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Micromachined catalytic combustible hydrogen gas sensor

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ABSTRACT

An integrated catalytic combustion H_2 sensor has been fabricated by using MEMS technology. Both the sensing elements and the reference elements could be integrated into the suspended micro heaters connected in a suitable circuit such as a Wheatstone configuration with low power consumption. Two sensitive elements and two reference sensors were integrated together onto a single chip. The size of chip was 5.76 mm^2 and the catalytic combustion sensor showed high response to H_2 at operating voltage of 1 V. The response and recovery times to 1000 ppm H_2 were 0.36 s and 1.29 s, respectively.

1. Introduction

Various types of chemical gas sensors have been developed in order to detect toxic gases, which can be classified into three categories according to their sensing mechanism: electrochemical sensors, catalytic combustion sensors, and semiconductor gas sensors [1]. Catalytic combustion gas sensors have been widely used for many years to detect flammable gases or flammable vapor. Modern catalytic gas sensors called as "pellistors" was suggested by Baker [2]. Typically, the Pt coil is covered by catalyst-impregnated alumina bead and the temperature rise by combustion reaction is measured via the increase in the resistance of Pt coil [3–5].

Catalytic combustion gas sensors are now commercially available and mainly used as methane detectors for coal mining security [6]. It is advantageous to get high gas response with no influence water vapor, but high power consumption, a very low internal resistance, high cost and low reproducibility are the major challenges to overcome [7,8]. The slow response due to the poor ratio of sensing surface to mass should be also resolved. To detect combustible gases, the catalytic combustion sensors are usually heated to ~500 °C using Pt wire and the electric power for heating ranges from 300 to 700 mW. Therefore, the catalytic combustion sensors with low power consumption are very important for the longer operation of sensor using battery.

Micro-electromechanical systems (MEMS) technology is very attractive integrating and fabricating method as building block in the application of gas sensors. MEMS-based gas sensors, which have a small volume, low power consumption, low manufacturing cost for mass production, showed good gas sensing performance such as high gas response, fast response and recovery times [9–12]. In particular, the miniaturization of catalytic combustion gas sensors using MEMS technology is a promising solution to decrease the power consumption significantly. To date, many catalytic combustion gas sensors in the integrated form have been fabricated using thin film or micromachining techniques [13–15], which include the CH_4 sensor with the heater power of 100–250 mW [13], flammable gas sensors with the heater power of 100 mW [14]. Besides catalytic combustion type, thermoelectric type of H_2 sensor has been also proposed to reduce the sensor operation temperature [16].

H_2 is being widely used in industry, such as chemical production and fuel cell technology. Thus, the reliable and efficient detection of H_2 is very important to avoid any disaster from flammable and explosive H_2 [17]. In this contribution, an integrated design of catalytic combustion H_2 sensor with low power consumption has been designed and fabricated using MEMS technology. For the thermal insulation, the suspended micro bridge design is employed. In the sensors with a Wheatstone bridge, the sensor signal of two sensing elements configuration is 2 time higher than that of the single sensing element configuration [18]. Thus, in the present study, two the sensing elements and two reference elements are coated on the four suspended micro heaters with a Wheatstone configuration. To the knowledge of authors, this is the first report to fabricate the combustion type gas sensors with two sensing elements

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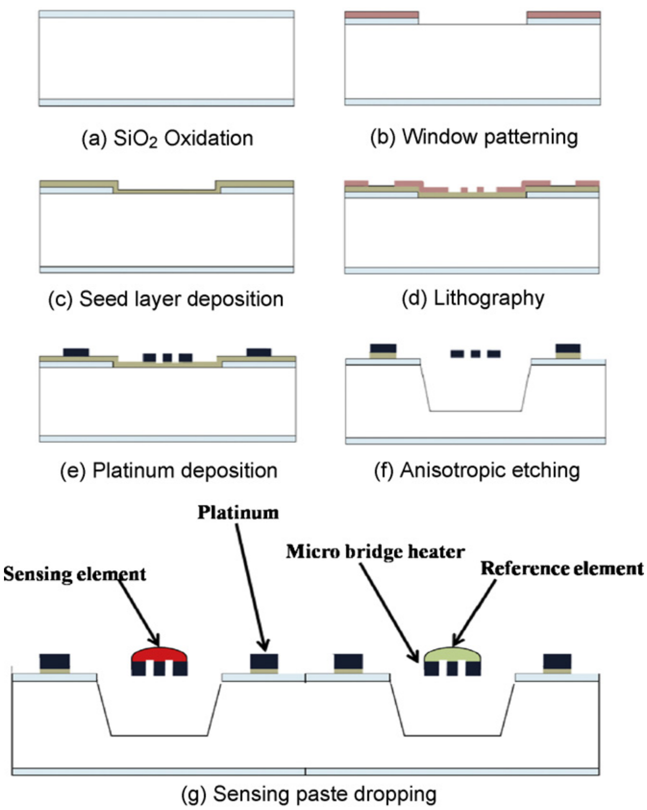

Fig. 1. Fabrication process flow for the catalytic combustion gas sensor.

