Full Factorial Design ($2^k$) for 45 Degree of Wall Angle in Anisotropic Wet Etching Process

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Abstract

This research aims to discover the optimal anisotropic wet etching condition in order to reduce hillocks that occur on the etched surface or reduce roughness and increase etch rate for 45 degree of wall angle (micro-mirror) of a silicon substrate by adopting a design of experiment (DOE) technique and the factorial $2^k$. Three potential factors which are an ultrasonic mode, a speed motor and a sample orientation, are employed in the factorial design. Analysis of variance (ANOVA) at a p-value significance level of 0.05 is used to assess the significance of the factors on an etch rate and a surface roughness ($Ra$). The silicon substrates were etched in 20 wt. % sodium hydroxide (NaOH) with Isopropyl alcohol (IPA) at 60 °C of solution temperature. An experiment with 24 runs, eight conditions and three replications for each condition, was performed and it was found that the ultrasonic mode was a significant factor which affected the etch rate. An ultrasonic mode and a speed motor were significant factors influencing the surface roughness ($Ra$). The optimal conditions of 45 degrees of wall angle of a silicon substrate, which were the maximum etch rate and the minimum roughness, were investigated by using a desirability optimization technique in sense of a soft mode of ultrasonic, a speed motor of 5 rpm and a vertical orientation with a desirability value of 0.7619. Finally, the optimal conditions were verified with experimental result.

Keywords : Anisotropic Wet Etching, Silicon and Silicon dioxide, Hillock, Ultrasonic Agitation, 45 ° Wall Angle, Design of Experiment
I. Introduction

Anisotropic Etching is a vital technique in the Micro Electro Mechanical Systems (MEMs) [II] and Micro-Opto-Electro-Mechanical Systems (MOEMS). The chemical wet etching is widely used in the electronics industries as it can provide a smooth etched surface [III] and control a wall angle with a low cost. So, it is commonly used in the small electronic devices fabrication [IV]. The important parameter in this manufacturing processes is the wall angle generated by the orientation of the structure on the wafer. The final structure (wall angle) is dominated by the slow etching plane. The wall angle is widely used in the electronic industry to be 45 degrees, 90 degrees or 54.7 degrees [XI]. To create a micro-mirror, the wall angle of substrate is always 45 degrees to reflect the light beam as it is easy to control the reflected beam direction [III] or use as an optical switching or an optical interconnection [X]. Moreover, it can be used as a light delivery system for heat-assisted magnetic recording [V]. The research related in this field is still in development. For example, An et al. [IX] tried to develop an optical mold application which has a 45 degree of wall angle. Pal et al. [VII] fabricate an optical micro-mirror which has a 45 degrees of wall angle. In the electronics application, if the wall angle is not equal to 45 degrees, the reflected beam light does not hit to the target which affects product reliability negatively [VII].

Furthermore, a smooth etched surface is also important because it affects the quality and the reliability of the product as well. Even though, an anisotropic wet etching technique provides a smooth etched surface, it has a limitation which is a long processing time. So, it is not appropriate to be used in a mass production which is typically for electronics industry. Nevertheless, the etching time can be reduced by increasing a solution temperature, but the hillocks will appear on the etched surface causing roughness on the surface. Currently, there are many researches that applied an ultrasonic technique to reduce these hillocks [I, VIII]. Pakpum [VI] etched a Si p-type (100) in a NaOH with IPA solution and adopted an ultrasonic cleaning machine which can reduce the amount of hillock to 2.64 % with Rq 6.77-22.6 nm. Yang et al. [XI] studied the use of ultrasonic agitation techniques and found out that it can increase the etch rate and the etched surface of a silicon wafer to be smoother by using KOH solution with ultrasonic 40 kHz and power 50 W. This caused a surface roughness less than 15 nm. Besides, some research applied an ultrasonic agitation with a surfactant to reduce surface roughness. A silicon (100) wafer with a silicon dioxide (SiO2) as a mask is etched in KOH with isopropyl alcohol solution which is a surfactant. The etching solution is in an ultrasonic bath with a frequency of 100 kHZ, ultrasound intensity 50 watts per liter (W/L) and a solution temperature of 60°C. The result showed that the etched surface roughness is smoother than the result from using the KOH solution with ultrasonic agitation solution which has 7.34 nm of roughness and 0.048 nm per second (nm/s) of etch rate [VIII].

