1-6-2012

EXP-SA: A Lateral Field Excited Acoustic Wave Sensor for Peroxide-Based Explosives

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Final Report for Period: 09/2010 - 08/2011
Submitted on: 01/06/2012
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Award ID: 0730753
Organization: University of Maine
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Vetelino, John - Principal Investigator
Title:
EXP-SA: A Lateral Field Excited Acoustic Wave Sensor for Peroxide-Based Explosives

Project Participants

Senior Personnel

Name: Vetelino, John
Worked for more than 160 Hours: Yes
Contribution to Project:

Name: Neivandt, David
Worked for more than 160 Hours: Yes
Contribution to Project:

Post-doc

Graduate Student

Name: Duy, Walter Scott
Worked for more than 160 Hours: Yes
Contribution to Project:
First year MS student in Electrical Engineering and is the primary graduate student involved in this project. Designs and implements experiments and processes data gathered.

Undergraduate Student

Name: Hackett, Brian
Worked for more than 160 Hours: Yes
Contribution to Project:
Fourth year BS student in Biochemistry. Assists the graduate student in experimental trials as well as data processing.

Name: Nadeau, Sara
Worked for more than 160 Hours: Yes
Contribution to Project:
Sara was a junior electrical engineering student who worked on testing various lateral field excited sensor platforms with different surface curvatures.

Name: FitzGerald, Michael
Worked for more than 160 Hours: Yes
Contribution to Project:
Michael was a junior majoring in physics who worked on the fabrication of lateral field excited devices.

Technician, Programmer

Other Participant

Research Experience for Undergraduates

Organizational Partners

SEACOAST SCIENCE, INC

Engineers at Seacoast Scientific worked with researchers at the University of Maine to develop films selective to peroxide based explosives. Walter Duy and Brian Hacket spent time at Seacoast Scientific working with Todd Mlsna and Steve Hobson in order to develop the technology necessary to deposit peroxide based explosive selective films. In particular they were involved in the design of polymer sensitive films to detect acetone and hydrogen peroxide vapors emitted from peroxide based explosives. They also discussed the gas testing apparatus and associated film responses.

Other Collaborators or Contacts

Activities and Findings

Research and Education Activities:

Since 9-11 and the Madrid and London Bombings, transportation security has become tighter in order to avoid future attacks. Through this, many potential threats have been averted. However, even with the increase in security, Peroxide Based Explosives (PBEs) still remain a very real and devastating threat to the safety of travelers. The only available test kits for PBEs require direct contact with the item in question. This makes for an inefficient and time-consuming process that will frustrate not only security fficials but passengers as well.

The major goal of this project is to develop a quick way to determine the presence of PBEs using a sensor that tests the air instead of direct contact (swabbing). The sensing platform being investigated is the Lateral Field Excited (LFE) acoustic wave sensor, hereafter referred to as an LFE, with a polymer film selective to PBE constituents.

To date the project has evaluated the performance of two polymer films, the constituents of which have been provided by Seacoast Science, Inc. Carlsbad, California. For succinctness the polymers are referred to as F102 and F108.

The LFEs primarily used to date have been 1 inch diameter quartz crystals with a resonant frequency of 5MHz. These LFEs were patterned with gold bite wing electrodes, which have been previously proven to have less capacitance than their half moon counterparts.

Experiments have been performed to determine how well the polymer films compatiblize with the quartz substrate of the LFE. Tests have also been performed to quantify the stability of the film over a wide range of temperatures. The sensing platform has been exposed to certain analytes (including acetone) to measure the response of the sensing platform through frequency shifts due to mass loading of the LFE.

Results to date have led to the postulation that using a holder that clamps the quartz crystal such as the Maxtech CHC-100 attenuates the sensor response as there are extraneous forces on the crystal edge that is translated to an electric field component due to the substrate's piezoelectric properties. The desire is therefore to reduce the stress on the crystal as much as possible, while maintaining the connection to the electrodes of the LFE and thus to maximize the sensor response.

In constructing a holder for the LFE that reduces the stress on the crystal due to clamping, several methods were explored. The first method was be to suspend the sensor on a platform and make a direct electrical contact to the electrodes via pogo pins. The amount of stress on the crystal is alleviated by a spring tightening mechanism. The drawbacks to this approach were that the holder is fairly large in size that the sensing face of the crystal is obstructed by the platform, making adapting this to a gas cell difficult.

The second approach was to directly wirebond the LFE to different lengths of coaxial cable. Theoretically, this would eliminate all clamping stress as there is only the direct connection to the electrodes that would be holding up the LFE. The two electrodes of the LFE would be wirebonded to the inner conductor and the outer sheath of the coaxial wire using a 2mm platinum wire. Different lengths of wire were used to test how much the length of the wire attenuated the LFE response. The method could not be implemented properly as the weight of the LFE could not be supported by the platinum wire.

