

FINAL TECHNICAL REPORT
Detection of Specific Gases by Metal Oxide Catalysis

28 June 2008

Sensor Tech, Inc.
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Strategic Technology Office (SRO)
Program: Detection of Specific Gases by Metal Oxide Catalysis
ARPA Order No. W200/00, Program Code: 6310
Issued by DARPA/CMO under Contract No. HR0011-06-C-0135
Contract duration: 29 September 2006 through 30 June 2008

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Abstract

Unambiguous detection of specific gases has been demonstrated to occur at specific temperatures by metal oxide catalysis. Potentially useful metal oxides and their combinations were deposited for evaluation by means of combinatorial material synthesis. The resulting material libraries were then tested for desired catalytic activity using thermal spectroscopy with a differential calorimetry detector. Experimental results suggest that some gases may be optimally detected with oxides of a single metal while other gases may be optimally detected with specific combinations of at least two metal oxides.

Subject terms

Gas detection, catalysis, metal oxides, gas sensors, gas transducers, calorimetry, differential calorimetry

Task Objectives

Gas detectors useful in the field suffer from marginal specificity – background gases can confuse the sensor yielding false positive and/or negative indications. Sensor Tech, Inc., Savannah, GA has demonstrated that simple, inexpensive gas detection sensors using selected 3D transition metal oxides as catalysts can unambiguously detect minute concentrations of specific gas molecules. This capability is retained in the presence of both high concentrations of background gases and high concentrations of closely related gas molecules.

Detectors with this capability are essential for the early sensing of toxic gases in building ventilation systems, other enclosed areas and outdoor but confined venues such as stadiums and public parks, where the deliberate or accidental release of toxic substances could affect public health and safety.

Sensor Tech is developing a new class of chemical sensors based on this technology and demonstrating its efficacy by detecting a predetermined toxic gas in a building's heating, ventilation, and air

conditioning systems (HVAC). Sensor Tech is delivering a set of three transducer assemblies that have been characterized for detection of ammonia and ethanol.

The contract requires that Sensor Tech accomplish the following tasks:

1. Identify the most suitable catalyst compounds for a target gas by using combinatorial material synthesis to create catalyst test specimens. Each set of different material compositions completed for each test specimen is termed a 'library' of materials.
2. Expose these test specimens to the target gas and the background gas in an apparatus that controllably varies the temperature of the catalytic test specimen and observe the thermodynamic response of these catalytic reactions over the x-y area of the test specimen as it cools to ambient.
3. Identify optimally-responsive catalytic compositions by noting and subsequently examining their x-y position on the test specimen.
4. Produce the optimally-responsive catalytic compositions for use in sensors.
5. Make and test operational sensors to verify and validate that the catalytic compositions identified meet the requirements for sensitivity and selectivity with respect to the test gas.
6. Report results and deliver a set of sensors to DARPA.

This work provides key enabling technology for the placement of highly sensitive and selective sensors in the HVAC of buildings critical to the continued operation of the Government in the event of deliberate chemical warfare attack. Such placement will provide the early detection of toxic gases minimize human morbidity and mortality and provide for the controlled and orderly investigation of such an event.

Technology Background

Sensor Tech's specific gas detection technology materially differs from the typical catalytic gas detection technology.

Typical Catalytic Gas Detection Technology

In general, catalytic gas detection in general use typically involves:

1. an electrically heated catalyst brought to a temperature such that
2. a catalytic reaction ensues yielding complete combustion
3. that raises the temperature of the gas surrounding the catalyst/heater system which continues to dissipate electrically generated heat and thereby
4. further raises the temperature of the catalyst/heater system to enable transfer of the electrically generated heat to the surrounding environment.
5. This temperature rise of the catalyst/heater system is correlated with the concentration of the gas undergoing combustion. (This is termed the offset detection technique.)

Typical detector structures sense catalyst temperature in a manner that yields an electrical signal that is a function especially of the temperature rise of the electrically powered catalyst/heater system in

proximity to the combustion, and is also a function of the temperature *distribution* throughout the sensing structure. The observed general temperature rise due to combustion is correlated with the concentration of gas(es) undergoing combustion.

Typical catalytic gas detection approaches (while sometimes claiming otherwise) tend to be inherently non-specific because other gas species in addition to the target gas may be simultaneously experiencing partial or complete combustion. Differential sensors can sometimes isolate the effects of a few combusting gases from the many gases simultaneously undergoing combustion.

