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Deposition and Etch Characterization of Low Stress Silicon Nitride Films Deposited via LPCVD

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Keywords

Silicon Nitride deposition, SiNx deposition, SiNx etch, Silicon Nitride etch, LPCVD SiNx

Goal:

The purpose of this document is to investigate deposition rate, uniformity, stress in LPCVD SiN_x film, and measure the etch rate using CF4 and CHF3/O2 in an RIE tool.

Materials:

- Prime 4 inch Si wafer

Tools:

- Sandevik LPCVD
- Oxford PlasmaLab PECVD
- Filmetrics F50 optical profilometer
- KLA Tencor P7 2D&3D/stress profilometer
- Oxford PlasmaLab 80 Plus RIE

Units:

- Gas flow rate: standard cubic centimeters per minute [sccm] and standard liter per minute [slpm]
- Pressure: Torr [Torr]
- Temperature: Kelvin [K] and degree Celsius [°C]
- Stress [MPa]

Protocol:

Note: stress measurement of thin films using KLA Tencor P7 2D&3D/stress profilometer requires a base measurement of the wafer prior to deposition. This can be done using the Standard Operating Procedure available *here*[1].

Once the baseline is measured, the wafers are transferred to the LPCVD SiN_x deposition tube.

LPCVD deposition:

- Using the Standard Operating Procedure available *here* [2] for the Sandvik system, deposit a SiNx film following the recipe below:
  1. Set furnace idle to 600 °C.
  2. Ramp up the temperature from 600 °C to 835 °C at a 6°C/min rate (~ 1 hour)
  3. Let the wafers stabilize at 835 °C for 1 hour.
  4. Process the wafers for 1 hour in the following conditions: 835 °C, 200 mTorr, DCS (dichlorosilane) flow 260 sccm, NH_3 flow 37 sccm.
  5. Ramp down the temperature from 835 °C to 600 °C in 1 hour (~ 6°C/min).
- Run the anneal process using the following steps:
  1. Load the wafers directly from the LPCVD. Ramp up the temperature from 600 °C to 1050 °C in 1 hour.
  2. Anneal the wafers at atmospheric pressure, with Nitrogen at 2 slpm for one hour.
  3. Ramp down the temperature to 600 °C in 2.5 hours.
  4. Extract the load arm and unload the wafers once they are cool enough to handle.

Thickness Measurement:

- Using the Standard Operating Procedure available *here*[3] measure the thickness and capture the wafer map on a Filmetric F50 optical profilometer. The thickness value is needed for the final stress measurement in the thin film.

Stress Measurement:

- Using the Standard Operating Procedure for KLA Tencor P7 2D&3D/stress profilometer available *here*[1] measure the stress. You will be asked to provide the thickness of the film, measured in the previous step

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[1] Yamamoto H., *SOP of MET-02* (2021) Standard Operating Procedure. Book 13, Scholarly Commons, University of Pennsylvania

[2] Azadi M., *Sandvik LPCVD and anneal tubes standard operating procedure.* Standard Operating Procedure. Book 6, Scholarly Commons, University of Pennsylvania

[3] Yamamoto H., *SOP of MET-03* (2021) Standard Operating Procedure. Book 15, Scholarly Commons, University of Pennsylvania

Etching:

- Using the Standard Operating Procedure for Oxford PlasmaLab 80 Plus RIE available *here*[4] run incremental etch steps using CF4 and CHF3/O2 recipes and measure the thickness (see thickness measurement protocol).

  Note: the recipe details are described in the following documents:
  - M. Metzler Reactive Ion Etch (RIE) of Silicon Nitride (SiNx) with Tetrafluoromethane (CF4). Scholarly Commons. 2016
  - M. Metzler. Reactive Ion Etch (RIE) of Silicon Nitride (SiNx) with Trifluoromethane and Oxygen (CHF3/O2). Scholarly Commons. 2016

Following the deposition protocol, we deposited SiN_x on 10 wafers and measured the deposition rate to be ~ 8 nm/min. Figure 1 shows the thickness for 10 wafers (Position 1 being closest to the loading lid and 10 being the wafer positioned in the back of the tube)