The approach we are currently using is a Teflon platform that exposes the sensing surface of the LFE. the LFE lays flat on the platform electrode side up. The electrodes are then wirebonded to copper strips that are soldered onto a coaxial connection. Initially, a BNC coaxial connection was utilized, but since there have been problems with the integrity of the response, the holder uses now uses two SMA coaxial connections. This substitution changes the measurement parameter from S11 to S12 and the responses observed are much cleaner. The compact size of the holder makes it adaptable to a test cell, and since there not a clamping stress on the LFE. Results from this approach have been very satisfactory.

A computer-controlled gas delivery system (GDS) was constructed to measure the response of the LFE/film sensor system to a controllable, known concentration of acetone gas. The stock concentration of the acetone gas is 100 ppm in dry air. The GDS uses the CIO-DDA8Jr Digital to Analog Converter and Analog to Digital Converter (ADC/DAC) chip connected to a Mass Flow Controller (MFC) box which consists of DACs used to control the MFCs. The MFCs used for the GDS are (2) Tylan 2900 and (2) Tylan FC-260, all are calibrated to output a max flow of 1000 SCCM. The interface of the GDS was programmed using VB6/Softwire and can be programmed to execute eight sets of different gas concentration/humidity settings.

The process in which the GDS executes the gas concentration/humidity settings is as follows: both a purge line and a target line are run continuously. A pneumatic valve controls which line the test cell is fed. Both the purge time and the target time can be changed to test for recovery time or to let the sensor reach a steady state response.

The sealed test cell is comprised of Duniway KF Flange parts. A 55mm KF Tee is used as the test chamber and gas is delivered to this via NPT fittings, the holder is connected to the outside via an RF bulkhead. The cell is sealed using KF clamps and O-rings.

Findings:

The major findings to date have centered around the develoment of processes for compatibilizing the polymer films with the LFEs, and the measurement of frequency shifts due to mass loading when the sensing platform is exposed to acetone.

The polymer films were initially applied onto bare LFE surfaces via a spin coater. This process however resulted in an uneven coating of the sensor. Two surface treatments of the quartz surface of the LFEs were performed in order to improve polymer/LFE compatibility. The first surface treatment was to react the LFE with octadecyltrichlorosilane (OTS) in order to render its surface hydrophobic. It was postulated that this would enhance compatibility with the hydrophobic F102 polymer. The second surface treatment was to employ aminopropyltrimethoxysilane (APTMS) to render the quartz LFE surface cationic in order to compatibilize it with the electronegative F108 polymer. It was found that the surfaces treatments successfully compatibilized the respective polymers with the LFEs and enabled the creation of uniform, high quality spin coated films.

Sensing platforms treated with OTS followed by F102 were employed to determine the response to the target analyte acetone. The sensing platform was exposed to a known amount of acetone (23.7% acetone vapor by volume) and the frequency change due to the exposure recorded. The frequency change due to acetone mass loading was approximately 120Hz, a very significant result. Further it was demonstrated that the response was reversible upon removal of the acetone.

Investigations of the temperature stabiltiy of the LFEs, both coated and non-coated, revealed excellent performance over the relevant range of 20C - 60C.

The LFE response due to the Teflon holder in comparison to the Maxtech CHC-100 holder was made: The magnitude response of the LFE was larger using the Teflon holder than the Maxtech CHC-100, although the value of the response is less. This validates that the clamping stress present in the the Maxtech CHC-100 attenuates the LFE response. The drawback to wirebonding directly to the LFE electrodes is that initially, the force and voltage used to wirebond was large and it etched through the electrodes to the crystal. Since then, the amount of force and voltage have been reduced, but when the wirebonding is removed, the gold deposited onto the electrodes comes off as well. This attenuates the LFE magnitude response as more and more of the gold is lifted off, with multiple uses. As such, the crystal needs to be redeposited with Chromium and Gold after several instances of wirebonding.

The responses of both F102 and F108 have been tested using the GDS. Both the magnitude and phase of the LFE/film platform were measured as a function on %acetone delivered to the sensor platform. The flow rate of the GDS was set 750 SCCM and the acetone concentration was set at 10% intervals over the range of 0-100ppm. The purge and target times were set at 2 minutes as it has been previously tested that the sensor platform responds within a minute of exposure to acetone flow. The frequency shift was tracked using the phase peak at every acetone concentration interval relative to the frequency of the phase peak at 0% acetone. The maximum response due to acetone for both films is at

50%-70% acetone concentration (50-70 ppm), but there is a measurable frequency shift at concentrations as low as 10% acetone. The magnitude of the frequency change is less at concentrations above 70% acetone which is not as expected, making the change in frequency vs. % acetone appear to be a parabolic curve for F108. This response is reproducible over several trials.

