A New Diagnostic Technique for Monitoring Plasma Reactor Walls: Multiple Total Internal Reflection Infrared Surface Probe

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Chemically Reactive Gas Plasmas for Etching







Motivation and introduction: the need for a "wall probe"

- Plasma reactor walls can play a crucial role in determining the plasma properties, such as the radical concentrations and ion densities, especially at low pressures.
- This well-known plasma-wall interaction has caused problems ranging from process drifts in IC manufacturing to irreproducible data in fundamental studies employing plasma diagnostics.
- Terms such as "wall conditioning" and "reactor seasoning" have become accepted language in literature to describe art of avoiding such effects.
- There are no diagnostic techniques to quantify the chemical state of the walls in a plasma reactor.





Objective

- Develop a surface probe based on multiple total internal reflection Fourier transform infrared (MTIR-FTIR) spectroscopy that can be used as a diagnostic tool to monitor films and adsorbates on walls of a plasma etching reactor (Lam TCP 9400[®]).
- Demonstrate applications of the probe
 - Effect of wall conditions on Cl/Cl₂ concentrations and uniformity of etching.
 - Monitoring deposits on reactor walls during Cl₂/O₂ etching of Si.
 - Monitoring efficacy of chamber wall cleaning in Shallow Trench Isolation (STI) etching.
 - Monitoring wafer-to-wafer reproducibility of wall conditions and its effect on process reproducibility.





Experimental Setup - Lam TCP 9400







MTIR-FTIR Wall Probe







Principles of MTIR-FTIR

Z Plasma $\mathbf{n}_{\mathbf{f}}$ film Film is probed through attenuated total reflection wall $\mathbf{\theta}$ 'n_{IRC} mode if $n_f < n_{IRC} \sin \theta$. **IR** from to detector spectrometer Z Plasma n_f Е **Film** is probed through multiple internal transmission n_{IRC} mode if $n_f > n_{IRC} \sin \theta$. $E = E_0 \exp\{-z/d_0\}$ Ο Ζ Surface species are probed C S Ε through attenuated total reflection.





Principles of MTIR-FTIR







Application: Si trench etching in a high density Cl₂/O₂ plasma

- Shallow Trench Isolation (STI) process is used to isolate transistors in ICs.
- > In STI process trenches with tapered sidewalls are etched into Si using Cl_2/O_2 .
- → While Cl_2 etches Si anisotropically, O reacts with reaction products (SiCl_x 0≤x≤4) to deposit a silicon oxychloride (SiO_xCl_y) film on the sidewalls.
- ➤ The feature profile shape depends on a delicate balance between etching and deposition.







Application: Si trench etching in a high density Cl₂/O₂ plasma



- SiO_xCl_y film is deposited not only on the trench sidewalls but also on the reactor walls.
- > Presence of the SiO_xCl_y film on the wall affects the radical wall recombination probabilities.
- SiO_xCl_y film must be etched from the reactor walls using SF₆ containing plasma in between every wafer. This cleaning process is referred to as Waferless Auto Clean (WAC).





Cl and Cl₂ concentration measurements

■ Effect of the wall conditions on the absolute Cl and Cl₂ concentrations were determined using optical emission spectroscopy in conjunction with actinometry as described by Donnelly and Malyshev. (*J. Vac. Sci. Technol.* A 14, 1076, 1996; *J. Appl. Phys.* 84, 137, 1998)

$$\frac{n_i}{n_{Ar}} = \alpha_i(T_e) \frac{I_i}{I_{Ar}}$$

Used 306 nm Cl₂, 822 nm Cl and 750.4 nm Ar emissions and corrected for the T_e dependence of α.

Calibrated α using as power $\rightarrow 0$, dissociation $\rightarrow 0$, $n_{Cl2} \rightarrow n_g = P_g/k_BT_g$

Used a simple model of the Cl mass balance with an adjustable Cl surface recombination probability, γ.

$$Cl_{2} + e^{-} \xrightarrow{k_{d}(T_{e})} 2 Cl + e^{-}$$
$$Cl + wall \xrightarrow{\gamma} \frac{1}{2} Cl_{2}$$
$$Cl \xrightarrow{S_{p}} pumped out$$





Cl concentration drift as a function of wall condition



- Drift in Cl concentration is due to changes in the Cl recombination probability on the walls. The drift is not related to chamber temperature, charge density or kT_e
 Expression to SE /O, plasma masta the walls to a perceducible condition.
- > Exposure to SF_6/O_2 plasma resets the walls to a reproducible condition





Resetting the chamber walls through "cleaning" with SF_6 plasma is essential for collecting reproducible data



☐ Cl concentration exhibits hysteresis with plasma power due to changes in the Cl recombination probability on the chamber walls with time.





