R2-B.2: Portable, Integrated Microscale Sensors (PIMS) for Explosives Detection

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II. PROJECT DESCRIPTION

Core funding for this project ends in Year 3 per the outcome of the Biennial Review process.

A. *Project Overview*

To successfully deter and detect explosives threats, a multimodal technical approach based upon an array of orthogonal or near-orthogonal sensing technologies (e.g. spectroscopic systems, imaging systems, swab-based sensors, etc.) is essential. The present effort seeks to develop a sensing system that can address one such detection vector, namely portable, integrated, microscale sensors (PIMS) that are suitable for vapor-phase explosives detection. These small-scale, cost-effective sensing systems are ideally suited for integration into existing baggage, cargo, and passenger screening portals, building ventilation systems or handheld portable devices, and, as subsequently described, exhibit performance metrics (e.g. false positive/negative rates, sensitivities, and power consumption metrics) that are expected to compare very favorably when integrated in operational environments.

The PIMS devices being developed within the context of ALERT are based on so-called bifurcation-based mass sensing principles, wherein vapor-phase target analytes chemomechanically interact with a functional layer (typically a polymer) deposited upon the oscillating surface of a microscale electromechanical resonator. This interaction renders a change in the resonator's effective mass, eliciting a shift in natural frequency and, given that the system is driven into a nonlinear response regime with two stable response branches, creates a marked change in amplitude (see Fig. 1 on the next page). Because this approach utilizes a nonlinear mechanism and a threshold technique for sensing, the associated control electronics can be greatly simplified (in comparison to sensors based solely on resonance-shift principles), which significantly aids the development of portable sensors with reduced form factors and favorable power consumption metrics. In addition, the sensitivity of the system can be widely tuned, as it is not based solely on the underlying physics of the device. In prior work by the investigators, which focused on vapor-phase alcohol sensing, bifurcation-based mass sensors were shown to yield superior performance metrics (i.e. false positive/negative rates, sensitivity and power consumption metrics) in laboratory environments and compared favorably to their more conventional counterparts [1, 2].

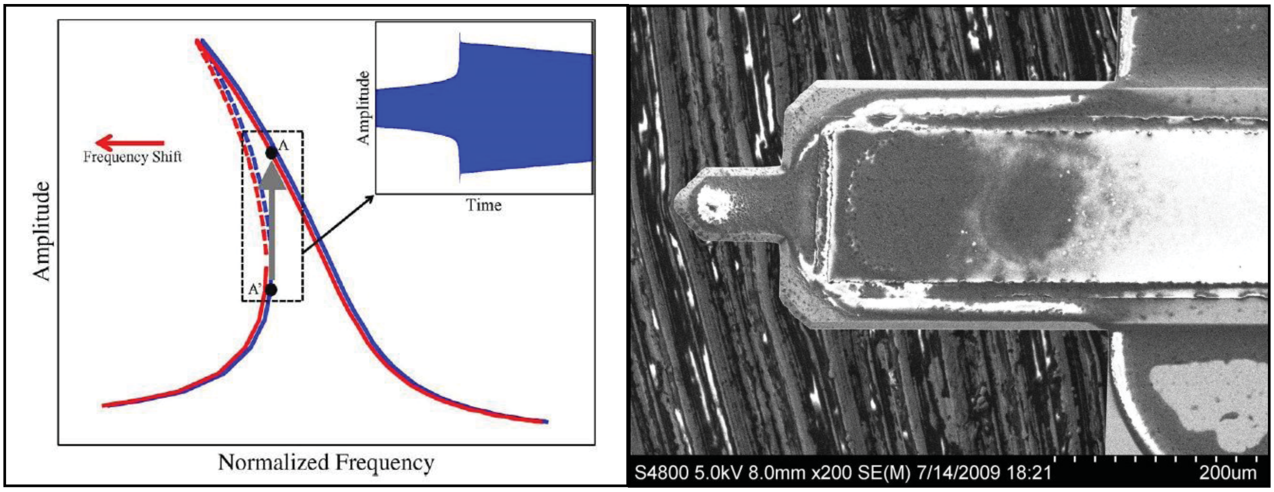

Figure 1: (left) Frequency response of a typical bifurcation-based sensor with a softening nonlinearity, before (blue) and after (red) adsorption of mass onto the electromechanical resonator's surface. The stable response solutions are represented by solid lines, while the unstable response solutions are represented by dashed lines. Points A' and A represent the resonant amplitudes prior to and post adsorption, obtained when the system is excited at a constant excitation frequency. As the mass adsorbs onto the resonator's surface, the frequency response shifts to the left, resulting in a sudden jump in the response amplitude (the inset shows the time response as the system moves across the bifurcation point). This transition, induced by chemomechanically-induced shifts in the natural frequency of the resonator, can be correlated to a mass detection event [1, 2]. (Right) Scanning electron microscope image of a representative functionalized Veeco DMASP probe – the first bifurcation-based sensor developed by the investigators [1, 2].

Given the investigators' prior research related to PIMS, the present effort was specifically focused on:

- Developing a new class of cost effective and tunable bifurcation-based mass sensors which are suitable for vapor-phase explosives detection. Though not included in the original statement of work, this task became essential when the commercial platform that the investigators had repurposed as a bifurcation-based mass sensor in prior work, the Veeco DMASP probe shown in Figure 1, was discontinued shortly before the onset of the initial performance period.
- Developing a new inkjet-based functionalization system which is capable of rapidly and precisely depositing functional surface layers on the sensors developed herein.
- Developing new, low-power control and signal processing electronics, designed to enable portable functionality, while maintaining performance.
- Validating sensor performance with real energetic materials within both laboratory and operational (wherein the impinging fluid flow becomes important) environments.
- Characterizing pertinent sensor metrics (e.g. false positive/negative rates, sensitivities, power consumption, etc.) and benchmarking these metrics against alternate sensing platforms.
- Overcoming the basic research challenges associated with integrating all of the sensing system's constituent pieces in a single, portable platform.
- Modeling the complete sensing system with an eye towards predictive design, performance optimization and, ultimately, technology transition.

B. Biennial Review Results and Related Actions to Address

This project was slated for sunset in the last Biennial Review and was concluded at the end of Year 3.

