Effect of SF$_6$ flow rate on the etched surface profile and bottom grass formation in deep reactive ion etching process

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Abstract. While deep reactive ion etching (DRIE) has proven to be a boon for silicon micromachining applications, certain factors still exist which affect the satisfactory performance of DRIE. Some of the process limitations include bottom grass formation, RIE lag, loading and notching effects and aspect ratio dependent etching. This paper presents the effect of SF$_6$ flow rate and etching/passivation cycle time on the etched shape profile and bottom grass formation. Rectangular trenches of varying widths are etched using Alcatel etching system. Critical DRIE process parameters, such as SF$_6$ flow rate and ratio of etching and passivation cycle time, are varied to explore the dependence of etched shape profile on these parameters. It is found that low SF$_6$ flow rate, i.e. 250 sccm, results in relatively smooth bottom surface. As SF$_6$ flow rate is increased, bottom surface roughness increases and grass forms on the bottom of etched trenches. Shape of etched surface profile was found to be changed from positive profile to negative profile, when the SF$_6$ flow rate was increased. Ratio of etching/passivation cycle was also found to be critical for prevention of bottom grass formation. DRIE process parameters were optimised to get smooth and vertical sidewalls.

1. Introduction

In last few years, time domain multiplexed deep reactive ion etching (DRIE-Bosch process) has gained popularity in high aspect ratio silicon etching for micro electromechanical systems (MEMS) applications [1]. Fluorine based inductively coupled plasma (ICP) using the Bosch process has been shown to achieve etch rates of 3–7 μm/min at room temperature [2-4]. DRIE is superior in practically every aspect to wet chemical etching of silicon, except in the areas of equipment cost and the ability to preferentially follow crystallographically defined etch directions. The principle advantages include higher etching rates, compatibility with photoresist masks, and the ability to produce vertical sidewalls on silicon substrates of any crystal orientation. The above advantages have led to DRIE being the most preferred method for producing high aspect ratio structures for various MEMS applications.

Deep silicon etch process developed and patented by Robert Bosch relies on many repetitions of alternating etch and passivation cycles. The principle of DRIE process is shown in Fig. 1. The first cycle is ion-assisted etching of the silicon substrate by an etching gas (SF$_6$) and the second is a sidewall passivation step using a polymer-producing gas (C$_4$F$_8$). At first, passivation cycle coats the side walls with a protective polymer, i.e. poly-tetra-fluoro-ethylene (PTFE) that prevents lateral etching. In successive etching step, a mixture of oxygen and SF$_6$ gas is passed. Oxygen ions etch the bottom polymer while fluoride ions etch silicon at the bottom of through-hole. Sidewalls of through-
holes remain protected by polymer. The continuous repetition of such etching and passivation cycles results in high etching rate and highly anisotropic through-hole etching.

![Diagram](image)

**Figure 1.** Principle of deep reactive ion etching (DRIE- Bosch) process

Despite the overwhelming success of this etching technology, certain factors exist which complicate its implementation. Such factors include dependence of etch rate on feature size [4-6], aspect ratio dependent etching [6], lateral etching at the insulating etch stop layer (notching effect) [7,10], dependence of etching rate on etching area (loading effect) and micromasking effect [8-10]. The aspect ratio dependent etching (ARDE) is a serious limitation in deep silicon etching: at high aspect ratios, excessive etch-rate reduction occurs with longer etch times. These factors can result in local etch rate variation (e.g., large feature has a greater depth than adjacent small feature), systematic variation of etch rate across the wafer, and the formation of dense accumulations of vertical filaments of silicon, known as ‘grass’ [8, 10].

As the factors causing above problems are strongly affected by the geometry of the pattern and process parameters, a detailed study of DRIE parameters is vital. There are several parameters which affect the etched shape and their profile; Figueroa et al [7] have shown the importance of oxygen in getting desired shape profile. However in this paper, we have presented the effect of etching gas (SF₆) flow rate in getting desired side wall slope profile. The two problems taken into account in the experiments are the control of etched shape profile and the grass formation at the bottom of etched profile. Experiments are performed to show the effect of flow rate of SF₆ on the formation of grass and on the etching profile.

