The Xsense project: The application of an intelligent sensor array for high sensitivity handheld explosives detectors

Kostesha, Natalie; Schmidt, Michael Stenbæk; Bosco, Filippo; Olsen, Jesper Kenneth; Johnsen, Carsten; Nielsen, Kent A; Jeppesen, Jan Oskar; Alstrøm, Tommy Sonne; Larsen, Jan; Thundat, Thomas

Total number of authors:
12

Published in:
Proceedings of IEEE Sensors Applications Symposium

Link to article, DOI:
[10.1109/SAS.2011.5739782](https://doi.org/10.1109/SAS.2011.5739782)

Publication date:
2011

Document Version
Publisher's PDF, also known as Version of record

[Link back to DTU Orbit](#)

Citation (APA):
Kostesha, N., Schmidt, M. S., Bosco, F., Olsen, J. K., Johnsen, C., Nielsen, K. A., Jeppesen, J. O., Alstrøm, T. S., Larsen, J., Thundat, T., Jakobsen, M. H., & Boisen, A. (2011). The Xsense project: The application of an intelligent sensor array for high sensitivity handheld explosives detectors. In *Proceedings of IEEE Sensors Applications Symposium* IEEE. <https://doi.org/10.1109/SAS.2011.5739782>

General rights

Copyright and moral rights for the publications made accessible in the public portal are retained by the authors and/or other copyright owners and it is a condition of accessing publications that users recognise and abide by the legal requirements associated with these rights.

- Users may download and print one copy of any publication from the public portal for the purpose of private study or research.
- You may not further distribute the material or use it for any profit-making activity or commercial gain
- You may freely distribute the URL identifying the publication in the public portal

If you believe that this document breaches copyright please contact us providing details, and we will remove access to the work immediately and investigate your claim.

The Xsense Project: The Application of an Intelligent Sensor Array for High Sensitivity Handheld Explosives Detectors

Natalie Kotesha¹, Michael Stenbæk Schmidt¹, Filippo Bosco¹, Jesper Kenneth Olsen¹, Carsten Johnsen², Kent A. Nielsen², Jan Oskar Jeppesen², Tommy Sonne Alstrøm³, Jan Larsen³, Thomas Thundat⁴, Mogens Havsteen Jakobsen¹ and Anja Boisen¹

¹Department of Micro- and Nanotechnology, Technical University of Denmark, Ørsted's Plads, Building 345east, DK-2800 Kgs. Lyngby, Denmark

²Department of Chemistry, University of Southern Denmark, Campusvej 55, DK-5230 Odense M, Denmark

³Department of Informatics and Mathematical Engineering, Technical University of Denmark, Building 321, DK-2800 Kgs. Lyngby, Denmark

⁴Department of Chemical and Materials Engineering, University of Alberta, 9107 - 116 Street Edmonton, AB T6G 2V4

Email: natalie.kotesha@nanotech.dtu.dk

Abstract-Multiple independent sensors are used in security and military applications in order to increase sensitivity, selectivity and data reliability. The Xsense project has been initiated at the Technical University of Denmark in collaboration with a number of partners in an effort to produce a handheld sensor for trace detection of explosives. We are using micro- and nano technological approaches for integrating four sensing principles into a single device. At the end of the project, the consortium aims at having delivered a sensor platform consisting of four independent detector principles capable of identifying concentrations of TNT, DNT, HMX and RDX at sub parts-per-billion (ppb) levels and with a false positive rate less than 1 parts-per-thousand. The specificity, sensitivity, reliability and the speed of responses are ensured by the use of advanced data processing, surface functionalization and nanostructured sensors and sensor design.

