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Design of experiment for the optimisation of deep reactive ion etching of silicon inserts for micro-fabrication

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ABSTRACT

The following paper describes a design of experiments investigation of the deep reactive of pillar structures on a silicon wafer. The etched wafers would subsequently be used as masters for the fabrication of nickel mould inserts for micro-injection moulding.
Undercuts occur when the pillar base has a smaller cross-section than the apex of the pillar. They therefore affect tolerances of the subsequent nickel mould, its strength and its de-mouldability from the silicon form.
The response measured in these experiments was the degree of undercut of micro-scale (10 μm x 10 μm x 40 μm, 5 μm x 5 μm x 40 μm and 2 μm x 2 μm x 40 μm)
The literature suggests that gas pressure, platen power, gas flow rate, phase switching times and mask size can all affect the degree of undercut. After examination of this literature, and of manufacturers guidelines, three parameters were selected for experimental testing: platen power, C_4F_8 gas flow rate during the passivation phase and switching times.
Switching times was found to be the only statistically significant parameter for both 10x10 μm and 5x5 μm pillars. The 2x2 μm pillars were not successfully replicated and could therefore not undergo statistical evaluation.

KEY WORDS LIST
Deep reactive ion etching, photolithography, microfabrication, design of experiment, platen power, C_4F_8 gas flow, switching times, undercut

1. INTRODUCTION

Undercuts, where the dimensions of the features are smaller at the base than at the apex, to surface features are an issue which needs to be examined when fabricating surfaces designed to be used in further processes. For example if the surface is to be used as a mould insert then undercuts can affect the accuracy of the replicate and in extreme cases render the mould de-mouldable.

Deep reactive ion etching (DRIE) is an anisotropic etching process used to fabricate surface features with vertical sidewalls in silicon. Using a time-multiplexed etching process [1; 2], in which a combination of etching and the deposition of a protective passivation layer is used, vertical etching of a sample can be controlled. This process is known as the Bosch process, in which the wafer is subjected to alternate etching and passivation phases and the silicon surface can be etched with micro-scale features with vertical sidewalls. The Bosch process produces these vertical sidewalls by continuously alternating between an etching phase and a passivation phase, resulting in an anisotropic etch pattern, Figure 1.

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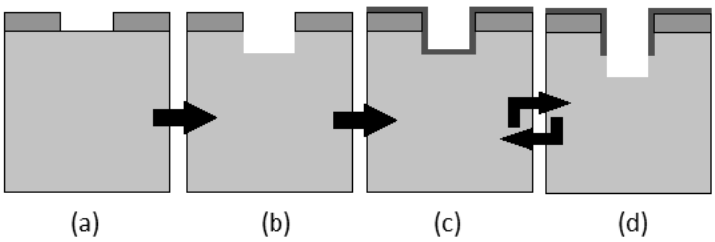


Figure 1, DRIE Bosch Process (a) Silicon wafer with photoresist, (b) Anisotropic etching phase, (c) Passivation phase, (d) Second anisotropic etching phase [3]

During the etching phase the silicon wafer is etched by directional bombardment with plasma ions. SF_6 gas is most commonly used when etching silicon. For the passivation phase the etched silicon is coated in a chemically inert layer. C_4F_8 gas is used to produce this inert layer when etching silicon, which protects the surface from further etching.

During the subsequent etching phase, due to the directional bombardment of plasma ions, the passivation layer at the base of the etched features is sputtered away and further etching of the silicon will continue in an orthogonal direction [4]. Only the passivation layer at the base of the etched features is removed, the sidewalls of the feature remain coated in the protective layer, which results in highly anisotropic sidewalls. Each phase lasts only a few seconds and the alternation between the two continues for a pre-set period of time until the desired etch depth is reached.

The literature suggests that several parameters which can affect the production of undercuts to surface features during DRIE notably: Pressure [2; 5-7], platen power [2; 8], gas flow [7-9], switching times [7; 9] and mask size [9].After examination of this literature and of manufactures guidelines[10] three parameters were selected for experimental testing: platen power, C_4F_8 gas flow in the passivation phase and switching times between phases. Platen power has a large influence on etch rate [2; 8], according to Evans & Beheim [2] when a high platen power is combines with high pressure during the etching phase the anisotropic nature of the profile is improved.

