VIII.3 Hydrogen Safety, Codes and Standards: Sensors

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Project Start Date: Fiscal Year (FY) 2008
Project End Date: FY 2014

Overall Objectives

- Develop a low-cost and low-power electrochemical hydrogen safety sensor for a wide range of vehicle and infrastructure applications with focus on high durability and reliability
- Continually advance test prototypes guided by materials selection, sensor design, electrochemical research and development (R&D) investigation, fabrication, and rigorous life testing
- Disseminate packaged sensor prototypes and control systems to DOE laboratories and commercial parties interested in testing and fielding advanced prototypes for cross-validation
- Evaluate manufacturing approaches for commercialization
- Engage an industrial partner and execute technology transfer

FY 2013 Objectives

- Perform third round of validation and verification at the National Renewable Energy Laboratory (NREL)
- Acquire new lot of sensor platforms from ESL ElectroScience and fabricate sensors for NREL testing
- Test heater control boards at LANL and validate performance and sensor temperature response at NREL
- Initiate industrial partner search for sensor field trials

Technical Barriers

This project addresses the following technical barriers from the Hydrogen Safety section (3.8) of the Fuel Cell Technologies Office Multi-Year Research, Development, and Demonstration Plan:

(A) Safety Data and Information: Limited Access and Availability

(C) Safety is Not Always Treated as a Continuous Process

(K) No Consistent Codification Plan and Process for Synchronization of R&D and Code Development

(L) Usage and Access Restrictions

Technical Targets

Technical targets vary depending on the application [1,2], but in general include:

- Sensitivity: 1-4 vol% range in air
- Accuracy: ± 1% full scale in the range of 0.04-4 vol%
- Response Time: <1 min at 1% and <1 sec at 4%; recovery <1 min
- Temperature Operating Range: -40ºC to 60ºC
- Durability: Minimal calibration or no calibration required over sensor lifetime (as defined by particular application)
- Cross-Sensitivity: Minimal interference to humidity, H_2S, CH_4, CO, and volatile organic carbons

FY 2013 Accomplishments

- Tested sensor power supplies together with cheaper, more simplified sensor platforms incorporating integrated, unitary heater/temperature sensor platforms.
- Developed, optimized, and characterized working electrode and electrolyte layers grown using electron beam evaporation methods. A significant reduction in sensor manufacturing time was demonstrated that would eventually mean a reduction in projected manufacturing costs.
- New sensors were prepared using e-beam methods and packaged for Round 3 testing at NREL.
- Identified and tested La-Sr-Cr-O electrode as a candidate for applications that may involve anaerobic operations: solves a weakness identified in Round 2 NREL testing last year.
- Conducted last round of verification and validation at NREL: data show that the sensor signal changes

due to changes in ambient temperature are effectively eliminated.

- Interference testing performed on sensors indicated negligible interference in the presence of H_2 for the standard interferences tested.
- Demonstrated that the electrochemical mixed potential sensor technology exhibits a high degree of sensor device-to-device response reproducibility.

INTRODUCTION AND APPROACH

Recent developments in the search for sustainable and renewable energy coupled with the advancements in fuel cell vehicles have augmented the demand for hydrogen safety sensors [2]. There are several sensor technologies that have been developed to detect hydrogen, including deployed systems to detect leaks in manned space systems and hydrogen safety sensors for laboratory and industrial usage. Among the several sensing methods, electrochemical devices [3-9] that utilize high temperature-based ceramic electrolytes are largely unaffected by changes in humidity and are more resilient to electrode or electrolyte poisoning. The desired sensing technique should meet a detection threshold of 1% (10,000 ppm) H_2 and response time of ≤1 min [10], which is a target for infrastructure and vehicular uses. Further, a review of electrochemical hydrogen sensors by Korotcenkov et al. [11] and the report by Glass et al. [10, 12] suggest the need for inexpensive, low-power, and compact sensors with long-term stability, minimal cross-sensitivity, and fast response. This view has been largely validated and supported by the fuel cell and hydrogen infrastructure industries at the NREL/DOE Hydrogen Sensor Workshop held on June 8, 2011 [13]. Many of the issues preventing widespread adoption of best-available hydrogen sensing technologies available today besides cost derive from excessive false positives and false negatives arising from signal drift and unstable sensor baseline; both of these problems necessitate unacceptably frequent calibration [13].

