DETECTION OF EXPLOSIVES USING A MICRO-CANTILEVER ARRAY NANO-CALORIMETER SYSTEM

A Senior Scholars Thesis

by

MATTHEW R. LANE

Submitted to the Office of Undergraduate Research
Texas A&M University
in partial fulfillment of the requirements for the designation as

UNDERGRADUATE RESEARCH SCHOLAR

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Approved by:

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ABSTRACT

Detection of Explosives Using a Micro-Cantilever Array Nano-Calorimeter System.
(April 2011)

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Over the past few decades, the likeliness of an attack from explosives has increased dramatically. Hence, the need to find a reliable device with the ability to detect explosives has increased even more. Sensing schemes based on micro-cantilever beams and their bending responses to specific explosives are considered to be an effective technique for explosives detection. In this study the bending responses of an array of micro-cantilever beams integrated with micro-heaters are recorded when they are exposed to various combustible materials. The results show that the micro-cantilever beams display a distinct response when exposed to different combustible materials. In the experiments it was observed that typically the deflection of the micro-cantilever beam in response to the thermal bi-metallic actuation was greater when exposed to acetone vapors than that for alcohol and air. Also, the deflection of the beam was greater for experiments using iso-propyl alcohol than that for air. These results are consistent with prior reports in the literature.

DEDICATION

To my loving parents.

ACKNOWLEDGMENTS

I would like to offer a special thanks to Dr. Banerjee for allowing me to work in his research group and providing me with an invaluable research experience. I would also like to thank Mr. Seokwon Kang, a graduate student in Dr. Banerjee's lab, who is the leader of the experiment and provided me with further information and data for the experiment. This project was sponsored by the Defense Advanced Project Agency (DARPA) – Mirco-Technology Office (MTO) through the Micro/Nano-Fluidics Fundamentals Focus Center (DARPA-MF^3). During the course of the study – the author was also supported through the Department of Energy (DOE) – Solar Energy Technology Program (SETP).

NOMENCLATURE

PETA	People for Ethical Treatment of Animals
P	Pressure
T	Temperature
t	Time

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CHAPTER I

INTRODUCTION

Recently there have been many concerns regarding civilian security and prevention of catastrophic events in highly populated areas as well as in military operations at foreign locations (e.g., at airports, border check points, detection of "Improvised Explosves Device" or "IED", etc.). Within the field of airport security the detection of explosive materials remains a highly important and daunting task. Current methods for detecting harmful or explosive devises in airports include but are not limited to the portal trace detection system, body scan wands, and trained canine units. Each device has a unique method of detecting explosives which can be implemented into civilian operations and help ensure the protection of people within the confines of a highly populated building or area.

Inherently, many of the commercial platforms for explosive detection are expensive and suffer from several deficiencies, which have created the need for a more reliable and economical explosives detection devices. Currently, the most accurate method for detection of explosives is through the usage of trained canine units due to their highly sensitive olfactory abilities. Alternative strategies under development at the Department of Defense (DOD) and the Department of Energy (DOE) include the use of trained bees

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This thesis follows the style of Journal of Heat Transfer.

for detection of specific explosives. However many concerns have been raised regarding the use of animals, including opposition from PETA (People for Ethical Treatment of Animals). Ideally, a reliable device needs to be created that can have an immediate impact on increasing the safety of people worldwide without causing harm to animals or compromising privacy. In this study detection of explosives is achieved by using a bimorph micro-cantilever array. This method relies on detection of vapors of combustible materials – which can emanate from both liquid and solid explosives. The sensing principle is based on monitoring the changes in deflections of a heated micro-cantilever due to change in thermo-mechanical stresses caused by the nano-scale combustion on the surface of the micro-cantilever that is catalyzed by a gold coating [1]. *Hence the micro-cantilever acts as a* nano-calorimeter *where the heat of reaction released during combustion is detected at a specific temperature (i.e., at the autoignition temperature) for a given vapor species (or mixture of vapor species).* The bimorph micro-cantilever is consists of two different thin film layers, which are composed of gold (400 nm) and silicon nitride (600 nm) [2]. Each micro-cantilever is individually heated using a micro-heater at the base of the micro-cantilever, fabricated in-situ in the gold thin film layer. The micro-cantilever array is procured commercially (Manufacturer: NanoInk Inc., Model: ActivePens®, Type: M3).

