Micro Sensors for Real-time Monitoring of Mold Spores and Pollen

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Keywords: Mold Spore, Pollen, Real–time Monitoring, Micro Sensor, Semiconductor Thin Film, Metal Oxide.

Abstract: Organic airborne particles such as mold spores and pollen cause a variety of diseases. Two types of micro sensors for real-time monitoring of such organic airborne particles have been developed using semiconductor thin-film. A basic type thin-film sensor has a simple configuration with a double-layered sensing film deposited on an alumina substrate. A MEMS type sensor is composed of two parts: a sensing element and a micro heater. Both parts are fabricated by using thin film technology, IC fabrication process and micromachining technique. The double-layered sensing film is deposited on a diaphragm formed on a Si substrate. A thin film heater is placed in parallel at a distance of about 50 μm. The resistance of both sensors steeply decreases and then recovers to the initial value when a mold spore or a grain of pollen adheres to the surface of the sensing film and burns on it. The resistance change and the recovery time depend on the size of the organic airborne particles. Thus it is possible to identify the species of the particle by the developed sensors. The sensors offer simple and inexpensive method to monitor organic airborne materials.

1 INTRODUCTION

Many people fall in pneumonia by inhalation of mold spores such as aspergillus fumigatus. At the same time, the number of people who are allergic to organic airborne particles such as pollen and house dust has been increasing. Although human noses are sensitive to some toxic gases and odors such as burnt odor and bad smell, human noses cannot detect organic airborne particles. Therefore, it is required to develop sensors for these airborne particles.

Some methods have been developed to monitor airborne materials so far. Various types of samplers are usually used to count the number of airborne mold spores (Hoisington et al., 2014; Whyte et al., 2007). However, they are disadvantageous in that it takes several days to culture them on agar. The gravitational method by a Durham's sampler is a common one to obtain the number of pollen (Konishi et al., 2014). It also takes a lot of time to get the number because they are observed by the human eye through an optical microscope. These two methods are not fit for real-time monitoring of airborne materials. Particle counters with laser optics are sometimes used (Weber et al., 2012). However, they cannot distinguish organic airborne particles from inorganic particles such as ashes and sands. In addition, they are complex and expensive in general.

This paper describes novel, inexpensive micro sensors that are capable of detecting organic airborne particles such as mold spores and pollen. Two types of micro sensors have been developed: a basic type sensor and a MEMS type sensor. The former has a simple structure with a relatively large sensing area that is suitable for detection of larger airborne particles. The latter has a smaller sensing area that is better suited for detection of smaller airborne particles.

Both sensors have configurations which are similar to thin-film gas sensors (Brunet et al., 2012; Sharma et al., 2011). In addition, similar sensing materials based on semiconductor metal oxides such as SnO_2 and Fe_2O_3 were used for the developed sensors. In general, these metal oxides are stable at elevated temperatures and their resistance changes by the redox reaction on the surface when a reducing gas or an oxidizing gas is introduced. In ordinary gas sensors, greater sensing area is better because it increases the sensitivity to these gases. In this study, however, the sensing area was reduced to fit for detection of fine airborne particles.

Tsuruzoe K. and Hara K..
Micro Sensors for Real-time Monitoring of Mold Spores and Pollen.
DOI: 10.5220/0005279301740179
In *Proceedings of the International Conference on Biomedical Electronics and Devices* (BIODEVICES-2015), pages 174-179
ISBN: 978-989-758-071-0

2 EXPERIMENTAL

2.1 Sensor Configuration

A schematic top and cross-sectional view of a basic type micro sensor is shown in Figure 1. A sensing film was deposited on an Al_2O_3 substrate. The sensing film had a double-layered structure. The first layer was Fe_2O_3+TiO_2 (5 mol%) + MgO (4 mol%)

