# VERY LOW-COST WIRELESS HYDROGEN LEAK DETECTION FOR HYDROGEN INFRASTRUCTURE

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### ABSTRACT

A unique hydrogen leak detection strategy is the use of powerless indicator wraps for fittings and other pneumatic elements within a hydrogen facility. One transduction mechanism of such indicators is a color change that is induced by a reaction between a pigment and released hydrogen. This is an effective way to detect hydrogen leaks and to identify their source before they become a safety event, however this technology requires visual (manual) inspection to identify a color change or leak. One improvement in this strategy would be to improve the communication of the visual response to an end-user. Element One (E1) has previously developed and introduced DetecTape®, a self-fusing silicone non-reversible hydrogen leak detecting tape for application to potential leak sites in hydrogen piping, valves and fittings, and it has been successfully commercialized with excellent feedback. Element One's sensors can be fabricated using either pigments or thin films which both change color and conductivity. Neither change requires an external power source. The conductivity change may be communicated as a wireless transmission such as passive radio frequency identification devices (RFID) to an appropriate receiving system where it may be remotely monitored to achieve higher levels of safety and reliability at low cost. Element One will report on its recent progress in the commercial development of remotely monitored hydrogen leak detection using several wireless protocols including passive RFID.

# **1.0 INTRODUCTION**

The realization that climate change is a major environmental issue, along with the recent energy crisis in Europe, has led to renewed global interest in hydrogen ( $H_2$ ) as an energy carrier. Undetected hydrogen gas leaks are a potentially severe hazard and difficult to detect due to high diffusivity and unique characteristics including its high propensity to leak. During a visit by Element One to NASA Kennedy Space Center, it was reported that active hydrogen sensors as little as 20 inches away from a leak in an outdoor environment often would not detect a leaking condition. Element One has developed DetecTape® a self-fusing color changing silicone tape that when applied to a lead site will encapsulate the leak site to allow detection of the smallest of leaks.

Element One has been developing both visual and electronic detectors for hydrogen with emphasis on low cost and durability. The possibility of combining the two by implantation of small sensing devices in the matrix of a self-fusing silicone tape is currently being investigated. This combination would allow remote discovery of a leak and visual confirmation of its exact location at closer range. E1 has previously tested both ideas separately with its thin film sensors and DetecTape® and is currently involved in uniting visual detection with a wireless sensor network using Zigbee and/or RFID technology. This research is being conducted under a U.S. Department of Energy Phase II SBIR award with an objective to validate the use of our sensors in hydrogen-natural gas blends.

Each method for remote sensing has advantages. Zigbee devices are internally powered, are less vulnerable to interference and can communicate directly with the internet whereas passive RFID devices require no power and are unlikely to serve as an ignition source making their deployment extremely safe and

affordable. E1 is currently involved with industrial partners for the development of both and has already generated some initial data using a prototype Zigbee device.

# 1.1 Objectives

The main objective of E1's current research is to combine the DetecTape® visual indicator with a remote sensing platform such as E1 resistive  $H_2$  sensors, which are ideally suited for a wireless network (Figure 1). The tape and electronic sensors have both been tested extensively in the laboratory and DetecTape® has been the subject of a rigorous test protocol [2]. Both are robust and should provide seamless integration into a continuous electronic monitoring system with the additional capability of close-up visual inspection from safer distances.

# 1.2 Approach

The E1 thin film sensor design is based on the chemochromic (color change induced by a chemical reaction) behavior of certain transition metal oxides. The device uses a proprietary thin film stack incorporating transition metal oxide(s) such as tungsten oxide (WO<sub>3</sub>, a well-known chemochromic material) or molybdenum trioxide (MoO<sub>3</sub>) with a superficial discontinuous layer of catalyst and a proprietary protective coating. The composition of the thin films stack determines whether the color change is reversible or non-reversible. The current thin film device is reversible in air, although it does not require oxygen to function (unlike typical combustible gas sensors) and is also hydrogen specific.

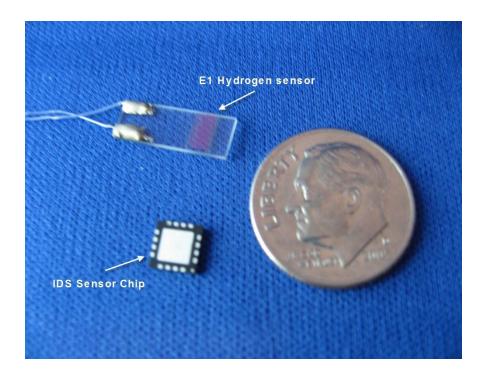


Figure 1. DetecTape® with imbedded thin film sensors as part of an integrated wireless hydrogen detection network.

