Optimization of KOH etching process for MEMS square diaphragm using response surface method

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ABSTRACT

Potassium hydroxide (KOH) wet etching is widely used in realizing microelectromechanical systems (MEMS) diaphragm due to its low cost, safe and easy handling. However, a variety of etching parameters such as etchant concentration, temperature, mask size and etching time need to be optimized thoroughly in order to save the time and costs of the etching process. This paper presents the numerical study and optimization of KOH etching process parameters using the response surface method (RSM) to realize the desired shape and size of MEMS diaphragm. Face central composite design (FCC) of RSM was employed as the experimental design to analyze the result and generate a mathematical prediction model. From the analysis, the temperature was identified as the most significant process parameter that affects the etching rate, thus affecting the thickness and size of the diaphragm. The results of RSM prediction for optimization were applied in this study. Particularly, 45% of KOH concentration, temperature of 80°C, 1735 µm2 of mask size, and 7.2 hours of etching time were implemented to obtain a square MEMS diaphragm with thickness of 120 µm and size of 1200 µm2. The results of RSM based optimization method for KOH wet etching offers a quick and effective method for realizing a desired MEMS devices.

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1. INTRODUCTION

Wet etching has been extensively used for the fabrication of microelectromechanical systems (MEMS) components including diaphragms based on single crystal silicon due to its cost effectiveness, simple experiment setup, and easy handling compared to dry etching [1]. Even though potassium hydroxide (KOH) and tetramethylammonium hydroxide (TMAH) are well known etchants used in wet etching process, KOH is more commonly used due to its high anisotropy reaction between the Si {111} and Si {100} planes [2]. Moreover, the etch rate of Si {100} is higher in KOH than in TMAH. Notably, this is indispensable for high productivity to reduce the cost of end products [3]. Nonetheless, the etching of (100)-oriented silicon using aqueous KOH creates V-shaped grooves problems with the (111) planes at an angle of 54.74° from the (100) surface, as illustrated in Figure 1. From Figure 1, the etch depth, *d*, and the width of the opening, *W*, can be determined when (100) wafers are used:

$$W = L - 2a = L - \frac{2d}{\tan 54.74^o} = L - \frac{2d}{1.414} \quad (1)$$

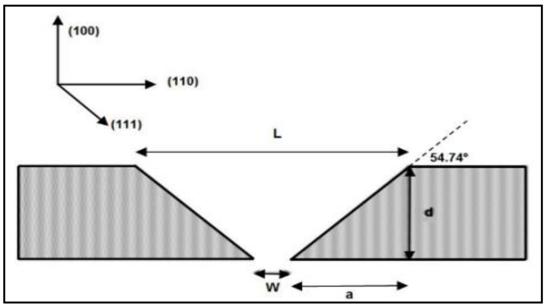

Figure 1. Formation of a V-shaped groove in (100) silicon using a KOH solution [4]

The etching characteristics of silicon wafers are vital for the fabrication of specific microstructural feature via wet etching technology. Even though the anisotropic etch characteristics of different types of wet etchants are well documented in the literature, the ideal condition for releasing a variety of complex, bulk micromachined structures are typically done using the trial-and-error method [5]. The classical one-factor-at-a-time (OFAT) method does not indicate the interactive effects of all parameters involved. Plus, it requires numerous experimental runs to attain the optimum levels and hence, consume too much time and has low cost effectiveness [6]. Many researchers had utilized statistically optimization in their research to conduct much more systematic approach [6]–[14]. One of the most well-known statistical design experiments is response surface method (RSM). RSM is a statistical technique to describe the behavior of set of data with the objective of making statistical previsions [9]. Generally, it is applied in statistical experimental design to determine the effect of factors, make prediction, search for optimum target, analyze interaction among variables, and propose a mathematical model that signify the whole process [10]. The major application of RSM is for optimization purposes. Therefore, the present study utilized the RSM method to investigate the characteristics of KOH wet etching with the aim to optimize the etching process parameters in realizing a simple square MEMS diaphragm. Four important parameters, namely temperature, KOH concentration, etching time, and mask size were investigated before designing the diaphragm. The interactions among the parameters were analyzed using the face central composite (FCC) experimental design.

