Safe Detector System for Hydrogen Leaks

Robert. A. Lieberman / Manal H. Beshay (PI/PM)
Intelligent Optical Systems, Inc.
MAY 11, 2011
Project ID # SCS014

This presentation does not contain any proprietary, confidential, or otherwise restricted information.
Overview

Timeline

♦ Start – June 2007
♦ Finish - November 2011
♦ 83% Complete

Budget

♦ Current Project Funding: $184,966
  (DOE $119,997; IOS Share $64,970)
♦ FY09/10 Project funding: $1,189,374
  (DOE... $951,500, IOS share...$237,874)
♦ FY08/09 Project funding: $1,230,000
  (DOE... $984,000, IOS share... $246,000)
♦ Funding received in FY07: $495,000

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)

Partners

♦ Intelligent Optical Systems, Inc.– Lead
♦ Dr. Gerald Voecks – Advisor
♦ Mr. Donald Allgrove – Market consultant
♦ NREL- Testing and Validation
♦ Sandia/LANL - Testing and Validation
♦ Intelligent Energy -- Commercialization
Relevance: Overall Project Goals
✓ Investigate fully distributed, integrated optic, and optrode sensor
✓ Chose initial sensor product and develop hardware and software
→ Demonstrate sensor prototype performance in real-world applications
→ Prepare to manufacture and sell hydrogen sensor products

Technical Objectives:

<table>
<thead>
<tr>
<th>Overall</th>
<th>CY10&amp;11</th>
</tr>
</thead>
<tbody>
<tr>
<td>Integrate IOS’ proprietary hydrogen indicator chemistry into a complete</td>
<td>Select and finalize hydrogen sensor components and outline scalable cost</td>
</tr>
<tr>
<td>optoelectronics package with well-defined sensing characteristics and a</td>
<td>analysis.</td>
</tr>
<tr>
<td>known end-use market</td>
<td>Finalize sensor data processing algorithms with minimum false alarms.</td>
</tr>
<tr>
<td></td>
<td>Fabricate, test, and validate performance of 14 fully packaged prototypes</td>
</tr>
<tr>
<td></td>
<td>Deploy prototypes at four different field test sites.</td>
</tr>
<tr>
<td></td>
<td>Collect and analyze real-time test data under various deployment conditions.</td>
</tr>
<tr>
<td></td>
<td>Reach end-users through field demonstrations and field trails.</td>
</tr>
</tbody>
</table>
Relevance: IOS Hydrogen Sensors Directly Address Multiple Barriers

- **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”)

Relevance: Product Specifications are Driven by Hydrogen Safety Needs

♦ Sensor Product Specifications:
- Range: 0 – 100% H\textsubscript{2}
- Sensitivity: (min) 0.1%H\textsubscript{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
**Approach:**

**Optical Waveguide Hydrogen Sensing**

**Colorimetric Detection**
- Immobilize hydrogen-sensitive indicator in optically transparent medium
- Indicator/matrix reversibly changes color in presence of H₂
- Intensity of light transmitted through matrix depends upon hydrogen concentration
- Intrinsically safe, electrically inert, wide temperature range, works in absence of O₂

**Optical Sensor Formats**

*Optrode*: Indicator immobilized in porous glass. Sensors can be packaged with electronics or remotely addressed through fibers.

*Integrated Optic Waveguide*: Indicator imbedded in waveguides fabricated on optical chip. Multiple channels improve performance.

