

# Nanoceramic MEMS Sensors for Universal Embedded Chemical Sensing

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## ABSTRACT

This paper describes recent developments in a nanoceramic MEMS platform used for chemical sensing. These advances enable the addition of environmental awareness to many other consumer and commercial devices, including smart phones.

Metal oxide semiconductor sensors are widely used as individual sensors and in arrays, and a variety of designs for low power microhotplates have been demonstrated.[1] Synkera Technologies has developed an embeddable chemical microsensor platform, based on a unique ceramic MEMS technology, for practical implementation in smart phones, other portable devices and wireless networks. Key features of this microsensor platform are (1) small size, (2) ultra-low power consumption, (3) high chemical sensitivity, (4) accurate response to a wide-range of chemicals, and (5) low cost. The sensor platform is enabled by a combination of advances in ceramic micromachining, and precision deposition of sensing films inside the high aspect ratio pores of anodic aluminum oxide (AAO).

**Keywords:** chemical sensor, air quality, gas detection, MEMs, nanotube

## 1 INTRODUCTION

Chemical sensors have long been used for the detection of industrial health and safety threats, as well as in a few select commercial and consumer level air quality applications, such as the detection of carbon monoxide (CO) and air quality contaminants, including volatile organic compounds (VOCs). While the performance of existing commercial devices is adequate for the intended applications, the cost, size and power prohibit many high volume applications.

Recent work by Synkera has focused on the miniaturization of the sensors, to enable a wide range of new applications, including use in portable and wireless devices, such as smart phones. Smart phones are an ideal host for chemical sensors, due to their ubiquitous presence, powerful computing capability, and ability to automatically summon help in the event the user is overcome from exposure to a hazardous gas, such as carbon monoxide.

## 2 EXPERIMENTAL

Synkera's approach to the development of a chemical microsensor platform for low power embedded applications

lies in the use of a micromachined, nanostructured ceramic, AAO. The resulting microsensor platform benefits from advances in nanotechnology and materials chemistry that facilitate robust and reliable detection of a wide range of chemicals. The nanostructured ceramic is AAO (Figure 1).

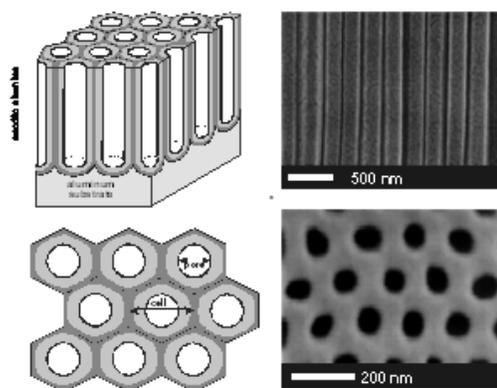


Figure 1. Cartoon and SEM image of anodic aluminum oxide (AAO), showing nanoporous structure. Top: Cross section. Bottom: surface image.

The nanoporous architecture of AAO is achieved via anodization of aluminum foils under precisely controlled conditions. Synkera has developed processes for well controlled fabrication of AAO featuring a range of parameters, including pore diameters of 5 to 300 nm. This platform is ideal for fabrication of MOS nanotubes for sensing (Figure 2). Further processing of nanoporous AAO via ceramic micromachining allows for multi-scale control - from nanometers to hundreds of micrometers - of the resulting ceramic MEMS components. Synkera leverages this capability to fabricate ultra-low power ceramic micro-hotplates, which in turn form the basis of its chemical microsensors.[2] The heating/sensing element is approximately  $100\mu\text{m}^2$ , robust to high temperature ( $>1000^\circ\text{C}$ ), and capable of high-frequency temperature modulation.

Ultra-thin films of semiconducting metal oxide are deposited throughout the nanopores of the micro-hotplate, and electrodes are used to detect the change in resistance of these films in response to chemical exposure.[3]

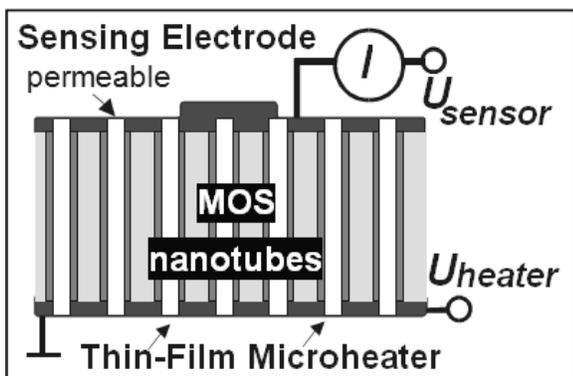


Figure 2. Schematic of AAO microhotplate.