Further testing has shown that the response of the film is attenuated over sequential trials. This is attributed to the film being saturated with acetone molecules. The recovery time of the sensor platform was then tested for the 50%-70% acetone concentration range. The film was first saturated with a target time of 10 minutes. After which, frequency shift is measured as a function of the purge time, keeping the target time at 2 minutes. The recovery time increases with an increase in concentration at a linear rate (3 minutes for 50%, 4 minutes for 60% and 5 minutes for 70%). Further tests need to be performed to detemine whether the trend follows for 80-100% concentration.

Current tests are focused on frequency change vs. time for different analyte concentrations to determine the signature and recovery time of the sensor. We have been able to develop a recovery time plot for 60 ppm acetone but have been having trouble reproducing it due to some film chemistry issues we are working out. The frequency change vs. time graphs will be compiled to create a dose response curve for acetone vapor. The same tests will be run on hydrogen peroxide vapor

Training and Development:

Walter Duy, Brian Hacket, and Michael FitzGerald have been trained in clean room protocol in addition to the operation of equipment including an ellipsometer, spincoater and network analyzer. Walter Duy has taken course work in related areas including sensor theory and polymer science. Each student has been trained in wire bonding of the LFE substrate to a holder that had been fabricated to reduce the stress on the crystal.

Sasha Alcott, a secondary school teacher, participated in research during the summer of 2010 as an RET, She has been trained in film preparation and chemistry. She has taken an introduction to sensors class to supplement her research experience.

Sara Nadeau and Michael FitzGerald participated in research during the summer of 2011 and continue their research into the 2011-2012 academic year. Sara has been examining the effect of surface curvature on the response of lateral field sensors. During the 2011 summer both students were NSF REU participants and took the introduction to sensors class to supplement their research activities.

Outreach Activities:

Several laboratory tours have been given by the project participants to high school students and state legislators.

Journal Publications

Duy, W; Hacket, B; Nadeau, S; Alcott, S; Mlsna, T; Neivandt, D; Vetelino, J, "A Lateral Excited Acoustic Wave Peroxide Based Explosive Sensor", IEEE Transactions Ultrasonics, Ferroelectrics and Frequency Control (in press 2012), p. , vol. , (2012). Submitted,

Duy, W; Hacket, B; Mlsna, T; Neivandt, D; Vetelino, J, "Detection of Peroxide Based Explosives Utilizing a Lateral Field Excited Wave Sensor", Sensor Letters, p. 1-3, vol. , (2011). Published,

Books or Other One-time Publications

W.D. Duy, B. Hackett, T. Mlsna,
D.J. Neivandt and J. Vetelino, "Detection of Peroxide Based
Explosives Utilizing a Lateral
Field Excited Acoustic Wave
Sensor", (2010). Conference Proceeding, Published
Bibliography: 13th International Meeting on Chemical Sensors, Perth Australia, July 11 to 14 2010 p. 274

Duy, W; Hacket, B; Mlsna, T; Neivandt, D; Vetelino, J

, "Detection of Peroxide Based Explosives Using a Lateral Field Excited Acoustic Wave Sensor", (2010). Conference Proceeding, Published
Bibliography: IEEE Ultrasonics Symposium, San Diego CA, Oct 11-14, 2010, pp 935-937

Web/Internet Site

Other Specific Products

Product Type:

Oral Presentation

Product Description:

Interfacial Chemistry: An Enabling Science Neivandt, Keynote Speaker, University of Tyler, American Chemical Society Student Chapter Chemistry Day, The University of Tyler, Tyler, TX, 15 Nov. 2008.

Sharing Information:

Disseminated Project Goals and Findings to Seminar Attendees

Product Type:

Conference Presentation

Product Description:

Lateral Field Excited Acoustic Wave Sensors for Peroxide Based Explosives. Vetelino, Neivandt, Mlsna, NSF EXP Grantees Conference, NSF &DHS S&T, National Science Foundation, Arlington, VA, 17-18 Jan. 2008.

Sharing Information:

Project Goals and Findings Disseminated to Conference Attendees

Contributions

Contributions within Discipline:

The project thus far has enable the collection of significant data regarding the performance of the LFE platform in air (as opposed to liquid environments). Having demonstrated the stablity of the platform in a gasous environment is a significant contribution to the field.

Contributions to Other Disciplines:

The findings of this project contribute to polymer science and film chemistry. If a film that specifically binds to both acetone and hydrogen peroxide were developed, the applications of these films on sensors to detect peroxide based explosives are numerous.

Contributions to Human Resource Development:

Three undergraduate students (Nadeau, FitzGerald, Hacket), one graduate student (Duy) and a secondary school teacher (Alcott) have received educational opportunites and training.

Contributions to Resources for Research and Education:

Masters thesis, Walter Duy, Lateral Field Excited Sensor for Peroxide Based Explosives, MSEE 2011, University of Maine

Contributions Beyond Science and Engineering:

The research on the peroxide based explosives sensor can be extended and commercialized to be used in transportation centers.

Conference Proceedings

Categories for which nothing is reported:

Any Web/Internet Site

Any Conference