#### **Design of Wireless Networks and Testing**

The wireless network sends the H2 exposure values and alerts from the sensor via the Zigbee mesh network to a central location for readout and alert generation. The data will hop from node to node and to the final destination (central station) which may be some distance away (Fig. E1). The central station is the network coordinator and will also process the data, including detection of alerts if the H2 concentration exceeds a threshold value. The station has a connection to the Internet (WiFi or Ethernet) where further data processing, storage and display is done (see Dashboard). While the range of individual nodes is limited (up to 1 km), the data hopping feature extends the range. While most nodes are sensors, some will be audio (e.g. horns) or visual (e.g. flashing light) alerts which are controlled by the central station. The setup will be done via a smart phone or laptop in communication with the central station via Bluetooth.

# **2.0 EXPERIMENTAL**

#### 2.1 Thin Film Sensors

Thin films were prepared by thermal evaporation using a Varian 3118 vacuum system. The sensor design consisted of WO<sub>3</sub>:Pd (500 nm/3 nm). Substrates were purchased commercially from Micrux, Asturias Spain. The substrates used were glass, dimensions 10 x 6 mm with interdigitated gold (150 nm Au) electrodes. Resistance measurements were made at the National Renewable Energy Laboratory (NREL), Golden, CO with a custom-built testing apparatus. Baseline resistances were on the order of 8-12 MΩ.

Initial data for the sensors were taken at NREL during the SBIR Phase 1 and consisted of testing in 2% hydrogen/air with varying levels of humidity (Figure 2). In a recent SBIR project, Element One's sensors were tested for applicability of hydrogen-natural gas mixtures of up to 20%. It was found that the presence of commercial natural gas has no adverse effect on the ability of the sensor to detect leaks. The sensors performed well, immediately responding to  $H_2$  and displaying a strong response at humidity levels up to 70%.

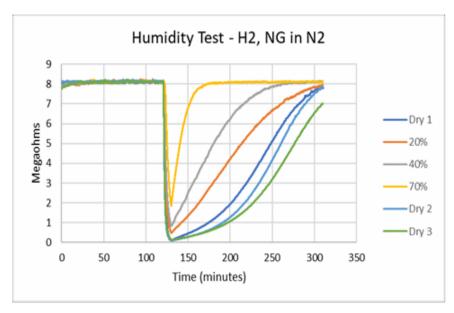


Figure 2. Thin film sensors tested in 2% H<sub>2</sub>/air with varying RH.

## 2.2 Detectape® (Self fusing silicone indicator tape)

DetecTape® samples made for Element One were obtained from Midsun Specialty Products, Berlin, CT

and were tested as received. The self-fusing silicone tape uses a pigment comprised of molybdenum oxide  $(MoO_3)$  mixed with platinum-ruthenium black (Pt) and is essentially irreversible.

# 2.3 Passive RFID

Circuitry and software for RFID experiments was obtained from Phase IV Engineering, Boulder, CO. The resistance range of the device was limited to approximately 70 k $\Omega$ . A thin film sensor was attached and monitored remotely. The sensor was then positioned on a mock flange assembly and wrapped with DetecTape® (Figure 3). Hydrogen was allowed to flow through the assembly at a very slow rate of 1 standard cubic foot per hour (scfh), equivalent to 8 standard cubic centimeters per minute (sccm).

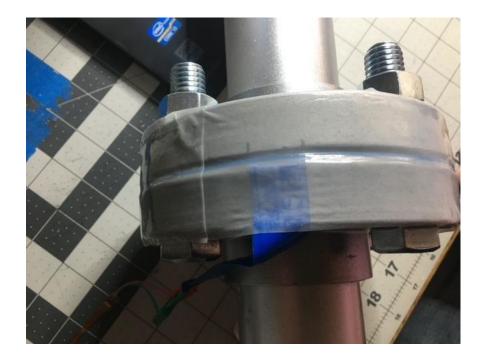


Figure 3. Mock flange assembly with an E1 resistive sensor wrapped in DetecTape®, remotely monitored with passive RFID and tested at a flow rate of 8 sccm

After less than 10 minutes the resistance reading had dropped to zero. The sensor was removed and inspected and found to be visibly colored as shown in Figure 4. The DetecTape® took considerably longer to show coloration, up to 20 minutes for a total amount of leaked hydrogen of less than one liter as noted in a separate experiment (Figure 5).