As part of the Hydrogen Codes and Standards sub-program, LANL and LLNL are working together to develop and test inexpensive, zirconia-based, electrochemical (mixed potential) sensors for H_2 detection in air. Previous work conducted at LLNL [9] showed that indium tin oxide (ITO) electrodes produced a stable mixed potential response in the presence of up to 5% H_2 in air with very low response to CO_2 and water vapor. The sensor also showed desirable characteristics with respect to response time, resistance to aging, and degradation due to thermal cycling.

In this investigation, the development and testing of an electrochemical H_2 sensor prototype based on '(ITO)/yttria-stabilized zirconia (YSZ)/platinum (Pt) configuration is detailed.

The device was fabricated using commercial ceramic sensor manufacturing methods on an alumina substrate with an integrated Pt resistance heater to achieve precise control of operating temperature while minimizing heterogeneous catalysis and loss of hydrogen sensitivity. Targeting fuel cell-powered automotive applications, the safety sensor was subjected to interference studies, temperature cycling, operating temperature variations, and long-term testing now exceeding over 6,000 hrs for some sensor configurations. In FY 2011, FY 2012, and FY 2013, the mixed potential electrochemical technology was independently validated at the hydrogen safety sensor-testing lab at NREL in three separate rounds of testing. In each round, two packaged pre-commercial prototypes were tested against a standard testing protocol, including the effects of changes in ambient temperature, pressure, humidity, and oxygen partial pressure (non-anaerobic) and sensor resistance to cross-interferences such as CO, CO_2, CH_4, and NH_3. In general, NREL testing showed a fast response to H_2 with exceptional low-level sensitivity and high signal-to-noise, very little deviation in sensor response to changes in ambient conditions such as humidity and barometric pressure, and minimal response to some common interference gases. However, potential weaknesses were found in the first two rounds of testing such as changes in sensor calibration with ambient temperature changes and complete sensor failure under the most harsh operating environment tested (anaerobic conditions).

FY 2011 testing at NREL uncovered an unanticipated interaction of the sensor element with the data acquisition system used in the hydrogen sensor testing system. The first sensor testing and validation experiments showed data with an anomalously high baseline (when no H_2 was present) and poor sensitivity to H_2 (when H_2 was present). These behaviors were never seen in LANL or LLNL laboratory sensor development work and could only be explained if there was insufficient input impedance on the data acquisition system. As a result, a high-impedance buffer (HIB) circuit board was designed and built to isolate the naked electrochemical sensor from stray electric currents that would generate a high baseline voltage which, depending on the direction of the current flow, would induce an offset voltage that would reduce the sensor voltage generated in response to H_2 exposure. The HIB is designed around a Burr Brown INA116 electrometer amplifier integrated circuit and is designed to minimize leakage between the electrodes and from the sensor itself to the electrometer circuit. The major success of FY 2012 were the successful verification and validation of the sensor/HIB package at NREL.

For FY 2013, a newly designed and constructed heater board was tested that changed sensor heater power according to ambient temperature. Scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS) analysis of the sensor that was exposed to anaerobic conditions were performed, and the post-testing evaluation was conducted to understand and interpret NREL findings.

The salient features of the H_2 sensor prototype developed by LANL and LLNL are (a) low power consumption, (b) compactness to fit into critical areas for some applications, (c) simple operation, (d) fast response, (e) a direct voltage read-out circumventing the need for complicated signal processing, (f) a low-cost sensor platform, and (g) excellent stability and reproducibility, all of which are conducive to commercialization using common ceramic manufacturing methods.

RESULTS REPORTED IN FY 2013

(a) Temperature feedback and heater control circuit development: In FY 2013, the principal goal for the third round of NREL testing was to test performance of the LANL/LLNL H_2 safety sensor prototype with active temperature feedback and control. NREL test results in FY 2011, Round 1 testing returned the anticipated results for ambient temperature testing. Because the sensor was tested using a fixed applied heater voltage throughout all of the Round 1 NREL testing, changes in the temperature of the NREL test chamber caused the sensor's temperature also to rise and fall commensurately. The variation in sensor voltage with temperature is well known since the response of mixed potential sensors is governed by electrode kinetics and the electrochemical reactions are a strong function of temperature. The small changes in the sensor Pt heater resistance were used to provide feedback to a heater control circuit designed and constructed for this project by Custom Sensor Solutions (Tucson, AZ). Figure 1 shows the experimental setup used for Round 3 testing at LANL. This circuit used a voltage output from a simple analog bridge to add/subtract to the heater voltage using the resistance from the sensor's Pt resistive heater as the control point. It is a very simple circuit and mode of operation that effectively maintained sensor temperature despite large changes in ambient T (over 90°C range in NREL test protocol) or local changes in sensor element temperature due to heat generated by H_2 combustion. While a 30°C change in test chamber temperature led to a 136% change in sensor voltage at 1% H_2 levels, this large temperature-induced shift in sensor voltage was not seen this year, indicating that the circuit worked very well.