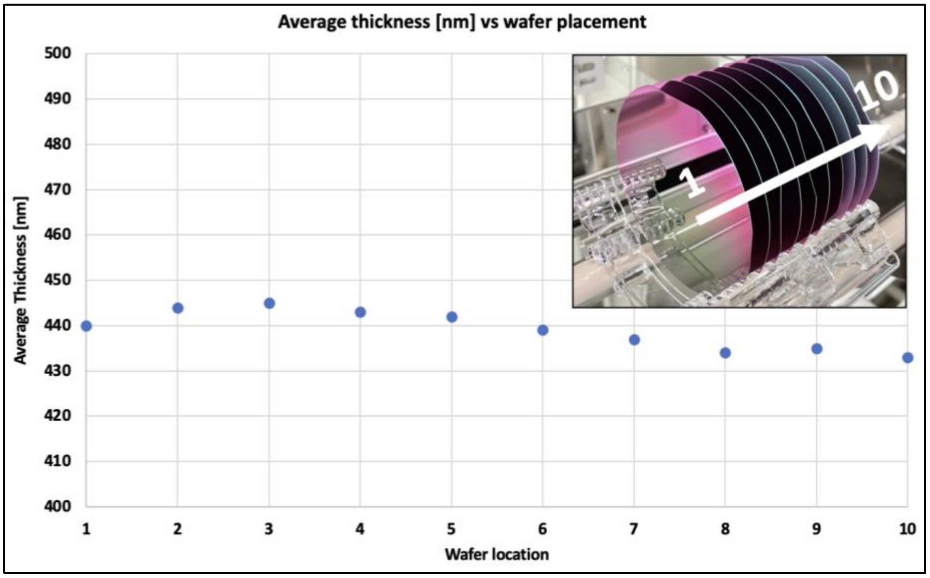

Figure 1. Film thickness vs wafer position for a 1 hour following the parameters described in the protocol section.

Wafers were place in the chamber at different orientations to track the effect of loading on the uniformity of deposition.

To measure the thin film stress, we scanned the surface of the wafers before deposition and after the post-deposition anneal. Using the difference between these two scans, and the film thickness we were able to calculate the stress in the annealed film. Figure 2 shows how these wafer scans (traces) change after thin film deposition for a typical thin film.

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[4] Azadi M., *Oxford PlasmaLab 80 Plus RIE standard operating procedure* Standard Operating Procedure. Book 6, Scholarly Commons, University of Pennsylvania

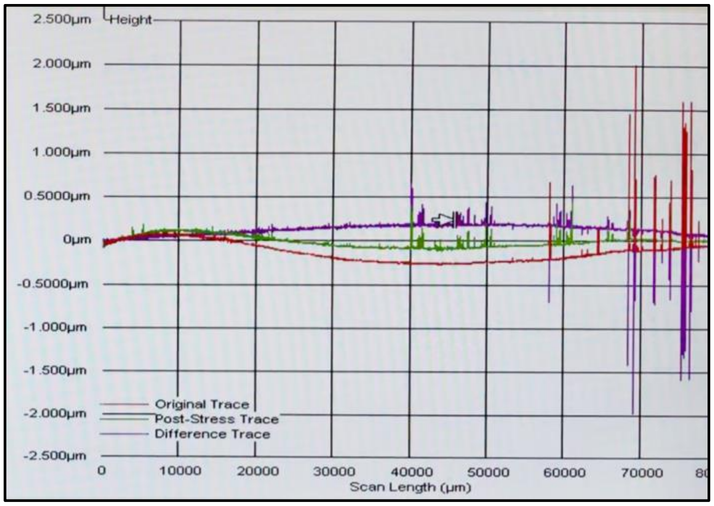

Figure 2. The induced curvature in the wafer (original trance), after deposition and annealing (Post-Stress Trace). And the Difference that is used to measure the stress.

Figure 3 show the thickness and uniformity map of wafers in positions 1, 2 and 3. Along with the original and post-anneal traces and their difference. The arrows indicate contact points between the wafers and the quartz boat during deposition and annealing.

