DETECTION OF EXPLOSIVES USING HEATED MICRO-CANTILEVER SENSORS

A Senior Scholars Thesis

by

JAMES RAYMOND KOSS

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

UNDERGRADUATE RESEARCH SCHOLAR

April 2008

Major: Mechanical Engineering

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Approved by:

Research Advisor:	Debjyoti Banerjee
Associate Dean for Undergraduate Research:	Robert C. Webb

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ABSTRACT

Detection of Explosives Using Heated Micro-Cantilever Sensors (April 2008)

James Raymond Koss
Department of Mechanical Engineering
Texas A&M University

Research Advisor: Dr. Debjyoti Banerjee
Department of Mechanical Engineering

The objective of this study is to demonstrate the ability to detect the presence of energetic materials by analyzing the bending response of an electrically heated micro-cantilever thermal bi-morph array. Heating the cantilevers that are made of materials with different coefficients of thermal expansion affects the bending and is measured using an optical device in real time. The detection scheme is based on the threshold value of current that results in a deviation (from the control value) of the actuation of the micro-cantilver. This threshold current is found to provide a unique signature to identify an equilibrium concentration of iso-propyl alcohol, acetone or gasoline vapors at room temperature. The threshold current is proportional with the vapor pressure of the volatile species and the ignition temperature. This shows sensors can be used for specific detection of different energetic material. The sensor array can be used to detect and identify volatile combustibles species in real time. Further, the sensor array can be multiplexed (i.e., detect multiple explosives simultaneously) and also allows redundancy checks so that false positive or false negative results can be eliminated. The sensor

permits detection without coming in contact with the contaminated surface or source of the combustible material because it detects the vapors effused by the explosive materials. Thus it can be used at a nominal distance away from the source.

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NOMENCLATURE

Pt	Platinum
H_2	Hydrogen
PMMA	Polymethylmethacrylate
m	Meters
µm	Micro Meters
nm	Nano Meters
mA	Milliamps
RVP	Reid Vapor Pressure

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CHAPTER I

INTRODUCTION

Detecting explosives is a crucial for saving lives. It is applicable both in the military and commercial security environments. Using a micro-cantilever based sensor is advantageous because of high sensitivity, real time responses, small size, and multiplexing (multiple analyte detections) capabilities. Cantilevers are usually hundreds of microns long, tens of microns wide, and less than a micron in thickness.[1] They are typically made of metals, glass, or silicon composites. In this study the cantilevers are made of silicon, gold, and silicon nitride.[2] Detection may involve recognizing mass absorption or molecules on the surface of the cantilevers. Recognition is achieved through mass sensing, or analyzing surface shear stress changes due to mass absorption causing a bending in a cantilever. This bending is observed using piezoelectric sensors,[3] or optically. A laser beam deflection from a cantilever can provide optical sensing. Figure 1 shows an example of optically sensing the deflection.

This thesis follows the style of Applied Physics Letters.

