Hydrogen Optical Fiber Sensors

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Project ID# SAP2

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Overview

Timeline

- Start - May 01, 2006
- Finish - April 30, 2007
- 90% complete

Budget

- Total project funding
  - DOE - $495K
  - Contractor- $124K
- Total funding in FY06
  - $236K
- Funding for FY07
  - $383K

Safety Sensor Development

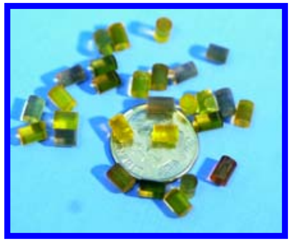
- Hydrogen Program Barriers Addressed
  1. Expense of data collection and maintenance
  2. Liability issues
  3. Safety is not always treated as a continuing process
- Sensor Performance Targets
  1. Measurement range: 0.1%-10%
  2. Gas environment: ambient air, 10%-98% RH range
  3. Interference resistant (e.g. moisture, hydrocarbons)

Technical Objectives

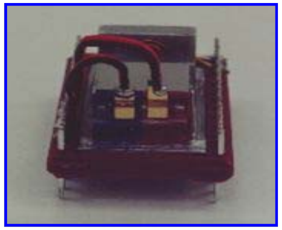
Overall	• Reduce or eliminate interferences from humidity and oxygen exhibited by virtually all current optically-based hydrogen detectors • Establish and fully characterize a compact hydrogen detector
FY2006	• Transfer existing indicator chemistry from commercial to in-house porous glass substrate and improve indicator performance • Transfer indicator chemistry from porous glass substrate to polymeric substrate • Establish ppm-level response to hydrogen in one or more candidate substrates
FY2007	• Establish good hydrogen sensitivity, response time, and sensor performance with little or no response to moisture and oxygen • Develop compact multi-channel detector/test system

Technical Approach:
Optical Detection of Hydrogen

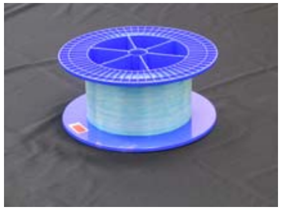
- **Colorimetric Detection**
  - Immobilize hydrogen-sensitive indicator in optically transparent medium
  - Indicator mixture changes color in presence of hydrogen
  - Transmitted light intensity depends on hydrogen concentration
- **Optical Formats**



» "Optrode:" Indicator immobilized in point sensors mounted on fiber tip. Sensors can be located far from electronics.



» Integrated Optic: Indicator embedded in waveguides on optical chip. Multiple channels improve performance.



» Distributed: Indicator coated on entire fiber. Wide area can be covered with a single cable.

Technical Plan

100% complete	***Task 1.0*** **Acquire reagents and substrate materials** • Maintain work flow
100% complete	***Task 2.0*** **Formulate porous glass sensors from silicate and/or silicone reagents** • Devise new indicator-immobilization techniques • Synthesize and characterize thin porous glass films
90% complete	***Task 3.0*** **Evaluate hydrogen diffusion in polymer materials** • Evaluate polymers and copolymers for oxygen and humidity barriers • Rank-order polymer materials tested
90% complete	***Task 4.0*** **Evaluate various techniques to produce thin-film and/or slab sensors from advanced polymers** • Survey waveguide fabrication methods • Develop waveguide-based sensor with enhanced optical performance
95% complete	***Task 5.0*** **Evaluate sensor response to hydrogen under inert conditions** • Measure baseline hydrogen response and sensitivity of candidate sensors • Measure response to hydrogen in the absence of water and oxygen • Select preferred operating wavelengths
90% complete	***Task 6.0*** **Evaluate sensor performance and resistance to moisture and oxygen** • Select best material for use in a waveguide-based hydrogen sensor • Test under operational conditions • Establish preliminary design of integrated optic waveguide-based hydrogen sensor
80% complete	***Task 7.0*** **Incorporate new sensors in compact hydrogen detector and test system** • Combine proprietary optoelectronic and software subsystems • Demonstrate a portable hydrogen detector unit
90% complete	***Task 8.0*** **Project management and reporting** • Document progress and provide deliverables