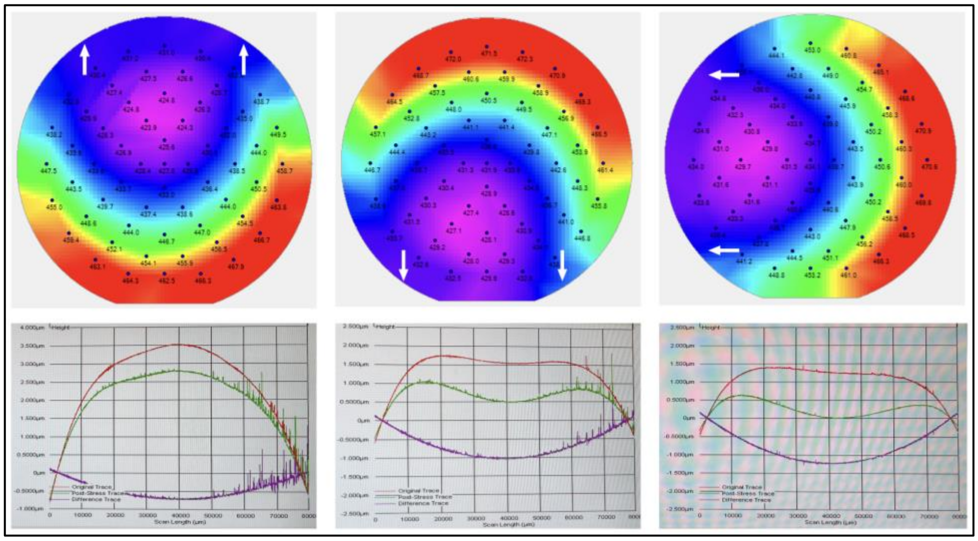

Figure 3. **Top row**: wafer map of thickness uniformity - arrows indicate contact points between the wafers and the quartz boat during deposition and annealing. **Bottom row**: surface trace for stress measurement (Before deposition, after annealing and difference)

Stress values in SiN_x films deposited using LPCVD are shown in figure 4. We used different scan directions to study directional dependance of stress.

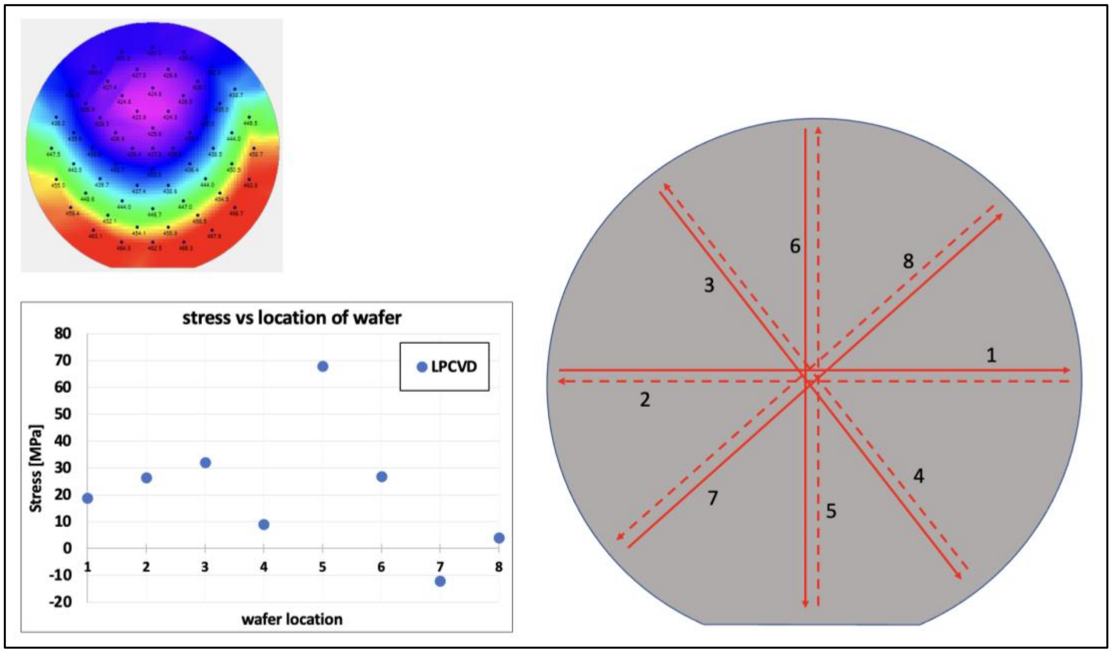

Figure 4. Stress value for LPCVD films after annealing. Numbers on the trace lines indicate the wafer location and the stress measurement trace with respect to the major flat.

