Safe Detector System for Hydrogen Leaks

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Project ID #
SCS014

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Overview

Time Line

- Start –June 2007
- Finish- June2011
- 95% Complete

MYPP Barriers/Targets

- **Delivery**: Barrier I. Hydrogen Leakage and Sensors
- **Storage**: Barrier H. Balance of Plant (BOP) Components
- **Safety:** Targets
- (Also: **Fuel Cells**, **Manufacturing**, and **Tech. Validation**)

Budget

- Current Project funding: $1,189,374 (DOE… $951,500, IOS share...$237,874)
- FY08/09 Project funding: $1,230,00 (DOE... $984,000, IOS share… $246,000)
- Funding received in FY07: $495,000

Partners

- Dr. Gerald Voecks – Advisor
- Dr. Angelo A. Lamola – Consultant
- Mr. Gerald Cole – Consultant
- NREL- Testing and Validation Provider
- Intelligent Optical Systems, Inc.– Program Lead

Project Goal:

- Select and finalize hydrogen sensor technology
- Design and fabricate scalable prototype sensors
- Investigate and establish end-user market size and cost analysis

Technical Objectives:

Overall	♦ Integrate IOS' proprietary hydrogen indicator chemistry into a complete optoelectronics package with well-defined sensing characteristics and a known end-use market ♦ Identify different formulations and physical embodiments to meet specific markets requirements
CY 07/08	♦ Develop polymer based hydrogen sensor chemistry ♦ Establish response to low levels of hydrogen in one or more candidate substrates ♦ Establish good hydrogen sensitivity and response time in ambient air environment ♦ Develop compact multi-channel test system
CY08/09	♦ Finalize indicator chemistry immobilization into porous glass optrodes; reduce or eliminate sensitivity to moisture and oxygen with polymeric barrier coating ♦ Develop sensor polymers for two distinct embodiments: point sensors and distributed sensors ♦ Optimize integrated optic sensor composition and fabrication ♦ Design and fabricate optoelectronic interface for integrated optic sensors ♦ Demonstrate feasibility of making intrinsically hydrogen sensitive fibers for distributed sensing
CY09/10	♦ Select and finalize hydrogen sensor chemistry that possess the optimum sensitivity, reliability, reproducibility, and aging performance ♦ Finalize and fabricate optoelectronic board for hydrogen leak sensor ♦ Assemble packaged prototype hydrogen point sensor that meets DOE specifications ♦ Test and validate the full packaged prototype performance at NREL testing laboratories.

Relevance

Barriers Addressed

- **Delivery:** Barrier I. Hydrogen Leakage and Sensors (MYPP page 3.2-20: "Low cost hydrogen leak detector sensors are needed")
- **Storage:** Barrier H. Balance of Plant (BOP) Components (MYPP page 3.3-14: "Light-weight, cost-effective… components are needed…These include… sensors")
- **Manufacturing:** Barrier F. Low Levels of Quality Control and Inflexible Processes (MYPP page 3.5-11: "Leak detectors… are needed for assembly of fuel cell power plants.")
- **Technology Validation:** Barrier C. Lack of Hydrogen Refueling Infrastructure Performance and Availability Data (MYPP page 3.6-8: "…the challenge of providing safe systems including low-cost, durable sensors [is an] early market penetration barrier")

Source: "DOE Hydrogen, Fuel Cells and Infrastructure Technology Multiyear Research, Development and Demonstration Plan" (MYPP), 2007 edition. http://www1.eere.energy.gov/hydrogenandfuelcells/mypp/

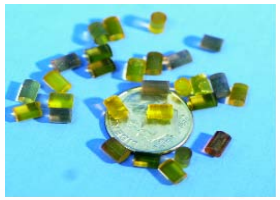
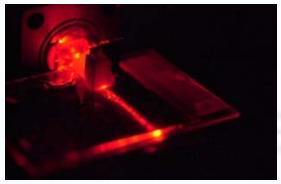
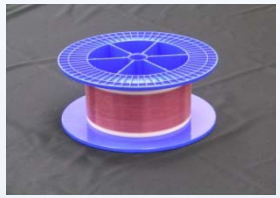
Relevance

Performance Targets

- Sensor Product Specifications (4Q 2010):
  - Range: 0 – 100% H_2
  - Sensitivity : (min) 0.1%H_2 - 4% of reading
  - Environment: Ambient air, 5-95%RH, and 0-55ºC range.
  - Interference resistant (e.g: moisture, hydrocarbons, oxygen)
- Applications:
  - Safety in distribution/production facilities
  - Leak detection
  - Home/garage safety
  - Vehicular safety

