A Persistent Surveillance Technique for the Detection of Explosives and Explosive Precursors

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Abstract

A gas detection platform was developed for explosive precursors using nickel micro-heaters coated with various metal oxide catalysts. Novel catalysts were developed for these gas sensors using combinatorial chemistry techniques in conjunction with co-sputtering from multiple oxide targets. Rapid screening protocols were facilitated by "printing" large arrays of sensor elements, so that a wide range of catalyst chemistries could be investigated for a specific target molecule of interest to DHS.

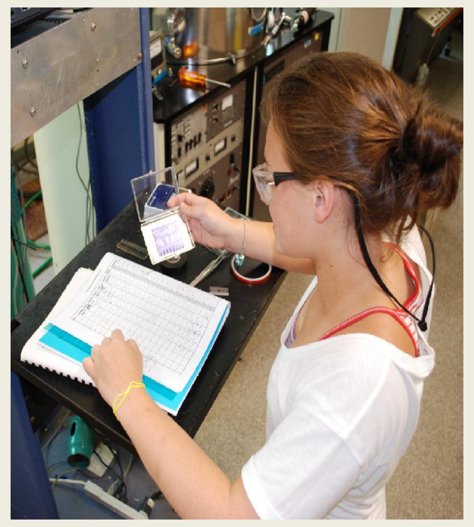
Very low concentrations of specific target molecules can be detected with our gas sensors, which rely on a thermodynamic response, rather than a conductometric transducer response. The thermodynamic response occurs at a specific temperature for a given target molecule and catalyst, as the sensor is thermally scanned from room temperature to elevated temperature, similar to microcalorimetry.

This calorimetry based detection scheme has improved long term stability relative to other sensors. The effects of oxygen partial pressure and humidity on the metal oxide catalyst are also minimized.

Relevance

The development of an inexpensive metal-oxide gas sensor that is capable of unambiguously detecting trace levels of target gases. Our sensor has the ability to detect explosives and explosive precursors in air, under ambient conditions, using optimized transition metal oxides as catalysts.

The sensor platform utilizes robust nickel microheaters that can be easily integrated with other sensor platforms to form a security network.



Technical Approach

General Fabrication Process

Nickel based microheaters were prepared on Al_2O_3 substrates, using standard photo-lithography techniques to form a large array of gas sensors. The microheaters were annealed in nitrogen at 900 C to improve electrical stability, prior to being coated with various metal oxide catalysts. Combinatorial chemistry techniques (Figure 1) were used to deposit a gradient of catalyst chemistries on microheater arrays which were later screened for optimal catalytic performance.

Testing and Sensor Characterization

The catalytic activity of the microheaters over a wide range of temperatures was determined by measuring the difference in electrical power required to heat the microheater to various setpoint temperatures; first in ambient air and then in the presence of trace amounts of various target gases including H_2O_2, NH_3, N_2O, Cl_2 and TATP (Fig 3-6).

The oxidation state and chemistry of the various metal oxide catalysts was determined before and after exposure using x-ray photoelectron spectroscopy. Figure 2 shows a sample XPS spectrum for a CuO and Cu_2O film produced from oxide and metal targets.

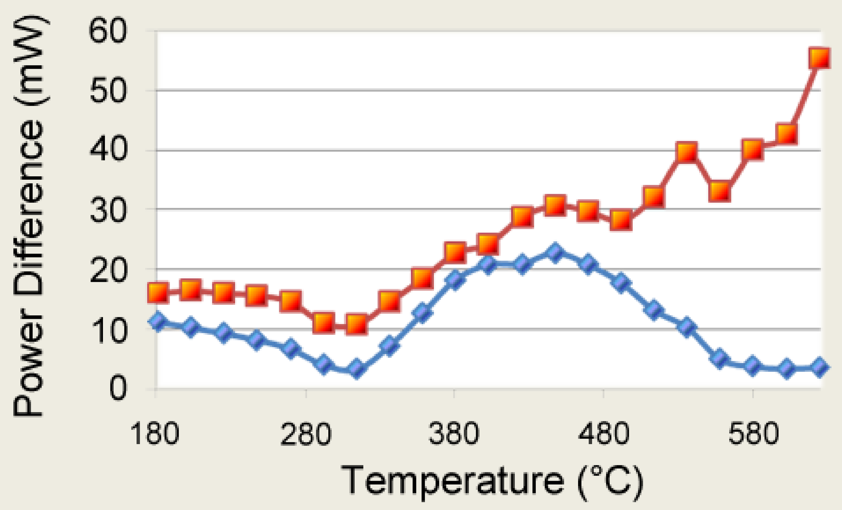


Figure 3. Response of a tungsten oxide coated microheater to H_2O_2 (red square) and TATP (blue diamond)