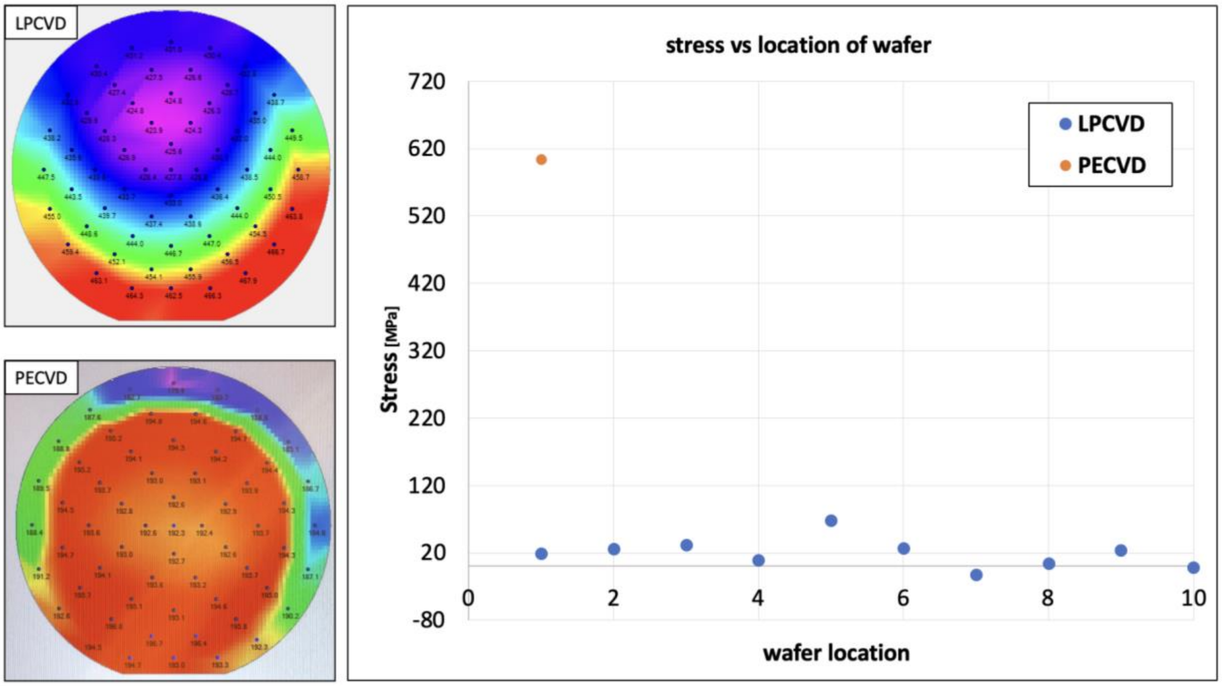

Figure 5. Comparison of thickness uniformity and stress in PECVD (not annealed) and LPCVD (annealed) grown films.

Etch rate of the annealed LPCVD SiN_x using CF4 and CHF3/O2 recipes is shown in figure 6.

Figure 7 shows the comparison of etch rate of annealed LPCVD and non-annealed PECVD SiN_x films, using the CF4 etch recipe.