From the above research, there are many essential parameters to reduce hillocks and increase etch rate for 45 degrees of wall angle such as the type of solutions, additive surfactants, temperature of the solution, agitation, etc. where these parameters should be the optimal values. Therefore, the aim of this research is to adapt the design of experiments (DOE) techniques to design an experiment, study the significant factors.
and construct a mathematical model to determine the optimal parameters for multi-response of the wet etching process combined with the desirability optimization technique according to the targets of maximum etch rate and minimum roughness. This can be a further alternative to improve the manufacturing process in the electronic industry fabrication.

II. Experiments

A 6-inch silicon wafer or silicon substrate, p-type plane (100) with 500 nm thickness of silicon dioxide (SiO2), was sliced to a square of 20 × 20 mm and the mask pattern is created by using the lithography technique on the substrate. This technique starts from coating a positive photoresist (AZ1512). The mask pattern are 45 degrees aligned to major {110} crystal axes as shown in Fig. 1 to create 45 degrees of wall angle [1]. The photoresist is exposed to UV light 80 per square centimeter (mJ/cm²) on the photoresist by using the EVG 610 Mask Aligner. After that, the photoresist is removed by using AZ Developer and the SiO$_2$ layer is eliminated by hydrofluoric (HF) solution.

A $2^k$ factorial design is applied with 3 factors which are i) frequency mode of ultrasonic cleaner, (ii) speed of the motor and (iii) a sample orientation. Each factor can be run at two levels in the experiment. Two response variables are interesting in this work which are a) etch rate and b) surface roughness. Three replicates of a 2$^3$ design were run in this research; the factor levels used in the design are shown in Table 1. The 24 experiments are run in random order as shown in Table 2.

![Fig. 1: Orientation of structures on (100) wafer. Alignment of 45° relative to flat lead to {110} walls](image-url)
Table 1: The level and symbol of the wet etching system

| Factors/Units                                      | Level          |
|---------------------------------------------------|----------------|
| Frequency mode of ultrasonic cleaner (mode)       | Low (-1)       |
| Speed of the motor (rpm) (speed)                  | High (1)       |
| Sample orientation                                |                |

Table 2: The 23 Design for the Wet Etching Experimental

| Run Order | Standard order | Mode | Speed | Sample orientation | Etch Rate (nm/min.) | Surface Roughness (Ra) (nm.) |
|-----------|----------------|------|-------|--------------------|---------------------|----------------------------|
| 1         | 14             | Normal | 0    | Vertical           | 257.691            | 26.6180                    |
| 2         | 8              | Normal | 5    | Vertical           | 250.565            | 16.9806                    |
| 3         | 21             | Soft   | 0    | Vertical           | 220.611            | 17.4140                    |
| 4         | 23             | Soft   | 5    | Vertical           | 223.203            | 8.9452                     |
| 5         | 24             | Normal | 5    | Vertical           | 235.784            | 17.4359                    |
| 6         | 5              | Soft   | 0    | Vertical           | 183.111            | 16.0768                    |
| 7         | 20             | Normal | 5    | Horizontal         | 251.707            | 23.1380                    |
| 8         | 10             | Normal | 0    | Horizontal         | 256.449            | 25.3853                    |
| 9         | 7              | Soft   | 5    | Vertical           | 206.418            | 9.7766                     |
| 10        | 12             | Normal | 5    | Horizontal         | 233.682            | 19.3210                    |
| 11        | 1              | Soft   | 0    | Horizontal         | 197.735            | 19.1080                    |
| 12        | 22             | Normal | 0    | Vertical           | 216.260            | 22.8028                    |
| 13        | 18             | Normal | 0    | Horizontal         | 251.707            | 23.1380                    |
| 14        | 17             | Soft   | 0    | Horizontal         | 179.182            | 16.9702                    |
| 15        | 11             | Soft   | 5    | Horizontal         | 214.144            | 11.0182                    |
| 16        | 16             | Normal | 5    | Vertical           | 236.686            | 17.1830                    |
| 17        | 3              | Soft   | 5    | Horizontal         | 212.176            | 10.2142                    |
| 18        | 19             | Soft   | 5    | Horizontal         | 202.071            | 12.7500                    |
| 19        | 15             | Soft   | 5    | Vertical           | 205.241            | 9.8729                     |
| 20        | 9              | Soft   | 0    | Horizontal         | 198.184            | 18.3197                    |
| 21        | 2              | Normal | 0    | Horizontal         | 252.054            | 23.0695                    |
| 22        | 4              | Normal | 5    | Horizontal         | 256.888            | 15.2631                    |
| 23        | 13             | Soft   | 0    | Vertical           | 186.006            | 16.3300                    |
| 24        | 6              | Normal | 0    | Vertical           | 256.702            | 24.0087                    |