Relevance

Technical Approach:
Selection of Optical Hydrogen Sensing Platform

IOS's Sensor Formats	Sensitivity Range (Level of detection, LOD)	Response Time@ 4%	Reproducibility
 Point	**0.2-10%** (0.1%)	2-3 seconds	>90%
 Integrated	**0.4-10%** (0.1%)	20 seconds	80-85%
 Fully Distributed	**1- 4%** (0.4%)	8-12 seconds	80-85%

Approach

FY 09-10 Technical Tasks/ Milestones

Completion	
100%	Task 1: Investigate Designs, Materials, and Indicators for Improved Hydrogen Sensors
100%	Task 2: Optimize, Fabricate, and Test Improved Hydrogen-Sensing Optrodes
100%	Task 3: Optimize, Design, Fabricate, and Test Multi-Channel Integrated Optical Waveguide Sensor Chip
100%	Task 4: Design and Integrate Optoelectronic Interface to Waveguide Sensor Chip
100%	Task 5: Test and Characterize Packaged Multi-Channel Integrated Waveguide Hydrogen Sensor
100%	Task 6: Investigate, Design, Fabricate, and Evaluate Prototype Distributed Hydrogen-Sensing Fiber
100%	Task 7: Perform Hydrogen Sensor Market Study under DOE Guidance
100%	Task 8: Continue FY07 Investigation of Irreversible Chemistry for Hydrogen Sensing
100%	Task 9: Optimize hydrogen sensor element fabrication process; finalize preparation protocol steps
100%	Task 10: Complete environmental testing for hydrogen sensor elements.
100%	Task 11: Identify potential cross-contaminants; test and optimize hydrogen sensor elements
Milestone 1	Complete cross-contamination testing of sensor elements and determine whether additional selective permeable membranes and/or scrubbing layers are needed.
100%	Task 12: Build optoelectronic interface to optrode sensor array modular package
95%	Task 13: Design and integrate software data processing including internal calibration curves
90%	Task 14: Perform complete test of packaged integrated hydrogen sensor, including sensitivity, response time, selectivity, false alarms, and temperature/humidity characteristics.
Milestone 2	Complete cross-contamination testing of sensor elements and determine whether additional selective permeable membranes and/or scrubbing layers are needed.
87%	Task 15: Perform hydrogen sensor market study.
85%	Task 16: Management and reporting

Approach

Project Plan: FY08 –FY10

- Identify critical sensor applications that mitigate hydrogen liability issues
- Research and develop reliable hydrogen sensors that fit these applications
- Engineer and commercialize cost-effective hydrogen detection systems

- Define hydrogen indicator chemistry
- Evaluate various optical sensor matrices
- Validate the sensor response/ cross-interference
- Develop integrated optic point sensor design
- Refine point sensor format based on market needs
- Develop hydrogen sensor fiber for cable-based system
- Select and finalize sensor format
- Finalize, fabricate, and test optoelectronic system
- Test complete packaged sensor prototype
- System certification and validation testing
- Establish commercial market and partnerships

Approach

Hydrogen Sensor Development

- **Optimization of hydrogen sensor chemistry:**
  - Improved sensitivity through controlled chemistry immobilization and activation steps
  - Improved reproducibility and higher yield of fabrication
- **Optoelectronic design and sensor prototype fabrication:**
  - Design and develop compact optoelectronic board featuring integrated user interface, low power consumption, audio and visual alarm, replaceable sensor chip, and rechargeable battery option
  - Assemble and test complete point sensor product prototype
- **Substrate testing :**
  1. Prepared hydrogen sensitive substrates are stored at ambient conditions
  2. Mount optrodes onto designed optoelectronic prototype board
  3. Measure intensity in real-time as a function of hydrogen concentration at various humidity, temperature, and flow-rates