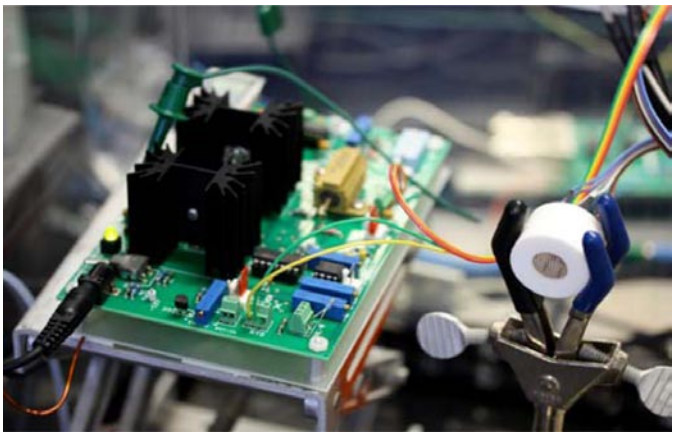

FIGURE 1. A packaged mixed potential, electrochemical sensor (shown in clamp on right) together with heater control electronics (left) used in FY 2013, Round 3 NREL testing to maintain precise sensor operating temperature commensurate with changes in ambient sensor temperature and temperature changes resulting from liberation of heat of combustion of the H_2 test gas.

(b) NREL testing: Figure 2 shows the results of NREL testing during the linear hydrogen range test performed as part of normal protocol. Given the exceptionally high signal-to-noise and high low-level sensitivity, NREL added several lower H_2 concentration levels to the test protocol. The sensors exhibited a normal baseline and response when measured at LANL or LLNL sensor laboratories in all cases. The HIB electronics developed in FY 2012 by Custom Sensor Solutions to counter undesirable sensor-data acquisition system interactions worked well, and a side benefit is the ability to control sensor baseline off-set and to adjust a desired amount of voltage amplification or gain. This latter feature is illustrated in Figure 2. The two sensors show different voltage levels for the same set of test gas concentrations because the gain was deliberately set at two different levels. The working voltage out is more than adequate for future final development and field trials, even for inexpensive recording of signal using either analog or digital circuits.

(c) Faster and less expensive methods of sensor fabrication demonstrated: In FY 2013, the methodology used to fabricate the safety sensor electrodes and electrolyte layers was changed from a combination of radio frequency magnetron sputtering (for the ITO working electrode) and electron beam evaporation (e-beam, for the YSZ electrolyte) to all e-beam. E-beam methods are used in commercial fabrication of sensors and devices. It is less expensive and a much faster method for film growth, although maintaining the stoichiometry of complex oxide materials may be more difficult. Figure 3a shows the EDS results of a test film grown in less than 10 minutes from a commercially sourced ITO target. The EDS confirmed the presence of the ITO film in a composition suitable for this application and similar in composition to those films prepared by sputtering. Also this FY, a new faceplate holder and machined shadow masks were procured to accommodate the ESL-prepared platforms and to apply the working electrode and electrolyte layers with more precision, leading to less change of substrate slippage resulting in unusable devices. Figure 3b is an SEM image using backscatter detector showing the ESL-prepared platform and wire bonding pads and counter electrode extension, together with the LANL-prepared ITO and YSZ layers.

