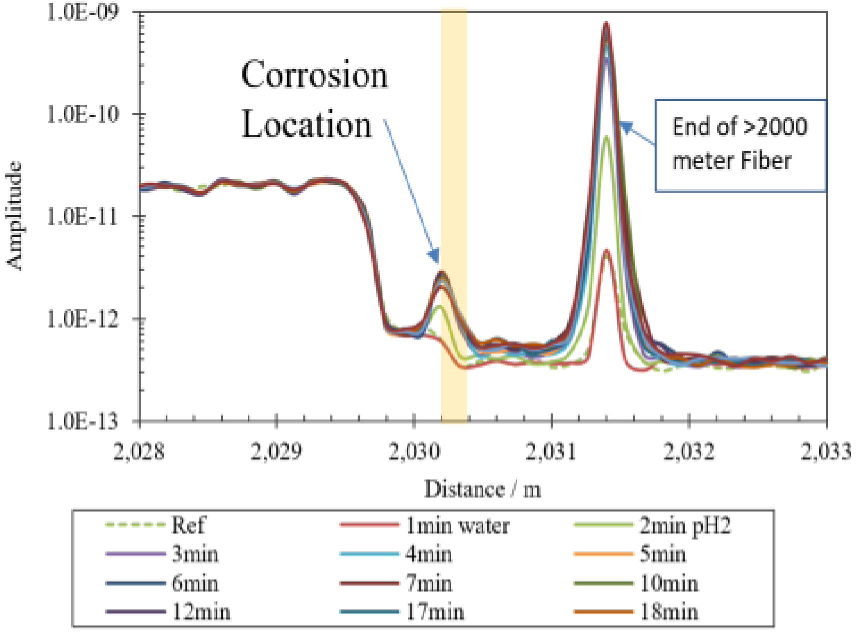
Optical Power Based

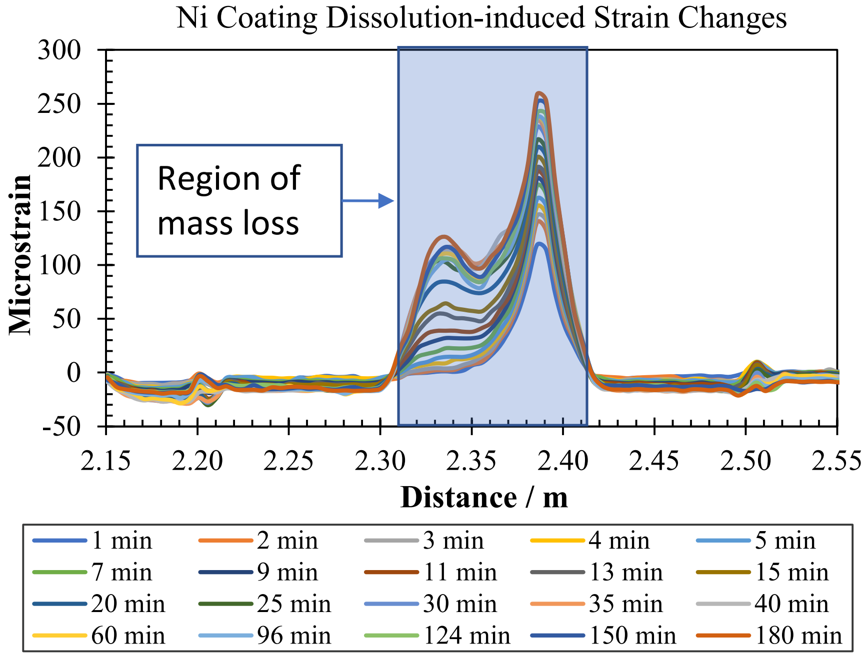


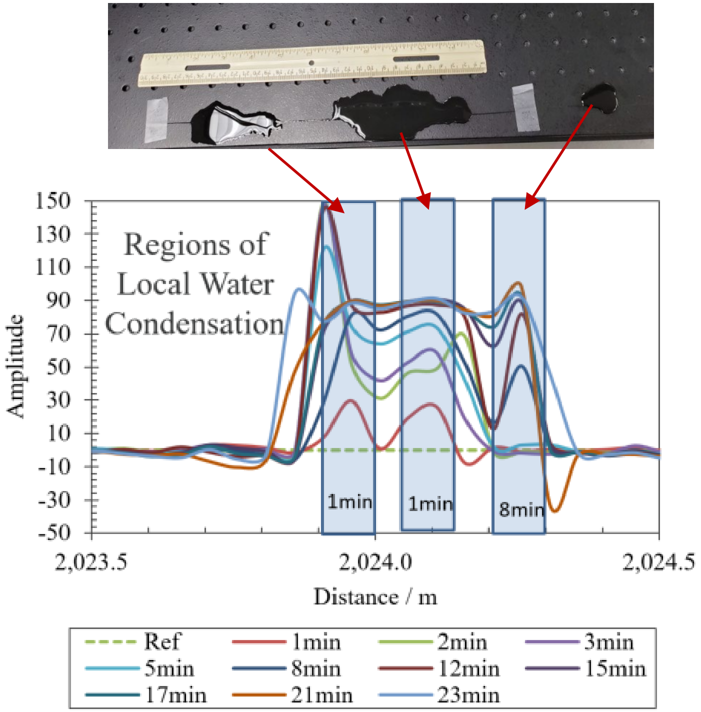
Strain Based



Water provides electrolytes for corrosion onset and is an indicator of potential corrosion.

