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

Abstract—This project seeks to develop portable, integrated, microscale sensors (PIMS), founded upon chemomechanically-induced response bifurcations, which are suitable for vapor-phase explosives detection. In prior work by the investigators, these sensing system were shown to yield superior performance metrics (i.e. false positive/negative rates and sensitivity metrics) in laboratory environments within the context of alcohol sensing. In the first period of performance, research efforts focused on the development of a new, low-cost bifurcation-based sensor platform (predicated by the commercial discontinuation of the platform utilized in prior work), the development of a new inkjet functionalization suite capable of rapidly and precisely depositing functional surface layers on the sensors developed herein and the development of new control and signal processing electronics designed to enable portable functionality. These efforts form a solid technical foundation for a second period of performance which will be focused on validating sensor performance with mock and real energetic materials within both laboratory and operational (wherein the impinging fluid flow becomes important) environments; characterizing pertinent sensor metrics and benchmarking these metrics against alternative sensing platforms; exploring and overcoming the basic research challenges associated with sensor integration; and, finally, modeling the complete sensing system with an eye towards predictive design, performance optimization, and, ultimately, technology transition.

I. PARTICIPANTS

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II. PROJECT OVERVIEW AND SIGNIFICANCE

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 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 transportation security 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, a marked change in amplitude (see Fig. 1). 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. 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 compare 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 is 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 mock and real energetic materials within both laboratory and opera-
tional (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; and

- Modeling the complete sensing system with an eye towards predictive design, performance optimization, and, ultimately, technology transition.

III. RESEARCH ACTIVITY

A. 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]. 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 formfactor metrics by eliminating the need to employ frequency tracking hardware, which is often attendant to conventional microscale mass sensor designs [1, 2, 14-17].

The present effort seeks to build upon the later body of research by developing portable, integrated microscale sensors (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 described in [17], 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 platforms capable of detection in operational environments, rather than solely performance metric enhancement; and (3) developing a scalable sensing solution which suitable for technology transfer.

B. Major contributions

In the first period of performance, research efforts focused on three key fronts: (1) the development of a new, low-cost bifurcation-based sensor platform; (2) the development of a new inkjet functionalization suite capable of rapidly and precisely depositing functional surface layers on the sensors developed herein; and (3) the development of new control and signal processing electronics designed to enable portable functionality. Research contributions made in each of these respective areas are outlined below.

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 because the commercial platform originally intended for repurposing and use, the Veeco DMASP probe shown in Figure 1, was discontinued by the company shortly before the start of the initial performance period. Fortunately, with challenge comes opportunity, and this temporary barrier allowed the investigators to explore and, ultimately, select a new resonator platform, which compares 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, depicted in Figure 2, is 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 (see Fig. 3) are used to signal a detection event. For purposes of this effort, the investigators explored the frequency response characteristics of representative resonators (see Fig. 3 and Fig. 4 on the next page), and determined that the torsional mode of vibration depicted in Figure 4d on the next page most frequently exhibits 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 (see Fig. 5 on the next page). Unfortunately, this nonlinear response was found to vary appreciable from resonator to resonator—a likely by-product of the fact that these devices, as originally conceived, are designed to operate within a linear response regime.

To overcome the device-to-device variability in the systems' nonlinear response and ensure that any randomly-selected resonator would exhibit the desired frequency response structure, the investigators developed a simple control strategy, dubbed feedback nonlinearization (see Fig. 6 on the next page). The antithesis of a feedback linearization system [18], this controller ensured that any quartz resonator, designed to exhibit a linear frequency response structure exhibits the nonlinear frequency response structure required for bifurcation-based sensing. The effectiveness of this novel control approach is clear from the results presented in Figure 7, which highlights the frequency response of five randomly-selected resonators. As evident, each resonator exhibits the same qualitative response, with quantitative variation resulting solely from how the resonator enclosure, or can, was opened to the environment, as required for sensing.

Figure 2: A second-generation bifurcation-based sensor – a repurposed quartz tuning fork which is driven into a nonlinear response regime.

Figure 3: Frequency response of the Kyocera ST3215SB tuning fork excited by a comparatively-low (3 V) pseudorandom excitation signal. Note the multiple clear resonances are exhibited.

