

# Hydrogen Desorption (TDS) in SiN<sub>x</sub> Thin Films using ESCO-TDS1200II IR

## Summary

Using an infrared-heating TDS system (Model: ESCO-TDS1200II IR), we evaluated hydrogen desorption in SiN<sub>x</sub> thin films. Three H<sub>2</sub> desorption peaks were observed at 450 °C, 570 °C, and 980 °C. The low-temperature peaks are consistent with surface-origin species on the native oxide (H<sub>2</sub>O or H), whereas the high-temperature peak reflects bulk hydrogen (N–H and Si–H). These results provide actionable guidance for pre-clean, deposition, and annealing optimization.

## Detailed Description

### 1. Sample & Instrument

Infrared-heating-type thermal desorption analyzer ESCO-TDS1200II IR) was used (See Fig. 1). The samples were heated under ultrahigh vacuum ( $<1 \times 10^{-7}$  Pa) with 60 °C/min up to 1100 °C. The sample temperatures were measured by a pyrometer.

We prepared two samples, A and B, with a SiN<sub>x</sub>/Si structure shown in Fig. 2. The SiN<sub>x</sub> films with a thickness of 17.9 nm were grown on p-type (100) silicon substrates using an LPCVD technique with the SiH<sub>2</sub>Cl<sub>2</sub>-NH<sub>3</sub> system at 650 °C.

H<sub>2</sub> desorption in the two samples, A and B, was investigated.

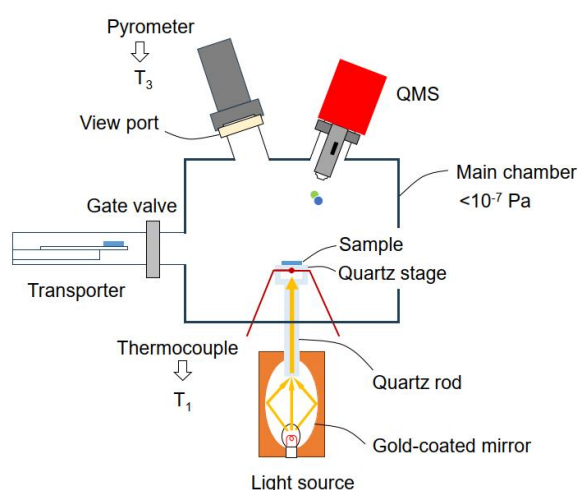


Fig. 1 Infrared-heating-type thermal desorption analyzer

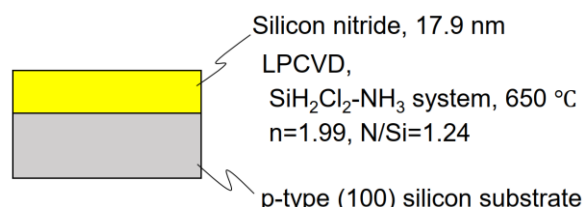


Fig. 2 Schematic cross-section of the sample structure.

## 2. Results

Trace (a) in Fig. 3 shows the H<sub>2</sub> desorption spectrum obtained from sample A. Three different peaks of the H<sub>2</sub> desorption rate appeared at 450, 570, and 980 °C.

