**IEE 572**
**DESIGN OF ENGINEERING EXPERIMENTS**

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# Design and Analysis study on etching rate of silicon nitride ($Si_3N_4$) layer via RIE etching process.

Project Report

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## List of Figures:

## 1..Objective of the experiment:

To maximize the etch rate in silicon nitride layer which is on top of silicon wafer using reactive ion etching with factors CF4, O2 and RF power. We would be employing the combinations of all these factors to maximize the response variable which is etch rate.

## 2..Introduction- Etching of Silicon Nitride layer on Silicon Wafer using RIE nitride etching process:

Silicon nitride is often created using the *chemical vapor deposition (CVD)* method or one of its derivatives, such as *plasma-enhanced chemical vapor deposition (PECVD)*, during the production process of semiconductor devices. Typically, this layer serves as an insulating layer for the device's construction. This substance offers a superior diffusion barrier than silicon dioxide against water molecules and sodium ions, which are two of the main causes of corrosion and instability during the manufacture of devices. Additionally, this substance is utilized in analogue chip capacitors as a dielectric between polysilicon layers. The technology for removing or patterning silicon nitride from a device has grown more relevant and crucial in tandem with the growing use of this material as a passivation layer throughout the device production process. One of the effective methods for reducing the regular usage of chemical etching agents and doing away with handling potentially harmful acids and solvents used in industry, such as HF, HN03, and alkaline solutions, is the dry etching process. Dry etching features isotropic and anisotropic etch profiles to accomplish directional etching without requiring the crystal orientation (i.e. Silicon) with superior process control and cleanliness, in addition to its simplicity in automation. In this study, silicon nitride layer on silicon substrate was etched downstream using a microwave plasma and tetrafluoromethane (CF4) with oxygen as the input gas. It can be argued that the etching mechanism for silicon utilizing CF4/O2 mixes is well understood. The Experiment of dry reactive ion etching (RIE) of silicon nitride layer under a CF4/O2 gas mixture will be reported in this.

## 3..Problem Description:

This experiment aims to examine and improve the silicon nitride (Si3N4) layer's etching rate utilizing the *Reactive Ion Etching (RIE) method*. Controlling the etching rate of silicon nitride layers, which are frequently employed in semiconductor fabrication for a variety of purposes, is essential for precision device manufacture.

The specific issue to be solved is to identify the major variables and their effects on the Si3N4 etching rate. In order to enhance the control and effectiveness of the RIE nitride etching process.

## 4..Experimental Procedure:

1. All the experiments were conducted on 1 x 1 cm2 wafers with a thickness of nitride layer range from 2400 A To 2500 A.
2. Etching process time and pressure was set constantly at 2 min and 500 mTorr, respectively. The thickness of the sample before and after the etching process was measured by Dektak XT profilometer.
3. RF Power, O2 and CF4 gas flow rate were selected as 3 main factors to determine the optimum etching rate for Si3N4 layer.

4. Table 1 below indicates the minimum and maximum values set in the JMP for each selected factor. A complete 2^3 factorial of 3 factors was designed, and a total of 8 experiments with different parameters were automatically generated by DOE to determine the effect of the selected factor.

## 5..Design Factors and Levels:

1. **CF4 Gas Flow Rates**: Variations in the flow rates of the Carbon Tetrafluoride (CF4 gas) can significantly affect the etching rate.
   Levels: Min & Max (Min: 40 sccm, Max: 50 sccm)
2. **O2 Gas Flow Rates:** Variations in the flow rates of the O2 gas can significantly affect the etching rate.
   Levels: Min & Max (Min: 5 sccm, Max: 10 sccm)
3. **RF Power**: The power supplied to the RIE system affects the plasma density and, consequently, the etching rate.
   Levels: 60W, 80W

## 6..Nuisance Factors:

1. **Pressure**: Variations in chamber pressure could affect the etching rate; therefore, it should be monitored and controlled.
   Levels: Low, Medium, High
2. **Sample Surface Preparation**: The quality of the Si3N4 layer and the cleanliness of the substrate may affect etching rate.
   Levels: Standard, High-quality, Contaminated

## 7..Factors which are constant:

Sample Material: Silicon nitride material is kept constants for all experiments.

