# Xsense - a miniaturised multi-sensor platform for explosives detection

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## ABSTRACT

Realizing that no one sensing principle is perfect we set out to combine four fundamentally different sensing principles into one device. The reasoning is that each sensor will complement the others and provide redundancy under various environmental conditions. As each sensor can be fabricated using microfabrication the inherent advantages associated with MEMS technologies such as low fabrication costs and small device size allows us to integrate the four sensors into one portable device at a low cost.

**Keywords:** Micro cantilever, Micro calorimetry, Surface Enhanced Raman spectroscopy, Colorimetric arrays, Data fusion.

## INTRODUCTION

In an attempt to develop a handheld explosives sensor, the Danish Agency for Science and Technology's, Program Committee on Nanoscience, Biotechnology and IT (NABIIT) has issued a €3.850.000 grant to fund the Xsense project [1]. Xsense started in 2008 and will culminate in 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 A/S and Serstech AB has been secured from the project onset and these partners contribute with equipment and knowhow. The ultimate goal of the Xsense project is based on the development of a reliable, sensitive, portable and low-cost explosives detector. The sensor system will be miniaturized and will therefore be highly suitable for the use in anti-terror efforts, border control and de-mining. At the end of the project, the consortium aims at delivering 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 a minimum of 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.

### 1.1 SENSOR TECHNOLOGIES

Our hypothesis is that only by merging several independent and sensitive measuring principles can reliability be ensured. 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. Secondly, the goal is to improve the reliability of the explosives detection significantly by establishing a network of four independent sensors. In the Xsense project we work towards the development and improvement of four individual miniaturized sensor technologies. The technologies include: cantilever-based sensors, a calorimetric sensor, surface enhanced Raman spectroscopy and a colorimetric sensor which each are capable of simultaneously detecting explosives in trace concentrations. Proof of concept has been confirmed for all four sensor technologies hence the ongoing effort is thus in optimizing the sensitivity and integrating all the sensors into one device. In the following a brief description of each sensing principle will be given.

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### 1.2 Microcantilever based sensing

Microcantilever sensors with optical read-out are capable of detecting extremely small changes in surface stress and offer promising opportunity for developing miniaturized sensors for explosive vapors. To increase the chemical selectivity, the top surface of a microcantilever is often modified with a selective layer which binds specific explosives. Upon binding of the molecules, the cantilever experiences a chance in surface stress and bends. The bending is detected optically as a deflection of laser light hitting the apex of the cantilever. Sensitive and selective sensors can detect trace amounts of explosives in real-time and the principle has been demonstrated to be able to detect for example PETN, RDX [2] and TNT [3]. Recently, polymer cantilevers fabricated at DTU Nanotech were used for the detection of the nerve gas model DMMP [4]. Our challenge is to find selective coating materials in order to bind the explosives and to minimize unspecific binding. Currently we have developed a readout method which is both robust and fast based on a novel DVD-ROM-like optical system [5].

In our novel system, hundreds of cantilevers can be placed onto a platform fabricated with photolithography in SU-8 on a glass wafer. The disk-like platform is spun at different angular velocities while a DVD-ROM pickup head (placed 1 mm below the glass) scans the cantilevers and measures vertical displacement using the DVD-ROM 4-quadrant photodetector [6,7].

Currently, we employ silicon cantilevers 500 µm long, 100 µm wide and 1 µm thick, coated with 20 nm Au layers that allow the laser beam to be reflected to the detection system. Au coating also allows thiol-based monolayer functionalization to be easily performed. Figure 1 shows a SEM image of the silicon cantilevers.

The profile acquisition of the cantilevers is performed scanning the laser path orthogonally to the cantilever beam direction at constant velocity. The profiles can be acquired scanning the laser at different velocities, where measurements up to 100 cantilevers per second have been proved. Each cantilever profile (Figure 1) is composed by around 1000 points, meaning that vertical displacement is measured every 100 nm along the width of the cantilever. Statistical analysis of the acquired bending profiles highly increases the reliability of the measurements.