C. State of the Art and Technical Approach

Chemical and biological sensors based on resonant micro- and nanoelectromechanical systems (MEMS and NEMS) have garnered considerable research attention over the past two decades [3-5]. Since the initial demonstrations of microscale resonant mass sensing in water vapor and mercury detection, the field has expanded to encompass a wide variety of applications ranging from medical diagnostics and environmental safety to national security and public safety [6-11]. Resonant mass sensors typically utilize chemomechanically-induced shifts in the frequency response of an isolated electromechanical resonator or an array of electromechanical resonators for analyte detection [7, 12, 13]. This approach has in fact been used with varying degrees of success in explosives sensing contexts in prior work [10, 14-21]. While a number of researchers have demonstrated the distinct utility of this approach in terms of both sensitivity and application space, sensors that exploit nonlinear behaviors have the potential to: (1) render improved performance metrics; (2) offer tunable sensitivities; and (3) simplify final device implementations and thus reduce power consumption and form factor metrics by eliminating the need to employ frequency tracking hardware, which is often attendant to conventional microscale mass sensor designs [1, 2, 22-25].

This effort sought to build upon the latter body of research by developing PIMS based upon the bifurcation-based sensing principles described above. This technical approach is markedly different from other known research, a possible exception being that research described in [22], in that it is focused on: (1) the vapor-based detection of explosives; (2) an engineering-based technical solution that emphasizes overcoming the hurdles associated with developing a cost-effective sensor platform capable of detection in operational environments, rather than solely performance metric enhancement; and (3) developing a scalable sensing solution which is suitable for technology transfer.

D. Major Contributions

D.1. Third performance period: 2015-2016

Though the initial laboratory tests focused on vapor-phase TNT detection (completed in Year 2) were promising (see Figure 10), upon false positive/negative characterization, it became clear that the sensor platform developed over the first two years of the study lacked the sensitivity and selectivity to perform optimally in field environments, and required further design refinement. To this end, the third, and final, period of performance for this project focused on enhancing (1) sensor selectivity and (2) sensitivity with a specific eye towards transitioning the sensors from the laboratory to realistic field environments. Technical details related to recent and ongoing research in each of these respective areas is delineated below.

D.1.a. Selectivity enhancement through refined surface chemistry selection

In the first two years of effort, functional surface chemistries suitable for the detection of TNT vapor, which were reported in prior literature, were screened based on a literature review and directly converted into an ink form and deposited onto the sensor. However, as noted above, preliminary false positive/negative testing revealed that some of these chemistries lacked the specificity required for field use. To address this concern, the investigators expanded their partnership with the Boudouris Group at Purdue University and experimentally pre-screened a number of functional ink variants suitable for vapor-based TNT detection. Figure 2 highlights the performance of a thin film of the TIPS-pentacene chemistry that was eventually selected for use with the 4^{th} and 5^{th} generation of PIMS, as characterized through absorption measurements obtained using UV-Visible Spectroscopy. As evident from the figure, there was a notable and rapid shift in the film's intensity spectra following brief exposure to trace amounts of TNT vapor, and this shift dissipated with time as the surface was exposed to ambient air absent of the energetic vapor.

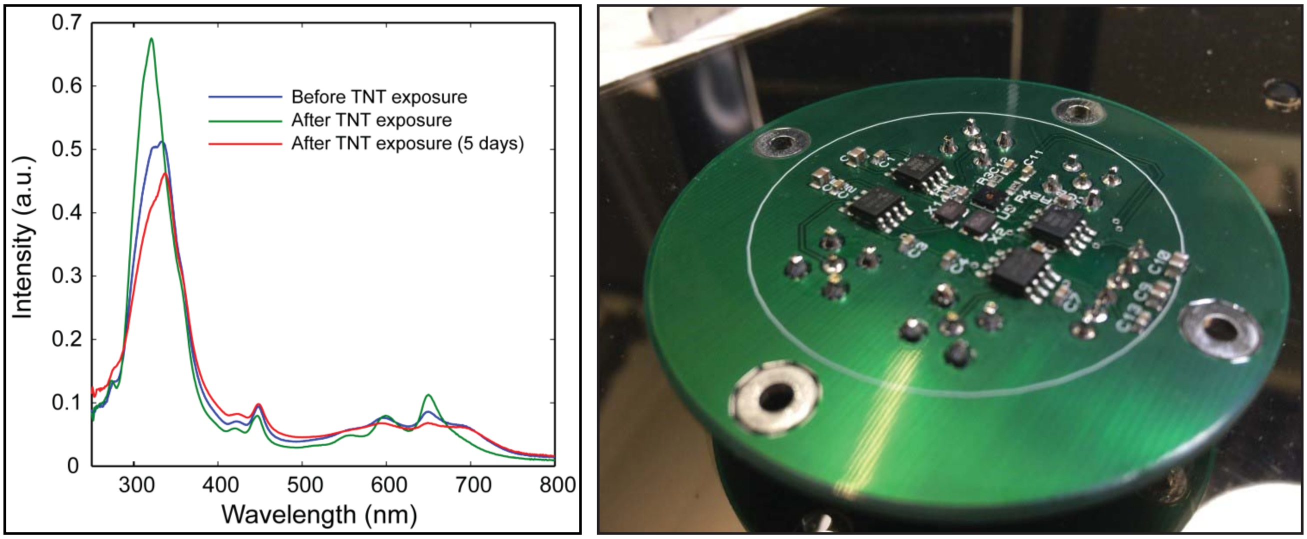

Figure 2: (left) Absorption measurement, obtained via UV-Visible Spectroscopy, from a thin film of TIPS-pentacene prior to and after exposure to a trace amount of TNT vapor (for 10 minutes). (Right) A 4th generation PIMS – this device is approximately the size of a US dollar coin.

Given the independent verification (via UV-Visible Spectroscopy) of the chemical functionality of the TIPS-pentacene, a layer of the material was deposited on a series of 4th generation PIMS, similar to that depicted in Figure 2. The sensors were then placed within the refined laboratory test set-up (see Fig. 3 on the next page) to experimentally characterize their performance in vapor-phase explosives detection. The results of a representative linear detection experiment are highlighted in Figure 3. As evident, the functionalized resonator exposed to the TNT vapor/ambient air mixture exhibited a noticeably larger shift in resonant frequency, as compared to the control resonator (which was exposed to the same mixture in the absence of a functional layer), indicating a positive detection event (note that coated and uncoated resonators exposed to ambient air demonstrated no measurable shift). Though the experiment detailed here lasted for 45 minutes (an infeasible screening time), notable differences between the outputs of the control and test sensors were seen in just a few minutes – a timescale deemed acceptable given the preliminary nature of the tests. The investigators believe these timescales can be shortened with further sensor development and implementation of the bifurcation-based sensing circuit in the 5th generation PIMS design.

