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Characterization of Silicon Isotropic Etch by Inductively Coupled Plasma Etcher for Microneedle Array Fabrication

JING JI
Mechanical Engineering National University of Singapore, 119260, Singapore

FRANCIS E.H. TAY
Mechanical Engineering National University of Singapore, 119260, Singapore

JIANMIN MIAO
MicroMachines Center, School of Mechanical and Aerospace Engineering, Nanyang Technological University, 50 Nanyang Avenue, 639798, Singapore

JIANBO SUN
MicroMachines Center, School of Mechanical and Aerospace Engineering, Nanyang Technological University, 50 Nanyang Avenue, 639798, Singapore
g0306040@nus.edu.sg

Abstract. This work investigates the isotropic etching properties in inductively coupled plasma (ICP) etcher for microneedle arrays fabrication. The effects of process variables including powers, gas and pressure on needle structure generation are characterized by factorial design of experiment (DOE). The experimental responses of vertical etching depth, lateral etching length, ratio of vertical etching depth to lateral etching length and photoresist etching rate are reported. The relevance of the etching variables is also presented. The obtained etching behaviours for microneedle structure generation will be applied to develop recipes to fabricate microneedles in designed dimensions.

1. Introduction
Microneedle arrays are widely used in drug and gene delivery in recent decade. Various approaches, such as dry and wet etching bath micromachining technologies, have been developed to fabricate silicon microneedles.[1] In KOH wet etching, the fabricated microneedles structure highly depends on the crystal planes of silicon. The etched product is hard to be controlled in wet etching also due to the wet etching results highly depend on temperature and components of enchant. [5] Plasma etching has been widely used in microneedle fabrication. Typically, the reactive ion etching (RIE) was used to achieve shape of microneedle, which is high aspect ratio structure with sharp tip. [6] Using the mask with cross pattern, side-opened microneedle was fabricated by the combination of isotropic etching and anisotropic etching process in ICP etcher. [7] The ICP high density plasma etcher is well known to carry out the DRIE, implemented with BOSCH process, which utilizes an etching cycle using SF$_6$ gas.
and then switches to a sidewall passivation cycle using C₄F₈ gas.[8] In the etching cycle based on SF₆ gas, the isotropic profiles is obtained, which can be used in microneedle fabrication to achieve the shape of needle tip.

Our work focuses on investigation of the isotropic etching behaviors in inductively coupled plasma (ICP) etching tool. The obtained results will be used in microneedles fabrication. In the experiment, the etch tool was operated only SF₆ gas without passivation cycle. Because of the complicate ICP etching mechanisms, the effects of process variables on etching results are nonlinear and correlated. Main process parameters include gas flow rate, platen power, coil power and automatic pressure control (APC) valve position. Due to the interaction influences of etching variable on etching results, some statistics methods have been used in plasma process characterization.[9][10] The design of experiments (DOE) technique was used in our process characterization to explore these correlated and interactional variables. The effects of multiple variables on output results (responses), including pressure, vertical etching, lateral etching, the ratio of vertical etching to lateral etching and photoresist etching rate were investigated.

| Table 1. Range of explored variables for isotropic etching |
|---------------------------------|
| Variable                      | Minimum | Maximum |
| SF6 flow rate (sccm)           | 30      | 150     |
| APV position (°)               | 30      | 75      |
| Platen power (W)               | 1       | 29      |
| Coil power (W)                 | 300     | 900     |

2. Experimental method

4-inch p-type silicon wafers with resistivity ranging from 1 ohmcm to 10 ohmcm, 450-500 um thick and (100) orientation were used in this study. The photoresist (AZ9260) were coated at 4000rpm (thickness ~3.5 um). Samples were exposed using mask aligner, developed with AZ400 and bake at 100 °C before ICP etching was carried out. The mask layout consisted of patterns with square arrays and dots arrays that exposed 12% of the total wafer surface. The size of the patterns is 80 μm in dimension with center distance 150 μm. All etches time in trials was 3minutes. The characterization work was performed using ICP etching tool from Surface Technology Systems (STS), which was used as an isotropic high-density plasma etcher for the passivation cycle was excluded.

Four variables were studied in this part of the experiment: SF₆ flow rate, automatic pressure control (APC) valve position, platen power and coil power. The ranges explored are presented in Table I. The selected ranges of mass flow and power were based on previous observations made by A.A.Ayon.[11][12] We used surface profiler to measure the depth of etched patterns. The etched photoresist thickness was calculated by measurement of depths before etching, after etching and after photoresist removing for each sample. A microscope integrated with video camera was use to take topside images of etched samples. Additionally, a scanning electron microscope (SEM) was used to obtain the etched structures under various etching recipes.

The commercial software Minitab® was used to create and analyze a matrix of 20 runs of process screening with DOE method. For the 4 factors full factorial design, 16 runs were carried out. The experimental design with 4 additional runs to verify the etching results. Totally 20 runs were executed to fit the quadratic model.