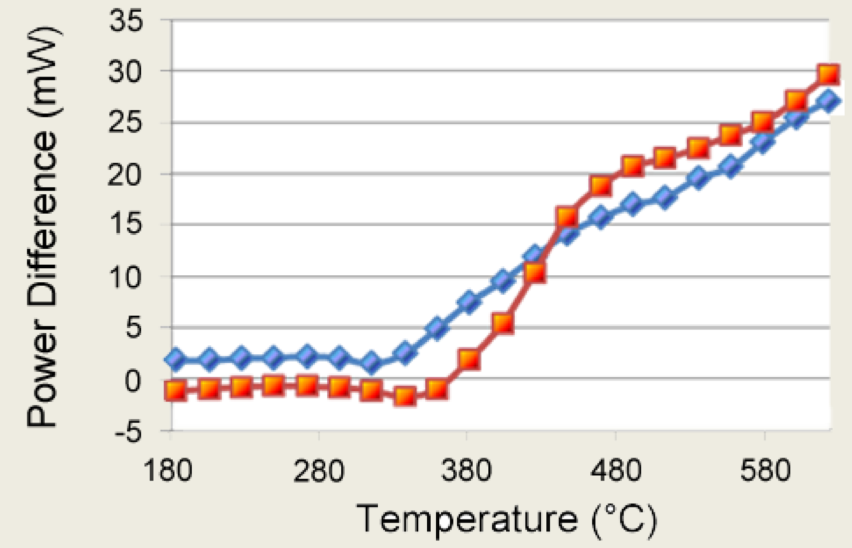


Figure 4. Response of a tin oxide coated microheater to H_2O_2 (red square) and TATP (blue diamond)

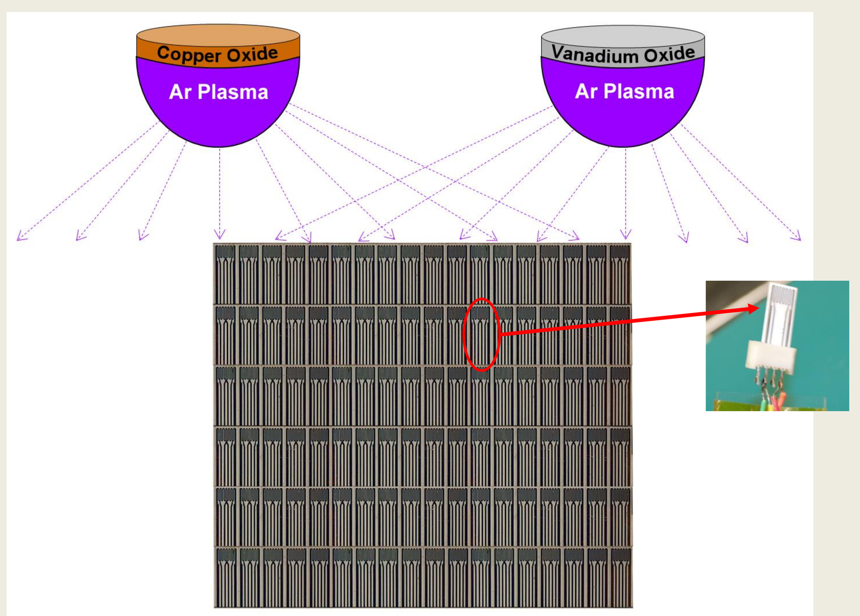


Figure 1. $CuO-V_2O_5$ catalyst libraries were deposited onto microheater arrays using co-sputtering in an oxygen plasma