Sensor Tech's Gas Detection Sensor Technology

Sensor Tech's detection approach is novel in that it uses a catalytic chemistry that operates at temperatures well below combustion temperatures (sometimes below the temperatures at which various noble metals become catalytically active). The detection approach operates in a manner that inherently produces a specific partial-oxidation chemical reaction. The specific reaction (typically but not necessarily exothermic) results in inherent detection specificity.

We carefully avoid target gas combustion because combustion tends to be a non-specific and is therefore an undesirable condition for the detection of specific target gases. In fact, noble metals can sometimes be used for passivation purposes in the detection structures enabled by Sensor Tech's technology because our sensors can in some cases operate well below the catalytic activation (combustion) temperatures of the noble metals.

Sensor Tech's novel approach to gas detection involves:

1. a catalyst raised with electrical power under feedback control to
2. a temperature specific to the target gas-catalyst combination that initiates
3. a specific non-combustion, partial-oxidation reaction that
4. takes place at a temperature slightly lower than the catalyst's temperature and
5. raises the temperature of the catalyst's surroundings
6. but is prevented from raising the temperature of the catalyst itself by
7. a feedback control system that
8. lowers the electrical power applied to heat the catalyst sufficient to
9. maintain the catalyst at the temperature specific to the target gas-catalyst combination. (This is termed the null-balance detection technique.)
10. The difference in electrical power required to maintain catalytic and non-catalytic surfaces of the same sensing structure design is directly related to the concentration of the target gas.

Sensor Tech's sensing devices in use today require a novel structure to enable our novel specific gas sensing technology to operate with the greatest precision and accuracy.

Sensor Tech's novel null-balance differential calorimetry detection approach is practical because the temperature specific to the target gas-catalyst combination is above the temperature of the ensuing reaction. Sensor Tech's catalyst is held above the reaction temperature by an amount that provides the activation energy for the specific non-combustion, partial-oxidation catalytic reaction. A useful feature of this situation is that the entire reaction can be easily quenched by simply reducing the electrical sensor heating power to zero which drops the catalyst to below the activation temperature.

It is crucial for detection specificity that the temperature specific to the target gas-catalyst combination is below the ignition temperature of the target gas. The ensuing reaction thereby accomplishes only a specific partial oxidation of the target gas rather than a more complete oxidation of the target (and likely other gases) that would result from ignition.

Sensor Tech's Catalytic Chemistry Detection Process

The uniqueness of 3D metal oxides is their ability to catalyze intermediate partial oxidations which are temperature and activation energy specific. Specificity for Ethanol for example, is determined by interactions and conditions which affect the properties of the catalyst-target molecule complex and the metal oxide's 3D orbital's electronic transition state, EA. The magnitude of the EA uniquely determines the specific target temperature for the reaction.

Many parameters can affect the magnitude of the EA, therefore the target temperature. Some of these parameters are: different ligand geometries, bond energies of metallic oxide catalysts as well as their polarities with respect to the target molecules and bond energies and varying probabilities of target molecule-metal oxide interactions, etc.

The one constant through all of the above is that even though the target temperature may vary, a given reaction's net free energy will not vary, i.e. the free energy of the oxidation of Ethanol to Acetal will always be -189.0 KJ/mol whereas the target temperature will vary with EA.

The electronic transition state properties of a particular metal oxide are not the only source of specificity for a particular gas. The unique properties of the 3D orbitals additionally provide geometric specificity.

The mechanism for the first step (Ethanol to Acetal) of Ethanol oxidation is dependent upon the specific properties of a specific 3D metal. For the case of copper oxide, the following properties are involved: The metal oxygen double bonds are shorter than the 3D ligands so that the oxide bond is enclosed in the volume containing the electronic properties of the copper 3D shell. This means that both oxygen and Ethanol must travel from the atmosphere and traverse the 3D shell in order to react with the copper oxide catalyst.

The second step is the oxidation of the aldehyde to higher oxidation products. 3D metal oxides at lower temperatures do not catalyze to the final combustion products water and carbon dioxide.

Sensor Tech's Signal Conditioning

Sensor Tech's signal conditioning electronics approach maintains essentially constant temperature operation of a sensor's catalytic area at a specific temperature in order to assure that a specific partial-oxidation reaction of a specific gas species is initiated by contact with a specific catalytic surface. All of these specific situations operate together as a system to enable specific gas species detection.

