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Characteristics of suspended planar-type gas sensor based on MEMS process

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Abstract: In order to simplify the fabrication process, distribute the temperature uniformly and reduce the power consumption of the micro-hotplate (MHP) gas sensor, a planar-type gas sensor based on SnO_2 thin film with suspended structure is designed through a MEMS process. Steady-state thermal analysis of the gas sensor and the closed membrane type sensor where the membrane overlaps the Si substrate is carried out with the finite element model, and it is shown that the suspended planar-type gas sensor has a more homogeneous temperature distribution and a lower power consumption. When the maximum temperature on the sensor reaches 383 ℃, the power consumption is only 7 mW, and the temperature gradient across the thin film is less than 14 ℃. To overcome the fragility of the suspended beams, a novel fabrication process in which the deposition of the gas sensing film occurs prior to the formation of suspended beams is proposed. The back side of the Si substrate is etched through deep reactive ion etching (DRIE) to avoid chemical pollution of the front side. The fabrication steps in which only four masks are used for the photolithography are described in detail. The Fe doped SnO_2 thin film synthesized by sol-gel spin-coating is used as the gas sensing element. The device is tested on hydrogen and exhibits satisfactory sensing performance. The sensitivity increases with the rise of the concentration from 50×10^{-6} to $2\,000 \times 10^{-6}$, and reaches about 30 at $2\,000 \times 10^{-6}$.

Key words: gas sensor; suspended planar-type; micro fabrication; gas sensing characteristic

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Gas sensors using semiconductor metal oxides (MOS) as the active material have been used to detect a variety of gaseous species[1-3]. In particular, SnO_2 has been extensively studied and used as the base material in devices such as the Taguchi. The integrated gas sensor with MOS thin film demonstrates better sensitivity, faster response, and lower power consumption than traditional thick film devices[4-5]. Its working principle is based on the change in conductivity that takes place after exposure to gases capable of reacting with chemisorbed oxygen. But the reaction generally occurs at temperatures in the range of 150 to 600 ℃ and, therefore, the sensor must be heated to obtain a suitable response.

The micro-hotplate (MHP), an important component, is used to heat the sensing thin film up to the operating temperature so as to make the sensor work in the best condition [6-7]. The thermal characteristics of the MHP influences the gas sensing property and the application of the sensor significantly. First, a uniform temperature profile across the MHP, especially within the sensitive area of the inter-digitated electrode (IDE), is essential in order to obtain a short response time, high sensitivity and good selectivity of the sensor[8]. Secondly, for most of the applications, it is necessary to use a sensor device with very low thermal conductivity to avoid the heat cross-talk with the other devices and the corresponding integrated circuits that compose the whole device[9]. Finally, the lower power consumption of the heater element is important to allow battery-power operation in portable detectors and long term operation of the device.

One way to improve the thermal characteristics of the MHP is to properly design the geometry of the isolation membrane. Many papers show a suspended membrane for this purpose[10-11]. The geometry of the membrane and the number of the arms can be varied.

In this paper, a new structured micro gas sensor named the suspended planar-type micro gas sensor, in which a heater, temperature sensor and an IDE are deposited on the same plane of the diaphragm, is proposed. This work is accomplished by deposition of a Fe doped SnO_2 thin film over the MHP as a sensing layer. Thermal FEM simulations are performed to achieve a homogeneous temperature distribution and lower power consumption.

1 Structure Design

1.1 Structure modeling

The whole chip size is about 2 mm × 2 mm × 0.35 mm. The heaters are two tapes of Pt 20 μm, folded into two double-track structures surrounding the temperature sensor which are located around the central IDE. The IDE

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has a 10 μm finger width and a 5 μm electrode spacing. The sketch of the electrode group is shown in Fig. 1. A window is opened in the Si substrate to lower the power consumption. The 700 nm thick SiO_2 thermal insulation between the Si substrate and the electrode group is etched to define a suspended structure. The sensitive area is connected with the Si substrate only through four suspended beams with a width of 50 μm and a length of 100 μm. The SnO_2 sensing film is deposited on the electrode group by the sol-gel spin-coating method. The area of the sensing film is about 520 μm × 520 μm. The structure of the sensor is shown in Fig. 2.