## 8..Factors which are uncontrollable:

1. Ambient Temperature and Humidity
2. Equipment Variability
3. Operator Variability
4. Gas source Variability

## 9..Main Factors under Consideration:

After analyzing the various factors affecting the etching rate, we consider the above-mentioned factors which majorly affect the etch rate. All these factors have 2 levels as shown:

**Table 1.**

| S.No. | Factor | Level 1 | Level 2 |
|---|---|---|---|
| 1 | O2 Gas (sccm) | 5 sccm | 10 sccm |
| 2 | CF4 Gas (sccm) | 40 sccm | 50 sccm |

| | | | |
|---|---|---|---|
| 3 | RF Power (W) | 60 W | 80 W |

1) **For O2 Gas:**
   a) One important consideration is the oxygen ($O_2$) flow rate into the etching chamber. A higher etching rate may result by increasing the flow rate of $O_2$, as $O_2$ is required for the oxidation and removal of silicon (Si) and silicon nitride ($Si_3N_4$). More oxidizing species are available to aid in etching at greater $O_2$ flow rates.
   b) Oxygen Concentration: The etching rate can be affected by the partial pressure or concentration of $O_2$ in the chamber. Elevated $O_2$ concentration has the potential to accelerate silicon nitride oxidation, hence augmenting the etching rate.

2) **For CF4 Gas:**
   **a)** Fluorine Flow Rate: An important consideration is the flow rate of $CF_4$, which supplies fluorine (F) ions. Since $CF_4$ plays a crucial role in the process that aids in the removal of silicon nitride, increasing the flow rate of this compound can improve the etching rate. A faster rate of etching could result from the introduction of more reactive fluorine species into the plasma by a greater $CF_4$ flow rate.

3) **For RF Power:**
   **a)** One important variable that can have a big impact on the etching rate is the amount of RF energy that is provided to the RIE system. The etching chamber's plasma may become more intense with higher RF power levels. This could therefore lead to a more vigorous etching procedure and a greater etching rate. Ions and radicals in the plasma are accelerated by the RF power, which increases their reactivity and material removal.
   **b)** Plasma Density: Another factor influencing plasma density is radiofrequency power. By supplying the etching process with additional reactive species, a *denser plasma* can *increase the etching rate*. The etching rate may be further impacted by a denser plasma, which is a result of increased RF power levels.
   **c)** Ion Energy: The energy of the radicals and ions produced in the plasma is related to radiofrequency power. Increased RF power can produce more energetic ions, which could have an impact on the material's physical sputtering and removal during etching.

## 10..Selection and Measurement of Response Variable:

The primary response variable is the **Etching Rate (μm/min)** of the silicon nitride layer. This will be measured using Dektak XT profilometer, which are standard techniques for evaluating thin film thickness.

To ensure measurement stability and consistency:

1. Calibration of the measurement equipment will be performed before each measurement session.
2. Measurements will be conducted at the same location on each sample to minimize variability.
3. Multiple measurements will be taken at each level to calculate an average etching rate and assess measurement repeatability.

## 11..Strategy of Experimental Design:

1. **Selection of Design of Experiments (DOE) Technique**:
   A **2^3 factorial design** will be used, considering all possible combinations of the defined factors and levels. This approach would help identify main effects and potential interactions between factors.

2. **Randomization and Blocking**:
   To account for any uncontrollable variations, randomization of experimental runs will be implemented. If any nuisance factors are identified during the preliminary analysis, blocking will be employed to control their influence.

3. **Sample Size**:
   A minimum of 2 replicates at each combination of factor levels will be conducted to ensure statistically meaningful results.

4. **Data Collection**:
   The etching rate measurements will be recorded, and any other relevant observations during the experiment will be noted for future analysis.

## 12..Randomization of Table (Experimental Setup Table)

A randomization table is produced to ensure unpredictability. The experiment is conducted, and blocking was done on the controllable nuisance parameters.