Figure 4d: Torsional mode of vibration depicted in Figure 4d on the next page.
Figure 4: Operational deflection shapes (ODSs) associated with the first four resonances of the tuning fork: (a) 22.05 kHz, (b) 32.73 kHz, (c) 162.0 kHz, and (d) 187.6 kHz. These operational deflection shapes were recovered via microscale laser Doppler vibrometry. The ODS depicted in (d) was deemed to be the most likely to be associated with a nonlinear behavior.

Figure 5: Frequency response of a representative tuning fork excited near its fourth natural frequency. Though the nonlinear response exhibited here is amenable to bifurcation-based sensing, this behavior was found to be quite variable between devices. This prompted the feedback nonlinearization strategy outlined herein.

Figure 6: Schematic of the feedback nonlinearization control scheme utilized to recover a suitable nonlinear response in arbitrarily selected tuning fork resonators or “crystals”.
With the functional backbone of the portable, integrated microscale sensor in hand, research efforts shifted towards the development of signal processing electronics suitable for sensor interrogation. Though still in a developmental stage, the first-generation control electronics, depicted in Figure 8, have been shown to successfully and rapidly recover the sensor's response – a necessary precursor to eventual sensor integration.

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 explosive vapors. In the present effort, the investigators are leveraging known surface chemistries, based on functional polymers, for explosives detection. As such, the technical challenge lies not in adsorption or adhesion chemistry, but rather material deposition.

To facilitate precise and rapid material deposition, the investigators have fabricated, tested and implemented a customized inkjet functionalization system (see Fig. 9 on the next page). This system, consisting of a 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 on the next page). As shown in Figure 10 on the next page, 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.
C. Future Plans

As noted above, the first year of the present effort focused on the development of a new, low-cost bifurcation-based sensor platform, the development of a new inkjet functionalization suite capable of rapidly and precisely depositing functional surface layers on the sensors developed herein, and the development of new control and signal processing electronics designed to enable portable functionality. With these efforts largely complete, the research focus can shift to the other hurdles which must be overcome to advance the development of portable, integrated microscale sensors and, ultimately, enable technology transfer. To this end, the next year of the proposed effort will focus on:

1. Validating sensor performance with mock and real energetic materials within both laboratory and operational (wherein the impinging fluid flow becomes important) environments;

2. Characterizing pertinent sensor metrics (e.g. false positive/negative rates, sensitivities, power consumption, etc.) and benchmarking these metrics against alternate sensing platforms;

3. Overcoming the basic research challenges associated with integrating all of the sensing system’s constituent pieces in a single, portable platform; and

Figure 9: The investigators’ customized inkjet functionalization system fabricated as part of this research effort. Key metrics of this system are highlighted in Table 1.

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<thead>
<tr>
<th>Parameter Name</th>
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<td>Registration Camera Resolution</td>
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Table 1: Performance metrics of the inkjet functionalization system developed as part of this effort.

Figure 10: A bifurcation-based sensor functionalized with black ink via the functionalization system developed as part of this effort.

Figure 11: Measurement Camera 1 and Inkjet Nozzle
4. Modeling the complete sensing system with an eye towards predictive design and performance optimization.

To accomplish the first two tasks, the investigators will work hand-in-hand with other ALERT researchers to select appropriate surface chemistries, and then leverage previous experimental protocols developed for vapor-phase alcohol sensing to validate sensor performance and develop quantitative performance metrics. Integration and modeling will present a more open-ended challenge, but are necessary to ensure the evolution of portable, integrated microscale sensors from laboratory to practice.

IV. EDUCATION & WORKFORCE DEVELOPMENT ACTIVITY

The impact of this project to date, from an education and workforce development perspective, has been limited to the two graduate research assistants who have worked on the project. In the upcoming year, this impact is expected to markedly increase as additional students join the effort, and the underlying research problem is introduced into existing curricula (e.g. the investigator's course on micro- and nanoelectromechanical systems – MEMS and NEMS, which contains an extended module on microscale sensors).

V. 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 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 transportation security 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 to their more conventional counterparts.

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. Anticipated end-user technology transfer

Portable, integrated microscale sensors (PIMS) may be amenable to a wide variety of public and private organizations, including those tasked with transportation portal security (e.g. the TSA), building security, and field-based detection (e.g. the ATF).

VI. LEVERAGING OF RESOURCES

To date, no commercial ventures related to this technology are being pursued. A closely related technology, which could expand upon the work described here, was recently included in a proposal to TSWG.
VII. REFERENCES


