

# **PPPL Summer Internship Project Report (Interim):**

## **Re-optimization of Deposition Parameters of Silicon-nitride Films**

### **Prepared by Plasma-Enhanced-Chemical-Vapor-Deposition Process**

#### **Introduction**

Silicon-nitride films are important in the fabrication of semiconductor microelectronic devices as an inter-level dielectric and for the final passivation of integrated circuits because of their excellent barrier property to moisture, metal ions and oxygen. A low-temperature (300 °C – 400°C) deposition process of silicon-nitride is required in order to be compatible with low-melting-point metals such as aluminum. For such applications, Plasma-Enhanced-Chemical-Vapor-Deposition (PECVD) is the deposition method of choice as nitride films can be deposited at temperatures below 400°C.

The silicon-nitride films that result from PECVD tend to be non-stoichiometric and they have a substantial hydrogen concentration. The physical properties of the  $\text{SiN}_x$  : H are largely determined by the stoichiometry (Si/N ratio), the hydrogen content in the film, and whether the hydrogen is predominantly bonded to the Si or the N, all of which may vary widely with the gas mixture and deposition conditions used. A slightly nitrogen-rich  $\text{SiN}_x$  gives better thin-film-transistor (TFT) characteristics and is more stable than the silicon-rich  $\text{SiN}_x$ . It is also reported that film density decreases and the dissolution rate in buffered HF increases as hydrogen content increases. Therefore, the films are preferably nitrogen-rich with a Si/N ratio ideally of 3/4, low hydrogen content, and the larger fraction of the hydrogen bound in N-H groups.

A PECVD system, Plasma-Therm / 790, is installed in the POEM Micro/Nanofabrication Facility (the “Clean Room”). The system recently was upgraded. In consequence the parameters for the deposition of best-quality films of silicon nitride have changed. Gas flow rates, gas pressure, RF power and substrate temperature will need to be re-optimized. This is to be done by running series of depositions coupled with film evaluation.

#### **Deposition Recipes**

- Original Recipe

The original recipe, which was optimized for the system before the upgrade, is saved in the file FSiN900.prc on the PECVD computer. During the deposition, the ratio of the gas-flow rate,  $\text{N}_2$  :  $\text{SiH}_4$  :  $\text{NH}_3$ , is 150 : 120 : 2 sccm. The substrate temperature is 250 °C, pressure is 900 mTorr, and the RF power is 20 W. See appendix A.

- New Recipe Series

New recipes are modified from the old recipe by varying one parameter at a time, while keeping everything else the same. The pressure, RF power and  $\text{N}_2$  and  $\text{NH}_3$  gas-flow rates

will be varied individually in 4 series; the substrate temperature and SiH<sub>4</sub>-flow rate will be fixed at 250 °C and 110 Sccm, respectively.

i. Pressure series:

Recipes	Pressure (mTorr)	Power (Watt)	N <sub>2</sub> (Sccm)	SiH <sub>4</sub> (Sccm)	NH <sub>3</sub> (Sccm)	deposition time (min)
FSIN900	900	20	150	110	2	60
FSIN700	700	20	150	110	2	60
FSIN500	500	20	150	100	2	60
FSIN300	300	20	150	100	2	120

ii. Power series

Recipes	Pressure (mTorr)	Power (Watt)	N <sub>2</sub> (Sccm)	SiH <sub>4</sub> (Sccm)	NH <sub>3</sub> (Sccm)	deposition time (min)
FSIN900	900	20	150	110	2	60
FSIN30W	700	30	150	110	2	100
FSIN10W	700	10	150	100	2	130
FSIN5W	700	5	150	100	2	120

iii. N<sub>2</sub> flow series

Recipes	Pressure (mTorr)	Power (Watt)	N <sub>2</sub> (Sccm)	SiH <sub>4</sub> (Sccm)	NH <sub>3</sub> (Sccm)	deposition time (min)
FSIN900	900	20	150	110	2	60
FSINN200	700	20	200	110	2	120
FSINN100	700	20	100	100	2	110
FSINN50	700	20	50	100	2	120

iv. NH<sub>3</sub> flow series

Recipes	Pressure (mTorr)	Power (Watt)	N <sub>2</sub> (Sccm)	SiH <sub>4</sub> (Sccm)	NH <sub>3</sub> (Sccm)	deposition time (min)
FSIN900	900	20	150	110	2	60
FSINN0	700	20	150	110	0	60
FSINN2	700	20	150	110	2	60
FSINN5	700	20	150	100	5	60