1. Randomize Output:

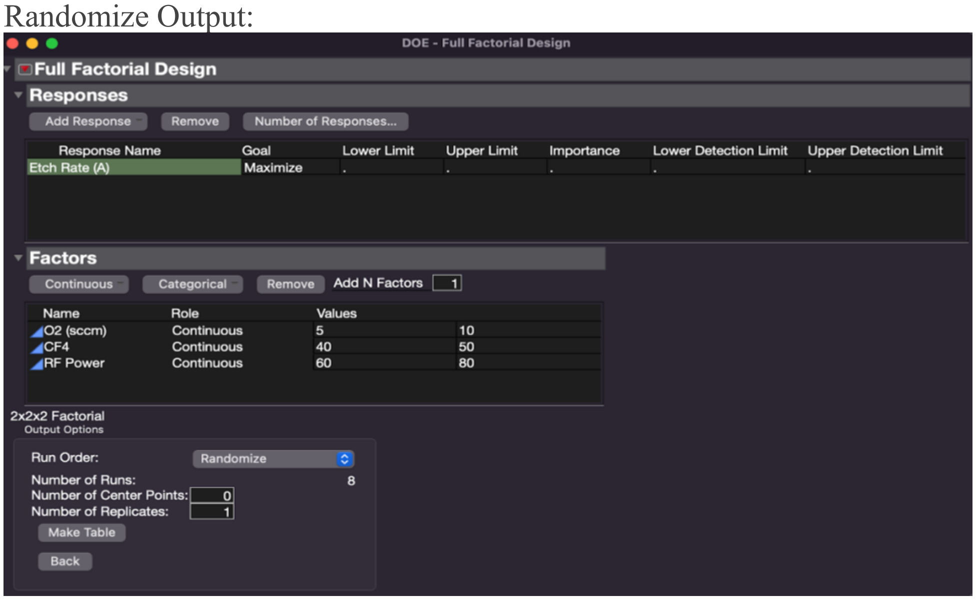
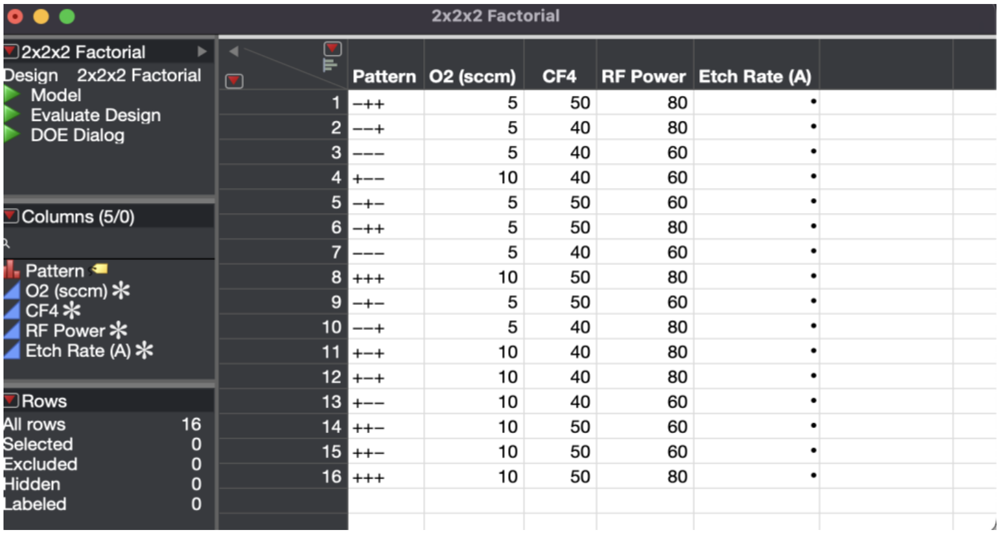



*Figure 1- Randomize run order Full Factorial Design*



| | Pattern | O2 (sccm) | CF4 | RF Power | Etch Rate (A) |
|---|---|---|---|---|---|
| 1 | −++ | 5 | 50 | 80 | • |
| 2 | −−+ | 5 | 40 | 80 | • |
| 3 | −−− | 5 | 40 | 60 | • |
| 4 | +−− | 10 | 40 | 60 | • |
| 5 | −+− | 5 | 50 | 60 | • |
| 6 | −++ | 5 | 50 | 80 | • |
| 7 | −−− | 5 | 40 | 60 | • |
| 8 | +++ | 10 | 50 | 80 | • |
| 9 | −+− | 5 | 50 | 60 | • |
| 10 | −−+ | 5 | 40 | 80 | • |
| 11 | +−+ | 10 | 40 | 80 | • |
| 12 | +−+ | 10 | 40 | 80 | • |
| 13 | +−− | 10 | 40 | 60 | • |
| 14 | ++− | 10 | 50 | 60 | • |
| 15 | ++− | 10 | 50 | 60 | • |
| 16 | +++ | 10 | 50 | 80 | • |