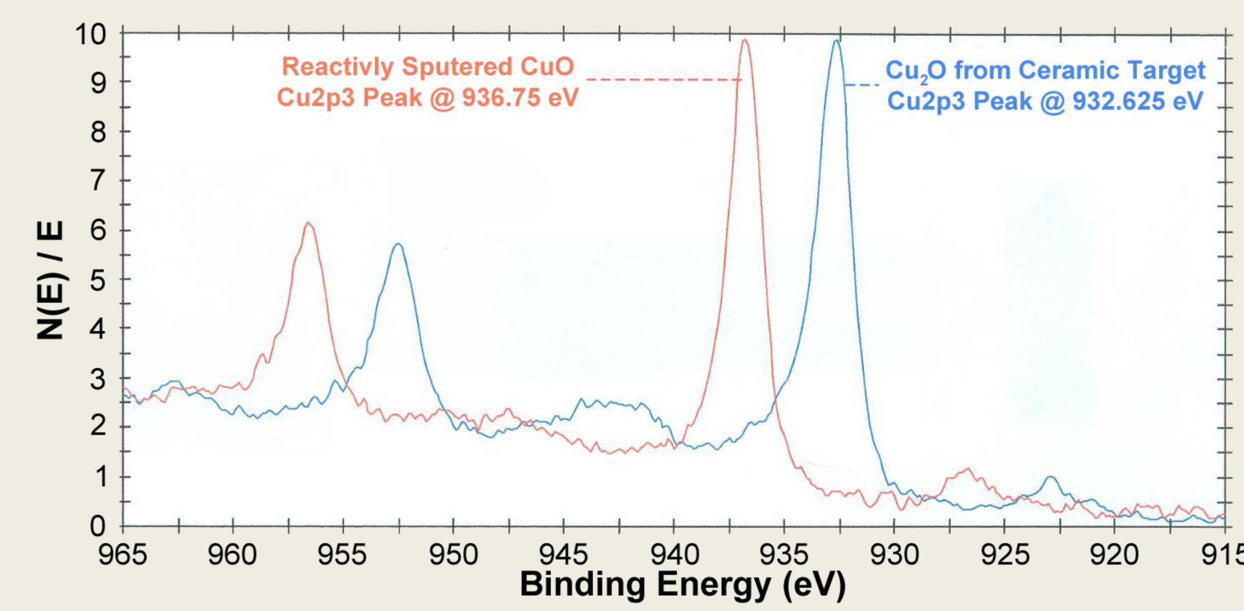


Figure 2. XPS spectra, showing shifts in binding energy observed for Cu_2O and CuO catalysts.

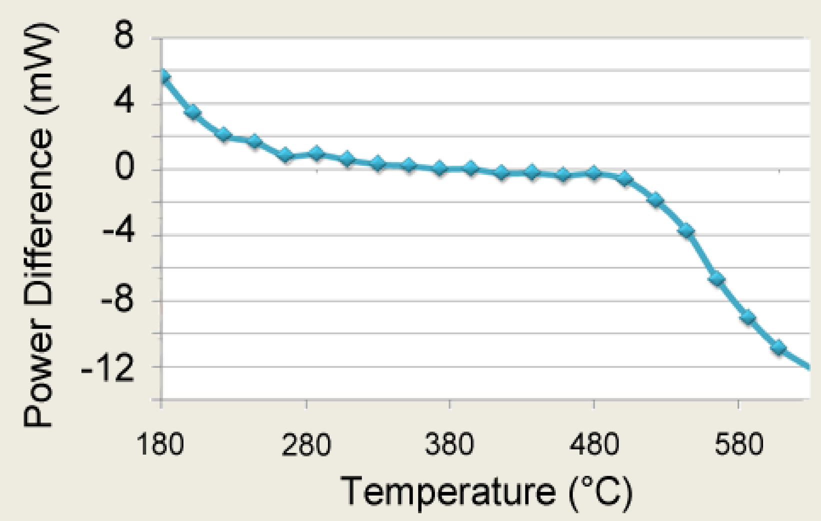


Figure 5. Response of a microheater coated with a copper oxide/ vanadium oxide alloy catalyst that was prepared by co-sputtering in an oxygen plasma from vanadium and copper metal targets and tested in H_2O_2.