After microheater fabrication, the sensing material(s) can be applied using either gas phase or liquid phase derived methods. In this work, metal oxide (e.g. tin oxide [4]) materials were deposited into nano-pores of the AAO. Metal oxide precursors, such as alkoxides, acetates or chlorides are dissolved in appropriate solvents. The resulting solutions are applied to the substrate, dried, and thermally treated to produce the desired metal oxide. By varying the concentration of the solution and the number of applications and the deposition method, the amount of metal oxide deposited can be controlled. In this fashion, the walls of the pores in the AAO can be coated without completely filling the pores. Precise control of film thicknesses results in outstanding reproducibility and sensitivity. Temperature modulation of the sensor films enables partially selective sensors and arrays.

Sensors can be packaged in a wide range of packages, including leaded headers (for replaceable sensors in the end product) or surface mount packages (for smallest size), as shown in Figure 3.

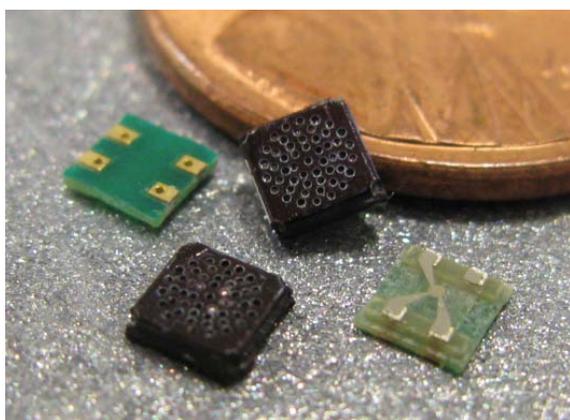
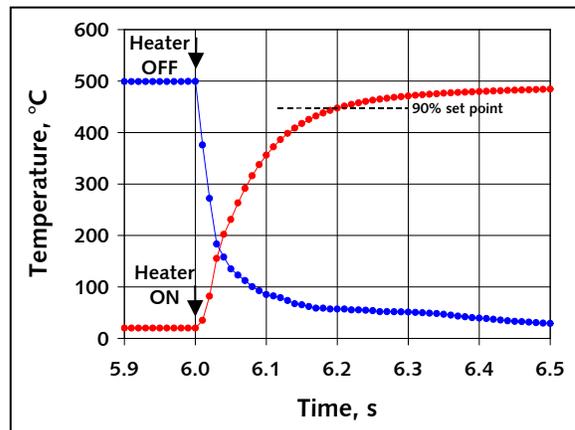


Figure 3. Nanoceramic microhotplates in SMT packages

### 3 RESULTS AND DISCUSSION

The use of the low mass ceramic microheater allows both very low power consumption and rapid heating/cooling of the Synkera microsensors, as shown in Figure 4. This rapid heating and cooling in turn enables dynamic thermal management (for increased sensitivity and selectivity), fast warm-up times and duty cycling for reduced overall power consumption, where the sensor is



kept off power for a majority of the time, and is heated only to make resistance measurements.

Figure 4. Rapid heating and cooling of the microsensors platform.

#### 3.1 Detection of chemical hazards

Our work has shown that extremely fine grained materials prepared inside the pores of AAO can result in detection limits ranging from part per billion (ppb), which is well below the commonly reported limits associated with metal oxide sensors, up to several percent.

In this paper, we focus on a hazard that is highly relevant to consumers. Carbon monoxide is an odorless, colorless tasteless gas that kills approximately 500 people in the United States alone every year.[5] The relevant levels of carbon monoxide for consumer applications, are in the range of 70 ppm (low alarm) and 400 ppm (high alarm). [6]

The response of a sensor to ppm levels of carbon monoxide is shown in Figure 5. The response function, as expected, is  $\log(\text{resistance})$  is linear with respect to  $\log(\text{concentration})$ . This large change in resistance is easily measured using common electronics.

The response of the sensor to a step change in the concentration of carbon monoxide is shown in Figure 6. The sensors are held in air for 5 minutes, then a source of 100 ppm carbon monoxide is introduced. After an exposure time of 10 minutes, the sensor background is again changed to air. In order to approximate an indoor application, the test gas background contains 45% humidity.