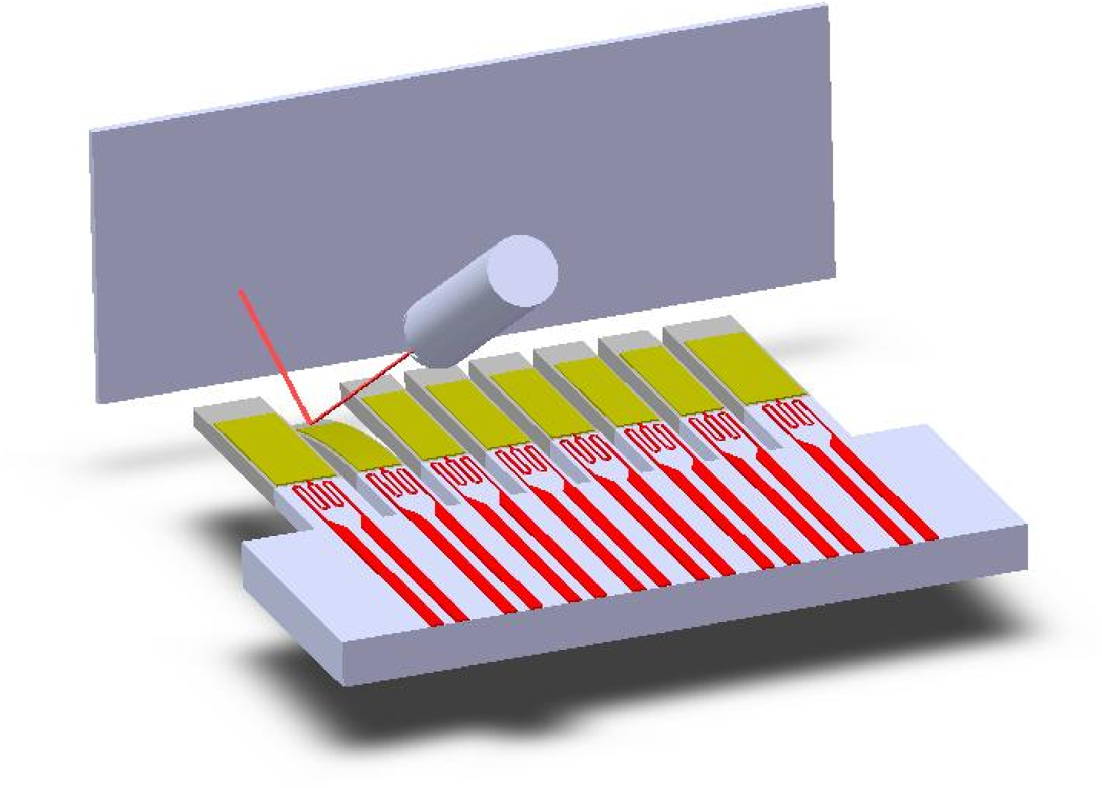

FIG. 1. Optical laser deflection used to sense the position of the cantilever.

Previous studies used micro-cantilevers with a layer of coating used for molecular recognition. These studies show cantilevers coated in Pt result in a different deflection then uncoated cantilevers, when both are exposed to H_2. Cantilevers coated in Polymethylmethacrylate (PMMA) display different shifts in resonance frequencies when exposed to various types of alcohols.[4-8] This study uses heated micro-cantilever arrays to sense surface reactions with different combustible specimens. The advantage of this method is the deflection is not based on mass absorption therefore coating the cantilevers is unnecessary. Electrical resistance micro-heaters are fabricated at the base of the cantilevers. The cantilevers are composed of silicon nitride with a layer of gold on top. The temperature of the micro-cantilevers can be manipulated by changing the current applied to the micro-heating elements attached to the cantilevers. The micro-cantilevers bend due to thermo-mechanical bi-morph actuation. The current activated micro-heaters attached to the cantilever removes the need for an outside heating source to cause the stress induced deflection mentioned in previous studies.[6]

The analyte vapors have different ignition temperatures. Heating the micro cantilever to the ignition temperatures of a specific volatile material causes different bending responses. The detection scheme is based on optically sensing these bending responses of the micro-cantilever exposed to a specific analyte compared to the control response in the absence of the analyte. The different bending responses provide a unique signature to identifying equilibrium concentration of iso-propyl alcohol, acetone or gasoline vapors at room temperature.

The objective of this study is to demonstrate the ability to detect the presence of energetic materials by analyzing the bending response of an electrically heated micro-cantilever thermal bi-morph array. The micro-heated cantilevers of the ActivePen™ array are exposed to the various energetic materials and are optically monitored in order to demonstrate the desired detection. Chapter II describes this experimental setup and procedures in detail. Chapter III reports the results of the experiment. Finally, Chapter IV summarizes the conclusions made from the study.

CHAPTER II

METHODS

Test apparatus

The essential components are the laser, the heated micro-cantilever array, and the projection screen shown in Figure 2. To provide an isolated environment for the testing procedure a Plexiglas box was used to house the test apparatus. The dimensions of the enclosure were 0.3 m × 0.3 m × 0.45 m. The ActivePen™ system (Manufacturer: NanoInk Inc., Skokie, IL)[2] consisting of a set of eight micro-cantilevers was mounted on a linear actuation stage with two degrees of motion. The stage was fixed to a steel platform base. To measure the deflection of the micro-cantilevers at varying currents, a laser diode (630-680 nm, Model: Lasiris™, Manufacturer: StockerYale Inc., Dollard-Des-Ormeaux, QC, Canada) was used as a light source. The laser was supported by a series of movable steel bars from the platform base. The angle and position of the laser was adjustable and was focused on a single cantilever of the array under observation. The laser is reflected from the polished gold surface of the cantilever. The reflected laser was monitored and recorded on the projection screen that was mounted on the vertical side of the enclosure for the different test conditions. The screen consists of a semi-transparent sheet of paper and the distance of the recorded positions of the reflected laser were measured. The displacement of the laser on the projection screen was directly proportional to the deflection of the laser when different currents were applied.