Approach

Hydrogen Sensor Testing Station

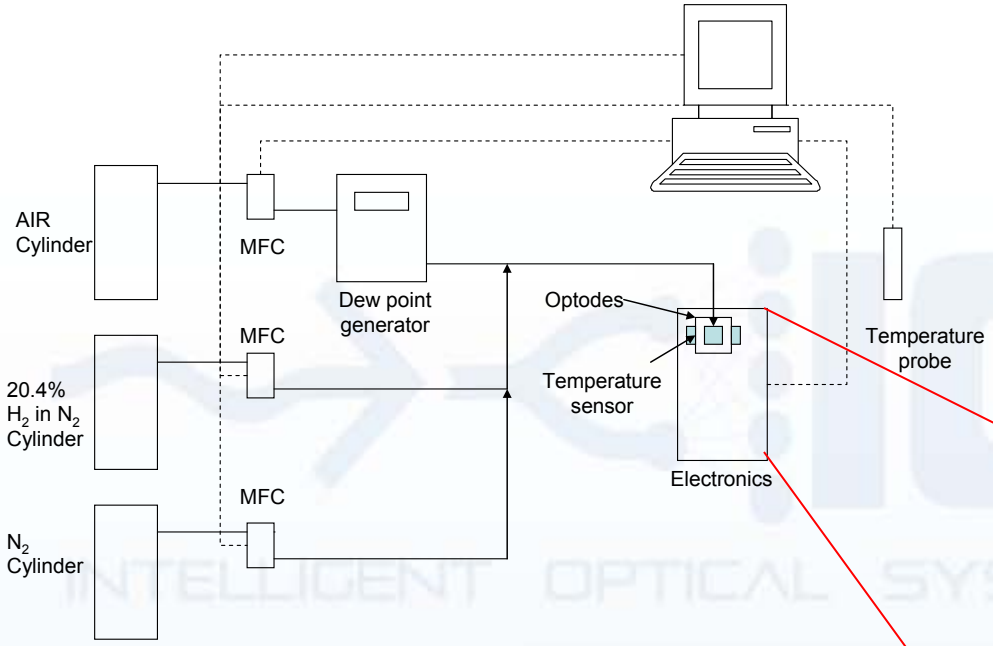


Illustration of the gas testing setup

hydrogen sensor in 4"X 6" testing prototype

Approach

IOS Approach to Hydrogen Sensing

Real-Time Monitoring of H2-Induced Color Change

Schematics of the envisioned Hydrogen Sensor.

Illustration of a potential usage in civilians' car garages as a Hydrogen leak detector.

Approach

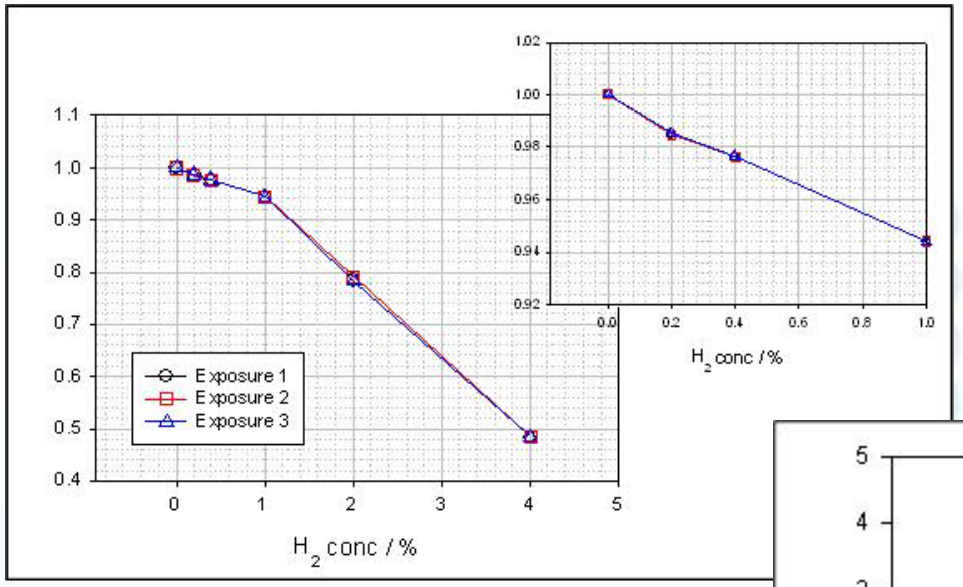
Technical Accomplishments: Sensor Prototype Specifications