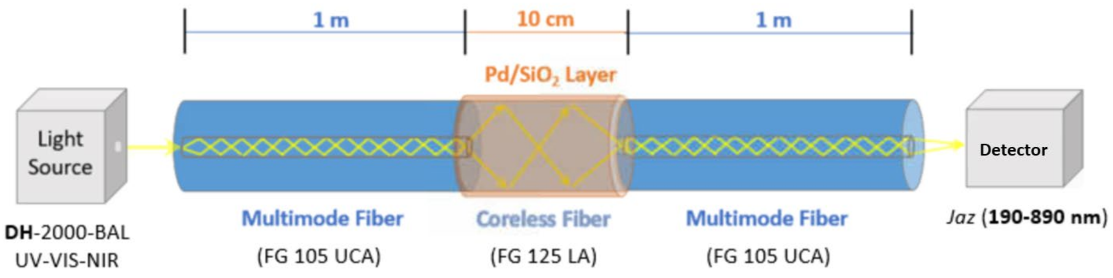






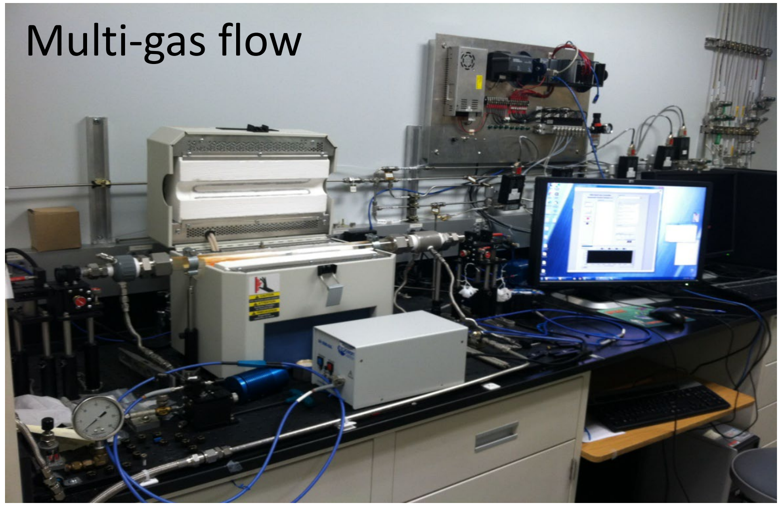
Corrosion can be detected and located along the optical fiber, which enables distributive corrosion monitoring for long-distance infrastructure.

