Fabrication of a MEMS micro-hotplate

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Abstract. With the development of microelectronics and micromachining technology, micro-hotplate as a micro-heater is a very important component in micro-sensor. So far, it has wide applications in pressure detection, gas detection and so on. In this paper, a micro-hotplate based on micro-bridge structure was designed and fabricated. Here we present a micro-hotplate with SOI (silicon-on-insulator) as the substrate and metal Au as the heating electrode material. It shows low thermal conductivity coefficient, good electrical insulation, low power consumption, and excellent resistance to high temperature and low pressure. Some IC (integrated circuit) processes such as magnetron sputtering of Ni/Cr, ion beam sputtering of Au, dry and wet etching processes are employed to fabricate the device. Anisotropic wet etching of silicon and thin film deposition were mainly studied. To protect the front side micro structure during the wet etching process, a special apparatus was designed and fabricated. The optimization process for anisotropic wet etching was obtained for releasing the micro-bridge after a large number of experiments and the micro-hotplate was successfully fabricated. The electrical, thermal characteristics of the micro-hotplate were tested and approved it a well functional device.

1. Introduction

MEMS (micro electro-mechanical systems) is a high-tech and cross field to combine microelectronics with micromachining technology to integrate micro components, micro sensors, micro actuators, signal processing and control circuit etc\cite{6}. In the common silicon substrate, micro-hotplates generally consist of a thin dielectric membrane suspended over an opening in a silicon substrate. In Microsystems technology, hotplates are mainly used for sensor applications where the sensing material is deposited onto the membrane and an electrical signal is also integrated in the hotplate\cite{7}. Hotplates operate at temperatures from 200 to 400 degree. With the development of MEMS technology, micro-hotplate based on MEMS has gradually gained its wide applications on gas sensors\cite{8}, infrared detectors, infrared sources\cite{9}, infrared emitters, actuators etc\cite{10}. By employing some thin films with good thermal insulation as the substrate, the micro-hotplate presents a series of advantages such as miniaturized size, low power consumption, fast response, high sensitivity, good reproduction and feasibility of sensor array\cite{11} integration.

As the core component of MEMS sensors, structure, materials, mechanical and thermal design, processing technology of micro-hotplate varies for different applications. Here we present a
micro-hotplate with silicon-on-insulator (SOI) as the substrate and metal Au as the heating electrode material. It shows low thermal conductivity coefficient, good electrical insulation, low power consumption, and excellent resistance to high temperature and low pressure. Au has a good conductivity, small electromobility, stable TCR (temperature coefficient of resistance) and chemical properties. In accordance with the principle of structural design and the conditions of laboratory, the fabrication processes are designed for the micro-hotplate.

In this work, some IC processes such as magnetron sputtering of metal Ni, ion beam sputtering of Au, dry and wet etching processes are employed to fabricate the device. Anisotropic wet etching of silicon and thin film deposition were mainly studied. Through the study of principle of the wet etching of silicon and a lot of corrosion experiments, the characteristics of wet etching were analyzed and the micro-bridge structures were designed to be compatible with the wet etching processes. TMAH is used to be the etchant. To protect the front side micro structures during the wet etching process, a special apparatus with fixture was designed and fabricated. After doing many experiments, we found it works very well, no TMAH solution was found to corrode the front side structures. The optimization process for anisotropic wet etching was obtained for releasing the micro-bridge after a large number of experiments and the micro-hotplate was successfully fabricated. The electrical, thermal characteristics of the micro-hotplate were tested and approved it a well functional device.

2. Design of the micro-hotplate

There are two types of micro-hotplate structure: the closed-type membrane, where the membrane overlaps the silicon substrate along its periphery and the suspended-type membrane, where the membrane is supported on the Si substrate by means of supporting beams. In both cases, the membrane lies over a cavity etched in the silicon substrate. In the latter case, the thermal losses to the substrate take place only through the supporting beams, and thus they are minimized compared to the closed-type membrane. Micro-hotplate designed here belongs to the suspended-type membrane structure. It can be used as the public heating source platform of many different narrow-band infrared emitter structures, such as based on two-dimensional photonic crystal.

Figure 1. Schematic diagram of heat transfer of micro-hotplate.