Technical Accomplishments

1. Porous glass sensor optimized
   -- Polymer coating provides resistance to humidity
   -- Commercial glass selected as most stable

2. Hydrogen chemistry modified and embedded in optical grade polymer
   -- Provides even greater resistance to humidity
   -- Properties suitable for fabrication of integrated optic sensor

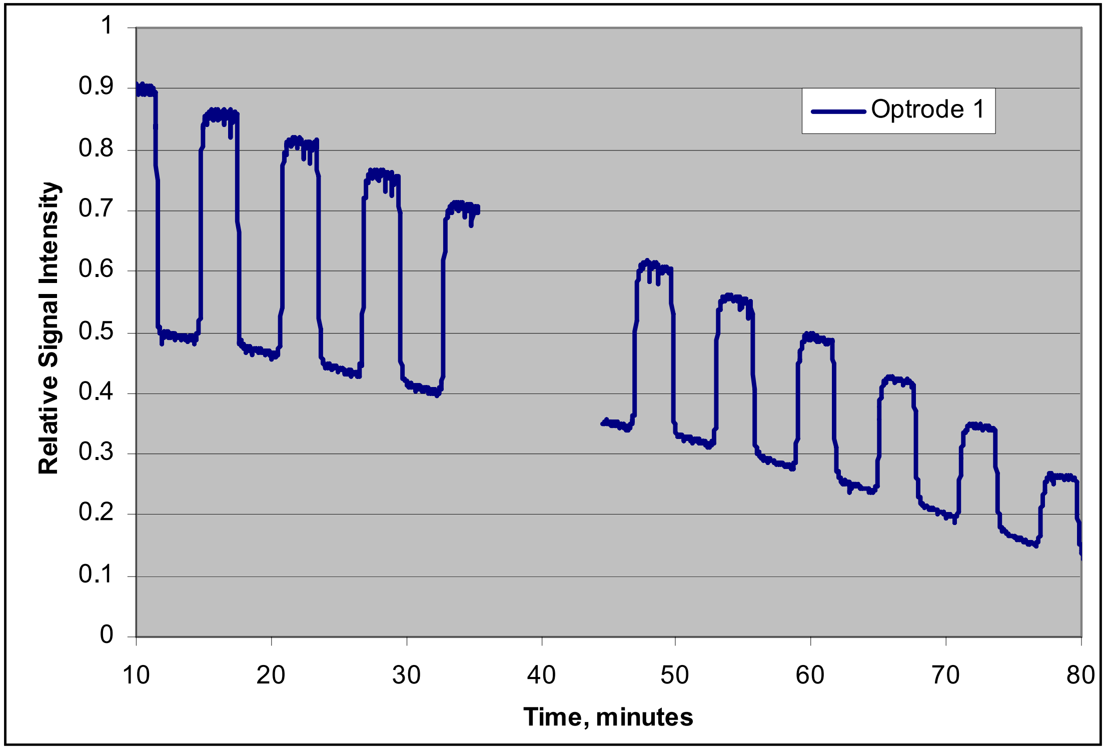
3. Multiplexed fiber optic test unit developed
   -- Incorporates low cost energy efficient LED light sources
   -- Basis for compact hydrogen sensor detector system

These three accomplishments all contribute to the Hydrogen, Fuel Cells, and Infrastructure Technologies Program's need for reliable, intrinsically safe, accurate, and cost-effective hydrogen detectors.

All FY06 Objectives have been met.