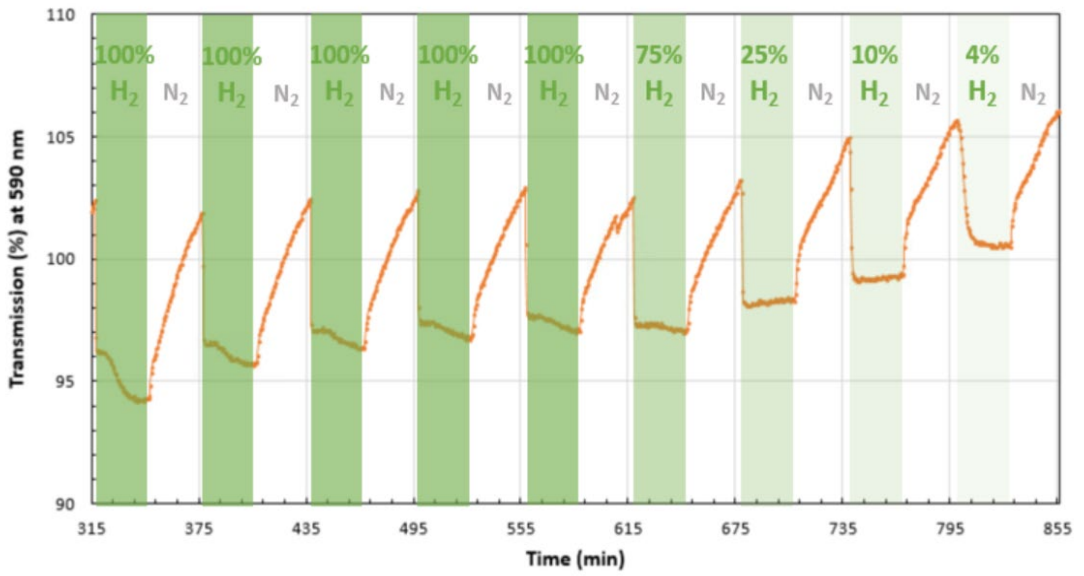
Progress: Optical Fiber H_2 Sensor



- Pd/SiO_2 as the hydrogen sensing material.
- Coreless fiber integrated structure allows sensing capability without etching the fiber cladding using hydrofluoric acid (HF).