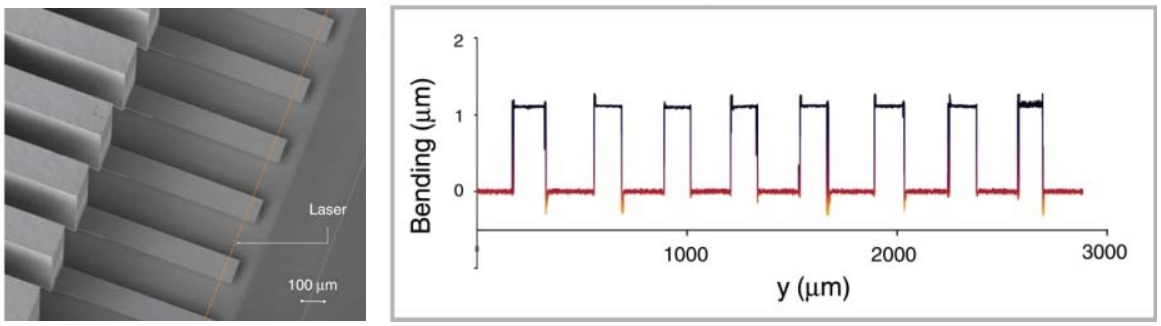


Figure 1: Left: SEM picture of the Au coated silicon cantilever array. Right: Single chip profile, consisting of around 1000 points for each of the eight cantilevers.

### 1.3 Microcalorimetry

The microcalorimetric approach is attractive for applications requiring the determination of the heat from chemical reactions or physical changes. As such it is ideal for explosives detection. In the Xsense project a micro differential total analysis system is being developed using similar principles as reported in [3,8-10]. However the concept being employed here differs by using a suspended membrane design, instead of using a cantilever or bridge design. Compared to other suspended membrane heaters such as [11] this membrane is thinner and has a larger surface area. The suspended silicon nitride membranes are 200 nm thick and have a 500x500 $\mu m^2$ surface area. The small membrane thickness ensures a low thermal mass, facilitating rapid heating and high sensitivity, while providing a large area for adsorption of molecules. To minimize heat loss by diffusion to the substrate, the membrane is suspended by a 50 µm wide beam at each corner. The heating and sensing resistors are identical and designed as spirals within each other as seen in Figure 2. This allows for a uniform heat distribution and temperature sensing across the entire membrane. The resistors are designed with thin vias

connected through the suspensions to dissipate more heat where the diffusion loss is predominant. Each resistor is connected with four vias to enable four-terminal sensing. By rapid heating of the structure, deflagration of explosives might be initiated. Deflagration will cause a significant heat development. Figure 2, shows an example of a schematic drawing of the chip as well as an example of a membrane onto which DNT has been deposited. Also, catalysts might be placed on the heater element to cause a stronger deflagration upon rapid heating.

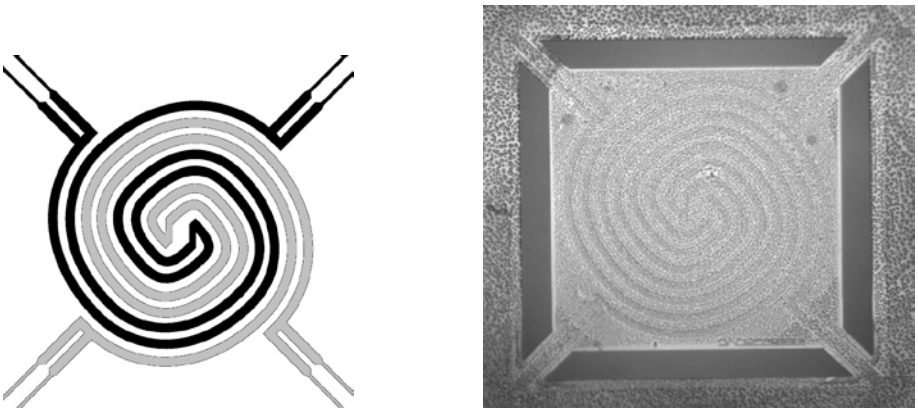

Figure 2: left: The silicon resistors are designed as two spirals inside each other with small vias to the connecting substrate. The small vias dissipate extra heat at the suspensions to the substrate compensating for diffusion losses. The spiral dissipates heat evenly across the membrane for a uniform temperature distribution. Right: Optical microscope picture of a membrane covered with 48 ng of TNT.

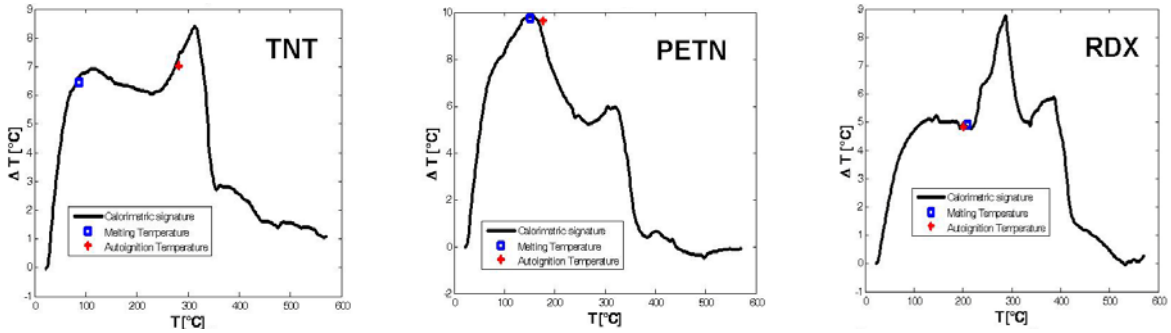


Figure 3: Calorimetric signature of respectively TNT, RDX and PETN. The melting temperature and the auto-ignition temperature is shown on the graph.

