Chamber Wall Effects on Polycrystalline-Si Reactive Ion Etching in Cl₂: A Multiple Real-Time Sensors Study

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Outline

- Multi-sensor Study of Cl₂ Etching of Poly-Si in Lam 9400 TCP / Variations with F-cleans
 - OES/Actinometry for CI
 - Broadband RF for Plasma Density
 - RTSE for Poly Si Etch Rate
- Wall Recombination Affects Both Neutral Species and Ion Concentrations
- Ion Density Measurement Control of Cl₂ etch of Si
- Interpretation of Actinometry Results Requires Careful Consideration of Gas Dilution Effects on Actinometer Concentration
- HBr-Cl₂ Mixtures

Motivation

- Chamber wall state as source of transient variations
- Loss rates at walls dependent on wall buildup
- Wall condition dynamically alters chemical and plasma densities
- Solutions for process drift: PMs, additional clean steps, test wafers



Control of plasma density will improve process tolerance limits & OEE!

Previous Wall State Work

- <u>Sawin</u>: 1st reported Etch Rate changes in Cl₂ due to O₂(1) & CF₄(1) chamber exposure. (*JECS* 1992)
- <u>Donnelly</u>: Increasing CI neutral conc. with time in a quartz tube helical resonator. (*JVSTA* 1996)
- <u>Aydil</u>: Atomic Cl drifts due to SiO₂ wall conditioning & SF₆ wall cleans. (*JVSTA* 2002)

This Work

- 1st experimental evidence of Cl₂ plasma density variation with F-cleans/wall prep.
- 1st direct correlation of real-time plasma density & real-time etch rate variations
- 1st direct real-time feedback control of plasma density to stabilize poly-Si etch rate in Cl₂
- Improved Understanding of Wall Effects and Actinometry Results

Time Stamped Sensor System



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RTSE



- Real-Time Spectroscopic Ellipsometer (RTSE)
 - Can optically model film etch depth, CD, sidewall slope
 - Use for real-time etch rate monitoring & transients

BroadBand RF





Remarks

- High frequency (GHz), low power (mW) sweep of plasma
- Plasma impedance spectroscopy
- Must analyze broad spectrum of data (Broadband RF Probe)
- Yields plasma density metric

BroadBand RF Circuit Analogy

ZA

TCP Coil

C_{coil}

C_{chuck}

 (ω)

 Z_{p2}

Z_{p1}

ε(ω)

Z_{p3}

Elec. Stat. Chuck

 C_w



- Loss paths give many resonance peaks in |Γ| for single ω_p
- Model peaks as RLC circuit resonances w/

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 \mathcal{O}_{ni}

Wall

BroadBand Signature



Signal sensitive to several important plasma outputs

- Plasma density
- Delivered plasma power
- Chamber wall state
- ***** Wafer surface chemistry

BB Peak Shifts & Density



- Two prominent resonance modes, ω_{n1} & ω_{n2}, for these chamber conditions
- Peak frequencies shift right for increasing density

FTIR Effluent Measurements



- <u>Fourier Transform InfraRed (FTIR) spectroscopy</u> measures volatile etch products in foreline exhaust
- Yields dynamic chemical state changes in SiCl₄ & SiF₄
- Used commercial INDUCTtm FTIR from On-line Tech.

Etch Conditions

- Lam 9400 TCP SE
- 10 mTorr
- 100 sccm Cl₂ flow
 - 100 sccm total etch gas flow for Cl₂/HBr experiments
- 5 sccm Ar flow
- 250 W TCP Power
 - Varied for Plasma Density Control (Closed Loop) Runs
- 100 W Bias Power
 - Bias Voltage Measurement Not Available
- Unpatterned 150 mm Poly-Si/30nm SiO₂/Si Test Wafers

Experimental Definition 1

- First project; 3 experiments
- Compensate for ion density losses due to Fcleaning of chamber walls
 - Nominal Etch: Run plasma chamber at steady state chlorine condition to establish real-time etch rate, BB peak position, and SiCl₄ effluent level
 - 2) <u>Open loop recovery</u>: Prep chamber walls using C_2F_6 clean to strip Silicon Oxychloride buildup, then run identical Cl_2 recipe.
 - 3) <u>Closed loop compensation</u>: Run identically as uncontrolled open loop etch, only now use TCP power to maintain BroadBand setpoint.