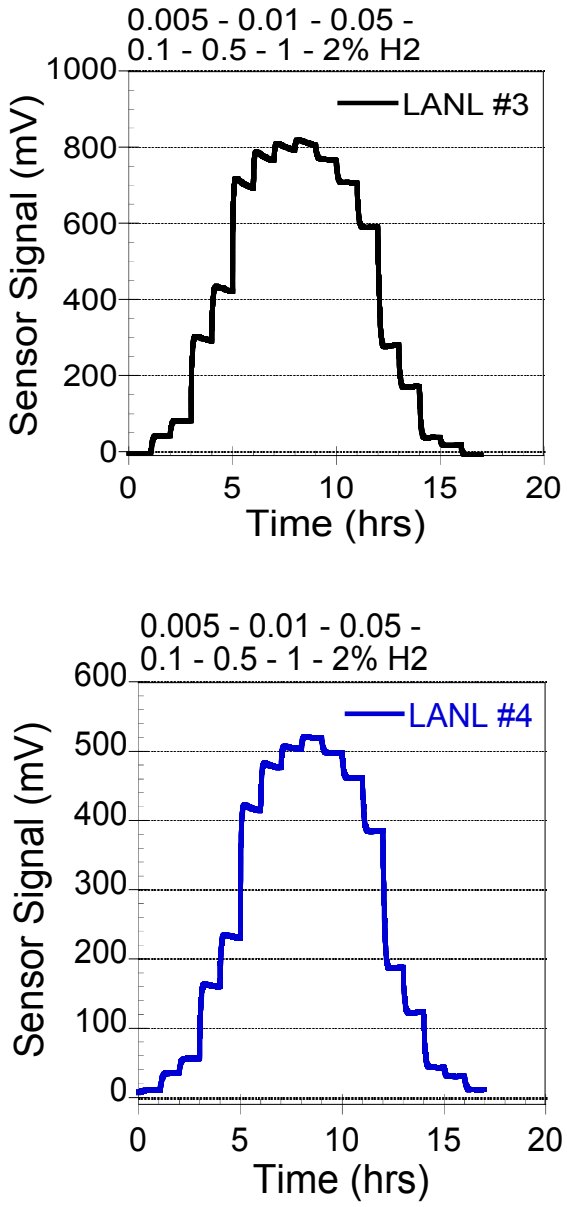

FIGURE 2. NREL "linear hydrogen test" performed on two packaged LANL/LLNL electrochemical hydrogen sensors. The use of HIB circuit boards isolated the two sensors from leakage currents in the data acquisition system at NREL discovered in Round 1 of testing. The HIB circuits isolate the sensors from stray leakage currents and permit normal sensor function. The different signal voltage levels for equivalent H_2 concentrations are a demonstration of the variable gain (signal amplification) built into the HIB circuitry.