The surface temperature of the gold layer on the micro-cantilever is instrumental in catalyzing the rate of oxidation/combustion when the beam is exposed to a combustible vapor. The heat of the reaction in turn affects the surface temperature which is apparent from the bending response of the micro-cantilever. The bending response is monitored from the deflection of a reflected laser beam that is incident on the surface of the cantilever array (and is reflected by the gold layer) [3]. The auto-ignition temperature of a particular explosive vapor is detected when a difference in the bending response of the micro-cantilever is detected compared to that of the control experiment performed in air (i.e., in the absence of the combustible vapor). In order to determine the efficacy of the sensing scheme using the micro-cantilever array, both simulations and experiments for the micro-cantilever sensor responses were performed in this study [4].

CHAPTER II

GOAL

The goal of this study is to determine the specificity of the sensor response for different combustible vapors. It is expected that each combustible vapor will have a distinct signature in terms of the unique bending response of the microcantilever, which is related to its auto-ignition temperature and is detected by the threshold current required to detect the difference in sensor response compared to the control experiment. Hence, each explosive is expected to have a distinct, clearly identifiable reaction response when detected using a micro-cantilever beam in the array. Fine-tuning this mode of detecting explosives can prove highly beneficial especially because the size of the micro-cantilever beam which is extraordinarily small, portable and much cheaper than the commercial devices currently in use. This portable device could easily be used in airports, ports, and train stations for providing a robust and economical platform for detection of explosives. Due to the small size and weight of this N/MEMS (Nano/Micro-Electro-Mechanical System) based device it can be used in situations not possible before. For example – since this micro-sensor array weighs only a few grams and has a form-factor less than a centimeter – it can therefore be mounted on various UAV (Unmanned Air-Vehicles) for remote monitoring and detection of explosive materials in civilian, urban and military settings.

CHAPTER III

METHODS

The micro-cantilever beam array is attached to a rotation stage that can rotate 360 degrees while providing high precision and control of motion. A laser beam is directed towards the micro-cantilever beams and the movement of the reflected laser beam is controlled automatically through computer-controlled motion stages, which allows the laser beam to be aligned using rectilinear (X, Y, and Z directions) and rotational/ angle motion control. The integrated stage (for both linear and rotational motion) is used to control the angle of incidence of the laser beam with the individual micro-cantilever beams in the sensor array. The motion of the reflected ray is monitored using a Position Sensitive Photo-Detector (PSPD) mounted using fixtures near the sensor array. The sensor platform consisting of the array of micro-cantilever beams, motion control stages, PSPD and the laser source are housed in an airtight chamber (environmental control chamber). The walls of the environmental control chamber are made of acrylic for visual access of the experimental platform. Visual access is needed to allow the user to control the position of the laser beam using the remote controlled motion stages.

The micro-cantilever beams are all pre-fabricated and obtained commercially (Model: ActivePens®, Manufacturer: NanoInk Inc., Skokie, IL). The microheaters to each sensor in the array can are connected to a power source and the micro-cantilevers in the array are actuated by increasing the actuation current. There are a total of 8 micro-cantilever beams within each array. Each beam is composed of a 600 nm thick layer of silicone nitride and a 400 nm thick layer of gold.

