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Plasma Enhanced Chemical Vapor Deposition (PECVD) of Silicon Nitride (SiNx) Using Oxford Instruments System 100 PECVD

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Abstract
This report discusses the deposition process of SiNx using the Oxford System 100 PECVD.

Disciplines
Nanoscience and Nanotechnology

1. Introduction

This report documents the study of deposition characteristics and film properties of silicon nitride (SiN_x) thin films deposited by plasma enhanced chemical vapor deposition (PECVD) using *Oxford PlasmaLab 100* system. Deposition rate, thickness non-uniformity, optical constant such as refractive index and in-plane stress of SiN_x films due to variation in duty cycle of high frequency and low frequency power during deposition were examined.

2. Tools and Techniques used

I. Oxford PlasmaLab 100 PECVD system was used for deposition of SiN_x films on 100 mm (4-inch) <100> orientation Si wafers of thickness 525 ± 25 µm.

II. Filmetrics F50 optical interferometer was used for measuring the thickness of deposited films, non-uniformity in thickness over the wafer and optical constants.

III. KLA Tencor P7 profilometer was used for measuring in-plane stress in SiN_x films.

3. Baseline Recipe

Following baseline recipe was used for film deposition after loading the wafer in to the chamber via loadlock:

Units:

- Gas flow rate: standard cubic centimeters per minute (sccm)
- Pressure: millitorr (mT)
- Temperature: degrees Celsius (°C)
- High frequency (RF) and low frequency (LF) power: Watts (W)

Step 1: System chamber is pumped at base pressure (below 5 mT) for 1 minute with electrode temperature at 350 °C.

Step 2: Chamber is pre-heated and purged with N_2 having flow rate of 700 sccm at pressure set point of 1400 mT and electrode temperature at 350 °C for 1 minute (for 4-inch wafer). *

*Step 2: If you are processing pieces mounted on a carrier substrate, it is recommended that the time in step 2 be increased to 10 minutes to ensure temperature stabilization of your samples.

Step 3: SiN_x is deposited in this step with following precursors and chamber conditions:

- Silane (10 % SiH_4 in Helium) flow rate: 90 sccm
- Ammonia (NH_3) flow rate: 45 sccm
- Nitrogen (N_2) flow rate: 1305 sccm
- Pressure: 1800 mT
- Low frequency LF power: 160 W, duration 's' seconds (transformer set to tap location labelled "500")
- High frequency RF power: 200 W, duration '20-s' seconds
- "Pulsed" ** is checked with "HF First" also checked in the system software.
- Capacitor starting points: Capacitor #1: 77 %, Capacitor #2: 26 %
- Electrode temperature: 350 °C
- Deposition time set point is hh:mm:ss (hours:minutes:seconds)

Step 4: Chamber is pumped to base pressure (below 5 mT) and wafer removed from loadlock.

** "Pulsed" deposition takes place where each deposition cycle of 20 seconds consists of 's' seconds of deposition at LF and '20-s' seconds of deposition at RF. For example, 8-12 duty cycle will consist of 8 seconds of deposition at LF and 12 seconds of deposition at RF. For a total deposition of 1 minute, 3 such cycles will take place.

4. Deposition characteristics and film properties

The following sections will discuss the deposition characteristics and film properties on varying duty cycle. As mentioned in the recipe, "pulsed" deposition takes place. In examining the effect of duty cycle, deposition at LF and that at RF is varied keeping the total duration of each cycle to be 20 seconds. Various duty cycle combinations examined are presented in table 4.1. For a total deposition of 2 minutes, 6 cycles of 20 seconds each will take place.

Total cycle time (s)	20								
LF time (s)	6	7	8	9	10	11	12	13	14
RF time (s)	14	13	12	11	10	9	8	7	6

Table 4.1: Duty cycles examined.