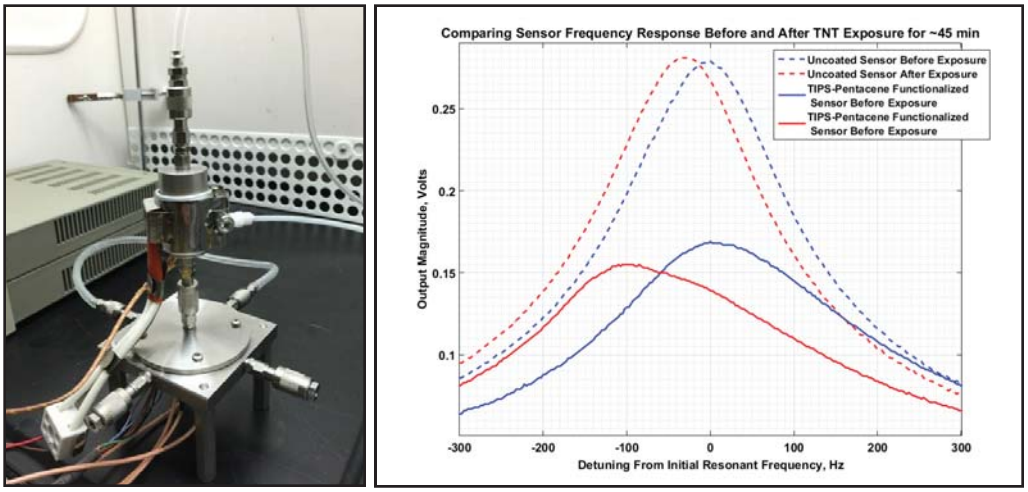

Figure 3: (left) Current laboratory-based experimental test set-up for PIMS characterization. (Right) Results obtained from a linear resonance shift detection experiment highlighting the efficacy of the tailored TIPS-pentacene chemistry.

D.1.b. Sensitivity characterization and enhancement

Concurrent with the drive towards better selectivity (detailed above), was a series of research activities aimed at improving the sensitivity of the PIMS. This push to obtain sensitivities deeper into the pg/Hz regime (specifically, sensitivities near 10 pg/Hz) was motivated by the desire to detect lower concentrations of TNT vapor in ambient air than those utilized in the original (Year 2) laboratory testing. Given the need for an appreciable functional surface area on the sensor, a conscious decision was made to realize this enhanced sensitivity through a push towards higher-frequency resonant elements, which utilized planar bulk modes in contrast to bending modes (a favorable change, due to their inherently higher quality factor), yet required higher-frequency control electronics (a distinct technical challenge, given the time delay issues inherent to the requisite circuitry). As detailed in Reference [26], this technical push ultimately proved successful, with the investigators realizing an open-loop controlled device with sensitivities of approximately 11 pg/Hz, when properly functionalized. In the course of this experimental endeavor, the authors also developed a new experimental technique capable of characterizing the spatially-dependent sensitivity exhibited by bulk mode resonators and their larger brethren (for example quartz crystal microbalances); see Figure 4 and Reference [26].

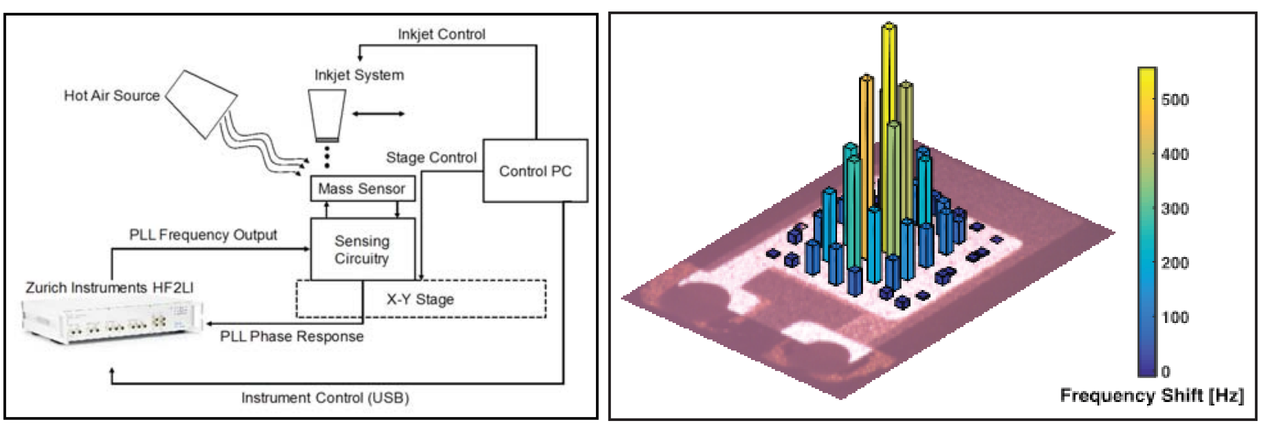

Figure 4: (left) Experimental set-up used to characterize the spatially-dependent sensitivity of the resonant element utilized in 5th generation PIMS. (right) Experimentally-obtained sensitivity map obtained from the resonant element used in 5th generation PIMS.

D.1.c. A translatable sensor platform

Given the aforementioned advancements in sensitivity and selectivity, research efforts in the fourth quarter of Year 3 have focused on: (1) updating the bifurcation-based sensing control electronics architecture to make them amenable for use with the higher-frequency resonant element; (2) adding on-board temperature and humidity compensation to the 5th generation PIMS sensors as a way of minimizing false positive/negative detections; and (3) continuing to characterize the performance of the 5th generation of PIMS in laboratory environments in the detection of both TNT and peroxide-based explosive threats. The investigators believe at least a portion of each of these endeavors will be completed by the project's conclusion later this summer.

D.2. Second performance period: 2014-2015

In the second period of performance for this project, research efforts focused on three key fronts: (1) refinement and characterization of the nonlinear feedback control underlying the PIMS platform; (2) sensor functionalization; and (3) characterizing sensor performance in laboratory environments. Technical details related to recent and ongoing research in each of these respective areas is delineated below.