(OL) Open Loop Drift Recovery



Nominal etch rate flat, OL rate increasing (upper plot)

- Nominal BroadBand ω_{n2} flat, OL ω_{n2} increasing (lower)
- OL signals do not recover in 60sec

(CL) Closed Loop Recovery



Both nominal & CL etch rate flat (upper plot)

- Both nominal & CL BroadBand ω_{n2} flat (lower plot)
- CL signals recover in ~5sec

SiCl₄ Effluent from FTIR



- Nominal SiCl₄ is flat with no disturbance (black)
- OL SiCl₄ effluent is suppressed = lower ER (green)
- CL SiCl₄ is mostly compensated by controller (blue)



 TCP power compensation in CL is very high at the start to make up for lost Cl⁺ ions to the walls

Experimental Definition 2

- <u>Second project; 2 experiments, OL vs. CL</u>
- 1st wafer effect elimination with plasma density compensation
 - Prep chamber walls using C₂F₆ clean
 - Follow with 3 open loop etches for 30s each in Cl₂ and measure etch depth
 - Prep chamber with C_2F_6 clean again
 - Follow with 3 closed loop etches for 30s each and compare etch depth variation with that in OL case

1st Wafer Effect Reduction Three 30s Cl₂ etches after single F-prep of chamber



- Open loop etch depth
- Etch rate increases, both in situ (RTSE) & ex situ (Reflectometer)
- Etch depth variation ~150Å

Closed loop etch depth with density correction Etch depth variation reduced to ~50Å

TCP Compensation R2R



 Closed loop TCP power compensation reduces with each successive run as chamber begins to season

Summary

- 1st evidence of real-time Poly-Si etch rate variation in Cl₂ due to F-exposure.
- 1st demonstration of ion density control in Cl₂ to compensate for Poly-Si real-time etch rate transients.
- Effluent SiCl₄ chemistry verifies both real-time performance drifts and feedback correction.
- Significant 1st wafer effect reduction after chamber cleans with density feedback control.
- Question: How Do We Explain the Results of Earlier Researchers?
 - Actinometry Results & Interpretations
 - Key Point Is That Even For Qualitative Conclusions, Actinometry/OES Results Must Be Carefully Analyzed Considering All Gasses Present In Chamber

Intensity Ratio I_{CI}/I_{Ar}

2 1.8 1.6 1.4 1.2 I_/I_Nominal I_/I_Ar OL 1 0.8 0.6 0 10 20 30 40 50 60 time(s)

I_/I_ Cl^ArNominal After F-disturbance, both controlled & uncontrolled cases show similar Clneutral suppression and recovery.
 Simple Conclusion is that Ions (not neutrals) control etch rate for this process.

λ_{Ar}: 750.4nm λ_{Cl}: 822.2nm

CI Intensity



Cl Intensity is Flat in Nominal/Seasoned-wall case & varies in Open Loop and Closed Loop Cases

Ar Intensity



I Nominal

- Intensity of Ar Being Nearly Flat Was Previously Taken By Some Researchers To Show that the Plasma Density Was Constant
- This led to the conclusion that neutral CI loss was responsible for Si etch rate variations
- We have shown that neither of these conclusions can be correct

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OES Setup Equations

d = Cl₂ dissociation fraction
 f_{Ar} = mole fraction of Ar in feed gas (5%)
 Mass balance: Cl₂ → 2dCl + (1-d)Cl₂

Raw optical intensity signals:

Detailed Look at Dissociation Diluation Effect on Ar

 $\underbrace{Cl_2}_{input \ gas \ mixture} \rightarrow \underbrace{2dCl + (1-d)Cl_2}_{gas \ composition \ in \ plasma}$ Chlorine Dissociation Now including the Ar actinometer concentration $\underbrace{\left(f_{Ar}\right)Ar + \left(1 - f_{Ar}\right)Cl_{2}}_{input \text{ gas mixture}} \rightarrow \underbrace{\left(f_{Ar}\right)Ar + 2d\left(1 - f_{Ar}\right)Cl + \left(1 - d\right)\left(1 - f_{Ar}\right)Cl_{2}}_{gas \text{ composition in plasma}}$ The concentration of Ar is diluted by Cl_2 dissociation So in the plasma, assuming all molecules, atoms, ions at the same temperature: $n_{Ar} = \left| \frac{f_{Ar}}{f_{Ar} + 2d(1 - f_{Ar}) + (1 - d)(1 - f_{Ar})} \right| n_{tot} = \left| \frac{f_{Ar}}{1 + d(1 - f_{Ar})} \right| n_{tot}$ $n_{Cl} = \left| \frac{2d(1 - f_{Ar})}{f_{L} + 2d(1 - f_{L}) + (1 - d)(1 - f_{L})} \right| n_{tot} = \left| \frac{2d(1 - f_{Ar})}{1 + d(1 - f_{L})} \right| n_{tot}$ Thus $\frac{n_{Cl}}{n_{+}} = \left| \frac{2d(1 - f_{Ar})}{f_{+}} \right| = 2d \left| \frac{(1 - f_{Ar})}{f_{Ar}} \right|$