The substrate was etched in a 20% wt. concentration of sodium hydroxide (NaOH) with 20 ml. of isopropyl alcohol (IPA) solution at 60 °C in the chemical wet etching system which modified an ultrasonic cleaner GT-1620 (2-liter) with a frequency of 40 kHz and a power of 50 watt by applying a motor and the substrate holder as shown in Fig. 2.

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In this work, cross section images were taken using the optical microscope and Bruker 3D Optical Microscope. The MB-Ruler software was used to measure the wall angle where the target is 45 degrees and the vision 64 program was applied to measure the etch depth and to calculate the etch rate (nm/min) (see Equation 1). For the roughness, three points for each substrate were measured randomly for each substrate and the vision 64 software provided the Ra value. Gauge repeatability and reproducibility analysis was applied to ensure stable and consistent measurements.

\[
\text{Etch Rate} = \frac{\text{Etch Depth}}{\text{Etching Time}} \times 1000
\]

(1)

![Fig. 2: The chemical wet etching system (A) a design of chemical wet etching system (B) a design of substrate holder (C) the chemical wet etching system used in this experiment](image)

The resulting data are shown in Table 2 and it was then analyzed by regression using Minitab Program version 18. The gradient search was adopted to solve the optimization problem which gives the optimal combination of parameters for maximum composite desirability value. The target for the etch rate is a maximum value and roughness is a minimum value. The predicted responses under the optimal condition are also compared with experimental results for the same set of parameters.

### III. Results and Discussions

After the 45 degrees of wall angle was created by aligning the mask pattern 45 degrees to major \{110\} crystal axes and performed the chemical wet etching following the conditions in Table 2. In the experimental result,
Table 2 analyses residual plots to determine the adequacy of models for regression and analyses the regression coefficients evaluated by using ANOVA. This approach determined the statistical significance of each factor in terms of two responses: a) etch rate (nm/min.) and b) surface roughness (Ra) (nm) as follows:

Residual normal probability plot for etch rate and surface roughness (Ra) is shown in Fig. 3. The figure includes normal probability plot, residual fitted values, histogram of the residuals and residuals versus order of data. In the normal probability plots, all residuals have normal distribution as the plot forms a straight line. The residual versus fitted value plot shows that residuals have equal variance. The histogram of the residuals shows a normal distribution. In addition, the residuals versus observation order of data shows that residuals are independent from observation order.