We take special precautions to observe and control the sensor temperature according to the specific gas we purpose to detect. We arrange by the design of the sensor structure and by feedback control for our catalytic surface to be at an essentially uniform and particular temperature.

We do not derive target gas concentration from the magnitude of sensor temperature change brought about by the intensity of combustion (the offset detection method discussed earlier). Rather, we derive target gas concentration from the reduction in electrical power required to maintain the catalytic surface at a specific temperature (the null-balance detection method).

We do these things independently for each of as many elements as may be operating in a transducer serving to detect various specific target gases by multi-differential calorimetry. Also, we may *vary* the temperature of a catalytic surface in order to specifically sense *different* target gases with the *same catalyst* by means of differential calorimetric thermal spectroscopy.

Others make catalytic sensors intended to initiate combustion and to use the intensity of any ensuing combustion (as indicated by the general temperature rise in the sensing catalyst and its nearby supporting structure) as the indication of target gas concentration. Their approach has no need for observing only a very specific region of temperature within their sensor's structure. Their sensing structures do not provide the means to respond *only* to the temperature of the catalytic area alone. The supporting structures developed by others that typically have resistance temperature detection means that include resistance variations *along the entire length* of their resistance temperature detection means.

Sensor Tech could operate these traditional sensors by using our catalytic chemistry and signal conditioning approach. However, the resulting indications are not likely to be gas species specific. To achieve the greatest detection specificity, Sensor Tech observes the electrical resistance change in *only* the isothermal region of the sensor where catalytic activity is enabled.

In Sensor Tech's specific gas detection technology, it is the temperature of a very specific catalytic region that matters. This temperature is used to effect feedback control and can be indicated by thermoelectric thermometry, resistance thermometry or any other thermometry means so long as the indicated temperature is derived *from, and only from,* this very specific catalytic region.

Sensor Tech typically observes the temperature of only the region of the heater that is in intimate contact with and at essentially the same temperature as our catalytic surface. Our preferred means for doing this has been by means of resistance thermometry by employing carefully designed 4-wire Kelvin sensing circuitry.

Technical Problems

IR screening of catalyst libraries produced by combinatorial material synthesis

Sensor Tech's proposed approach to screening a combinatorial library for catalytic activity level by using IR video techniques proved to be non-trivial to implement. It is difficult to achieve a sufficiently uniform temperature over a large fraction of an alumina sheet.

A successful alternative has been depositing combinatorial libraries on an array of nickel wires formed into individual coils that can be mounted in connectors for insertion into a sample gas flow. We then compare the responses of the sensors representing various material combinations achieved by co-sputtering. While useful for identifying catalyst activity, this configuration lacks the required specificity for field use. We were unable to progress beyond wire-based elements for variable resistance heating elements because our resources were limited.

Moisture contamination

Early attempts at coating wires with catalytic oxides did not yield useful sensing devices. Moisture present during a sensor seasoning protocol was identified as the cause.

Control crosstalk due to low thermal resistance to radiation heat transfer

Early mounting schemes for sensing and reference elements based on nickel wire coils employed ceramic material to act as a shield for heat transfer between the elements. However, this ceramic material did not provide a sufficiently high thermal resistance to radiation heat transfer between the sensing and reference elements. This situation enabled excessive thermal crosstalk between sensing and reference elements which had the effect of confusing the sensor and reference temperature control systems, thereby producing invalid data.

Gold-coated aluminum foil was substituted for the ceramic thermal shield material. This thermal shielding approach markedly increased the resistance to radiation heat transfer between the nickel wire coil sensing and reference elements. This increase in thermal resistance enabled the acquisition of valid differential calorimetric measurements as the sensor and reference temperatures were varied during transducer characterization.

When Sensor Tech recognized that we were working with invalid data we requested a no-cost extension to the contract to provide time to resolve the invalid data matter. Fortunately, the issue was resolved rather quickly and useful work was able to proceed after only a short delay.

Insufficiently isothermal operation of the catalyst

We attach catalytic material to an electrical resistance that increases in resistance as its temperature increases and use an electrical current to heat this assembly to bring the catalyst material to its desired operating temperature. The variable-resistance heater (VRH) can take many different forms.

One convenient form for a VRH is a suitable length of nickel wire arranged in a coil with space between its turns and attached to a standard electrical connector.

