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The Black Silicon Method VIII:
A study of the performance of etching silicon using SF_6/O_2 -based chemistry
with cryogenical wafer cooling and a high density ICP source

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ABSTRACT

This paper presents a study of the performance of current trends in high speed, highly controllable anisotropic plasma etching of silicon for its use in micro- and nano-engineering. Optimisation rules for tuning the equipment are formulated to enable maximum results with respect to etch rate, etch selectivity, and profile control (e.g. anisotropy). The optimisation process uses the black silicon method (BSM) to allow for an easy to use design-of-experiment method. After optimisation, etch rates of silicon up to $15\mu\text{m}/\text{min}$ were obtained at relatively high pressure ($4\text{Pa} = 30\text{mTorr}$) and ICP power (2kW) while keeping the anisotropy high. At the same time, the erosion rates of thermal silicon dioxide and ordinary photoresist (Shipley S1805) were around $7\text{nm}/\text{min}$ (i.e. selectivities up to 2000). Increasing the pressure to 20Pa , the selectivity increased to 10000, although bottling became more pronounced. The high etch rate and high selectivity are especially important in case of micro engineering, where wafer through etching with the help of plasmas will become a standard in the near future. In case of nano engineering, however, profile control is the main concern. To prevent undercut in such cases, in particular bottling due to a broad ion angular distribution, the pressure should be sufficiently low. The best results were found at pressures down to $0.2\text{Pa} = 1.5\text{mTorr}$ and at a low ICP power of 350W to prevent a too strong mask erosion caused by the low pressure. The silicon etch rate decreased to $1\mu\text{m}/\text{min}$ and the erosion rate of the oxide and resist were both approximately $20\text{nm}/\text{min}$ giving a selectivity of 50. Reproducibility (wafer-to-wafer) and uniformity were also output factors of prime concern. Surprisingly enough, for two different equipment manufacturers the results were almost identical when using the same parameter setting. This indicates, that the SF_6/O_2 -based chemistry was optimised rather than the equipment itself.

INTRODUCTION

A prerequisite for the successful construction of nano- and micro-engineered structures is a reliable and reproducible technology. The dimensions of such parts can range from submicron up to millimetres and are generally fabricated using wet chemical (crystal anisotropy) or dry plasma (ion-bombardment anisotropy) etching. Especially in micromachining for through-wafer-etching, a high etch rate, while maintaining a good anisotropy, is a must. For this reason high density sources and helium backside wafer cooling are more and more incorporated into single wafer diode etchers.

We have recently demonstrated that trenches (up to $200\mu\text{m}$) with high aspect ratios (up to 10), were etched at high etch rates (up to several $\mu\text{m}/\text{min}$) and low ion energies ($15\text{--}90\text{eV}$) using an SF_6/O_2 -based conventional reactive ion etch (RIE) system and the black silicon method (BSM) [1]. The BSM uses the parameter settings where black silicon appears, as a guideline to optimise the equipment quickly for maximum anisotropy. This is because black silicon only shows up when the profile is near vertical with barely no undercut. The low ion energy prevents substrate damage (electronics), mask erosion, and makes it easy to change the profile of the trench. However, a disadvantage of such systems is that the temperature control of the wafer is usually insufficient. Moreover, the etch rate is directly controlled by the rf power supply which, on its turn, directly controls the bias voltage and, therefore, the ion energy and mask selectivity. For this reason it would be convenient to have an independent control of the bias voltage with respect to the rf power and an appropriate heat sink.

Currently, at least four vendors are selling plasma systems aiming especially at the MEMS/ MNE market which can meet the above mentioned demands. All do have helium backside cooling to stabilise the wafer temperature while etching and all come with double powered plasma sources. One is an inductively coupled plasma source (ICP) to create a high radical and ion density. The other is a capacitively coupled plasma source (CCP) to direct the ions from the plasma glow region towards the wafer surface. This enables us to control independently the energy-flux of radicals and ionic species. Two of them, the Plasmalab 100 system of Oxford Plasma Technology and the METlab system of Alcatel, are using cryogenic wafer cooling for profile control and an SF_6/O_2 -based chemistry for a high etch rate. The high density plasma is created into a quartz dome and the wafers are clamped mechanically. The other two, the SLR system of Plasmatherm and the ASE system of STS, have incorporated the Bosch patent in their system to control the anisotropy. In these systems the wafers are clamped electrostatically.