*Figure 2- Randomize run order Full Factorial Design- 2x2x2 Factorial Design*

2. Right to left Run order:

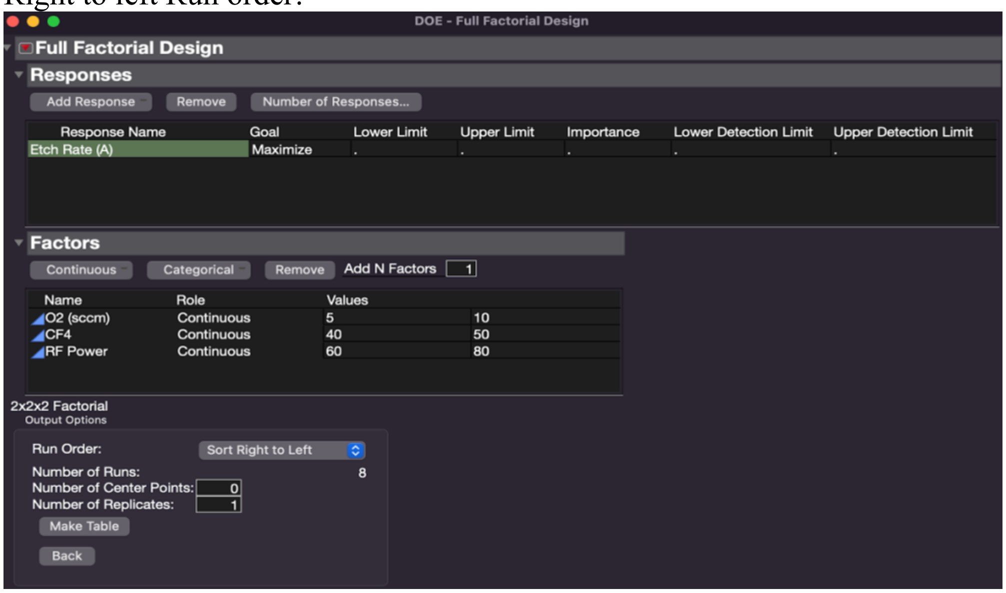
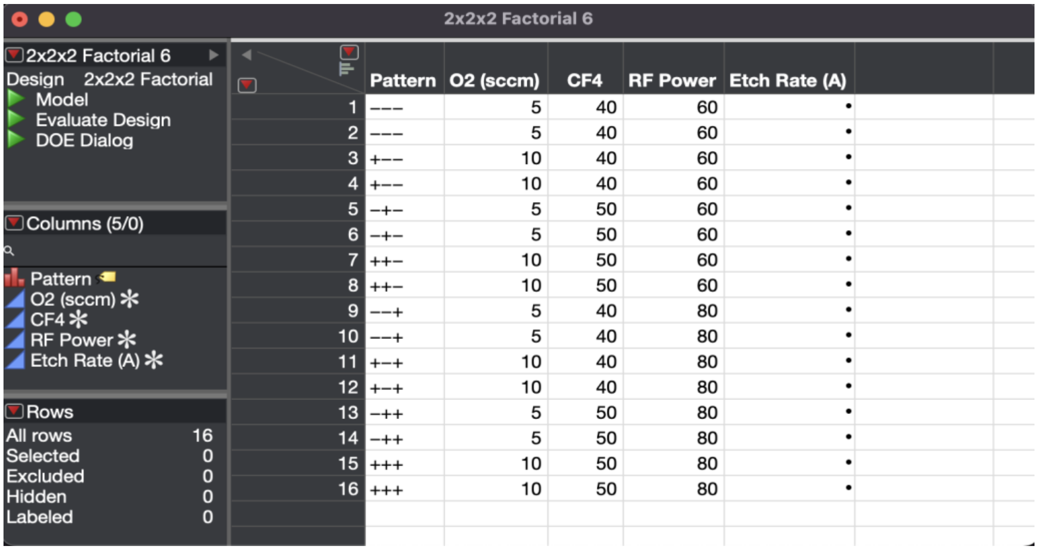