Potential Long-Term Humidity Effects

(above 90% RH uncoated glass substrate fails)



- Repeated exposure to 5% hydrogen in air at 90% RH results in loss of sensor response
- Primary cause: Humidity-fouling of porous glass substrate

Porous Glass Sensor Response

-- *Inert* Environment

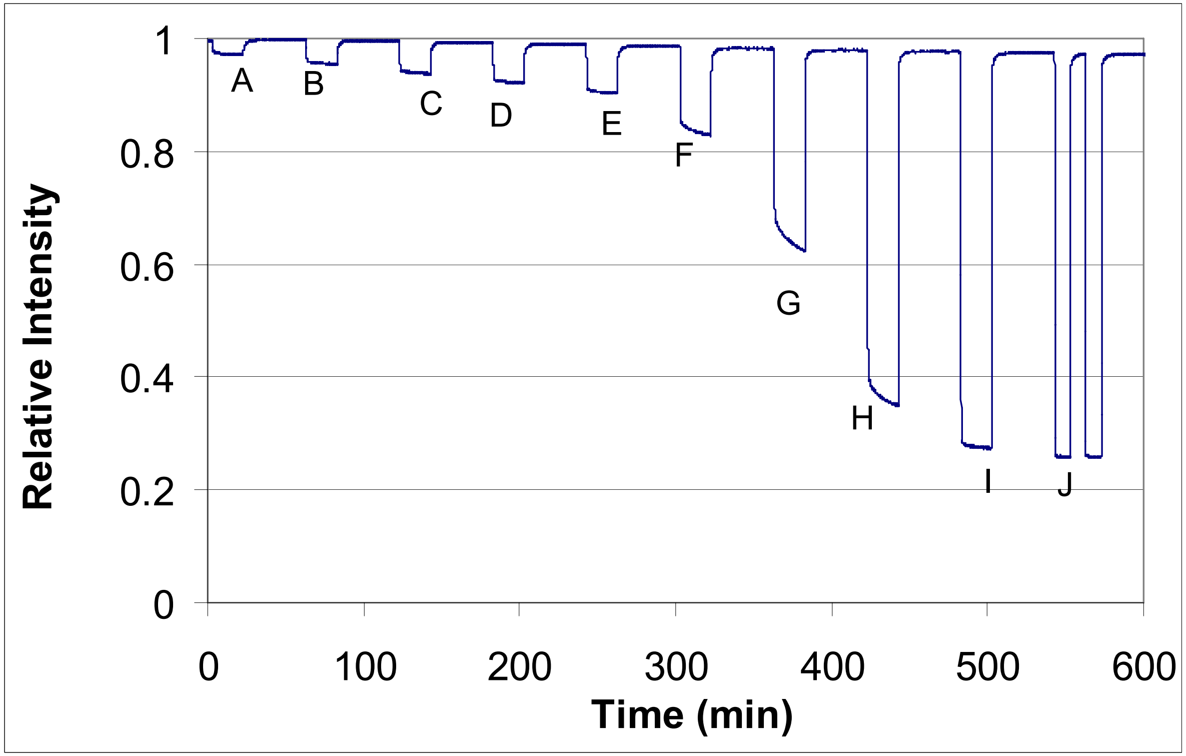
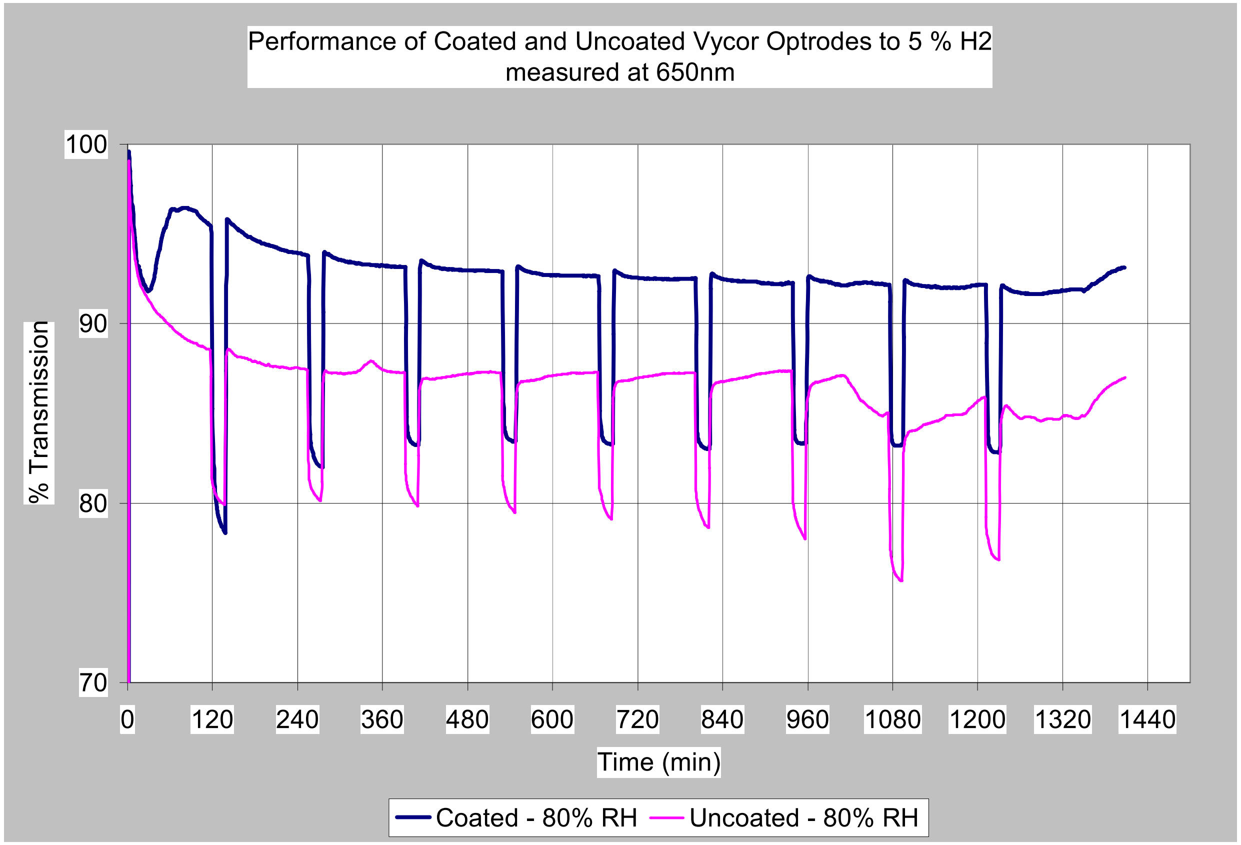