Keywords- Xsense; explosives detection; surface enhanced Raman scattering spectroscopy; colorimetry; calorimetry; cantilever-based sensor; DNT; TATP; RDX; HMX; data fusion

I. INTRODUCTION

The detection of hidden explosives is a major challenge which requires fast, portable, reliable, selective and sensitive detection methods for explosives and explosive related illicit materials. Major areas of applications for explosives sensors include: anti-terrorism (screening luggage, mail packages, checking suspects and mass transit systems), demining (clearance of mines and unexploded ordnance (UXO) in current or former war zones) and in the field of environmental monitoring of hazardous compounds. For example trinitrotoluene

(TNT) can easily enter the groundwater and is classified as toxic to all life forms in concentrations above 2 ng/L.

Today, hidden explosives are mainly detected by trained dogs. Dogs are capable of detecting TNT, nitroglycerin-based dynamites, plastic explosives in concentrations as low as parts per trillion (ppt). However, the sniffing dog has some limitations; negative influences of environmental distractions - olfactory, audio, visual can disrupt the work and provoke unpredicted behavior. Also, dogs are expensive to train and maintain, and can only work for a few hours at a time. Furthermore, dogs require an equally skilled handler whose training is also expensive and time consuming. Furthermore, dogs often need retraining and/or time for acclimatization and certification when moved to a new environment. Dog's responses are not selective, since animals can only indicate the presence or absence of explosives.

In an effort to address some of these issues the Danish Agency for Science and Technology's, Program Commission on Nanoscience Biotechnology and IT (NABIIT) has issued a € 3.850.000 grant to fund the Xsense [1] project which will be presented here. Xsense is projected to run for four years from mid 2008 to 2012. Four Ph.D. students, three post doctoral researchers in addition to four tenured professors distributed between the Technical University of Denmark and the University of Southern Denmark participate in the ongoing research. A close working relationship with the industrial partners Unisensor and Serstech AB has been secured from the project onset and these partners contribute with equipment and knowhow. The industrial goal is to provide Danish industry with

new ideas for products and to assist them in further developing their current technologies in the sensor area. Furthermore, it is anticipated that new business ideas will emerge from the project, creating a technology platform for start-up companies. The ultimate goal of the Xsense project is based on the development of a reliable, sensitive, portable and low-cost explosives detector, as well as sensor engineering and its validation will be performed. The sensor system will be miniaturized, hence portable, and will therefore be highly suitable for the use in anti-terror efforts, border control, environmental monitoring and demining. At the end of the project, the consortium aims at having delivered a sensor platform consisting of four independent detector principles capable of detecting concentrations of TNT and other explosives at sub parts-per-billion (ppb) concentrations and with very infrequent false alarms. The inherent design qualities of the finished device should enable its use by personnel with minimal training thus giving these persons explosives detection capabilities otherwise only available to trained dog teams.

II. SENSOR TECHNOLOGIES

An intelligent array of sensors tends [2] to be very good at identifying a broad range of explosives, mixture of explosives and related compounds which are necessary for explosives manufacturing. Our hypothesis is that only by merging several independent and sensitive measuring principles can reliability be improved. The basic scientific goal of Xsense is to focus on the development of miniaturized sensors in order to achieve the detection limit towards explosives (TNT is the major test molecule) of 1 ppb. The data reliability is being addressed by a concentrated effort in data processing (reducing noise and pattern recognition). The improvement in sensitivity will be performed by surface functionalization and design. Secondly, the goal is to improve the reliability of the explosives detection significantly by establishing a network of the independent sensors. Hereby, a false positive rate less than 1 parts-per-thousand is sought. In the Xsense project we are working towards the development and improvement of four individual miniaturized sensor technologies. Technologies include: surface enhanced Raman scattering (SERS) spectroscopy, cantilever-based sensors, a calorimetric sensor and a colorimetric sensor which are each capable simultaneously to detect explosives in concentrations of at least 1 ppm. Proof of concept has been demonstrated for all four sensor technologies hence the ongoing effort will be in optimizing the sensitivity. The project will allow us to compare the

technologies in terms of sensitivity, reliability, response time, ease of use and cost. Based on the analysis of the individual sensors and on input from the data processing activities a sensor network will be established. The idea is that the amount of false alarms can be significantly reduced by increasing the number of independent sensing methods. The individual sensors as well as the network of sensors will be used for detection of explosives and related compounds in air. Also, mixtures of explosives and influence of changes in explosive concentrations, humidity and temperature will be analysed.

