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Fabrication of low power micro-heater for micro-gas sensor
II. Characteristics of micro-gas sensor

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Abstract

A new planar-type microsensor, which had a platinum heater and a sensing layer on the same plane was fabricated on silicon substrate with stress-relieved PSG(phosphosilicate glass)/Si_3N_4(800nm/150nm) diaphragm. The proposed planar-type microsensor could be fabricated by simple silicon process using only 3 masks for photolithography process compared with 5 or 6 masks of the typical micro-gas sensor. The thermal properties of the microsensor from thermal simulation were compared with those of the fabricated microheater. Although there are some discrepancy between the simulation result and the result from the fabricated microheater, the thermal simulation by FEM was proved to be an useful method to evaluate the thermal properties of microheater. The sensing characteristics of the fabricated microsensor with the planar-type heater were investigated also.

I. Introduction

Most metal-oxide gas sensors for detecting hydrocarbon or combustible gases work at high temperatures between 100 and 400℃ in order to operate efficiently and be adequately sensitive to gases[1]. This implies rigid restrictions on the available technologies. Therefore in-situ heating is one of the basic requirements for a micro-gas sensor to maintain it at the elevated temperature with low power consumption.

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For low power consumption and good thermal isolation from the surrounding circuits, the microheater of the sensor has to be mounted on a thermal insulation layer. Until now, two kinds of membrane-type structures were widely used for thermal insulation. One structure uses the substrate silicon removed by etching from the back side[2~4] and the other uses the substrate silicon removed by etching from the front side, that is, micro-bridge structure[5~7]. Among both structures, the former structure was more frequently used due to easy fabrication process and mechanical strength.

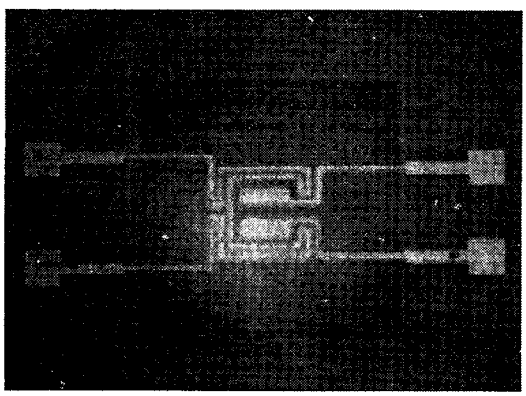
Recently, we have reported research on thermal distribution analyses of the platinum microheater with PSG/Si_3N_4 diaphragm. The simulations were performed by using a commercially available finite element method(FEM) package[8]. In the report,

two kinds of micro-gas sensors, the planar-type structure ,which has a heater and a sensing layer on the same plane, and the stack-type structure, which has the stacked structure of sensing layer/insulator/heater, were designed. The thermal analyses including temperature distribution over the diaphragm and power consumption of the heater were compared with each other. From the simulation result, we concluded that the planar-type heater consumed about 10% more power than the stack-type heater for the same operating temperature of sensing layer. The temperature uniformity over the sensing layer was, however, relatively good in the planar-type heater compared with in the stack-type heater. First of all, the planar-type heater proposed in this study could be fabricated by only 3-masks compared with 5 or 6-Masks of the stack-type heater.

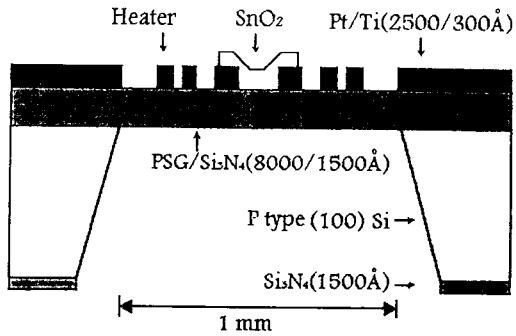
In this study, the simulated micro-heater, the planar structure, was fabricated. The heating properties of the heater were compared with the results of simulation and the CO gas sensing characteristics of the microsensor with the heater were investigated.