*Figure 3- Right to Left Run Order Full Factorial Design.*



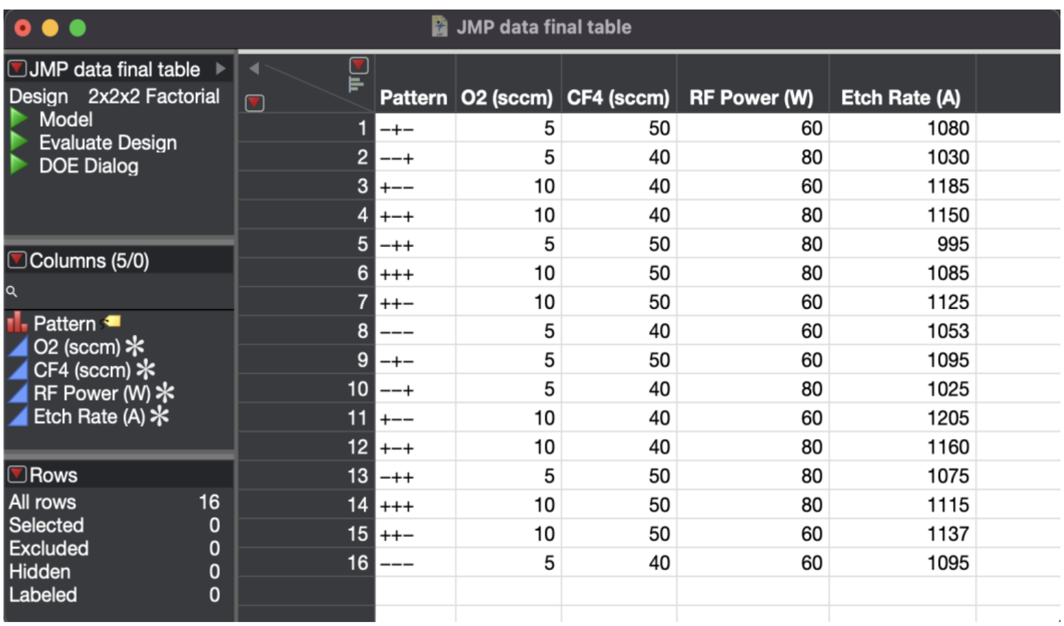


| | Pattern | O2 (sccm) | CF4 | RF Power | Etch Rate (A) |
|---|---|---|---|---|---|
| 1 | −−− | 5 | 40 | 60 | • |
| 2 | −−− | 5 | 40 | 60 | • |
| 3 | +−− | 10 | 40 | 60 | • |
| 4 | +−− | 10 | 40 | 60 | • |
| 5 | −+− | 5 | 50 | 60 | • |
| 6 | −+− | 5 | 50 | 60 | • |
| 7 | ++− | 10 | 50 | 60 | • |
| 8 | ++− | 10 | 50 | 60 | • |
| 9 | −−+ | 5 | 40 | 80 | • |
| 10 | −−+ | 5 | 40 | 80 | • |
| 11 | +−+ | 10 | 40 | 80 | • |
| 12 | +−+ | 10 | 40 | 80 | • |
| 13 | −++ | 5 | 50 | 80 | • |
| 14 | −++ | 5 | 50 | 80 | • |
| 15 | +++ | 10 | 50 | 80 | • |
| 16 | +++ | 10 | 50 | 80 | • |

*Figure 4- Right to left run order Full Factorial Design- 2x2x2 Factorial Design*

## 13..Statistical Analysis of Data

After conducting the experiments and collecting all the data, using JMP we analyze the data. The data used in JMP is shown in the below figure.





| | Pattern | O2 (sccm) | CF4 (sccm) | RF Power (W) | Etch Rate (A) |
|---|---|---|---|---|---|
| 1 | −+− | 5 | 50 | 60 | 1080 |
| 2 | −−+ | 5 | 40 | 80 | 1030 |
| 3 | +−− | 10 | 40 | 60 | 1185 |
| 4 | +−+ | 10 | 40 | 80 | 1150 |
| 5 | −++ | 5 | 50 | 80 | 995 |
| 6 | +++ | 10 | 50 | 80 | 1085 |
| 7 | ++− | 10 | 50 | 60 | 1125 |
| 8 | −−− | 5 | 40 | 60 | 1053 |
| 9 | −+− | 5 | 50 | 60 | 1095 |
| 10 | −−+ | 5 | 40 | 80 | 1025 |
| 11 | +−− | 10 | 40 | 60 | 1205 |
| 12 | +−+ | 10 | 40 | 80 | 1160 |
| 13 | −++ | 5 | 50 | 80 | 1075 |
| 14 | +++ | 10 | 50 | 80 | 1115 |
| 15 | ++− | 10 | 50 | 60 | 1137 |
| 16 | −−− | 5 | 40 | 60 | 1095 |

*Figure 5- JMP Data*

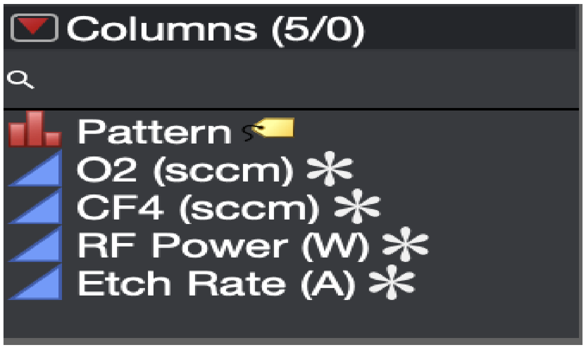



*Figure 6- Factors chosen in JMP.*

## 14..Results:

**Effect summary:**

An Effect Summary succinctly describes the primary outcomes or consequences of a phenomenon, action, or event, providing a concise overview of its impact or implications.