The experimental results and analysis of variance for etch rate and surface roughness are shown in Table 3 and 4. Based on the experimental results, single regression models were developed to predict etch rate and surface roughness. The general polynomial equation (Equation 2) for three factors considered in this case is:

\[ y_i = \beta_0 + \sum_{j=1}^{3} \beta_j x_j + \sum_{j=1}^{3} \beta_{jj} x_j^2 + \sum_{i=1}^{3} \sum_{j=i+1}^{3} \beta_{ij} x_i x_j \]

From the analysis of variance in Table 3 and 4, the corresponding parameter is promising where \( P \) values are less than 0.05. Mode (x1) is the promising factors for etch rate, at the 95% confidence level. For surface roughness, mode (x1) and speed (x2) are promising factors at the same confidence level. In the reduced model, it was found out that the coefficients of the variables affecting the responses were the same. These coefficients are used (Table 4 and 5) to
Alonggot Limcharoen Kaeochotchuangkul et al. develop the mathematical model. The formulas obtained from the linear regression model for the two responses (Equation. 3 and 4) are:

\[ Y_1 = 224.33 + 22.01 x_1 \]  
\[ Y_2 = 17.547 + 3.648 x_1 - 3.223 x_2 \]  

Where \( y_i \) is the response and \( x_j \) represents mode and speed and sample orientation, respectively.

Based on the analysis of variance from Table 3 and experimental results, the etch rate depends on the ultrasonic mode (which is normal mode and soft mode). The normal mode is generating a higher etching rate than the soft mode because it generates a strong and a huge frequency wave that can scrub hydrogen bubbles (H\(_2\)) on the silicon surface. The hydrogen bubbles are generated on the etched surface called the ‘pseudo-mask’ phenomenon, which inhibits the chemical reaction between the etchant and silicon atoms on the surface and this causes the lower etching rate [VIII] and let the NaOH solution (Etchant) bombard the silicon surface faster. This will increase the etch rate of the wet etching process. For the soft mode, the etch rate was lower than normal mode as the soft mode is a single frequency ultrasonic wave having a soft frequency and thus leading the NaOH reach the silicon surface slower than the normal mode.

Table 3: Estimated effects and coefficients for etch rate

| Term                        | Effect | Coef | SE Coef | T-Value | P-Value | VIF |
|-----------------------------|--------|------|---------|---------|---------|-----|
| Constant                    |        | 224.33 | 2.77    | 80.87   | 0.000   | 1.00|
| Mode (x₁)                   | 44.03  | 22.01 | 2.77    | 7.94    | 0.000   | 1.00|
| Speed (x₂)                  | 6.09   | 3.05  | 2.77    | 2.23    | 0.028   | 1.00|
| Sample orientation (x₃)     | -2.29  | -1.15 | 2.77    | -0.81   | 0.685   | 1.00|
| Mode*speed (x₁*x₂)          | -10.35 | -5.17 | 2.77    | -1.87   | 0.081   | 1.00|
| Mode*Sample orientation (x₁) | -5.84  | -2.92 | 2.77    | -1.05   | 0.308   | 1.00|
| speed*Sample orientation (x₂) | 0.16   | 0.08  | 2.77    | 0.03    | 0.977   | 1.00|
| Mode*speed*Sample orientation (x₁*x₂) | 1.56   | 0.78  | 2.77    | 0.58    | 0.783   | 1.00|

\[ S = 13.5894 \quad R\text{-sq} = 81.18\% \quad R\text{-sq(adj)} = 72.95\% \quad R\text{-sq(pred)} = 57.66\% \]
Table 4: Estimated effects and coefficients for etched surface roughness (Ra)

| Term                                    | Effect | Coef  | SE Coef | T-Value | P-Value | VIF |
|-----------------------------------------|--------|-------|---------|---------|---------|-----|
| Constant                                | 17.547 | 0.359 | 48.91   | 0.000   |         | 1.00|
| Mode (x₁)                               | 7.296  | 3.648 | 0.359   | 10.17   | 0.000   | 1.00|
| Speed (x₂)                              | -6.445 | -3.223| 0.359   | -8.98   | 0.000   | 1.00|
| Sample orientation (x₃)                 | 1.188  | -0.594| 0.359   | -1.66   | 0.117   | 1.00|
| Mode*speed (x₁*x₂)                      | 0.495  | 0.248 | 0.359   | 0.69    | 0.500   | 1.00|
| Mode*Sample orientation (x₁*x₃)        | 0.473  | 0.237 | 0.359   | 0.66    | 0.519   | 1.00|
| speed*Sample orientation (x₂*x₃)        | 0.731  | -0.365| 0.359   | -1.02   | 0.324   | 1.00|
| Mode*speed*Sample orientation (x₁*x₂*x₃)| -0.596 | -0.298| 0.359   | -0.83   | 0.419   | 1.00|

