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Silicon nanostructures with very large negatively tapered profile by inductively coupled plasma-RIE

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Dry etching of silicon has been extensively studied, mostly with a goal of obtaining perfectly vertical sidewalls with high aspect ratio. Yet, sloped sidewall with a negative taper angle (i.e., diameter/width decreases linearly with depth) may find various applications. However, the systematic study on the etching process development to obtain such a profile is rather scarce. In this work, the authors present a controlled and reproducible fabrication process to achieve silicon nanostructures with negatively tapered sidewall profile using inductively coupled plasma-reactive ion etching with C_4F_8 and SF_6 gas. The plasma etching parameters have been thoroughly optimized in order to avoid the undercut or curved reentrant profile due to isotropic etching, so as to achieve a negatively tapered profile. The influence of the plasma etching parameters, especially the radio frequency power and C_4F_8/SF_6 gas flow ratio, on the etching rate and the sidewall taper angle has been analyzed. With an optimal etching recipe, the silicon nanostructures with an unprecedented large 10° negative taper angle were achieved. These results were demonstrated on different structure sizes of 500 nm, 700 nm, and 1.2 μm diameters. © *2016 American Vacuum Society.*
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I. INTRODUCTION

The plasma etching combined with lithography techniques represents one of the most important patterning techniques, capable of fabricating micro and nanostructures with a good control of the feature size and shape, as well as the structures' sidewall profile. This is because dry plasma etching can achieve anisotropic etching due to directional physical bombardment by the ions, whereas wet chemical etching is usually isotropic. Apparently, silicon is the most frequently studied material for dry etching, and so far, most of the research efforts on Si plasma etching have been focused on the obtaining of perfectly vertical sidewalls with high aspect ratio, which is defined by the depth to width structure ratio.[1–3] The vertical sidewall is usually attained by finely balancing the etching and sidewall passivation by inhibitors such as fluorocarbon polymer or SiF_xO_y for cryogenic etching.[4] Deviation from the balancing point will lead to either a positively tapered profile when the passivation dominates or an undercut/reentrant profile when the etching dominates. Previously, using nonswitching Bosch process with C_4F_8/SF_6 gas, we obtained a positive taper angle varying from 4° to 22° when increasing the C_4F_8/SF_6 gas flow ratio to 59/1.[5] Unlike anisotropic etching of silicon by potassium hydroxide, which gives only a fixed taper angle of 35.5°,[6] dry etching is crystalline orientation independent with tunable taper angle and is thus a more versatile process and may find applications for highly efficient light capturing for imaging and solar energy harvesting.[7,8]

However, only few studies in the literature were reported regarding the means of obtaining structures with negatively tapered profile, which can be used for many applications. One application is to use negatively tapered silicon structures to make hydrophobic and omniphobic surfaces that repel both water and oil.[9] This is because that the pillars with negative profile can either generate a net force for lifting the liquid droplet upward to overcome the gravity when the liquid is suspended on the top of microstructures (Cassie-Baxter state), or offer a larger effective surface area than pillars with positive profile, which amplifies more the hydrophobicity when the liquid is in intimate contact with microstructures (no trapped air, Wenzel state).[10] Another potential application for negatively tapered structures is to fabricate reentrant atomic force microscopy (AFM) tips for critical dimension measurement and imaging sidewalls or overhang structures.[11] These kinds of structures can also be very useful as nanowires to fabricate nanodevices such as multigate transistors or single electron transistors.[12,13] The challenge comes from the fact that an undercut/reentrant profile (becomes positively tapered or curved with the narrowest part in the middle after mask removal), rather than a negatively tapered profile, usually results when decreasing the sidewall passivation. In the extreme case, without passivation by C_4F_8, pure SF_6 etching of silicon is isotropic like wet etching, because etching of silicon in fluorine-based plasma is spontaneous without the need of damage by ion bombardment, and the sticking coefficient of the etching species (free radicals) is only ∼0.01, so they mostly bounce back after hitting the etch bottom/sidewall to etch the silicon right below the mask.

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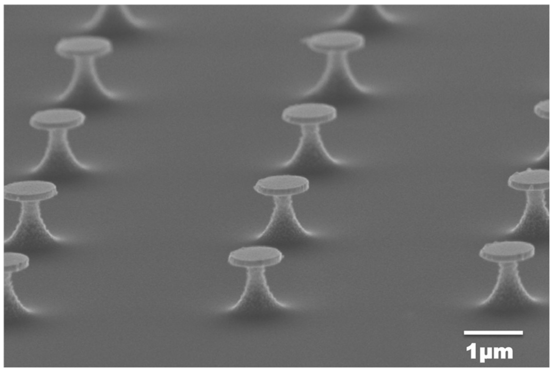

FIG. 1. SEM image of Si nanostructures with a pronounced undercut profile obtained with 65% of SF_6 gas in the C_4F_8/SF_6 gas flow mixture. The RF power, ICP power, and pressure are, respectively, 60 W, 1200 W, and 35 mTorr.