Figure 4. E1 resistive sensor remotely monitored with passive RFID showing visible coloration after 10 minutes exposure to 100% H<sub>2</sub>.



Figure 5. Mock flange assembly wrapped in DetecTape® showing before exposure (left) and after a small leak of <1 ml/s H<sub>2</sub> for 19 minutes.

Initial work to attach the Element One thin film leak detection sensor to an RFID tag was completed as shown in Figure 6. This work was done for Element One by Telaeris, San Diego, CA who was recommended by HID, a global manufacturer of RFID tags. Telaeris also makes RFID tracking software and is able to monitor hundreds of tags remotely. It is planned that this software and RFID solution will be modified for use with Element One's thin film hydrogen leak detection sensors.

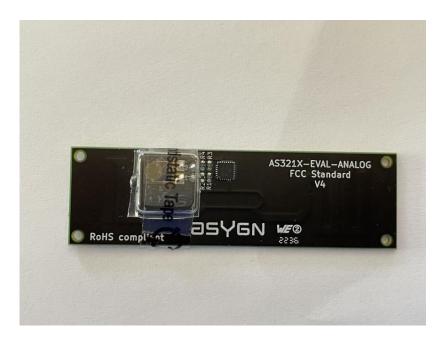


Figure 6 Element One's Thin Film Leak Detection Sensor Mounted on RFID Tag

## 2.4 Wireless Zigbee

Wireless results were obtained at NREL with a prototype Zigbee circuit assembled by Esensor, Buffalo, NY. The tests were performed in 100% H<sub>2</sub> inside a vented enclosure, with the simulated leak located several centimeters away from the sensor element. Results are presented in Figure 7, with the sensor showing a very strong response within seconds and dropping to a resistance of < 1 M $\Omega$ .

An additional test was performed on an archived sensor using 2% H<sub>2</sub>/air. The sensor was fabricated in 2019 and was over three years old. The sensor reacted immediately although the rate of response was slowed (Figure 8). These results demonstrate the longevity and sensitivity of E1 sensors as well as providing proof of concept for both state-of-the-art wireless technologies. The final design for the sensor mount will be compatible with either platform.

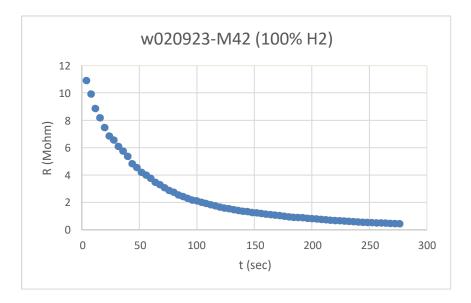


Figure 7. Response of thin film sensor tested in 100% H<sub>2</sub> using remote Zigbee device.

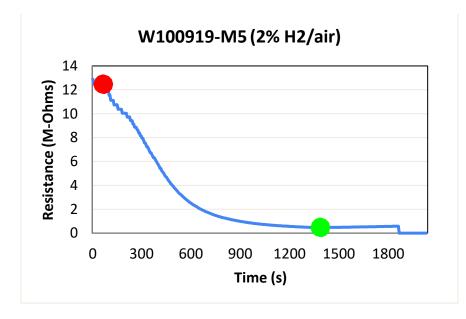


Figure 8. Response of thin film sensor tested in 2% H<sub>2</sub>/air using remote Zigbee device after 3 years ambient exposure.

# **3.0 CONCLUSION**

Having successfully tested both constituents of the design, the next step is to combine them into a single entity. E1 is currently collaborating with two industrial partners to bring the concept to the field-testing stage essentially using off-the-shelf wireless technology. Once that is complete, the visual and electronic components will be further refined to produce a final product with the benefits of both remote sensing and visual leak detection. Large-scale filed testing is scheduled for late 2023 and early 2024.

E1 thin film sensors and DetecTape® visual indicating tape can still be used separately, but when used together they can be even more effective at detecting and locating hydrogen leaks either in enclosed spaces where an explosion hazard is possible or outdoors in pipelines and refineries.

# 4.0 REFERENCES

- 1. Hoagland, W., Hydrogen gas indicator system, US Patent 6,895,805, May 24, 2005.
- 2. OSTI.GOV Technical Report: Passive Leak Detection Using Commercial Hydrogen Colorimetric Indicator, 2016.

#### **5.0 ACKNOWLEDGEMENT**

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