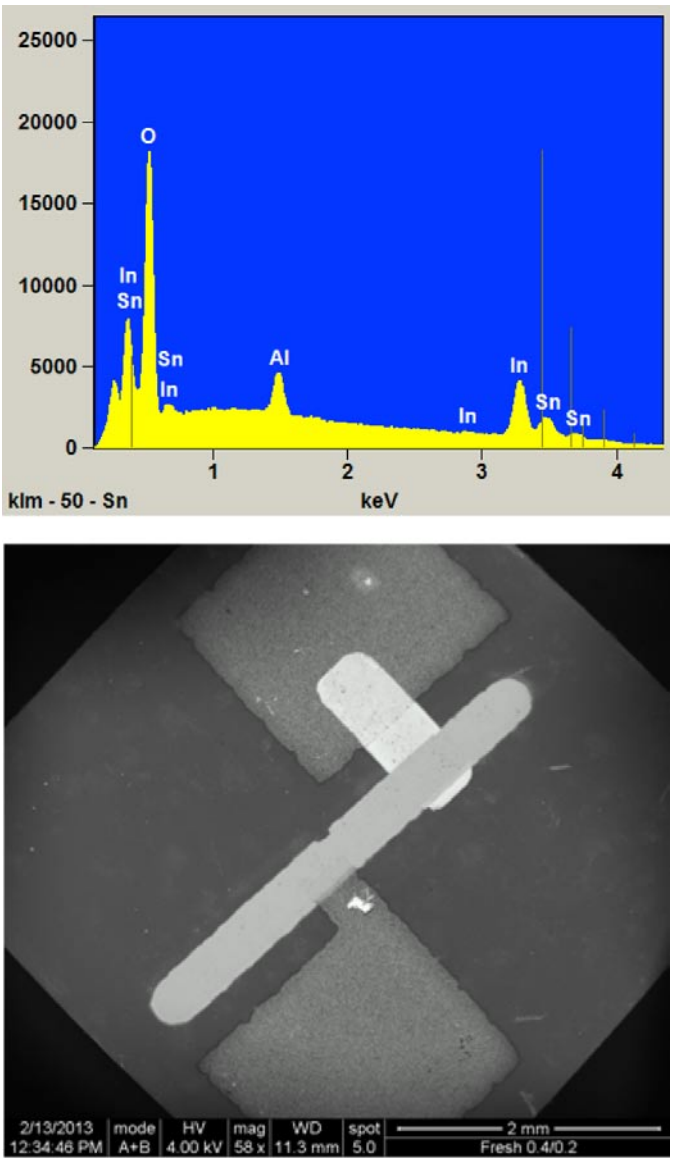

FIGURE 3A (TOP) AND 3B (BOTTOM). EDS spectra showing the elemental analysis of an ITO working electrode prepared from a single target, electron beam evaporation source (3a) and an electron backscatter detector image of the an actual H_2 sensor prepared at LANL on a commercially prepared, ESL sensor platform (3b). The final sensor layer (YSZ) can be seen in this image to cover the ITO working electrode (bright contrast) and the Pt counter electrode extension from the Pt pad used for electrical connection to the sensor.

(d) Comparison of FY 2012 and FY 2013 devices and demonstrated device-to-device reproducibility: One of the LANL/LLNL sensors prepared for NREL testing in FY 2012 was retrieved and placed into an improved sensor test stand constructed in FY 2013 at LANL. A newly prepared and packaged sensor was also tested under identical conditions. Not only were the working electrode and electrolyte prepared using different methods and equipment, the sensors tested at NREL in FY 2013 were also fabricated using a new ESL platform production lot. Despite the opportunity for substantial variations in sensor construct, the fundamental underpinnings of the sensor design and flexibility imparted though use of the HIBs permits exceptional high-level device-to-device reproducibility. This is shown in Figure 4a. Moreover, Figure 4b illustrates that precise temperature control during operation reveals a true logarithmic response not seen up to now. In past reports, a plot of sensor voltage vs. H_2 concentration would show deviations from logarithmic behavior with increasing H_2 levels. This was because

the sensor heater voltage was not automatically reduced commensurate with heating due to heat of combustion of the hydrogen. The data in Figure 4b are another indication as to how well the combined sensor/heater control has improved the performance of the safety sensor.