Custom Gas Sensor Development Reactors

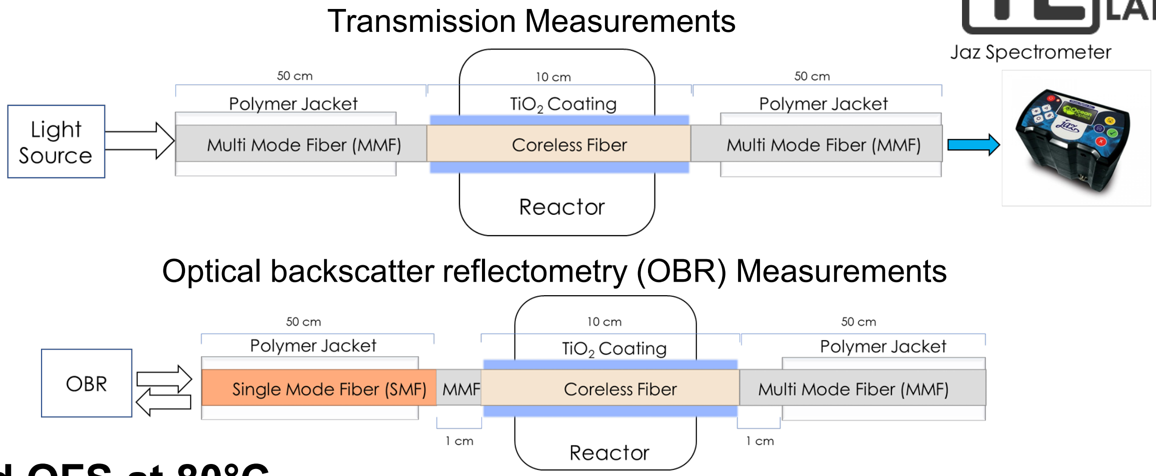




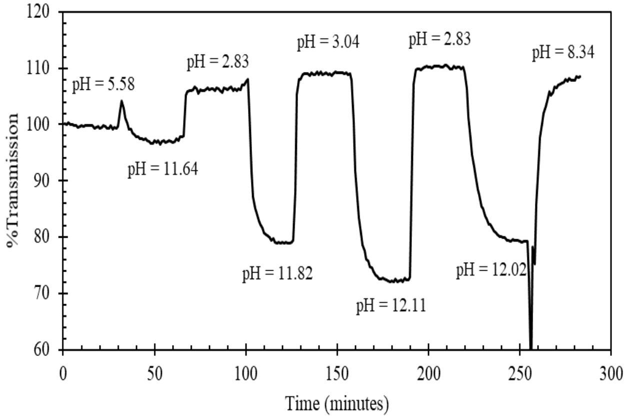
Pd/SiO_2 coated optical fiber H_2 sensor demonstrated reversible sensitivity for a wide range of H_2 concentrations (4%-100%).

Progress: Optical Fiber pH Sensor

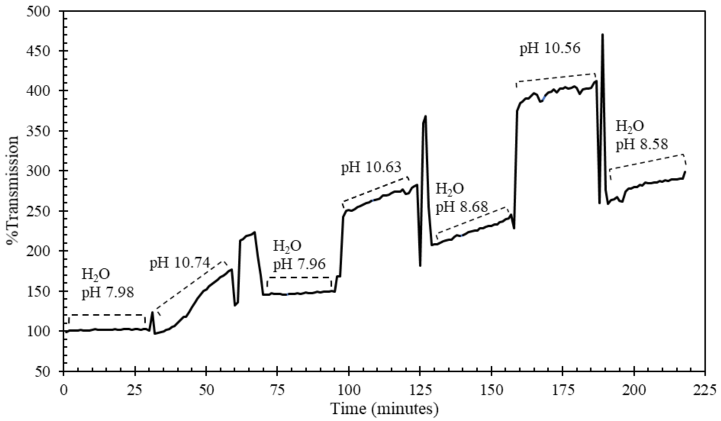
Multi-phase high pressure high temperature autoclave for pH sensor testing

