Optimization of deep silicon etching process for microstructures fabrication

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Abstract. The paper presents the study of cyclic process of deep anisotropic silicon etching, called Oxi-Etch, in which the steps of etching and oxidation alternate, allowing deep etching of silicon with an anisotropic profile. This process forms typical for cyclic etching process sidewall profile called scalloping. Opportunities for modification and optimization of the process for specific application were investigated. The effects of optimization of the bias voltage and the duration of the etching step on the parameters of the resulting structures, such as the etching depth, wall roughness, and the accuracy of transferring the lithographic size, are considered. Balance between etch rate and scalloping was established.

1. Introduction

Plenty of microstructures relevant for various applications are fabricated by deep silicon etching. Some devices of droplet microfluidics [1] can be based on thin trenches made in silicon. Array of holes in silicon can form two-dimensional photonic crystal, which can be applied for terahertz communication devices [2]. Another application is microstructures for 3D integrated capacitors using in dynamic random-access memory (DRAM) [3]. In order to increase capacitance and consequently density of the stored information, the area of the capacitor plates is increased, so usually integral capacitors are formed by conformal deposition of films on sidewalls of high aspect ratio structures in silicon. Manufacturing of all these high aspect ratio structures requires technology of deep anisotropic plasma etching of silicon. Different approaches of anisotropic silicon etching have their own features. One of these technologies is well-known Bosch process, in which anisotropy is achieved by protecting sidewalls of structure with fluorocarbon film deposited form plasma. The idea of this process consists in the cyclic repetition of the steps of passivation of the surface with a polymer film in a fluorocarbon plasma and anisotropic etching with a DC bias in a fluorine-containing plasma. Structures etched in this process have typical sidewall profile with periodical notches called “scallops”, which could be smoothed by further optimization of the process, i.e. shortening of both steps duration.

Here an alternative room temperature approach of deep anisotropic silicon etching known as Oxidation-Etching (Oxi-Etch approach) was studied. Earlier, it was proposed [4] and patented [5]. The idea is based on the replacement of polymer passivation by plasma oxidation of silicon. Silicon oxide 1–2 nm thick on the sidewall has sufficient plasma etch resistance for one etching cycle. The process studied in this work is a cyclical repetition of the following basic steps: 1) passivation in the O$_2$ plasma - the formation of a SiO$_2$ layer several nanometres thick on all open areas of silicon (oxidation step), 2)
etching in SF₆ plasma - removal of passivation from the bottom with accelerated ions, and spontaneous isotropic etching (etching step). The method we are investigating has several advantages over the Bosch process. In the etching process, no hard-to-remove polymer film remains on the side walls, which facilitates subsequent technological operations. Furthermore, fluorocarbon gases used in Bosch process for passivation have significant global warming potential, which means that increased content of these gases in atmosphere leads to an increase in the greenhouse effect. Passivation by O₂ plasma is more eco-friendly.

In addition to the above, there are ample opportunities for process optimization. For example, in addition to the basic steps of etching and passivation, intermediate steps can be introduced in which the gas composition in the reactor is changed; in these steps, the value of the input power and bias voltage can also be changed. This is especially true in reactors in which continuous wave plasma is used for reasons of reproducibility. In this case, the consideration of processes in a mixture of gases between steps is also important.

At present work approaches of modification and optimization of the oxidation-etching process are examined. In studied process DC bias is applied for a short time only for breakthrough passivation and then etching of exposed silicon regions is isotropic without bias. As bias-stimulated etching break the passivation layer predominantly on horizontal areas, sidewalls are preserved from isotropic etching. Minimization of duration of DC bias should increase selectivity in process.

2. Experimental

The experiment was carried out in the PlasmaLab 100 plasma etching tool (Oxford Instruments Plasma Technology, UK) on silicon samples 2x2 cm with a SiO₂ mask 2 µm thick. A feature of this reactor equipped with a Faraday shield is that the ignition of an ICP discharge is possible only when a bias voltage is applied to the sample. For this reason, the charge is ignited once at the very beginning of the process and more ICP power is not interrupted.

Mask has round holes 2, 4, 8, and 12 µm in diameter and 16 µm width trenches formed by photolithography and plasma etching. In this work basic Oxi-Etch process, described in details earlier [4], was modified in following way. Typical step duration time, gas flows and applied powers diagram is shown on figure 1.

![Figure 1. Diagram of variation of parameters in process.](image-url)
Oxidation step was conducted in O\textsubscript{2} plasma at high ICP power (2000 W) without DC bias and step time was varied 4-8 s. Then was short (2 s) transition step at which oxygen in chamber was replaced by SF\textsubscript{6}/Ar mixture. And right after that during 2 s breakthrough step DC bias was applied in plasma mixture and passivation on the bottom of the structure was broken. Then it was 3-6 s etching step, when in SF\textsubscript{6} plasma 1750 W ICP power structures were etched isotropically without DC bias. Finally, it was 2 s transition step from SF\textsubscript{6} to O\textsubscript{2} plasma with low ICP power 1200 W. During the process the sample was glued to the 100 mm silicon wafer fixed in chuck cooled to room temperature. As a result, we obtained etched structures and used SEM (Carl Zeiss Supra 55) to characterize features such as undercut and mentioned above scalloping.

It can be seen that the switching on of the bias power in the process is performed very scarcely. This is due to the fact that ion-stimulated etching is required only to remove passivation from the bottom of the trench. During the rest of the time, the excessive flow of accelerated ions will not stimulate the silicon etching process, but will only lead to additional erosion of the mask. Moreover, if oxygen ions undergo acceleration due to DC bias, this will lead to their deep penetration into the bottom of the silicon etching structure and the creation of a deeply oxidized layer, which can even cause an etching-stop effect.