D.2.a. Refinement and characterization of the nonlinear feedback control

As noted above, though it was not included in the original statement of work, it became imperative for the investigators to develop a new bifurcation-based sensor platform in the first year of effort because the commercial platform originally intended for repurposing and use as a resonant sensing element, the Veeco DMASP probe shown in Figure 1, was discontinued by the manufacturer shortly before the start of the initial performance period. To this end, the PIs, working in conjunction with their research assistants, developed a simple control strategy dubbed "feedback nonlinearization" (see Fig. 5), which could morph the commercially-designed-for, linear frequency response structure of any quartz or MEMS resonator (e.g., those depicted in Fig. 6 on the next page) into the nonlinear frequency response structure required for bifurcation-based sensing. While this control strategy was successfully implemented near the end of the first performance period, it became clear during testing that additional refinement was needed in the second year of the effort. To this end, the analog circuitry paired with the resonators went through a series of design iterations, eventually yielding the circuit design depicted schematically in Figure 7 on the next page and pictorially in Figure 8 on the following page. While a complete set of technical details related to the design and implementation of this system can be found in Reference [27], for present purposes, suffice it to note that this system contains three main subsystems: (1) the actuation and sensing subsystem, which is used for resonator transduction; (2) the filtering subsystem, which is used for on-chip signal conditioning; and (3) the cubic feedback generation system, which is used to realize the desired nonlinear frequency response characteristic. When properly designed and implemented on-chip, these subsystems provide the functional backbone necessary for bifurcation-based sensing.

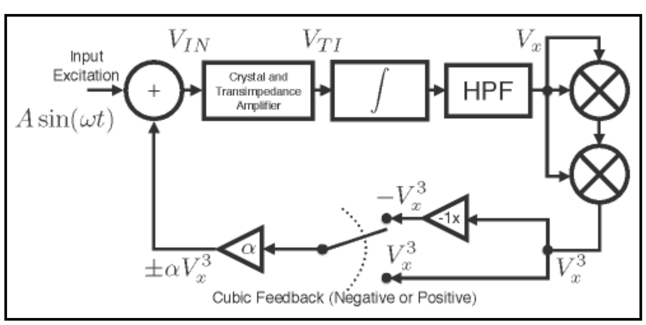

Figure 5: Schematic representation of the final feedback control concept.

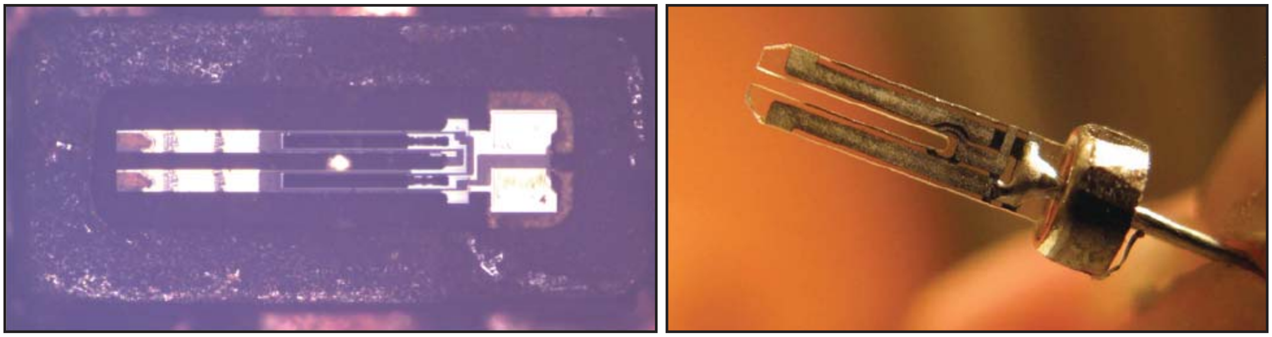

Figure 6: Examples of quartz tuning fork devices: (left) Epson FC-135 and (right) Abracon AB38T.

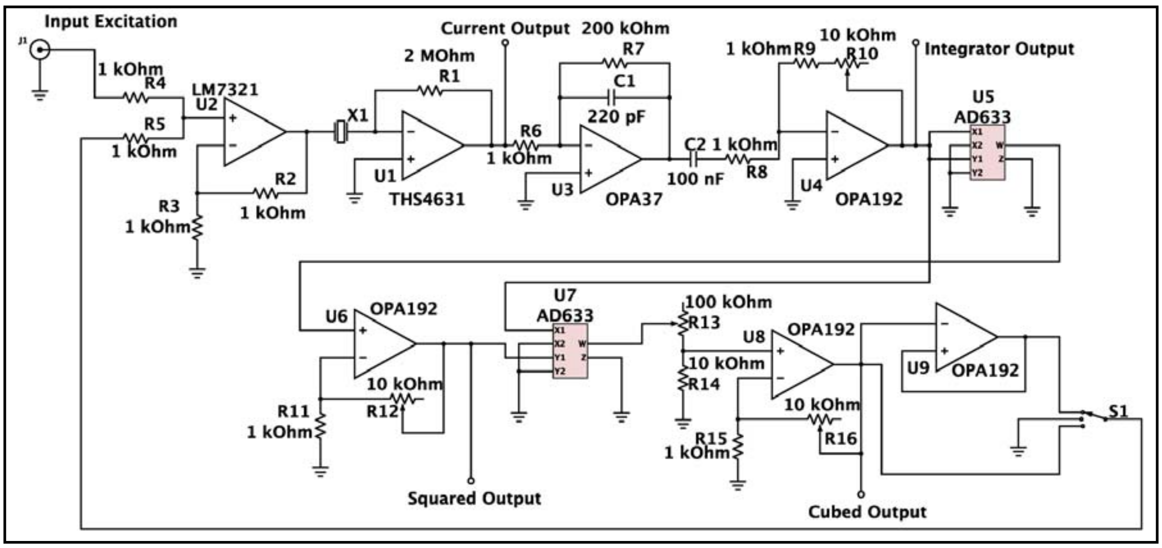

Figure 7: Detailed circuit diagram of the bifurcation-based sensing platform. Note that power supply lines are omitted aside from ground, as are buffer operational amplifiers from the output connections and decoupling capacitors.

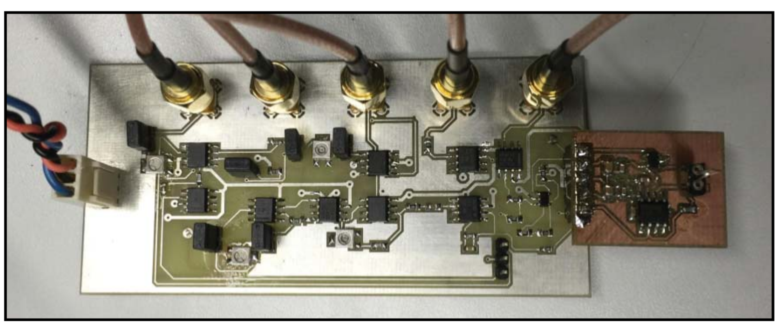

Figure 8: Implementation of the design depicted in Figure 7. The resonant sensing element can be seen on the smaller PCB (on the right), along with the drive and transimpedance amplifiers.