In this paper, the advantages of double powered high density sources and cryogenic helium backside wafer cooling will be treated. Important optimisation criteria are to maximise the etch rate, the etch selectivity, anisotropy, uniformity, and reproducibility.

Figure 1b: The influence of the temperature on the profile (Plasmalab 100). Initial etch rate ca. $4.2\mu\text{m}/\text{min}$.
 $P_{\text{ICP}} = 600\text{W}$; $P_{\text{CCP}} = 2\text{W}$ ($\Rightarrow$ self-bias = 15V); $Q_{\text{SF}_6} = 120\text{SCCM}$ ($\Rightarrow p_{\text{work}} = 10\text{mTorr}$); $p_{\text{He}} = 20\text{mbar}$.

In contradiction, the smaller holes exceed $\text{AR}=1$ after 4 minutes of etching. The consequence of such a high AR is shown in figure 1b for the Plasmalab 100 where the influence of the temperature on the profile is shown with the help of SEM. The parameter setting is given in the figure also. The average silicon etch rate was ca. $3\mu\text{m}/\text{min}$ and that of the resist mask ca. $4\text{nm}/\text{min}$ giving an average selectivity of ca. 800. However, the initial etch rate was ca. $4.2\mu\text{m}/\text{min}$ giving an initial etch selectivity of 1000. Clearly, the slope of the trench changes from negative at 160K to positive at 180K . At even higher temperatures, the profile becomes isotropic. It should be noted, however, that the profile is not under all circumstances becoming more negative while decreasing the temperature. For example, for a setting directly at the right side of the black silicon line in figure 0, marked with "A", the profile becomes more positive while decreasing the temperature. As it should, of course, because the line represents the settings for profiles going straight down. It is worthwhile noting that the METlab shows an identical result for almost the same setting.

Anyhow, an undercutting of the mask, be it negative or isotropic, will deplete reactive species for etching into the deep, resulting in a lower etch rate. Especially in case of the smaller holes where the amount of sidewall area is relatively high with respect to the size of the opening, i.e. in case of a high AR. Returning to figure 1a, the dependency of the temperature on the silicon etch rate is shown for the smallest hole. Starting from the lowest temperature, at 100K , the etch rate first quickly increases because the SF_6 is almost not condensing at the wafer surface anymore which otherwise blocks the etching. At 140K it reaches its maximum because the profile turns from negatively tapered into straight down. Then, at 160K , the etch rate has a dip due to the forming of black silicon. At 170K , the profile becomes a little isotropic and, therefore, the black silicon disappears and the etch rate increases again. Beyond 170K , the profile becomes more and more isotropic which decreases the etch rate until it saturates when the profile is fully isotropic.

Figure 2: Potentials inside the plasma.

CCP power & bias voltage: The main function of the CCP source is to create a bias voltage to accelerate ions with a predetermined energy to the substrate. The ions will gain an energy in their travel from the plasma glow region, which is at the so-called plasma potential (with respect to the anode, i.e. grounded electrode), towards the cathode where the wafers are situated as depicted in figure 2. However, the bias voltage is developed between the anode and cathode of the capacitor plates from the CCP source. Therefore, we are not able to determine the plasma potential directly and thus the plasma-to-cathode potential. In such cases, so-called Langmuir probes are in use.