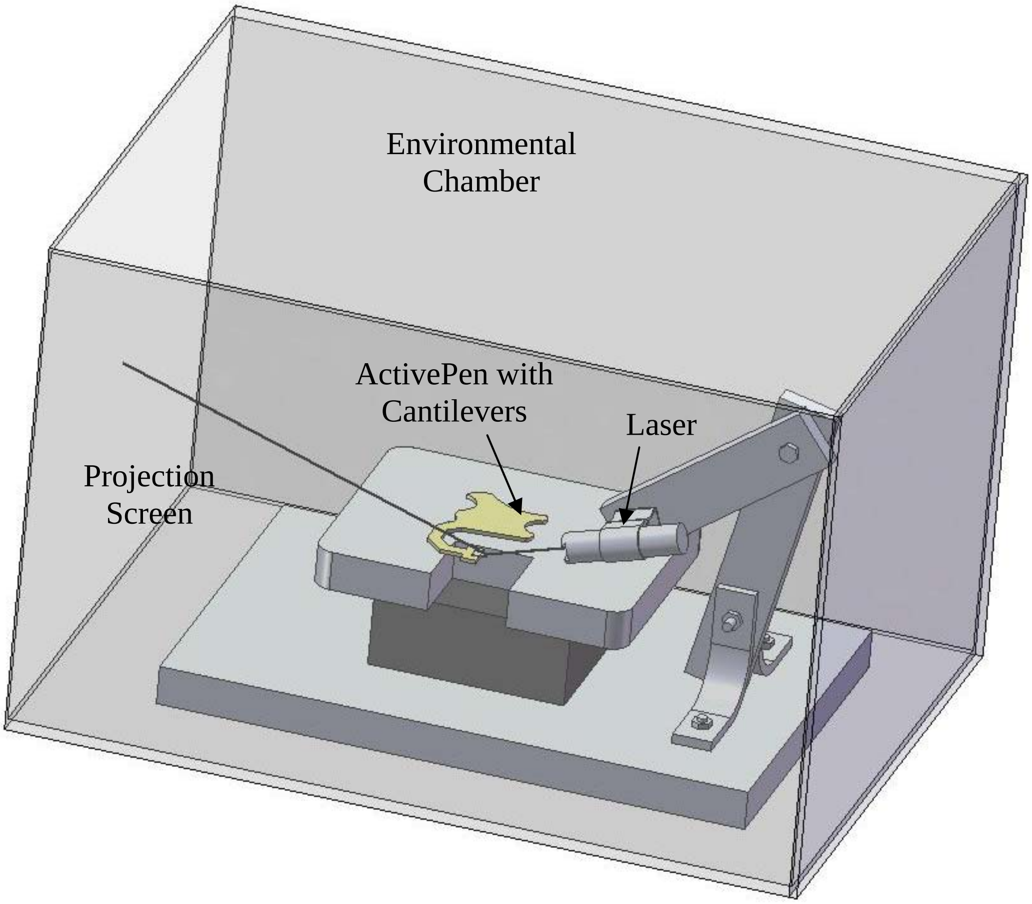

FIG. 2. Schematic of experimental test setup.

The ActivePen™ array (Model: T3,.Manufacturer: NanoInk Inc.) consists of six writer probes and two reader probes that are 150 microns long shown in Figure 3.[2] The width of the probes are 30 microns for the writer and 40 microns for the reader with 10 microns between each probe. The ActivePen™ system consists of a platform that the micro-cantilever array was mounted on. The platform connects to a variable current or voltage control DC power supply. The heated cantilevers were controlled by varying the current supplied to the ActivePen™ array.[1] The circuit configuration consisted of an Ammeter in series with the power supply to measure the current supplied to the micro-cantilevers, and a 100 Ohm resistor in series for added safety as well as control of the current.