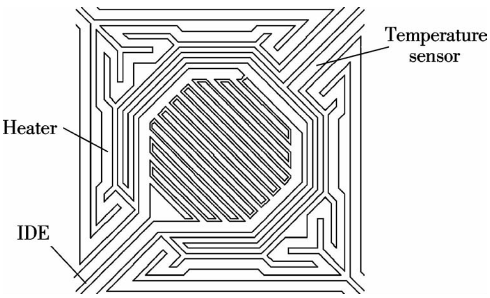

Fig. 1 Layout of the electrode group

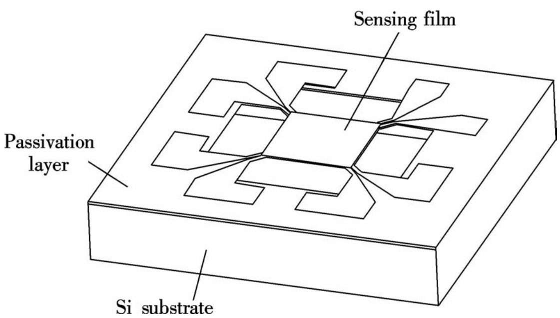

Fig. 2 Sketch of the suspended planar-type micro gas sensor

To compare with the suspended structure, a closed membrane type sensor where the membrane overlaps the Si substrate along its periphery is considered in this paper as shown in Fig. 3.

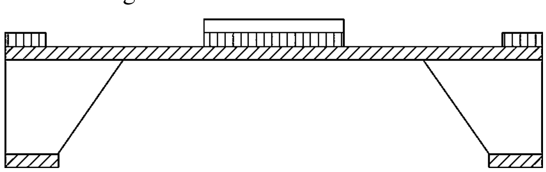

Fig. 3 Cross-section of the closed membrane type sensor

1. 2 Simulation results and discussion

The design of the device is supported by thermal simulations with the FEM analysis tool ANSYS. For the simulation only convective energy losses are taken into account since the irradiative energy losses are very small due to the small area of the heated plate[12]. The heat is obtained through loading the heat generation rate on the heater. The simulation results of the closed membrane type sensor and the suspended planar-type sensor can be seen in Fig. 4 and Fig. 5.

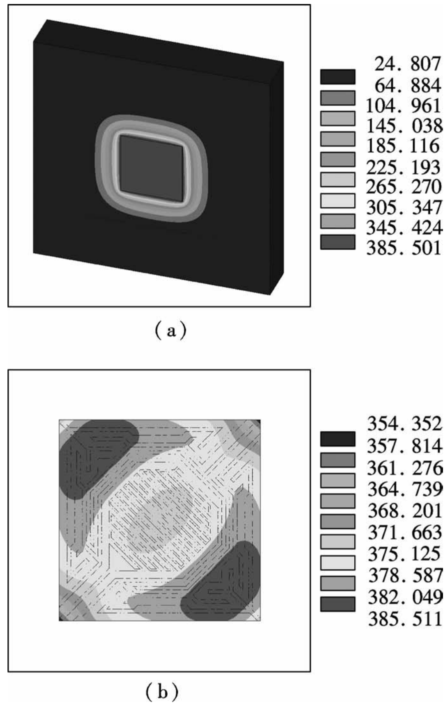

Fig. 4 Temperature distribution of the closed membrane type sensor. (a) Temperature distribution of the device; (b) Temperature distribution across the thin film

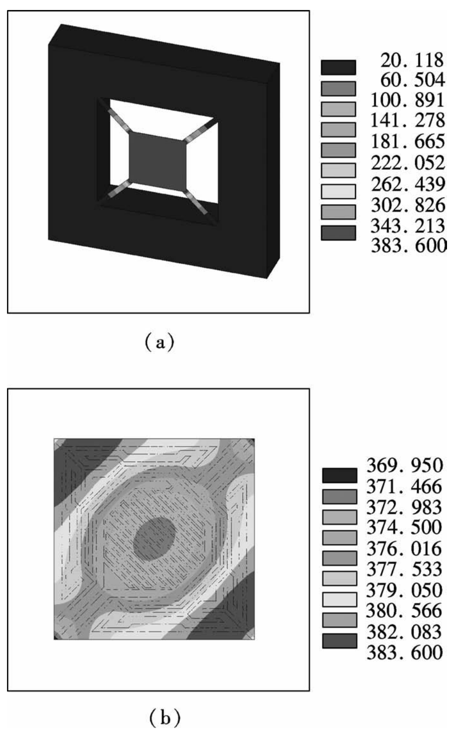

Fig. 5 Temperature distribution of the suspended planar-type sensor. (a) Temperature distribution of the device; (b) Temperature distribution across the thin film

From Fig. 4 we can see that the sensor obtains a reasonable temperature distribution on the whole device. The maximum temperature which appears on the sensing film reaches 385 ℃ and the power consumption is about 15 mW. The temperature on the Si substrate is about

24 ℃. But the temperature difference on the sensing film is more than 30 ℃, which may interfere the efficiency of the sensing film obviously.