Following the development of the aforementioned closed-loop system, its frequency response behavior was characterized using forward and backward sweeps of the input excitation. Key results from this study are included in Figure 9. As evident from this figure, the feedback nonlinearization strategy is not only capable of developing the desired softening frequency response characteristic, but is capable of doing so in a tunable fashion (the saddle-node or cyclic-fold bifurcation can be moved with increasing excitation amplitude). Furthermore, even in the absence of tuning, the control system is capable of producing the desired structure in multiple devices of the same variety and, though the results are not included here for the sake of brevity, devices of multiple varieties. The control architecture also was found to provide an additional capability to enhance the robustness of the PIMS platform against non-target analytes and external disturbances, as needed to reduce false-positive events. Given these positive results, the PIs determined that the newly-developed, closed-loop resonator platform was sufficient for implementation in a PIMS platform and, as such, the PIs redirected efforts in the remaining portion of the performance period towards sensor functionalization and laboratory characterization.

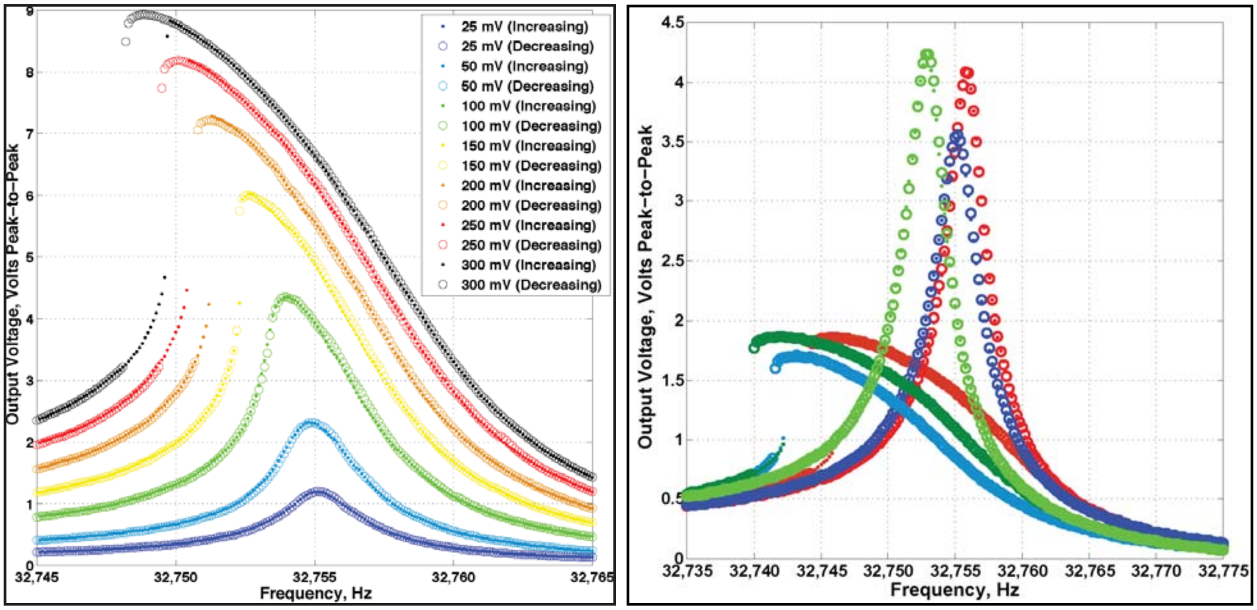

Figure 9: (left) Demonstration of the tuning of the Duffing-like frequency response in the softening feedback mode, with a fixed gain and increasing excitation amplitude. (Right) Softening responses of three different AB38T tuning fork crystal devices, with their respective linear responses shown as well. These devices were excited with the same fixed feedback gain (for the softening case) and the same input excitation.

D.2.b. Sensor functionalization

With a robust resonator platform in hand, research efforts shifted to sensor functionalization. As detailed below, in the first year of this research effort, the PIs, working in conjunction with their research assistants, developed a customized inkjet functionalization system (see Fig. 10 and Table 1) capable of depositing picoliter-scale drops of functional polymers with micron-level accuracy and precision. Accordingly, research efforts in the second period of performance focused on the development and selection of appropriate surface chemistries, as well as deposition process control and optimization.

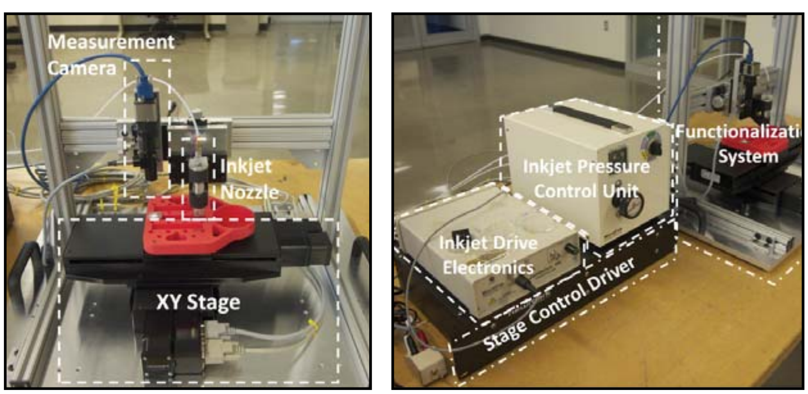

Figure 10: The investigators' customized inkjet functionalization system fabricated as part of this research effort. Key metrics of this system are highlighted in Table 1.

Parameter Name	Value	Units
Stage Travel – X	200	mm
Stage Travel – Y	200	mm
Maximum Speed	50	mm/s
Positioning Accuracy	3	μm
Positioning Repeatability	$\pm$0.5	μm
Nozzle Orifice Diameter	20 - 80	μm
Recommended Fluid Viscosity	<20	cp
Recommended Fluid Surface Tension	20 – 70	dynes/cm
Registration Camera Resolution	2.65	μm/pixel

Table 1: Performance metrics of the inkjet functionalization system developed as part of this effort.