For each experimental run, two different measurements are performed. The first measurement (control experiment) is performed in air - in the absence of the combustible vapor. The second measurement is performed in the presence of the explosive vapor. The bending response of the micro-cantilevers is monitored using the PSPD where the change in location of the reflected beam is monitored. In order to perform the experiment, the laser beam is pointed directly onto the 8 micro-cantilever beams in the array. The incident laser beam is reflected by the gold coated surface of the micro-cantilever beams that are undergoing thermo-mechanical deformation induced by the change in surface stresses. Alternately, the position of the reflected laser beam is marked manually on a screen. As the input voltage is increased up to 20 volts with 2 volt increments, the temperature in a beam rises due to Joule heating in the micro-heaters that are fabricated in-situ at the base of the micro-cantilever. The increase in temperature causes the gold layer to expand differentially by a larger amount than the silicon nitride layer at the bottom - thus inducing a bending response of the micro-cantilever by thermal bimorph actuation. Also, as the temperature increases, the rate of chemical reaction catalyzed on the surface of the gold layer is also accelerated. The catalytic activity of the gold layer is different for different species of vapors – causing heat release on the surface of the micro-cantilever due to combustion. The auto-ignition of the combustible vapors thus occur at different temperatures which is monitored by the change in bending response (compared to the control experiment) of the micro-cantilevers that occur at different actuation currents (i.e., threshold value of the actuation current). The reflection of the beam is recorded along with the resistance for each actuation current for each micro-heater in the array. The bending response is measured by either: (1) change in the location of the reflected laser beam on the screen or by PSPD; or (2) change in the resistance of the micro-heater. Initially, The bending response is plotted as a function of the actuation current for the sensor array in air – i.e., in the absence of combustible vapors – which serves as the control experiment and provides the baseline for the sensor response. A similar plot is obtained for the sensor response when exposed to the combustible vapor material of interest. The difference in sensor response is then compared to detect the threshold value of the actuation current at which the bending response deviates from the control experiment. This threshold value of the actuation current corresponds to the auto-ignition temperature of the combustible vapor, which in turn is expected to depend on the species concentration, vapor pressure for the combustible vapor, ambient temperature and humidity. The results from the experiments are used to explore if the threshold current for actuation is distinctly different for different vapor materials, either as a pure species or as a mixture of combustible vapor species.

CHAPTER IV

RESULTS

Acetone and Iso-Propyl Alcohol were used as test materials. The results show the deflection response of the micro-cantilever beam in the presence vapor and in air (absence of the vapor). For predicting the response of the beam when exposed to vapor species, there are two characteristics of the vapors species that need to be incorporated in the numerical models [5]. First of all, the vapor pressure of Acetone (186 mmHg) is much higher than that of Isopropyl alcohol (33 mmHg). Therefore, the combustion and auto-ignition for acetone is expected to be initiated at much lower values of threshold currents than that for iso-propyl alcohol. The second important characteristic of the vapors is the activation energy required to initiate the combustion reactions. It has been found that the activation energy of isopropanol (301.1 × 106 [J/kgmol]) is higher than that of Acetone (137.7 × 106 [J/kgmol]).

Figure 1 shows the deflection of the micro-cantilever beam as a function of the actuation current supplied to the micro-heaters. In the figure, the deflection response when exposed to acetone is compared to that of air. The deflection response is monitored in the experiments is monitored by measuring the position of the reflected laser beam on a screen (i.e., using the principles of an optical lever). The position of the laser beam was measured at actuation currents ranging between 0 mA- 20 mA, with increments of 2 mA. The results in Figure 1 suggest that the micro-cantilever beam has a greater deflection when exposed to acetone than when exposed to air, as the actuation current is increased to 20 mA. The threshold current for acetone was observed to be 18 mA.

Similar experiments were performed using the micro-cantilever beam that was exposed to isopropyl alcohol vapor and the results are plotted in Figure 2. The results in Figure 2 suggest that the beam has a greater deflection when exposed to air than when exposed to isopropyl alcohol, as the actuation current is increased to 20 mA. It was observed that at an actuation current of 18 mA the bending response was higher for isopropyl alcohol than for air. The threshold current for the isopropyl alcohol was observed to be in the range of 12-16 mA [6].