4.1 Deposition rate

To study the effect of duty cycle on deposition rate and film properties, 9 deposition runs were carried out with various LF and RF cycles as presented in table 4.1. Each run consisted of 1 minute of deposition, thus running 3 full cycles of alternating LF and RF power. Figure 4.1 shows the resultant deposition rate and standard deviation of film thickness. Data used in figure 4.1 is presented in table 4.2 as measured by *Filmetrics F50*. The blue curve shows the deposition rate which ranges between 47 nm/min. to 52 nm/min. The black vertical bars at each point on the blue curve denotes the maximum and minimum SiN_x deposition rate on the wafer. It should be noted that while change in deposition rate with RF power cycle time is small (range of blue curve is ~5 nm/min.), the black vertical bars can have range as much as 9 nm/min. (for RF power cycle time of 7 s) which wouldn't be desirable for a deposition rate of 48 nm/min. The red curve shows standard deviation for each RF power cycle time. The deposition rate is calculated based on the average film thickness on the wafer as measured by *Filmetrics F50*. *Filmetrics F50* is equipped with a motorized stage allowing for the collection of full wafer maps as shown in figure 4.2 (wafer map of deposition run for 1 minute at RF power cycle time of 6 s). Thickness at 115 points per wafer was measured with 5 mm edge exclusion. The standard Si_3N_4 "universal" material file supplied in the software was used for these measurements.

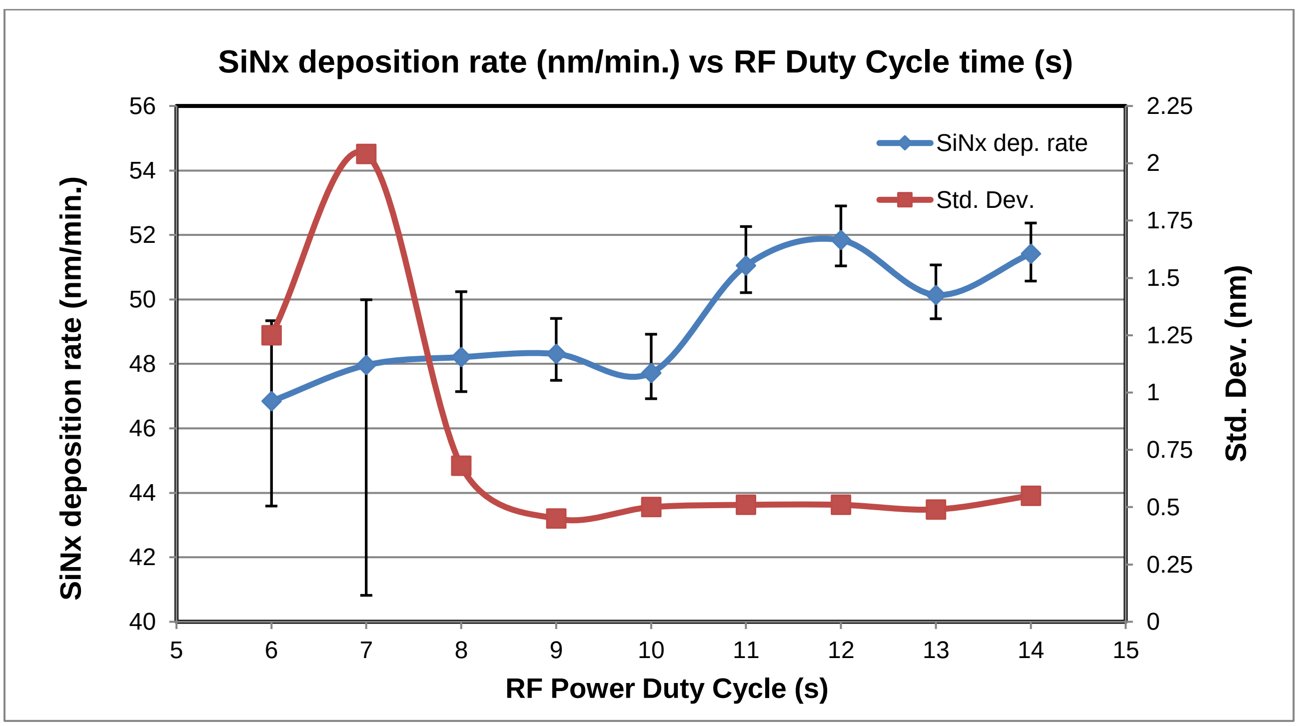

Figure 4.1: Variation of deposition rate with duty cycle as measured by Filmetrics F50.