2. RESEARCH METHOD

First and foremost, KOH wet etching simulation was conducted using AnisE, the etch simulation tool in IntelliSuite. Then, the output simulation values were statistically analyzed using RSM. Finally, the MEMS square diaphragm was fabricated to verify the reliability of the statistical model.

2.1. Simulation of KOH Wet Etching using IntelliSuite Software

Etch simulation tool of IntelliSuite, AnisE was used to generate the 3D model for anisotropic etching of silicon. With AnisE, the simulation of etching was conducted under various parameters of temperature, KOH concentration, etching time and mask size. Conducting etching simulation significantly reduced the fabrication cost and time. Most importantly, it help researchers to investigate etching behaviors, make prediction, and optimize the MEMS devices. Figure 2 shows the sample structure of MEMS square diaphragm simulated using AnisE.

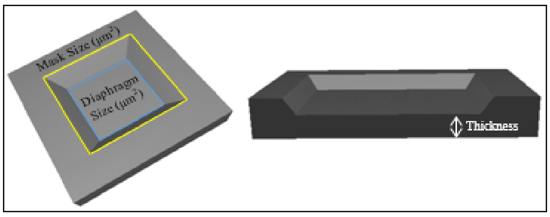

Figure 2. Model of MEMS square diaphragm

2.2. Statistical Analysis using Response Surface Method

In order to investigate the KOH etching parameters, RSM using Design–Expert software was chosen as the statistical method. In this paper, the etching factor parameters, namely temperature, KOH concentration, etching time, and mask size for square MEMS diaphragm were selected to investigate their interaction to the output responses namely diaphragm's thickness, etch rate, and diaphragm's size. Table 1 presents the levels of etching parameters and the targeted output responses. The levels of each factor in the current research were selected based on previous related studies [4], [15-19]. The main target in the present study is to develop MEMS square diaphragm with size of 1200 µm2 and thickness of 120 µm.

Table 1. Etching Factor Parameters and Output Response with Their Respective Codes

Code	Factor/ Response	Etching Parameters	Low level (-1)	Center (0)	High Level (+1)
A	Factor	Temperature (°C)	40	60	80
B	Factor	KOH Concentration (%)	25	37.5	50
C	Factor	Etching Time (h)	1	3.35	5.7
D	Factor	Mask Size (µm2)	1200	1600	2000
E	Response	Diaphragm Thickness (µm)		Target = 120 µm	
F	Response	Etch rate (µm/h)		Target = Maximum	
G	Response	Diaphragm Size (µm2)		Target = 1200 µm2	

2.3. Fabrication of Square Diaphragm

In this research, a <100> oriented n-type silicon wafer of 4-inch diameter, 525±25 µm thickness, with double-sided 200 nm coated silicon nitride was used. After dicing the wafer into 2.5 cm x 2.5 cm size, the substrates were cleaned to remove any organic contaminants. The fabrication of square diaphragm utilized KOH wet etching with prior steps of photolithography and Buffered oxide etchant (BOE) etching process [15]. Before the photolithography process, the sample was spin coated with AZ 1500 positive photoresist using spin coater with a spin setting of 500 rpm for 5 s, followed by 3000 rpm for 35 s. Then, the resist was baked at 100°C for 1 min. In order to transfer the pattern of square diaphragm to the sample, the sample was exposed to UV exposure for 35 s. Next, the sample was baked at 100°C for 2 min. Then, the pattern was developed by immersing the sample in AZ300K developer for 1 min. In order to prepare the window pattern for the KOH wet etching, the silicon nitride layer was etched. BOE etching method was used to remove the nitride layer with the etching rate of 45 nm min^{-1} at 80°C [20]. In this study, the sample was immersed in BOE solution by double boiling at constant 80°C temperature for 10 min to completely remove the 200 nm nitride layer on unprotected photoresist layer [15]. Finally, wet anisotropic etching was performed by double boiling process as shown in Figure 3. The double boiler heating method was preferred due to its controllability and its ability to provide consistent heating to the etchant [21].