The overriding advantage of using wire-based sensors at this stage is their ease of production and substantial catalytic response. The disadvantage of wire-based sensors is the wide power bandwidth of their response to catalytic activity.

A heated wire is hottest at its center and decreases in temperature from either side of the wire's center to its lowest temperature at the wire's mounting structure. This results in catalyst material operating over a range of temperatures, hottest at the center and cooler as the wire mounting structure is approached from either side of its center. With increasing electrical power applied to the wire, a response is first detected at the hottest location, the center of the wire. Additional power applied to the wire moves the responsive zone outward to twin locations on either side of the center that are now at the most responsive temperature. This is why a particular catalytic response is observed in a wire sensor over a wide range of applied electrical power.

Best sensitivity and specificity is achieved when the catalytic material is all at essentially the same temperature. The performance of a wire-based sensor is limited by the temperature variations along the wire.

We have verified that a heater using a tapped serpentine pattern on an alumina substrate can be vastly superior to a wire heater because it can maintain a uniform catalyst temperature in the specific area where its electrical resistance is observed using the Kelvin (4-wire) resistance measurement circuit topology. This configuration yields a very sharp catalytic power response with respect to the sensor electrical resistance observed between the serpentine taps.

Sensor Tech is transitioning to the tapped-serpentine-on-alumina sensor structure for upcoming work. However, we could not obtain enough alumina sensors for statistically relevant results with the time and resources still available within the scope of this effort. So we will present data from wire sensors in this Final Report.

General Methodology

The proposed methodology was to deposit catalytic material libraries by means of combinatorial material synthesis onto thin ceramic sheets that were some 10 in² in area and screen these libraries for catalytic response by heating them uniformly and changing their temperature as the sample gas flowed over the catalyst. We planned to observe the catalytic activity by using IR imagery and identify specific compounds that performed well using SEM with EDS and XPS. As discussed above, this approach did not prove to be useful. We eventually produced useful combinatorial libraries on nickel wire coils.

URI, Kingston RI and HPI, Ayer, MA, prepared a set of sensors with different copper-iron oxide catalysts and URI determined which catalyst combinations were most responsive to various target gases.

Responsive oxide catalyst combinations of oxides of iron and copper as well as single oxides of iron and copper deposited on nickel wire coils were analyzed using SEM with EDS and XPS to determine the composition of the most responsive libraries.

Using combinatorial material synthesis, the most responsive “catalyst libraries” were screened for catalytic activity using integrated power curves that were established for each catalyst-coated coil. The most responsive libraries were tested using the latest version of the Sensor Tech signal conditioning and data acquisition instrument and later analyzed for chemical composition using SEM with EDS and XPS.

The coil transducers (catalyst supports) were produced from very fine nickel wires, which were subsequently annealed in ultra low oxygen partial pressures and then coated with different copper oxide-iron oxide combinations using co-sputtering techniques. In this way, chemical composition gradients are produced in substrate space that are spatially dependent on the distance from the respective sputtering targets.

The catalyst-coated transducers were placed in the test fixture with a gold-plated aluminum foil separator that is used to thermally isolate the reference coil from the catalyst-coated transducer such that extremely small changes in power could be sensed and quantified. The small differences in power required to keep the catalyst and reference coils at the same temperature determine the resolution limit and sensitivity of the measurement.

Technical Results

Summary

The combinational material synthesis approach for catalyst development yielded good results.

IR screening techniques hold significant promise for catalyst library screening, but were not useful within the scope of this project.

The most optimally-responsive catalytically active material for reacting with Ethanol gas was 100% CuO. The most optimally-responsive catalytically active material for reacting with Ammonia gas was a iron oxide-copper oxide combination.

Detailed results

There are several steps involved when depositing a useful catalytic surface and each has exacting requirements for process time, device temperature, surrounding gases, etc. The process must be under the control of a detailed protocol in order to achieve consistent results. We use the term ‘seasoning’ to refer to the protocol that takes the mechanically finished device through the subsequent steps that make the catalytic surface useful.

Integrated power curves were established for each catalyst-coated microheater using the original Sensor Tech signal conditioning instrument. In this manner, catalyst response is being correlated with catalyst chemistry.

Using this sensor platform and testing protocol, a number of catalyst libraries were screened. Test results from five catalyst libraries after repeated thermal scanning are shown in Figure 1 below.

