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MEMS Hydrogen Gas Sensor for the Entire Concentration Range of Hydrogen Gas

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We have proposed a new type of hydrogen gas sensor that is a combination of two types of hydrogen sensor, in which one is for a lower hydrogen gas concentration of less than 4% and the other is for a higher hydrogen gas concentration of more than 4%. The hydrogen gas sensor for low concentration works basically at room temperature based on the exothermic reaction of a Pd film formed on the cantilever and that for high concentration works during heating to about 150°C as a thermal conductance-type sensor by using the high thermal conductivity of hydrogen gas. This hydrogen gas sensor with a cantilever as a sensing area is fabricated using micro-electromechanical systems (MEMS) technology and silicon-on-insulator (SOI) substrate. A nichrome (NiCr) microheater and three thin-film thermocouples composed of an SOI layer of heavily doped n-type impurity and a Ni film layer are formed on the cantilever, and two areas, namely, a sensing area (region B_s) with the Pd film and a reference area (region B_R) without the Pd film, are formed with the same size in the vicinity of the tip of the cantilever. We have demonstrated that the proposed hydrogen gas sensor can measure hydrogen concentration at a very wide range from 0.1 to 100% within about 10 s.

1. Introduction

Hydrogen gas is focused on as a new energy source and as a carrier gas in metal organic chemical vapor deposition (MOCVD) for semiconductor devices. Since hydrogen has an explosion risk at a volume ratio of about 4% (vol.%) in air, a hydrogen gas sensor for low-concentration hydrogen gas detection of less than about 4% is necessary to ensure safety. On the other hand, a high-concentration hydrogen gas sensor is also necessary to control its concentration for carrier gas and fuel.

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Various types of hydrogen gas sensor based on different principles have been studied. Traditionally, the contact combustion-type hydrogen gas sensor has been well known.^(1,2) In these hydrogen gas sensors, hydrogen is burned by a heater with a catalyst, such as Pt heated to about 100°C or more. The temperature difference between the sensing area and the substrate is measured using a thermopile. However, a relatively high temperature is needed to achieve the contact combustion in the atmosphere containing oxygen. Since the sensitivity to hydrogen gas depends on the state of the catalyst surface, we have faced problems such as a change in the catalyst surface over time, selectivity to flammable gas such as methane, and the formation reproducibility of the catalyst surface.

A hydrogen gas sensor using the metal-oxide-semiconductor field-effect transistor (MOSFET), which has the catalyst on the gate oxide and is based on the mechanism of the change in the work function including the Nernstian behavior, was reported.⁽³⁻⁶⁾ This sensor must be heated, such as to about 50°C, by a heater owing to a decrease in the effects of ambient temperature and humidity, and to expedite the response. However, it had a problem in terms of the relatively large power consumption required to heat it up because of the large heat capacity of the sensor chip.

We have proposed a cantilever-type hydrogen gas sensor formed by MEMS technology, which is based on the exothermic reaction at the occlusion of hydrogen and the selectivity of the Pd film occluding only hydrogen.⁽⁷⁾

In this study, the hydrogen gas sensor composed of a cantilever having a microheater and three thermocouples formed by MEMS technology, which can cover the measurement of the entire hydrogen gas concentration range (0.1-100%) with only one sensor, is proposed and demonstrated.

For a low hydrogen concentration of 4% or less (the low-hydrogen-concentration mode), we use the exothermic reaction at the occlusion of hydrogen and hydrogen selectivity of the Pd film to the other gases operating at near room temperature. On the other hand, for a high hydrogen concentration of 4% or more (the high-hydrogen-concentration mode), we use the sensor as a thermal conductance-type sensor since hydrogen gas has the highest thermal conductivity of all gases.