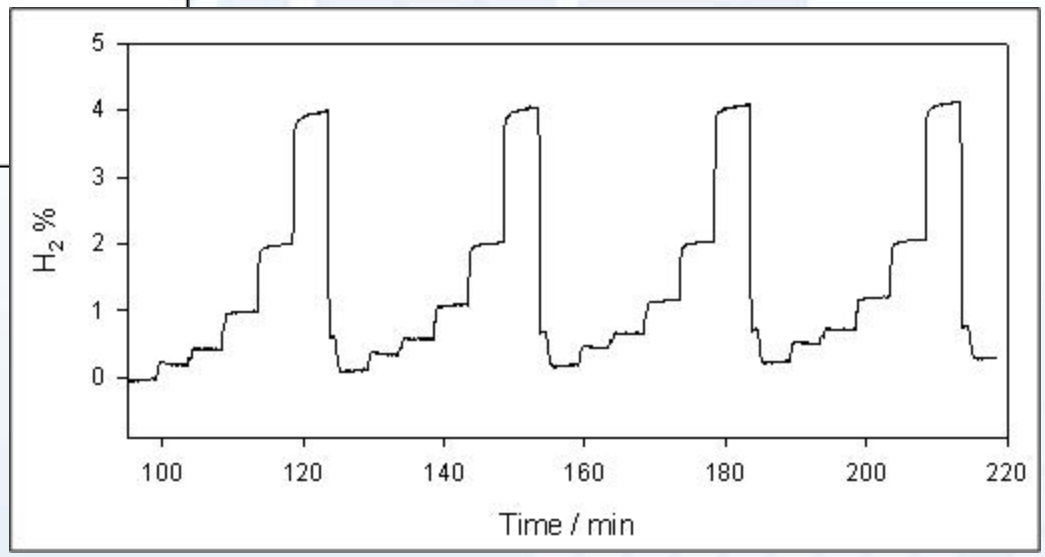
Operating Range	100 ppm to 10%
Accuracy	2% of Concentration (0 to 3% Range) 5% of Concentration (3 to 10%Range)
Response Time	Less than 5 seconds
Recovery Time	Less than 5 seconds
Warm up	2 seconds
Power	Battery, USB, or AC adapter
Battery type	Lithium Ion rechargeable
Battery Life	10 hours
Charge Time	From full discharge -- 1.5 hours Operational immediately
Enviromental Correction	Compensated for temperature and humidity
Temperature Range, Operating	10°C to 55°C (compensated)
Temperature Range, Storage	-30°C to 100°C
Humidity Range, Operating	0 - 90%RH, Non-Condensing (compensated)
Product Life	>5 Years
Calibration Frequency	Every 3 months

Sensor Type	Hydrogen Gas Leak in Air
Application	Hand held or wall mounts
Technology	Intrisically safe optical sensing
Display	2-line backlit LCD
User Interface	Four buttons or serial interface
Alarm Indicatorss:	Flashing LED and Buzzer
Auxillary Indicator	3-color LED for charging and battery
Serial Communication	USB 2.0, RS-232
Enclosure	Anodized Aluminum
Input Voltage	5VDC 10W