A. Surface Enhanced Raman Scattering

In Raman spectroscopy it is possible to identify a molecule by mapping its vibrational states. The molecule of interest is illuminated with laser light, which is scattered by the molecule. Part of the scattered laser light has been modified by the vibrational states of the molecule i.e. has undergone a shift in frequency. The substance can be identified by collecting the scattered laser light and analyzing the various shifts in frequency originating from the different vibrational states. We have developed a maskless fabrication process [3] shown schematically in Fig. 1 in order to generate Raman enhancing surfaces. A silicon wafer is etched by reactive ion etching. The etch process is tuned such that it results in micromasking of the surface which is then used to define small and highly reproducible nano-pillars on a full wafer surface.

Figure 1. Schematic of the fabrication process. a) A blank silicon wafer is structured by maskless reactive ion etching to form free standing nano-pillars. b) The pillars are coated with metal c) The resulting structure are free standing metal coated nano-pillars with rough surfaces.

Recently, nano-structured surfaces have been developed that enhance the Raman cross section of explosives molecules by 9 orders of magnitude [3] (Fig. 4). The signal enhancement allows for a miniaturized detection system using chip-based micro spectrometers (supplied by Serstech AB) and cost efficient CCD elements.

Figure 2. Scanning electron microscope image of nanostructured silicon surface optimised for enhancing the Raman effect. The silicon surfaces are coated with 50-400 nm thick layers of silver / gold. Note the large surface area.

B. Cantilever based sensing

Micro-cantilever sensors with integrated piezoresistive or optical read-out are capable of detecting extremely small changes in surface stress and offer promising opportunities for developing miniaturized sensors for explosive vapors. To enable chemical selectivity, the top surface of a micro-cantilever is modified with a selective layer which binds specific explosives. Upon binding of the molecules, the cantilever bends and the bending is detected electrically as a change in the resistance of the integrated piezoresistor or optically as a deflection of a laser light hitting the apex of the cantilever. Sensitive and selective sensors can detect trace amounts of explosives in real-time, as demonstrated for example with PETN, RDX [4] and TNT [5]. Recently, polymer cantilevers fabricated at DTU Nanotech were used for the detection of the nerve gas model DMMP [6]. Our challenge is to find selective coating materials in order to bind the explosives and to minimize unspecific binding. Also, the readout method needs to be robust and fast. Currently we are investigating cantilevers for optical read-out and methods of integrating the cantilevers[7], colorimetric and SERS sensors into the same solid support structure. An example of an array of silicon cantilevers is shown in Fig. 3. The cantilever bending will be read-out using the pick-up head from a DVD [8].

Figure 3. Top view of microcantilever sensors array

C. Micro-calorimetry

The micro-calorimetric approach can be used to determine phase transitions, and fast exothermic chemical reactions characteristic for explosives [9]. A heater element in the micro-calorimetric sensor is placed on a micrometer sized bridge. The bridge furthermore contains a temperature sensitive resistor [10]. In this way it is possible to perform calorimetry on trace particles. Using rapid heating ramps it is possible to deflagrate explosive particles (exothermic reaction) causing a significant heat development. Explosives molecules that have been detected by the micro-calorimetric sensor include TNT, PETN and RDX [11]. Fig. 4 shows an example of a schematic drawing of the chip as well as an example of a DNT coated bridge.

In this method the explosives are passively adsorbed on the surface before heating is initiated. During a rapid heating ramp all explosives will start to melt, evaporate and finally deflagrate. This is recorded using the built in temperature resistor and the resulting temperature profile will give a unique signature for different explosives and non explosives. This work is in collaboration with researchers from Oakridge National Laboratory, USA.