Altering the switching times, the ratio between the etching and passivation phases, has been found a major effect on the etch profile and presence of undercuts. As shown in Figure 2, the proposed mechanism is that by increasing the etch time the likelihood of a negative etch profile, and therefore undercuts, is increased. Whereas if the passivation rate is increased the thickness of the passivation layer is also increased resulting in a reduced etch rate and a more positive etch profile [2], Figure 2.

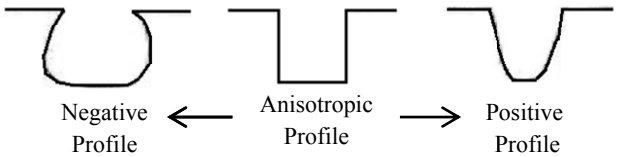


Figure 2, Schematic of etch profiles

Alterations to the C_4F_8 gas flow in the passivation phase have been found to effect the production of a more positive etch profile [10] due to the increased thickness of the passivation layer.

The aim of this paper is to examine the effect of these factors on the undercut sustained by the various pillar features during the DRIE process. Pillar features of 10x10 μm, 5x5 μm and 2x2 μm were used.

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2. METHOD

2.1. Experimental Design

The deep reactive ion etching process was investigated using a design of experiment (DOE) approach. The DOE approach to process optimisation was developed as a statistical method capable of highlighting the interactions between various factors to determine the optimum levels required for experimental work [11]. As well as being more practical regarding time and resources when compared to methods which focus on the alteration of a single factor per run, the DOE approach also provides a route to observe how various factors may interact to affect the response [12].

The factor levels used for this DOE are displayed in Table 1. The criteria for the selection of these levels are explained in Table 2.

Factor	Low Level	High Level
Platen Power (W)	10	18
C_4F_8 Gas Flow in Passivation Stage (sccm[†])	70	100
Switching times – Etching: Passivation (s:s)	5:5	9:5

Table 1, Factor examined; high and low levels

Factors	Low Level	High Level
Platen Power (W)	The minimum value at which ion directionality could be controlled for the scale of the features examined – taking into account equipment limitations	The maximum value at which ions directionality could be controlled for the scale of the features examined – taking into account equipment limitations
C_4F_8 Gas Flow in Passivation Stage (sccm)	The minimum value for obtaining a sufficient passivation layer after consideration of the passivation layer required and system pressure – taking into account equipment limitations	The maximum value at which deposited passivation layer would not hinder etching of features after consideration of the passivation layer required and system pressure – taking into account equipment limitations
Switching Times – Etching: Passivation (s:s)	The minimum level chosen due to feature dimensions and passivation layer thickness (if the etching time is too short, in comparison to the passivation time, it will not completely remove the passivation layer and the likelihood of the uneven etching increases)	The maximum level chosen after taking into account the dimensions of the features and the effect on the passivation layer (if the etching time is too long, in comparison to the passivation time, it will quickly remove the passivation layer and begin to isotropically etch the silicon)

Table 2, Reasons for DOE levels selected

The DOE scheme used was devised using a full factorial 8 run design generated using Minitab®15. The order of the DOE runs, Table 3, was generated using a built-in randomizer function in Minitab.

[†] sccm: standard cubic centimetre per minute

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Run	Platen Power (W)	C_4F_8 Gas Flow in Passivation Stage (sccm)	Switching Times – Etching: Passivation (s:s)
1	-	+	-
2	+	+	-
3	-	+	+
4	-	-	+
5	+	-	+
6	+	+	+
7	+	-	-
8	-	-	-

Table 3, Randomised 8 run full factorial DOE sequence

The response examined was the undercut of the features, Figure 3, and was calculated using equation 1. Where W_1 is the width of the pillar apex, W_2 is the width of the pillars base and U is the degree of undercut. For each run, 10 pillars were measured and the average of these was taken as the response value.

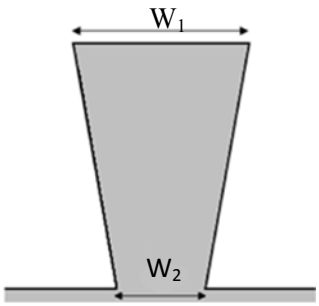


Figure 3, Schematic of pillar undercut

$$W_1 - W_2 = U \qquad (1)$$

The experimental data gathered was analysed using main-effects plots, Pareto charts and normal probability plots generated using Minitab®15.