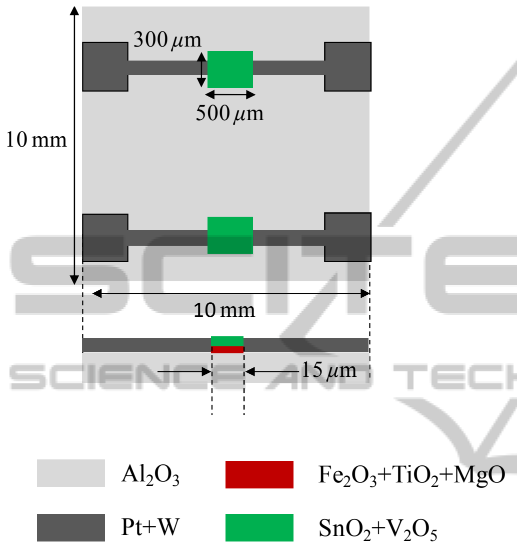

Figure 1: Schematic top and cross-sectional view of a basic type sensor.

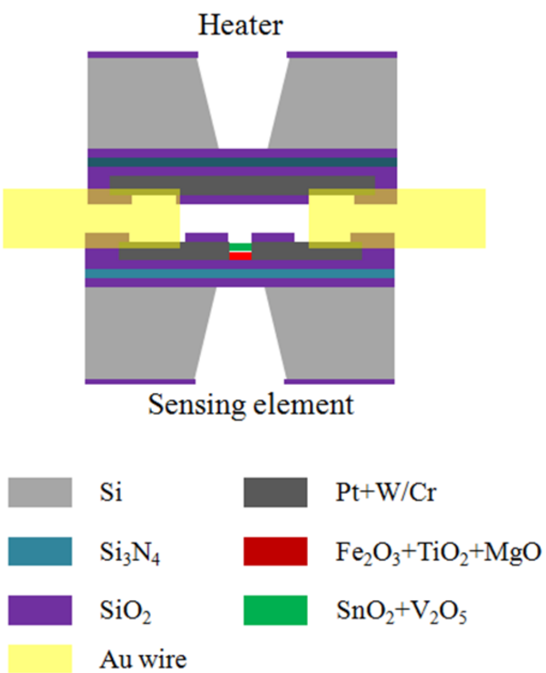

Figure 2: Schematic cross-sectional view of a MEMS type sensor.

and the second layer was SnO_2 + V_2O_5 (4 mol%). Its sensitivity and stability have been improved by adopting a double-layered structure (Hiwatari and Hara, 1998). The thickness of the first and the second layers was 100nm and 100nm, respectively. The length and the width of the sensing film between the electrodes were 15 μm and 100 μm, respectively. The sensor was heated by a commercially available Pt heater covered by alumina ceramics when the sensor response was tested.

A schematic cross-sectional view of a MEMS type micro sensor is shown in Figure 2. The sensor is composed of two parts: a sensing element and a micro heater. A sensing film was deposited on a SiO_2/Si_3N_4/SiO_2 diaphragm formed on a Si substrate. A thin film heater was also made on a similar diaphragm formed on another Si substrate. The sensing element and the micro heater were placed in parallel at a distance of about 50 μm by inserting gold wires.

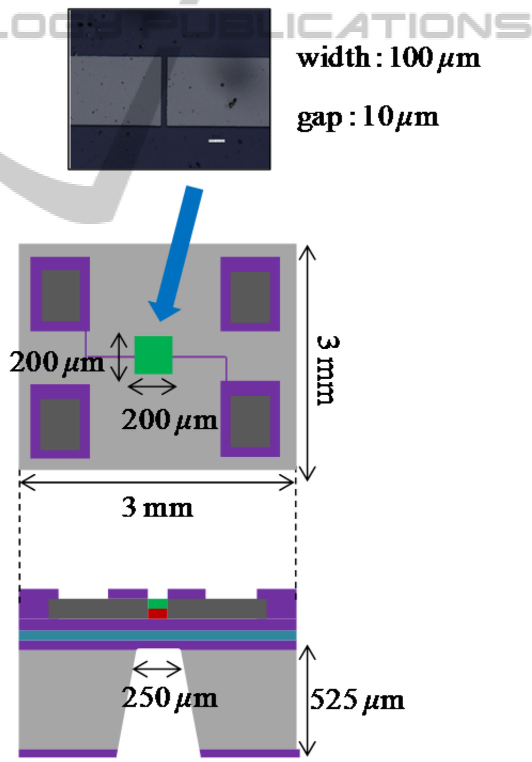

Figure 3: Schematic top and cross-sectional view of the sensing element.