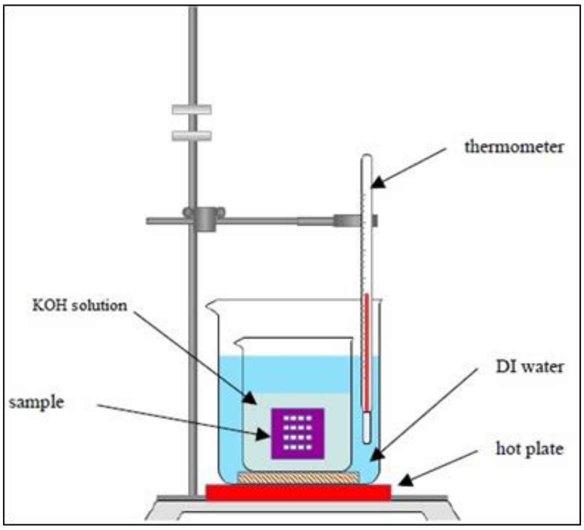

Figure 3. KOH etching process setup [11]

3. RESULT AND DISCUSSION

This section presents the etching simulation results, statistical analysis and optimization using RSM, in addition to the comparison of RSM prediction with simulated and experimental etching results of fabricated MEMS square diaphragm.

3.1. Etching Simulation Result by IntelliSuite

First, the etching of square (100) silicon MEMS diaphragm was carried out over a range of etching parameters as shown in Table 1 using IntelliSuite, AnisE software. Next, the output responses obtained from the simulation were collected and tabulated as illustrated in Table 2.

Table 2. Experimental Design of KOH Etching Simulation

Run\Code	A	B	C	D	E	F	G
1	0	0	0	0	436	21.175	1520
2	0	-1	0	0	421.5	25.98	1480
3	0	0	-1	0	494.2	21.175	1580
4	-1	1	-1	0		3.78	
5	1	1	1	-1	247	46.06	840
6	-1	1	-1	-1		3.78	
7	0	0	0	0	436	21.175	1520
8	1	-1	-1	-1	436	84.11	1100
9	-1	-1	-1	-1		6.91	
10	1	0	0	0	276.2	68.555	1280
11	1	-1	1	-1	14.5	84.11	520
12	-1	0	0	0	494.2	5.63	1580
13	0	1	0	0	465.1	14.23	1540
14	-1	1	1	-1	494.2	3.78	1180
15	0	0	1	0	392.4	21.175	1440
16	-1	-1	1	-1	479.6	6.91	1160
17	1	1	-1	1	465	46.06	1940
18	-1	-1	1	1	479.6	6.91	1960
19	0	0	0	0	436	21.175	1520
20	1	-1	1	1	14.5	84.11	1320
21	0	0	0	1	436	21.175	1900
22	-1	-1	-1	1		6.91	
23	0	0	0	0	436	21.175	1520
24	0	0	0	-1	436	21.175	1100
25	0	0	0	0	436	21.175	1520
26	1	-1	-1	1	436	84.11	1900
27	1	1	1	1	247	46.06	1640
28	1	1	-1	-1	465.1	46.06	1140
29	0	0	0	0	436	21.175	1520
30	-1	1	1	1	494	3.78	1980

*run number 4, 6, 9, and 22 were subjected to simulation constraints to measure diaphragm's thickness and size

3.2. Statistical Analysis of Response Surface Method

The process of statistical analysis was started with analysis of variance (ANOVA) in order to evaluate the adequacy of the response surface model developed. Table 3 shows the ANOVA for the data generated by the RSM model for KOH wet etching simulation. The p-value of $p<0.0001$ indicated that the model was significant. The value of R-squared (R2) describes up to what extent a model can perfectly estimate the experimental data points, whereas the adjusted R2 measures the amount of variation on the mean values explained by the model [22]. From Table 3, the predicted R2 values were close to the actual R2 values. This reveals that the experimental data fitted well with the predicted values of the model.