3. Results and discussion

The measured vertical etching depth, lateral etching length and photoresist etching rate were characterized; and the corresponding responses were produced. Calculated the ratio of vertical etching
depth to lateral etching length (V/L ratio), which indicates the final length of the fabricated needle, was also characterized. Figure 1 shows the relevant variables of microneedle profile.

In DOE, the effect of a factor, which is the change in response produced by a change in the level of the factor, weights the influence of a factor to responses. The significance of factors in the full factorial experiment can be constructed based on a normal probability plot of the effect estimate. By the factorial analysis in Minitab®, the effects of variables for various responses, such as pressure, vertical etching, lateral etching, ratio of vertical etching to lateral etching and photoresist etching rate, were achieved.

Fig. 1. The dimension used in microneedle fabrication (Drawing for the main dimension of microneedle fabrication used in this experiment. Vertical etching depth is V. Lateral etching length is L. V/L ratio is the vertical etching depth divided by lateral etching length. Photoresist etching rate is obtained by the thickness difference of photoresist between before and after etching divided by etching time.)

3.1. Pressure
In the STS ICP etching tool, the chamber pressure normally is controlled by varying the APC valve or changing the flow rate at the fixed APC position. The obvious effects of these two factors are verified in figure 2. Higher pressures correspond to higher values of SF6 flow rate and APC valve positions in degrees, as shown in figure 3. The response also benefits with higher coil power. In ICP, power is transferred from the electric fields to the plasma electrons near the surface by collisional dissipation and by collisionless heating process. [13] Therefore, higher coil power may cause higher surface temperature, which causes higher pressure.

Fig. 2. Normal probability plot of effects as pressure

Fig. 3. Pressure (mTorr) dependence on SF6 flow rate (sccm) and APV position in degree

3.2. Vertical etching depth (V)
The normal probability plot of the effect estimates for vertical etching depth from the ICP isotropic etching experiments is shown in figure 4. The vertical etching depth dependence on coil power and SF$_6$ flow rate is illustrated in figure 5. Clearly, coil power is the most significant variable which controlled the vertical etching depth because the ion flux reached to the etched silicon surface is determined by this variable. This response also benefits with higher SF$_6$ flow rate due to that higher level of this variable helps to remove the byproduct.[14] In addition, vertical etching depth increase at higher value of APV position, because the residence time of reactive gases is longer at higher pressure. The residence time is proportional to PV/f, where P is the pressure, V is the chamber volume and f is the gas flow rate.

![Fig.4. Normal probability plot of effects as Vertical etching depth](image1)

![Fig.5. Vertical etching depth (μm) dependence on SF6 flow rate (sccm) and coil power (W)](image2)

3.3. Lateral etching length (L)

Figure 6 shows the effect of variables on the lateral etching length. The effects distribution is similar to vertical etching depth because the isotropic profile is obtained using SF$_6$ gas in plasma etching. Based on the analysis of this response, it indicates that lateral etching increases with respect to vertical etching when the ion fluxes increase. However, the lateral etching rate is lower than the vertical etching rate. Lateral etching most benefits from SF$_6$ flow rate may due to higher flow rate take away more etching products and make less deposition on the side wall.

![Fig.6. Normal probability plot of effects as Lateral etching length](image3)
3.4. Ratio of vertical etching to lateral etching (V/L ratio)
This response is important because it is expected to play a role in determining the height of needle. The longer needle will achieve under higher value of V/L ratio. Based on this response analysis, higher platen power and low SF₆ flow rates contribute to increase the V/L ration as shown in figure 8. The V/L ratio dependence on platen power and SF₆ flow rate is illustrated in figure 9.

3.5. Photoresist etching rate
Figure 10 shows the normal probability plot of effects on the photoresist removed rate. Platen power (electrode power) is the dominate variable as it is far from the line passing through the other points. For the ion bombardment energy is determined by the platen power, higher applied platen power contributes to high ion bombardment energy that caused larger photoresist etching rate. Figure 10 also illustrates the larger SF₆ flow rate and higher APV position contribute to the less photoresist etching for these two standardized effects have negative value factors. It due to higher chamber pressure lowers the ion energy. At higher SF₆ flow rate and APV position, the higher pressure is achieved, as illustrated in figure 2.
4. Conclusions

Using full factorial factors design method, each effect of variable for experimental responses was investigated in isotropic etching in ICP etcher. Based on DOE analysis, the vertical etching and lateral etching, have a predominant dependence on coil power and SF$_6$ flow rate, which decide the ion flux in etching; the higher V/L ratio benefit from lower SF$_6$ flow rate and higher platen power; and the photoresist etching rate increase with platen power increasing. Therefore, under the followed etching condition, higher coil power, higher APV position, higher platen power and lower SF$_6$ flow rate, the higher aspect ratio needle structure will achieved with short fabrication time. In addition, a quantitative model to present the process is also obtained with the effect of variables on a response in DOE model analysis. In the future work, reduced model will be set up based on the analysis of the quantitative model, and it will be used in fabrication of designed needle structure.

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