## Film Evaluations

Silicon-nitride films of each recipe are deposited on silicon, glass slides and chromium-coated glass slides. The methods we use for film characterization include: inspection of the color of the films deposited on the glass slides, analysis of etch-rate in buffered-HF, and ellipsometry to determine the refractive index.

a. Color inspection

The color of the films deposited on the glass slides is a rough indication of the Si/N ratio, since the silicon content affects the refractive index and hence the color of the film. The higher the silicon content, the more yellowish and less transparent the film looks. This is a quick qualitative way of assessing the Si/N ratio, although the exact proportion has to be determined from the ellipsometry experiments.

b. Etch-rate analysis

The etch-rate of the films in buffered-HF is related to film density and the hydrogen content. This analysis is performed on the films deposited on the chromium-coated glass slides, because chromium protects the glass substrate being dissolved in HF.

Each chromium-coated glass slide is cut into small pieces, after the deposition. Four or five lines of photoresist are painted on each piece. These pieces are submerged in 1:6 buffered-HF for different durations. The surface profiles of the pieces are then analyzed with the surface-profiler, after stripping off the photoresist. The step-height of the lines and their time-dependence are included in Appendix B.

c. Ellipsometry

To be done...

d. Electrical properties analysis

This is a last resort, due to its complexity. We may not need to do this, depending on the results of other methods.

**Results**

Pressure series:

- Color inspection:	transparent				yellow	→
- Pressure:	300	500	700	900	mTorr	
- Etch rate	2099	1754	1650	1570	Ang/min	
	←					
	fast etch			slow etch		

Power series:

- Color inspection:	transparent				yellow	→
- RF power:	5	10	20	30	Watt	
- Etch rate	2147	2310	1876	1794	Ang/min	
	←					
	fast etch			slow etch		

N<sub>2</sub>-flow series:

(samples ready, analysis to be done)

NH<sub>3</sub>-flow series:

(samples need to be prepared and analysis needs to be done)

**Conclusion**

## Appendix A Original Recipe

FSiN900.prc

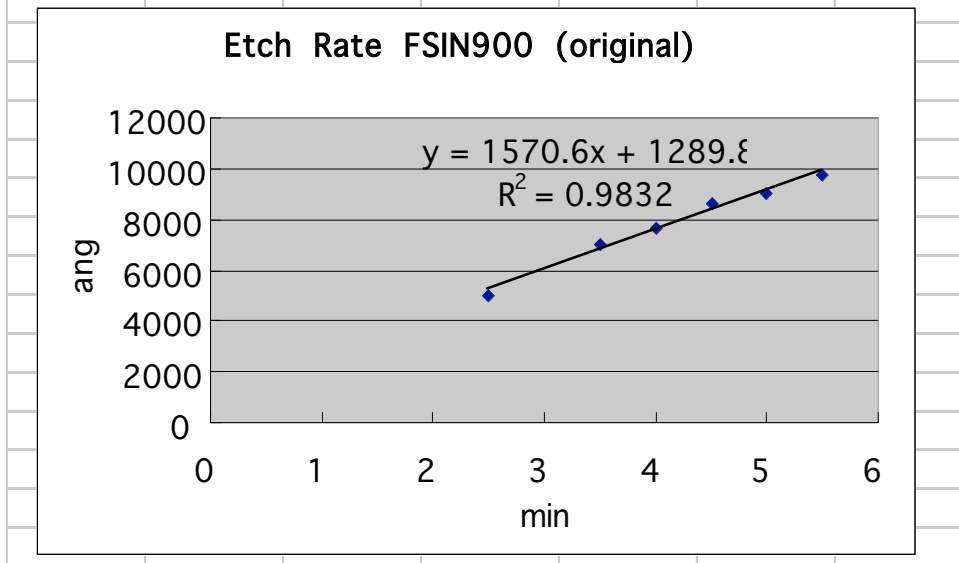
1. Initial: Hold time 5 min
2. Ar flow: 30 sec, 500 mTorr, 0 Watt, Ar 50 sccm
3. Ar plasma: 3 min, 500 mTorr, 10 Watt, Ar 50 sccm
4. N<sub>2</sub> preflow: 30 sec, 500 mTorr, 0 Watt, N<sub>2</sub> 200 sccm
5. Preflow gases: 30 sec, 900 mTorr, 0 Watt, N<sub>2</sub> : SiH<sub>4</sub> : NH<sub>3</sub> =150:120:2
6. deposit SiNx: 60 min, 900 mTorr, 20 Watt, N<sub>2</sub> : SiH<sub>4</sub> : NH<sub>3</sub> =150:120:2
7. N<sub>2</sub> chamber purge: 60 min, 900 mTorr, 0 Watt N<sub>2</sub> 200
8. End