Table 3. Analysis of Variance (ANOVA)

Response	p-value	R2	Adjusted R2	Predicted R2	Mean
Diaphragm Thickness	<0.0001	0.9959	0.9939	0.9831	396.31
Etch rate	<0.0001	1.0000	1.0000	1.000	1.28
Diaphragm Size	<0.0001	0.9988	0.9982	0.9953	1450

The final regression models in terms of actual factors for diaphragm's thickness, etch rate, and diaphragm's size predictions are outlined as:

Diaphragm Thickness = 590.29280 + (10.51111 Temperature) – (17.44854 KOH Concentration) + (17.83930 Time) – (5.35714x10-5 Mask Size) + (0.21363 Temperature KOH Concentration) – (1.85479 Temperature Time) + (1.69434 KOH Concentration Time) – (0.14128 Temperature2)

Log10(Etch Rate) = – 0.57981 + (0.036901 Temperature) + 9.63770x10-3 KOH Concentration + (4.62548x10-7 Temperature KOH Concentration) – (8.14864x10-5 Temperature2) – (2.6845x10-4 KOH Concentration2)

Diaphragm Size = 31.72340 + (17.47264 Temperature) –(24.01191 KOH Concentration) + (24.60993 Time) + (1.00000 Mask Size) + (0.29400 Temperature KOH Concentration) – (2.55319 Temperature Time) + (2.33191 KOH concentration Time) – (0.21944 Temperature2)

The relative differences among the parameters are illustrated through perturbation plots in Figure 4, Figure 5, and Figure 6 to provide notable summary for the responses. By default, the Design–Expert software sets the reference point at the midpoint (coded value 0) of all factors. The perturbation plot can be used to find the factors that affect the responses the most. Figure 4 shows the perturbation plot for the etch rate (100) and evidently, it was mainly influenced by temperature (A), with small influence from KOH concentration (B). Moreover, Factor A was directly proportional with the etch rate (100) and this was different for Factor B which was inversely proportional to the etch rate. Meanwhile, the etching time (C) and mask size (D) did not give any effect on the etch rate (100).

Figure 5 illustrates the perturbation plot for the diaphragm thickness. Notably, the etching temperature (A) and etching time (C) had the most significant effect on the diaphragm thickness. Thus, these two parameters should be well controlled to obtain a very thin diaphragm. In contrast, varying the KOH concentration (B) did not give significant effect on the thickness. This showed a positive correlation between the etch rate (100) and diaphragm thickness as the temperature increase would increase the etch rate (100), thus, decreasing the diaphragm's thickness.

Figure 6 portrays the perturbation plot for the diaphragm size. From Figure 6, the mask size (D) was found to be the most important parameter that should be controlled to realize the desired diaphragm size. Apart from that, temperature (A) and etching time (C) had small influences, whereas KOH concentration (B) had no statistical significance to the diaphragm size. Figure 7 shows the combined effect of KOH concentration and temperature on the etch rate (100). From Figure 7, KOH concentration did not affect the etch rate, but when combined with high temperature (80˚C), it would give significant effect to the etch rate. Referring to Figure 7, the etch rate increased with the rise of temperature, and it accelerated starting from 60°C to 80˚C.