In Fig. 5(a), it is deduced that the temperature drops radically at the anchors of the beams. When the temperature of the micro-hotplate is set to 380 ℃ in the simulation tool, the power consumption is about 7 mW, much lower than that of the closed membrane type. The Si substrate around the structure has a temperature about 20 ℃, being very close to the ambient temperature. From Fig. 5 (b), it can also be seen that more uniformity of the temperature is achieved in the sensing film. The temperature varies from 383.6 ℃ of the heater to 369.95 ℃ of the periphery. Furthermore, the temperature non-uniformity across the sensitive area of the IDE is about 6 ℃, meaning that the sensing film can be more sensitive to target gas.

Although the closed membrane type sensor shows good thermal characteristics, there is still a lot of heat transmitted to and wasted on the Si substrate. It will lead to a higher power consumption of the device and a higher temperature on the Si substrate. The suspended type sensor exhibits better thermal characteristics. Both the power consumption of the device and the temperature on the substrate are lower than those of the closed membrane type. It is because the sensitive area is connected with the substrate only through four suspended beams. Heat transmitted to the Si structure will be limited both by a small thermal conductivity coefficient of the SiO_2 and by a low heat transfer area on the beams. So when the sensor array is used, the thermal cross-talk between the individual sensors can be minimized. This also assures that the temperatures of the surrounding regions are low, so that the sensor peripheral circuits can work reliably under low temperatures.

2 Device Fabrication

In spite of the high quality performance of the thermal characteristics, the application of the suspended structure still may be restricted due to some drawbacks related to fragility. In the traditional fabrication process, the sensing film is deposited after the fabrication of the MHP. But the suspended beams are too fragile to support some processes such as spin-coating, lithography, washing and so on. So, when the gas sensing film is deposited on the top of the electrodes, the small arms break after some time.

To solve the question above and improve the yield of the device, the gas-sensitive material can be deposited prior to the Si and SiO_2 micromachining. In this way, the suspended structure can be formed in the last step. The basic technological steps for the device fabrication (see Fig. 6) can be summarized as follows:

1) A SiO_2 layer with a thickness of 700 nm is thermally grown on each side of the Si substrate. This layer is used as a thermal insulation layer[13] on the front side as well as a passivation layer during etching on the back side (see Fig. 6(a)).

2) A bi-layer of Ti/Pt (20 nm/200 nm) for the electrode group is deposited by magnetron sputtering. The geometry of the heater is defined by the lift-off technology (see Fig. 6(b)).