Technical Accomplishments	Relevance

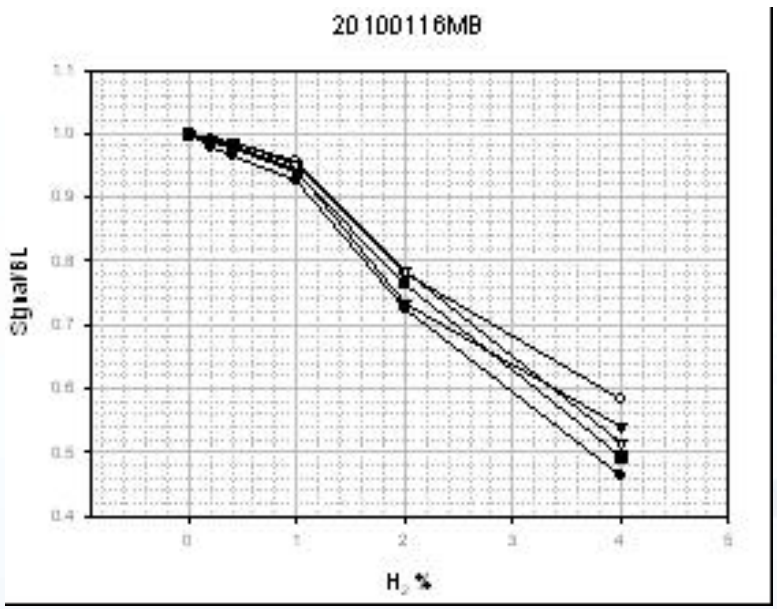
•Hydrogen Sensor:
- Repeatability

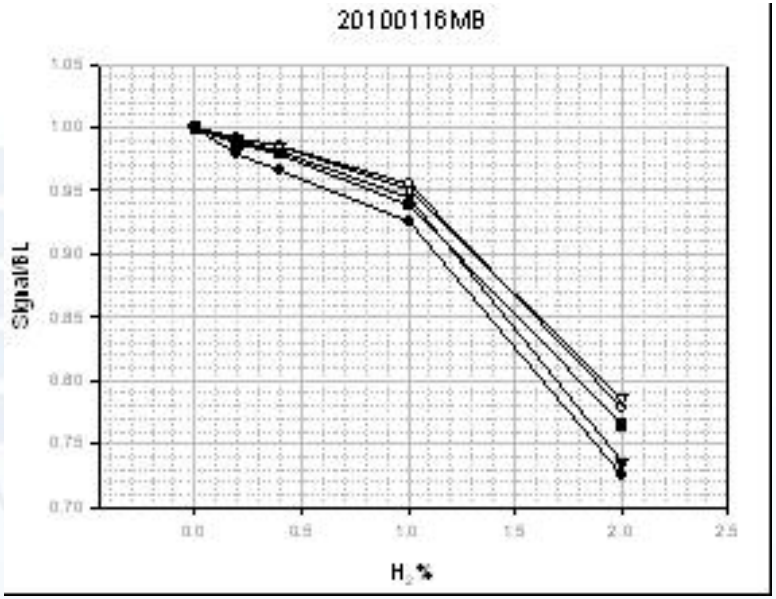




Technical Accomplishments

Hydrogen Sensor: -Fabrication Reproducibility

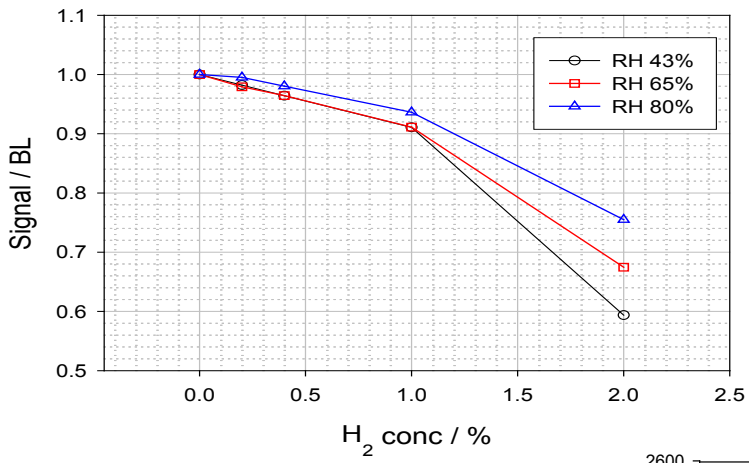




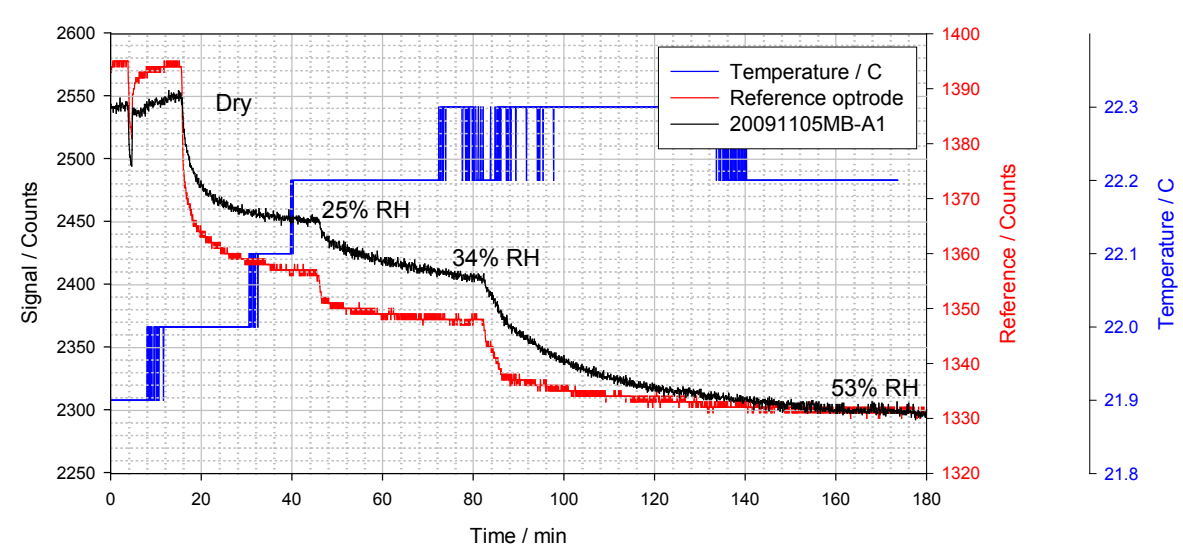
Sensor response of 5 different substrates from two different batches to 0 – 4% H2 in air at 40-45% RH.