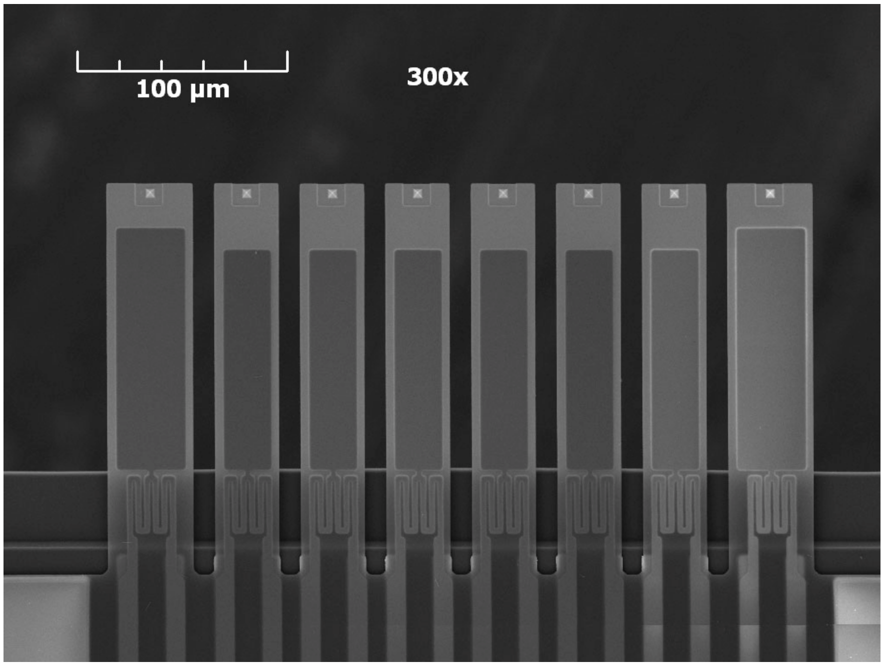

FIG 3. ActivePen™ array of cantilevers.

The ActivePen™ cantilevers are made of silicon nitride. A serpentine electrical micro-heating element is located at the base of the cantilever and is attached to a gold heat spreader on the cantilever. Heat is conducted along the heat spreader and into the silicon nitride cantilever. The different coefficients of thermal expansion cause the cantilever to bend when the current is increased and the heat is applied, known as thermal bi-morph.[2]

Test procedure

Control test

The control tests for the experiments were performed in ambient air in the absence of any combustible vapors. The laser was focused on the cantilever under consideration. The reflection was marked on the projection screen before a current was sent through the micro-heater in order to obtain a zero for the proceeding deflections. A current was applied to the cantilever incrementally up to a maximum of 25 mA. The reflection of the laser was marked at each increment of current on the projection screen. The displacements of the marks on the projection screen from the zero were measured to proportionally represent the deflection of the cantilever. The process was repeated using the same cantilever again in ambient air conditions to serve as control data for comparing the deflection of the micro-cantilevers when exposed to different volatile vapors.

Experiments with combustible vapors

Isopropyl Alcohol, Acetone, and Gasoline were the three combustible substances considered in the experiments. Prior to introducing the substances while the system is only exposed to ambient air in the container baseline marks were recorded using a method similar to the one described in the ‘Control Test’. The reflection of the laser was marked on the projection screen as the supplied current was incrementally increased from 0 to 25 mA in a single cantilever. The test substance was then introduced into the environmental chamber by placing a small container filled with the liquid under the cantilevers. The door of the test chamber was closed to isolate the test apparatus. The seams of the door and any other gaps in the chamber were sealed in order to stop the combustible vapor from leaking. The substance was left for 15 minutes in order to allow the combustible vapor to reach its thermodynamic equilibrium concentrations at room temperature (about 27°C). During the experiment the apparatus is left undisturbed in order to ensure that no outside effects contribute to the deflection chance in the cantilever or the reflected position of the laser on the projection screen. After the thermodynamic equilibrium was reached the same cantilever that was used to achieve the baseline while exposed to ambient air, was monitored. A current was applied to the micro-cantilever incrementally in steps of 1-2 mA up to 25 mA. The position of the reflection was recorded on the projection screen at each increment beginning with the mark when no current is supplied. The height of each point from the base point of no current is proportional to the deflection of the cantilever and was measured as a function of input current. The initial position of the reflected laser exposed only to ambient air is then compared to the position of the laser exposed to the combustible vapor.

CHAPTER III

RESULTS

The response of the sensor is recorded as a change of height as a function of actuation current for the three combustible materials used for this study: Acetone, iso-pryl alcohol, and gasoline. As the actuation current is increased the surface temperature of the cantilever increases and can reach around 500°C.[9] At low actuation currents the deflection of the cantilever in the presence of volatile material was similar to the control. The bending response diverged from that of the control experiments at a current identified as the threshold value of actuation current. The threshold value of actuation current was found to scale with the ignition temperature and vapor pressure of the combustibles. The value is used to identify the specific combustible material.