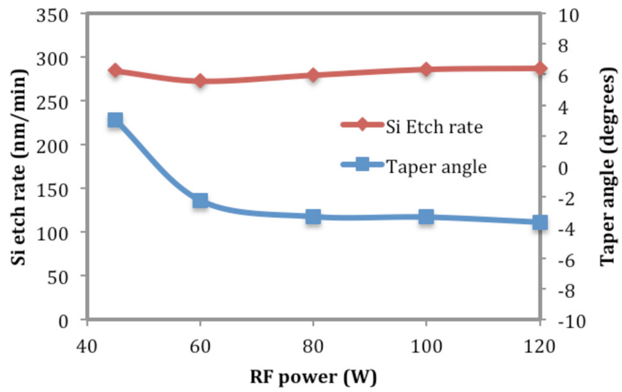

FIG. 2. (Color online) Si etch rate and taper angle as a function of the RF power.

In this study, we finely tuned the etching recipe using nonswitching Bosch process with SF_6/C_4F_8 gas and successfully attained a very large negative taper angle of 10°.

II. EXPERIMENT

Our fabrication process consists of two main steps: (1) electron beam lithography (EBL) and pattern transfer by the lift off technique to fabricate the Cr mask, followed by (2) inductively coupled plasma (ICP)-RIE to etch the underneath silicon with Cr as mask. A double resist layer was used in order to create an undercut profile for easy liftoff of thick metals.[14,15] ZEP-520A of 270 nm and 260 nm PMMA resists were spin coated on a Si wafer with the more sensitive ZEP resist as the bottom layer. After baking to drive off the solvent, EBL was performed using Raith 150[TWO] with exposure doses varying from 70 to 130 μm/cm^2, depending on the required sizes of the structures. After exposure, the resist film stacks were developed by amyl acetate, which is a good developer for both resists. Then, 150 nm of Cr was deposited by e-beam evaporation, followed by liftoff using anisole in an ultrasonic bath.

The silicon plasma etching was carried out using an Oxford ICP380 instrument in a gas mixture of C_4F_6/SF_6. Si wafer was used as a carrier during the plasma etching. In order to understand the effect of the different etching parameters on the etching rate and sidewall profile angle, the SF_6 percentage was varied from 25% (15 sccm) to 40% (24 sccm) with the total gas flow fixed at 60 sccm, and the RF power was varied from 45 (resulted in a bias voltage reading of 277 V) to 120 W (bias voltage 463 V) at a fixed ICP power of 1200 W, a pressure of 25 mTorr, an etching time of 10 min, and a temperature of 15 °C. The pressure is a critical parameter in the plasma etching. Generally, higher pressure leads to more reactive species (mainly free radicals) in the plasma to speed up the etching, and more collisions of the ions to decrease the etching anisotropy.[16,17] That is why in our first experiments, we tried to increase the pressure in order to promote the lateral etching for obtaining negatively tapered profile; however, it turned out that, for the range of RF powers fixed in this work (40–120 W), the ICP plasma failed to strike for pressures higher than 30 mTorr. Therefore, we fixed the pressure to 25 mTorr to ensure the stability of the ICP plasma.

After Si plasma etching, the etch rate of all the samples was measured by a Veeco Dektak profilometer, and the etched structures were examined by LEO 1530 scanning electron microscopy with the samples mounted on a 60° tilted stubs.

III. RESULTS AND DISCUSSION

As a reasonable starting point, we adopted the etching recipe that we reported previously, which gave a large positively tapered angle, except that we drastically increased the SF_6/C_4F_8 gas flow ratio (50%–65% of SF_6 in the gas mixture) in order to promote etching and suppress sidewall passivation. However, as shown in Fig. 1 for 65% of SF_6 gas