The use of a Langmuir probe is an already established technique to measure the plasma-to-floating potential, V_{pf} . This potential is directly related to the electron temperature as can be found in many textbooks treating plasma physics. Although there is some disagreement around the exact formulation, we will take the simplest form because this is enough to make our statement clear:

$$V_f - V_p = \frac{kT_e}{2q} \ln \left(2.3 \frac{m_e}{m_i} \right) \quad [\text{V}] \quad (1)$$

Figure 4b: Plot showing that CCP current is not depending on CCP power

The ion flux is related to the ion density in the glow region, which is proportional to the total power consumed by the plasma: $P_{tot} = P_{CCP} + P_{ICP}$ and, due to an increasing number of gas collisions, inversely proportional to the pressure at constant residence time (Note: When the residence time is chosen constant, then the pressure is proportional to the flow Q). Therefore:

$$ER_{ox} = k \left(V_p - V_{thres} + V_{bias} \right) \frac{(P_{ICP} + P_{CCP})}{Q} \quad (3)$$

The threshold voltage, V_{thres} , has to be incorporated because ions need a certain energy to be able to break chemical bonds. The constant k is a kind of "matching factor". Indeed, with respect to fig.4a, for zero CCP power the erosion rate is still:

$$ER_{ox} = k \left(V_p - V_{thres} \right) \frac{P_{ICP}}{Q} \quad (4)$$

Matching this formula with figure 4a, it reveals that $k \sim 0.15$ (SCCM*nm)/(min*V*W), for both SiO_2 and resist, and $(V_p - V_{thres}) \sim 10V$. The voltage closely matches the theoretical value of 12V when V_{thres} is taken a few volt. However, a remark has to be made: The electron temperature decreases with increasing pressure because the electron mean free path decreases as well. As the pressure is raised, the electrons undergo more collisions and are unable to gain energy. As a consequence, the plasma potential should decrease with increasing pressure, since V_p is a function of the electron temperature. Why this effect is not observed is not yet understood, but probably it is still too small.

To check the just described theory, we plotted at the same time the bias as a function of the CCP power in figure 4b. Now the straight line indicates that the current, i.e. flux of charged particles towards the wafer, is constant for a constant pressure and ICP power. The current did not depend on CCP power, temperature, nor oxygen content. Of course, when the CCP power becomes too large with respect to the ICP power, the current will raise by the CCP power also. Giving the figure a closer look, there is an offset voltage of approximately 10 volts found for zero CCP power. Clearly, this is impossible: The offset is caused by the bias measuring unit. The voltage meter measures up to

1000 volts with an accuracy of 1%, i.e. $\pm 10V$. This is not acceptable in our case and, therefore, we used the reading of the CCP power monitor directly.

So, we have found that the erosion rate is proportional to the CCP current. How is this current depending on the ICP power and SF_6 flow? In figure 4c, the CCP current is plotted as a function of both parameters (Note: The current is given in arbitrary units because only the bias is monitored and not the top-top voltage of the CCP power source: $V_{tt} = P_{CCP}/I_{tt}$. However, V_{tt} is proportional to V_{bias} , which makes this technique reliable). It is found that the figure has a good match when we take the current proportional to the ICP power and inversely proportional to the pressure (or flow). Note that the erosion rate would still be proportional to the CCP current, even when the dependency of the CCP current on the SF_6 flow or ICP power would have been entirely different.

SF_6 flow, pressure & residence time:

In case of the pressure and flow, the difficulty arises that these two input variables are directly related to another important variable; the residence time:

$$\tau_{res} = V \frac{p}{Q} \quad (5)$$

with V the plasma volume in litres, p the work pressure in Torr, and Q the flow in Torr*litre/sec (1Torr/lit/sec= 76SCCM). For both the Plasmalab 100 and the METlab, ($V \sim 15$ l) the minimum residence time of ca. 70msec is achieved when the vacuum conductance is at a maximum, i.e. the throttle valve between the reactor and the turbo is completely opened. Only in this case, $\tau_{res} = \gamma V/S$, with S the (turbo) pumprate and γ a constant depending on the reactor geometry (gas flow restriction), the efficiency of the turbo, but in particular upon the plasma gas temperature and the degree of dissociation. For example, in case of a fully dissociated plasma the population of free moving particles might be doubled ($SF_6 \Rightarrow SF_5 + F$) and the temperature could be twice as high too. Therefore, $\gamma=4$ is a reasonable value giving $\tau_{res}=60msec$.

Note that it is impossible two change one of the variables, p, τ_{res} , or Q, without changing at least one of the other two (at constant volume V, of course).

Figure 4c: Dependency of CCP current on ICP power and pressure (~flow).