The sensing film was heated by the heater through the air that existed between the two parts when the response was examined (Hara, 2013). The sensing film had a double-layered structure; the material of the sensing film is the same as that of the basic type sensor. The thickness of the first and the second layers was 100 nm and 100 nm, respectively. The length and the width of the sensing film between the electrodes were 10 μm and 100 μm, respectively. The dimension of the Si substrates and the diaphragms was 3 mm × 3 mm × 0.5 mm and 250 μm × 250 μm × 7 μm, respectively, for both elements.

The detailed configuration of the sensing element and the micro heater is shown in Figure 3 and Figure 4, respectively.

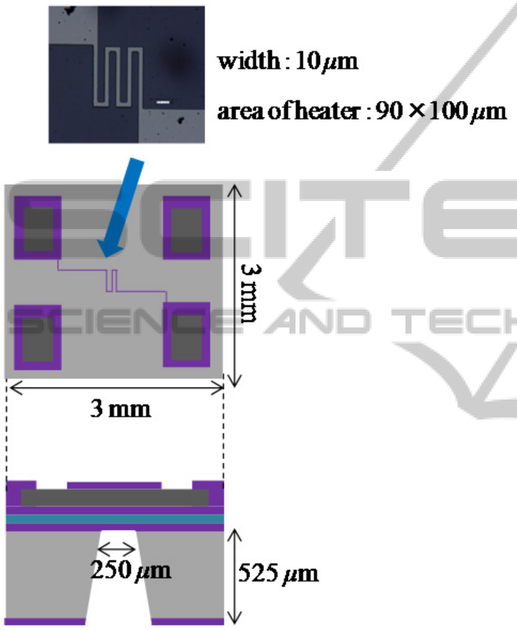

Figure 4: Schematic top and cross-sectional view of the micro heater.

2.2 Sensor Fabrication

The fabrication process of a basic type micro sensor is as follows. A Pt+W (5 mol%) film was deposited on an Al_2O_3 substrate for use as an electrode and defined by photolithography. Next, a layer made of Fe_2O_3+TiO_2 (5mol%) + MgO (4 mol%) and another layer made of SnO_2 + V_2O_5 (4 mol%) were successively deposited to form a sensing film. Finally, the sensing film was patterned by lift-off technique. All these thin films were deposited by r.f. sputtering technique.

The fabrication process of a MEMS type sensor is described below. A multi-layered SiO_2/Si_3N_4/SiO_2 film was successively deposited on a Si substrate. The thickness of the SiO_2, Si_3N_4, and SiO_2 films was 4 μm, 2 μm and 1 μm, respectively. Next, a part of Si was removed by wet etching to make a diaphragm structure. A triple-layered Cr/Pt + W (5 mol%)/Cr film was deposited as a sensor electrode and patterned by photolithography and subsequent sputter etching. The thickness of Cr, Pt + W and Cr films was 35 nm, 200 nm and 35 nm, respectively. Finally, a sensing film was deposited and patterned by lift-off technique. This process yielded a sensing element as shown in Figure 3.

A triple-layered thin film heater (Cr/Pt + W (5 mol%)/Cr) was made on a diaphragm formed on another Si substrate with a similar process. The heater had a meander pattern, whose total length and width were 500 μm and 10 μm, respectively. This process yielded a micro heater as shown in Figure 4. All these thin films were deposited by r.f. sputtering technique to fabricate a MEMS type sensor.