* Distributed Sensing Fiber*: Indicator coated on entire length of sensing fiber. Wide area continuous coverage with a single cable.
Approach: Product Design is Driven by Hydrogen Sensor Market

- Vehicle Safety
- Stationary Power
- Storage & Transport
- Refueling
Approach: Rigorous Testing to Validate Packaged Hydrogen Sensor Performance

- IOS Sensor Test Facility:
  - Computer-controlled gas delivery
  - Compatible with NREL facility
  - NIST-traceable gas concentrations
Approach: Project Plan: FY08 – FY11

- 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
- Perform field testing at multiple beta sites to validate the sensor performance

- Define sensor chemistry
- Evaluate various optical sensor matrices
- Validate the sensor response/cross-interference
- Develop and build the optoelectronic interface
- Select and finalize sensor format
- Finalize, fabricate, and test optoelectronic system
- Develop and optimize signal processing algorithms
- Test complete packaged sensor prototype
- Field demonstration and validation testing
- Refine algorithms and release finalized units
- Establish commercial market and partnerships
Accomplishment: Hydrogen Safety Sensor Prototype Refined/Ruggedized

Hardware Package Evolution

FY10

FY11
Accomplishment: Sensor Repeatability/Reversibility Validated

- Optrode response to 0.2; 1.0; 2.0%, of hydrogen at IOS
- Optrode response to 0.22, 1.08 and 2.15% of hydrogen at NREL

- Optical signals self-consistent within 2%
- Optical signals at NREL within 2% of IOS signals
Accomplishment: Rapid Response
Hydrogen Alarm Algorithm Developed

Hydrogen release detection algorithm repeatedly responds to presence of 2% hydrogen in air in less than 5 seconds.
Accomplishment: Hydrogen Sensor Alarm Algorithm Detail
Accomplishment: Optrodes Have High Sensitivity and Rapid Alarm Capability

Response Time ($T_{90}$) at Various Hydrogen Levels

<table>
<thead>
<tr>
<th>H2 (%)</th>
<th>Detection Time (s)*</th>
</tr>
</thead>
<tbody>
<tr>
<td>4.0</td>
<td>3</td>
</tr>
<tr>
<td>2.0</td>
<td>3</td>
</tr>
<tr>
<td>1.0</td>
<td>3</td>
</tr>
<tr>
<td>0.5</td>
<td>3</td>
</tr>
<tr>
<td>0.1</td>
<td>10</td>
</tr>
<tr>
<td>0.05</td>
<td>10</td>
</tr>
<tr>
<td>0.02</td>
<td>30</td>
</tr>
<tr>
<td>0.01</td>
<td>120</td>
</tr>
</tbody>
</table>

*RH = 45%; P = 1 atm.; T = 23 ºC; Flow Rate = 1.0 L/min.

Response time < 5 sec. at 10% LFL
Accomplishment: Rapid Response Demonstrated in Field Tests

("Simulated leak:" Hydrogen release into building at Sandia/Livermore open-air test site.)

- Release detected within 2 seconds
- De-alarming algorithms need further calibration
Accomplishment: Field Tests Show Potential for High-Accuracy Concentration Measurement

(“Simulated leak:” Hydrogen release into building at Sandia/Livermore open-air test site.)

Optical signal closely tracks increasing hydrogen concentration in building
**Future Work: Remaining Technical Tasks/Milestones**