2. Materials and Methods

2.1 Structure and mechanism of hydrogen gas sensor

In Fig. 1, a photograph of the fabricated hydrogen gas sensor is shown. This hydrogen gas sensor is composed of an SOI cantilever having regions A and B, and region B is divided into two areas: region B_s (sensing area) and region B_R (reference area) with the same size. In this prototype hydrogen sensor, the SOI cantilever is about 5 mm long. In region B_s , a Pd film of about 1 μ m thickness as a hydrogen occlusion material is deposited by RF sputtering to occlude hydrogen, while no Pd film is present in region B_R . A film composed of Ti/Ni/Ti (Ti: ~0.05 μ m thick; Ni: ~0.5 μ m thick), which is also used as one of the thermoelectric materials for a thin-film thermocouple, underneath the Pd film occluded by hydrogen, because the Pd film has a tendency to peel when exposed to hydrogen gas. In region A, which is located in the vicinity of the



Fig. 1. Photograph of the fabricated hydrogen gas sensor.

center of the cantilever, a NiCr film as a microheater is formed and region B, including regions B_s and B_R , is located near the tip of the cantilever.

In regions A, B_s , and B_R , hot junctions of the thermocouple are formed, and their cold junctions are formed in the Si substrate (Si chip). These thermocouples are composed of thermoelectric materials of a heavily n-type impurity-doped SOI (phosphorous P, 1,100°C, 1 h, thermal diffusion) and a Ni film (about 0.5 µm thick) formed on it via a thermally grown SiO₂ film (about 1 µm thick). These thermocouples have a Seebeck constant of about –160 µV/K. The Ni film is sandwiched by very thin Ti films, as Ti/Ni/Ti, deposited by RF sputtering to increase adhesion with the SiO₂ film and to prevent the overetching of the Ni film during its patterning.

Figure 2 shows a diagrammatic illustration of the measurement circuit of the hydrogen gas sensor. The total resistance of series-connected thermocouples to measure the temperature difference ΔTb between regions B_s and B_R was about 33 Ω . In this measurement circuit, the temperature of region A can also be measured by using thermocouple A by turning on the switch for thermocouples. The feedback resistance R_f is 100 k Ω in the OP amplifier circuit shown in Fig. 2. We can obtain the amplified output voltage V_0 corresponding to the temperature difference ΔTb between region B_s, where the temperature rises by exothermic reaction when occluding hydrogen in the Pd film, and region B_R, where no thermal reaction occurs, by using thermocouples formed in these regions.

In our proposed hydrogen sensor working at room temperature, the exothermic reaction will stop when the Pd film is saturated by occluded hydrogen, and then the temperature will decrease. This will lead to a peak in the output characteristics. It is necessary to discharge hydrogen from the Pd film periodically, after hydrogen is



Fig. 2. Diagrammatic illustration of the measurement circuit of the hydrogen gas sensor.

occluded. In these sensor measurements, the hydrogen gas sensor is initialized to discharge the occluded hydrogen from the Pd film by heating the microheater to about 150°C by applying a rectangular waveform voltage for 5 s with an electric power of about 255 mW.

First, we had planned to measure the hydrogen gas concentration only during the heating cycle by measuring the temperature difference ΔTb between region B_s with a Pd film and region B_R without a Pd film, because the Pd film will increase its temperature exothermically when hydrogen is occluded. However, we have found that the output voltage V_0 corresponding to the temperature difference ΔTb is decreased when the hydrogen concentration exceeds 4%, as shown in Fig. 3. The reason is that the temperature of the cantilever will decrease under the same power consumption of the microheater because of the thermal conduction to a surrounding gas as the concentration of hydrogen gas, which has the highest thermal conductivity of all gases, increases.

In the low hydrogen concentration of less than 4%, we have found that it is better to measure the hydrogen concentration during the cooling cycle, after the heating to discharge hydrogen from the Pd film is stopped. In this cooling cycle, we can apply the null detection method, which is well known as a highly precise measurement.

Thus, we have decided to adopt two measurements methods for lower and higher hydrogen concentrations with different principles: one is in a hydrogen concentration lower than about 4% (the low-hydrogen-concentration mode), and the other is in a hydrogen concentration higher than about 4% (the high-hydrogen-concentration mode).



Fig. 3. Waveforms of output voltage V_0 corresponding to the temperature difference ΔTb of region B_s (sensing area) and region B_R (reference area) for various hydrogen concentrations in air when the microheater is heated by a constant rectangular waveform voltage.