The sensing element and the micro heater were placed in parallel at a distance of about 50 μm by inserting gold wires between the sensing element and the micro heater as shown in Figure 2.

2.3 Experimental Setup

The sensor was set in a closed test box made of acrylic. The inner volume was 5.4 L. The responses to organic airborne particles such as mold spores and pollen were examined; the change of the sensor resistance was measured by using a digital multimeter.

Both sensing films need to be heated for operation. The temperature of the basic type sensor was measured to be 425 °C. The estimated sensor temperature of the MEMS type sensor ranged from 330 °C to 360 °C, while the estimated heater operating temperature ranged from 550 °C to 600 °C, corresponding to the consumed power that ranged from 125 mW to 140 mW.

2.4 Samples for Detection

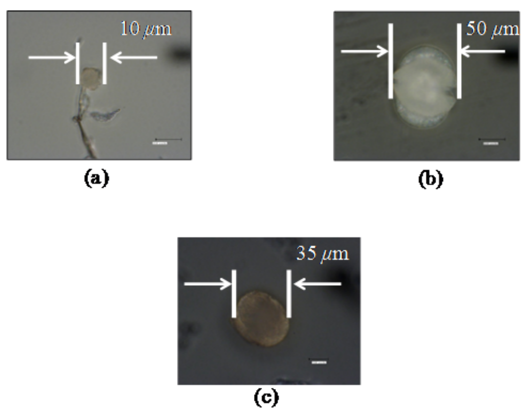

Figure 5: Photographs of (a) mold spores, (b) pine pollen, and (c) cedar pollen.

Mold spores, pine pollen and cedar pollen were used as test samples. The photographs of these particles are shown in Figure 5(a), 5(b) and 5(c), respectively. The typical diameter was 10 µm, 50 µm and 35 µm, respectively.

3 SENSING PERFORMANCE

3.1 Response to Mold Spores

The response to mold spores of the basic type sensor and the MEMS type sensor is shown in Figure 6(a) and 6(b), respectively. The operating temperature of the sensing films was about 425 °C and 330 °C, respectively. The sensor resistance steeply decreased after adhesion of mold spores and then gradually recovered to the initial value as the mold spores combusted on the sensor surface for both sensors.

For the basic type sensor, the relative resistance decrease was 34.2 % when a mold spore adhered to the surface and it was about 70 % when a bundle of mold spores adhered. The recovery time was 3.2 s when a mold spore adhered to the surface and it was about 12 s when a bundle of mold spores adhered. The repeatability of the response was satisfactory.

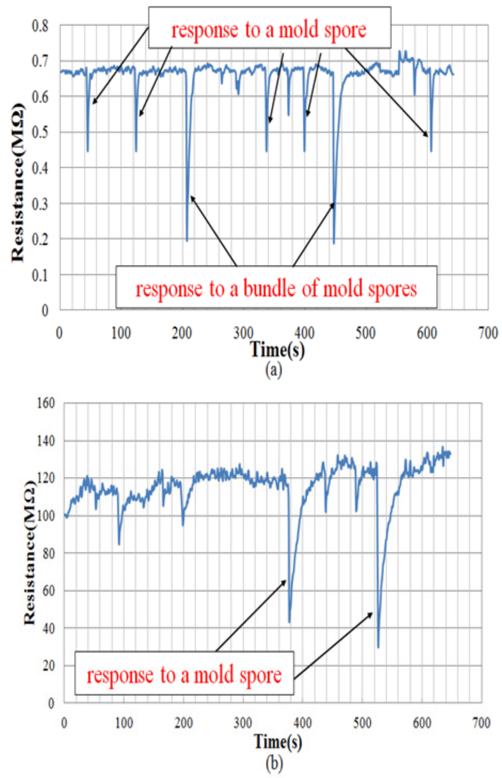

Figure 6: Response to mold spores: (a) basic type sensor and (b) MEMS type sensor.