OES Fits

- Clean Chamber / High Recombination Case Yields Actinometry Data with Enough Structure to Extract α_{Cl} ' & K_{Ar}' by Nonlinear Regression
- Dissociation Fractions for Other Runs Estimated by Assuming α_{CI} ' is the same as the Clean Chamber Result
 - Possible T_e variations Errors
 - Possible Window Variations

Fitting of OES Data

Fitting 2 constants allows quantitative extraction of d from OES data

$$\begin{split} I_{Ar} &= K_{Ar}(T_{e})\omega_{n}^{2}n_{Ar} = K_{1}\omega_{n}^{2}\left[\frac{f_{Ar}}{1+d\left(1-f_{Ar}\right)}\right]n_{tot} \\ I_{Cl} &= K_{Cl}(T_{e})\omega_{n}^{2}n_{Cl} = K_{2}\omega_{n}^{2}\left[\frac{2d\left(1-f_{Ar}\right)}{1+d\left(1-f_{Ar}\right)}\right]n_{tot} \\ \left[\frac{I_{Cl}}{I_{Ar}}\right] &= \left(\frac{K_{Cl}}{K_{Ar}}\right)2d\left(\frac{1-f_{Ar}}{f_{Ar}}\right) \rightarrow d = \frac{1}{2}\left(\frac{K_{Ar}}{K_{Cl}}\right)\left(\frac{f_{Ar}}{1-f_{Ar}}\right)\left[\frac{I_{Cl}}{I_{Ar}}\right]_{meas} \\ I_{Ar} &= K_{Ar}n_{tot}\omega_{n}^{2}\left[\frac{f_{Ar}}{1+\frac{1}{2}\alpha_{cl}\left(\frac{f_{Ar}}{1-f_{Ar}}\right)\left[\frac{I_{Cl}}{I_{Ar}}\right]_{meas}\left(1-f_{Ar}\right)}\right] = K_{Ar}n_{tot}\omega_{n}^{2}\left[\frac{f_{Ar}}{1+\frac{1}{2}\alpha_{cl}f_{Ar}\left[\frac{I_{Cl}}{I_{Ar}}\right]_{meas}}\right] = K_{Ar}'\omega_{n}^{2}\left[\frac{f_{Ar}}{1+\frac{1}{2}\alpha_{cl}f_{Ar}\left[\frac{I_{Cl}}{I_{Ar}}\right]_{meas}}\right] \\ I_{Cl} &= K_{2}(T_{e})\omega_{n}^{2}n_{Cl} = K_{Cl}n_{tot}\omega_{n}^{2}\left[\frac{\alpha_{cl}f_{Ar}\left[\frac{I_{Cl}}{I_{Ar}}\right]_{meas}}{1+\frac{1}{2}\alpha_{cl}f_{Ar}\left[\frac{I_{Cl}}{I_{Ar}}\right]_{meas}}\right] = K_{Cl}'\omega_{n}^{2}\left[\frac{\alpha_{cl}f_{Ar}\left[\frac{I_{Cl}}{I_{Ar}}\right]_{meas}}{1+\frac{1}{2}\alpha_{cl}f_{Ar}\left[\frac{I_{Cl}}{I_{Ar}}\right]_{meas}}\right] = K_{Ar}'\omega_{n}^{2}\left[\frac{f_{Ar}}{1+\frac{1}{2}\alpha_{cl}f_{Ar}\left[\frac{I_{Cl}}{I_{Ar}}\right]_{meas}}}{1+\frac{1}{2}\alpha_{cl}f_{Ar}\left[\frac{I_{Cl}}{I_{Ar}}\right]_{meas}}\right] = K_{Cl}'\omega_{n}^{2}\left[\frac{\alpha_{cl}f_{Ar}\left[\frac{I_{Cl}}{I_{Ar}}\right]_{meas}}{1+\frac{1}{2}\alpha_{cl}f_{Ar}\left[\frac{I_{Cl}}{I_{Ar}}\right]_{meas}}\right] = K_{Ar}'\omega_{n}^{2}\left[\frac{f_{Ar}\left[\frac{I_{Cl}}{I_{Ar}}\right]_{meas}}{1+\frac{1}{2}\alpha_{cl}f_{Ar}\left[\frac{I_{Cl}}{I_{Ar}}\right]_{meas}}\right] = K_{Ar}'\omega_{n}^{2}\left[\frac{f_{Ar}\left[\frac{I_{Cl}}{I_{Ar}}\right]_{meas}}{1+\frac{1}{2}\alpha_{cl}f_{Ar}\left[\frac{I_{Cl}}{I_{Ar}}\right]_{meas}}\right] = K_{Ar}'\omega_{n}^{2}\left[\frac{f_{Ar}\left[\frac{I_{Cl}}{I_{Ar}}\right]_{meas}}{1+\frac{1}{2}\alpha_{cl}f_{Ar}\left[\frac{I_{Cl}}{I_{Ar}}\right]_{meas}}\right] = K_{Ar}'\omega_{n}^{2}\left[\frac{f_{Ar}\left[\frac{I_{Cl}}{I_{Ar}}\right]_{meas}}{1+\frac{1}{2}\alpha_{cl}f_{Ar}\left[\frac{I_{Cl}}{I_{Ar}}\right]_{meas}}\right] = K_{Ar}'\omega_{n}^{2}\left[\frac{f_{Ar}\left[\frac{I_{Cl}}{I_{Ar}}\right]_{meas}}{1+\frac{1}{2}\alpha_{cl}f_{Ar}\left[\frac{I_{Cl}}{I_{Ar}}\right]_{meas}}\right]$$