For the low hydrogen concentration, we will measure the temperature difference ΔTb between B_s and B_R during the cooling cycle after stopping the heating. On the other hand, for the high hydrogen concentration, we will measure the temperature of region A based on that at zero hydrogen concentration during heating as a thermal conductance-type sensor. Since we have always carried out both measurement methods during heating and cooling cycles, we can compare these two output voltages and we can judge the real hydrogen concentration.

2.2 *Measurement in low-hydrogen-concentration mode*

A hydrogen storage metal such as palladium (Pd) will generate a metal hydride when reacting with hydrogen. In the thermal equilibrium state of the hydrogen storage metal, the reaction of hydrogen occlusion will be given by⁽⁸⁾

$$Pd + H_2 \rightleftharpoons PdH_x \pm \Delta H, \tag{1}$$

where ΔH is the heat of the reaction, and x is the amount of occluded hydrogen.

The principle of hydrogen gas (H_2) sensing in this low-concentration region is that we measure a small temperature change based on the exothermic reaction when a material such as Pd occludes hydrogen, and let the temperature change correspond to the hydrogen concentration in air by calibration.

To detect the minute temperature change, we have fabricated a free-standing cantilever structure with a small thermal capacity using Si-MEMS technology, as shown

in Fig. 1, so as to achieve very high temperature sensitivity.

We measure the hydrogen concentration during the cooling cycle after stopping the heating of a microheater formed on the cantilever in air including hydrogen. In this process, region B_R will immediately return to room temperature as an ambient temperature because it has no Pd film on it, while the temperature in region B_S will remain higher than that of region B_R because of the exothermic reaction based on the occlusion of hydrogen in the Pd film, which will last until occlusion in the Pd film is saturated. Therefore, the measurement is executed at the time when it seems that the output voltage, corresponding to the temperature difference between region B_R and the Si substrate (chip) being at room temperature, becomes almost zero.

General metallic alloys for hydrogen storage are easily oxidized and so cannot be used in air. However, a Pd film of a noble metal as a hydrogen occlusion material works with stability even at a high temperature in air. Thus, the Pd film will be the best material for the occlusion and discharge cycles of hydrogen.

2.3 Measurement in high-hydrogen-concentration mode

The thermal conductivity of hydrogen gas is the largest among gases. Therefore, as shown in Fig. 3, the heat radiation from the microheater increases as the hydrogen concentration in air is increased above 4% because of the effect of the thermal conductivity even if the microheater is heated by the same electric power, and the temperature decreases more than the temperature of the heater when heating it in air.

In Fig. 3, waveforms of output voltage V_0 corresponding to the temperature difference ΔTb for various hydrogen concentrations in air are shown, when the microheater is heated by a constant rectangular waveform voltage. In the low hydrogen concentration of 4% or less, we have adopted the measurement of the temperature difference ΔTb between B_s and B_R ; however, we cannot use this experimental method for the high hydrogen concentration above 4%, we have adopted the other experimental method to measure the temperature difference using thermocouple A between region A, where the microheater is formed, and the Si substrate working as a thermal conductance-type sensor. We let the output voltage of thermocouple A correspond to each hydrogen concentration above 4% as a thermal conductance-type hydrogen gas sensor based on the output voltage of thermocouple A when the hydrogen concentration is zero.

3. Results

3.1 *Output characteristics for injections of various hydrogen gas concentrations in air*

After we filled a metal container of about 30 mL, in which the hydrogen sensor is placed, with pure air at a room temperature of 20°C, hydrogen occluded in the Pd film was discharged by heating region A to about 150°C for 5 s with a power of 255 mW applied to the microheater formed on the cantilever of the sensor device shown in Fig. 1. Then, we filled the container with hydrogen gas of concentrations of 0, 1, 5, 10, 20, 60, and 100% relative to air by slowly injecting it over 2.5 s. In Fig. 4, the output voltage



Fig. 4. Output voltage waveforms corresponding to the temperature difference ΔTb when various hydrogen gas concentrations diluted by pure air are injected.

waveforms are shown when we have measured the temperature difference ΔTb under the above-mentioned conditions. The horizontal axis is all 2.5 s/div. As for the vertical axis, we have adopted a unit of 100 mV/div. for 0 and 100% hydrogen concentrations to compare them at the initial setting. However, for the other hydrogen concentration, a unit of 50 or 100 mV/div. is adopted depending on the output intensity.