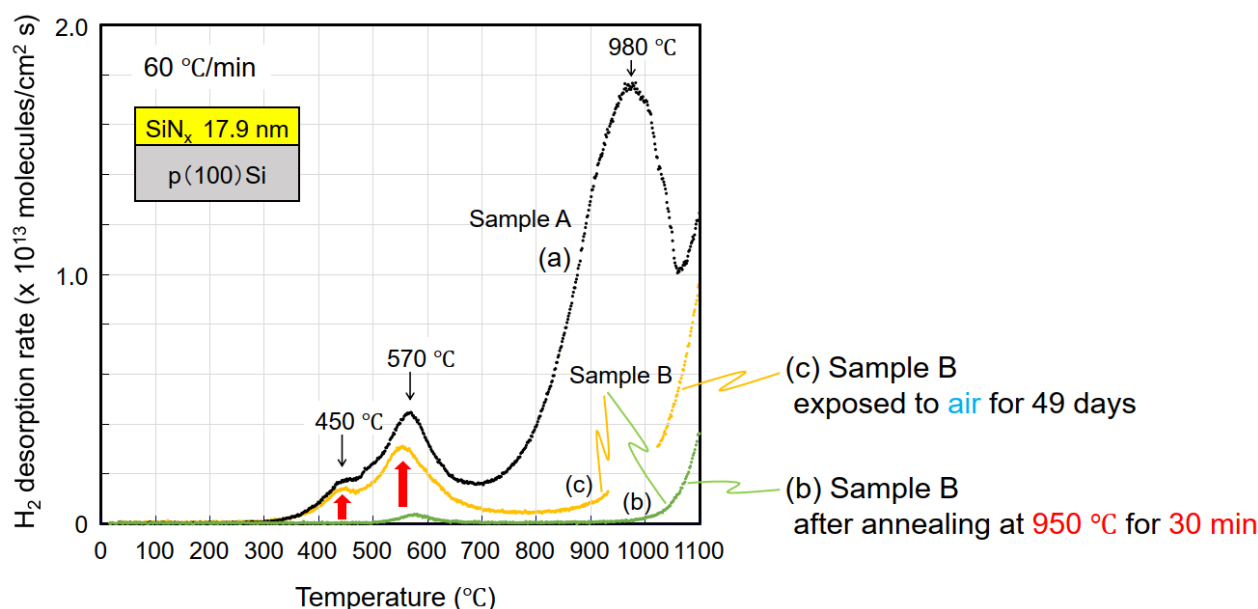


Fig. 3 TDS spectra of H<sub>2</sub> molecules obtained from samples A and B.

Sample B was annealed under ultrahigh vacuum ( $<1 \times 10^{-7}$  Pa) at 950 °C for 30 min. As shown in Fig. 3, trace (b) of sample B indicates that the three peaks of the H<sub>2</sub> desorption rate were significantly attenuated. Almost H atoms were released from sample B during the annealing. Subsequently, sample B was exposed to air for 49 days at room temperature and the TDS measurement was conducted again. As seen from trace (c), the two peaks at 450 and 570 °C clearly appeared again.

This result suggests that the two peaks are mainly caused by H<sub>2</sub>O or H<sub>2</sub> adsorbed on the native oxides existing on the surfaces of the SiN<sub>x</sub> film and Si substrate.

On the other hand, the peak at 980 °C did not recover after air exposure. This indicates that the peak at 980 °C was caused by hydrogen released from the SiN<sub>x</sub> bulk, which would be originated with H atoms dissociated from N-H and Si-H bonds in the film, according to Ref. [1].

## 3. Interpretation

Low-temperature regime (450 °C and 570 °C) — Surface-origin desorption

High-temperature regime (around 980 °C) — Bulk-bonded hydrogen in SiN<sub>x</sub>

For further details regarding the above report, please refer to the following reference [2].

Thermal desorption spectroscopy (TDS) is effective for probing thermally activated processes in thin films such as SiN<sub>x</sub>. By separating and detecting surface-origin and bulk-origin species, it enables precise evaluation of the diverse films employed in semiconductor devices.

## References

- [1] K. Kobayashi, R. Miyauchi, and K. Kimoto, "Influence of high-temperature thermal annealing on paramagnetic point defects in silicon-rich silicon nitride films formed in a single-wafer-type low-pressure chemical vapor deposition reactor," J. Vac. Sci. Technol. A, vol. 42, pp. 050402-1 - 050402-6, July 2024. <https://doi.org/10.1116/6.0003778>.
- [2] K. Kobayashi and K. Kimoto, "Characterization of Hydrogen Desorption and Charge Traps in Silicon Nitride Films," 2024 International Symposium on Semiconductor Manufacturing (ISSM), Tokyo, Japan, 2024, pp. 1-4, doi: 10.1109/ISSM64832.2024.10874887.