2.2. Experimental Procedure

Silicon wafers underwent photolithography to pattern the wafer surface prior to DRIE. The photolithography process coated the wafer in a positive photoresist layer which was then exposed to UV light through a photomask. Areas of the photoresist which were exposed to UV light through gaps in the photomask were dissolved during development leaving areas of the wafer surface susceptible to etching during the DRIE process. Areas of the photoresist not exposed to UV light were not removed during development and acted as a protective layer from the etching process.

The wafers were first placed in a Polaron PT7160 RF plasma barrel etcher for a time of 120 s , and then placed on a hot-plate (115^o) for 120 s, to ensure the wafer surface was clean. The wafers were then placed in a hexamethyldisilazane environment for 15 minutes before being coated with S1818 positive photoresist in a spin-coater with a rotational speed of 4000 rpm for a duration of 60 se. The wafers were then placed back on the hot-plate for 90 s.

The wafers were placed in an MS56 mask aligner and exposed to UV light through a chrome-on-glass photomask, using a hard contact exposure programme, to pattern the resist surface with the features to be etched. Each wafer was exposed for 25 seconds.

The wafers were placed in a developer solution of MF319 for 25 seconds, and then rinsed in distilled and deionised water.

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The wafers were then placed back into the plasma barrel etcher for a further 120 s.
A deep reactive ion etcher was used to process the experimental runs. Each run consisted of 100 cycles, and due to the various switching times not all runs took the same amount of time, Table 4.

Run	Samples Name	Switching Time (s:s)	Total Run Duration (min)
1	DOE 1	5:5	16:45
2	DOE 2	5:5	16:45
3	DOE 3	9:5	23:29
4	DOE 4	9:5	23:29
5	DOE 5	9:5	23:29
6	DOE 6	9:5	23:29
7	DOE 7	5:5	16:45
8	DOE 8	5:5	16:45

Table 4, Duration of each DRIE run

The surface of each wafer was imaged, post etch, using an optical microscope. Wafers then underwent Oxygen plasma cleaning to remove any remaining photoresist, in order that the features could be examined in a field emission gun scanning electron microscope (SFEG-SEM). The conditions of cleaning are shown in Table 5.

Wafers were then re-examined using an optical microscope so a comparison of the features, pre and post Oxygen plasma cleaning, could be made. Upon comparison it was noted that there was no difference between the pre and post Oxygen plasma cleaned samples.

Pressure (mTorr)	45.0
O2 Gas Flow (sccm)	45
Coil Power (W)	800
Platen Power (W)	0
Cycle Time (min)	5

Table 5, O_2 plasma cleaning Parameter settings

The pillar features on the wafer surfaces were imaged and examined using an SFEG-SEM. The width at the base and the apex of the pillars were measured and compared to determine the degree of undercut the features obtained in each DOE run using equation 1.

3. RESULTS

3.1. Appearance of features

Figures 4a and 4b (run 3 and run 4) show examples of severe undercutting achieved on 5x5 μm features. The degree of undercut is such that no pillars could be observed standing on these samples. In contrast, figures 4c and 4d (runs 1 and 2) show examples where the undercut is either positive or minimal.

Figures 5a to 5c (runs 2, 1 and 8) show examples of uneven etching, where features are present between the pillars. For contrast an example of an evenly etched surface is shown in Figure 5d (run 7). A potential mechanism for the uneven etching observed for runs 2, 1 and 8 is the incomplete removal of the passivation layer during the etching phase. Residual particles of the passivation layer will act as randomly placed nano-scale masks resulting in the uneven etching pattern[13; 14]. In extreme cases this can result in the "black silicon" effect, shown in Figure 5c (run 8) where the uneven etching of the surface has become gradually rougher through the etching cycles so that "black silicon" has been formed. ("Black silicon" is a needle-like surface structure comprised of silicon spikes which absorbs light reducing the reflective properties of the silicon surface [14].)

