

Micromachined Combustible Gas Detector

Overview

In collaboration with the University of Utah, MIT, and the University of New Mexico, Sandia National Laboratories' Microsensors S&T department has modeled, designed, fabricated, and tested a combustible gas sensor that uses a resistively heated, noble metal-coated, micromachined polycrystalline Si filament to calorimetrically detect the presence and concentration of combustible gases. A thin catalytic Pt film was selectively deposited by chemical vapor deposition (CVD) onto resistively heated microfilaments. Using a constant-resistance-mode feedback circuit, Pt-coated filaments operating at ca. 300° (35 mW input power) respond linearly to H₂ concentrations between 100 ppm and 1% in air.

Operational Concept and Applications

Combustible gases such as methane, hydrogen, and the vapors of gasoline and other fuels pose a threat to safety when they accumulate at explosive concentrations. A relatively well developed technology for the detection of such combustible species in air is the use of a heated noble metal filament, typically Pt, the temperature of which increases when combustion occurs on its surface. A slightly more complex variation of this device is known as a "pellistor": the Pt wire is embedded in a high-surface-area ceramic pellet, which may itself contain catalytic metal particles. With any of these devices, the change in filament resistance resulting from a temperature increase

due to combustion on the filament or pellet surface is detected to indicate the presence and concentration of combustible gases.

We are miniaturizing this technology, taking advantage of the economy of silicon microfabrication technology and the feasibility of integrating control and measurement circuitry, in addition to utilizing small size as a means to reduce power consumption and response time. We are utilizing surface micromachining technology, fabricating microfilaments from polycrystalline Si films, with the finished microfilaments separated from the underlying Si substrate by a gap to provide thermal isolation. Similar microfilaments have been explored as potential incandescent light sources and hot-wire anemometers, but we are the first to exploit them for chemical sensing purposes.

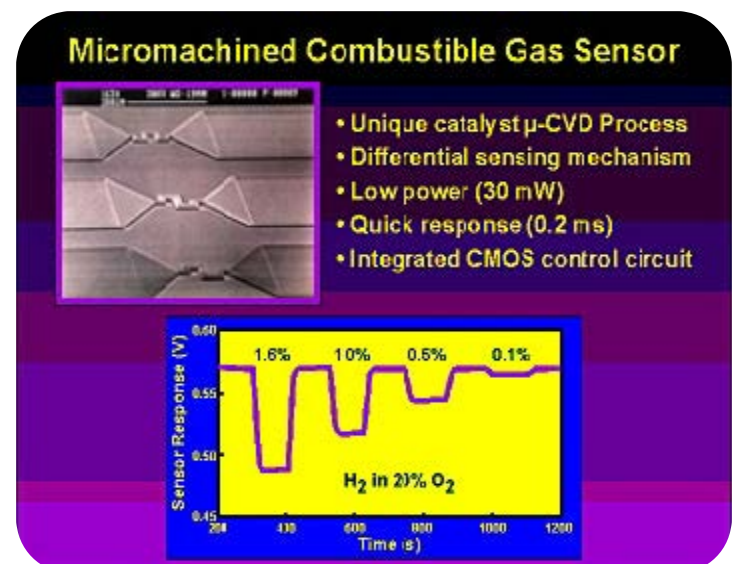


Figure 1. Micromachined Combustible Gas Sensor.

Our design strategy includes a 0.25 μ CVD Si₃N₄ overlayer to protect the poly-Si filaments from long-term thermal oxidation, which would occur at the elevated temperatures required for the detection of some combustible species. The filaments are 2 μ thick x 10 μ wide, ranging from 0.1 to 1 mm in length; several are shown in Figure 1.

To catalyze the combustion process, a thin film of noble metal is deposited using "microCVD": one or more selected filaments is heated resistively (to approximately 500°) in an inert gaseous ambient that includes a volatile organometallic precursor for the desired noble metal. We have used Pt(acac)₂ as the precursor. Metal deposition occurs only on the heated filament(s), allowing different metals and/or different film thicknesses to be deposited on each filament. We have discovered that control of the filament temperature, the total time of deposition, and particularly the nature of the duty cycle (ratio of "on time" to "off time", as well as the absolute values of the on time and off time), during catalyst deposition provides a means to control the morphology and microstructure of the catalyst layer.

There are two keys to the effective operation of the catalytic microfilament gas sensor: (1) the use of a feedback-based control circuit to keep the microfilament at a constant temperature at all times; (2) measurement based on the use of a differential pair of filaments, one of which bears a catalytic coating to facilitate surface combustion of the species to be detected, the other passivated so that no combustion occurs on its surface. The circuit we use maintains a constant temperature by adjusting the supplied current to keep the total filament resistance at a constant value (temperature and resistance are directly related). There are two main reasons for operating both the sensing filament and its passivated reference filament at the same, constant temperature: first, variations in ambient conditions, including but not limited to gas

flow rate, gas thermal conductivity, temperature, and relative humidity affect the rate of heat loss of both the sensing and reference filaments in precisely the same manner, so that the changes in input power to the sensing and reference filaments that occur as a consequence of such ambient variations will be equal and therefore cancel one another when the two filaments are operated in a differential mode; second, identical thermal histories increases the likelihood that any gradual aging of the sensing microfilament will be mirrored in the reference microfilament, and therefore cancel. With operation in this differential mode, a sensor signal results only if combustion occurs on the catalytic microfilament, in which case the input power decreases in relation to the concentration of combustible species; no similar input power decrease is recorded for the passivated (noncatalytic) reference filament.

Results

- The control circuit was designed, built, and used successfully for the CVD of Pt onto a number of individual filaments and also for testing in combustible H₂/O₂ mixtures.
- Pt-coated filaments operating at ca. 300° (35 mW input power) were found to respond approximately linearly, in terms of the change in supply current required to maintain constant filament resistance (temperature), to H₂ concentrations between 100 ppm and 1% by volume in an 80/20 N₂/ O₂ mixture.
- An added benefit of the very low thermal mass of the microfilaments is rapid response time. The measured response time for the imposition of a current step that raises the temperature of the filament several hundred degrees above ambient is about 200 μ .

For additional information or questions, please email us at MGA@sandia.gov.