We have ascertained that no influence of the flow during gas injection was observed at all because of the fact that no output change is observed during injection of 0% hydrogen concentration (pure air). It is understood that a wavy peak grows as the hydrogen concentration is increased; moreover, the wavy peak width broadens in a low hydrogen concentration.

In this paper, we will not describe the detailed characteristics of the hydrogen concentration in pure nitrogen gas; however, we want to add that a similar tendency in air is also observed in pure nitrogen gas. Therefore, we can measure the hydrogen concentration even in the state without oxygen.

3.2 Output characteristics when hydrogen gas of 100% is injected at various ambient temperatures

The above-mentioned container of about 30 mL is filled with 100% hydrogen gas by slow injection over 5 s, after the container, which is put in a constant-temperature tank set at various ambient temperatures, is filled with pure air. In Fig. 5, output voltage waveforms depending on the temperature difference of B_s and B_R at various ambient temperatures are shown, where the unit of the horizontal axis is 5 s/div. It is understood that the shape of the waveforms sharpens and the peak increases as the ambient temperature increases.

3.3 *Output characteristics of low-concentration hydrogen gas*

After the injection of hydrogen gas of various concentrations has been completed, the microheater is heated in the square wave voltage for 5 s by an electric power of 255 mW. Output voltage waveforms corresponding to the temperature difference of the B_s and B_R regions for various low hydrogen concentrations are shown in Fig. 6. The time when the heating of the microheater began is graphed as 0 in this figure. The temperature of region A during heating is estimated to be about 150°C. The detection of an extremely small amount of hydrogen gas becomes important because hydrogen is an explosion hazard when its concentration in air becomes about 4%.

In the measurement of a low hydrogen concentration of 4% or less and that of a high hydrogen concentration of 4% or more, different methods are used. In the heating period in this low hydrogen gas concentration, the temperature rise is obtained as a result of diverse effects existing together, such as exothermic effects based on the occlusion of hydrogen and the catalyst in the Pd film, and cooling effects based on heat conduction depending on the hydrogen concentration, etc. as the concentration of hydrogen gas increases. However, it is observed that the effect of heat radiation on the thermal conductivity of hydrogen gas increases when the hydrogen gas concentration exceeds 4%, and the output voltage becomes small. That is, the output voltage of the hydrogen gas concentration of 5% has become smaller than that of 4%, as shown in Fig. 6.



Fig. 5. Output voltage waveforms depending on temperature difference ΔTb in various ambient temperatures.

When the temperature difference ΔTb between regions B_s and B_R , which has a slight difference in shape with region B_s , is measured during the heating-on cycle, there is a problem in that the output appears even if the hydrogen concentration is 0. Therefore, we consider that the error margin may be large in the measurement of an extremely small concentration of hydrogen gas.

To solve the above-mentioned problem, the temperature difference ΔTb of the sensing area and reference area should be measured during the cooling cycle after the heating of the microheater is stopped. Figure 7 shows the magnified output voltage waveforms in an extremely low hydrogen gas concentration range (0.5% or less) among the



Fig. 6. Output voltage waveforms corresponding to the temperature difference ΔTb for various low hydrogen concentrations.



Fig. 7. Magnified output voltage waveforms in an extremely low hydrogen gas concentration range (0.5% or less) during the cooling process after the heating is stopped.

characteristics shown in Fig. 6 during the cooling process after the heating has stopped. In a low hydrogen concentration, it is understood that the temperature of the sensing area is still above room temperature because of hydrogen occlusion, whereas the temperature of the reference area completely returns to room temperature at a time of about 8τ , where τ is a thermal time constant (about 500 µs in this prototype sensor) of the cantilevertype microheater, measured after the heating is stopped (at about 4 s after the heating is stopped). This temperature difference ΔTb rises with an increase in the hydrogen concentration in this low-concentration area. Since the temperature of the sensing area should be room temperature when the hydrogen concentration is 0, the null detection method with extremely high accuracy and high sensitivity can be used to measure the temperature difference ΔTb of the sensing area and reference area at room temperature. Therefore, an extremely highly accurate measurement of the hydrogen concentration becomes possible for this hydrogen gas sensor.