A thin SiO₂ film deposited on the chamber walls decreases Cl surface recombination probability, γ

- ~ 5 Å thick SiO₂ film is deposited on reactor walls even when Si wafer or O₂ are not present in the chamber: quartz window is the source of Si and O.
- ❑ Wall recombination probability of Cl is lowered drastically when walls are coated even with ~ a monolayer of SiO₂.
- γ calculated by fitting measured Cl concentration to a model is ~ 0.03 on SiO₂ covered walls.
- □ SF₆/O₂ plasma plasma removes the SiO₂ film.



 $\Box \quad \gamma \sim 1 \text{ after the SiO}_2 \text{ is removed.}$





Cl₂ dissociation under identical plasma operating conditions can be drastically different depending on the wall conditions



□ % dissociation depends on pressure

$$n_{Cl_2} = \frac{n_g}{1 + \frac{8k_d n_e V_d}{S \psi_{Cl} + 4S_p}}$$





With chamber reset

- Cl loss from the reactor is limited by transport (diffusion to the walls).
- **Low % dissociation, higher** γ **.**
- □ % dissociation is independent of pressure

$$n_{Cl_2} = \frac{n_g}{1 + \frac{8k_d n_e V_d}{V_d D / \Lambda^2 + 4S_p}}$$



Cl concentration drifts slowly as the reactor walls are covered with a thin layer of SiO₂ and γ decreases







Effect of a thin SiO₂ film on the walls (and the resulting change in the Cl recombination probability) on Si etching uniformity



50 mTorr, 150 sccm Cl₂, 600 W TCP power, 100 W rf-bias





Plasma Etching Uniformity with WAC (SF₆ plasma cleaning in between wafers)



Initial Clean Wall

After SF₆ Cleaning step

Removal of the silicon oxychloride with SF₆ resets the reactor wall back to the reproducible condition.
 Etch rate and uniformity return to their initial values.





Detection of adsorbed $SiCl_x$ etch products on the reactor walls during Si etching with Cl_2 plasma using the MTIR- FTIR surface probe

Experiment:

- □ Start with ~ 20 Å thick SiO₂ film on the MTIR crystal.
- □ Turn on a Cl₂ plasma with Si wafer in the reactor but without rf bias applied to the chuck ⇒ no etching ⇒ no SiCl_x
- □ Turn on the rf bias: Si wafer etches at 4000 Å/min.
- □ SiCl_x absorptions are not observed after step 2 \Rightarrow Cl does not adsorb easily on SiO₂ surface. This is consistent with low γ.
- SiCl_x absorptions observed after step 3 ⇒ SiCl_x adsorbed onto the wall are produced by etching reactions.







Deposition of SiO_xCl_y film on the walls during Cl₂/O₂ etching of Si



During Cl₂/O₂ etching of Si, SiCl_x etch products react with O and deposit a chlorinated SiO₂ (SiO₂:Cl) film on the chamber walls.





Effect of bias power on chamber wall deposition



- Under otherwise similar etching conditions, an increase in bias power, increases the etch rate and hence the SiCl_x production.
- > Increasing SiCl_x production causes an increase in deposition on the chamber walls \Rightarrow chamber wall deposition depends on amount of SiCl_x etch products generated.





Effect of oxygen on chamber wall deposition



- ➤ Under otherwise similar etching conditions, and constant total flow rate an increase in O₂ inlet flow, increases the deposition on the chamber wall.
- At higher O₂ flows, wall deposition decreases because etch rate decreases (SiCl_x limited), until etching finally stops and there is no deposition on the chamber walls.





Chamber Walls can be Cleaned with F-Containing Plasma



> Sufficiently long SF_6/O_2 plasma (WAC) removes the SiO₂:Cl deposits.