**Appendix B Etch-rate Analysis (Incomplete yet)**  
**Step-height of the lines and their time-dependence (etch-rate)**

The step-heights are measured in ang and time in min. The unit for etch-rate is ang/min.

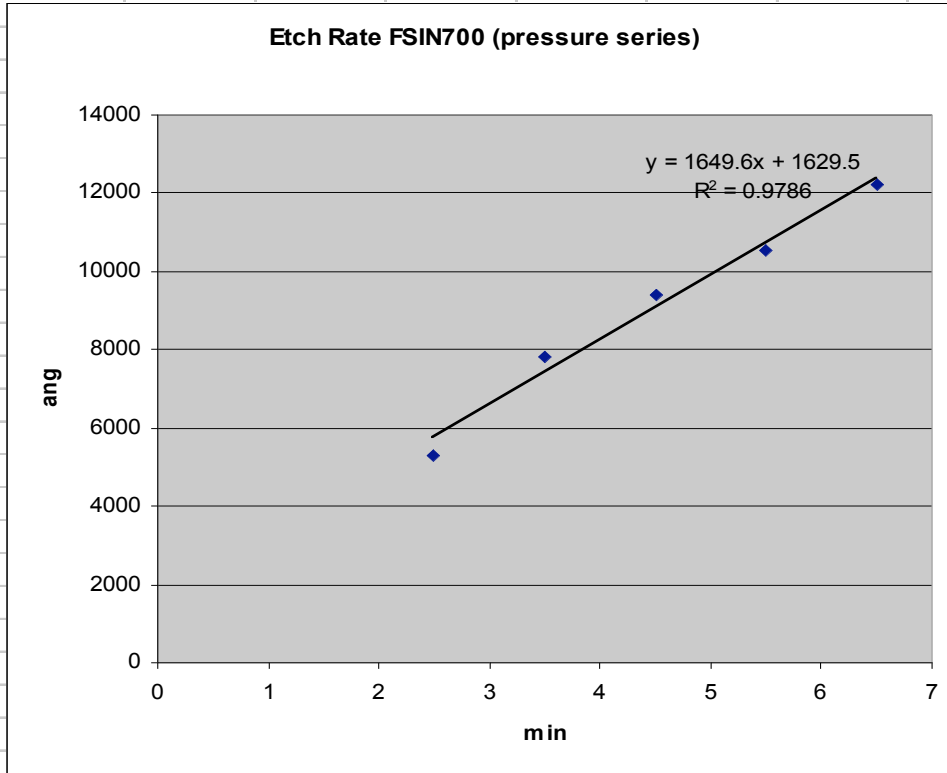
FSIN900

number	line1	line2	line3	line4	time	average
O	10670	10740	10750	10720	6	1072
A	9107	9143	9001	8864	5	9028.7
B	9659	9720	9707	9834	5.5	973
C	7580	7582	7665	7747	4	7643.
D	7039	7086	6901	6981	3.5	7001.7
E	4925	4970	5003	4956	2.5	4963.
F	8514	8517	8630	8885	4.5	8636.