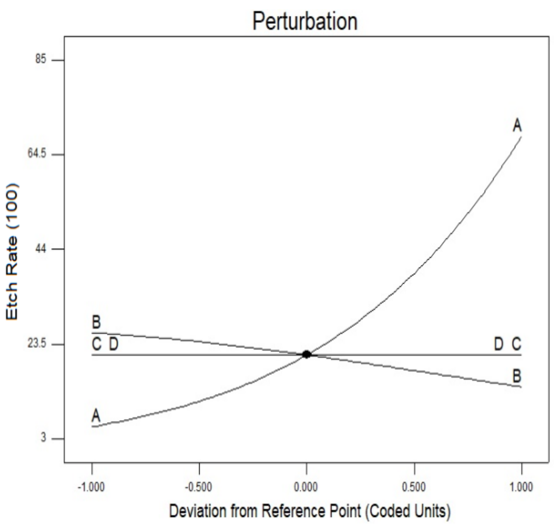

Figure 4. Perturbation plot for etch rate

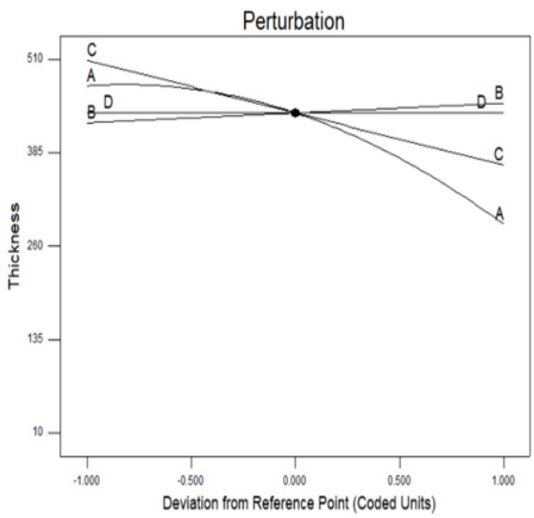

Figure 5. Perturbation plot for thickness

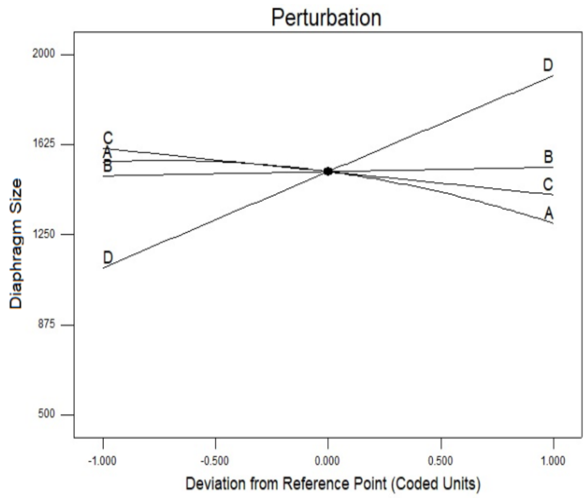

Figure 6. Perturbation plot for diaphragm's

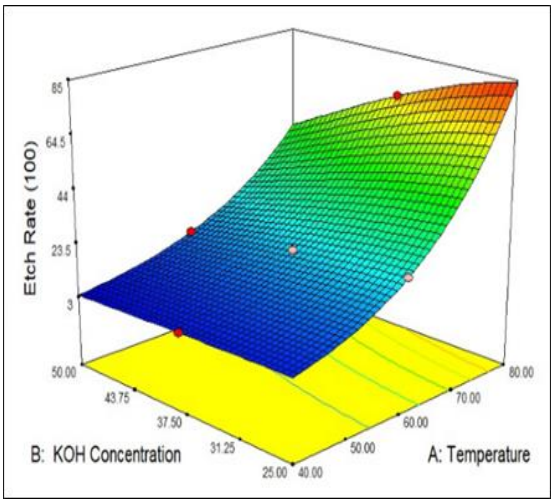

Figure 7. Combined effect of concentration and temperature on etch rate

3.3. Optimization of MEMS Square Diaphragm Size

The discussion depicts that all of the factors had a significant effect on the responses. Hence, all the factors should be set at optimum condition so the MEMS structure could be realized for desired thickness and size with minimum etching time. In this study, the goal for multi-objective optimization was to minimize the etching time with the targeted diaphragm's thickness of 120 µm and targeted diaphragm's size of 1200 $µm^2$. In this optimization process, the KOH concentration was fixed at 45% in order to avoid rough surfaces of the silicon etched [11]. Even though low KOH concentration was better in achieving high etching rate (100), this parameter should be compensated with surface roughness parameter to get a smooth surface. Moreover, KOH concentration had less significant effect to the etching rate (100) if compared to the temperature. Table 4 shows the best four predicted values to achieve multi-objective optimization goals via the RSM model developed. The ideal technique for selecting the optimum operating characteristics of the etching process was by referring to the desirability approach. Higher desirability score of the resulting response reflects better optimization of the etching characteristics [23, 24]. These results demonstrate that in order to obtain a desired optimization target, the controlled factors should be set approximately at temperature (A) ≈ 80°C; KOH concentration (B) = 45%; etching time (C) ≈ 7.2 h; with mask size (D) = 1735 $µm^2$. At this condition, the diaphragm's thickness (E), etch rate (F), and mask size (G) were predicted to be approximately 120 µm, 55 µm/h, and 1200 $µm^2$, respectively. These controlled values were applied in IntelliSuite simulation and experimental etching process, and consecutively, compared with the RSM prediction.