Sample	Power LF, RF (W, W)	Duty Cycle LF, RF (s, s)	Total dep. time (min.)	Dep. Rate (nm/min.)	Std. Dev. (nm)	Non-uniformity (%)
1	160, 200	6, 14	1	51.42	0.55	1.8
2		7, 13		50.14	0.49	1.7
3		8, 12		51.84	0.51	1.8
4		9, 11		51.05	0.51	2
5		10, 10		47.72	0.5	2.1
6		11, 9		48.31	0.45	2
7		12, 8		48.21	0.68	3.2
8		13, 7		47.96	2.04	9.6
9		14, 6		46.84	1.25	6.1

Table 4.2: Deposition rate and thickness non-uniformity as measured by Filmetrics F50.

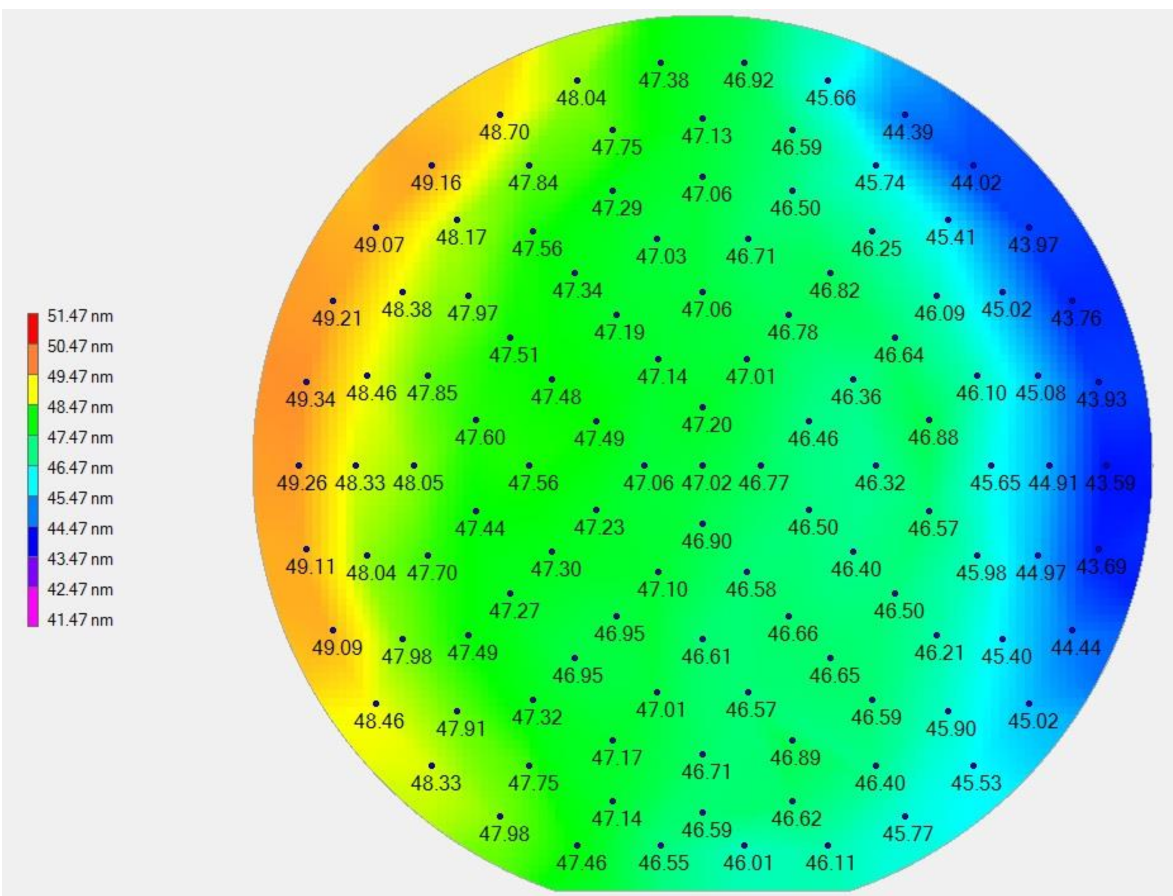

Figure 4.2: 2D map of thickness profile as measured by Filmetrics F50 (RF cycle time of 6 s).