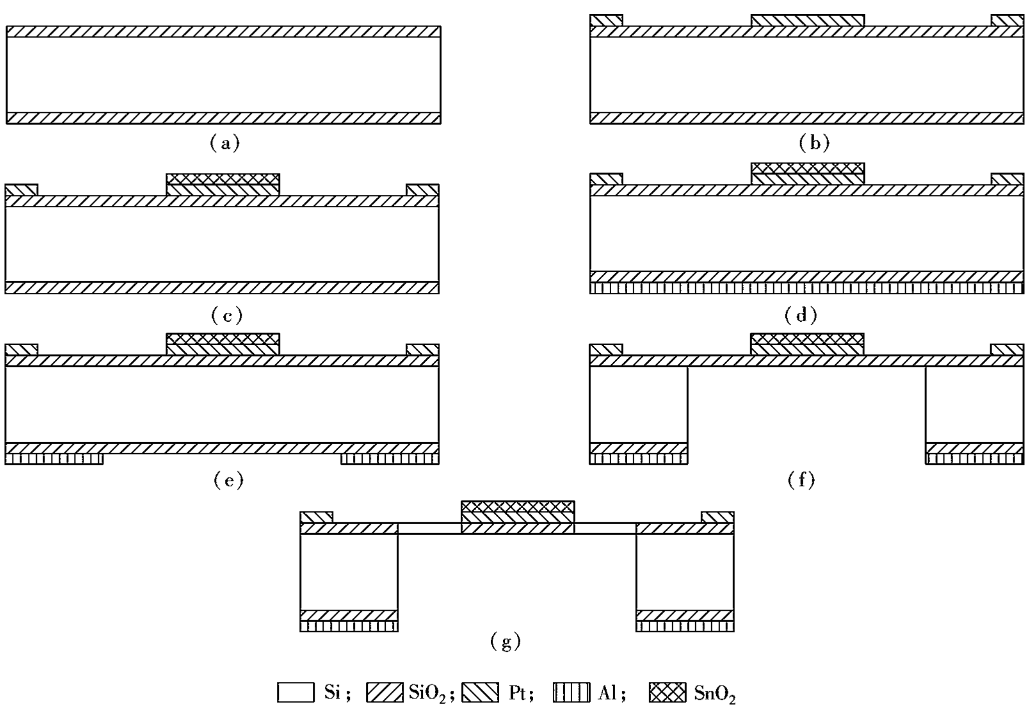

Fig. 6 Cross-sections of the fabrication process

3) A layer of Al is deposited and patterned on the bottom of the Si/SiO_2 through magnetron sputtering and lift-off. This layer is used to be a mask layer for further dry etching(see Fig. 6(c)).

4) The SiO_2 layer on the bottom is etched through RIE with the Al mask (see Fig. 6(d)).

5) The Fe doped SnO_2 thin film is deposited on the top of the electrode group through the sol-gel spin-coating method and wet-etched to form a sensing film (see Fig. 6(e)).

6) The backside of the Si substrate is removed by deep reactive ion etching (DRIE). The vertical walls can be obtained by DRIE of the silicon. The densities of the devices fabricated through this technology are increased as compared with using wet anisotropic etching of silicon. DRIE also gives more flexibility in the design of the device in such a way that the crystal planes of the silicon substrate do not restrict the geometry of the membrane. Moreover, chemical pollution during the wet etching can be avoided, so no protection of the device front side is required during the silicon etching when the sensing film is deposited prior to the silicon micromachining(see Fig. 6 (f)).

7) The suspended structure is defined in the front side by RIE(see Fig. 6(g)).

The micro gas sensor can be fabricated by using four masks for the photolithography processes. The micro image of the device is shown in Fig. 7.

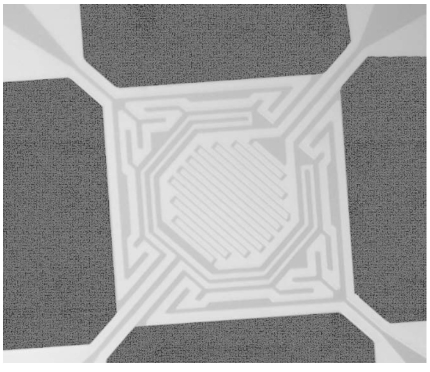

Fig. 7 Micro image of the device

3 Fe Doped SnO_2 Film Preparation

In this study, Fe doped SnO_2 nanoparticles are chosen as the sensing film. Among the semiconducting metal oxides used for gas sensors, SnO_2 is the most widely used material due to its low cost, long life and good reproducibility. Up to now, tin oxide sensors can be fabricated into three types of devices, i. e. sintered block, thick film and thin film. Among them, thin film devices have attracted a wide range of attention due to their fast response, low fabrication cost, and suitability for the suspended structure[14].

To improve the gas-sensing characteristics, semiconductor based gas sensors doped with Cu, Fe, Pd, Ag, Pt, or their composites are used. In particular, the transition metal oxide additives are known to enhance surface adsorption sites by inhibiting crystallite growth in the basic SnO_2 matrix. However, among the various transition metal oxides, iron oxide is preferred due to its wide solid solubility and formation of a stable compound over a wide range of Sn/Fe ratios[15-16].

The Fe doped SnO_2 gas sensing film is deposited onto the MHP by the sensing layer deposition method called spin-coating. The synthesis of the thin film is based on processing with $SnCl_2 \cdot 2H_2O$, $FeCl_3$ and C_2H_5OH. The most noteworthy property of this procedure, in comparison with the method such as CVD, sputtering, etc., is the simplicity of the method. Other remarkable characteristics are the repeatability, mass-production and low cost of this method. Then the chemical reagent is mixed in the ethanol in a certain ratio and stirred for 3 h in a closed vessel. This solution is kept for 24 h at room temperature and the transparent hydrosol is obtained. The gel is spin-coated onto the Si/SiO_2 substrate at a speed of 3 000 r/min for 30 s. The specimen is dried at 100 ℃ for 15 min to evaporate the solvent and to remove organic residuals, and then a new layer is deposited. The coating and the drying processes are repeated ten times until the desired thickness is reached. And then the film is sintered in air for 2 h. As a result, the 2% Fe doped SnO_2 thin film is obtained. It can be clearly revealed from Fig. 8 that the average gain size of the thin film is about 40 μm and the thickness is about 200 nm.