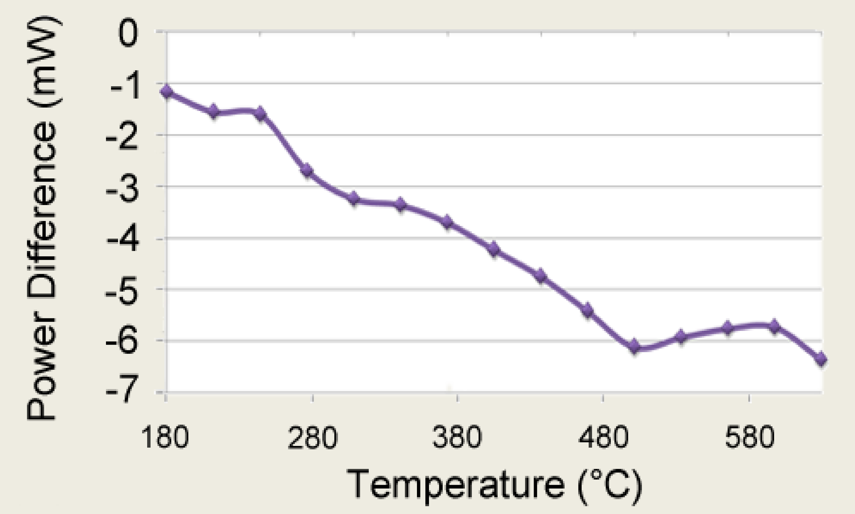


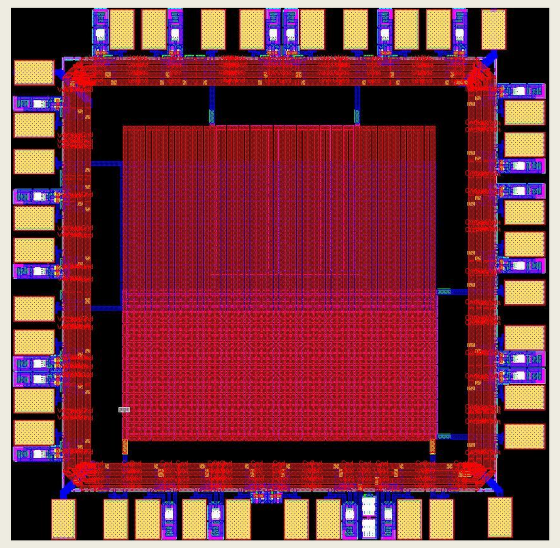
Figure 6. Response of a copper oxide (Cu_2O) coated microheater prepared from copper oxide target and tested in H_2O_2.

Accomplishments Through Current Year

A cost effective, robust gas sensor, capable of detecting TATP at the parts per million level, was developed using nickel microheaters coated with various metal oxide catalysts including tungsten oxide, vanadium oxide, copper oxide, zinc oxide and tin oxide. Using combinations of the aforementioned oxides, novel catalysts were developed for TATP sensing using combinatorial chemistry techniques, whereby rapid screening protocols were used in conjunction with co-sputtering from multiple oxide targets.

Future Work

Partnering with engineers from the Navy (NUWC) in Middletown RI, we are designing and developing a mixed-mode MEMS catalytic gas detector that incorporates free standing diaphragms with embedded microheaters in a silicon architecture to improve the response time and sensitivity of the sensor. This MEMS based sensor platform will initially be demonstrated using TATP as the target gas to show the improvement in performance metrics with this approach.



1.5 mm x 1.5 mm MEMS chip

Opportunities for Transition to Customer

We have collaborated with SensorTech, Inc. (Savannah, GA) over the past few years on gas detection protocols for the US Army and DARPA. Raytheon, Smiths and Draper Labs have expressed a keen interest in our technology and key personnel from both organizations are updated on a regular basis.

Patent Disclosures

"Thermodynamic Based Gas Sensors Using Metal Oxide Catalysts", O.J. Gregory, M. Platek and A. Cote, URI Patent Disclosure filed Feb. 20110

Publications Acknowledging DHS Support

O.J. Gregory, H. Ghonem, M.J. Platek, J. Oxley, J. Smith, M. Downey, C. Cummiskey and E. Bernier, "Microstructural Characterization of Pipe Bomb Fragments", Materials Characterization, Vol.61, No. 3, p.347-354, (2010).

219th Electrochemical Society Meeting 2011, Montreal, Canada, "Detection of TATP Using Thermodynamic Based Gas Sensors with Metal Oxide Catalysts", Y. Chu, K. Waterman, C. Hurley, M. Amani and O.J. Gregory.

Microscopy and Microanalysis 2010, Portland , OR, "Characterization of Pipe Bomb Fragments using Optical Microscopy and Scanning Electron Microscopy", M.J. Platek, O. J. Gregory, T. Duarte, H. Ghonem, J. Oxley, J. Smith, E. Bernier.

Y. Chu, K. Waterman, C. Hurley, M. Amani, M. J. Platek and O. J. Gregory, "Catalytic Decomposition of Triacetone Triperoxide (TATP) Using Transition Metal Oxide Based Gas Sensors", submitted to Sensors Letters

Other References

G. Eranna, B. C. Joshi, D. P. Runthala, R. P. Gupta, "Oxide Materials for Development of Integrated Gas Sensors- A Comprehensive Review", Critical Reviews in Solid State and Materials Sciences.

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