Being cognizant of both nitroaromatic and organic peroxide threats, surface chemistries suitable for the detection of both classes of compounds were explored in conjunction with the Boudouris Group in the School of Chemical Engineering at Purdue University. With regard to the detection of nitroaromatic explosives, such as trinitrotoluene (TNT), the PIs and their partners leveraged the wealth of existing chemistries developed in prior work, including those that reversibly bind to specific molecules due to hydrogen bonding interactions (see, for example [28]), as well as self-assembled monolayers of 4-mercaptobenzoic acid (4-MBA) and 6-mercaptonicotinic acid (6-MNA), which provide carboxylic acid end groups that can reversibly bind with the nitro groups on analytes such as TNT [29]. Likewise, for organic peroxide sensing, the PIs and their partners have leveraged the fact that the weak O-O bond in these molecules breaks down into free radicals that are highly reactive. Accordingly, it is believed that these radicals can be easily detected through the interaction with another radical bearing species, specifically a radical polymer. Radical polymers are macromolecules bearing a stable radical site on the pendant groups of a non-conjugated carbon backbone [30]. The radical site on these polymers will react with the radicals generated from organic peroxide molecules to irreversibly bind them to the sensor. The radical polymer currently under investigation is poly (2, 2, 6, 6- tetramethylpiperidinyloxy methacrylate), abbreviated PTMA, which has been synthesized through RAFT polymerization in the Boudouris Group [31].

For each of the diverse chemistries detailed above, the PIs have worked in conjunction with their partners to develop ink-like solutions through the careful selection of solvents and solids loading. While the technical merit of the “inks” can only be determined through extensive testing, the PIs believe that the underlying science is sufficiently mature for these to be a success. Accordingly, recent research efforts have focused on the rapid deposition of these chemistries onto a wide array of resonator platforms (see Fig. 11 on the next page), as well as further refinement of the inks themselves.

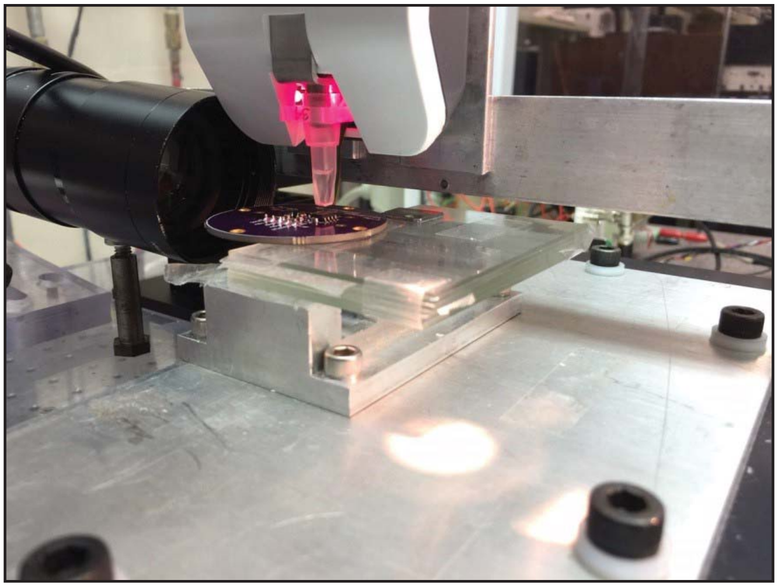

Figure 11: A PIMS platform being functionalized with PTMA.

D.2.c. Characterizing sensor performance in laboratory environments

With the electromechanical development of the PIMS platform effectively complete, efforts in Q4 of 2014 focused on characterizing the PIMS' performance in a laboratory environment, with an eye towards transition to the field. Given the desire to further leverage Purdue University's extensive experience with real energetic materials and the PI's more recent experience with this same class of materials, significant effort was expended to certify a new laboratory building, secure a magazine for explosives storage, and certify a subset of associated staff and research assistants in safe chemical and explosives handling. Likewise, significant effort was expended to develop two new scaled test chambers (see Fig. 12), each of increasing volume, which could be used to scale up the sensor's operational environment from a few milliliters to a >50,000 ft^3 open room. This was done with the belief that if the sensor platform failed to meet key performance metric thresholds in the low-volume test environments, it was sure to fail in a larger opens space, even with proper sensor placement and flow control.

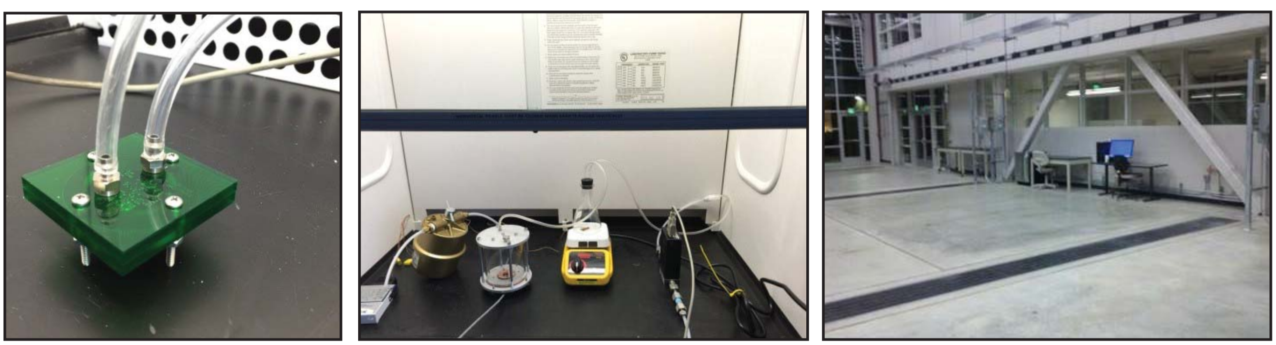

Figure 12: The investigators' test environments ranging in volume from a few milliliters to > 50,000 ft^3.

Though sensor testing was still in a nascent stage, preliminary results were promising. Figure 13 (on the next page), for example, highlights the result of one PIMS test conducted using the test environment depicted in the center of Figure 12. Here, a small amount of TNT (approximately five flakes) was placed within a warmed beaker to create a vapor source. This vapor was then mixed via precision mass flow controllers with ambient air and fed into the test chamber. As evident from the acquired results, a positive detection event was recorded, albeit not at the temporal rate desired by the PIs, due in part, to the non-ideal nature of the employed pentacene chemistry. Subsequent efforts were squarely focused on building upon this result and others like it, developing statistically-significant performance data and characterizing pertinent performance metrics.