II. Device Fabrication

The structure of a proposed planar-type microsensor is shown in Fig.1. The dimensions of



(a)



(b)

Fig. 1. The structure of a planar-type microsensor. (a) SEM of over view (b) Schematic cross sectional view

the sensitive tin oxide film were 260 ㎛×260 ㎛, those of the square diaphragm were 1.5 ㎜×1.5 ㎜ and those of the fabricated whole silicon chip were 3.7 ㎜×3.7 ㎜.

A double-side polished p-type(100) silicon wafer with a thickness of 320 ㎛ and a resistivity of 10~20Ω · ㎝ was used as a substrate. However, a stress-relieved dielectric layer[9], composed of a low-pressure chemical-vapor-deposited(LPCVD) silicon nitride(150 nm) and atmosphere-pressure chemical-vapor-deposited (APCVD) phosphorous silica glass(PSG, 800 nm), is used as a membrane on the front side and as a passivation layer during back etching on the backside. Figure 2 shows flow sequence of fabrication process for the planar-type microsensor. The process steps were as follows:

(1) A 230 nm thin film of Pt with 30 nm Ti as adhesion layer was deposited by r.f. magnetron sputtering.

(2) The PSG/Si_3N_4 layer of backside was patterned by wet etching and reactive ion etching(RIE) subsequently using Mask I , and the Pt/Ti layer of front side was patterned using Mask II by double side alignment.

(3) The Pt/Ti layer was etched to realized a resistance heater and electrode pairs for the sensing layer.

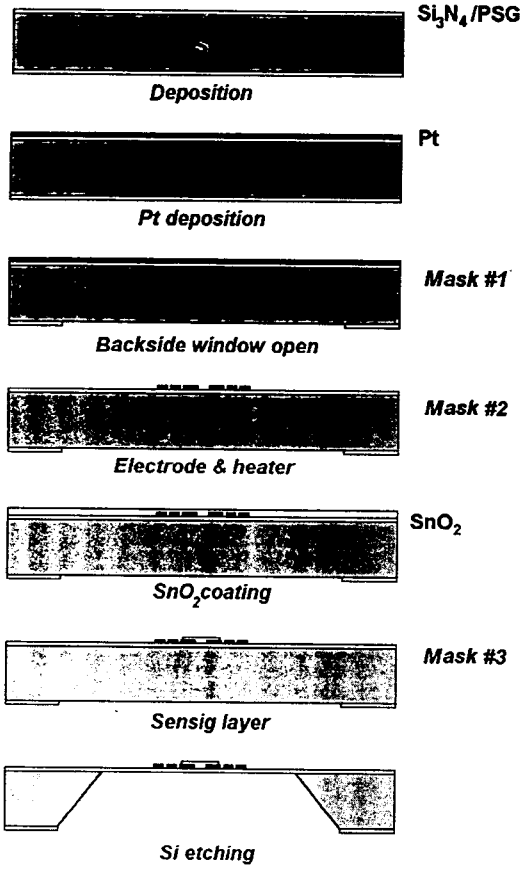


Fig. 2. The flow sequence of fabrication process for the planar-type microsensor.

(4) The SnO_2 solution synthesized by hydrothermal method[9] was spin coated over the silicon substrate with platinum electrode pairs and etched to form sensing layer using Mask III, and the sensing layer was heat treated at 600℃ for 30 min subsequently.

(5) Finally, the backside of the Si substrate was removed by anisotropic KOH etching, and subsequently thermal isolation structure with a 950 ㎚ thick square diaphragm was fabricated.