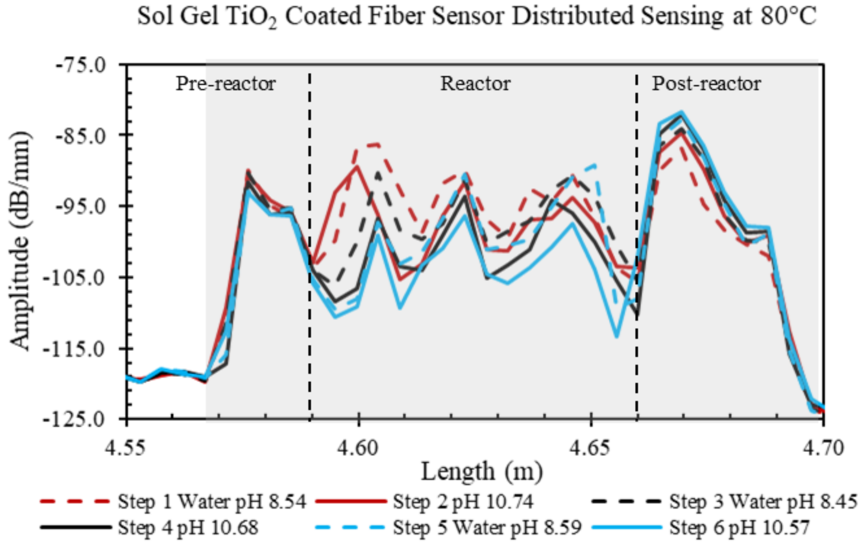


SiO_2 coated OFS at room temperature



TiO_2 coated OFS at 80°C



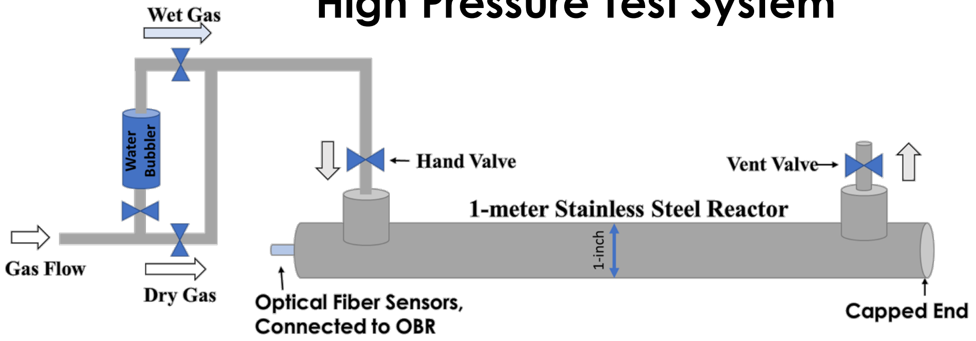


Ref: Shumski et al, Proc. SPIE 12105, SPIE Defense + Commercial Sensing (SI22), 12105-21, 2022.

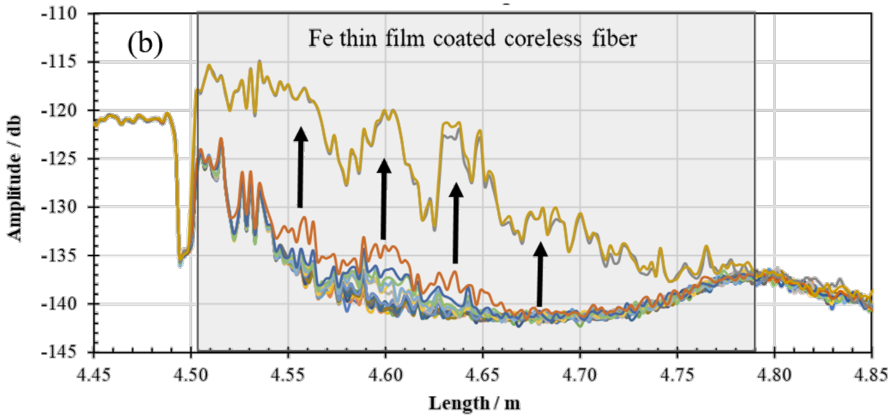
- **Oxide-based sensing layers were developed for pH sensing with stability at room temperature and elevated temperature.**
- **Sol-gel TiO_2 coated optical fiber was demonstrated for distributed pH sensing at high temperature relevant for subsurface condition.**

