Development of Silicon Nitride Etch Process

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Abstract—Silicon nitride has been extensively used as a hard mask for the local oxidation of silicon (LOCOS) for the processing of CMOS devices. Plasma etching is an ideal etch process for patterning the silicon nitride. An optimized etch process was developed for silicon nitride using the LAM-490 plasma etcher. A designed experiment was created, executed, and analyzed. The parameters investigated were SF_6 flow, He flow, pressure, power, and electrode gap. The responses examined were silicon nitride uniformity, silicon nitride etch rate, silicon nitride to oxide selectivity and silicon nitride to photoresist selectivity. Through analysis of the experimental results optimal factor settings were determined. A recipe was created on the LAM-490 with the optimal factor settings and endpoint detection was incorporated into the optimal recipe.

1. BACKGROUND

Silicon nitride has been extensively used in the processing of CMOS devices for passivation layers and LOCOS. The LAM-490 plasma etcher was recently purchased from Xerox and will be used to etch Silicon Nitride and Polysilicon. The Gaseous Electronic Conference (GEC) Plasma Cell has been replaced with the LAM-490. The GEC Plasma Cell had one chamber that used parallel plate electrodes for reactive ion etching (RIE). RIE is anisotropic due to the directionality of the ions. Currently silicon nitride etching is being done using the DryTek Quad. The DryTek Quad has four chambers with a common loadlock and is a RIE process. Unlike the DryTek Quad, the LAM-490 is fully automated. The LAM-490 is easier to use and faster than the DryTek Quad. The LAM-490 also has endpoint detection. Currently the DryTek Quad does not have endpoint detection, therefore the etch time is calculated using previous etch rates. A post-etch verification is done by measuring the oxide thickness in the etched areas. The oxide thickness is measured to determine if the wafer was over-etched or under-etched. Using the LAM-490 will allow for endpoint detection and eliminate over- and under-etching. A process needs to be developed to etch silicon nitride for sub-micron CMOS devices using the LAM-490. There are many variables that need to be considered in developing and optimizing an etch process. Due to the complexity of etch tools an etch recipe cannot be transferred from one etch tool to another. A new recipe needs to be developed.

The LAM-490 is a fully automated, plasma dry etch tool that will be used to etch silicon nitride and polysilicon. The LAM-490 allows up to 15 steps for each etch recipe. The recipes that will be used by students will contain approximately five steps. The five steps include stabilization, main etch, over-etch, reset, and recipe end. The stabilization step stabilizes the pressure, gas flow, and electrode gap before the RF power is turned on and etching begins. The RF power is turned on and a plasma is ignited in the main etch step. The over-etch step is used to complete the etching when endpoint detection is used for the main etch step. The reset step sets the parameters back to their idle mode settings and stabilizes. The fifth step, recipe end, ends the recipe.

Silicon nitride will be etched using SF_6 and He. Helium is used to help in ionization and help in the ignition of the plasma. The fluorine reacts will silicon to form silicon tetra-fluoride. The balanced chemical reaction is shown below.

$$6\ SF_6 + Si_3N_4 \rightarrow 3SiF_4 + 6SF_4 + 2N_2$$

A central composite design was used to develop and optimize a silicon nitride etch process on the LAM-490. There are many important etch parameters including pressure, power, gap, SF_6 flow, and He flow. There are also many responses that can be analyzed including etch rate, uniformity, selectivity, and sidewall profile. An optimal etch rate is one that is not too fast or too slow. If the etch rate is too fast then it is uncontrollable and it will lead to poor endpoint detection. If the etch rate is too slow then throughput will be low. For an optimized process the best uniformity should be chosen, in this case less than 5% non-uniformity is desired. High silicon nitride to oxide selectivity and high silicon nitride to photoresist selectivity are desired.

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Endpoint detection is an integral part of silicon nitride etching. Dry plasma etching has poor selectivity of silicon nitride to oxide. Therefore oxide is not a good stopping layer for silicon nitride since SF_6 will also etch oxide. To prevent etching through the underlying oxide, optical emission endpoint detection is used for silicon nitride etching.

2. PROCEDURE

A. Wafer Preparation

Starting with bare silicon substrates a 2000Å thermal oxide layer was grown. Then a 3500Å layer of silicon nitride was deposited using low pressure chemical vapor deposition. The wafers were then coated with photoresist and half of each wafer was exposed. The wafers were then developed, removing the photoresist from half of the wafer. The nitride exposed on half of each wafer was then etched down to the oxide. The photoresist was ashed. Then the wafers were patterned again using the CMOS submicron active mask, resulting in photoresist, oxide, and silicon nitride being exposed to the surface of the wafer. The CMOS submicron active mask is used for LOCOS in RIT's submicron CMOS process. It is used to pattern the silicon nitride before the field oxide growth. After the lithography was completed the wafers were ready to be used in the actual experiment.