4.2 Thickness non-uniformity

Figure 4.3 shows non-uniformity in film thickness across the wafer as measured by *Filmetrics F50*. Except for the deposition at RF power cycle time of 6, 7 and 8 s; non-uniformity is around 2 %. Data used in figure 4.3 is presented in table 4.1.

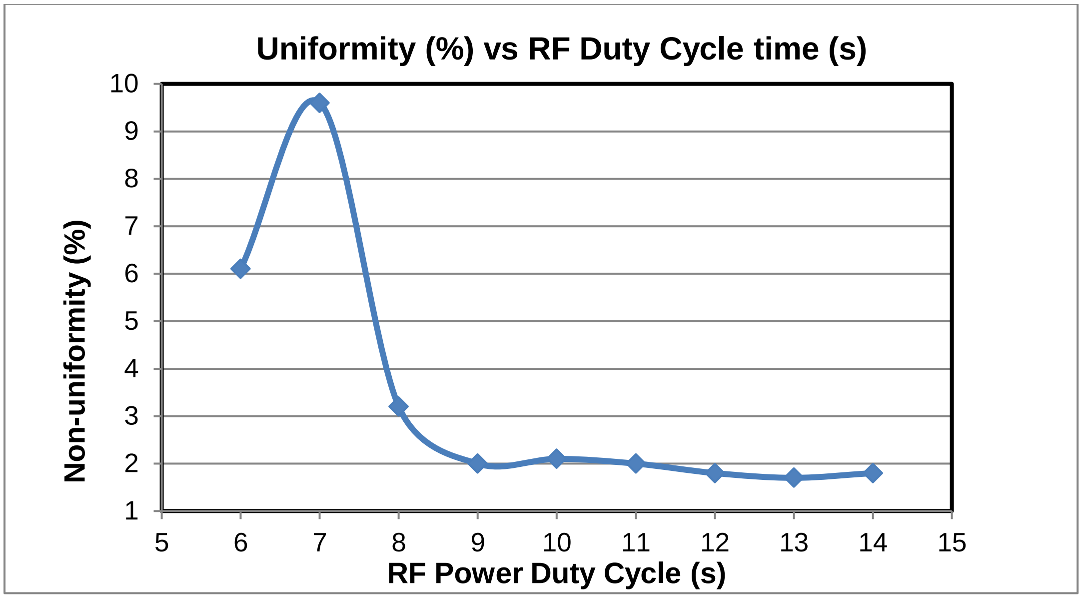

Figure 4.3: Thickness non-uniformity as measured by Filmetrics F50.