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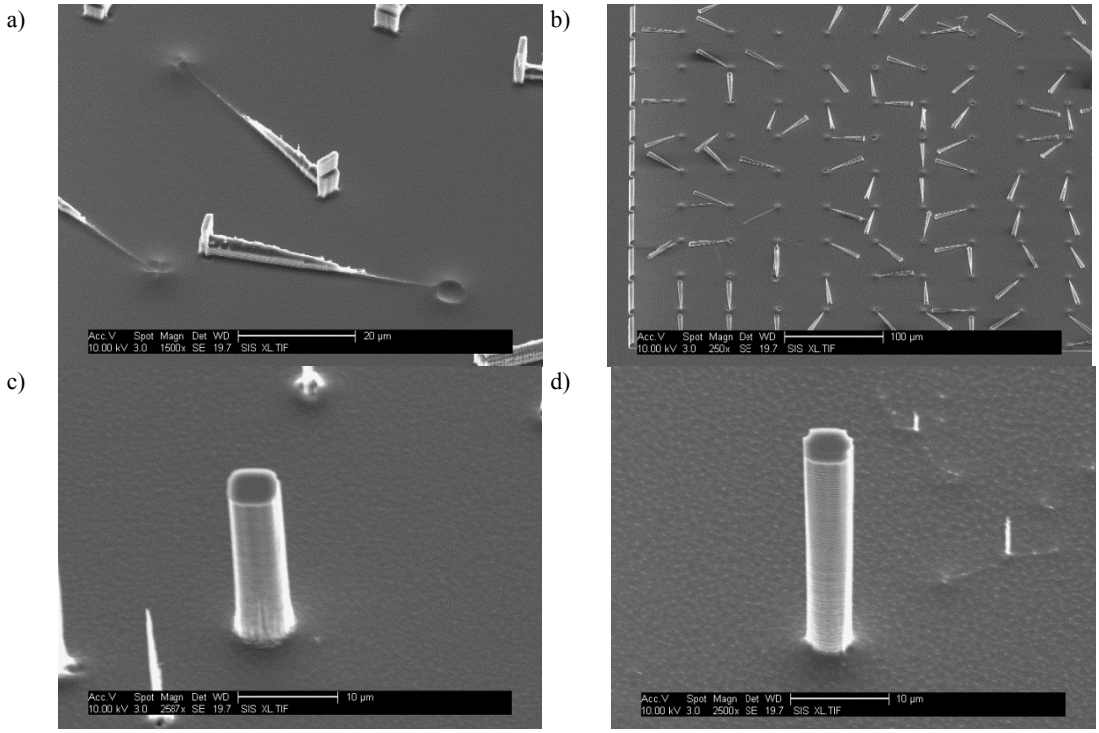