Acetone experiments

The deflection recorded after introducing acetone into the enclosure and allowing it to reach equilibrium the same as the ambient air control up to 10 mA, seen in Figure 4. At 12 mA the deflection is different from the control experiments, and slightly different for the subsequent currents.

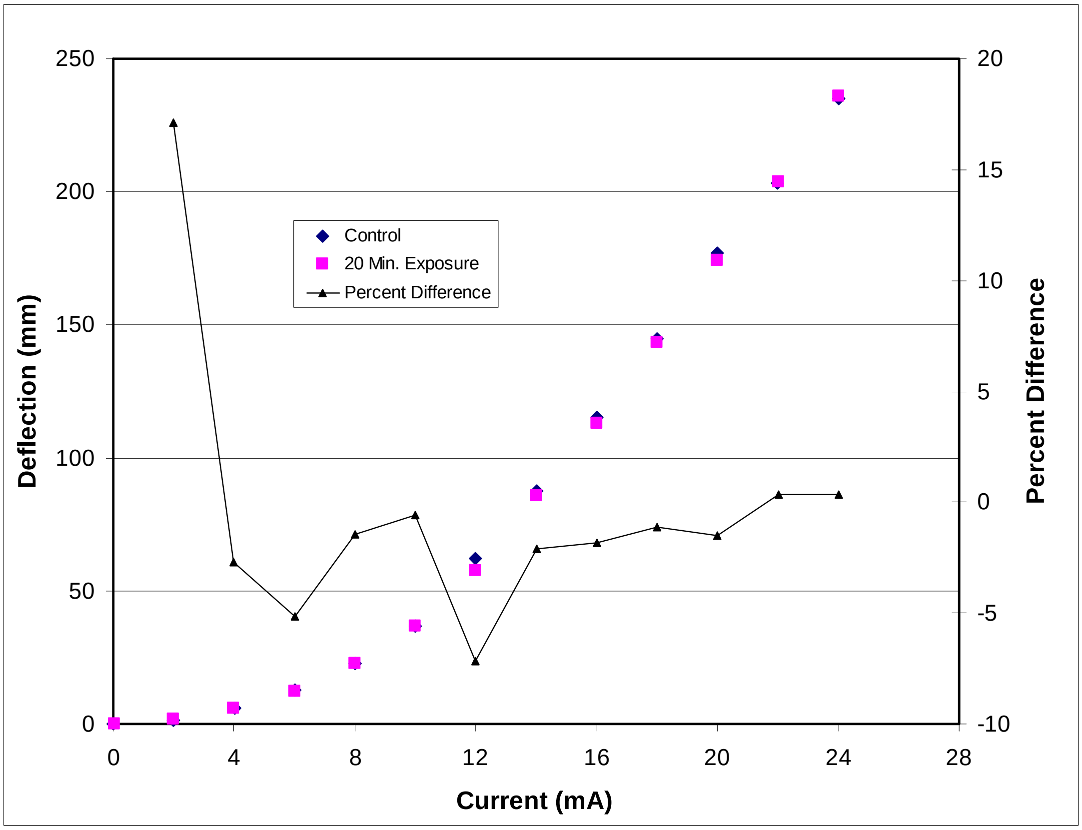

FIG. 4. The deflection response of the sensor for acetone relative to current.

Iso-propyl alcohol experiments

Iso-propyl alcohol caused the sensor deflection to diverge from the control in ambient air at around 8 mA. Most of the successive input currents also resulted in a deflection different from the control. 8 mA is established as the threshold temperature. The response of Iso-propyl alcohol is shown in Figure 5.