B. Execution of Experimental Design

A central composite design was designed and executed. This design varied 5 factors: pressure, power, gap, SF_6 flow, He flow. The experiment consisted of 32 treatment combinations. Each factor contains five factor settings. The factor settings are shown in table 1 below.

Table 1: Factor Settings

Factor	- Star Point	-1	Zero point	+1	+ Star Point
Pressure (mT)	260	285	310	335	360
Power (W)	125	150	175	200	225
Gap (cm)	0.65	0.9	1.15	1.4	1.65
SF_6 Flow (sccm)	60	95	130	165	200
He Flow (sccm)	0	25	50	75	100

Nitride, oxide, and photoresist thickness measurements were taken prior to etching using reflection spectroscopy (Nanospec AFT). The wafers were then etched as specified by the central composite designed experiment. The nitride, oxide, and photoresist thickness were measured again and the etch rates, uniformity, and selectivity were calculated.

C. Endpoint Detection

The optimized factor settings obtained from the experimental analysis of the central composite design were used to set up endpoint detection. Three wafers were etched using the optimized recipe, to determine the endpoint parameters needed on the LAM-490. The parameters needed are delay time, time period to normalize, and % of normalized value to trigger the endpoint. The incorporation of endpoint detection is the final step in this project.

3. ANALYSIS

The data was analyzed using JMP software and optimal factor settings were determined. Nitride uniformity was the first response examined. The analysis shows that the only significant effects on nitride uniformity are He flow and power. A model equation was obtained from the analysis and is equation 1 below.

$$Y = 6.89 + 1.52(Power) + 1.45(He)$$

From the analysis it was found that the best uniformity was obtained when the Power was set to 125 W and the He flow was set to 0 sccm. As the power increased and as helium flow increased the uniformity decreased. This is illustrated in figure 1 below.

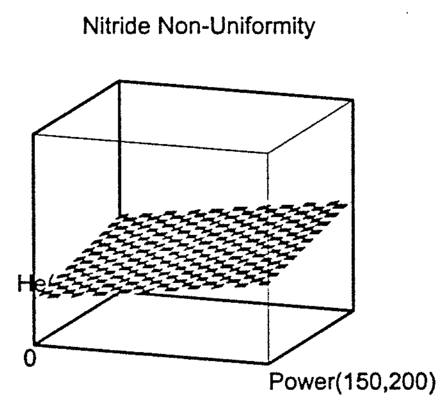

Figure 1: The effects of He and Power on Nitride Non-Uniformity

The nitride etch rate was analyzed using the optimal etch parameters for He flow and power determined from the nitride uniformity analysis. It was found that there were many significant effects and interactions on the nitride etch rate. The model equation obtained for the nitride etch rate is quite extensive and can be found in the appendix. What should be noted is that the gap does not have a significant effect on nitride etch rate. Pressure and SF_6 flow were found to have a significant effect on nitride etch rate and the effect is illustrated in figure 2 below. As the pressure increases the etch rate decreases and as the SF_6 flow increases the etch rate increases. From this analysis it was determined that the optimal setting for pressure is 260 mTorr and the optimal setting for SF_6 flow is 200 sccm. These settings are at the edge of the design space, however the design space was determined from preliminary experimentation and investigation on the tool limitations. It was found that for an SF_6 flow of 200 sccm the lowest pressure that can be achieved is 260 mTorr.