Figure 4. a) Graphic illustration of the sensing principle. Explosives attached to the micrometer sized bridge structure will deflagrate as the temperature of the bridge is rapidly increased using integrated heater elements. The resulting temperature increase as a function of time is used to map the presence of different explosives. b) Optical microscope image (top view) of bridge structure with adsorbed DNT on top. The DNT forms a uniform coating of small droplets.

D. Colorimetric array

The fourth sensor principle for the detection of explosives is colorimetric sensing technology. It has been already shown that colorimetric sensors are highly sensitive, fast in response and low in cost; colorimetric sensors demonstrated the ability to detect traces of molecules with performance levels similar to that of trained canines [12-15].

We have discovered a new class of chemical compounds that can distinguish between closely related compounds (methanol, ethanol and propanol), explosives and other classes of compounds [13]-[16] (patent pending). The colorimetric sensing technique

is based on an array of chemo-selective compounds immobilized on a solid support. Upon exposure to an analyte in gas phase or in the liquid phase each compound - or dye - changes color. Each dye is chosen as to react selectively towards various analytes or analyte mixtures. This change of color is based on non-covalent host-guest interaction and redox interaction between the dye (host) and analyte (guest) [17, 18]. A change in a color signature indicates the presence of explosives (Fig. 5) and VOCs [13]. By creating a color difference map it is possible to create a unique fingerprint for each compound of the interest. The difference map is generated after a mathematical analysis of the color changes. A difference map presents the difference in absolute value of RGB colors obtained from the absolute value of red, green or blue color after the analyte exposure minus the absolute value of red, green or blue color before the analyte exposure.

Figure 5. A difference map of the colorimetric sensor array obtained before and after the exposure of DNT in the gas phase. The image was generated after the mathematical analysis of the color changes.

A simple colorimetric sensor array can be used for detection and identification of explosives like DNT, HMX, RDX and TATP (Fig. 6). Furthermore colorimetric arrays have been shown to be able to detect illegal drugs or drug precursors, volatile organic compounds (VOCs) at different humidity and various temperature conditions. This sensor array is inexpensive, and can potentially be produced as single use disposables.

Figure 6: A difference map of the colorimetric sensor array obtained before and after the exposure of RDX, DNT, HMX and TATP in the gas phase (experiments were performed at elevated temperature 100° C). The image was generated after the mathematical analysis of the color changes. Control experiments were performed at room temperature; Control experiment-100° C was performed without exposure of explosives at elevated temperature 100° C.

E. Signal processing

Data processing in the field of explosives is the most challenging part of the survey and requires careful data analysis and interpretation. To obtain reliable and robust detection and quantification of explosives requires optimization of all steps in the data processing pipeline. The data processing pipeline includes: 1) Pre-processing: preparation/conditioning, noise removal and feature extraction of individual sensor data. 2) Data fusion: learning the optimal nonlinear combination of pre-processed data with the aim of predicting presence of explosives. The learning phase is done from sensor data sets assisted by reference measurements of explosive concentration. 3) Data presentation and evaluation: quantification of system's decision, uncertainty, robustness and reliability. We have extensive experience with such pipelines in diverse areas such as neuro-imaging [19] skin cancer detection from Raman spectroscopy [20] and land mine detection [21].

CONCLUSION

In light of the sensory systems presented herein, the Xsense project aims at introducing micro- and nanotechnological approaches to the field of explosives detection. The concept is based on the development of a gas sensor by combining four sensor techniques in a hand held device coordinated by advanced mathematical signal processing. We believe that this is a promising approach for

explosives detection with low false alarms to be used in for example landmine clearance operations. A fully developed sensor system could be successfully applied in airports, by police, military and border control bases who all demand detection systems with fast, simultaneous and efficient detection of explosives.

ACKNOWLEDGEMENTS

Danish Agency for Science and Technology's, Program Commission on Nanoscience Biotechnology and IT (NABIT) for funding. Case number: 2106-07-0031 – Miniaturised sensors for explosives detection in air.