A test fixture used in these laboratory tests is pictured in Figure 2. It is a 15.24 cm (6 in) length of plastic water pipe with a 2.54 cm (1 in) inside diameter. A longitudinal hole is cut in the center of its length to accept a DB-9 connector. A nickel wire coated with a catalyst library serves as a sensing element and a bare nickel wire coil serves as a reference element. These two coils are mounted to the solder cups of the connector and a gold-coated aluminum foil sheet is positioned to act as a thermal shield between the coils. This assembly of wires and thermal shield are inserted through the opening in the side of the pipe and sealed with museum putty. The target gas mixture arrives and departs through one-hole stoppers at either end of the pipe.

While effective and convenient for laboratory tests, this test fixture is obviously inappropriate for field use. We were limited by the scope of the contract to this mechanical arrangement for gas sensing transducers.

SEM images of these libraries are included as Figure 3. The responsiveness of each catalyst (combinatorial) library was based on the following criteria: (1) the lowest threshold temperature for which a power difference could be measured (or an indirect measurement of temperature such as electrical resistance) and (2) the development of fine structure in the resulting spectra.

Based on these criteria, Coil #4 was determined to be the most responsive catalyst library of those investigated in the catalyst system having varying ratios of iron oxide-copper oxide. When using ammonia as the target gas, Coil #4 exhibited the lowest threshold temperature of any catalyst library and also showed considerable fine structure in the electrical resistance (temperature) spectra. It is worth noting that a sign change in the power response between coils #3 and #4 was observed that might be explained in terms of the difference in thermodynamic response for the various oxides; i.e. exothermic vs endothermic behavior. A drop in power was observed for coils #1, #2 and #3 at the threshold condition consistent with an exothermic response whereas an increase in power was required to keep coil #4 at the same temperature as the reference, consistent with an endothermic response.

The chemical composition of the various catalyst libraries was analyzed using SEM with EDS, and the results are summarized in Table 1 below. This analysis suggests that the copper oxide-rich catalysts are more sensitive to ammonia than the iron oxide-rich catalysts and that additional, higher-resolution combinatorial synthesis might yield new combinations with even greater response and sensitivity. When similarly prepared catalyst libraries were used to detect the presence of ethanol vapor in the gas phase, chemical analysis yielded another important finding. EDS analysis of the reference coils indicated that there was considerable carbon residue on the surface of the coils relative to the catalyst coated coils; i.e. EDS analysis of the catalyst coated coils revealed that only trace amounts of carbon were present on the

surface. This finding further supports the premise that the oxides play a key role in the catalytic activity of the sensors, whether prepared from individual oxide catalysts or combinations of oxides using co-sputtering methods, all of which suggests that further studies using XPS are warranted to better understand the nature of the chemical bonds and responsiveness of the oxides using this sensor platform.