For the MEMS type sensor, the relative resistance decrease was 69.3 % when a mold spore adhered to the surface. The recovery time was 29.5 s. The response was larger and the recovery time was slower compared to those for the basic type sensor.

3.2 Response to Pine Pollen

The response to a grain of pine pollen of the basic type sensor and the MEMS type sensor is shown in Figure 7(a) and 7(b), respectively. The temperature of the sensing film was about 425 °C and 330 °C, respectively. The sensor resistance steeply decreased after adhesion of a grain of pine pollen and then gradually recovered to the initial value as a grain of pine pollen combusted on the sensor surface for both sensors.

For the basic type sensor, the relative resistance decrease was 65.9 % when a grain of pine pollen adhered to the surface. The recovery time was 9.5 s.

For the MEMS type sensor, the relative resistance decrease was 95.1 %. The recovery time was 99 s. The response was larger and the recovery time was slower compared to those for the basic type sensor.

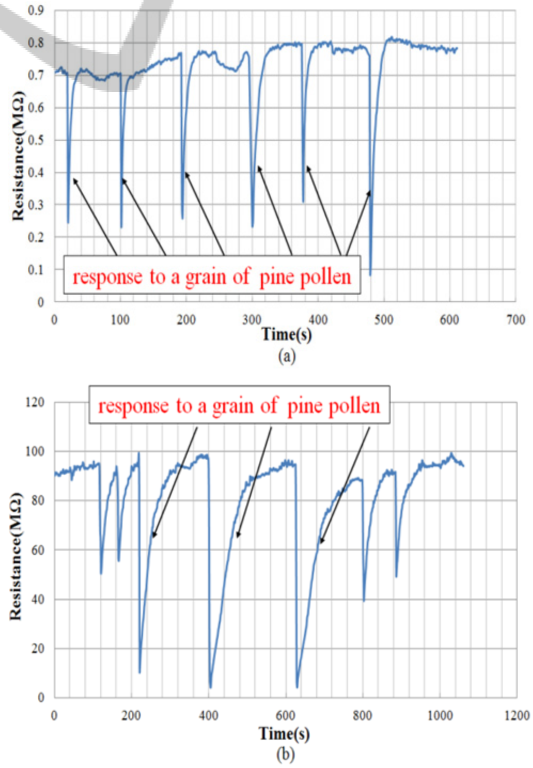

Figure 7: Response to pine pollen: (a) basic type sensor and (b) MEMS type sensor.

3.3 Response to Cedar Pollen

The response to cedar pollen is shown in Figure 8 for the MEMS type sensor. The temperature of the sensing film was about 330 °C. The sensor resistance steeply decreased and then gradually recovered to the initial value. The relative resistance decrease was 88.9 %. The recovery time was 67 s.

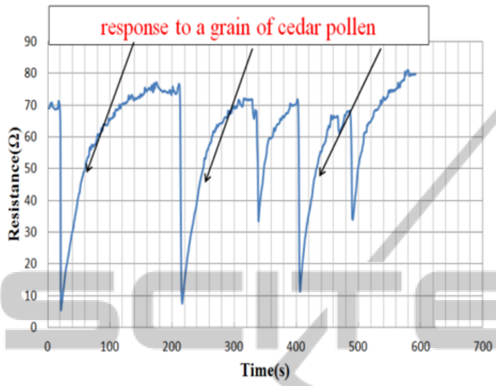

Figure 8: Response to cedar pollen for the MEMS type sensor.

4 DISCUSSION

4.1 Sensing Principle

The sensing principle for organic airborne particles is similar to that for reducing gases (Heiland and Kohl, 1988); hydrogen and carbon atoms in the mold spores or pollen react with chemisorbed and/or lattice oxygen on the surface of the metal oxide film, emitting electrons into the conduction band of the film. Thus the sensor resistance decreases after adhesion of organic particles. The resistance value gradually recovers to the initial one as the particle burns out on the surface of the sensing film.