III. Results and Discussion

3.1 Heating characteristics of the heater

The heating characteristics of the planar-type platinum heater were examined. The platinum resistor was prepared for common use of heater and temperature sensor. The resistance of the platinum heater was measured as a function of the temperature on a hot plate in order to calibrate the temperature scale. From the relationship between temperature and resistance of the platinum resistor, the measured positive temperature coefficient(PTC) of the platinum heater was about 3500 ppm/℃. It has been reported that the PTC is 0.385 Ω/K[11] for industrial platinum resistance thermometers which resistance value is 100 Ω at 0 ℃. This means that the electrical resistance raises with 0.385 Ω if the temperature increase with 1 K. The discrepancy between the measured PTC and standard PTC may be come from the facts that the thickness of the platinum resistor of the temperature sensor in this experiment is too thin to show bulk effect and the fabricated platinum resistor has some different qualities.

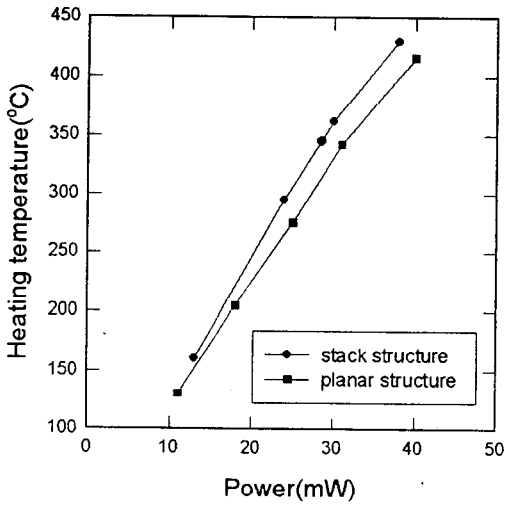


Fig. 3. Simulated heating characteristics of microheaters.

Figure 3 shows simulated heating characteristics of two types of microheaters, that is, the planar-type and the stack-type microheaters. In

the planar-type structure, the sensitive film was heated by the platinum heater on the outer side of the sensitive layer. The heating energy of the heater might be transferred in the form of thermal conductive energy. Therefore, some convection energy might be dissipated during thermal conduction from platinum heater to sensitive layer in the planar-type structure. On the other hand, in the stack-type structure with the structure of sensing layer/oxide(insulator)/ heater, the sensing layer could be heated by the heater situated just below of the sensing film. Consequently, the stack-type structure could save the consuming power for the high operating temperature. The simulated power consumption of the planar-type heater for the operating temperature of 350 ℃ of the sensing layer was 33 ㎽(or 34 ㎽ with consideration of radiation effect) and this power is about 10% more than that of the stack-type heater for the same operating temperature.

The simulated relationship between power and temperature of the sensing layer was compared with that of the fabricated microheater in Fig. 4.

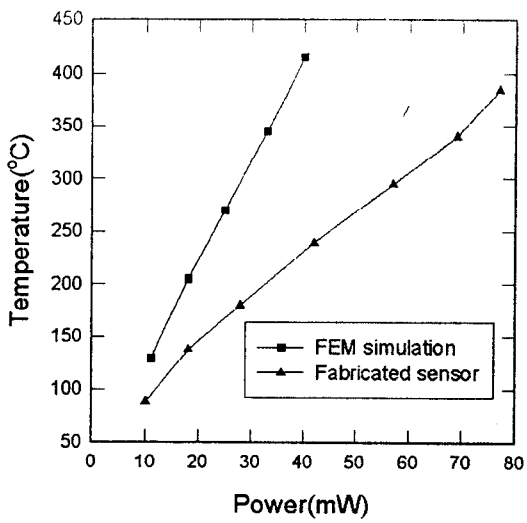


Fig. 4. The heating properties of the planar-type microsensor from the computer simulation and the fabricated microheater.