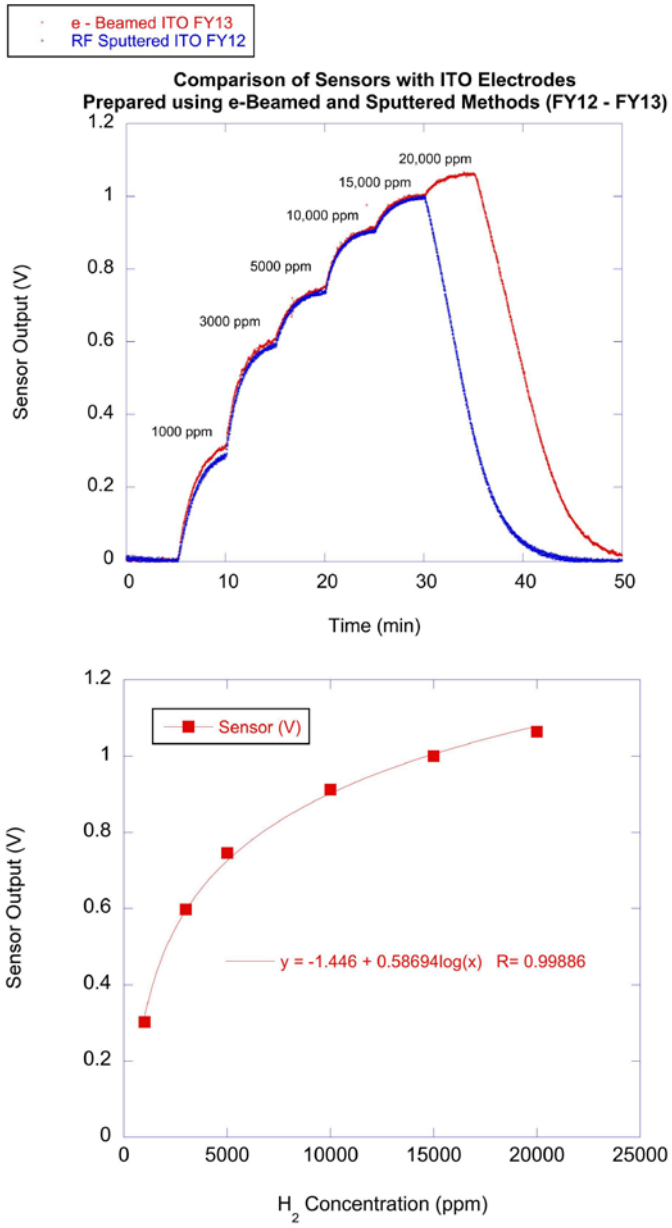

FIGURE 4A (TOP) AND 4B (BOTTOM). H_2 response comparison of a packaged H_2 safety sensor prepared and tested in Round 2 NREL testing in FY 2012 to a sensor prepared in FY 2013 using all electron beam evaporation methods (4a). The FY 2012 ITO sensor-working electrode was prepared using radio frequency magnetron sputtering and more than an order of magnitude slower deposition rate. Bottom, 4b: a plot of H_2 voltage level vs. H_2 concentration of a packaged LANL/LLNL H_2 safety sensor prepared in FY 2013; this year the high quality of fit to a logarithmic curve is the result of using temperature feedback from the sensor to control heater voltage applied to the sensor.

(e) Interference testing protocol at NREL: The packaged H_2 safety sensor prototype was tested at NREL using its interference protocol. The standard NREL test focuses on four interference gases: CH_4, CO_2, CO, and NH_3. Figure 5 shows the NREL testing results for 1%, 0.5%, 50 ppm, and 50 ppm, respectively. In these tests, the sensor baseline is first established, and 1% of H_2 is cycled. The interference gas is then turned on for 1 hr, after which 1% H_2 is reestablished together with the interference gas. After an additional hour, the H_2 is switched off and, after one more additional hour, the flow of interference gas is terminated. Finally, the sensor is cycled with one more exposure of 1% H_2. With this protocol, the sensor performance before and after interference exposure is measured together with the potential to measure the effects of simultaneous target/interference gas exposure. Figure 5 shows minimal interference effects for CO_2, CO, and ammonia. Methane produces an interesting response curve. The sensor shows a response to 1% CH_4; however, when hydrogen is introduced, Figure 5 shows that the voltage level for neat 1% H_2 is recovered and the apparent response to CH_4 is rejected. All interference gases tested impart no deleterious effects or poisoning to the sensor.

(f) Oxygen partial pressure testing; NREL anaerobic testing component of protocol: The effect of changing oxygen partial pressure on sensor H_2 sensitivity was investigated as part of the NREL standard test protocol. What was not conveyed to us was the fact that part of this protocol looked at testing in the absence of oxygen. Mixed potential electrochemical sensors using zirconia electrolytes rely on oxygen in the environment to establish competing electrode reactions and mediate the transfer of electrons that enable communication of electric potentials using a potentiostat or sourcemeter. However, the sensor will function in oxygen partial pressures ranging from roughly a fraction of a percent to 100% PO_2. This was confirmed by NREL. Figure 6 shows the sensor response to 1% H_2 cycling while the fraction of air was reduced in the experiment leading to PO_2 levels of 20%, 10%, 5%, and finally 0%. The sensor response to 1% H_2 remained unchanged as the PO_2 was reduced. However, as expected, the sensor voltage became unstable in the absence of oxygen. Moreover, the sensor response did not recover, and post mortem investigations quickly showed that the indium tin oxide sensor working electrode was not stable in anaerobic conditions, and the reducing environment produced by lack of oxygen, 1% H_2, and roughly 450-500°C sensor temperature led to reduction of the oxide to metal. The anaerobic testing duration was on the order of 5 hours, so it is quite possible that short periods of operation (e.g., <10 or 20 minutes) may not produce these irreversible effects. Once this occurred, SEM/EDS analysis showed that the metal cations diffused into the platform or alloyed with the Pt sensor pad. While the sensor cannot tolerate extended periods in such reducing conditions, the sensor nevertheless

