Development of RIE Processes for the Etching of Single Crystal Silicon, Silicon Dioxide

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Abstract: Etching of micro-structures in a single crystal Si, SiO₂ has been obtained using reactive ion etching with SF₆/O₂ and CF₄/O₂ gas mixtures respectively. The variation in etch rate of Si and SiO₂ has been observed by varying gas composition, reactants flow rate, pressure and duration of plasma process. The reactive ion etching is normally carried out using RF (Radio frequency) plasma. In the present work, we have utilized LF (Low frequency) for the RIE (Reactive ion etching) processes. High etch rate of Si of 0.344µm/min has been achieved with SF₆/O₂ plasma and etch rates of SiO₂ are 0.0316µm/min achieved using CF₄/O₂ at 40 KHz. The results show that O₂ concentration and pressure have strong effect on etch rates. Also the variation in etch rate is influenced by variation in power. The etching processes developed in this work are aimed for surface micro-machining of silicon for the fabrication of MEMS (micro electro mechanical system) devices.

Keywords: reactive ion etching, Low frequency.

1. Introduction

Si is a promising material for development of high temperature solid-state electronics and having properties like larger band gap, smaller leakage current, strong, brittle and immune to noise. Silicon has also been used to fabricate MEMS micro devices due to its excellent mechanical properties. In order to create micro structures in Si, we need to develop Si etching processes. Silicon dioxide is a successful material because of its stable oxide. Various forms of Silicon oxide are used in micromachining due to excellent electrical and thermal insulating properties. Reactive ion etching is a combination of physical and chemical processes that leads to formation of micro-structures like micro-needles with surface micro-machining. Isotropic profile is most common in dry etching when halogen etchants are used. Si is etched by halogens at room temperature and at milli-torr pressure because of strong halogen bond (550KJ/mol). Fluorine plasmas are milder and resist selectivity is better. Fluorine based gases are the most popular reactive gases for the Etching of Si. The free radical F⁺ spontaneously react with Si to produce a volatile product. But, SF₆ based processes use 10-40% of added O₂. Oxygen reacts with SF₆ and keeps fluorine concentration high by preventing fluorine recombination with the fragments. SF₆ etches a few microns of Si but isotropic under-cutting is sometimes desirable and even necessary in order to fabricate free standing beams.

Silicon etching can be done by fluorine based plasma and chlorine based plasma. In fluorine based plasma CF₄, SF₆, C₂F₆, C₃F₈ etc. any gas can be used. All the gases contain fluorine free radicals that are the most important species for silicon etching. The reaction between silicon and F⁺ is spontaneous. The profile that is achieved is very selective (Si: SiO₂=40:1) but isotropic.

\[
\begin{align*}
\text{e}^- + \text{CF}_4 &= \text{CF}_3^+ + \text{F}^+ + \text{e}^- + \text{F}^+ + \text{Si} = \text{SiF}_4 \\
\text{O} + \text{CF}_3^+ &= \text{COF}_2 + \text{F}^+
\end{align*}
\]

The extra electron that is obtained maintain the charge neutrality and oxygen is added to increase the etch rate. In chlorine based plasma, mixture of C₂F₆ and Cl₂ is used. Where C₂F₆ serves two functions, one to etch Si and SiO₂ and other is to create a passivation layer in order to make the profile anisotropic. For undoped Si, the reaction in chlorine based plasma is not spontaneous but for doped Si, the reactions are spontaneous. For the etching of SiO₂, hydrogen is used with CF₄ instead of oxygen. HF etches silicon dioxide. Also hydrogen suppresses the formation of fluorine free radical and very good selectivity is achieved (SiO₂: Si=35:1).

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\text{CF}_4 + \text{H}_2 = \text{CF}_3^+ + \text{HF}
\]

The results of RIE of single crystal Si and Silicon Dioxide are obtained using tetra-30. It is a parallel plate reactor having software PRS (plasma reaktor steuerung) for the control of plasma machine. In this, a recipe is created having parameters like pumping down time, gas supply, reactor consists of two parallel plates’ electrodes, where the wafers are kept on powered electrodes. There are two power supplies, one is LF and other is magnetron. There is vacuum system connected at one side in order to create vacuum and

2. Experimental SETUP

TETRA-30 is a photo resist stripper that is used to remove the photo resist from the substrate surface. The photo resist can be removed by burning it in oxygen plasma. But it can also be used for the purpose of reactive ion etching. The reactor consists of two parallel plates’ electrodes, where the wafers are kept on powered electrodes. There are two power supplies, one is LF and other is magnetron. There is vacuum system connected at one side in order to create vacuum and...
evacuation system for the removal of by products.