4.3 Optical constant

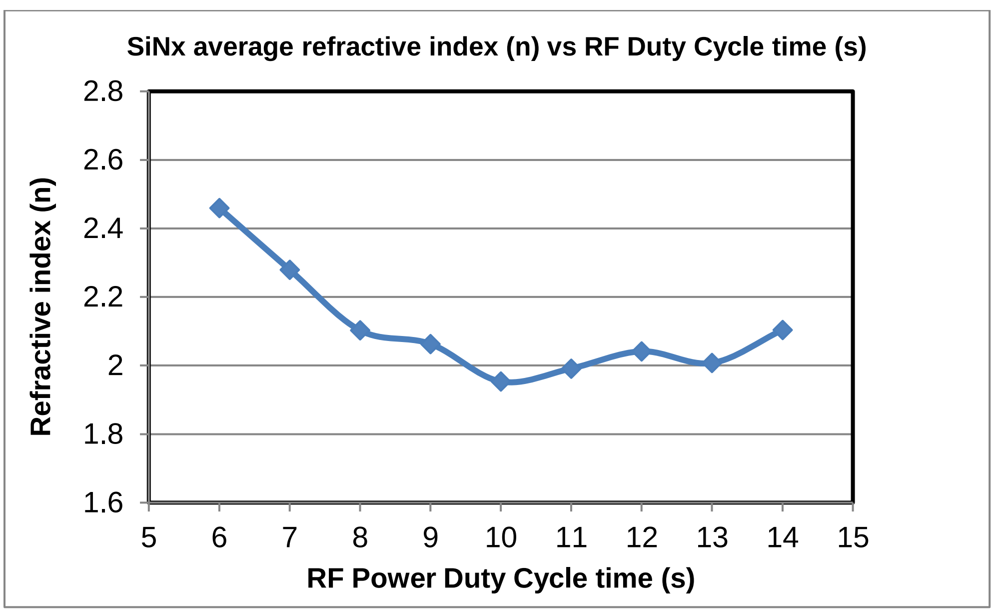

Figure 4.4: Variation in refractive index 'n' with duty cycle as measured by Filmetrics F50.

The refractive index 'n' of the samples is measured using *Filmetrics F50*. Figure 4.4 shows refractive indices of the films for varying RF power cycle time. Refractive index shown for each sample is the average of refractive index measured at 115 points per wafer with 5 mm edge exclusion, similar to the thickness measurement. Data used in figure 4.4 is presented in table 4.3. It can be seen that changing the duty cycle can have significant effect on the optical quality of the film.

Sample	1	2	3	4	5	6	7	8	9
RF time (s)	6	7	8	9	10	11	12	13	14
n	2.4598	2.2796	2.1028	2.0631	1.9535	1.9914	2.0415	2.0075	2.1040

Table 4.3: Refractive index 'n' as measured by Filmetrics F50.

4.4 Mechanical Stress

In-plane stress is measured to study the effect of duty cycle on film stress, which can be affected by factors such as dissolved gases such as Ar, He and H from the deposition process and stoichiometric ratio of Si and N. To measure in-plane stress, 2D stress measurement option in *KLA Tencor P7* profilometer is used. Film stress is measured in two perpendicular directions in center: one (MFDWN) parallel to the major flat axis of the substrate (MFDWN) and second (MFRT) perpendicular to the major flat axis of the substrate as shown on the right in figure 4.5.

Before depositing SiN_x film, radius of curvature of the Si substrate is measured using the 2D stress option. After the deposition, radius of curvature of the deposited film is measured. The software in *P7* calculates the stress using the pre- and post-deposition radius of curvature and the input film thickness. The average film thickness as measured by *Filmetrics F50* is used to calculate stress. Since the stress calculation uses average thickness and does not consider the non-uniformity, stress calculated is approximate. Figure 4.5 shows the stress across MFDWN and MFRT for various duty cycles. Data used in figure 4.5 is presented in table 4.4. The film stress is majorly compressive in nature and anisotropic in major cases (different stress across MFDWN and MFRT).