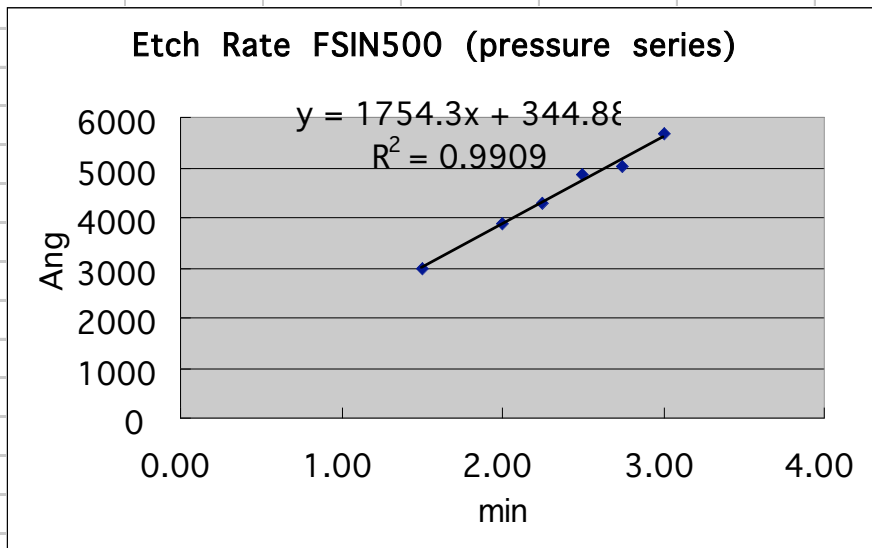
FSIN700

number	line1	line2	line3	line4	time	average
O	13100	12920	13020	13060	7	13025
A	12340	12300	12220	11920	6.5	12195
B	10440	10460	10560	10630	5.5	10523
C	9357	9194	9546	9576	4.5	9418
D	7740	7859	7836	7916	3.5	7838
E	5090	5398	5385	5285	2.5	5290



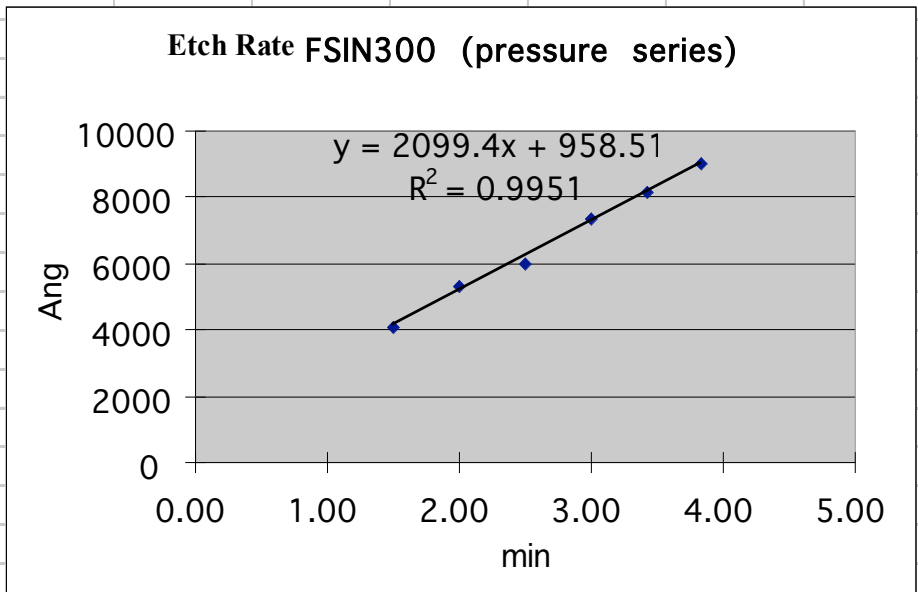
FSIN500

number	line1	line2	line3	line4	min	average
D	5753	5642	5780	5676	3.17	5713
B	4884	4856	4816	4799	2.50	4839
C	3859	3816	3906	3857	2.00	3860
E	5030	5031	5050	4938	2.75	5012
F	4312	4277	4306	4190	2.25	4271
G	2980	2955	2938	3018	1.50	2973
A_	5725	5640	5687	5647	3.00	5675