![Diagram of TETRA-30](image)

**Figure 1:** Schematic of TETRA-30

TETRA-30 operates in two different modes. In automatic mode, a program consists of different processes is created through process parameters. A program can be built by sub-programs and consists of up to 10 sub-programs. The program parameters can be saved. A sub-program consists of:

- Pumping down
- Gas supply
- Plasma process
- Flushing period
- Venting period

Pumping down is used to create vacuum in the chamber and the maximum pumping down time is set. If this time exceeds, an error message is generated, entry in alarm list appears and display field of the switch turns red. As soon as pumping down starts, display switch turns green. Gas supply input is made in scmm. Different MFC (mass flow controller) are connected to the system. Plasma process duration defines the duration for which plasma is created in the chamber. During this time dissociation, ionization, excitation all the processes take place. Plasma process time in hour, minutes, seconds is set. It also depends upon the material to be etched. For some material plasma process duration is kept in hours but for some it is kept even in minutes. The set power and maximum absolute deviation is entered. If the value exceeds, entry in alarm list appears. The minimum and maximum temperature must be entered. The process starts if the minimum temperature is reached. Flushing period is entered and during this time all the gases are flush out. Flushing time is set in minutes. Venting is done in order to set the chamber pressure equals to atmospheric pressure so that the door of chamber can easily be opened. Venting period should be chosen such that atmospheric pressure is reached in vessel and display field of switch turns yellow.

**2.1 Development of RIE processes for Si**

<100> oriented, n type Si wafers were used as substrate and piranha cleaning was done. Positive photo-resist S-1818 was deposited using spin on method at 2000 rpm, soft baking at 90°C. Photo resist of 2.8 µm thickness was deposited on wafer. Then the pattern was defined by UV exposure. The pattern was recovered using photo resist developer CD-26. After development, the wafer was hard baked in oven at 120°C. Then wafer was placed in tetra-30. Reactive ion etching at LF was done using suitable recipe. The gases used for this purpose were SF₆ and O₂. Dry etching has been performed in TETRA-30. The etch rates were measured using Surface Profiler and Zeta. The etch rates were found to be dependent upon the power and pressure. Etch rates at two different pressures, 0.15mbar, 0.1mbar were found 0.344µ/min and 0.304µ/min respectively.

**2.2 Development of RIE processes for SiO₂**

<111> oriented p-type wafers were used as substrate and piranha cleaning was done. PECVD oxide of 0.8micron was deposited. Positive photo-resist S-1818 of 2.8 micron thickness was deposited using spin on method at 2000 rpm rate. Soft baking at 90°C and a pattern was defined by UV exposure. Then pattern was recovered using photo resist developer CD-26. After development, the wafer was hard baked in oven at 120°C. Then wafer was placed in tetra-30. Reactive ion etching at LF was done using suitable recipe. CF₄/O₂ gas mixture was used for this purpose. Different etch rates of 0.3.16µm/min at 0.1mbar and 0.23µm/min at 0.3 mbar were obtained. The etch rates were found dependent on pressure in side the chamber and power.

**3. Results and Discussion**

Different recipes were developed for different wafers. Nine wafers of silicon and nine wafers of silicon dioxide were
taken. On three wafers, same recipe was run. Then the recipe was changed. Then the modified recipe was run on other three wafers. In this way the different etch rates were obtained by changing the power and pressure. For first three samples, when the pressure is 0.5mbar then at different power i.e. 400W, 600W, 800W, different etch rates were obtained. Similarly same process was carried out for 0.1mbar and 0.15mbar pressure. But for silicon dioxide, for first three samples when the pressure is 0.1mbar and next three samples when pressure is 0.15mbar, the power is kept 400W, 500W, 600W but for last three samples when pressure is 0.3mbar power values are kept 600W, 700W and 800W. By varying the power, different etch rates were obtained. Etch rates also depends upon the duration of plasma process.

For these experiments, etch rates were evaluated as the function of pressure of the chamber and power. As power increases, etch rate increases but as pressure increases, etch rates decreases. For Si, when the pressure increases, the etch rate increases and achieves the maximum value of 0.344µm/min and after a certain pressure value i.e. 0.1mbar, etch rate decreases. For SiO₂, etch rates increases initially and achieves the maximum value of 0.0316µm/min but after 0.3mbar pressure etch rates decreases. When power increases beyond 70%, the etch rate decreases because the pressure is goes on increasing simultaneously. For 40% of power no etch rates were observed independent of pressure values.

4. Conclusion

For better etch rates, the pressure for Si should be kept 0.1 mbar and also power should be greater than 40%. For SiO₂, the pressure should be kept 0.3mbar and then the power should be increased. The increase in power leads to increases in ionization that leads to more number of free radicals and more etching takes place. But increase in pressure, decreases the mean free path of radicals. So, the ions collide with each other rather than collision with the
surface. Ions crowding take place just above the surface that decreases the etch rate.

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