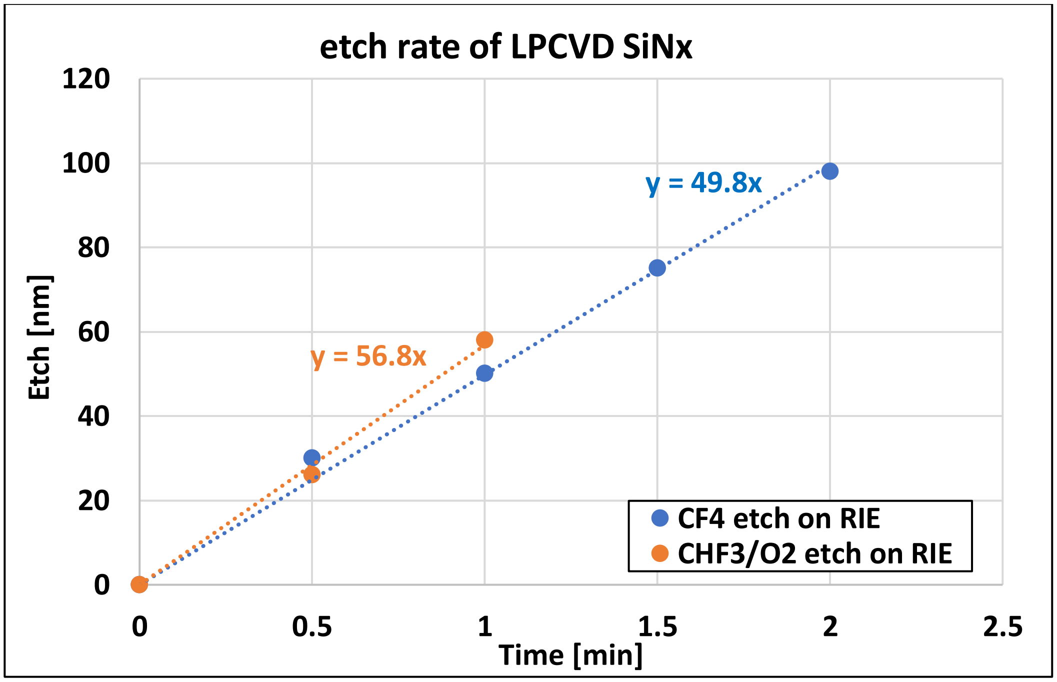

Figure 6. Etch rate of annealed LPCVD SiN_x

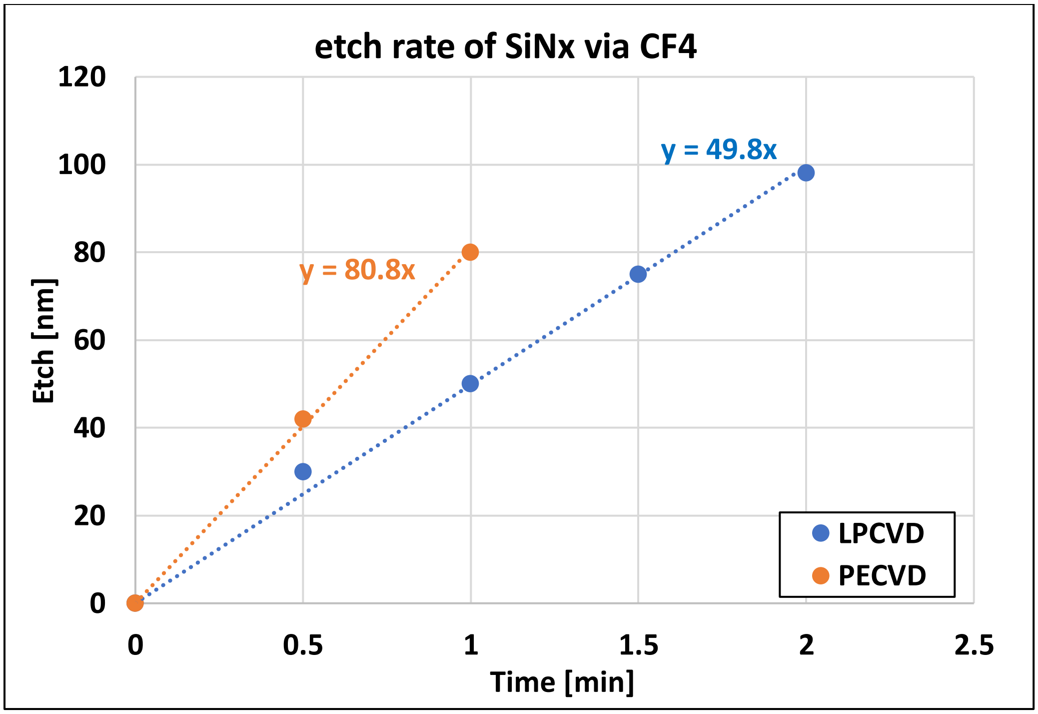

Figure 7. Etch rate comparison of annealed LPCVD and non-annealed PECVD SiNx using CF4 RIE etch

The etch rate of annealed LPCVD SiN_x using 49% HF at room temperature (no agitation) is measured to be ~ 2.5 nm/min, though we do not guarantee the uniformity and accurate repeatability of the wet etch processes.