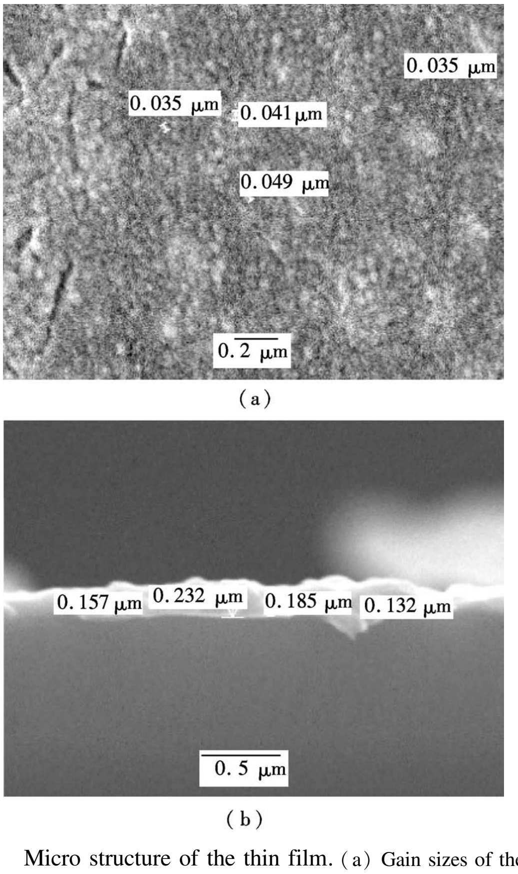

Fig. 8 Micro structure of the thin film. (a) Gain sizes of the thin film; (b) Thickness of the thin film

4 Gas-Sensing Properties

The gas test of the gas sensor is carried out in a sensor test system designed by our group[17]. Briefly, a desired gas concentration is obtained in a stainless steel test chamber, where the sensor is placed, by means of several mass flow controllers (MFC). USB6215 of National Instruments performs the gas sensor data acquisition. The pure SnO_2 thin film and Fe doped SnO_2 thin film have been prepared, respectively, and the micro sensor has been tested in a hydrogen atmosphere.

Fig. 9 gives the sensitivity curve of the sensor with Fe doped SnO_2 to the hydrogen concentration between 50×10^{-6} and $2\ 000 \times 10^{-6}$ at operation temperatures of 250, 300 and 350 ℃. We can see from Fig. 9 that the best response property appears at a temperature of about 300 ℃ and this temperature is used in all measurements. The sensitivity to hydrogen depends on the concentration almost linearly from 50×10^{-6} to $2\ 000 \times 10^{-6}$ at the three temperatures. In this paper, the gas response sensitivity is defined as $S = R_a / R_g$, where R_a and R_g are the resistance values of the thin film measured in clean air and air containing test gas, respectively.

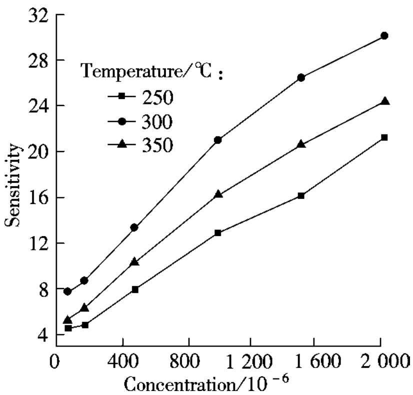

Fig. 9 Sensitivity at 250 and 300 ℃

The sensitivity of the micro sensor with pure SnO_2 thin film and Fe doped SnO_2 thin film in the hydrogen concentrations between 100×10^{-6} and $2\ 000 \times 10^{-6}$ is shown in Fig. 10. It is clearly seen that the response is significantly higher for Fe doped SnO_2. The sensitivity at the low concentration of 50×10^{-6} is still 8.