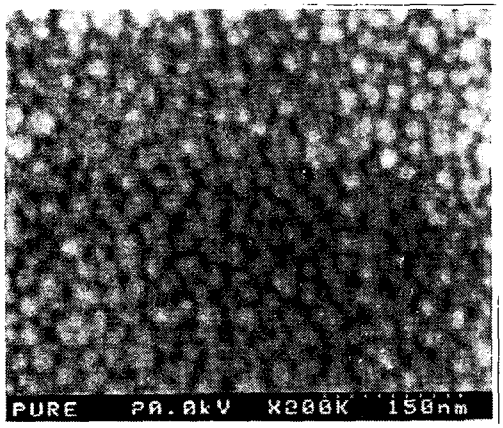
The consumed power of the planar-type heater for the operating temperature of 350 ℃ is about 68 ㎽ and this is about 2 times of the simulated result. This discrepancy between the simulated result and the tested result from the fabricated microheater might come from the simplification of the simulation condition, that is, disregard of the effects of bonding pad, lead wire and touch of the silicon sample with package die.

Though there are some discrepancy between the simulation result and the result from the fabricated microheater, the computer simulation method using FEM looks very useful method to evaluate the thermal distribution and thermal consumption of the micro silicon heater.

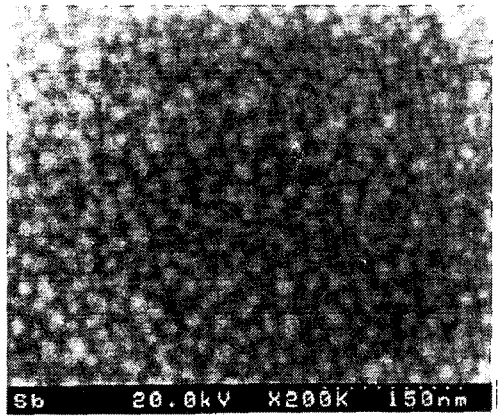
3.2 Gas sensitivity characteristics of the microsensor

The most commonly used methods for the preparation of sensing film of micro-gas sensor are physical methods such as r.f. sputtering and thermal evaporation[12~14]. Though it is not easy to be compatible with microelectronic process, the chemical preparation method such as sol-gel method[10,15] and spray method[16] for the preparation of gas sensitive film has many advantages over the physical ones because of the follows; (i)catalyst can easily incorporated, (ii)additive can exactly controlled, (iii)no special apparatuses are required, (iv)the resultant films may possibly be characterized by an ultrafine and porous structure with a large specific area.

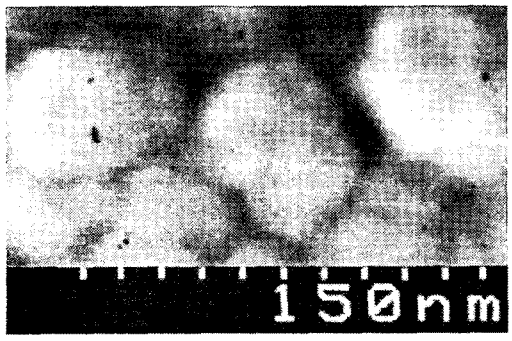
The coating SnO_2 and Sb-doped SnO_2 solutions were colloidal SnO_2 solution(SnO_2 content 8 wt.%, mean diameter of particles : about 3 ㎚) and SnO_2(Sb) solution(SnO_2 content 10 wt.%, Sb content 0.6~0.7 wt.%, mean diameter of SnO_2 particles : about 3 ㎚), and these are commercially available solutions(Taki Chemical Co., Ltd., Japan). These solutions were prepared by means of the hydrothermal method described in detail elsewhere[10,15]. The mixed solution of SnO_2 and