Duration of both oxidation and etching steps were varied in order to research ability to tune etch rate and scalloping of the process. Effect of DC bias during breakthrough step was studied. SEM pictures of microstructures were obtained after 50 cycles process. Using modified process high aspect ratio structures with vertical sidewall profile were achieved in 530 cycles of etching process.

3. Results and discussion
At first oxidation and etching without bias steps durations were varied. Values of etch rate, undercut and scalloping in 50 cycles process on 16 μm trench are shown in table 1. Etch rate is increasing with decrease of oxidation step duration and with the increase of etching step duration due to increasing the proportion of etching time in the cycle. Undercut grows with decreasing of oxidation time and increasing of etching time due to lack of passivation of the top of structure. Scalloping is decreasing with the decrease of oxidation duration and increase of etching duration probably because of improving the break-up of oxide at the edges of the bottom of the structure. As a result, optimized process with acceptable etch rate and fine undercut and scalloping is process with decreased duration both oxidation and etching steps.

| Columns: t\textsubscript{oxid} s | 4       | 6       | 8       |
|--------------------------------|---------|---------|---------|
| Lines: t\textsubscript{etch} s | Rate=1.14 μm/min | Rate=1.04 μm/min | Rate=1.06 μm/min |
|                                | EPC=0.25 μm/cycle | EPC=0.26 μm/cycle | EPC=0.35 μm/cycle |
| 3 Undercut=158 nm              | Undercut=100 nm | Scalloping=88 nm |
| 6 Scalloping=64 nm             | Rate=1.31 μm/min | Rate=1.27 μm/min | Rate=1.06 μm/min |
|                                | EPC=0.35 μm/cycle | EPC=0.38 μm/cycle | EPC=0.35 μm/cycle |
|                                | Undercut=400 nm  | Undercut=374 nm | Undercut=247 nm |
|                                | Scalloping=59 nm  | Scalloping=67 nm | Scalloping=78 nm |

DC bias during passivation breakthrough step was varied. On figure 2 results of etching of 2 μm holes in 50 cycles process for different bias voltage in the range of 38-83 V are shown. As one can see at low DC bias with increase of aspect ratio leads to incomplete passivation breakthrough and consequently nanograss formation at the bottom of the structure. Increasing of DC bias leads to more...
complete breakthrough in the depth of the structure, but structures still have positive sidewalls angle. At high DC bias in first 50 cycles complete breakthrough is achieved which gives vertical sidewalls profile. On the other hand, with growth of DC bias mask etch rate is also increasing. In processes with 38 V, 65 V and 83 V DC bias mask etch rate is growing from ~ 0 nm/min, 4.6 nm/min and 7.4 nm/min respectively. This is consistent with the known concepts that the silicon oxide of which the mask is made is etched in a fluorine-containing plasma in a threshold process after 40 eV, with a bias exceeding 60-80 volts, the etching rate does not change so significantly. It is noteworthy that the effect of the formation of roughness at the bottom does not appear immediately, but after reaching a certain etching depth and aspect ratio of the hole. This means that it is possible to investigate the possibility of changing the bias voltage depending on the cycle number with increasing etching depth.

**Figure 2.** SEM pictures of 2 μm holes after 50 cycles processes with a) 38 V, b) 65 V and c) 83 V DC bias during breakthrough step.

Using modified Oxi-Etch approach deep silicon etching process was carried out to achieve high aspect ratio structures. In order to obtain vertical profile and achieve deep structures with 2 μm mask 530 cycles process with 4 s oxidation time and 3 s etching time was chosen. DC bias during breakthrough step was increased every few cycles from 65 V at start to 130 V in the end. SEM pictures of 16 μm trench and 8 μm hole are shown at figure 3.

**Figure 3.** SEM pictures of a) 16 μm trench and b) 8 μm holes after 530 cycles of optimized Oxi-Etch process.

Both structures have vertical sidewalls. For example, trench structure on the figure 3a width variation less than 1% over the entire etching depth. Aspect ratio for trench and hole are 5.6 and 7 respectively. Effect of aspect ratio dependence etching was observed, for example holes with
diameters of 4 μm, and 8 μm have depths 45.4 μm, and 56.9 μm respectively. This typical for deep silicon etching effect is that with increasing of aspect ratio transport of active particles to the etching region becomes more complicated. As a result, narrower structures have lower etch rate.

4. Conclusions
In present work cyclic Oxidation-Etching deep silicon etching process was modified, studied and optimized. This approach has some advantages over well-known deep plasma etching processes. Influence of steps duration and DC bias during breakthrough step were studied. It is shown that there is a trade-off between the etching rate and undercutting depending on the ratio of the etching time and the oxidation time in the cycle. It is shown that with a decrease in the bias voltage, a significant increase in the selectivity of the Oxi-Etch process is possible. At the same time, when etching high-aspect-ratio structures, a small bias voltage may be insufficient to break through the passivation layer at the bottom; this can lead to the formation of an inclined wall and even, with incomplete breakthrough of the passivation layer, to the formation of nanogras at the bottom. It is necessary to develop processes with variable characteristics depending on the cycle number to obtain maximum selectivity and vertical profile.

Using the process high aspect ratio structures with acceptable sidewalls profile were achieved. For example, for a process with a duration of 530 cycles, trench coats with strictly vertical walls were obtained. Etch selectivity, i.e. the ratio of the etching depth to the mask consumption was 67 for trench coats with a width of 16 μm, and 42 for holes with a diameter of 8 μm. Researched method can be used for manufacturing structures for different applications.

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