Technical Accomplishments

Hydrogen Sensor:
- Humidity/ Temperature Effect

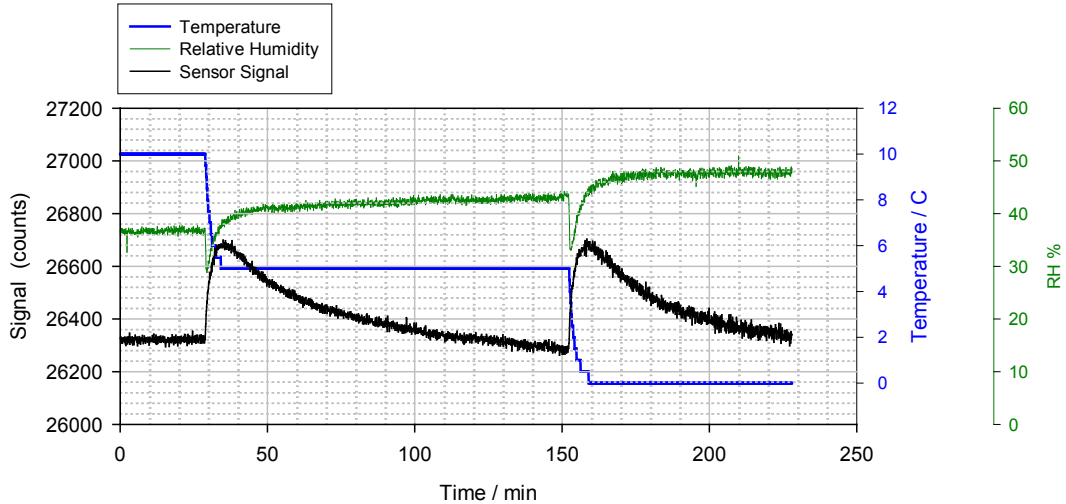


- Humidity dependence is corrected via a reference channel
- Temperature effect was noticed through humidity changes

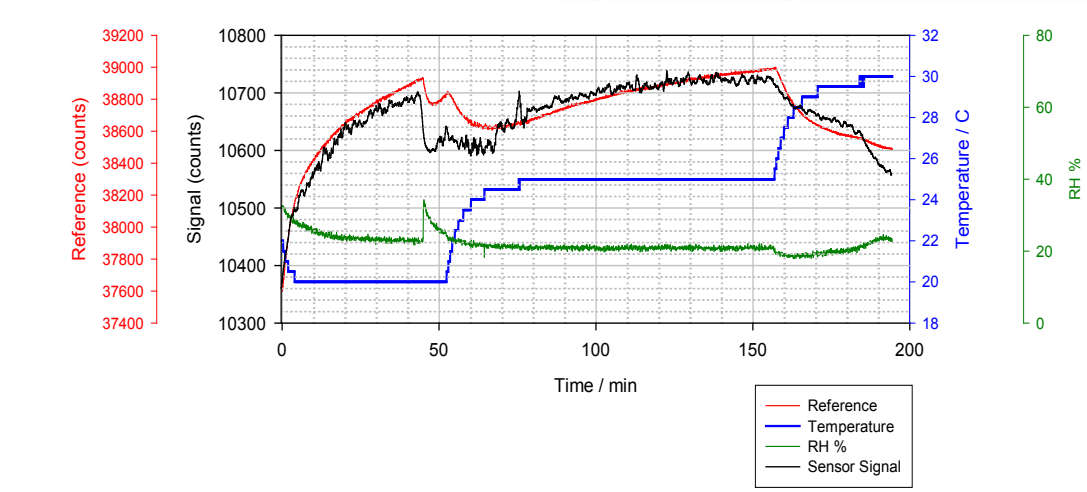


Technical Accomplishments

Hydrogen Sensor:
- Humidity/ Temperature Compensation

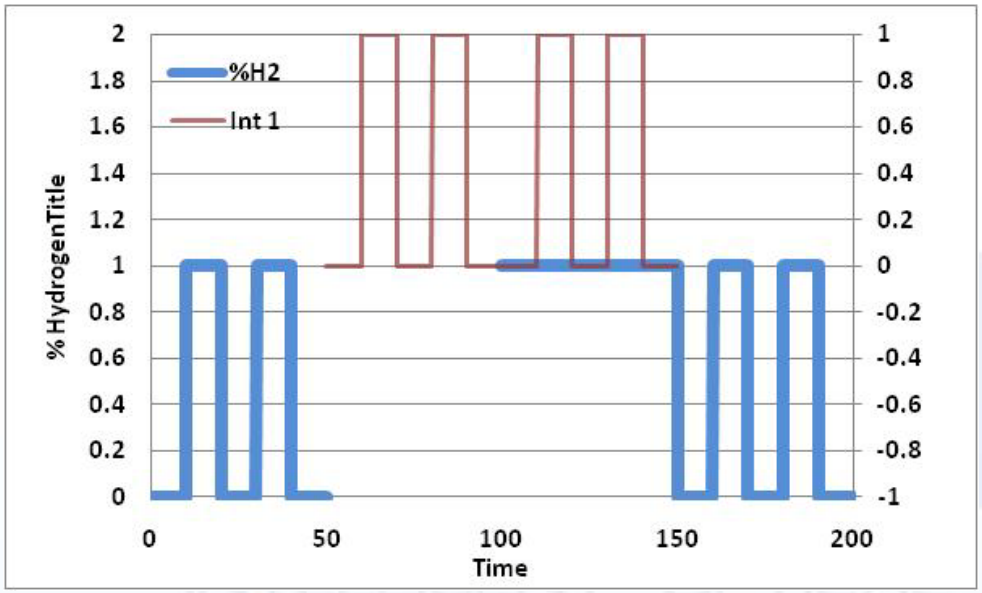


Clear correlation between sensor and reference channel in various temperature and humidity conditions.