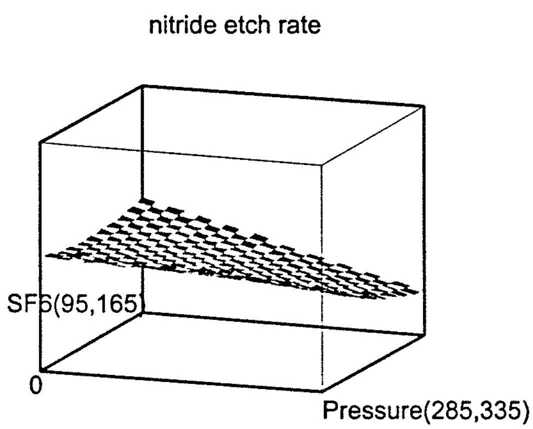

Figure 2: The effects of SF6 and Pressure on Nitride Etch Rate

The nitride to oxide selectivity was analyzed using the optimal etch parameters for He flow and power determined from the nitride uniformity analysis and the optimal etch parameters for pressure and SF_6 flow determined from the nitride etch rate analysis. It was found that there are many significant effects and interactions on the nitride to oxide selectivity. The model equation is quite extensive and can be found in the appendix. The gap is the only factor that did not have a significant effect on the nitride uniformity or the nitride etch rate. It was found from the analysis of nitride to oxide selectivity that as the gap increased the nitride to oxide selectivity also increased. This is illustrated in figure 3 below.

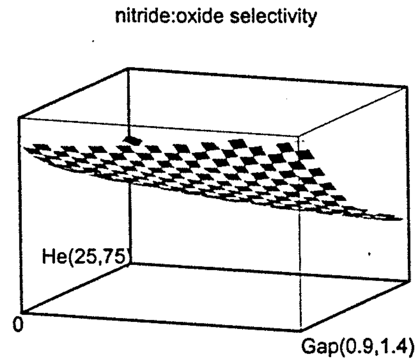

Figure 3: The effects of He and Gap on Nitride:Oxide Selectivity

From the analysis of oxide to photoresist selectivity it was found that there were many significant effects and interactions. However, using the optimal factor settings found from the analysis of nitride uniformity, nitride etch rate, and nitride to oxide selectivity gave a nitride to photoresist selectivity of 0.61. A nitride to photoresist selectivity of 0.61 is adequate for this process since the photoresist thickness is at least three times the nitride thickness.

The optimal factor settings are shown in table 2 below.

Table 2: Optimal Factor Settings

Factor	Setting
Pressure	260 mTorr
Power	125 Watts
Gap	1.65 cm
SF6 Flow	200 sccm
He Flow	0 sccm

The optimal factor settings determined from this analysis were used in the model equations to determine the responses shown in table 3 below.

Table 3: Responses at Optimal Factor Settings.

Responses	
Nitride Etch Rate	1555 A/min
Nitride Uniformity	2.44%
Nitride:Oxide Selectivity	3.06
Nitride:PR Selectivity	0.61

Once the optimized process had been determined optical endpoint detection was incorporated into the optimized recipe. There is a delay time of 10 seconds and then the intensity signal is normalized over the next 20 seconds. When the intensity falls to 90% of the normalized value the endpoint is triggered. There is an over-etch step of 20% of the etch time to reach endpoint. The over-etch step ensures that all the nitride is removed from the wafer.

4. CONCLUSIONS

An experiment was designed, executed, and analyzed to determine the optimal factor settings for a silicon nitride etch process on the LAM-490. From the analysis it was found that as He flow increases and as power increases silicon nitride uniformity decreases. It was also found that as pressure decreases and SF_6 flow increases silicon nitride etch rate increases. When the He flow is set to zero and the electrode gap increases the silicon nitride to oxide selectivity increases. The responses determined from the optimal factor settings are a silicon nitride etch rate of 1535 A/min, silicon nitride non-uniformity of 2.44%, silicon nitride to oxide selectivity of 3.06 and silicon nitride to photoresist selectivity of 0.61. Endpoint detection has been incorporated into the optimal etch recipe. As a result of this experiment, students will be exposed to an automated etch tool.

5. APPENDIX

Table 4: Model Equation for Nitride Etch Rate:

	Coefficient
Constant	1705.62
Pressure	-220.875
Power	377.75
SF6	-207
He	263.375
Pressure*Power	-73.3125
Pressure*SF6	-87
Power*SF6	-103.125
Pressure*He	80
Power*He	115.31
SF6*He	79.875
Power*Power	67.639
SF6*SF6	51.701

Table 5: Model Equation for Nitride to Oxide Selectivity:

	Coefficient
Constant	1.956
Pressure	0.1358
Power	-0.079
Gap	0.0175
SF6	0.0808
He	-0.2292
Pressure*Gap	0.11
Power*He	0.10875
Gap*He	0.0825
Power*Power	0.1278
He*He	0.1053

Table 6: Model Equation for Nitride to Photoresist Selectivity:

	Coefficient
Constant	0.705
Pressure	-0.0217
Power	0.00583
Gap	0.0092
SF6	-0.035
He	0.0192
Pressure*Gap	0.03375
Power*He	0.02125
SF6*He	0.03875
Power*Power	0.03