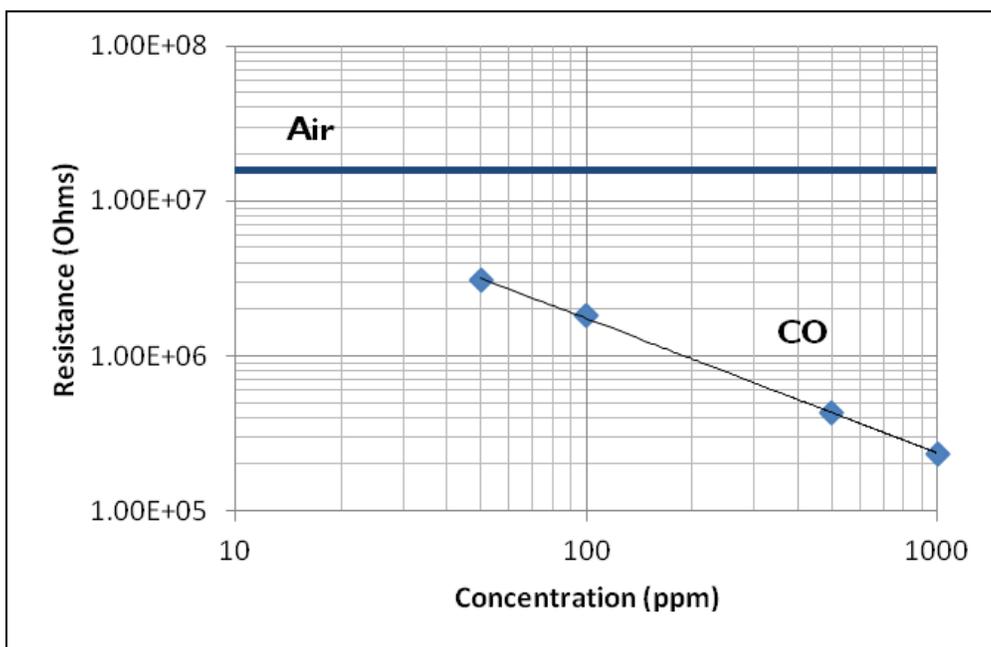


Figure 5. Sensor Response Function - (Log) Sensor Resistance is proportional to (Log) VOC (Ethanol) Concentration

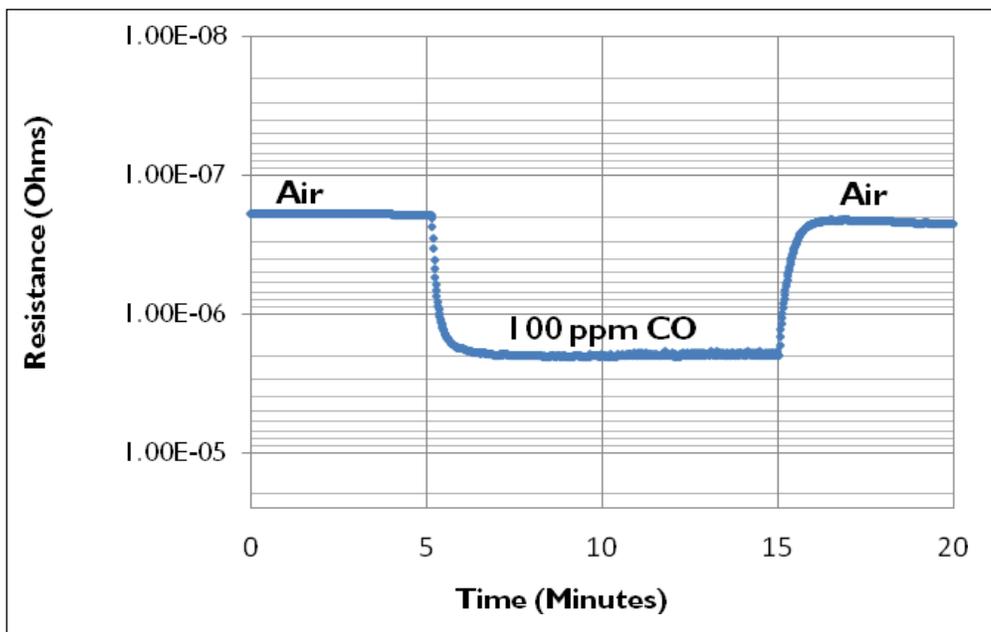


Figure 6. Sensor response to 100 ppm CO in humid air (45% RH) when operated continuously at 20 mW

### 3.2 Reducing power consumption

In order to reduce power consumption, we have experimented with duty cycling the sensor, or turning the heater on only to make a measurement of the carbon monoxide level. Prior studies have shown that the length of the heater on pulse should be in the range of 0.5 to 1 second, in order to allow stabilization of the sensing films.

An example of the sensor response when the heater is turned on is shown in Figure 7. In this plot, the heater is on from approximately 94 seconds to 95 seconds, for 1 second total. The sensor output stabilizes in approximately 0.5 seconds.