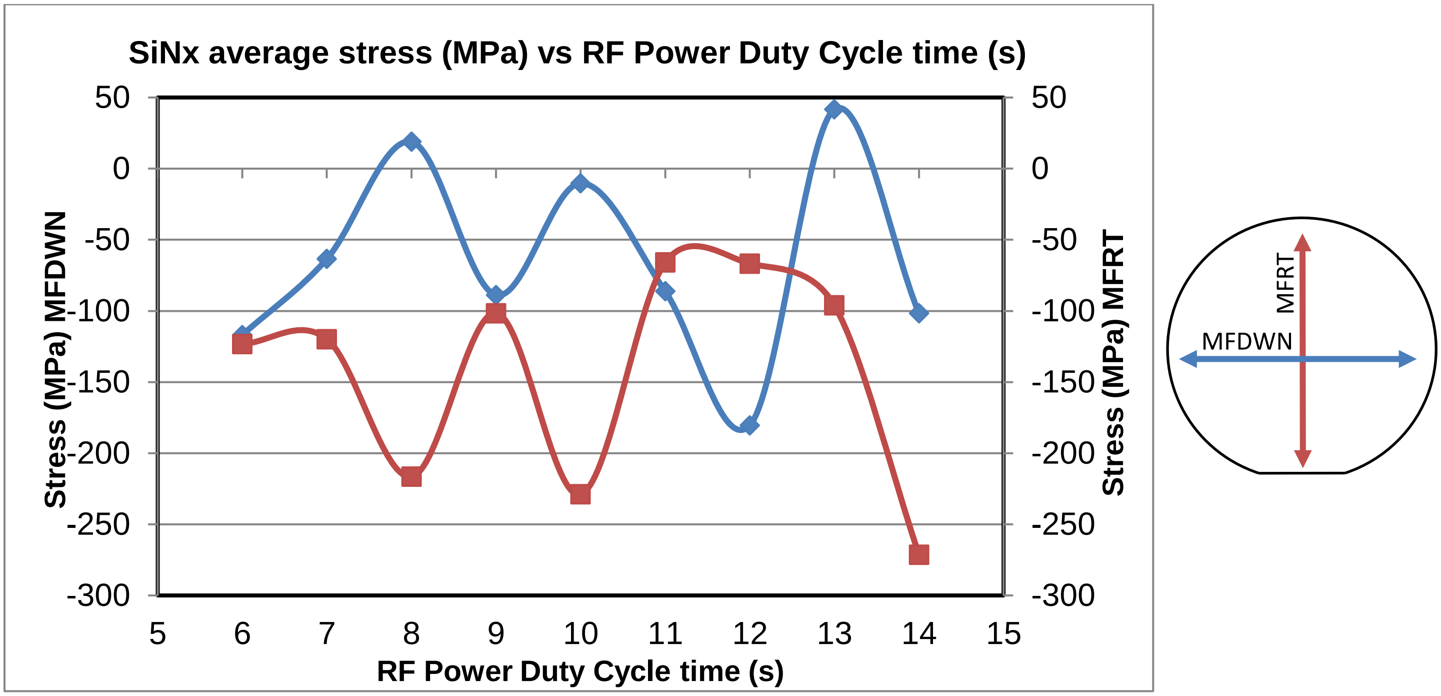

Figure 4.5: In-plane stress in SiN_x films as measured by KLA Tencor P7.

Sample	Power LF, RF (W, W)	Duty Cycle LF, RF (s, s)	Avg. thickness (nm)	Stress (MPa) MFDWN	Stress (MPa) MFRT
1		6, 14	51.42	-101.7	-271.3
2		7, 13	50.14	41.59	-96.15
3		8, 12	51.84	-180.4	-66.78
4		9, 11	51.05	-86.26	-65.93
5	160, 200	10, 10	47.72	-10.26	-228.9
6		11, 9	48.31	-88.86	-101.6
7		12, 8	48.21	18.87	-216.5
8		13, 7	47.96	-63.41	-119.8
9		14, 6	46.84	-116.9	-123.3

Table 4.4: In-plane stress as measured by KLA Tencor P7.

5. Summary

Deposition rate and film properties of SiN_x films deposited by PECVD using *Oxford PlasmaLab 100* are examined. Tools such as *Filmetrics F50* (interferometer) and *KLA Tencor P7* (profilometer) are used to measure thickness, optical properties and film stress. To examine effect of duty cycle on deposition rate, PECVD is carried out for varying duty cycles as shown in figure 4.1. The deposition rate is calculated using thickness measurement obtained by *F50*. The thickness measurement also provides information on non-uniformity of film thickness across the wafer. The user is advised to take into account the deposition rate and its standard deviation, the non-uniformity for full wafer deposition, refractive index ‘n’ and in-plane stress in selecting the duty cycle to be used for the deposition process for desired film properties.