Table 7: Final Optimized Etch Recipe for Sub-micron CMOS LOCOS:

	Step #1	Step #2	Step #3	Step #4	Step #5
Pressure (mTorr)	260	260	200	0	0
RF Top (W)	0	125	75	0	0
Gap (cm)	1.65	1.65	1.65	1.35	1.35
CF_4 (sccm)	0	0	0	0	0
Oxygen (sccm)	0	0	5	0	0
Helium (sccm)	0	0	0	0	0
SF_6 (sccm)	200	200	40	0	0
Oxygen (sccm)	0	0	0	0	0
Compl	Stability or time	Endpoint or time	Over-etch	Time	Recipe
max	30 sec.	3 min.	20%	10 sec.	-------

Table 8: Experimental Data

tc	Pattern	Pressure (mTorr)	Power (W)	Gap (cm)	SF6 (sccm)	He (sccm)	nitride etch rate (A/min)	nitride uniformity	oxide etch rate (A/min)	PR etch rate (A/min)	Nitride:Oxide Selectivity	Nitride:PR Selectivity
1	-+-+-	285	200	0.9	165	25	1872	3.74	927.9	2874	2.02	0.65
2	00a00	310	175	0.65	130	50	1639.8	3.89	706.2	2353.2	2.32	0.70
3	0A000	310	225	1.15	130	50	2791.5	5.95	1213.8	3299.4	2.30	0.85
4	00A00	310	175	1.65	130	50	1792.5	3.44	975.3	2678.7	1.84	0.67
5	0	310	175	1.15	130	50	1807.5	3.56	921	2547.6	1.96	0.71
6	000a0	310	175	1.15	60	50	2368.5	12.57	1301.4	2979.3	1.82	0.79
7	+++-+	335	200	1.4	95	75	2706	15.34	1362.9	3365.4	1.99	0.80
8	-+--+	285	200	0.9	95	75	3085.5	4.67	1535.1	3752.4	2.01	0.82
9	-++--	285	200	1.4	95	25	2334	11.95	1205.4	3077.4	1.94	0.76
10	0	310	175	1.15	130	50	1800	3.96	945.3	2541.6	1.90	0.71
11	a0000	260	175	1.15	130	50	2161.5	8.36	1201.2	2805	1.80	0.77
12	+-+++	335	150	1.4	165	75	1537.5	3.83	651.9	2038.8	2.36	0.75
13	---++	285	150	0.9	165	75	1680	3.47	864.6	2222.4	1.94	0.76
14	000A0	310	175	1.15	200	50	1651.5	5.75	804.6	2397.3	2.05	0.69
15	0a000	310	125	1.15	130	50	1356	6.13	512.1	1658.4	2.65	0.82
16	0	310	175	1.15	130	50	1720.5	13.78	912	2354.4	1.89	0.73
17	++---	335	200	0.9	95	25	1824	6.88	879.3	2591.7	2.07	0.70
18	0000a	310	175	1.15	130	0	1408.5	2.29	479.1	2070.3	2.94	0.68
19	0	310	175	1.15	130	50	1906.5	21.44	926.4	2500.8	2.06	0.76
20	+---+	335	150	0.9	95	75	1513.5	7.88	813	2160.6	1.86	0.70
21	--++-	285	150	1.4	165	25	1362	3.42	538.2	1932.6	2.53	0.70
22	++-++	335	200	0.9	165	75	1993.5	10.33	950.4	2757.6	2.10	0.72
23	0	310	175	1.15	130	50	1558.5	6.44	887.7	2359.8	1.76	0.66
24	0	310	175	1.15	130	50	1633.5	4.67	878.4	2458.5	1.86	0.66
25	A0000	360	175	1.15	130	50	1426.5	3.4	633.9	2111.1	2.25	0.68
26	-++++	285	200	1.4	165	75	2613	12.79	1331.4	3335.4	1.96	0.78
27	+--+-	335	150	0.9	165	25	505.5	2.97	200.4	989.1	2.52	0.51
28	+-+--	335	150	1.4	95	25	1335	5.44	449.7	1585.2	2.97	0.84
29	--+-+	285	150	1.4	95	75	1647	5.21	1007.7	2348.1	1.63	0.70
30	0000A	310	175	1.15	130	100	2002.5	9.04	1092	2763.6	1.83	0.72
31	++++-	335	200	1.4	165	25	879	5.56	314.1	1342.2	2.80	0.65
32	-----	285	150	0.9	95	25	1531.5	2.3	671.7	1821.9	2.28	0.84

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