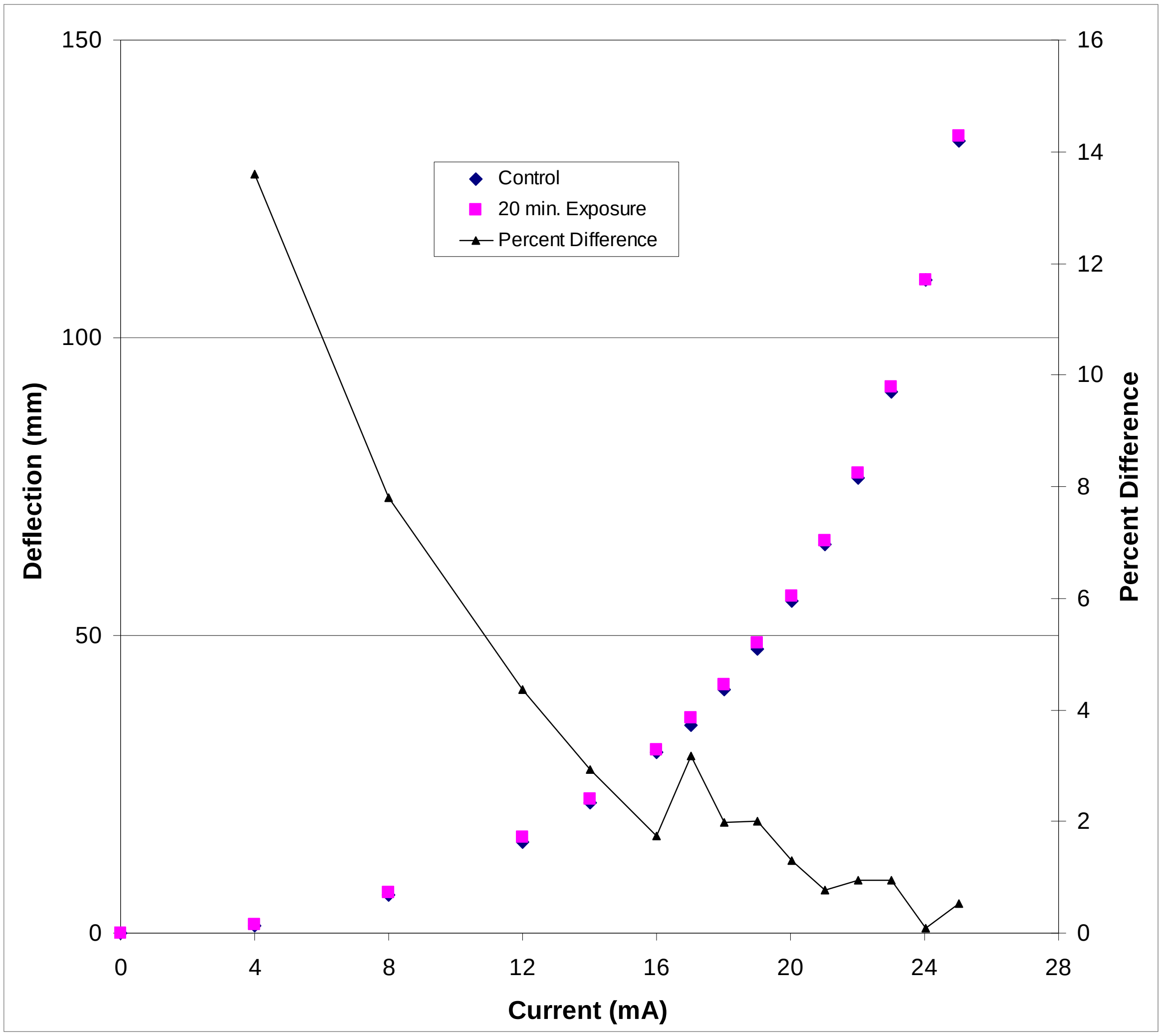

FIG. 5. The deflection response of the sensor for iso-propyl alcohol relative to current.

Gasoline experiments

When exposed to gasoline, the sensor experienced a significant change in deflection from the control at around 18 mA of input current, shown in Figure 6. The percent difference between the control and the gas exposure deflections for the following currents, experienced large variation. However, 18 mA can be determined to be the threshold current.