Table 4. Optimal Etching Parameters for Targeted Diaphragm Structure

Run	A (OC)	B (%)	C (h)	D (um2)	E (um)	F (um/h)	G (um2)	Desirability
1	80.0	45.0	7.19	1735	120	55.29	1200	0.96
2	80.0	45.0	7.22	1735	120	55.04	1200	0.96
3	80.0	44.9	7.18	1747	120	55.27	1212	0.96
4	80.0	45.0	7.20	1756	120	55.27	1221	0.96

3.4. Comparison of Simulation and Experimental Etching Results

The simulation and fabrication of MEMS square diaphragm were conducted by adopting the predicted parameters as estimated by RSM. Figure 8 shows the KOH etched square diaphragm by IntelliSuite simulation. The simulated results indicated that the thickness and size of the diaphragm were 102 µm and 1240 $µm^2$, respectively. Figure 9 presents the experimental results of anisotropic etched silicon on a square diaphragm. From Figure 9, the diaphragm's size was shown to have a dimension of approximately 1275 $µm^2$, while the thickness of the diaphragm was approximately 120.2 µm. Table 5 shows the comparison of the diaphragm's size and thickness between the RSM prediction, simulation, and experimental results. For the simulated results, the diaphragm's size and thickness showed small dimensional deviation, with 15% thickness variation and 3.3% size variation. The comparison between the experimental and predicted results indicated that the error was 0.17% in terms of diaphragm's thickness and 6.25% for the diaphragm's size. In order for statistical analysis to be reliable, error values must be less than 20% [25]. Hence, from the results, it was concluded that the model developed using RSM could accurately predict the KOH etching process.

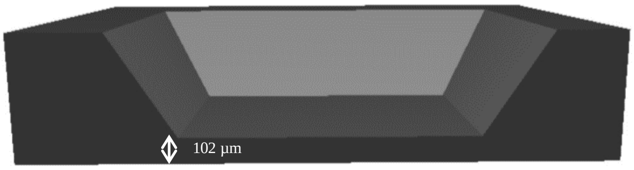

Figure 8. Simulated result of cross-sectional view of the MEMS square diaphragm structure

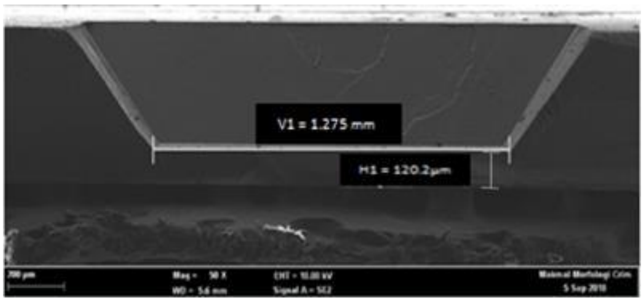

Figure 9. SEM picture of experimental results of anisotropic etched silicon on a square diaphragm

Table 5. Comparison of the Predicted Optimum Conditions

Method	Diaphragm size	Diaphragm Thickness	Deviation error with RSM prediction
RSM Prediction	1200 µm2	120.0 µm	-
Intellisuite Simulation	1240 µm2	102.0 µm	15.0% (diaphragm thickness) 3.3% (diaphragm size)
Experimental KOH Etching	1275 µm2	120.2 µm	0.17%(diaphragm thickness) 6.25% (diaphragm size)

4. CONCLUSION

The implementation of RSM based design optimization for MEMS square diaphragm structure is presented. The mathematical models have been developed for silicon etch rate (100), thickness and size of the diaphragm. The statistical RSM results based on these mathematical models were satisfactory and in accordance with the results of the simulation and the experiment. The optimization results demonstrate that in order to obtain the square MEMS diaphragm with thickness of 120 µm and size of 1200 µm2, the controlled factors should be set approximately at etching temperature ≈ 80°C; KOH concentration = 45%; etching time ≈ 7.2 h; with mask size = 1735 µm2. The difference between the validated value and the RSM predicted value was within 0.17 - 15 %, indicating that RSM method was able to predict the optimum parameters of KOH etching with low error. Hence, RSM was found to be an effective method to predict the wet etching process parameters for realizing MEMS structure, thus reducing the time consumed and the fabrication cost of refining those etching parameters.

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