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# Metal-coated glass microfiber for concentration detection in gas mixtures using the 3-Omega excitation method

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ABSTRACT

The 3-Omega measurement technique was applied to a fabricated metal-coated glass fiber for the purpose of low power gas sensing. The sensor performance was evaluated for mixtures of $CO_2$, Ar, He and $CH_4$ in $N_2$ in an isothermal chamber, where mass flow controllers precisely controlled concentrations. The metal-coated fiber was fabricated by depositing a thin layer of gold (∼150 nm) onto a glass fiber, using a custom designed deposition lathe installed in a standard sputtering system. A custom 3-Omega conditioning circuit controls the AC heating current and detection of the 3-Omega voltage signal. The amplitude and phase lag, and the in-phase and out-of-phase components of the 3-Omega voltage signal are presented for different gas mixtures and are related directly to their concentrations. Using this gas sensing technique, we have demonstrated the uncertainty in concentration (*i.e.*, sensitivity) to be better than 50 ppm, and as low at 10 ppm for some gases. The dependence of the different 3-Omega signals on the thermophysical properties of the system is briefly described. The low power, high sensitivity nature of the sensor is also demonstrated as the metal-coated fiber sensor consumes ∼10% of the power consumed by conventional thermal conductivity detectors (TCDs), and unlike those, it operates at near room temperatures.


## 1. Introduction

Gas sensors have been widely used to determine the composition of gas mixtures and to detect the presence of a particular species in gases [1]. Thermal conductivity detectors (TCDs) are a common type of gas sensors, which use the difference in thermal conductivity of gases to detect the composition of gas mixtures. Miniature TCDs are highly desirable for gas chromatography due to their excellent sensitivity demonstrated to 1 ppm. Nanoscale bridge type sensors using nanotubes [2], nanowires [3], and nanobelts [4] have also been fabricated. These TCDs often require high power consumption (hundreds of milliWatts to a few Watts) to provide sensitivities in the few ppm range. Smaller variation of microTCDs are fabricated that offer better sensitivity and power consumption [5]. However some of these TCDs operate at high temperatures [6], which necessitate routine re-calibration and may be destructive to the detected sample. More recently, we have demonstrated the ability to perform low power gas sensing using the 3-Omega technique on a polysilicon microbridge geometry while still maintaining good sensitivity [7]. However, in the microbridge design a significant portion of the heat was conducted along the microbridge to the substrate, which reduced the heat input to the surrounding gas medium, thereby limiting the sensitivity of 3-Omega technique to 1000 ppm. Herein, we present a gas sensing technique that overcomes not only these challenges, but offers other advantages such as ease of fabrication, near room temperature measurements while still consuming power in the order of milliWatts. The design and fabrication technique of the sensor used in this work was first developed by Schiffres et al. [8] for the measurement of thermal conductivity of liquids and gases.

### 1.1. Principles of 3-Omega

The 3-Omega technique has been extensively used in thermal conductivity measurements [9] and is well described in literature [10], but it also offers an approach to low-power gas sensing that we have recently investigated [7]. In a standard 3-Omega measurement, a sinusoidal electrical current of amplitude $I_\omega$ at frequency $\omega = 2\pi f$ is driven through a metal heater line, resulting in a Joule heating at frequency $2\omega$. The periodic heating creates a thermal wave that penetrates the surrounding medium. The thermal wave attenuates over a penetration depth, given by $L_p = \sqrt{\alpha/2\omega}$, where $\alpha$ is the thermal diffusivity of the surrounding medium. This gives rise to a temperature oscillation at the source, which has a fre-

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**Table 1**
Dimensions and properties of the different layers of the metal-coater fiber sensor[8]. The values for the fiber layers were provided by the manufacturer and the properties of the titanium and gold layers are obtained from Ref. [11].

| | Layer 1 (Schott F2[a] leaded glass) | Layer 2 (Schott 8250[a] borosilicate) | Layer 3 (Titanium) | Layer 4 (Gold) |
|---|---|---|---|---|
| Diameter (d)/Thickness (t) | d = 24 μm | t = 1.1 μm | t = 10 nm | t = 150 nm |
| Thermal Conductivity (W/m-K) | 0.78 | 1.18 | 21.9 | 317 |
| Density (kg/m$^3$) | 3600 | 2280 | 4500 | 19300 |
| Specific Heat (J/kg-K) | 557 | 720 | 522 | 129 |

[a] Properties and dimensions were supplied by the manufacturer.

quency $2\omega$ but lags the heating current by a phase lag $\varphi$ due to the finite time it takes for a temperature response. The temperature oscillation causes the resistance of the heater to oscillate at $2\omega$. Since the current is driven at $1\omega$, this resistance oscillation results in a voltage component at $3\omega$. The amplitude, $V_{3\omega}$ and phase lag, $\varphi$ of the voltage signal can be directly measured and the voltage amplitude can be directly related to the amplitude of temperature oscillation by Eq. (1).

$$\Delta T_{2\omega} = \frac{2}{dR/dT} \cdot \frac{V_{3\omega,\mathrm{RMS}}}{I_{\omega,\mathrm{RMS}}} \quad (1)$$

where $dR/dT$ is the temperature rate of change of the sensor's resistance and the subscript RMS refers to the root-mean-square value.