The system always utilizes two membranes on two individual chips. One membrane is used as a reference and one holds the measurement sample. Due to the small scale, rapid heating and fast analysis times are possible. Fast temperature ramping is also a requirement to reach deflagration temperature before the sample is evaporated. The system is capable of analyzing and detecting trace amounts of explosive compounds. Calorimetric signatures for TNT, RDX and PETN were obtained and were easily distinguishable from each other, Figure 3. Furthermore, it has been shown that as little as 12ng of explosives results in a calorimetric signature that can be used to distinguish the type of explosives.

### 1.4 Surface Enhanced Raman Spectroscopy

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 undergoes inelastic scattering when interacting with the molecule i.e. the light undergoes 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 two step fabrication process which generates Raman enhancing surfaces [12]. A silicon wafer is etched by maskless reactive ion etching. The etch process is tuned such that it results in

the formation of highly reproducible nanopillars on a full wafer surface. Hereafter the silicon nanopillars are coated with silver or gold rendering the surfaces highly Raman active. Explosives molecules adsorbed to this surface will give rise to a very large Raman signal. The large signal enhancement allows for a miniaturized detection system using chip-based micro spectrometers supplied by Serstech AB.

Examples of Raman spectra of DNT obtained on the nanostructured silicon surfaces with silver coatings are shown in Figure 4, with the results obtained on a commercially available SERS substrate [13] overlain for comparison. It is seen that the signal measured on the silver coated silicon nanopillars display an increase in Raman signal two orders of magnitude larger than the commercial substrate. Gold covered silicon nanopillars also display two orders of magnitude better Raman enhancement (not shown here) than the commercial substrate.

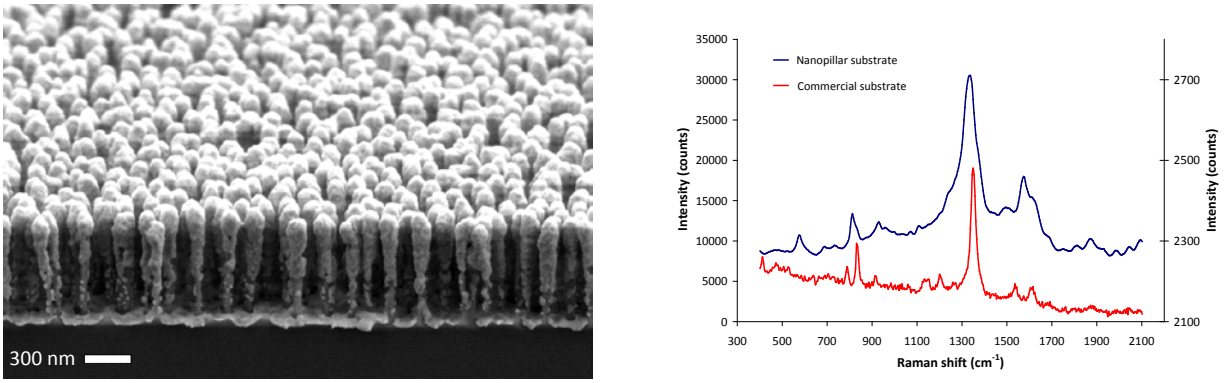


Figure 4: Left: Scanning electron microscope image of nanostructured silicon surface optimized for enhancing the Raman effect. The silicon surfaces are coated with thin film layers of silver / gold. Note the large surface area. Right: Raman spectra (785 nm excitation, 1s acquisition) of DNT evaporated onto the nanopillars substrate with a commercial substrate inserted as comparison. The nanopillars substrate has a two orders of magnitude stronger signal. Note the two different y-axis.

### 1.5 Colorimetric Arrays

The final sensor for detecting explosives is based on the application of colorimetric sensing technique. It has been shown that colorimetric sensing technology is highly sensitive, fast in response and low in cost. Colorimetric sensors have demonstrated the ability to detect trace levels of molecules with performance levels similar to that of trained canines [14-16]. We have already shown that a new class of chemo-selective compounds can distinguish between closely related compounds (organic and inorganic acids), alcohols, explosives and other classes of compounds [14, 17-19].

