

# Differential Thermal Analysis Measurement technique for trace explosive detection

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## 1. Introduction

Explosive threats are a major concern in today's society. To insure public safety fast and reliable screening methods for explosives are necessary [1]. The leading technology in terms of sensitivity and selectivity remains to be ion mass spectroscopy [2], but other types of sensors are emerging. We propose differential thermal analysis (DTA) of trace explosive particle as a fast and reliable technique for detecting and determining potential explosive threats. This technique has some clear advantages compared to other selective gas sensors. Selectivity of gas sensors is normally achieved by immobilizing selective receptors on the sensor surface [3]. These receptors will bind only to the desired target molecule, and the binding can be detected via for example mass changes, surface stress changes or by fluorescent labeling. However it is difficult to achieve reliable and reproducible data and regeneration of the sensor surface in order to allow for new measurements is challenging. Using DTA a selective surface coating can be avoided, and the surface is regenerated after each measurement. DTA is a calorimetric technique where the temperature difference is monitored between sample and a reference during a thermal ramp. In this abstract a micro DTA system is presented used for measuring on DNT (2,4-Dinitrotoluene).

## 2. Experimental

A micro heater designed as a bridge is fabricated using standard cleanroom processing techniques measuring (w x l x h) 100 $\mu$ m x 400  $\mu$ m x 400 nm. The bridge is made of silicon nitride with integrated heating elements and temperature measurement resistor made of doped silicon. A SEM picture can be seen in Fig 1. The small size enables a very low thermal mass of only 30 nJ/ $^{\circ}$ K that is needed for measuring trace amounts of explosives. The DTA system is using a reference and a measurement micro heater. To increase the sensitivity and decrease the effect of contact resistances the resistance change of the measurement resistor is determined using 4 terminal sensing as seen in Fig. 2. The sensing region is then limited to the 300  $\mu$ m that is the distance between the terminal contacts.

Small fabrication variation can make temperature response and starting resistance differ from heater chip to heater chip. To make pairing of reference and measurement chip possible different current levels can be applied to either chip to cancel any difference. A difference in temperature response is canceled by applying a calibrated heating profile to the reference chip making the resistance change identical for both chips. In order to achieve high sensitivity amplification electronics has been made by company Unisensor where reference and measurement output of the measurement resistor is subtracted and amplified 500 times, made possible by the close pairing of the two chips. When measuring a fast heating ramp is used, and the temperature change due to melting, evaporation, decomposition and deflagration of the sample is measured giving a unique signature for different explosives as shown in [4].

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### 3. Results and Discussion

The sensitivity has been determined by heating up the bridge using the integrated heaters and measuring the resulting change in resistance using the 4 terminal sensing technique, and by direct measurement of the resistance change. The result can be seen in Fig. 3. Temperature is calibrated by measuring resistance change in an oven. Using the 4 terminal technique a TCR value of  $1.2 \cdot 10^{-3}/^{\circ}\text{K}$  was obtained. Measuring the resistance change directly resulted in a TCR value of  $1.0 \cdot 10^{-3}/^{\circ}\text{K}$  giving an improvement of 20% in sensitivity.

DNT has been adsorbed on the sensor surface by flowing nitrogen containing DNT vapours around the sensor. The adsorbed amount is approximately 0.5 ng a minute, and measurements are done for 0.5, 1, 2 and 2.5 ng. Measurements are presented in Fig 4. showing the resulting DNT signature and that the signal is scalable with the amount of material on the bridge.

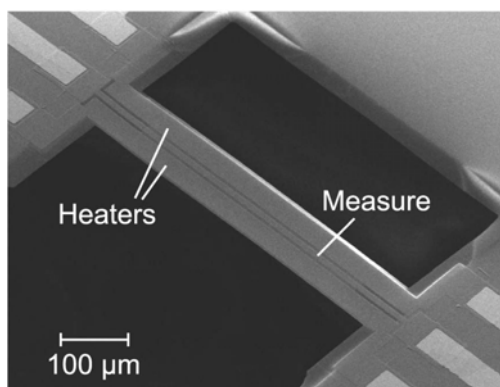


Fig 1. SEM image of a fabricated calorimetric sensor. The heating resistors and the measurement resistor can be seen under the silicon nitride.

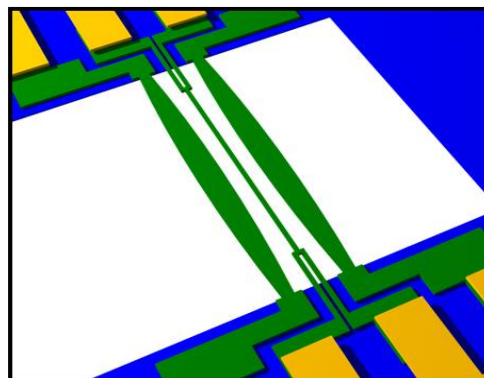


Fig 2. Illustration of the 4 terminal contact system where the resistance change of only the center part of the bridge is measured.

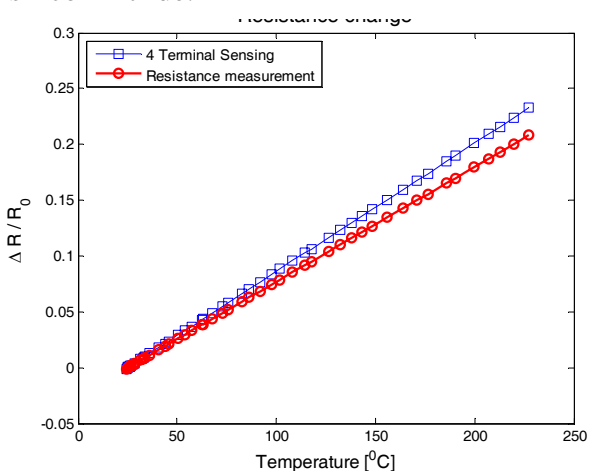


Fig 3. Resistance change as a function of temperature, using a 4 point terminal and a direct measurement.

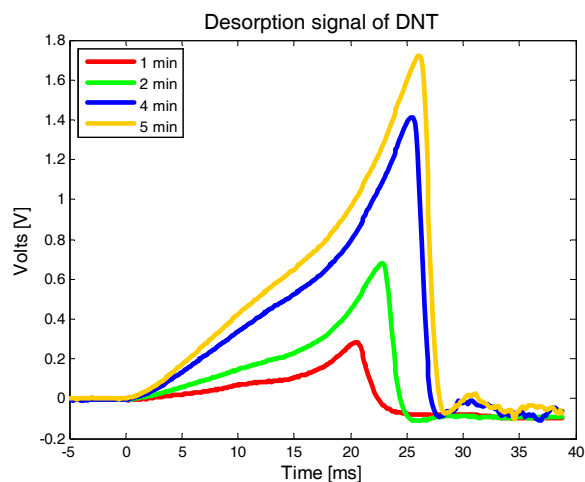


Fig 4. DNT desorption signatures for 0.5 to 2.5 ng of material.

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