The response and recovery time curves of the micro sensor with pure SnO_2 thin film and Fe doped SnO_2 thin film in the concentration range of 50×10^{-6} to $2\ 000 \times 10^{-6}$ at 300 ℃ are shown in Fig. 11 and Fig. 12. The response and recovery time of the Fe doped SnO_2 thin film sensor are found to be about 15 s and above 100 s, respectively, for all the concentrations, much shorter than those of the pure SnO_2 thin film sensor. But the response and recovery time are still a bit long. It may result from the compact structure of the thin film which can be seen in Fig. 8. The response and recovery process will be shorter by improving the porosity of the film.

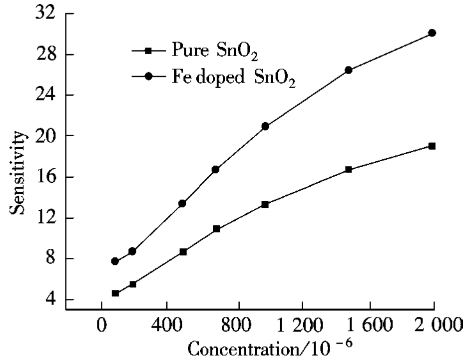

Fig. 10 Sensitivity of the sensor attached to Fe doped SnO_2 thin film and pure SnO_2 thin film

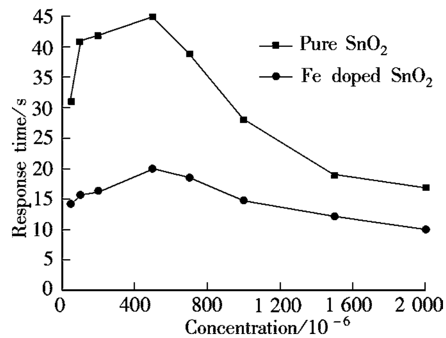

Fig. 11 Response time of pure and Fe doped SnO_2 thin film sensor

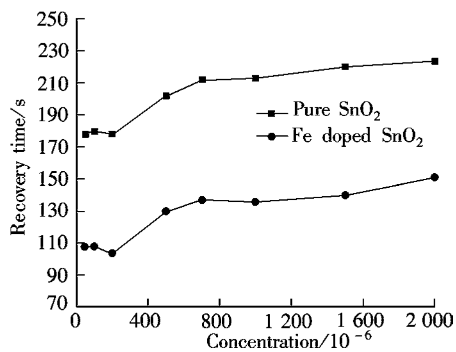

Fig. 12 Recovery time of pure and Fe doped SnO_2 thin film sensor

5 Conclusion

A suspended planar-type gas sensor based on Fe doped SnO_2 thin film is designed and fabricated. Both the thermal insulation layer and the four suspended beams are made of 700 nm of SiO_2. The uniformity of temperature distribution over the sensing layer and the low power consumption are confirmed to be satisfactory as designed by the thermal FEM simulation. To solve the drawback related to the fragility, a novel fabrication process in which the deposition of gas sensing film occurs prior to the formation of the suspended structure is proposed. The gas sensor attached to the Fe doped SnO_2 thin film exhibits satisfactory sensitivity to hydrogen at 300 ℃, but the response and recovery time is a bit long.

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基于 MEMS 工艺的悬臂共面式气体传感器性能分析

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摘要：为了简化制作工艺，使温度分布均匀以及降低功耗，设计了一种基于 MEMS 制造工艺的悬臂共面式 SnO_2 气体传感器. 使用有限元法对这种传感器及膜结构堆积于硅基底上的封闭膜式气体传感器进行了稳态热分析，结果表明悬臂共面式传感器拥有更均匀的温度分布和更低的功耗. 当最高温度为 383 ℃时功耗仅为 7 mW，敏感薄膜上的温差低于 14 ℃. 为解决悬臂易碎的问题，提出了一种新的制造工艺，该过程在正面刻蚀 SiO_2 层形成悬臂结构前沉积 SnO_2 敏感薄膜，并采用深反应离子刻蚀的方法对硅基底进行体刻以避免湿法刻蚀对传感器表面的化学污染. 整个过程总共需要 4 块掩模板. 采用旋涂法溶胶凝胶法将掺有 Fe 离子的 SnO_2 薄膜沉积于基底上作为敏感元件. 该器件对氢气表现出了良好的气敏性能，随着氢气浓度从 50×10^{-6} 上升到 $2\,000\times10^{-6}$，灵敏度逐渐提高，在 $2\,000\times10^{-6}$ 时的灵敏度为 30.

关键词：气体传感器；悬臂共面式；微加工；气敏性能

中图分类号：TP212.2