(a) SnO_2 film



(b) SnO_2(Sb) film

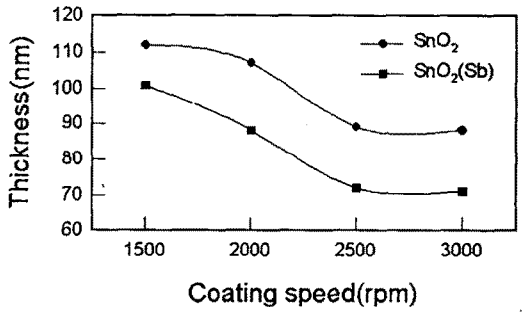


(c) expanded micrograph of SnO_2 film

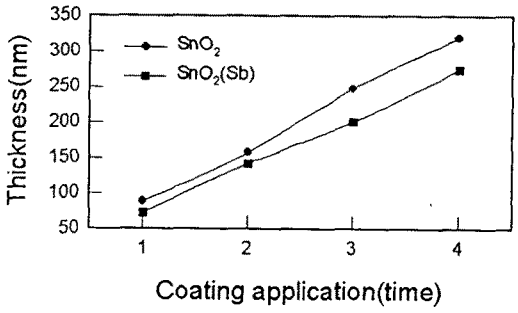
Fig. 5. High-resolution SEM micrographs of SnO_2 film(a) and SnO_2(Sb) film(b) spin-coated at 3000rpm and 2 times application.

SnO_2(Sb) was used to control the Sb contents of a coated film.

The surface morphologies were observed by high resolution SEM. Figure 5 shows the surface morphologies of SnO_2 and SnO_2(Sb) films, which were spin-coated at 3000 rpm, at two times and heat treated at 600 ℃ for 30 min in air. Figure 5(c) is the expanded micrograph of the SnO_2 film in Fig.5(a). A comparison of Fig.5(a) with Fig.5(b) suggested that there are no great microstructural differences between undoped and Sb doped SnO_2 films, and that the sol-gel spin-coating technique can produce homogeneous SnO_2 films with crystallites very regular in size. The film was composed of very fine, below 150Å, particles of SnO_2 crystallite and was well condensed flat film. And we can see that interparticle neck formation in the SnO_2 film is minimal in Fig.5.



(a)

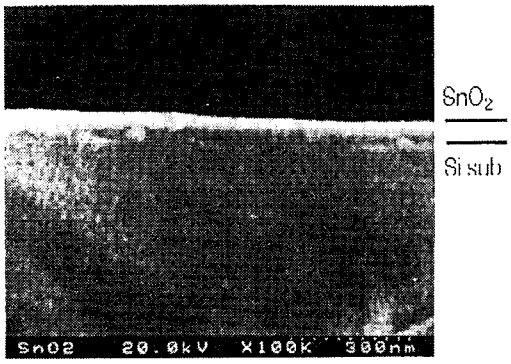


(b)

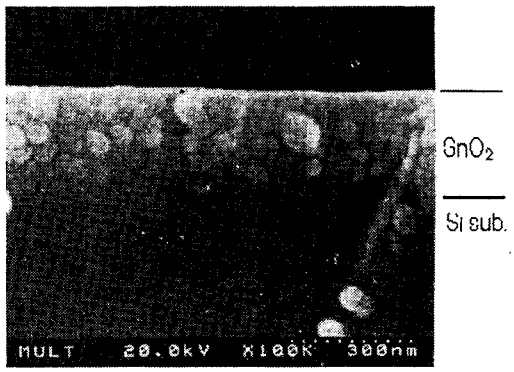
Fig. 6. Dependence of film thickness on (a) spin-speed of coating and (b) the number of coating applications.

The film thickness was controlled by spin-speed variation between 1500 rpm to 3000 rpm by step of 500 rpm and the number of coating application. The film thickness was monitored by stylus method and ellipsometer, and confirmed by cross sectional SEM view. The film thickness measured by SEM shows good agreement with the thickness values measured by stylus and ellipsometer. Figure 6 shows the relationship of SnO_2 and SnO_2(Sb) films between the film thickness and spin-speed. Film thickness decreased with increasing spin-speed. It was also found that there was a linear relationship between the film thickness and the number of coating application. In case of SnO_2 film, film thickness increased with an interval of about 74 ㎚ by increasing the number of applications from 1 to 4 at the spin-speed of 3000 rpm.

Figure 7 is typical cross sectional views of the SnO_2 films spin-coated at 3000 rpm on silicon substrate at 1 time and 4 times respectively. These SEM were photographed over the sample placed vertically. The thickness of the film, which was coated at a time, is about 70 ㎚ and that of the film, which was coated at 4 times, is about 300 ㎚. Figure 7 indicates the primary particles(=below 15 ㎚ diameter) form the large secondary tin oxide agglomerate(=averagely 50 ㎚ diameter). This agglomerate may possibly make the coated film into the porous film.