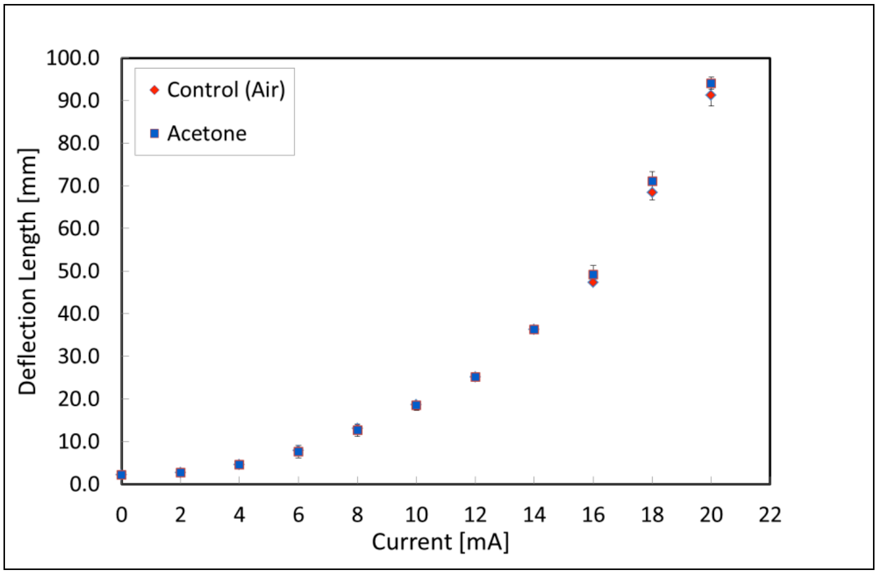

Figure 1. Deflection of the laser beam as a function of the input current for the sensor array in air and acetone vapor environment.

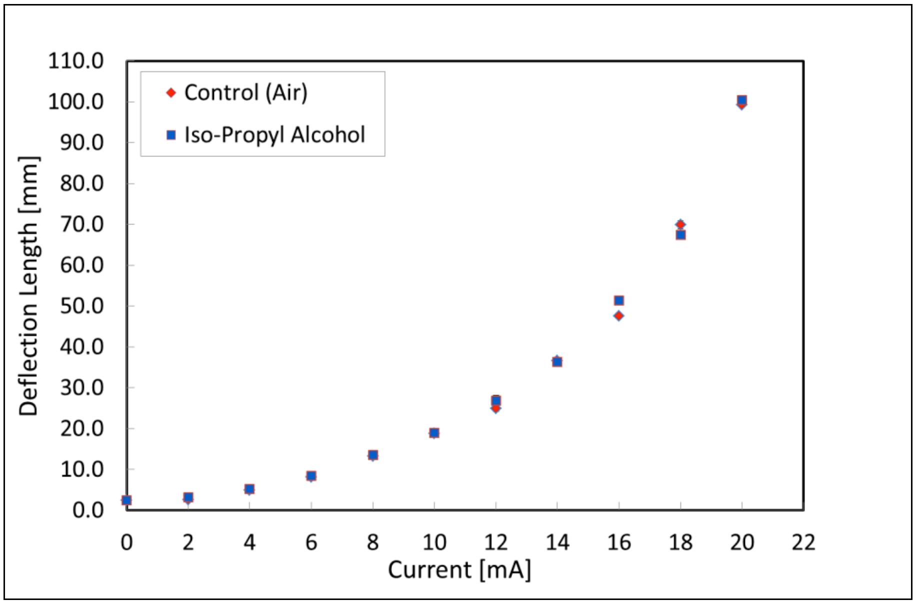

Figure 2. Deflection of the laser beam as a function of the input current for the sensor array in air and iso-propyl alcohol vapor environment.

CHAPTER IV

SUMMARY AND CONCLUSIONS

Experiments were performed using a nano-calorimeter apparatus. The nano-calorimeter apparatus consists of an array of thermal bimorph micro-cantilevers that are actuated using individual micro-heaters that are fabricated in-situ at the base of each micro-cantilever. The bending responses of the micro-cantilevers are monitored using the principle of an optical lever. The experiments show that the bending response was unique for each explosive vapor for the experiments conducted using Alcohol and Acetone.

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