**Effect Summary**

| Source | LogWorth | PValue | |
|---|---|---|---|
| O2 (sccm)(5,10) | 4.015 | 0.00010 | |
| RF Power (W)(60,80) | 2.033 | 0.00926 | |
| O2 (sccm)*CF4 (sccm) | 1.639 | 0.02299 | |
| CF4 (sccm)(40,50) | 1.070 | 0.08511 | ^ |
| O2 (sccm)*RF Power (W) | 0.229 | 0.59004 | |
| O2 (sccm)*CF4 (sccm)*RF Power (W) | 0.113 | 0.77136 | |
| CF4 (sccm)*RF Power (W) | 0.021 | 0.95353 | ^ |

Remove Add Edit FDR ('^' denotes effects with containing effects above them)

**Effect Tests**

| Source | Nparm | DF | Sum of Squares | F Ratio | Prob > F |
|---|---|---|---|---|---|
| O2 (sccm)(5,10) | 1 | 1 | 31862.250 | 51.1946 | <.0001* |
| CF4 (sccm)(40,50) | 1 | 1 | 2401.000 | 3.8578 | 0.0851 |
| RF Power (W)(60,80) | 1 | 1 | 7225.000 | 11.6088 | 0.0093* |
| O2 (sccm)*CF4 (sccm) | 1 | 1 | 4900.000 | 7.8731 | 0.0230* |
| O2 (sccm)*RF Power (W) | 1 | 1 | 196.000 | 0.3149 | 0.5900 |
| CF4 (sccm)*RF Power (W) | 1 | 1 | 2.250 | 0.0036 | 0.9535 |
| O2 (sccm)*CF4 (sccm)*RF Power (W) | 1 | 1 | 56.250 | 0.0904 | 0.7714 |

*Figure 7-Effect Summary & Effect Tests*

After looking at the Effect Tests, we can see that it captures the key effects in a brief and informative manner.

**Analysis of variance:**

**Analysis of Variance**

| Source | DF | Sum of Squares | Mean Square | F Ratio |
|---|---|---|---|---|
| Model | 7 | 46642.750 | 6663.25 | 10.7062 |
| Error | 8 | 4979.000 | 622.38 | Prob > F |
| C. Total | 15 | 51621.750 | | 0.0017* |

*Figure 8- Analysis of variance*

**Summary of Fit**

| | |
|---|---|
| RSquare | 0.903548 |
| RSquare Adj | 0.819153 |
| Root Mean Square Error | 24.94744 |
| Mean of Response | 1100.625 |
| Observations (or Sum Wgts) | 16 |

*Figure 9- Summary of fit*

**Actual Predicted Plot:**

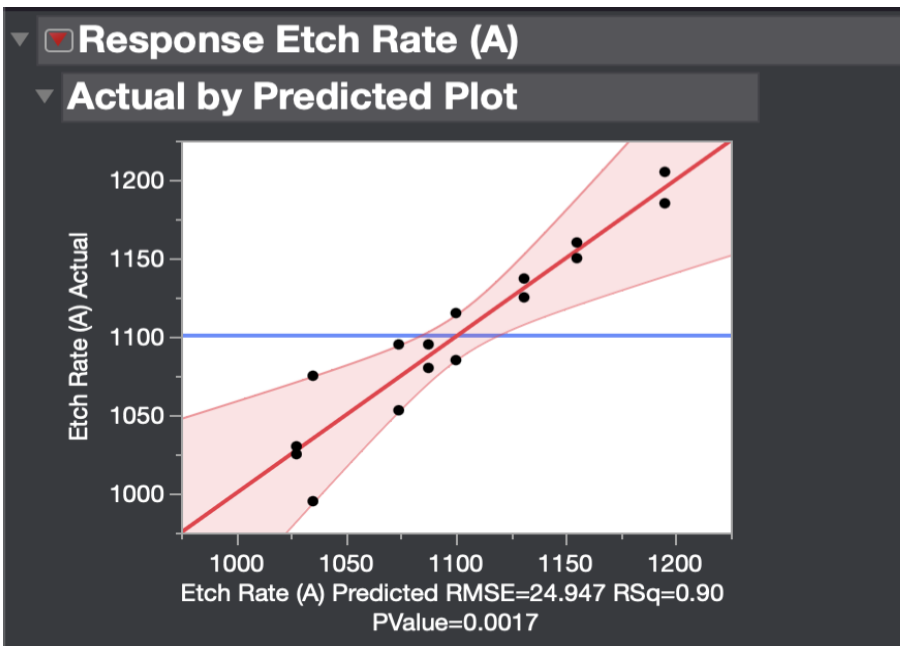



*Figure 10- Actual Predicted Plot*

After looking through the graph we can assess the relationship between actual and predicted values of Etch Rate. The graph shows that there's a strong linear relationship between the predicted and actual etch rates, with the predictions being quite accurate given the high R-squared value and the low p-value.