(a)



(b)

Fig. 7. Cross-sectional views of spin-coated SnO_2 films.

Aqueous solution of hydroiodic acid(57% HI in water, 99.99%) from Aldrich Chemical Company, Inc. was used as etching solution of SnO_2 film. The 150 ㎚ thick SnO_2 film made by sol-gel method was etched at 80 ℃ by photolithographic process using Posi-PR. As is shown in Fig.8. a high-resolution pattern(about 5 ㎛) could be realized. The etch rate was about 50 ㎚/min at 80 ℃. The etching of SnO_2 films in HI gives excellent pattern resolution and the film patterned by HI solution shows no sensitivity decreasing after etching.

The gas sensitivity of microsensor to CO gas was tested to evaluate the heater operation. The gas sensitivity(S) was defined as the ratio of the resistance in air(Ra) and in CO gas(Rg). Figure 9

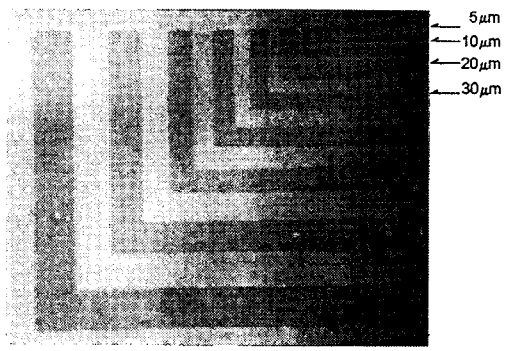


Fig. 8. Result of wet etching of SnO_2 film on silicon substrate.

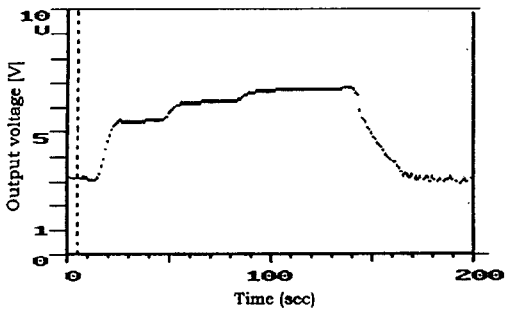


Fig. 9. Time response of the microsensor with SnO_2(Sb : 0.5 wt.%) film to CO gas.

shows the response characteristics of microsensor with SnO_2(Sb : 0.5 wt.%) film to three CO concentrations(2000, 3000 and 5000 ppm). The CO sensitivity of SnO_2(Sb) film is about 4 to 2000 ppm CO gas at 250 ℃ and the film shows fast response below 10 sec.

Ⅳ. Conclusions

Thermal properties of the two types of microheaters, the stack-type and the planar-type, were simulated by FEM commercial package and the planar-type heater was fabricated by silicon process technique. The stack-type structure with sensing layer/oxide/heater could save the power consumption for the high operating temperature. In this structure, the sensing layer could be heated by the heater situated just below of the sensing film. However, the planar-type structure with a sensing layer and a platinum heater on the same plane could be fabricated simple process using only 3 masks for photolithography process. In the planar-type structure, the sensing layer was heated by the heater situated out of the sensing film and the heating energy probably was transferred by thermal conduction mechanism of the PSG/Si_3N_4 diaphragm.

The planar-type heater structure proposed in this study could be available technology for simple fabrication process and comparatively low power consumption heating.

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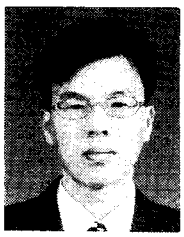

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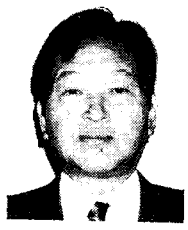

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