Technical Accomplishments

Hydrogen Sensor: - Cross interference

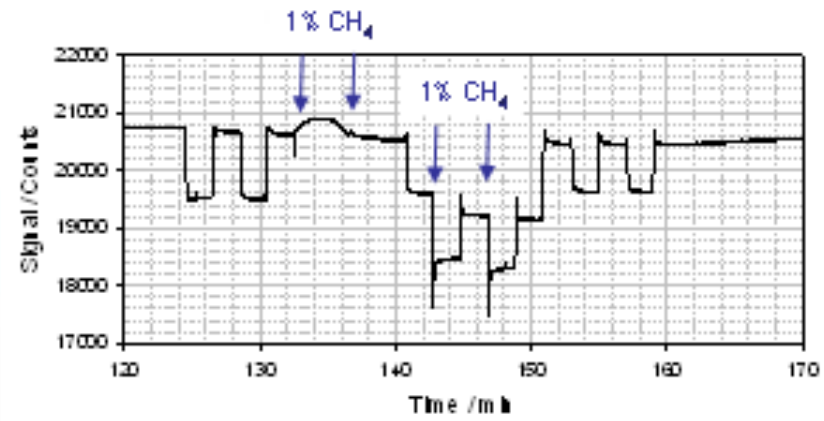


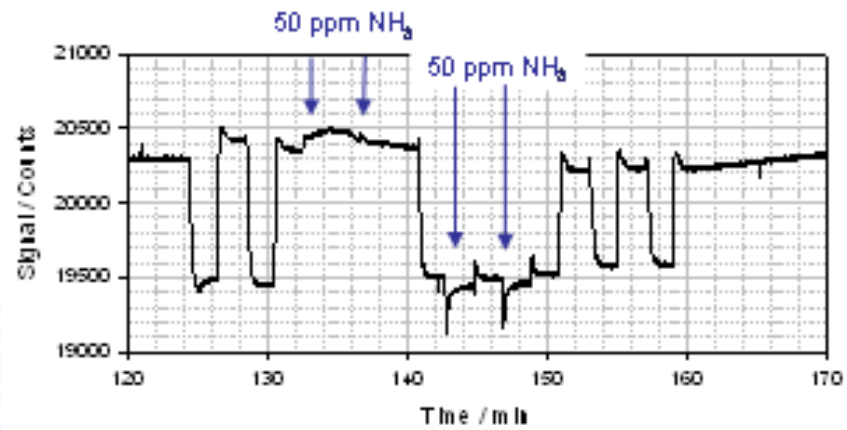
Cross-interference testing scheme (NREL Test Protocol)

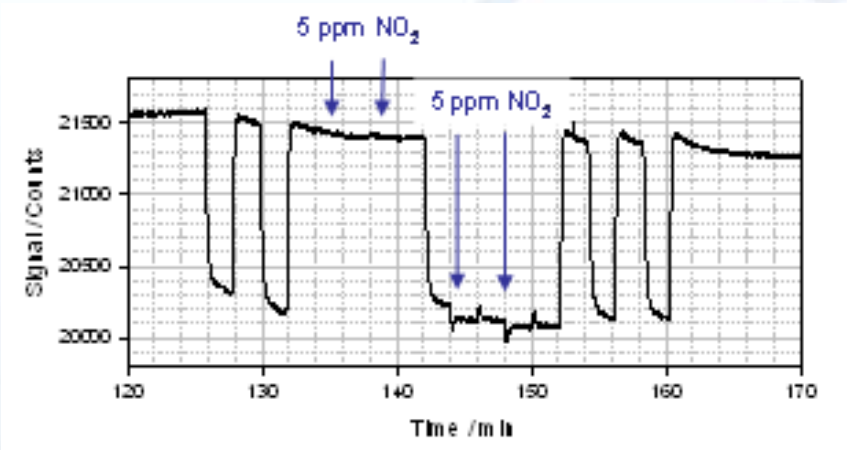
Potential Interferent	Test Conc. (in air)	Test Conc. (in 1% H_2)
Carbon Monoxide	50 ppm	50 ppm
Nitrogen Dioxide	5 ppm	5 ppm
Hydrogen Sulfide	20 ppm	20 ppm
Methane	1%	1%
Ammonia	50 ppm	50 ppm