Figure 4, 5x5µm pillars, a) Run 3, b) Run 4, c) Run 1, d) Run 2

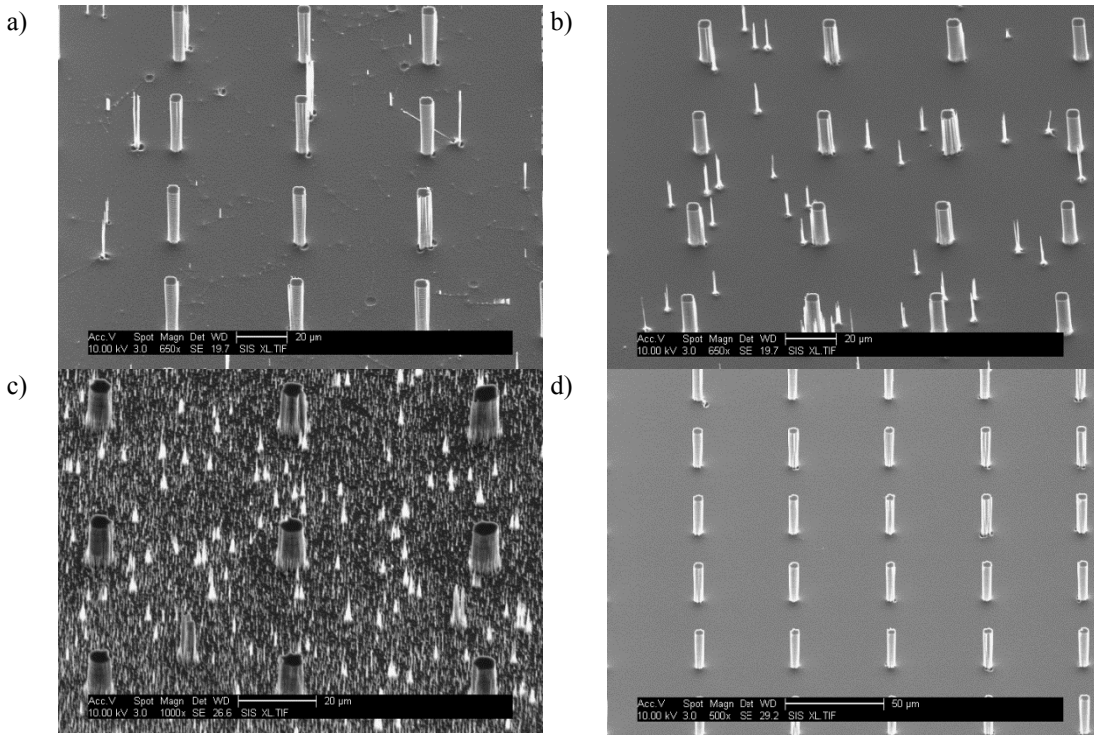


Figure 5, 5x5mm pillars, a) Run 2, b) Run 1, c) Run 8, d) Run 7

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Upon examination of the 10x10 μm features it was noted that incomplete etching of the wafer surfaces was observed for the same runs as those displayed for the 5x5 μm pillars. It should also be noted that the runs which resulted in the severely undercut 5x5 μm pillars also resulted in 10x10 μm pillars with large undercuts, although all the 10x10 μm pillars remained standing,

3.2. Measured undercuts

Table 6 shows the average undercut for the 10x10 μm and 5x5 μm pillars. None of the 2x2 μm pillars were successfully etched.

Run	Platen Power (W)	C_4F_8 Gas Flow in Passivation Stage (sccm)	Switching Times – Etching: Passivation (s:s)	Undercut Averaged from 10 pillars	
				10x10 μm	5x5 μm
1	-	+	-	0.028	-0.349
2	+	+	-	0.354	0.441
3	-	+	+	1.271	-
4	-	-	+	1.572	3.986
5	+	-	+	2.545	-
6	+	+	+	1.800	-
7	+	-	-	0.087	0.444
8	-	-	-	-0.729	-1.091

Table 6, Average undercut for 10x10 μm and 5x5μm Pillars

3.3. Statistical analysis

Figures 7-9 show the main-effects plots and Pareto charts for the 10x10 μm and 5x5 μm pillars.

Figures 6 and 8 are Pareto charts which display the significance of either an individual factor or of factor interactions using a bar chart. The bars are ordered in descending magnitude according to the effect on the response. The vertical line on the plot represents the point beyond which a factor becomes statistically significant; the value of alpha (α - 0.05) indicates a chosen confidence limit of 95%. The following symbols were used to represent the various factors on the Pareto charts: (A) platen power, (B) C_4F_8 gas flow and (C) switching time.

Figures 7 and 9 are main-effects plots of each factor and display the factors' affect on the magnitude of the average undercut.