The total heat loss of the micro-hotplate consists of conduction, convection and thermal radiation. As shown in figure 1, it includes: (1) heat conduction through the supporting beam to the surrounding frame region, (2) conduction, convection and thermal radiation through the high-temperature bridge surface (here assumed to be atmospheric environment, therefore the thermal conduction of air is also included).
The total heat loss can be expressed with the following formula (1), the first describes the heat conduction through the supporting beam, the second is conduction and convection of air, the third is thermal loss brought from the thermal radiation.

\[
Q_{\text{total}} = G_m \lambda_m (T_{\text{hot}} - T_{\text{amb}}) + G_{\text{air}} (h_f + \lambda_{\text{air}})(T_{\text{hot}} - T_{\text{amb}}) + G_{\text{rad}} \sigma (T_{\text{hot}}^4 - T_{\text{amb}}^4)
\]

(1)

Where \( \lambda_m \) and \( \lambda_{\text{air}} \) are thermal conductivity of membrane and gas respectively, \( h_f \) is the gas convection coefficient, \( G_m, G_{\text{air}} \) and \( G_{\text{rad}} \) are geometric factor related to the membrane structure, \( \sigma \) is the Stefan-Boltzman constant \( (5.67 \times 10^{-8} \text{W/(m}^2\text{T}^4)) \), \( \varepsilon \) is the surface emissivity, which range from 0 to 1, \( T_{\text{hot}} \) and \( T_{\text{amb}} \) are temperature of hotplate and ambient environment respectively.

We hope to obtain relatively high infrared emissivity, so the size of the micro-hotplate we design is larger, whose centre area is \( M \) order of magnitude. For such a large size, it is very difficult for us to use coating method to fabricate the non-closed membrane structure. In order to get the higher mechanical strength, improve device yield and simplify the production process, we adopt the SOI substrate to produce micro-hotplate, so the simulation of structure is also based on SOI substrate.

The micro-hotplate structure is shown in figure 2, to get the uniform heating effect, electrode use hole-like structure, as shown in Figure 3. For SOI we use, the thickness of underlying silicon is 675nm, the middle buried oxide layer is 500nm, and the top silicon is 10\( \mu \)m. The thickness of upper \( \text{SiO}_2 \) is 500nm, and gold resistance wire film is 100nm. The centre size of micro-hotplate is 2\( \text{nm} \times 2\text{nm} \), the width of supporting beam is 50\( \mu \)m, and the length is 1400\( \mu \)m.

![Figure 2. Schematic diagram of micro-hotplate structure.](image)

(a) Two-dimensional sectional view; (b) Heating electrode of front side.

3. Fabrication of the micro-hotplate
The fabrication process is based on standard technologies used in semiconductor industry; some IC processes such as magnetron sputtering of metal Ni/Cr, ion beam sputtering of Au, dry and wet etching processes are employed to fabricate the device. The process flow is shown schematically in figure 3. It starts with \(<100>\) orientation SOI wafers with a 500\( \mu \)m silicon oxide film grown by thermal oxidation both at front side and back side. The basic process steps are stated as follows:
Figure 3. The process flow chart of micro-hotplate.

3.1. Fabrication of Au electrode
Metal film hotplate offer precise temperature control, which can also be used as thermometer, it greatly simplifies the device structure and fabrication technology. Due to metal gold some good electrical and thermal features, we select Au as the heating electrode. Here we adopt the lift-off process. First, the ENPI negative photoresist is spin coated on the front side of substrate, the inverse electrode pattern is formed by photolithography process, so the photoresist section comes into being. Then a RF magnetron sputtering of Ti layer is deposited on top of the formed oxide to improve adhesion between Au and SiO$_2$ substrate, the thickness of Ti is about 10nm. Au layer is deposited via ion beam sputtering, process parameter is shown in table 1, and its thickness is about 110nm. Metal layer covers the remaining photoresist as well as photoresist opening. The last step is to use stripper to remove the photoresist. Metal film attached to photoresist is stripped from the substrate, which deposited directly onto the substrate will be retained.