F abstracts and replaces Cl in the SiO₂:Cl film converting it to SiO₂:F



- □ Si-F stretching absorption initially increases during SF₆/O₂ plasma cleaning as F abstracts and replaces Cl in the SiO₂:Cl film. It undergoes a maximum and decreases as the film is removed.
- **Si-O** peak shifts to high wavenumber | F incorporation.
- □ If the SF₆/O₂ WAC step is short, the wall deposits are not removed completely and F incorporated into the film could leach out in next processing step.





Wall as a virtual gas source



- □ SiF₄ is detected in the reactor exhaust and in the plasma even though no fluorine containing species are introduced into the chamber.
- □ The decay in the SiF₄ concentration in the exhaust indicates that an exhaustible amount of F is present in the reactor.
- □ F trapped in the SiO₂ walls as well as in the quartz window during SF₆/O₂ plasma cleaning step is released into the plasma in the subsequent steps.





Application of MTIR-FTIR wall probe to monitoring

shallow trench etching repeatability

- □ 10 wafers etched with Cl₂/O₂ plasma with cleaning (WAC) of the walls in between each wafer.
- **Etch rate is very reproducible.**
- Reproducible wall conditions ensured by the WAC: SiO_xCl_y film is completely removed during WAC.
- However, close observation of the trench profiles show a subtle drift in the slope of the sidewalls.







Etch Profile Drift



After etch Stripped Wafer 1

 ❑ Amount of deposition on the trench sidewalls
 <u>decreases</u> from wafer 1 to wafer 10 ⇒ sidewall angle increases.







Application of MTIR-FTIR wall probe to monitoring shallow trench etching repeatability: deposition on the chamber walls



- □ Infrared spectra collected after etching each wafer show that the amount of film deposited on the walls increases even though the walls are cleaned completely between etching steps and etch rate is constant.
- □ This trend is opposite that of the deposition on the trench sidewalls.





Wafer-to-wafer drift in plasma properties during etching



- □ With every successive wafer processed in the chamber
 - the amount of wall deposition due to each wafer <u>increases;</u>
 - emission from SiCl_x etch products <u>increase</u>;
 - etch rate and SiCl₄ concentration in reactor exhaust remain <u>constant;</u>
 - deposition on the trench sidewalls <u>decrease</u>.
- □ Other experiments show that the drift is related to the amount of residual F in the reactor, most likely trapped F in the quartz window.
- We still do not understand the detailed mechanism of the drift and the role of residual F.





Source of sidewall deposition

- The amount of etch products leaving the bottom of the trench is <u>not changing</u>
- Gas phase SiCl_x species concentration <u>increases</u>
- Deposition on chamber wall increases
- Etch product deposition on sidewall of trench <u>decreases</u>







Wafer-to-wafer drift is related to residual Fluorine level



- > Amount of SiF_4 in exhaust of the reactor indicates the residual Fluorine level in the chamber.
- > More Fluorine is incorporated in the chamber when multiple WAC steps are run
- > Fluorine level in the chamber is **not replenished** to the same level by the succeeding clean-1
- Residual Fluorine level in the chamber <u>decreases</u> as more wafers are processed in the chamber and finally reaches a steady state





Fluorine level maintained - No wafer-to-wafer drift



- Fluorine level in the chamber is replenished to the same level by the succeeding Clean-2's
- Residual Fluorine level in the chamber is <u>constant</u> as more wafers are processed in the chamber
- > Deposition on the chamber wall is **not changing**
- Deposition on the sidewalls of the trench is also invariant







Fundamental Understanding of Plasma-Wall Interactions and Managing Wall Conditions Reduces Wafer-to-Wafer CD Variability

Gate Etching Process with \succ WAC Every Wafer

	Variation (nm 3sigma)
run-to-run 3sigma (with WAC)	2.403
run-to-run 3sigma (w/o WAC)	7



Wafer #

Data Courtesy of H.K. Chiu and H.J. Tao Taiwan Semiconductor Manufacturing Company, Taiwan





Summary

- ❑ A novel diagnostic technique based on multiple total internal reflection (MTIR) FTIR has been developed and used to monitor wall conditions during plasma etching of Si with Cl₂/O₂ plasmas.
- This new probe was used to develop a detailed and fundamental understanding of the interactions between the plasma and the reactor walls.
- Fundamental understanding of plasma-wall interactions obtained through MTIR-FTIR and other plasma diagnostics were used to
 - Improve wafer-to-wafer reproducibility
 - > optimize wall cleaning processes
 - minimize wall cleaning time to decrease emissions