Figure 8 shows a graph of the output voltage in a hydrogen concentration of less than 1% at the time indicated by the dotted line shown in Fig. 7. The dotted line in Fig. 7, which is lined at 9 s in the horizontal axis, is at 4 s later $(0.5 \times 8 \text{ s})$ since the heater was turned off.

In our experiments, the mixing ratio of air and hydrogen gas was adjusted by using the gas flow meter. Therefore, we could calibrate the hydrogen concentration only above 0.1%.

3.4 Output characteristics of high-concentration hydrogen gas

In hydrogen gas concentration above 4%, the temperature difference between the substrate and region A, in which the microheater is formed, of the hydrogen gas sensor as shown in Fig. 1 is measured using thermocouple A as a thermal conductance-type hydrogen gas sensor, which is based on the fact that hydrogen gas has the highest thermal



Fig. 8. Graph of the output voltage V_0 of the hydrogen concentration in less than 1 vol.% at the time indicated by the dotted line shown in Fig. 7.

conductivity of all gases. Figure 9 shows the output voltage waveform of the hydrogen gas sensor for each hydrogen gas concentration when working in the high-hydrogenconcentration mode, and Fig. 10 shows the output characteristics of the hydrogen gas sensor for each hydrogen gas concentration at that time, based on the output voltage of 0% hydrogen gas concentration.

4. Discussion

In Fig. 4, it is thought that a wavy peak will grow as the hydrogen concentration is raised due to an increase in the caloric value, because the hydrogen occlusion reaction per unit time increases as the hydrogen gas concentration increases. A peak in the output voltage will appear when the hydrogen occlusion in the Pd film reaches saturation and the exothermic reaction stops, and then the temperature begins to fall. When the hydrogen concentration is low, the quantity of hydrogen occlusion per unit time to the Pd film becomes small and the time until saturation will be extended. This fact may be the reason why the waveform becomes wide when the hydrogen concentration is low.

In contrast, when the hydrogen concentration is high, a sharp peak and large height can be expected because hydrogen occlusion is achieved in a short time and the exothermic reaction is also quickly finished.



Fig. 9. Output voltage waveform of the hydrogen gas sensor for each hydrogen gas concentration when working in the high-hydrogen-concentration mode.



Fig. 10. Output characteristics of the hydrogen gas sensor to each hydrogen gas concentration based on the output voltage of 0 vol.% hydrogen gas concentration.

In general, if the length of a cantilever becomes half, it is understood that the thermal time constant becomes 1/4. Therefore, it is thought that the high-speed detection of less than 1 s is enabled by the miniaturization of the cantilever of our proposed hydrogen sensor.

In Fig. 5, it is thought that, in air, the reason why the shape of waves sharpens and the output peak grows as the ambient temperature rises is that the reaction of the injected hydrogen gas with oxygen adsorbed on the Pd surface becomes active as the temperature increases. The output peak is observed to grow as the ambient temperature decreases in pure nitrogen gas, although it is not shown here.⁽⁷⁾ Because a temperature rise was observed in a pure nitrogen gas atmosphere by injection of hydrogen gas, it is understood that these phenomena must be based on an occlusion effect of hydrogen and that oxygen is not necessary for this reaction.

Figure 11 shows the output voltage of the hydrogen gas sensor when the container was filled with pure methane (CH_4) gas as a flammable gas by injection over 2.5 s after the container was substituted by pure nitrogen (a) or air (b). It is understood that the output voltage, when methane gas is injected, is not observed at all with the same hydrogen gas sensor (as shown in Figs. 4 and 5). We may conclude that the methane gas did not respond at all to our hydrogen gas sensor when working at the low-hydrogen-concentration mode. From these results, as for this hydrogen sensor, it is confirmed that there is a high selectivity for hydrogen but no response to methane gas, because of the method using the effect of the exothermic reaction based on hydrogen occlusion of Pd film.