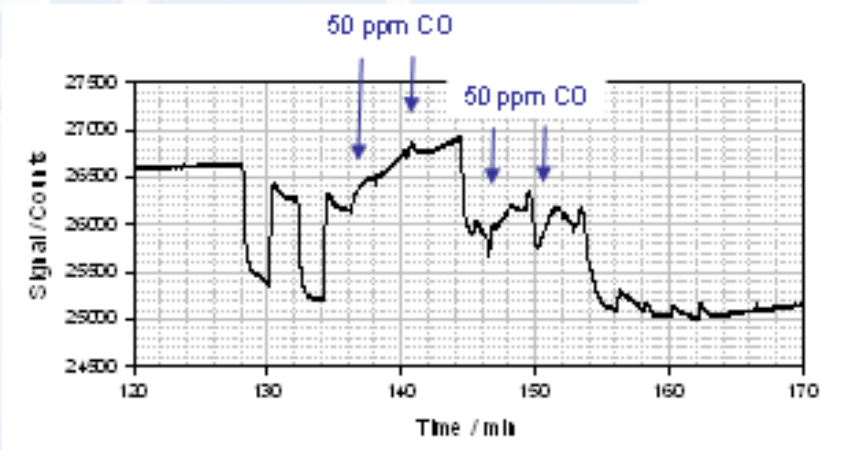
Technical Accomplishments

Hydrogen Sensor:
- Cross interference









Technical Accomplishments

Collaborations/Acknowledgements

- **Gerald Voecks – Advisor**
  - Fuel cell applications and commercialization
- **Angelo A. Lamola – Consultant**
  - Photochemistry/indicators
- **William Buttner, Robert Burgess, Matthew Post – NREL Testing Collaborators**
  - Hydrogen sensor testing and validation
- **Intelligent Energy**
  - Customer/commercialization partner
- **DOE Codes and Standards:** We thank Antonio Ruiz, DOE for his continued support of this work.

Collaborations

Summary

Relevance:

- Reliable, cost-effective hydrogen sensors are needed for the Delivery, Storage, Manufacturing, Fuel Cell, and Safety Key Activities of the DOE Hydrogen Program. Applications range from garage and passenger compartment safety to leak detection in production facilities and refueling stations.

Approach:

- High performance, low cost optical sensors based on indicator chemistry can meet projected needs
  - Integrated optic sensors and optrodes are ideal for single-point or multiple-point detection
  - Compact optoelectronic prototype is suitable for hand-held and wall mounting applications

Technical Accomplishments:

- Improved indicator chemistry performance (scalable and reproducible fabrication technique)
- Compensated humidity and temperature effects on sensor signal
- Integrated sensor elements into a compact prototype optoelectronic package
- Tested and validated sensor capabilities at National Renewable Energy Laboratory NREL
- Executed market size and cost analysis for sensor commercialization

Collaborations:

- Consultants/Advisor: Gerald Voecks, Angelo Lamola
- Customer/Commercialization Partner: Jadoo Power
- Collaborators: William Buttner, Robert Burgess, and Matthew Post (NREL Testing laboratory)

SUPPLEMENTAL SLIDES

Multiyear Program Plan:
Sensor Performance Targets to be achieved by 4Q 2012

- **Fuel Cells:** MYPP page 3.4-20 (Table 3.4.9)

Hydrogen in fuel processor output	▪ Measurement range: 25%–100% ▪ Operating temperature: 70°–150°C ▪ Response time: 0.1–1 sec for 90% response to step change ▪ Gas environment: 1–3 atm total pressure, 10–30 mol% water, 30%–75% total H_2, CO_2, N_2 ▪ Accuracy: <2% full scale
Hydrogen in ambient air	▪ Measurement range: full confidence of the ability to detect half of the lower explosion limit ▪ Temperature range: -30°C to 80°C ▪ Response time: under 1 sec ▪ Gas environment: ambient air, 10–98% relative humidity range ▪ Lifetime: 10 years ▪ Interference resistant

- **Safety:** MYPP page 3.8-7 (Table 3.8.2)

Table 3.8.2. Targets for Hydrogen Safety Sensor R&D
• Measurement Range: 0.1%-10% • Operating Temperature: -30 to 80°C • Response Time: under one second • Accuracy: 5% of full scale • Gas environment: ambient air, 10%-98% relative humidity range • Lifetime: 10 years • Interference resistant (e.g., hydrocarbons)

Relevance

Partners – Potential Markets

Hydrogen handling

– Hydrogen production , storage, and testing facilities (Aerovironment, Intelligent Engineering)

- **Fuel cells**
- – Stationary fuel cells, Vehicle fuel cells, production and transport (Jadoo Power, Intelligent Energy)
- **Aerospace**
- – Launching pad fueling, liquid rocket hydrogen sensor (Boeing)

Collaborations