The amplitude and phase signals can be directly related to the thermal properties (*e.g.,* thermal diffusivity) of the sensor and the gas medium. Since the thermal diffusivity is a function of the gas mixture, the 3-Omega signals vary with gas composition and can be used to resolve gas compositions. The phase lag is only a function of the geometry and thermophysical properties of the heater and its environment, and is independent of the amplitude of driving current thus uniquely alleviating the need for routine calibration.

## 2. The 3-Omega sensor

In our previous study on 3-Omega gas sensing, a polysilicon microbridge suspended on a silicon dioxide sacrificial layer was used which could readily resolve gas concentrations of 1000 ppm in binary mixtures [7]. Since a significant portion of the heat was conducted within the microbridge and dissipated to the solid substrate, the sensitivity to the gas medium was limited. In this study, a glass fiber coated with a thin layer of metal (gold) is used as the 3-Omega heater and the sensing element. The raw glass fiber has a 26 μm diameter, and consists of a leaded glass core (Schott F2) and a borosilicate cladding (Schott 8250) [8]. The diameters and properties of the different layers are provided in Table 1.

### 2.1. Fabrication of sensor

The metal layer was deposited using a conventional sputtering system (Unifilm Multisource Sputtering System, IEN, Georgia Tech). To achieve uniform cylindrical coating, the fibers were strung tautly onto a spool, which was uniformly rotated with the motor in the sputtering system as shown in Fig. 1 [8]. The deposition lathe ensured uniform circumferential coating. Since the deposition crystal monitor is calibrated for deposition in a rectangular plane, the thickness deposited on the sensors is smaller by a factor of π, which results from the ratio of the fiber's actual surface area (π × diameter × length) to its projected area (diameter × length). The sputterer was programmed to deposit a 10 nm titanium adhesion layer, followed by a 150 nm of gold. The fiber was characterized using a SEM to determine the exact thickness and to verify the uniformity of the gold layer along the circumference of the fiber. This was done by measuring the metal layer's thickness at over 10 points along the fiber's circumference. The resulting thickness of the metal

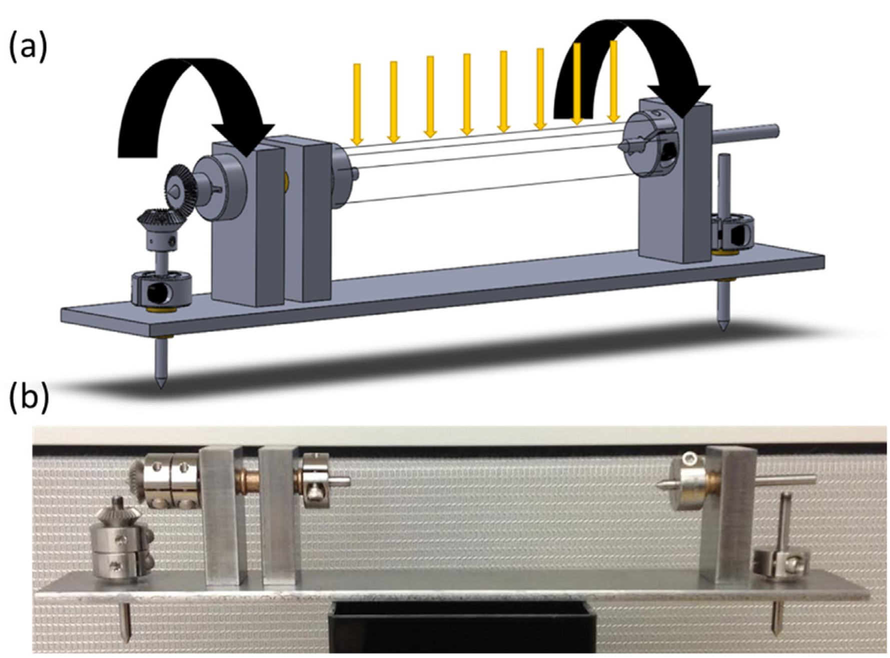


**Fig. 1.** (a) Schematic showing the deposition lathe with fibers strung onto the rotating spool. The black arrows indicate the direction of rotation of the spool and the golden arrows indicate the direction of gold sputtering. A bevel gear is used to convert the plane of rotation from the default horizontal plane to the vertical one with the spool. (b) A picture of the deposition lathe used.