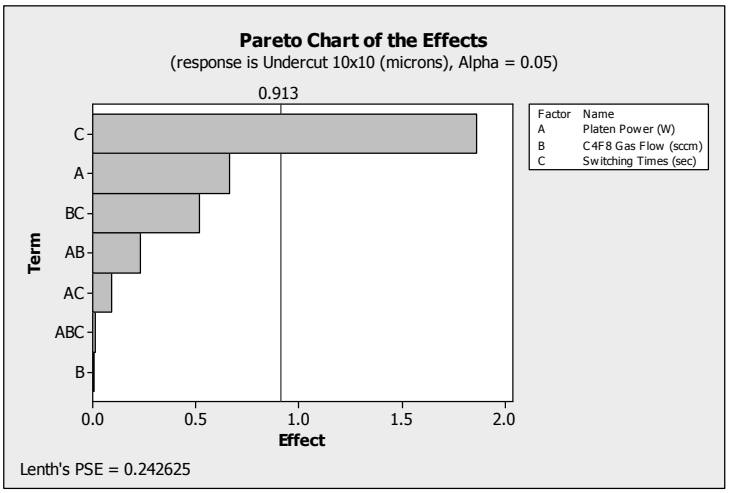


Figure 6, Pareto Chart for 10x10 μm pillars

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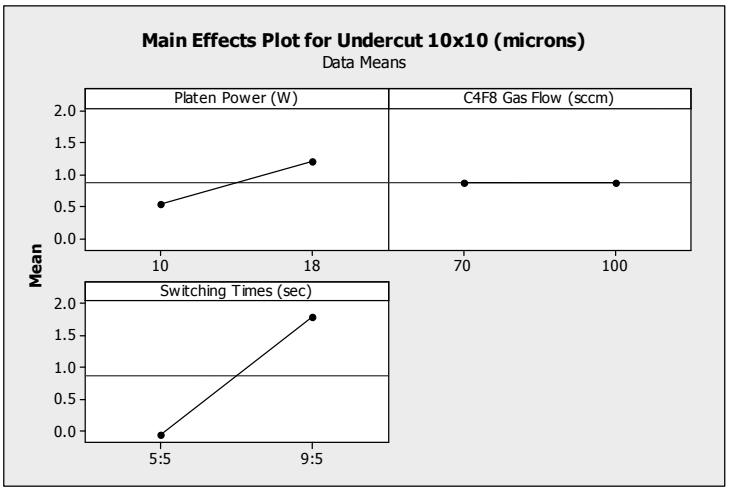


Figure 7, Main-Effects Plot for 10x10 μm pillars

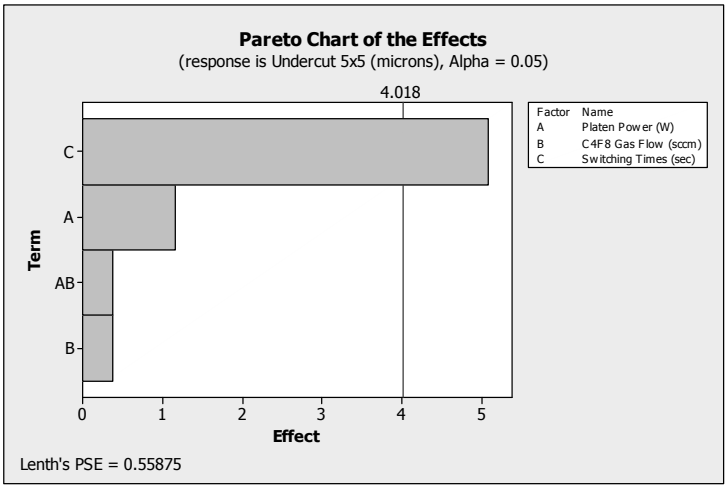


Figure 8, Pareto Chart for 5x5 μm pillars

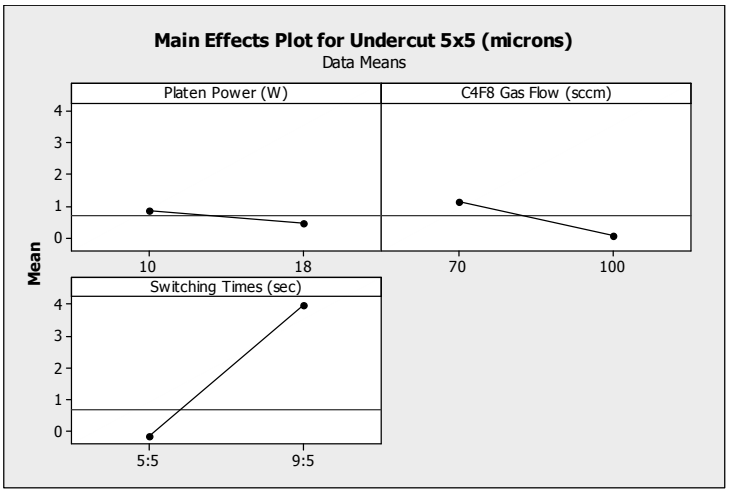


Figure 9, Main-Effects Plot for 5x5 μm pillars

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4. DISCUSSION

The 2x2 μm pillars were not successfully replicated. This is most likely due to the feature limitations of the photomask and the etching limitations of the DRIE.

For both the 5x5 μm and 10x10 μm pillars it was found that the switching time was the only statistically significant factor.

A possible mechanism for the observed significance of switching time is the rate at which the passivation layer is removed during the etching phase. The longer the etching phase the greater the silicon etch rate, which results in a more negative etch profile. Whereas for a shorter etching phase less time is available for the passivation layer to be completely removed prior to the silicon etching. This can result in an uneven etch surface and the black silicon effect.

5. CONCULSIONS

In this paper the effect of DRIE factors on feature undercut has been examined for three pillar dimensions: 2x2 μm, 5x5 μm and 10x10 μm, using undercut as the measured experimental response.

A deign of experiment approach was used to evaluated the degree of feature undercut in correlation to the process factors. It was shown that switching times was the only statistically significant factor affecting the undercut of both 5x5 μm and 10x10 μm pillars. The 2x2 μm pillars were not successfully replicated.

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