<table>
<thead>
<tr>
<th>Task 11:</th>
<th>Identify potential cross-contaminants; test and optimize hydrogen sensor elements</th>
</tr>
</thead>
<tbody>
<tr>
<td>11.1</td>
<td>Fabricate and test optrodes for inclusion in demonstration units.</td>
</tr>
<tr>
<td>Milestone 1</td>
<td>Complete cross-contamination testing of sensor elements and determine whether additional selective permeable membranes and/or scrubbing layers are needed.</td>
</tr>
<tr>
<td>100%</td>
<td>Task 12: Build optoelectronic interface to optrode sensor array modular package</td>
</tr>
<tr>
<td>78%</td>
<td>Task 13: Design and integrate software data processing including internal calibration curves</td>
</tr>
<tr>
<td>Task 13.1</td>
<td>Define and integrate internal calibration parameters.</td>
</tr>
<tr>
<td>Task 13.2</td>
<td>Optimize detection and quantification algorithms.</td>
</tr>
<tr>
<td>Task 13.3</td>
<td>Run simulation testing on optimized signal processing protocol.</td>
</tr>
<tr>
<td>32%</td>
<td>Task 14: Perform complete test of packaged integrated hydrogen sensor, including sensitivity, response time, selectivity, false alarms, and temperature/humidity characteristics.</td>
</tr>
<tr>
<td>Task 14.1</td>
<td>Conduct long term testing at various environmental conditions.</td>
</tr>
<tr>
<td>Task 14.2</td>
<td>Analyze and tune algorithms.</td>
</tr>
<tr>
<td>Task 14.3</td>
<td>Demonstrate the final packaged test unit at NREL.</td>
</tr>
<tr>
<td>Milestone 2</td>
<td>Finish complete testing of the sensor package and validate system performance. Provide report on shelf life of aged and packaged sensor components</td>
</tr>
<tr>
<td>27%</td>
<td>Task 15: Perform hydrogen sensor market study.</td>
</tr>
<tr>
<td>Task 15.1</td>
<td>Acquire components and material for fabricating units.</td>
</tr>
<tr>
<td>Task 15.2</td>
<td>Fabricate and test sensor units.</td>
</tr>
<tr>
<td>Task 15.3</td>
<td>Calibrate demonstration units.</td>
</tr>
<tr>
<td>Milestone 3</td>
<td>Complete fabrication, assembly, and testing of 14 units for field demonstration</td>
</tr>
<tr>
<td>Task 15.4</td>
<td>Deliver test units to each field test site and support tests.</td>
</tr>
<tr>
<td>Task 15.5</td>
<td>Provide technical support.</td>
</tr>
<tr>
<td>Task 15.6</td>
<td>Analyze data from test sites.</td>
</tr>
<tr>
<td>Milestone 4</td>
<td>Analyze field testing data and finalize sensor algorithms.</td>
</tr>
<tr>
<td>Task 15.7</td>
<td>Prepare manufacturing/design recommendations.</td>
</tr>
<tr>
<td>85%</td>
<td>Task 16: Management and reporting</td>
</tr>
</tbody>
</table>
Future Work: Hydrogen Sensor Concentration Algorithm Being Finalized

Reproducible sensor response requires hysteresis compensation
Future Work: Humidity/ Temperature Compensation Being Finalized

- Excellent response at all RH levels
- Reproducible effect

- Good response at all temperatures
- Reproducible effect
Future Work: Field Tests Begin May 2011

- 14 Complete hardware prototypes fabricated
- Deployment in multiple test sites
- Data collection
  - May-June 2011: Data for final algorithm development
  - July-September 2011:
    - Verification of low false alarms in non-release sites
    - Verification of rapid alarm in controlled-release tests
    - Final characterization of concentration measurement
Collaborations: Acknowledgements

♦ Gerald Voecks – Advisor
  - Fuel cell applications and commercialization

♦ William Buttner, Robert Burgess, Matthew Post – NREL Testing Collaborators
  - Hydrogen sensor testing and validation

♦ Daniel E. Dedrick, Erik Merilo- Sandia National Lab.
  - Hydrogen sensor field simulation testing

♦ Intelligent Energy
  - Customer/commercialization partner

♦ DOE Codes and Standards: We thank Antonio Ruiz and Katie Randolph for their continued support and valuable insights.
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 will 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
- Developed framework for detection and measurement algorithms
- Tested and validated sensor capabilities at third-party test site (NREL)
- Executed market size and cost analysis for sensor commercialization

Collaborations:
- Consultants/Advisors: Gerald Voecks, Donald C. Allgrove
- Commercialization Partner: Intelligent Energy
- Collaborators: William Buttner, Robert Burgess, and Matthew Post (NREL) Daniel E. Dedrick, Erik Merilo (Sandia National Lab, Field simulation testing)
Back-Up 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 H₂, CO₂, N₂
| | · 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>
<thead>
<tr>
<th>Table 3.8.2. Targets for Hydrogen Safety Sensor R&amp;D</th>
</tr>
</thead>
</table>
| · 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)
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 (Intelligent Energy, Quantum, Ballard)

♦ Aerospace

– Launching pad fueling, liquid rocket hydrogen sensor (Boeing, NASA)