The colorimetric sensing technique is based on an array of chemo-selective compounds immobilized on a solid support, such as a membranes or microstructure. Upon exposure to an analyte in gas the phase or in the liquid phase each chemo-selective compound - or dye - changes color. Each of the selected dyes reacts 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) [18, 19]. Changes in a color signature indicate the presence of explosives and volatile organic compounds VOCs [14]. 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 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.

A simple colorimetric sensor array can be used for detection and identification of explosives like DNT, HMX, RDX and TATP as shown in Figure 5. Furthermore, the colorimetric sensor has been shown the capability to detect illegal drugs or drug precursors, VOCs at different conditions: humidity and temperature.

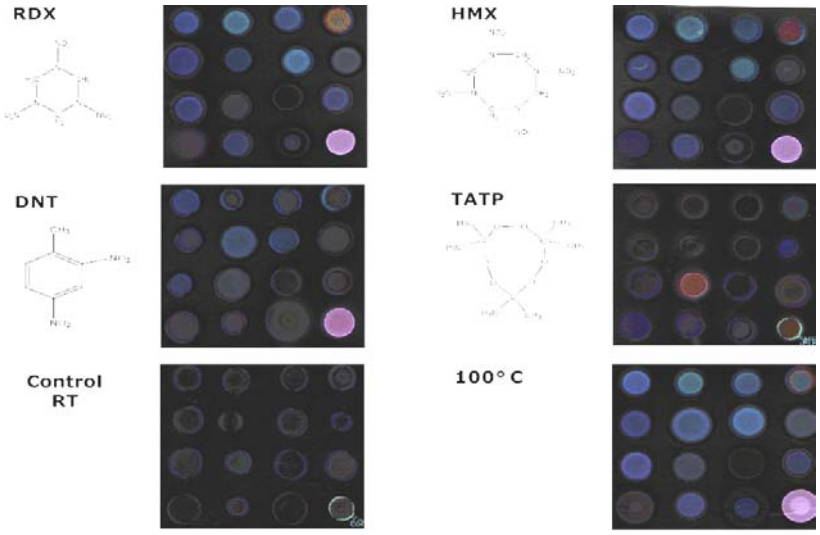


Figure 5. Difference maps of the colorimetric sensor array obtained in the presence of DNT, RDX, HMX, and TATP at elevated temperature 100 °C. Images were generated after the mathematical calculations of the color changes; and presented as the difference map obtained from the absolute values of RGB values of each dye spot before and after the exposure of targets.

## 1.6 DATA PROCESSING

The objective of data processing is reliable and robust detection and quantification of explosives and requires careful optimization of all steps in the data processing pipeline. The data processing pipeline includes: *Pre-processing*, *Data fusion*, and *Data presentation and evaluation*. Pre-processing involves preparation/conditioning, noise removal and feature extraction of individual sensor data. A significant part is to establish knowledge of sensor response from careful statistical design of experiments. The goal is to enhance sensor sensitivity as well as robustness to environment conditions using response surface methodology. Early data fusion includes learning the optimal nonlinear combination of pre-processed and aligned data with the aim of predicting presence of explosives. An alternative strategy is late or decision fusion where sensors are processed individually and their decisions are later combined. One might hypothesize that an optimal strategy is a mix of these approaches. The learning phase is done from sensor data sets assisted by reference measurements of explosive expressions; hence, this step involves considerable experimentation as well. Data presentation and evaluation concerns the quantification of various - often conflicting properties - of the overall system with respect to decision capability, uncertainty, robustness and reliability.

We have extensive previous experience with such pipelines in such diverse areas as neuroimaging [20], skin cancer detection from Raman spectroscopy [21], and land mine detection [22]. Within the Xsense project data processing has been applied to colorimetric data [14], and polymer coated Quartz Crystal Microbalance (QCM) data [23], which is of similar nature to cantilever based data. In colorimetric data analysis we invented a new color difference map representation. This representation has been evaluated for classification of various analytes [14]. A number of supervised (e.g. artificial neural network and Gaussian processes) and unsupervised learning (principal component analysis) techniques have been investigated for analysis of QCM data [23].

## 1.7 CONCLUSION

In light of the sensor systems presented here, the Xsense project aims at introducing micro- and nanotechnological approaches to the field of explosives detection. The concept based on the development of a trace level sensor by combining four sensor techniques in a hand held device coordinated by advanced mathematical signal processing appears to be a promising approach for explosives detection with a low rate of false alarms. Such a sensor system could be applied in airports, in law enforcement, and military applications where fast, simultaneous and efficient screening of explosives are necessary.

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

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