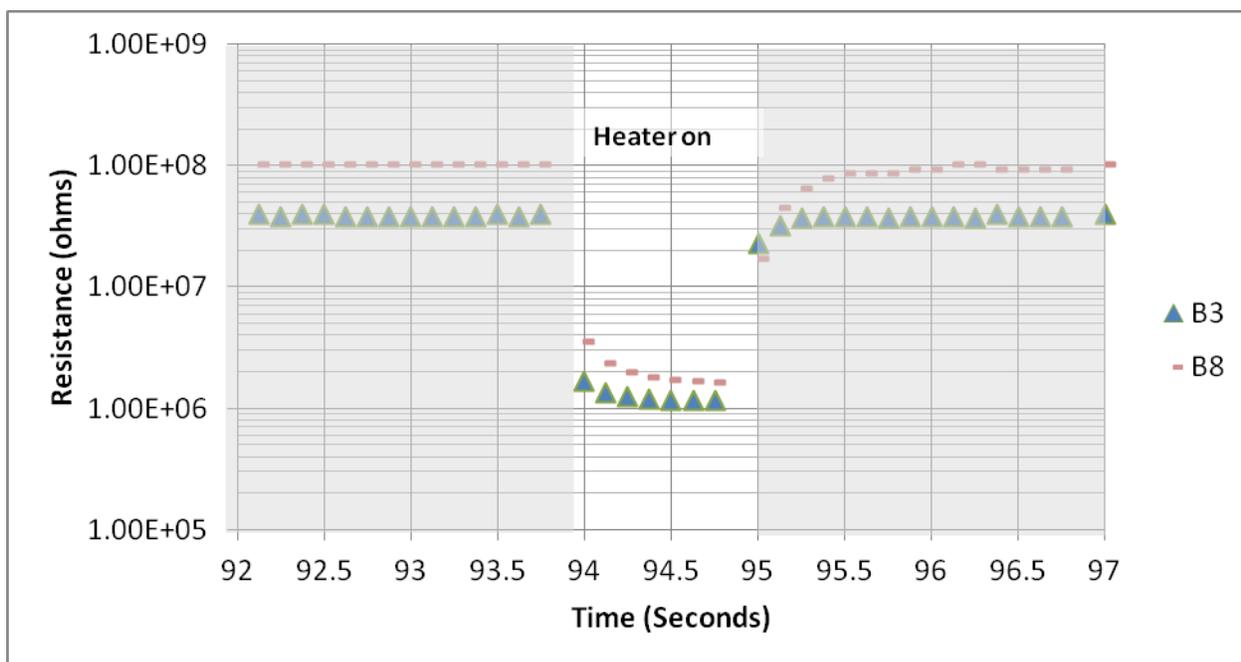


Figure 7. Sensor Response during heater “on” pulse.

A summary of the parameters which have been successfully explored for detection of carbon monoxide are shown in Table 1.

Table 1: Summary of conditions used for pulsing.

Period	10 to 60 seconds
Peak Power	15 to 25 mW
Length of Heater Pulse	0.5 to 2 seconds
Average Power Consumption	<0.5 mW to 5 mW

#### 4 CONCLUSIONS

In this work, Synkera has shown a unique, nanostructured ceramic MEMS platform that offers outstanding, reliable performance in a highly miniature, low cost, low power design. This paper presents results of the detection of carbon monoxide; however, the sensors have been shown to detect a wide range of hazards, including volatile organic compounds (VOC’s), ethanol, flammable gases and hydrogen sulfide. Future work will focus on other components of air quality, including carbon dioxide, ozone, NOx and many other gases.

#### 5 ACKNOWLEDGEMENTS

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#### REFERENCES

- [1] S. Semancik, et al, Sensors & Actuators B: Chemical 77, 579-591 (2001).
- [2] D. Routkevitch, et al, U.S. Patent No. 6,705,152 B2, granted 03/16/04, filed 10/24/2000.
- [3] D. Routkevitch, et al, “Anodic Alumina MEMS: Applications and devices” in MEMS – 2000, Proc. ASME Int. Mech. Eng. Congress 2000, Orlando, FL, Vol. 2, ASME, New York, pp. 313-318, 2000.
- 4 A. Govyadinov, P. Mardilovich, K. Novogradez, S. Hooker, D. Routkevitch, Anodic Alumina MEMS: Applications and devices, MEMS - 2000, Proc. ASME Int. Mech. Eng. Congress, Nov. 5-10, 2000, Orlando, Florida, Vol. 2, ASME, New York, 313-318 (2000).
5. <http://www.ul.com/global/eng/pages/offerings/perspectives/consumer/productsafety/co/>, accessed March 10, 2013
- 6 UL 2034, Standard for Single and Multiple Station Carbon Monoxide Alarms