Progress: Optical Fiber Corrosion Sensing at High Pressures

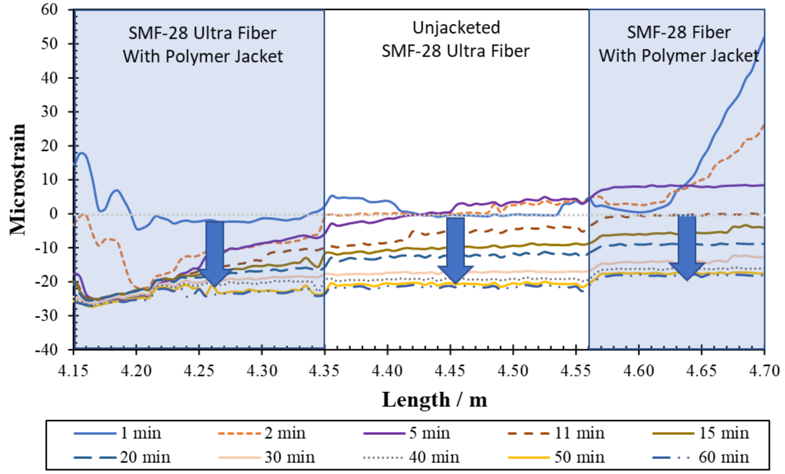
High Pressure Test System



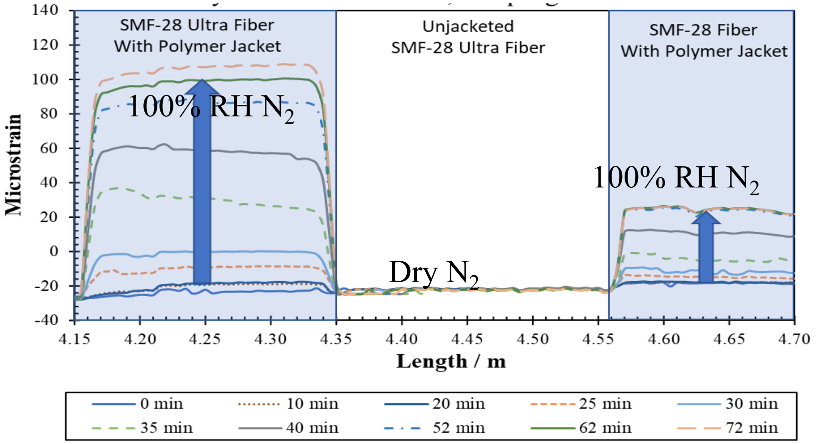
Corrosion Monitoring in 850 psi humid CO_2



Pressure Monitoring from 14.7 to 900 psi N_2



Humidity Monitoring at 900 psi



Ref: Wright et al, AMPP 2022 Annual Conference + Expo, Paper C2022-17804

- **Metallic coated OFS successfully demonstrated corrosion sensing at high pressures.**
- **Pressure and humidity monitoring were demonstrated using commercial single-mode fibers.**

Accomplishments and Future Plans

Accomplishments

- An abstract was submitted to 2022 AIChE Annual Meeting (November 13-18, 2022), titled "Pd-nanoparticle enabled optical fiber hydrogen sensor for subsurface storage conditions" authored by D. Kim, N. Diemler, R. Wright, M.P. Buric, P.R. Ohodnicki.
- A presentation and a conference paper: "TiO_2 Coated Optical Fibers for Distributed Real-Time pH Monitoring in Wellbore Conditions" authored by Shumski, A., Diemler, N., Wright, R., Lu, F., Ohodnicki, P. and Su, Y., " was presented at SPIE Defense + Commercial Sensing 2022 conference (April 3-7) and accepted to *Proc. SPIE 12105,* SPIE Defense + Commercial Sensing (SI22), 12105-21, 2022.
- A presentation and a conference paper: "Metallic Film-Coated Optical Fiber Sensor for Corrosion Monitoring at High Pressures," authored by Wright, R.F., Diemler, N., Baltrus, J., Ohodnicki, P.R., Jr., Ziomek-Moroz, M., and Buric, M., was presented at 2022 AMPP Annual Conference + Expo, March 6-10.
- A non-provisional patent was field on April 26, 2022, titled "Metal Oxides Enabled Fiber Optic pH Sensor for High temperature High pH Subsurface Environments" invented by F. Lu, R. Wright, P. Lu, P. R. Ohodnicki.

Future Plans for Sensor Development and Testing

EY2021: Room temperature and ambient pressure → EY2022: Humid, mixed gas conditions → EY2023: High pressure, high temperature conditions

Acknowledgement

Program Manager: Evan Frye (DOE, FECM)

Technology Manager: Jared Ciferno (DOE, FECM)

Technical Portfolio Lead: Angela Goodman (NETL)

Sensor Team: Ruishu Wright (PI), Nathan Diemler, Daejin Kim, Alexander Shumski

This work was performed in support of the U.S. Department of Energy's Fossil Energy Oil & Natural Gas Research Program. The Research was executed through the NETL Research and Innovation Center's Subsurface Hydrogen/Natural Gas Storage FWP. Research performed by Leidos Research Support Team staff was conducted under the RSS contract 89243318CFE000003.

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