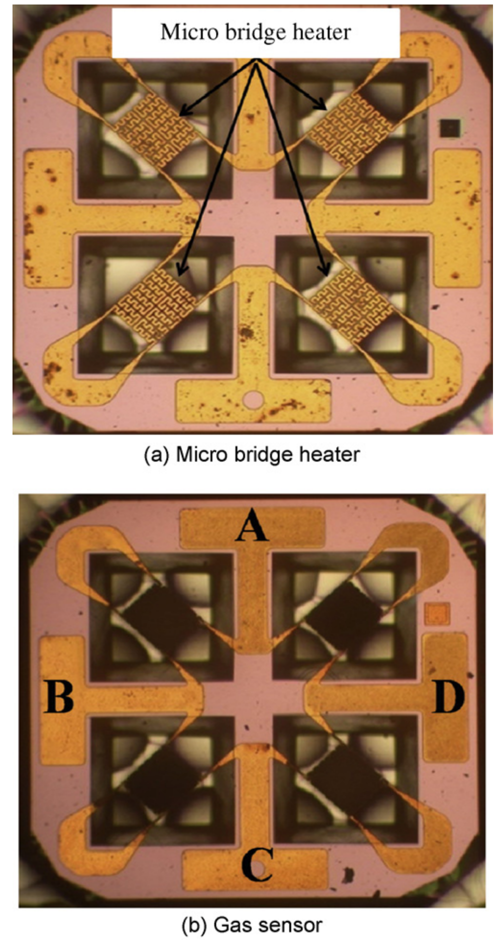

Fig. 2. A photograph of the fabricated sensor device (a) micro bridge heater and (b) gas sensor.

and two reference elements using MEMS technology. The fabrication process uses only two photo masks, which provides a simple and cost-effective approach to fabrication MEMS-based catalytic combustion sensor in a mass-production scale. The sensor with 2.4 mm × 2.4 mm in area selectively detects the low concentration of H_2 (20 ppm) with low total power consumption 55.68 mW. The sensor shows the responses in proportional to H_2 concentration and rapid response and recovery kinetics at the heater voltage of 1 V.

2. Experimental

2.1. Sensor preparation

The catalytic combustion gas sensors for H_2 has been fabricated on silicon wafers via the fabrication processes shown in Fig. 1. The substrate for the sensors device is a 4 in. n-type silicon wafer with a thickness of 520 μm and resistivity of 1–10 Ω cm. The substrate should be a good heat conductor and a good electric insulator. The Si wafers coated with silicon dioxide and silicon nitride are very suitable for integration with other components or for micromachining. After a standard cleaning procedure, a silicon dioxide film of ∼1.6 μm was deposited by thermal oxidation at 1100 °C. A photoresist was spin coated and then the window was patterned by exposure and development. An adequate hardbake process is important for the stability during the subsequent processing. The first mask was patterned in the onside of the substrate and the window was defined. A standard lithographic process was employed to pattern the cavity to be isolated and a Ti/Au seed layer for the electroplating was deposited by an E-beam evaporator. The thicknesses of the Ti and Au were 50 and 100 nm, respectively. An AZ 5214 photoresist is patterned on the seed layer, while the pure platinum layer is grown by an electroplating method. The thickness of the electroplated platinum film was 2.8 μm. Subsequently, the seed metal stripping is performed in wet etching solution and the front side of heater was removed by anisotropic etching to form a micro bridged structure. Fig. 2 showed the photograph of the fabricated gas sensor. The micro bridge heaters were used to heat the sensing elements and reference elements electrically to their operating temperature. And the sensor signal was measured between the heater. The temperature of the Pt heater at a given heater voltage was measured by observing the melting of temperature-sensitive crayons. In order to reduce the power consumption required to heat the sensor, the region on the side of the sensor was anisotropically etched using KOH solution onto the micro bridge heater of 2.5 μm in thickness. The micro heaters are suspended across a 200 μm deep cavity by platinum bridges and the area of the micro heater is 200 μm × 200 μm. The line width of the platinum bridge is 10 μm and the dimension of our sensor is 2.4 mm × 2.4 mm.

2.2. Sensing element

Catalytic combustion type sensors have gas sensing elements and compensation elements. To increase the response to H_2 and the number of detectable molecules, a catalyst such as platinum was added. The catalytic effects play an important role in improving the response to target gases and this is important for catalytic combustion type gas sensor. As sensing elements, the commercial γ-alumina (Sigma–Aldrich Co., Ltd.) was used. The 5 wt% Pt was loaded by the impregnation with H_2PtCl_6 (Heesung Metal., Ltd.) solution. In order to fabricate slurry containing sensing materials, the γ-alumina (reference element) and Pt loaded γ-alumina (sens-