Table 1. Process parameters of ion beam sputtering Au film.

| Parameter            | Screen-grid voltage | Screen-grid current | Accelerating-grid voltage | Air pressure | Substrate temperature | Time   |
|----------------------|---------------------|---------------------|---------------------------|--------------|-----------------------|--------|
| Value                | 500V                | 50mA                | 100V                      | $3 \times 10^2$Pa | 80°C                  | 20min  |

In order to strip effectively, the following requirements must be met: (a) the thickness of photoresist mask is greater than the thickness of metal layer; (b) after developing, the profile of the photoresist mask section goes to the next narrow width of Chinese character “八” shape, so the metal deposited on the substrate and metal on the photoresist film will be disconnected completely. When the photoresist layer is washed away, the metal film on it is lifted-off and washed together with ENPI photoresist layer. After this, the metal Au remains only in the region where it has a direct contact with substrate.

3.2. Fabrication of Ni/Cr mask and dry etching of Si
During the experiment, we found that Au layer will also be etched while RIE etching of Si, the surface
of Au layer becomes rough. If etching time is long enough, Au layer will be etched completely. This is due to the hardness of Au is relatively low; the physical bombardment etches the Au as well as Si. So a Ni/Cr protective layer is added above Au layer. RIE etching rate of Ni/Cr is small, 100nm Ni/Cr protective layer can be enough.

Table 2. Process parameters of DC magnetic sputtering Ni/Cr film.

| Parameter          | Current | Base pressure | Air pressure | Substrate temperature | Time |
|--------------------|---------|---------------|--------------|-----------------------|------|
| Value              | 0.5A    | $3 \times 10^3$Pa | $3 \times 10^2$Pa | Room temperature     | 5min |

The etching mask of Ni/Cr is deposited via the DC magnetron sputtering, the process parameter is shown in table 2. The Au and the Ni/Cr layer are both less than 200 nm, so ordinary lift-off can fulfill the requirement. After that, front side etching window is opened. Subsequently, the front side silicon dioxide and the upper silicon of SOI are etched via RIE process. Dry etching of Si and SiO$_2$ is performed with fluorine-based chemistries, such as CHF$_3$ or SF$_6$, which break up into free fluorine atoms in the course of glow discharge, and then react with silicon to generate gaseous substance, finally achieve the purpose of etching. After several experiments, we have the best process flow. RIE dry etching process is as follows:

3.2.1. Remove the residual photoresist: O$_2$: 80SCCM, power: 100W, time: 1min;

$$C_2H_6 + O_2 + e^- \rightarrow CO + H_2O$$  \hspace{1cm} (3.2.1)

3.2.2. SiO$_2$ etching: CHF$_3$:O$_2$ = 80SCCM:15SCCM, power: 100W, time: 3min;

$$CHF_3 + e^- \rightarrow CHF_2^+ + F + 2e^-$$  \hspace{1cm} (3.2.2)

$$SiO_2 + 4F \rightarrow SiF_4 + O_2$$  \hspace{1cm} (3.2.3)

3.2.3. Si etching: SF$_6$:O$_2$ = 40SCCM:5SCCM, power: 100W, time: 15min.

$$SF_6 + e^- \rightarrow SF_5^+ + F + 2e^-$$  \hspace{1cm} (3.2.4)

$$Si + 4F \rightarrow SiF_4$$  \hspace{1cm} (3.2.5)

Hence, the front side processes are achieved. The SEM photographs of structure after RIE etching is shown in figure 4.

Figure 4. The SEM photographs of structure after top silicon RIE etching.

3.3. SiO$_2$ wet etching of back side etching window

To release the micro-bridge structure, we will remove the underlying silicon. So it is necessary to etch
the back side. Before doing this, the front side must be protected by spin coating a photoresist layer. Then the back side alignment is processed. Wet etching of SiO2 is carried out at room temperature, so photoresist can be used as mask layer. The adhesion of photoresist is to be very good, and it must be fully dried to prevent lateral corrosion. In order to enhance the adhesion of photoresist, we adopt two steps as following: (1) Place the substrate in the baking oven for 20~30 min to remove water on the surface thoroughly; (2) Spin coating a tackifier layer of HMDS (Hexamethyldisilizane) prior to photolithography.