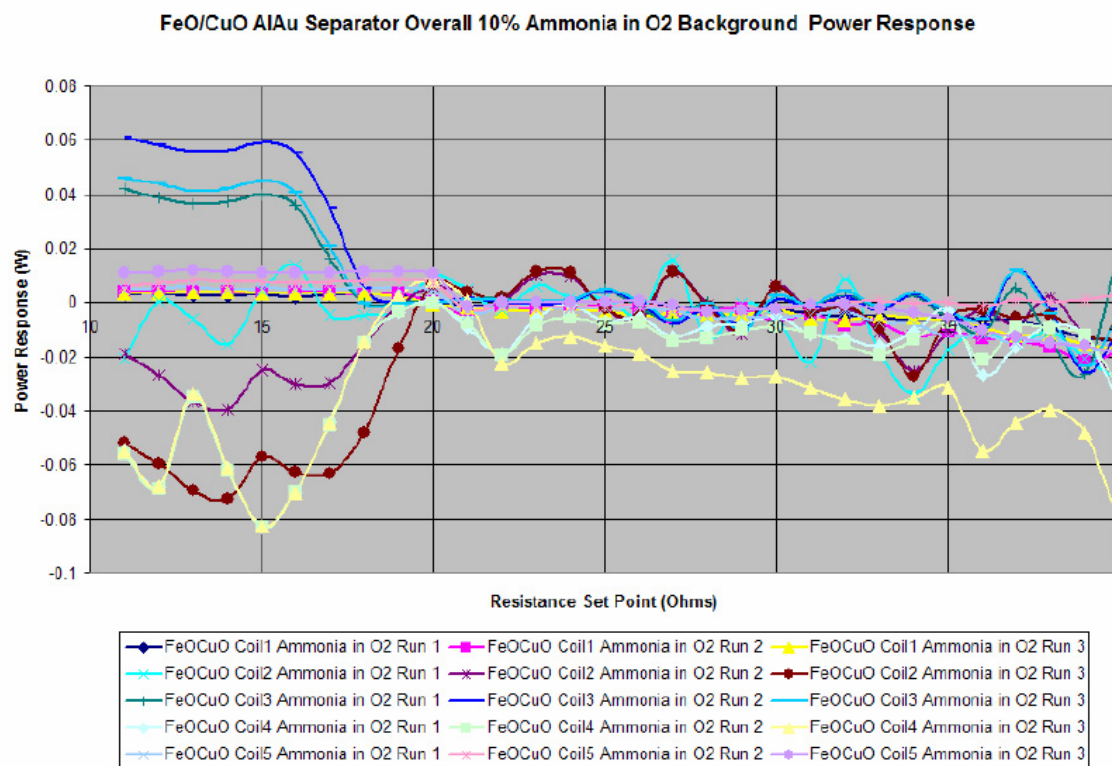


Figure 1. Thermal response of five iron oxide-copper oxide catalyst combinatorial libraries.

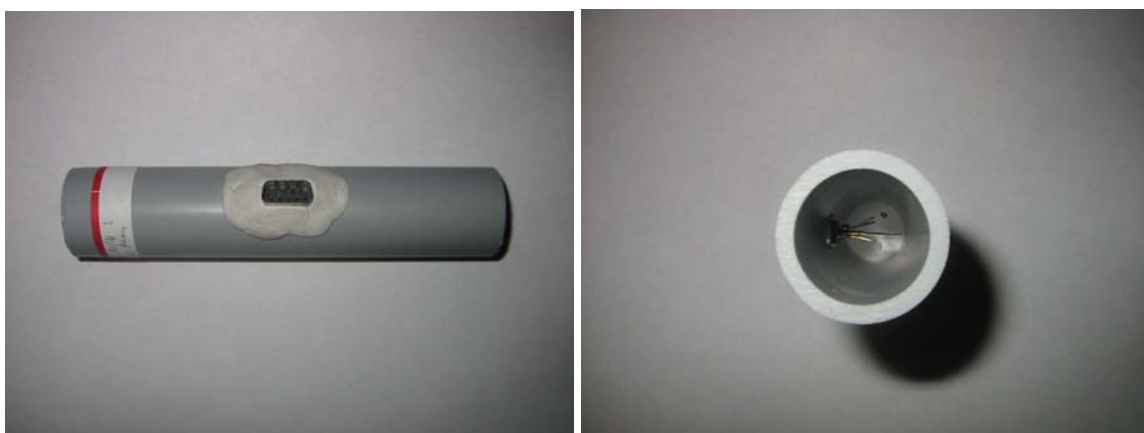


Figure 2 Catalyst test fixture
length = 15.24 cm (6 in), inside diameter = 2.54 cm (1 in), DB-9 connector.