### 2. Experimental details

All experiments were performed on 4” diameter, p type, <100> orientation, 0.1-0.01 Ω-cm, 500 μm thick silicon wafers. 10 μm thick AZ9260 photoresist was used as the mask layer. Rectangular slots of varying width from 40 μm to 80 μm were designed in the plastic mask. Wafers were patterned using Karl-Suss mask aligner. Alcatel A601E ICP etch system was used for silicon etching. Different DRIE experiments were performed by varying the flow rate of SF₆, etching and passivation cycle time. Brief details of the experiments are given in table 1. A total of 13 experiments were performed in which SF₆ parameters were varied.

In first 5 experiments, SF₆ flow rate was varied from 250 sccm (exp no 1) to 400 sccm (exp no 5), to find out the effect of SF₆ flow rate on grass formation and etched surface profile. Ratio of etching and passivation cycle was also changed to explore its effect on the etching characteristics. Each sample was etched for 90 min. Once etching was completed, samples were washed in acetone to remove the photoresist. Samples were cut in center by diamond cutter, and scanning electron microscope (SEM) was used to check the cross sectional profile.

### 3. Experimental results & discussion

From the SEM characterization of each DRIE etched sample, geometrical dimensions have been found out. Details of these etched dimensions; profile angle and etching rate are given in table 2. Profile angle represents the slope of etched side walls with horizontal surface and is measured by the ratio of difference in top (d₁) and bottom diameter (d₂) of trenches and depth of trench (h) i.e. profile angle \( \theta = (d₁-d₂)/h \). Profile is termed as negative (opening) if profile angle \( \theta \) is less than 90°. If profile angle \( \theta \) is more than 90°, profile is termed as positive (closing) profile.
Table 1. Details of DRIE process parameters used in the experiments.

| Exp. No. | SF₆ flow rate (sccm) | C₂F₈ flow rate (sccm) | Etch/Pass time (s) | Coil/Platen power (W) | Temp (°C) | Etch time (min) |
|----------|----------------------|-----------------------|-------------------|-----------------------|-----------|-----------------|
| 1        | 250                  | 200                   | 3.0/1.0           | 1800/100              | 10        | 90              |
| 2        | 300                  | 200                   | 3.0/1.0           | 1800/100              | 10        | 90              |
| 3        | 325                  | 200                   | 3.0/1.0           | 1800/100              | 10        | 90              |
| 4        | 350                  | 200                   | 3.0/1.0           | 1800/100              | 10        | 90              |
| 5        | 400                  | 200                   | 3.0/1.0           | 1800/100              | 10        | 90              |
| 6        | 250                  | 200                   | 5.0/2.0           | 1800/100              | 10        | 90              |
| 7        | 300                  | 200                   | 5.0/2.0           | 1800/100              | 10        | 90              |
| 8        | 350                  | 200                   | 5.0/2.0           | 1800/100              | 10        | 90              |
| 9        | 400                  | 200                   | 5.0/2.0           | 1800/100              | 10        | 90              |
| 10       | 250                  | 200                   | 4.0/2.0           | 1800/100              | 10        | 90              |
| 11       | 300                  | 200                   | 4.0/2.0           | 1800/100              | 10        | 90              |
| 12       | 350                  | 200                   | 4.0/2.0           | 1800/100              | 10        | 90              |
| 13       | 400                  | 200                   | 4.0/2.0           | 1800/100              | 10        | 90              |

Table 2. Geometrical dimensions and profile angle of trenches etched at different SF₆ flow rate