Figure 6: Initial selectivity, S , between silicon and oxide. Note that S decreases due to RIE lag and higher loading.

ICP power, oxygen content, & anisotropy: We already found the dependency on the ICP power in the forgoing section. However, there is a special remark to bear in mind: During operation, the dome is creating a steady stream of oxygen into the gas mixture. This flux is increasing with ICP power. This effect is highly unwanted because a) our results are directly influenced by this flux, b) the dome has a finite lifetime, and c) it might consume fluor. To demonstrate this effect, a sample was etched at 500W ICP power and 200SCCM SF_6 flow. This point is marked "A" in figure 5a. The profile turned out to be partly isotropic. The undercut could be prevented by decreasing the SF_6 flow to 100SCCM, increasing the ICP power to 1000W, or by adding 10SCCM oxygen flow. This results indicates that the dome generates a steady flux of oxygen. We can overcome this problem by replacing the quartz dome by an alumina dome which is known to have a very low erosion rate and produces no oxygen, although it might result in particle generation (sputtered aluminum) resulting in micro roughness. Of course, when the dome is replaced, extra oxygen is needed to tune for the correct recipe again.

So, it seems to be that oxygen is, at least partly, responsible for the etch anisotropy. But, it is found that there exists a peculiar turn point in the black silicon line at 130K as found in figure 0. Could there be another inhibiting layer working effectively at lower temperatures? For example, the condensing of SF_6 at the wafer surface could act as a passivating layer [6,7]. Alternatively, the condensed SF_6 gas is dissociated under ion bombardment forming reactive and passivating species very locally. The alumina dome could reveal this information.

Etch rate, erosion rate & selectivity

We already found an expressions for the oxide erosion rate at zero CCP power. Combining this formula with the expression for the initial silicon etch rate ($\text{AR} \ll 1$) gives us the initial selectivity:

$$S = \frac{ER_{Si}}{ER_{ox}} \approx 12 \cdot \frac{Q_{SF6}^2}{4Q_{SF6} + P_{ICP}} \quad (8)$$

where the flow is given in SCCM and the power in W. Of course, when we apply a bias voltage we should incorporate eq.3 in stead of eq.4. Note that the constants

"12" and "4" are depending on the equipment. Moreover, this equation suggests that the flow is ruling the selectivity. In fact, it is the pressure which controls the erosion rate, but, for a constant residence time these two variables are directly related to each other. Therefore, when the pressure is increased by decreasing the gas conductance, i.e. operating the pressure valve, the selectivity is increasing. However, this will increase the amount of bottling also. The best choice for setting the parameters will depend on what selectivity we need and how much bottling we can take. So, in case of relatively high power, the selectivity increases almost quadratic with flow. On the other hand, at lower powers, the selectivity increases only proportionally with flow. Figure 6 shows a plot of the selectivity (for $\text{AR} \ll 1$).

Profile: The etched profile is influenced by all parameters and becomes more positively tapered for lower SF_6 flow, higher oxygen content, higher ICP power, lower self-bias, and smaller mask openings. Quite surprisingly, the roughness of the sidewalls after etching were very good at the maximum etch rate. As an example, in figure 7a an optimised profile is shown etched at $15\mu\text{m}/\text{min}$. A remarkable effect is observed at the trench bottom: The originally circular mask opening has turned into a square! The sidewalls are found to be $\langle 110 \rangle$ planes and, therefore, the etch rate turns out to be depending on the crystallographic structure of the silicon material. So, the etching changed from ion bombardment anisotropy into crystal anisotropy at extremely low bias and plasma potentials. This crystal behaviour is even more pronounced by increasing the pressure as found in figures 7b and 7c. The figures clearly show the $\langle 111 \rangle$ faces created due to the difference in activation energy of the crystal orientation.

At a minimum achievable SF_6 flow (25SCCM) and ICP power (350W), the undercut and bottling effect decreases to practical zero. In figure 8 and optimised profile is shown. The corners of the mesa structures are quite eccentric eroded. This phenomena is caused by the double attack of incoming ions at these spots; two streams arriving from both faces. To decrease this effect, we should approach the black silicon region more closely.

Figure 8: Etching at low pressure and low ICP power.