Figure Key	% Hydrogen
A	0.1
B	0.2
C	0.4
D	0.6
E	0.8
F	1.0
G	1.5
H	2.0
I	3.0
J	5.0

- 0.1 % hydrogen detected with excellent signal-to-noise ratio
- Projected sensitivity <100 ppm

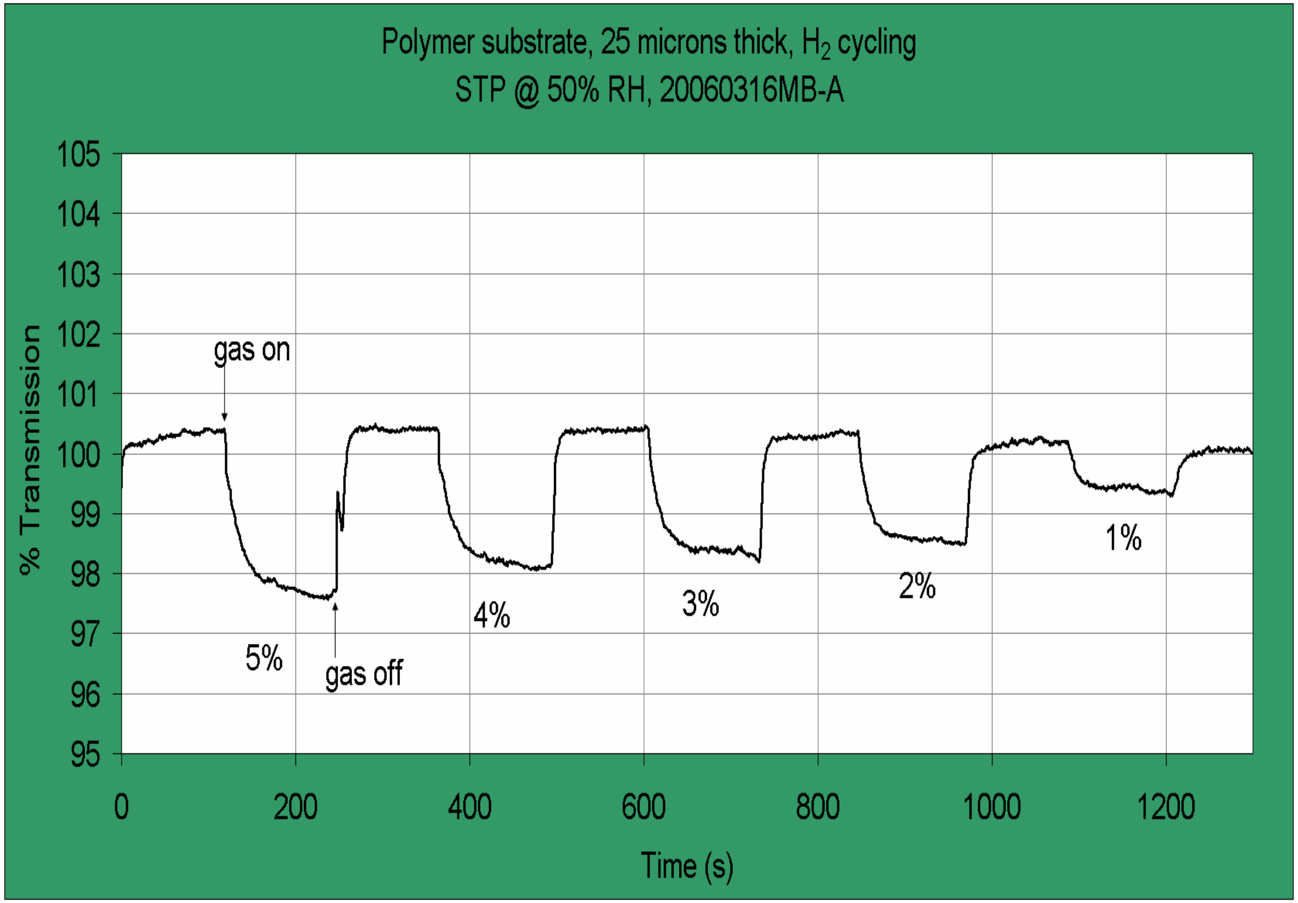
Optimized Porous Glass Substrates



Sensor with barrier coating developed in project (blue line above) has:

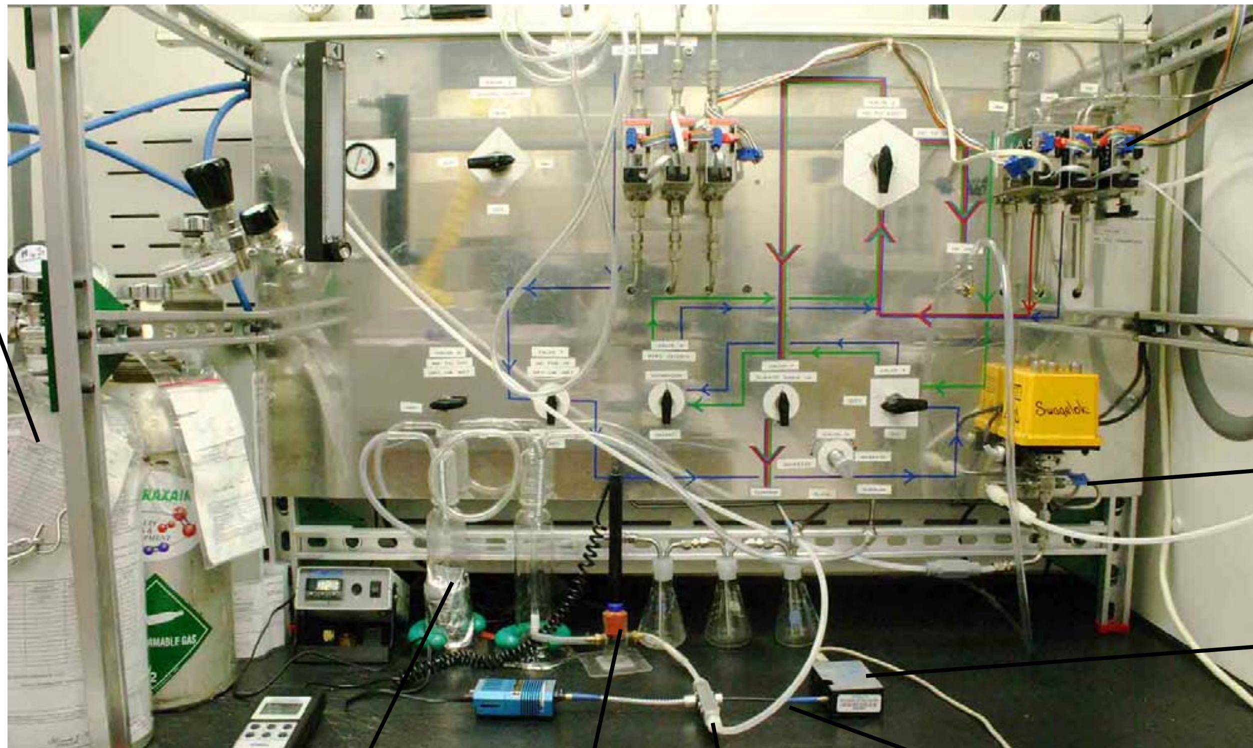
- More stable response (consistent peak-to-peak values)
- Faster equilibration in 80% RH environment

Water Resistant Polymeric Substrate



- Optical grade polymer supports hydrogen indicator chemistry
- No observable interference from O_2 or humidity
- Chemistry shows good sensitivity over range of H_2 concentrations