FSIN300

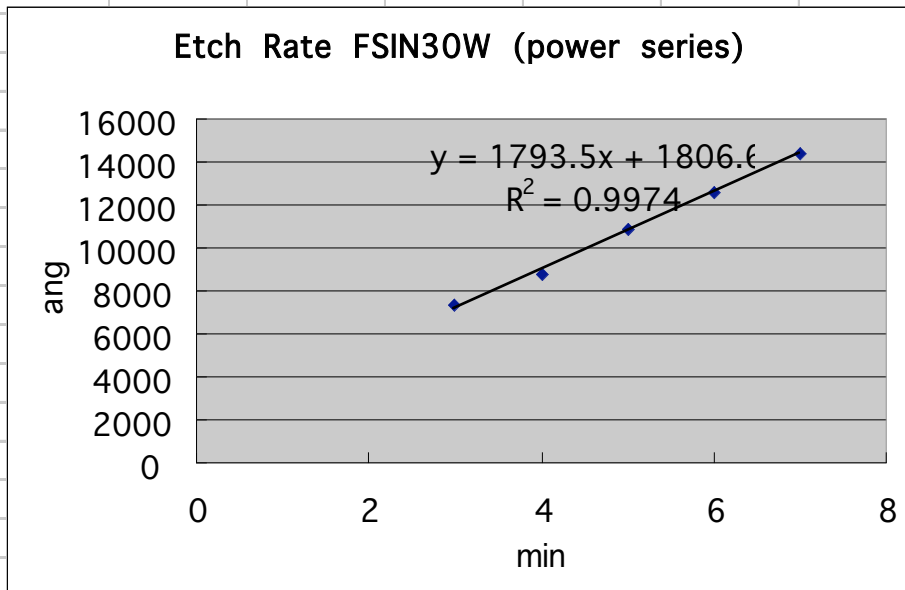
number	line1	line2	line3	line4	min	average
O	9698	9790	9751	9825	4.15	9766
A	9028	8995	9040	9067	3.83	9033
B	7404	7278	7298	7333	3.00	7328
C	6014	6073	6021	5814	2.50	5981
D	5477	5344	5314	5140	2.00	5319
E	4012		4148	4105	1.50	4088
F	7966	8147	8230	8131	3.42	8119





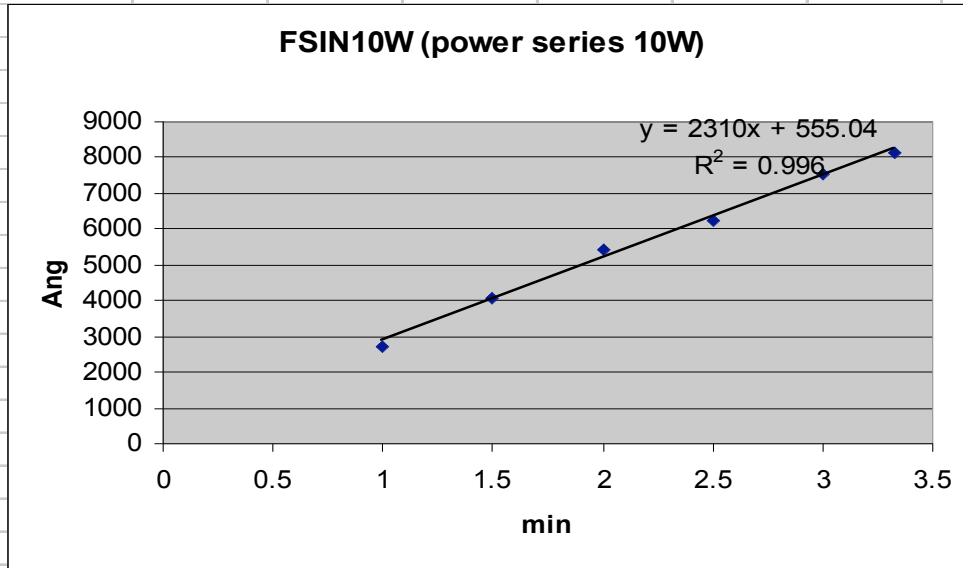
FSIN30W

number	line1	line2	line3	line4	time	average
O	15730	15670	15570	15210	7.42	15545
A	14470	14400	14290	14220	7	14345
B	12590	12490	12630	12640	6	12588
C	11050	10870	10890	10770	5	10895
D	8705	8689	8705	8868	4	8742
E	7187	7216	7390	7409	3	7301
					0	0



FSIN10W

number	line1	line2	line3	line4	time	average
A	7748	7421	7472	7495	3	7534
B	6315	6242	6234	6227	2.5	6254.5
C	5316	5391	5431	5450	2	5397
D	4116	4124	4038	3923	1.5	4050.25
E	8139	8130	8134	8207	3.33	8152.5
F	2759	2740	2680	2760	1	2734.75
O	9143	9149	9195	9226	3.5	9178.25



FSIN5W

number	line1	line2	line3	line4	time	average
O	5680	5690	5689	5653	2.5	5678
A	4547	4520	4466	4452	2	4496
B	3753	3773	3774	3801	1.67	3775
C	3036	3041	3018	3040	1.33	3034
D	2333	2307	2315	2263	1	2305
E	1646	1646	1661	1678	0.67	1658