| Exp. No. | SF₆ flow rate (sccm) | Designed diameter (μm) | Top diameter d₁ (μm) | Bottom diameter d₂ (μm) | Etching depth h (μm) | Profile angle θ (°) | Etch rate (μm/min) |
|----------|----------------------|------------------------|----------------------|------------------------|---------------------|---------------------|--------------------|
| 1        | 250                  | 40                     | 63.9                 | 42.3                   | 142.3               | 85.66               | 1.581              |
|          |                      | 50                     | 84.5                 | 50.2                   | 151.6               | 83.55               | 1.684              |
|          |                      | 60                     | 85.9                 | 65.4                   | 161.7               | 86.37               | 1.797              |
| 2        | 300                  | 40                     | 47.6                 | 57.1                   | 146.5               | 91.86               | 1.628              |
|          |                      | 50                     | 50.0                 | 58.0                   | 155.3               | 91.48               | 1.726              |
|          |                      | 60                     | 60.0                 | 67.5                   | 169.8               | 91.37               | 1.887              |
| 3        | 350                  | 40                     | 42.9                 | 50.0                   | 153.9               | 91.32               | 1.710              |
|          |                      | 50                     | 50.0                 | 55.4                   | 182.5               | 90.85               | 2.028              |
|          |                      | 60                     | 60.8                 | 63.5                   | 185.4               | 90.42               | 2.060              |
| 4        | 400                  | 40                     | 45.3                 | 59.5                   | 156.8               | 92.59               | 1.742              |
|          |                      | 50                     | 53.6                 | 67.2                   | 186.1               | 92.10               | 2.068              |
|          |                      | 60                     | 63.2                 | 73.0                   | 196.9               | 91.43               | 2.188              |

3.1.1. Effect of SF₆ flow rate on the side wall profile (opening/closing profile)

Effect of SF₆ flow rate on the etched shape profile of the trenches is clearly shown in Fig. 2. Fig. 2a shows negative (opening) profile (profile angle ~92°) of a trench that is etched at SF₆ flow rate of 400 sccm. At a flow rate of 350 sccm, almost vertical profile is obtained. Shape profile is changed from positive to negative, when the flow rate is reduced to 250 sccm.

The negative taper of the side walls is caused by a lateral etching rate, which increases with the depth of the trench. This negative taper is caused by a decreasing passivation quality of the layer with the etching depth, by an increased sidewall impact by fluoride ions. This impact can either be the result of ion deflection due to mirror charges on the sidewall or ion back-sputtering from the trench bottom. When the SF₆ flow is increased (~400 sccm) with all the other parameters kept constant, the passivation quality of the layer diminishes. Fluoride ions have isotropic nature of etching, i.e. etching rate remains the same in all directions. More number of fluoride ions starts etching the side walls and
thus lateral etching start. Due to sideway etching, bottom diameter of trench increases and negative profile is formed.

**Figure 2.** Shape profile of trenches etched with variable SF$_6$ flow rate (a). Negative (opening) profile at 400 sccm (profile angle ~92°) (b). Straight profile at 350 sccm (c). Positive (closing) profile at 250 sccm (profile angle ~86°)

Etching of the bottom of the trench depends on the influx of neutrals, ions, electrons, and inhibitor precursors, as well as the removal of the reaction products. In normal cases, there is equilibrium between transport of etchant towards and the transport of products away from surface; however at large depth, this equilibrium does not remain balanced and transport mechanism of reactants and waste products get complicated. Reactant species (fluoride ions) try to go down to etch the bottom surface, while the gaseous products (SiF$_x$O$_y$) try to come out of the trench. In this transport process, some of the fluoride ions lose their etching capability.

When the SF$_6$ flow rate is reduced gradually, overall etching capability of fluoride ions reduces. As a result of reduced etching capability of fluoride ions, etching rate reduces and bottom opening starts closing, thus results in positive profile (Fig. 2c). An optimum SF$_6$ flow rate is needed to get perfectly straight profile. In this case, we have found that straight etched shape profile is achieved at a flow rate of about 350 sccm.

### 3.1.2. Effect of SF$_6$ flow rate on grass formation

In the experiments, samples are etched at different SF$_6$ flow rate i.e. 250, 300, 350 and 400 sccm. Other DRIE parameters remain the same and are given in table 1. Cross section of trenches are characterised by SEM and their cross sectional images of etched trenches are given in Fig. 3.