BHF (buffered hydrofluoric acid) solution is used to etch the back side oxide layer; it is a mixture of 5 part 40% NH4F: 1 part 49% HF by volume. To obtain stable etch rate, the prepared BHF solution must be sit at least 1 hour. In the etch process, we can clearly see the color change of substrate, the purple SiO2 disappear gradually, exposing the Si color. After etching in BHF for 5 min, the back side etching window is opened. The chemical reaction as

\[ SiO_2 + 4HF + 2NH_4F \rightarrow (NH_4)_2SiF_6 + 2H_2O \]

(3.3)

3.4. Anisotropic wet etching of Si

TMAH (tetramethyl ammonium hydroxide) is the most preferred ammonium hydroxide based silicon etchant, it is non-toxic to humans, no damage to integrated circuits, and compatible with CMOS technology. In addition, while using TMAH to etch silicon; thermal oxidation of silicon dioxide can be a good mask medium.

Schematic of equipment for silicon wet etching system is shown in figure 5, which is set up by thermostatic magnetic stirrer. It has function of temperature control and magnetic stirring to facilitate uniform etching. With the process of etching, due to chemical reaction and volatilize along with heat, solution concentration and components will change. In order to maintain the stability of TMAH solution concentration, we add a water cooled condensation-reflux system. Reflux can reduce the loss of volatile TMAH solution by the condenser. All experiments were carried out in a closed glass vessel with constant temperature bath.

![Figure 5. Schematic diagram of silicon anisotropic wet etching system.](image)

The thickness of SOI wafer we used is about 675nm, so it takes 6~7 hours to etch fully. Substrate
is immersed in TMAH solution so long that the front side structure will be destroyed, even the metal electrode will come off, and therefore it is necessary to take some measures to protect front side structure. A fixture is designed and made. The fixture material is Teflon, besides; all fluoride or Teflon coated O-seal ring is adopted. This fixture can be used repeatedly, just only replace the washer. The fixture we designed is shown in figure 6.

![Figure 6. Structure diagram of Si TMAH wet etching fixture.](image)

It mainly consists of three parts, as we can see from the figure 6, part A is a lid with screw thread, part B is a circular gasket, and part C is a sleeve with screw thread as well as a hole in the middle of it. The gasket and sleeve both have a groove which can place O-ring washer. Place the back side of substrate down in the middle of the sleeve, then cover the gasket on the substrate, finally tighten the lid. To prevent substrate from moving during the tightening process, we usually apply a small amount of glue-502 to the washer of sleeve to fix the substrate. In addition, packaged in the water can reduce the probability of breaking of substrate caused by the stress changes during the wet etching. After doing many experiments, we found it works very well, no TMAH solution was found to corrode the front side structure.

Wet anisotropic etching of silicon is accomplished by using 25% TMAH solution. After six-hour 90 degree water bath heating, the underlying silicon is etched entirely. The back side etching diagram is shown in figure 7. As we can see from the figure, the result of wet release is good. But due to etching time is too long, (111) plane is also be etched partly. Therefore, the etch window’s size can be designed larger, so as not to effect the device function.

![Figure 7. The SEM photographs of back side after wet etching.](image)
3.5. Etching of the middle buried oxide layer and removal of Ni/Cr mask

The etching of buried oxide layer can be completed by wet etching as well as dry etching, specific methods has been introduced in the previous section. While Ni/Cr mask layer is removed by Ni/Cr etchant TFN $^{[8]}$, which is a mixture of 20% $(\text{NH}_4)_2\text{Ce(NO}_3)_6$, 6% HNO$_3$ and 74% H$_2$O. The reaction occurs at room temperature, etching rate is fast, is about 80nm/min. Because gold’s color is yellow we can see clearly, there is no need to control etching time precisely to control the thickness of nichrome. Only observe the surface color of substrate in the etching process, if gold color emerges, prolong a few seconds, then clean the substrate carefully. Eventually, the micro-hotplate is successfully fabricated. The SEM graphic of micro-hotplate we make is shown in Figure 8.

![Figure 8. The SEM photographs of finished micro-hotplate.](image)

4. Conclusion

A micro-hotplate with silicon-on-insulator (SOI) as the substrate and metal Au as the heating electrode material is designed and fabricated. Some IC processes such as magnetic sputtering, ion beam sputtering, dry and wet etching processes are employed to fabricate the devices. Anisotropic wet etching of silicon and thin film deposition were mainly studied. Through the study of principle of the wet etching of silicon and a lot of corrosion experiments, the characteristics of wet etching were analyzed and the micro-bridge structures were designed to be compatible with the wet etching processes. The optimization process for anisotropic wet etching was obtained for releasing the micro-bridge after a large number of experiments and the micro-hotplate was successfully fabricated.

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