**Residual Against Predicted plot:**

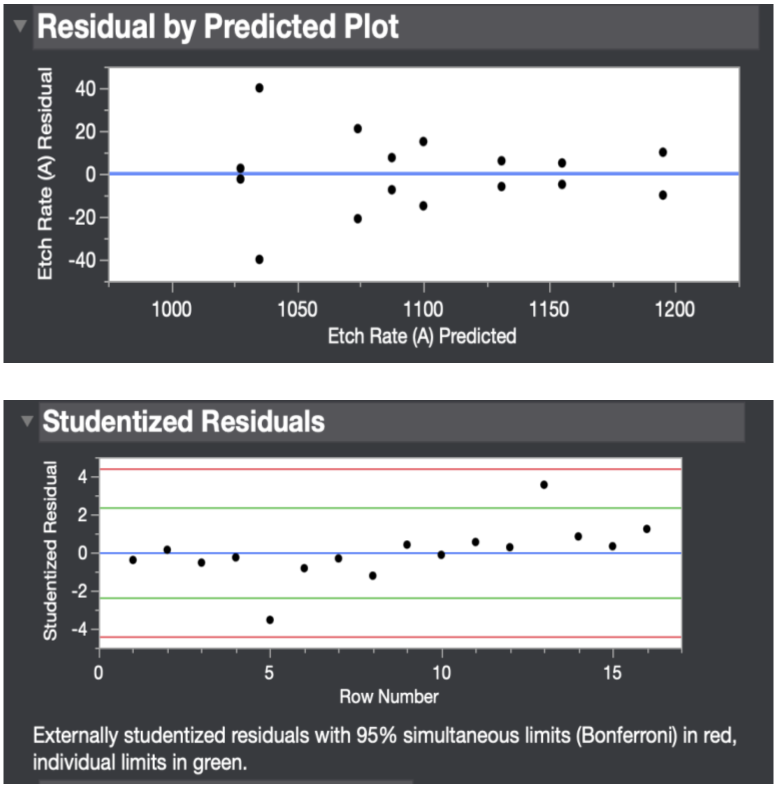



*Figure 11-Residual by predicted Plot and Studentized Residuals*

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The above is a plot of the residuals versus the ascending predicted response values.