From Fig. 7, it is understood that the output voltage does not vanish even if it passes by several times the thermal time constant after the heating of the heater stops in the



Fig. 11. Output voltage of the hydrogen gas sensor when the container was filled with pure methane (CH_4) gas (H: 2.5 s/div., V: 100 mV/div.) (a) in nitrogen gas and (b) in air.

atmosphere of various hydrogen gas concentrations. These facts will come from the exothermic reaction due to the discharged hydrogen gas during the heater-on state and the reocclusion during the cooling state after the heating stops. This may be clear from the fact that the time until hydrogen is saturated by hydrogen occlusion becomes longer as the hydrogen concentration decreases, as shown in Fig. 4.

In Fig. 7, the mixing ratio of hydrogen gas with pure air to measure the hydrogen concentration was determined by calibration using the gas flow meter; however, its accuracy of calibration was 0.1%. Therefore, we could not measure the hydrogen concentration of less than 0.1%. It is thought that the measurement of at least 100 ppm (0.01%) is possible even using this present hydrogen gas sensor if we use a more accurate calibration, because we can use the null detection method for our hydrogen sensor.

In combustion-type hydrogen gas sensors using catalysis, it is necessary to heat the sensing area and to measure the hydrogen concentration during its heating. However, the temperature difference ΔTb between the reference area and sensing area will surely exist during heating owing to a minute difference in their thermal capacity and conductance, and it is hard to compensate for this temperature difference completely. Therefore, in these types of sensor, it is essentially hard to measure a hydrogen concentration of about 0%.

On the other hand, a hydrogen concentration in the vicinity of 0% can be measured in detail using our proposed hydrogen gas sensor because the concentration measurement is carried out during the cooling process and by the null method, which is that the output voltage essentially becomes 0 when the hydrogen concentration is 0%.

Sensing in the low-hydrogen-concentration mode that uses the exothermic reaction

based on occlusion of the Pd film and sensing in the high-hydrogen-concentration mode using the thermal conductivity of hydrogen can be carried out at the same time using only one hydrogen gas sensor during a heating cycle.

In the low-concentration hydrogen gas, it takes a lot of time to return to the baseline at 0%, as shown in Figs. 3 and 6. However, we have experimentally confirmed that characteristic waveforms are almost stable and fixed when the heating cycle of heating-on for 5 s and heating-off for 5 s is repeated. The heating-on cycle up to about 150° C is used to refresh the sensor by discharging the occluded hydrogen from the Pd film.

5. Conclusions

We have proposed a new type of hydrogen gas sensor that is a combination of two types of hydrogen sensor, in which one is for a lower hydrogen gas concentration of 4% or less (the low-hydrogen-concentration mode), and the other is for a higher hydrogen gas concentration of more than 4% (the high-hydrogen-concentration mode). The hydrogen gas sensor for low concentration works basically at room temperature based on the exothermic reaction of the Pd film formed on the cantilever and that for high-concentration works during heating to about 150°C as a thermal conductance-type sensor by using the high thermal conductivity of hydrogen gas.

We have demonstrated that the proposed hydrogen gas sensor can be used to measure hydrogen concentration in the range from 0.1 to 100% within about 10 s. If we use a more accurate gas flow meter for calibration, detection of the concentration of about 0.01% (100 ppm) of hydrogen gas will be expected to be carried within about 10 s.

It is confirmed that our hydrogen gas sensor working in the low-hydrogenconcentration mode has a high selectivity for hydrogen but no response to methane gas, because of the method using the effect of the exothermic reaction based on hydrogen occlusion of the Pd film.

Since this hydrogen gas sensor has a very small thermal capacitance and thermal conductance because of the use of the cantilever formed by MEMS technology, a quicker response and lower power consumption will be achieved by its further miniaturization. Studies on long term stability and hysteresis for various hydrogen concentrations are necessary.

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