The sensor is not selective to a mold spore or a grain of pollen but sensitive to all organic particles. However, both the resistance change and the recovery time are dependent on the size of the particle.

The observation by the naked eye revealed that cedar pollen got burned black soon after they adhered to the sensing surface and then gradually disappeared. The TDS (Thermal Desorption Spectroscopy) data on cedar pollen placed on the surface of the sensing film showed that a peak by H_2O appeared at around 150 °C, which was likely to be derived from the absorbed water in the pollen. Another peak by H_2O appeared at around 300 °C, which was likely to be one of the combusted gases. Two peaks by CO and CO_2 appeared at around 300 °C and 320 °C, respectively. Both were supposed to be combusted gases. These results indicate that the pollen burned on the surface of the sensing film. Liquid components usually evaporate from the sensing surface because it is maintained above 330°C.

4.2 Identification of Species

A larger organic particle contains more hydrogen and carbon atoms and consumes more oxygen atoms on the surface of the sensing film, emitting more electrons into the semiconductor film. The resultant relative resistance decrease is greater for a larger particle. In addition, it takes a longer time to combust a larger particle. So the recovery time is slower for a larger particle.

The relative resistance decreases and the recovery times are summarized in Table 1 and 2 for the basic type sensor and the MEMS type sensor, respectively. The smallest relative resistance change and the fastest recovery time were observed for a mold spore with a diameter of 10 µm that was the smallest particle in the experiment, while the largest relative resistance change and the slowest recovery time were observed for a grain of pine pollen with a diameter of 50 µm that was the largest particle. The medium relative resistance change and the recovery time were observed for a grain of cedar pollen with a diameter of 35 µm. Thus it is possible to estimate the particle size from the resistance decrease and the recovery time so as to identify the species of the particles. For example, a grain of cedar pollen that may cause allergy can be distinguished from a grain of pine pollen that may not cause allergy based on the resistance change and the recovery time.

Table 1: Average decrease of resistance and average recovery time for the basic type sensor.

Measured substance	Decrease of resistance (%)	Recovery time (s)
Mold spore	34.2	3.2
Pine pollen	65.9	9.5

Table 2: Average decrease of resistance and average recovery time for the MEMS type sensor.

Measured substance	Decrease of resistance (%)	Recovery time (s)
Mold spore	69.3	29.5
Pine pollen	95.1	99
Cedar pollen	88.9	67

4.3 Features of Two Types of Micro Sensors

The basic type sensor has a larger sensing area. So it is suitable for detection of a larger particle such as pollen. On the other hand, the MEMS type sensor has a smaller sensing area. So it is better suited for detection of a smaller particle such as mold spores. The experimental results reveal that the MEMS type sensor exhibits larger relative resistance change to a mold spore compared to the basic type sensor. Thus it is essential to minimize the sensing area for detection of a small particle.

The recovery time of the MEMS type sensor was much slower than that of the basic type sensor for both mold spores and pollen. This characteristic feature results from the lower operating temperature of the MEMS type sensor. Some other experiments show that both the resistance decrease and the recovery time strongly depend on the temperature of the sensing film. So it is necessary to optimize the temperature of the sensing film for specific particles to be detected.

The consumed power of the MEMS type sensor was reduced to about 125 mW by adopting heat-insulated structure with use of diaphragm. It is small enough for a portable or wearable detector of airborne particles.

5 CONCLUSIONS

Two types of micro sensors for real-time monitoring of organic airborne particles have been developed using semiconductor thin-film: a basic type thin-film sensor a MEMS type sensor. Both sensors successfully detected a mold spore or a grain of pollen. Based on the resistance change and the recovery time, it is possible to identify the species of the particle by the developed sensors. The repeatability of the sensor response was satisfactory. The MEMS type sensor was better suited for detection of smaller particles. Both sensors offer simple and inexpensive method to monitor organic airborne materials. Since the consumed power of the MEMS type sensor is about 125 mW, it can be used as a portable or wearable detector.

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