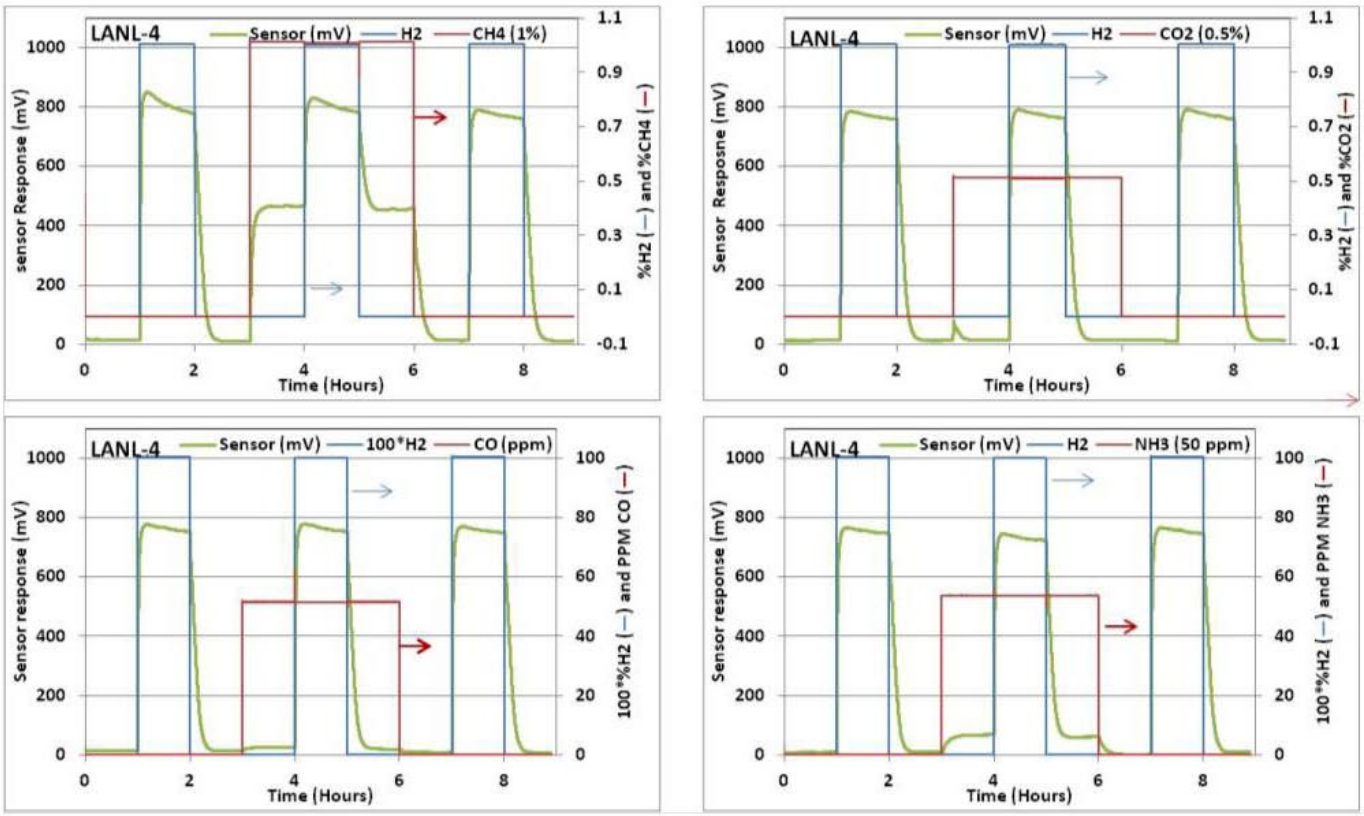

FIGURE 5. The results of standard NREL interference tests for methane (upper left), CO_2 (upper right), CO (lower left), and ammonia (lower right) at 1%, 0.5%, 50 ppm, and 50 ppm, respectively. The sensor showed minimal interference to CO_2, CO, and ammonia in these tests. The unusual response to methane (the complete rejection of CH_4 interference in the presence of H_2) is under investigation.

performed very well, and response was essentially invariant to PO_2 changes short of 0% O_2.

In FY 2013, a new working electrode material candidate was identified as a possible substitute for ITO. Lanthanum strontium chromite is a very stable electronically conducting oxide material that has been used for other applications (e.g., solid oxide fuel cells and automotive electrochemical sensor research and development) where exceptionally harsh conditions are anticipated. LANL investigations strongly indicate that this material may be substituted for the ITO for sensors that require extended anaerobic exposure periods. Though not required for the present safety application, it is available as an option for future development.