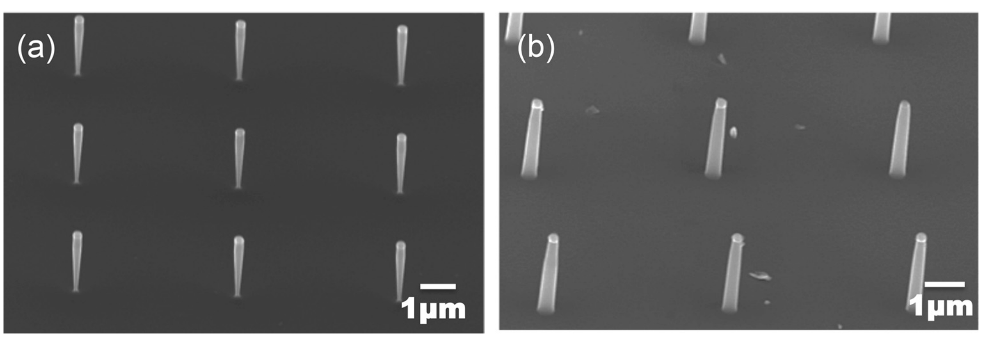

FIG. 3. SEM image of 300 nm diameter Si structures obtained at 33% of SF_6 ($C_4F_8/SF_6 = 40/20$) and RF power of, respectively, (a) 45 W (taper angle of 3°) and (b) 60 W (taper angle of −2.2°).

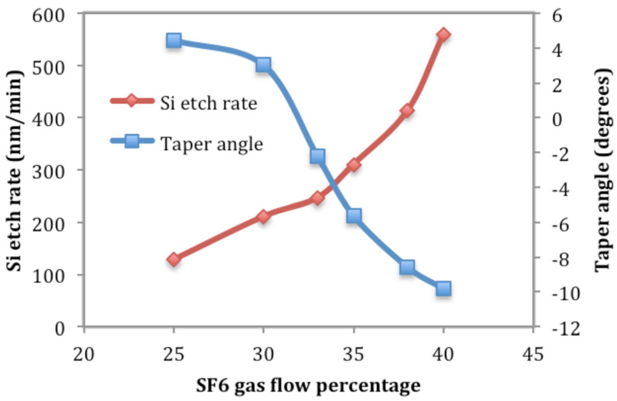

FIG. 4. (Color online) Si etch rate and tapered angle variation as function of the SF_6 gas flow percentage in the mixture.

percentage in the C_4F_8/SF_6 gas mixture, a clear undercut profile was obtained instead. Therefore, we systematically studied the synergic effect of RF power and gas flow ratio, as detailed in Secs. III A and III B. Other etching parameters, including pressure, ICP power, and substrate temperature, are not systematically investigated in this study. Generally, higher pressure results in more frequent collision of ions with the gas molecules and smaller bias voltage for a fixed RF power, which both decrease anisotropy; ICP power determines the plasma density and affects more the etching rate than the etching profile; and the substrate temperature affects the etching profile only when the volatility of the passivation layer varies significantly for the temperature range under study.

A. Effect of RF power

The effect of the RF power was studied at a fixed gas flow of $C_4F_8/SF_6 = 40/20$, an ICP power of 1200 W, and a pressure of 25 mTorr. Figure 2 shows the Si etch rate and the sidewall taper angle as function of the RF power. For the effect of RF power, on one hand, by increasing the RF power, the DC bias is increased and ions are then more accelerated toward the sample, which promotes physical sputtering and more directional etch. On the other hand, higher RF power leads to larger dark space (sheath) thickness above the sample, and thus, the ions have to travel a longer distance to reach the sample surface with higher chance of collision with gas molecules in the chamber, which promotes less directional etch.[18] The net effect is the tradeoff of those two competing effects. In this work, as seen in Fig. 2, we noticed a relatively small dependence of the Si etch rate on the RF power. Instead, the Si etch rate remained almost constant at around 280 nm/min for RF powers between 45 and 120 W. This indicates that here ions have little effect on the etching rate, since etching is mainly done by free radicals that are much more abundant (by 2–3 orders) than ions in typical plasma. This behavior was also observed by other researchers.[9,19] However, Fig. 2 shows that the sidewall taper angle decreased considerably from $+3.1°$ at 45 W to $-2.2°$ at 60 W, to saturate then at approximately $-3°$ between 80 and 120 W. Above RF power of 80 W, the balance of the two competing effects of RF power as mentioned above led to a nearly constant $-3°$ taper angle. As such, tuning the RF power alone did not give a large desired negative taper angle of close to $-10°$ (Fig. 3).

B. Effect of SF_6 gas flow percentage

As mentioned above, in the C_4F_8/SF_6 gas mixture, the SF_6 gas is destined to etch the Si whereas the C_4F_8 is used as a passivation to protect the Si structure sidewalls so as to achieve a good control of the etching profile.[20,21] Indeed, the SF_6 percentage can impact the structure sidewalls from a positive to a negative profile.[17] Therefore, the SF_6 gas flow percentage represents a key parameter to achieve a good control of the sidewalls profile. However, all the plasma etching parameters should be well optimized collectively to avoid the undercut profile usually obtained by only increasing the SF_6 gas flow, as shown in the example presented in Fig. 1.