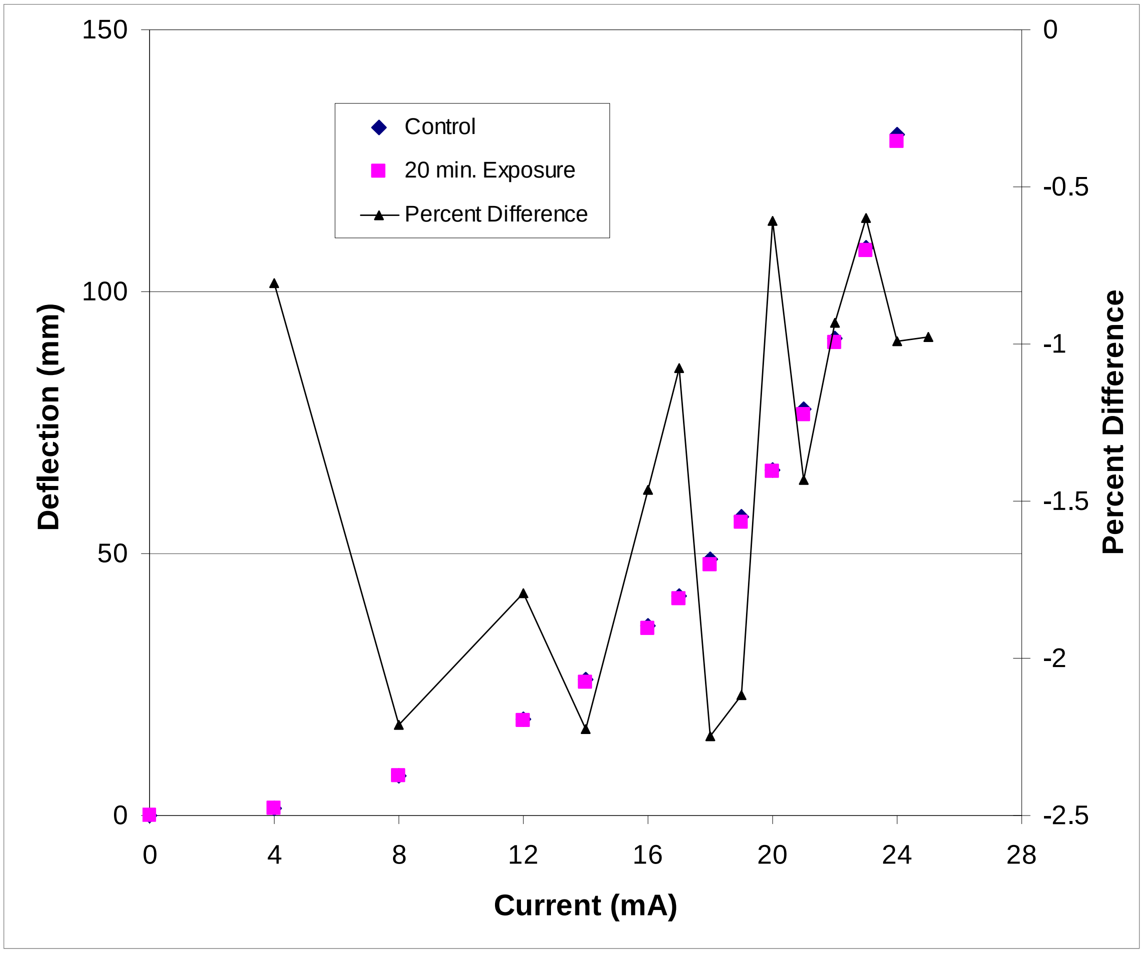

FIG. 6. The deflection response of the sensor for gasoline relative to current.

Discussion

Acetone vapors have an ignition temperature of 465°C[10] and a vapor pressure of 186 mm of Hg at 20°C.[11] The equilibrium mass fraction of acetone for these experiments is around 0.25 mole / mole of air.[1] Isopropanol vapors have an ignition temperature of 465°C[10] and a vapor pressure of 186 mm of Hg at 20°C.[12] The equilibrium mass fraction of acetone for these experiments is around 0.25 mole / mole of air.[1] Gasoline has an ignition temperature of 280°C.[10] Gasoline is a mixture of volatile components and its vapor pressure must be characterized using Reid Vapor Pressure. The RVP for gas is between 213 and 623 mm of Hg at 37°C.[13,14] The relationship between each substances calorimetric properties and its threshold current can be seen in Figure 7.