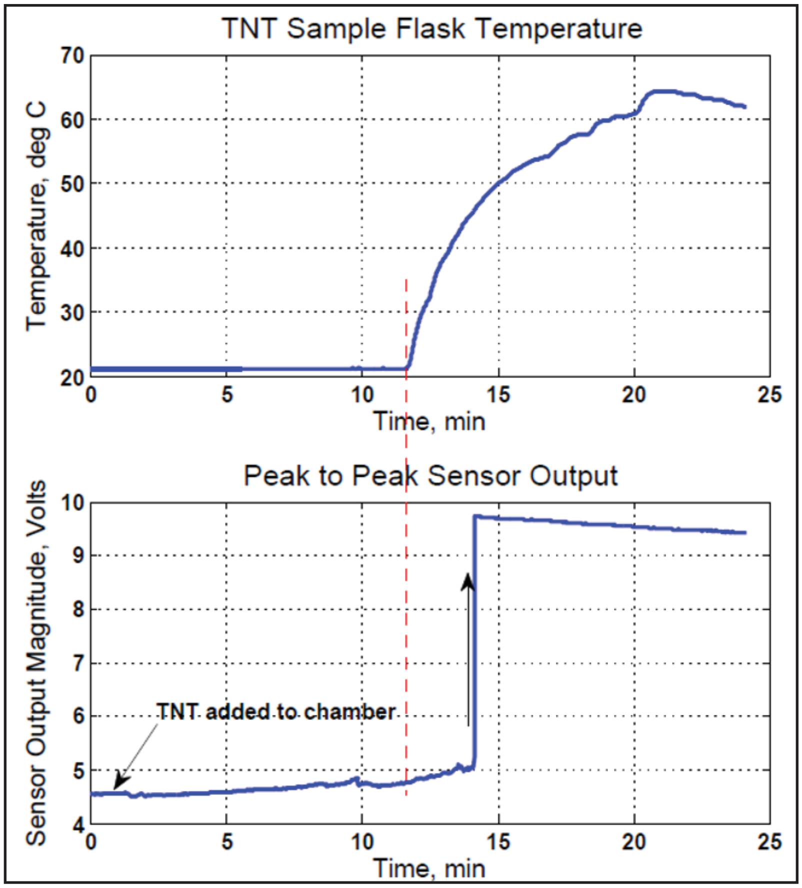

Figure 13: A representative, preliminary result obtained during a positive detection event.

D.3. First performance period: 2013-2014

In the first period of performance, research efforts focused on three key fronts:

D.3.a. Development of a new, low-cost bifurcation-based sensor platform

As noted above, though it was not included in the original statement of work, it became imperative for the investigators to develop a new bifurcation-based sensor platform at the onset of this project. Fortunately, with challenge comes opportunity, and this temporary barrier allowed the investigators to explore, and ultimately select a new resonator platform which compared very favorably in terms of both cost (retail costs are on the order of a few cents in comparison to more than $75) and device-to-device repeatability.

The selected platform, two examples of which are depicted in Figure 6, was a repurposed quartz tuning fork originally designed to be a timing reference in commercial electronics. This electromechanical system, which utilizes piezoelectric mechanisms for both actuation and sensing, offers exceptional stability and is widely understood when operated in conventional (linear) modalities. Though prior works have explored the use of these tuning forks as sensors, all known works have exploited linear sensing principles akin to those described above, wherein shifts in one or more of the system's resonances are used to signal a detection event. For purposes of this effort, the investigators explored the frequency response characteristics of a series of representative resonators and determined that the first torsional mode of vibration most frequently exhibited the nonlinear frequency response characteristics requisite for bifurcation-based mass sensing (see Fig. 1) when driven beyond the typical excitation range; a softening nonlinearity with a notable jump in response amplitude as the system transitions across the sub-critical bifurcation point. Unfortunately, this nonlinear response was found to vary appreciably from resonator to resonator; a likely by-product of the fact that these devices, as originally conceived, were designed to operate within a linear response regime.

D.3.b. Development of new control and signal processing electronics to enable portable functionality

To overcome the device-to-device variability in the systems' nonlinear frequency response noted above, and to ensure that any randomly-selected resonator would exhibit the desired frequency response structure, the investigators developed a simple control strategy dubbed "feedback nonlinearization". The antithesis of a feedback linearization system [32], this controller ensured that any quartz or MEMS resonator designed to exhibit a linear frequency response structure, exhibits the softening nonlinear frequency response structure required for bifurcation-based sensing (additional details can be found above). The effectiveness of this novel control approach is clear from the results presented above.

D.3.c. Development of a new inkjet functionalization suite capable of rapidly and precisely depositing functional surface layers on the sensors developed herein

The transition from resonator to functional sensor is not only dependent on signal processing and control electronics, but also sensor functionalization. Specifically, the resonant mass sensors described herein require a functional surface layer to detect explosives vapors. To facilitate precise and rapid material deposition, the investigators fabricated, tested and implemented a customized inkjet functionalization system (see Fig. 10). This system, consisting of an inkjet nozzle, precision positioning stages, a measurement camera, and associated control electronics is capable of depositing picoliter-scale drops of functional polymers with micron-level accuracy and precision (see Table 1). This system enables the investigators to deposit various functional materials on the surface of the bifurcation-based sensors, or any other ALERT sensor platforms in a highly-controlled fashion.

E. Milestones

The following milestones were largely completed within the scope of Year 3 research:

- Investigated advanced inkjet-based functionalization strategies, new ink formulations, and their influence on performance metrics.
- Overcame the basic research challenges associated with integrating all of the sensing system's constituent pieces in a single, portable platform.
- Collaborated to develop a fluid delivery strategy to ensure vapors reach the sensor.

The following key milestones must be achieved for this project to reach its full potential. Note that given the sunset status of the project, some of these tasks may not be completed.

- Validate sensor performance with real energetic materials (solid and liquid) within both laboratory and operational (wherein the impinging fluid flow becomes important) environments.
- Characterize pertinent sensor metrics (e.g. false positive/negative rates, sensitivities, power consumption, etc.) and benchmark these metrics against alternate sensing platforms. Model the complete sensing system with an eye towards predictive design and performance optimization.

F. *Future Plans*

This project will be sunset at the conclusion of Year 3.