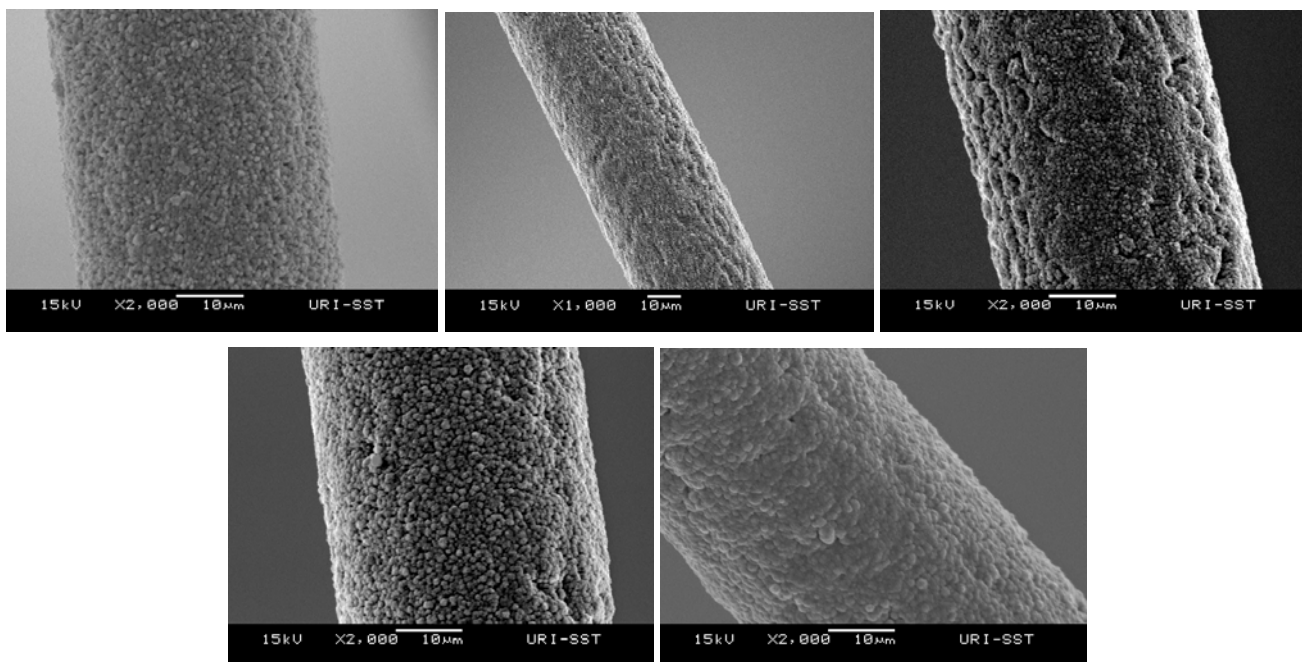


Figure 2. SEM Images nickel wire combinatorially coated with various mixtures of oxidized iron and copper, Coils 1 (upper left) through 5 (lower).

<u>Coil ID</u>	<u>Fe:Cu Composition (atomic %)</u>
1	88.0:12.0
2	33.0:67.0
3	6.00:94.0
4	0.50:99.5
5	0.01:99.9

Table 1. Chemical Composition of iron-copper oxide catalysts

Important Findings and Conclusions

Catalyst libraries deposited using combinatorial material synthesis can, but will not necessarily, yield transitional metal oxide catalyst compounds that are more responsive to a particular gas than the oxide of a single metal.

Catalyst library screening by means of IR area thermography to observe the heat transfer caused by catalytic activity did not prove useful within the scope of this contract. Another technique for screening catalyst libraries was developed and performed well, but it required depositing the library onto a set of individual sensing elements and independent signal conditioning electronics for each element. Screening with high-resolution libraries in this manner will require signal conditioning many elements simultaneously.

Careful and thorough documentation and implementation of all process and test protocols is imperative for obtaining consistent results.

Significant Hardware Development

A multi-sensor signal conditioning system for differential calorimetric operation of gas detection sensors was developed and used to obtain the data presented above. Previous signal conditioning could accomplish differential calorimetric spectroscopy with only two variable-resistance heating elements.

The new multi-channel device simultaneously and independently operates up to 16 VRH devices, providing each with over one watt of electrical power under closed-loop electrical resistance control. Each of the 16 elements can serve either a sensing or a reference element.

A LabVIEW program accomplishes operator interface, test scenario programming, three-mode direct digital feedback control and data recording. The operator enters numbers to define the proportional, differential and integral feed back parameters for each temperature control loop and can vary these parameters during a test run.

The design of this multi-sensor signal conditioning system is easily adaptable to operate as many sensing elements as may prove useful and to hand-held equipment operating only a few VRH devices.

Special Comments

While no patents were filed during the period of performance of this contract, future disclosure activity is anticipated as Sensor Tech's research continues under Army Research Office Contract Number W911NF-08-C-0059.

The gas detection transducers used to develop the results presented here are laboratory devices and not mechanically appropriate for typical field use.

The electronic signal conditioning used with the gas detection transducers was designed and produced to operate the gas detection transducers for laboratory testing. Sensor Tech anticipates modifying this hardware and software design for subsequent gas detection transducer designs and for use in the field.

Implications for Further Research and Development

There is a need for automated catalyst deposition and seasoning on sensing elements to achieve more uniform sensors. Sensor Tech expect this will reduce the cost of producing catalytic libraries for use in R&D and the cost of catalytic gas detecting transducers targeted to specific applications. Sensor Tech intends to pursue micro-machining methods and new combinatorial material synthesis methods to manufacture sensing elements for both purposes.

There is also a need for automated implementation of manufacturing and test protocols to maximize manufacturing productivity and testing consistency.

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