It is apparent from Fig. 3 that amount of bottom silicon grass increases when the SF$_6$ flow rate is gradually increased. No bottom grass is formed when the trench is etched at SF$_6$ flow rate of 250 sccm (Fig. 3d). As soon as flow rate is increased to 300 sccm, needle like silicon grass formation starts (Fig. 3c). At a flow rate of 400 sccm, bottom grass formation is very stern among all samples (Fig. 3a). Density and height of bottom silicon grass increases with SF$_6$ flow rate (Fig. 3c) and found to be the maximum at 400 sccm. Density of silicon grass is also more than any other samples. Close view of the silicon grass is shown in Fig. 4. Typical height of silicon grass is about 10-15 μm (Fig. 4b).

The exact reason behind grass formation is not clear yet, however it is thought that formation of bottom grass is due to the localised concentration of incoming fluoride ions. It is observed that in case of high SF$_6$ flow rate, fluoride ion angular distribution is localised at some bottom points. In the beginning of etching process when the etching depth is low, fluoride ions dispersion is high enough to prevent silicon grass, but during the ongoing etching process, ion angular distribution flux sharpens, enabling grass to form at the bottom. Thus grass is formed when the flux of incoming ions is highly collimated. In the case of low SF$_6$ flow rate, fluoride ions do not localised and hence, bottom grass does not form.
Figure 3. Effect of SF$_6$ flow rate on bottom grass formation (a). 250 sccm (b). 300 sccm (c). 350 sccm and (d). 400 sccm. Total etching time in all cases is 90 min.

Figure 4. Close view of bottom grass formation in the trench etched at SF6 flow rate of 400 sccm.

3.1.3. Effect of etching/passivation cycle time on grass formation (3/1, 4/2 and 5/2)

Bottom grass formation is also found to be affected by the ratio of etching and passivation cycle time. Experiments are performed at three different cycle timings i.e. at etching/passivation cycle time 3.0/1.0, 5.0/2.0 and 4.0/2.0 seconds respectively. Cross sectional images of trenches etched by these recipes are shown in Fig. 5. SF$_6$ flow rate is 300 sccm, while C$_4$F$_8$ gas rate is 200 sccm. Coil and platen power is 1800 W and 100 W respectively.

It is clear from these images that an optimum ratio of etching and passivation cycle time produces smooth grass free bottom. In the case of small etching cycle time, i.e. 4 seconds, problem of bottom grass formation is severe. This effect is termed as ‘micromasking effect’. Due to small etching period, bottom polymer layer is not completely etched away and the remaining polymer layer acts as a secondary mask and protects bottom silicon from etching. This results in long needle kind of silicon ‘grass’ structures (Fig. 5a). When the etching cycle time is increased to 5 seconds, bottom grass formation is completely eliminated (Fig. 5b), and smooth bottom surface is obtained. However, if the ratio of etching and passivation is increased to 3, bottom grass formation starts once again. Hence, optimum cycle time parameters are critical for getting satisfactory DRIE results. In these experiments, a cycle having etching and passivation time of 5 and 2 seconds respectively is found to be optimum.
3.1.4. Effect of SF$_6$ and etching/passivation cycle time on etch rate

It is clear from the Table 2 that silicon etching rate strongly depends upon the SF$_6$ flow rate. At flow rate of 400 sccm, etch rate is about 2.2 μm/min which is reduced to 1.8 μm/min. Increment in etching rate is due to availability of fluoride ions. Etching rate is also found to be more for wider opening trenches than narrower ones.

4. Summary

In this paper, we have shown the effect of two of the critical DRIE process parameters, i.e. SF$_6$ gas flow rate and ratio of etching to passivation cycle time. Shape profile of trenches is changed from negative to positive profile at two extreme SF$_6$ flow conditions. Effect of SF$_6$ on bottom grass formation is also shown here. At a flow rate of 250 sccm, no bottom grass is found. When SF$_6$ flow rate is increased gradually, formation of bottom grass start. Experimental results are separated in two parts: grass formation on the bottom of etched trenches and the variation of etched profile (negative to positive profile). Till now, we have considered only the effect of SF$_6$ flow rate and etching cycle time. Experiments are going on to consider additional parameters, such as chamber pressure, oxygen flow rate and C$_4$F$_8$ flow rate, and to study the combined effects of all considered parameters on bottom grass formation and etched shape profile evaluation.

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