S = 1.75758 R-sq = 92.21% R-sq(adj) = 88.80% R-sq(pred) = 82.48%

The promising factors affecting the surface roughness (Ra) in the wet etching system are a) ultrasonic mode and b) speed of motor. P-Value is used for ultrasonic mode and speed of motor factors are less than 0.05 (α = 0.05) as shown in table 4. The ultrasonic waves and rotating agitation (speed of motor) generate the waves in the wet etching process and this can remove H₂ bubbles on the surface quicker, so it will improve the surface roughness.

Mathematically, the optimum etching condition leading to the maximum total desirability is shown in Table 5 and Fig. 7. Fig.7 shows the response of each parameter (mode, speed and sample orientation on the x-axis) to the individual corresponding values (composite desirability, etch rate and roughness on the y-axis). The far-left column presents the effects of the multi-parameters to composite desirability, etch rate and surface roughness.

To confirm the results, the wet etching system was adjusted to follow the optimal condition suggested in Table 6 in order to obtain the results as shown in Fig. 4 - 6. Three experiments to confirm the results were run at 60 °C for 90 minutes. It was found out from the tests, that the etch rate and surface roughness values were very close to the target values. Based on this, the optimal etch rate should be 211.6205 nm/min and the surface roughness should be 9.5316 nm. The wet etching system
IV. Conclusions

The design of experiment method combined with optimization and desirability function technique was adopted to determine the optimal parameter setting in wet etching systems. Two responses with a single material are obtained in this work, in contrast to prior research that used basic statistics to analyse a single response with the same material and no replication in the experiment [VI]. The significant factors of this research are different from prior work, where researchers postulate that orientation affects surface roughness. In this research, the experiments have performed three replications for each condition and the analysis of statistical results shows, that the orientation does not affect etch rate and surface roughness. Mathematically, the multi-responses with a single material correspond to linear regression, while there is no mathematic model or analysis of variance from prior research. Etch rate and surface roughness responses employing wet etching system have been discussed in this research. Parameter optimization for multi-response was shown and composite desirability function was applied according to the targets of maximum etch rate and minimum surface roughness. This produced an optimal condition for wet etching system as follows:

- ultrasonic mode = soft,
- speed of motor = 5 rpm and
- sample orientation = vertical.

With this optimal condition, the response obtained from the mathematical model correlates very well with the wet etching experiments showing only a small error factor. Therefore, the proposed optimization methodology in this research could be proofed to be effective.

Fig. 4: Top view of SEM image (A) showing etched surface of the Optimal condition (B) showing etched surface of the {110} planes with 45° angles to the (100) surface (C) showing etched surface (D) showing etched surface of NaOH 20%wt., Temperature 60 °C and no ultrasonic
Fig. 5: Bruker 3D Optical Microscope pictures of 3D surface plot etched surface

Fig. 6: Bruker 3D Optical Microscope pictures of 3D surface plot etched surface
Table 5: Estimated effects and coefficients for etched surface roughness (Ra)

| Response          | Mode | Speed (rpm) | Sample Orientation | Desirability Value | Composite desirability |
|-------------------|------|-------------|--------------------|--------------------|------------------------|
| Etch Rate (Maximum) | Soft | 5           | Vertical           | 0.90316            |                        |
| Roughness (Minimum) |      |             |                    | 0.64279            | 0.7619                 |

Fig. 7: The optimal value of parameters for each response
Table 6: The results confirm using the optimal parameters

| Run | Etch Rate (nm/min.) | Ra (nm) | Wall Angle (°) |
|-----|---------------------|---------|----------------|
| 1   | 196.799             | 7.905   | 45.05          |
| 2   | 206.027             | 9.740   | 45.09          |
| 3   | 203.356             | 10.040  | 45.41          |
| Average | 202.061           | 9.228  |                |

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