**Residual normal Quantile plot:**

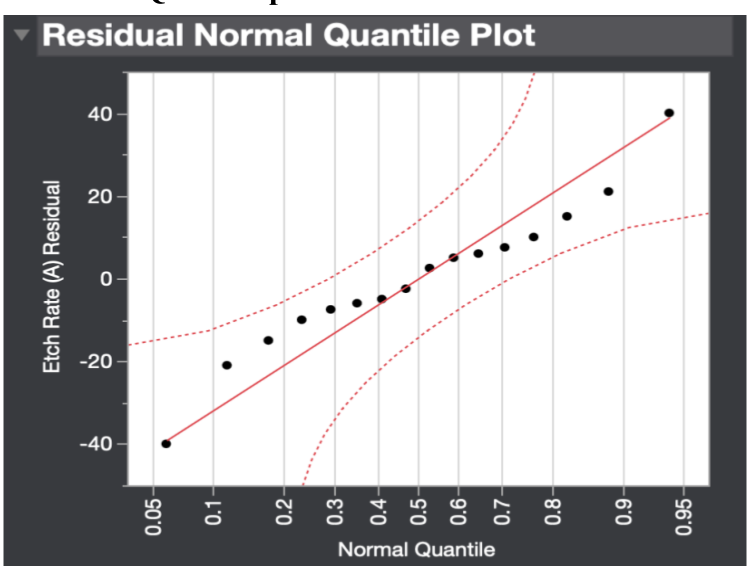


*Figure 12- Residual normal Quantile plot*

**Prediction Profiler:**

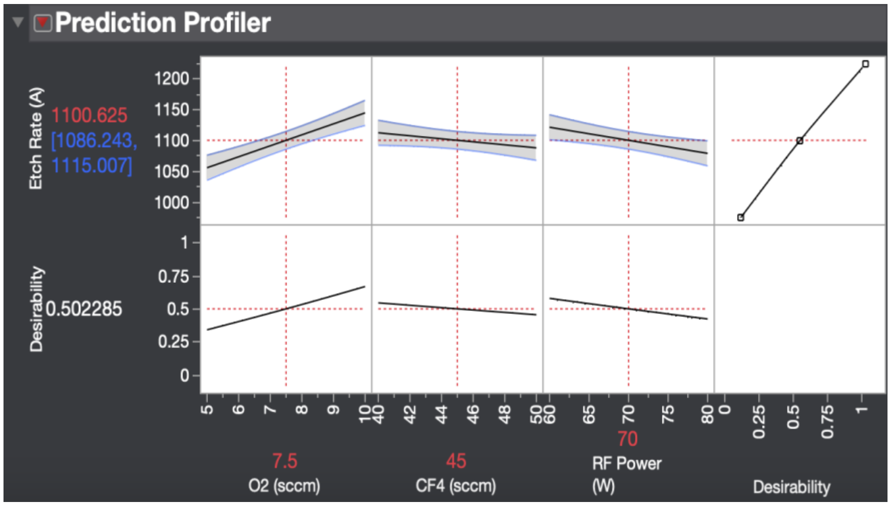


*Figure 13- Prediction profiler Graph*

The prediction profiler's aim is to display all the connections between various causes and various answers. This is achieved through using a matrix of plots, where the number of rows represents the number of responses, and the number of columns represents the number of factors.

**Parameter Estimates**

| Term | Estimate | Std Error | t Ratio | Prob>\|t\| |
|---|---|---|---|---|
| Intercept | 1100.625 | 6.236861 | 176.47 | <.0001* |
| O2 (sccm)(5,10) | 44.625 | 6.236861 | 7.16 | <.0001* |
| CF4 (sccm)(40,50) | -12.25 | 6.236861 | -1.96 | 0.0851 |
| RF Power (W)(60,80) | -21.25 | 6.236861 | -3.41 | 0.0093* |
| O2 (sccm)*CF4 (sccm) | -17.5 | 6.236861 | -2.81 | 0.0230* |
| O2 (sccm)*RF Power (W) | 3.5 | 6.236861 | 0.56 | 0.5900 |
| CF4 (sccm)*RF Power (W) | 0.375 | 6.236861 | 0.06 | 0.9535 |
| O2 (sccm)*CF4 (sccm)*RF Power (W) | 1.875 | 6.236861 | 0.30 | 0.7714 |

*Figure 14- Parameter Estimation*

**Normal Plot:**

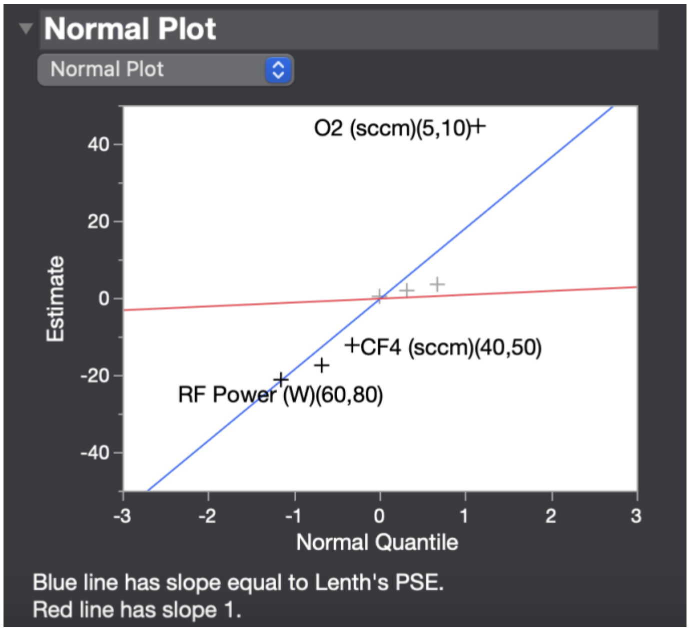


*Figure 15- Normal Plot*

## 15..Conclusion:

In conclusion, this project has successfully investigated the etching process of silicon nitride, employing a systematic approach through Design of Experiment (DOE). The Project focused on evaluating the influence of key factors, namely O2, CF4, and RF power, on the silicon nitride etching process. Notably, the experimental results show the substantial **impact** of **O2 in enhancing the etching rate** of the silicon nitride layer.

Based on the comprehensive analysis, a set of optimized parameters, it has been proposed for silicon nitride with thicknesses ranging from 1000 to 3000 angstroms. This optimal parameter set includes an RF power of 60W and a specific CF4 at 40 sccm and O2 at 10 sccm. The rationale behind this proposal is twofold: it achieves effective silicon nitride etching and does so with a **relatively low consumption of RF power**. Furthermore, the specified gas flow ratio ensures a balanced and efficient mixture of O2 and CF4.