REFERENCES

1. <http://www.xsense.dk/>.
2. Senesac, L. and T.G. Thundat, "Nanosensors for trace explosive detection". *Materials Today*, vol. 11, pp. 28-36, 2008.
3. Schmidt, M.S., A. Boisen, and J. Hubner, "Towards Easily Reproducible Nano-structured SERS Substrates", in *2009 IEEE Sensors, Vols 1-3*, pp. 1685-1689, 2009.
4. Pinnaduwa, L.A., "Sensitive detection of plastic explosives with self-assembled monolayer-coated microcantilever", *Applied Physics Letters*, vol. 83, pp. 1471-1473, 2003.
5. Pinnaduwa, L.A., "Detection of trinitrotoluene via deflagration on a microcantilever", *Journal of Applied Physics*, vol. 95, pp. 5871-5875, 2004.
6. Pinnaduwa, L.A., "Miniature sensor suitable for electronic nose applications", *Review of Scientific Instruments*, vol. 78, p. 4, 2007.
7. Bosco, F.G., "Self-aligned cantilever positioning for on-substrate measurements using DVD pickup head", *Microelectronic Engineering*, vol. 87, pp. 708-711, Sep. 2009.
8. Hwu, E.T., "Simultaneous detection of translational and angular displacements of micromachined elements", *Applied Physics Letters*, vol. 91, pp. 3, 2007.
9. Senesac, L.R., "Micro-differential thermal analysis detection of adsorbed explosive molecules using microfabricated bridges", *Review of Scientific Instruments*, vol. 80, Mar. 2009.
10. Greve, A., "Micro-calorimetric sensor for vapor phase explosive detection with optimized heat profile", *Microelectronic Engineering*, vol. 87, pp. 696-698, 2010.
11. Yi, D.C., "Detection of adsorbed explosive molecules using thermal response of suspended microfabricated bridge", *Applied Physics Letters*, vol. 93, p. 3, 2008.
12. Feng, L., "A colorimetric sensor array for identification of toxic gases below permissible exposure limits", *Chemical Communications*, vol. 46, pp. 2037-2039, 2010.
13. Kostasheva, N.V., "Development of the colorimetric sensor array for detection of explosives and volatile organic compounds in air", *Proc. SPIE*, vol. 7673, doi: 10.1117/12.850219, 2010.
14. Rakow, N.A. and K.S. Suslick, "A colorimetric sensor array for odour visualization", *Nature*, vol. 406, pp. 710-713, 2000.
15. Suslick, B.A., L. Feng, and K.S. Suslick, "Discrimination of Complex Mixtures by a Colorimetric Sensor Array: Coffee Aromas", *Analytical Chemistry*, vol. 82, pp. 2067-2073, 2010.
16. Jakobsen, M.H., "Sensor array for detection of volatile compounds in air", 2010. Patent is pending.
17. Nielsen, K.A., "Tetra-TTF calix[4]pyrrole: A rationally designed receptor for electron-deficient neutral guests", *Journal of the American Chemical Society*, vol. 126, pp. 16296-16297, 2004.
18. Nielsen, K.A., "Binding studies of tetrathiafulvalene-calix[4]pyrroles with electron-deficient guests", *Tetrahedron*, vol. 64, pp. 8449-8463, 2008.
19. Strother, S., "Optimizing the fMRI data-processing pipeline using prediction and reproducibility performance metrics: I. A preliminary group analysis", *Neuroimage*, vol. 23, pp. S196-S207, 2004.
20. Sigurdsson, S., "Detection of skin cancer by classification of Raman spectra", *IEEE Transactions on Biomedical Engineering*, vol. 51, pp. 1784-1793, 2004.
21. Larsen, J., DeFuse - Demining by Fusion of Techniques, Nordic Demining Research Forum research project with the objective of investigating the improvement of the mine clearance operation by combining several existing methods, in *NDRF Summer Conference*. Scientific Foundations of the Demining by Fusion of Techniques Project: Varde, Denmark, 2007.