In summary, the third round of NREL testing was completed in April 2013, and all concerns identified in Rounds 1 and 2 were successfully ameliorated. Data produced in FY 2013 indicates that the technology has been validated to advance to field trials in collaboration with commercial hydrogen partners. Optimization of support electronics and improvements to the sensor technology continue to progress with the available funding.

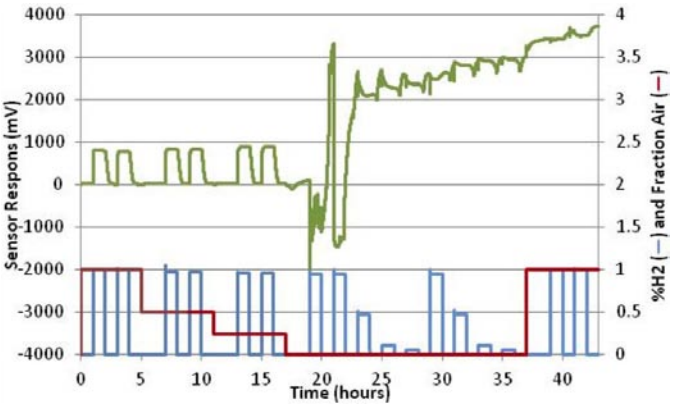

FIGURE 6. NREL anaerobic testing experiment that tests ability of H_2 sensors to withstand operation without air or oxygen. The LANL/LLNL electrochemical sensor H_2 response is invariant to PO_2 changes, but some O_2 is required based on sensor design incorporating use of oxygen ion-conducting solid electrolyte (YSZ).

CONCLUSIONS

- All FY 2013 milestones were completed this year.
- A viable H_2 safety sensor technology has been developed on an advanced sensor platform that continues to improve. An advanced H_2 sensor prototype was fabricated on an alumina substrate with ITO and Pt electrodes and YSZ electrolyte with an integrated Pt heater to achieve precise operating temperature and minimize heterogeneous catalysis.
- Multiple sensors were prepared using new electron beam vapor deposition methods and packaged. Devices may be fabricated in a fraction of the time as devices prepared by a combination of radio frequency magnetron sputtering (working electrode) and e-beam evaporation (electrolyte). Sensors prepared in FY 2013 showed identical response to sensors prepared in FY 2012, e.g., excellent response, signal-to-noise, and device-to-device reproducibility.
- Two sensors, heater control boards, and impedance buffers were tested in a third round of validation/verification conducted at NREL.
- The new heater control feedback effectively eliminated the temperature response reported by NREL in Round 1 testing.
- A combination of NREL Round 2 and 3 testing shows excellent sensitivity to H_2, reproducible device response with high signal-to-noise, minimal interferences to changes in relative humidity and barometric pressure, minimal response to changes in ambient temperature, and good to excellent rejection of potential interference gases CO_2, CO, NH_3, and CH_4.
- NREL testing showed minimal changes in sensor response over a wide range of oxygen partial pressures. H_2 response was constant until all oxygen was removed from the test chamber, showing oxygen displacement will not affect sensor response. Post mortem analysis of the device subjected to NREL anaerobic testing revealed that the highly reducing environment at operating temperature affected the ITO electrode. A new working electrode material was identified and tested and is available for applications where periods of anaerobic operation may be envisioned.

FUTURE DIRECTIONS

- Identify potential partners to plan and conduct field trials and testing at a commercial H_2 production or filling station in collaboration with NREL.
- Identify commercialization partners and plan for a path forward.
- Develop new version of sensor electronics optimized for field-testing.
- Reduce size and power consumption of the sensor element.
- Work with NREL partners to develop testing protocols for mixed potential type, electrochemical gas sensors.
- Prepare and test new sensors for field trials, test with new electronics and field packaging.
- Place sensors and data acquisition computers/data loggers at field-test location(s).

COLLABORATION AND COORDINATION WITH OTHER INSTITUTIONS

- National Renewable Energy Laboratory
- ESL ElectroScience, Inc.
- Custom Sensor Solutions, Inc.
- BJR Sensors, LLC

FY 2013 PUBLICATIONS AND PRESENTATIONS

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