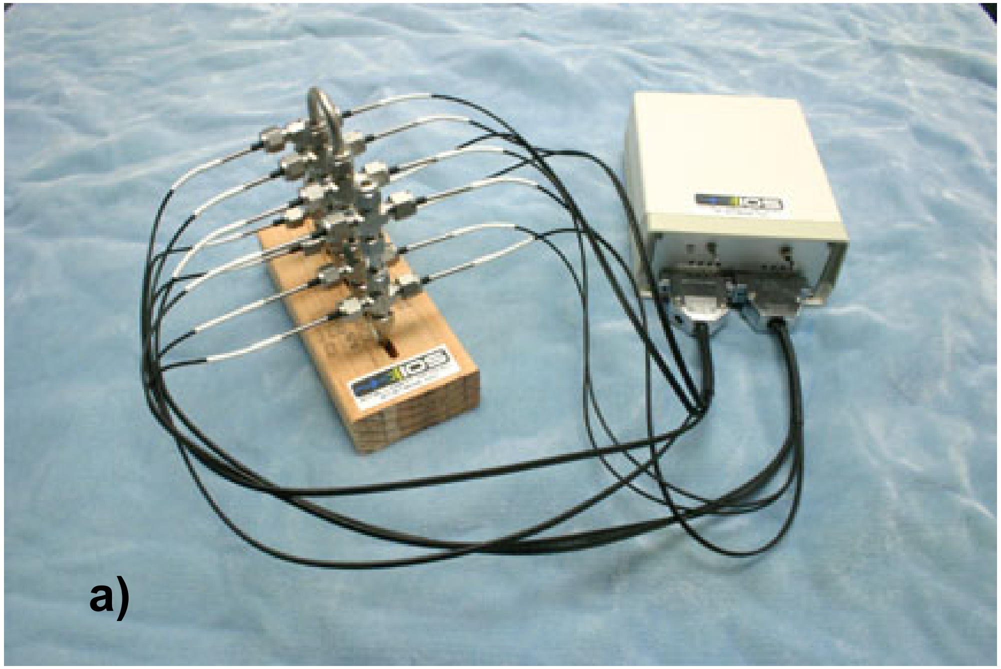
IOS Hydrogen Sensor Test Facility



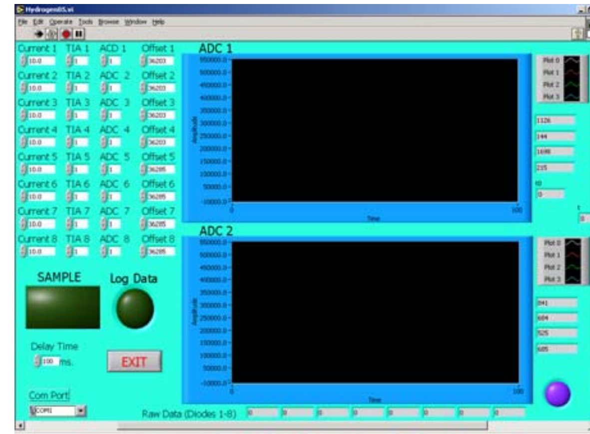
Fully automated test equipment:

- Computerized mass flow controllers for gas mixing
- Online humidity measurement
- Detailed test protocols established

Multichannel Optoelectronic System



a)



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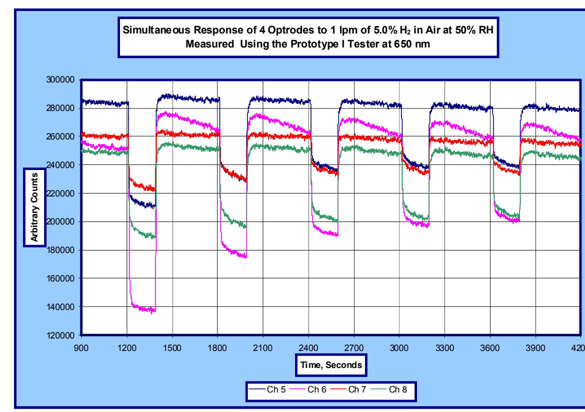


c)

Fiber optic multi-sensor analyzer

a) Proprietary optoelectronics
   - High sensitivity and stability
   - Suitable for fiber optic or integrated optic readout

b) PC-enabled graphical user interface

c) Simultaneous data acquisition for 8 sensor channels

Future Work

FY 2007

-- Develop and characterize polymer waveguides
-- Finish optoelectronic system development
-- Test response to hydrogen, oxygen, and humidity

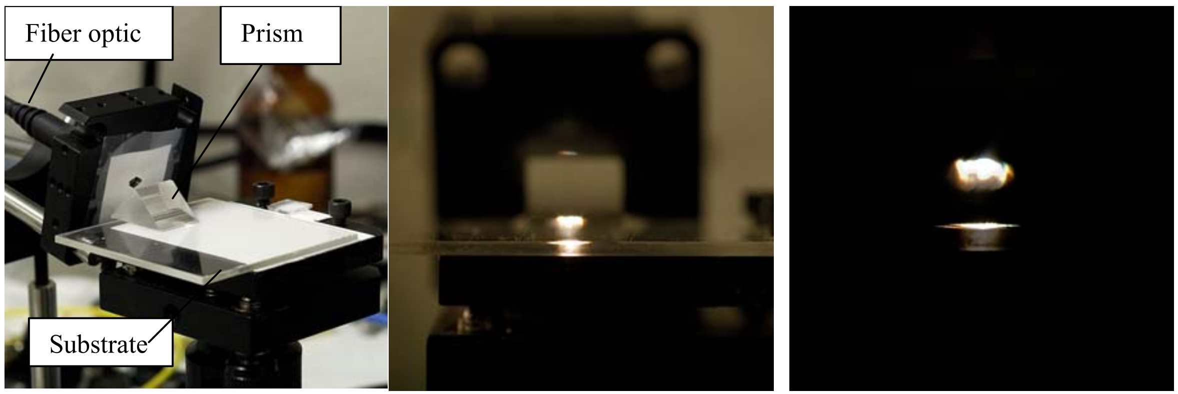
Final milestone: Create and characterize an optical hydrogen sensor with improved humidity and oxygen resistance

FY 2008 (Proposed)