III. RELEVANCE AND TRANSITION

A. *Relevance of Research to the DHS Enterprise*

As noted above, to successfully deter and detect explosives threats, a multimodal technical approach based upon an array of orthogonal or near-orthogonal sensing technologies (e.g. spectroscopic systems, imaging systems, swab-based sensors, etc.) is essential. The present effort seeks to develop a sensing system which can address one such detection vector, namely PIMS that are suitable for vapor-phase explosives detection. These small-scale, cost-effective sensing systems are ideally suited for integration into existing baggage, cargo, and passenger screening portals, as well as building ventilation systems, or handheld portable devices, and are expected to exhibit performance metrics (e.g. false positive/negative rates, sensitivities and power consumption metrics) that compare very favorably when integrated in operational environments to their more conventional counterparts.

Our present goal is to develop a sensing system capable of:

- < 0.5% false positive/negative rates
- < 50 pg/Hz sensitivity
- Operating for >24 hours using its own household battery or power from a host platform
- Fitting within a package measuring <2 in^3

It is important to note that, though the proposed sensors are being developed with vapor-based explosives detection in mind, the underlying technology could plausibly be used for a wide variety of DHS-relevant missions, including the detection of hazardous chemicals, contraband, etc. In essence, only the functional surface layers added to the resonating sensor elements need to be changed to enable the detection of a new substance or class of substances.

B. *Potential for Transition*

The PIs believe that the transition potential of the sensing platform described herein is high. To this end, the PIs competed for and subsequently received $50,000 of support from the Purdue Research Foundation's TRASK Innovation Fund. This support, which is specifically allocated to support the direct costs of applied research that is complementary to existing basic research, was used to push PIMS down the transition pathway through: (1) the evaluation of sensor performance in a more holistic way than originally proposed in this basic research effort; (2) the integration of the sensing system with a mock screening portal (ongoing); and (3) the development of a more refined prototype PIMS system than would be achievable given the financial constraints of the present effort. The PIs hope that through this march down the transition pathway there can be greater engagement with end users to ensure that the final prototype(s) will truly meet their needs.

C. *Data and/or IP Acquisition Strategy*

The PIs hold all intellectual property relevant to this study. With respect to data, as the project nears its conclusion, the PIs hope to gather additional information related to key sensor performance metrics from DHS partners to ensure that those realized with the PIMS platform are both appropriate and acceptable.

D. *Transition Pathway*

As detailed above, the PIs are pushing the PIMS platform down the transition pathway through engagement with the Purdue Research Foundation. This process, which started in June 2015, is designed to produce a start-up company or licensable technology. It should be noted that the Foundation has deep, working relationships with DHS and the Department of Defense, which are being leveraged to ensure the success and relevance of this technology.

IV. PROJECT ACCOMPLISHMENTS AND DOCUMENTATION

A. *Education and Workforce Development Activities*

1. Course, Seminar, and/or Workshop Development
    a. Apart from the graduate and undergraduate research experiences highlighted below, the project has also strived for impact through the classroom, or perhaps more accurately, the virtual classroom. To this end, new educational modules have been developed for the PI's graduate-level course on the Mechanics of MEMS and NEMS, which are related to micro/nanoscale transduction and sensing, and subsequently released via the Purdue NExT online portal: http://purduenext.purdue.edu/. Since being released in early 2015, 25 students across the globe have taken the course (for credit), deepening their skills in this emergent technical area.
2. Student Internship, Job, and/or Research Opportunities
    a. Since its initiation in September 2013, the impact of this project from an education and workforce development perspective has been tangible. Cognizant of the fact that our long-term deliverable is well-trained students with homeland security expertise, the project has incorporated two graduate students and six undergraduate students. To date, one of these graduate students (Dr. Andrew B. Sabater) has graduated. He subsequently assumed a position at the US Navy's Space and Naval Warfare Systems Command (SPAWAR), working in the area of advanced sensor development. Likewise, three of the project's undergraduate students (Lillian Miles, Allison Murray, and Trevor Fleck) have completed their undergraduate studies, and based on their positive undergraduate research experience, have decided to pursue graduate studies at Oregon State University, Purdue University, and Purdue University, respectively. Of note here, all of the remaining undergraduate students have expressed a keen interest in graduate studies, and one (Brittney Scifres) is slated to join Purdue University as a graduate student in the spring of 2017.

B. *Peer Reviewed Journal Articles*

1. N. Bajaj, A. B. Sabater, J. N. Hickey, G. T.-C. Chiu, and J. F. Rhoads. "Design and Implementation of a Tunable, Duffing-Like Electromechanical Resonator via Nonlinear Feedback." Journal of Microelectromechanical Systems, 25(1), pp. 2-10. February 2016. DOI:10.1109/JMEMS.2015.2493447.

Pending –

1. N. Bajaj, J. F. Rhoads, and G. T.-C. Chiu. "Characterization of Resonant Mass Sensors Using Inkjet Deposition." Submitted to the Journal of Dynamical Systems, Measurement, and Control. 2016.

C. Other Presentations

1. Seminars:
   a. J. F. Rhoads. "Impediment or Opportunity: Examining the Role of Complexity and Nonlinearity in Resonant Micro- and Nanosystems." University of Washington, Department of Mechanical Engineering. November 17, 2015. Seattle, Washington.
2. Interviews and/or News Articles:
   a. E. Bender. "Explosive Progress: Interdisciplinary Academic Research is Advancing Our Understanding of Energetic Materials and Our Security." Discovery - Innovation at Purdue Engineering. October 13, 2015.

D. New and Existing Courses Developed and Student Enrollment

New or Existing	Course/Module/ Degree/Cert.	Title	Description	Student Enrollment
Existing	Course	Mechanics of MEMS and NEMS	New online modules on MEMS/NEMS transduction principles and sensing were developed for this course, which was released to select institutions worldwide in 2015 via Purdue University's NExT platform: http://purduenext.purdue.edu/	25 students

E. Technology Transfer/Patents

1. Patent Applications Filed (Including Provisional Patents)
   a. J. F. Rhoads, G. T.-C. Chiu, N. Bajaj, and A. B. Sabater. Nonlinear Mass Sensors Based on Electronic Feedback and Methods of Using the Same. U.S. Patent Application Number US 14973262. December 17, 2015.

F. Requests for Assistance/Advice

1. From Federal/State/Local Government
   a. The project PI (Rhoads) served as an external reviewer for the Science of Signatures research pillar at Los Alamos National Laboratory between May 15-18, 2016. This comprehensive, biennial review spanned the Laboratory's numerous research activities related to signature acquisition, interpretation, and processing for pre- and post-nuclear and/or explosive events. Note that a substantial portion of the reviewed research was funded by the Department of Homeland Security and, in particular, the Domestic Nuclear Detection Office.

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