The effect of the C_4F_8/SF_6 gas flow ratio on the etch rate and sidewall taper angle was studied at an optimal RF power of 60 W, an ICP power of 1200 W, and a pressure of 25 mTorr. The total gas flow is fixed to 60 sccm. In order to avoid dominance by chemical etching that promotes an undesirable undercut profile, a lower percentage of SF_6 (i.e., $<50\%$ of the total gas flow) than C_4F_8 gas was always used. Figure 4 shows the variation of the Si etch rate and the sidewall taper angle as a function of the SF_6 gas flow percentage in the mixture.

As seen, by slightly increasing the SF_6 gas flow from 20% ($C4F_8/SF_6 = 48/12$) to 40% ($C_4F_8/SF_6 = 36/24$), the etching rate is increased from 128 to 558 nm/min, and the taper angle is considerably decreased from 4.4° to $-9.7°$. The balancing point between etching and passivation to give a vertical sidewall profile was obtained at 33 SF_6 gas flow percentage. Examples of unique Si structures etched at different SF_6 gas flow percentages varying from 25% to 40% are shown in Fig. 5. A low magnification SEM image of an array of Si nanostructures obtained at 40% of SF_6 with a

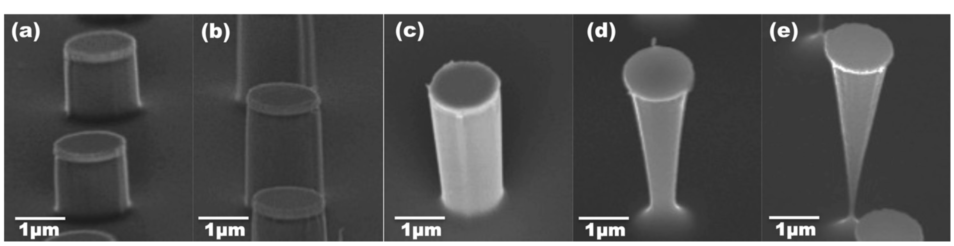

FIG. 5. SEM images of Si structures (Cr mask still on, mask diameter 1.2 μm) as a function of SF_6 gas flow percentage in the C_4F_8/SF_6 mixture with respective taper angles: (a) 25% of SF_6 (4.4°), (b) 30% of SF_6 (3°), (c) 33% of SF_6 ($-4.6°$), (d) 38% of SF_6 ($-8.5°$), and (e) 40% of SF_6 ($-9.7°$).

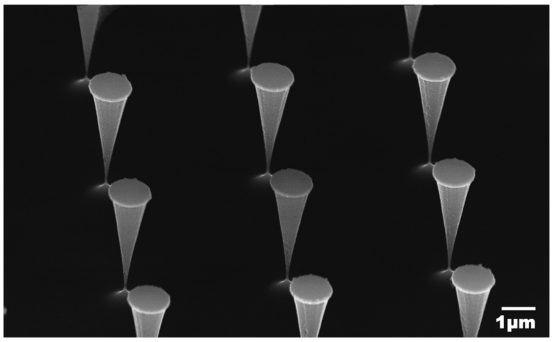

FIG. 6. SEM image of Si structures (Cr mask still on, mask diameter 1.2 μm) obtained at 40% of SF_6 and RF power of 60 W, showing a large negative taper angle of −9.7°.

large negative taper angle of −9.7° is shown in Fig. 6. To the best of our knowledge, this is the first time such a straight (not curved) and large negative taper angle is achieved by dry etching.

IV. SUMMARY AND CONCLUSIONS

In this work, we developed a reproducible ICP-RIE process to fabricate Si nanostructures with large negatively tapered sidewall profile. The influence of the plasma etching parameters (the chamber pressure, the RF power, and the gas flow ratio C_4F_8/SF_6) on the sidewall profile has been discussed. A low SF_6 percentage in the gas mixture (<50%) is found necessary to avoid the undercut or curved profile due to excessive isotropic etching by SF_6. By optimizing the plasma etching parameters, a good control of the Si structure sidewall with taper angle from positive to negative values was realized. In particaular, Si nanostructures with an unprecedented large negative taper angle of 10° have been achieved. These results are promising for many applications such as water and oil repelling surfaces, Si nanowires for single electron or multigate transistors, and lateral re-entrant AFM tips.

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