-- Test sensor longevity and response to potential “interferants”
-- Fabricate multichannel waveguide chips
-- Develop advanced signal acquisition and processing
-- Miniaturize optoelectronic system

IOS Optical Waveguide Technology

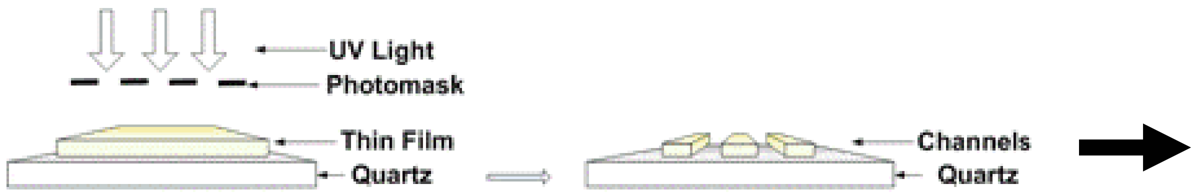
Step 1: Fiber coupled waveguide <u>film</u>

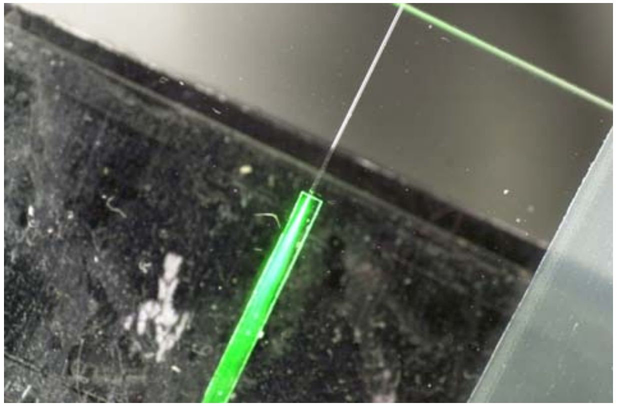


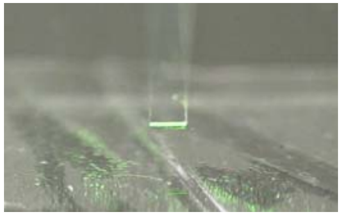
- Light travels horizontally through sensor film
  - –Path length increases by 2-3 orders of magnitude
  - –Sensitivity increases
- Hydrogen enters through top of film
  - –Diffusion length unaffected
  - –Sensor response time stays the same

IOS Optical Waveguide Technology

Step 2: Fiber coupled waveguide channel







- Light confined in two dimensions
  - –Horizontal launch preserves path length increase
  - –Side confinement improves light throughput
  - –Multiple channels can share same chip
- Hydrogen enters through top and sides of film
  - –Average diffusion length shortens
  - –Sensor response time improves

Summary

Relevance:
- Reliable, cost-effective hydrogen safety sensors are required for generation, storage, transport, and (eventually) home safety applications

Approach:
- Optical sensors based on indicator chemistry can be designed for high performance and low cost

Technical Accomplishments:
- Developed barrier coating for moisture resistance
- Improved indicator chemistry performance
- Embedded Indicator chemistry in optical grade polymer
- Hydrophobic material suitable for waveguide fabrication

Proposed Future Work:
- Fabricate hydrogen sensitive waveguides
- Analyze longevity, specificity, and moisture response
- Package system and include final corrections for temperature and humidity

Final Goal:
- A miniaturized sensor using multiple channels on a single optical chip will achieve an extremely high probability of detecting dangerous hydrogen levels and an extremely low false alarm rate

Target Summary

Hydrogen Safety Sensor Targets			
Metrics	**2008 System Target**	**FY 06 Results**	**FY 07 Results**
Dynamic range	0-100%	0-5%	0-10%
Response time	1 sec	3-50 s Depending on substrate	1-10 s
Accuracy (noise)	+/- 10% signal	+/- 10% signal	+/- 10% signal
1% hydrogen detection in ambient atm	Yes	Yes	Yes
Possible interferences	none	T, Humidity, CO	CO

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