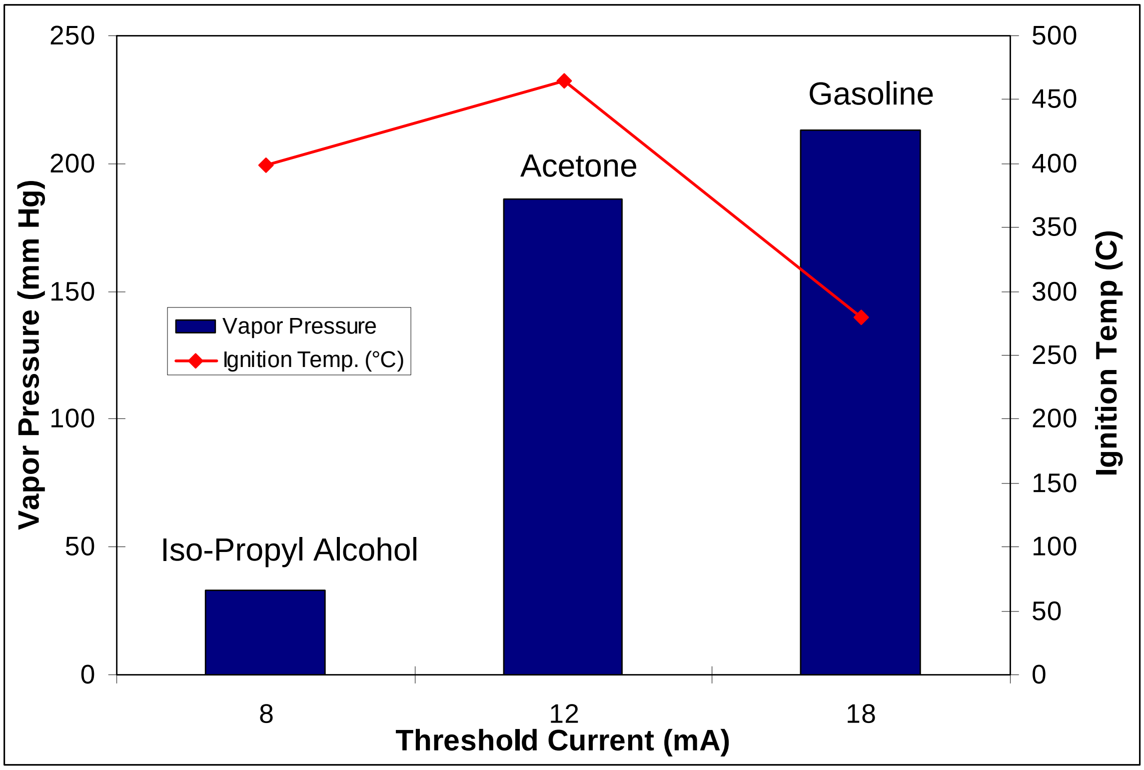

FIG. 7. The sensor response for the three materials in relation to their calorimetric properties.

When the applied current is increased the surface temperature of the cantilever also increases. As the surface temperature approaches a specific value related to the ignition temperature and vapor pressure of the combustible vapor present in the test environment, a reaction takes place responsible for a change in surface stress and the bending response. Absorption, catalytic oxidation, or other surface reactions are most likely responsible for the change in surface stress on the cantilever. The cantilevers can be coated with a material to enhance the sensitivity with respect to a specific mechanism (e.g. absorption or catalytic oxidation.) or a specific combustible to identify calorimetric signatures. It is possible to supply each cantilever with a different actuation current simultaneously. This would allow the sensor to instantly test for up to eight different combustibles at the same time by supplying the known threshold currents for vapor to a different cantilever. This would immediately determine which vapor is present. Or multiple cantilevers could be supplied the same current to provide redundancy in order to reduce uncertainty. This sensor platform could be integrated with control electronics that would analyze the data and display the results for real time identification of the combustible present.

CHAPTER IV

SUMMARY AND CONCLUSIONS

The experiments show that exposing the sensor to acetone, iso-propyl alcohol, and gasoline results in a change in the deflection of the cantilevers after reaching an unique threshold actuation current for each vapor species as the actuation current is increased (compared to control response obtained from exposure to ambient air). Each combustible vapor can be uniquely detected by monitoring the threshold actuation current that is unique to each vapor. The deflection in the presence of iso-propyl alcohol diverged from the control at a threshold actuation current of 8 mA, and had an increased deflection at higher actuation currents. Acetone had a threshold current of 12 mA, producing a deflection less then that of the control. Gasoline caused the deflection of the cantilevers to significantly change at 18 mA. The results show that the threshold current is closely related to the vapor pressure of the combustible. Based on these results it can be concluded that the volatile vapors of the three combustibles change the deflection of the cantilever compared to an open air control. This deflection can be detected using an optical sensing technique. It can also be concluded that the sensor can be used to detect each specific combustible based on the unique deflection signatures. This sensor can be implemented in many different applications. The applications can range from permanent instruments in home based environments to applications for occupational safety and also in portable devices for military applications. The performance of the sensor array can also improved for detecting a specific combustible, by coating the cantilevers with reagents for specific analytes of interest.

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CONTACT INFORMATION

Name: James Raymond Koss

Professional Address: c/o Dr. D. Banerjee
Department of Mechanical Engineering
Texas A&M University
3123 TAMU
College Station, TX 77843-3123

Email Address: jrkoss@neo.tamu.edu

Education: B.S. Mechanical Engineering, Texas A&M University
May 2008
Cum Laude
Undergraduate Research Scholar
Pi Tau Sigma