Ar OES Signal & Fit: SiCl₄ Ignored



CI OES Signal & Fit: SiCl₄ Ignored



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Cl₂ Net Dissociation: SiCl₄ Ignored



Ar Fraction: SiCl₄ Ignored



 $I_{Ar}(t)$ ~const. due to opposing effects of dilution (\downarrow) & ne (\uparrow)

Dissociation Fractions: SiCl₄ Ignored



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Intensity Ratio I_{CI}/I_{Ar}

λ_{Ar}: 750.4nm λ_{Cl}: 822.2nm



Why is feedback controlled I_{Cl}/I_{Ar} still low? – <u>Our</u> <u>Next AVS Paper</u> : GENERATION of Cl Is Increased but COMSUMPTION by Si Etching & Dilution by SiCl₄ Offset Generation

Key Reactions

 $\begin{array}{ll} Cl_2 \rightarrow 2Cl & \text{Dissociation} \\ 2Cl \rightarrow Cl_2 & \text{Recombination (wall & bulk gas phase)} \\ Cl + e^- \rightleftharpoons Cl^+ + 2e^- & \text{Ionization & Bulk Deionization} \\ Si + 4Cl \rightarrow SiCl_4 & \text{Etch} \\ SiCl_4 + SiO_2 \rightarrow SiO_xCl_y + Cl + Cl_2 \\ SiCl_4 + Al_2O_3 \rightarrow Al_ySi_2O_xCl_y + Cl + Cl_2 \\ \end{array} \right\} \text{Deposition Reactions (unbalanced)}$

Simplified Reaction Set

Assuming Cl ionization and Si-species deposition Reactions have small effects on gas species concentrations, the other remaining reactions yield: $F_{Cl_2}Cl_2 + F_{Ar}Ar + F_{Si}Si \rightarrow xCl + yCl_2 + F_{Si}SiCl_4 + f_{Ar}Ar$ chamber gas phase molecules molecules in $\frac{1}{2}x + y + 2F_{Si} = F_{Cl_2}$ for Cl_2 mass balance $y = (1 - d) F_{Cl_2}$ where d = Net Dissociation Fraction of Cl_2 $F_{Si} = \{Si \text{ atoms/s consumed by etching}\}$ known from measured etch rate & flows So $x = \left\lceil 2dF_{Cl_2} - 4F_{Si} \right\rceil$

Result of Simplified Reaction Set

$$\begin{split} n_{g} &\propto x + y + F_{Si} + F_{Ar} \\ n_{g} &\propto 2dF_{Cl_{2}} - 4F_{Si} + (1 - d)F_{Cl_{2}} + F_{Si} + F_{Ar} \\ n_{g} &\propto (1 + d)F_{Cl_{2}} + F_{Ar} - 3F_{Si} \\ n_{Cl} &= \left[\frac{x}{x + y + F_{Si} + F_{Ar}}\right] n_{g} = \left[\frac{2dF_{Cl_{2}} - 4F_{Si}}{(1 + d)F_{Cl_{2}} + F_{Ar} - 3F_{Si}}\right] n_{g} \\ n_{Ar} &= \left[\frac{F_{Ar}}{x + y + F_{Si} + F_{Ar}}\right] n_{g} = \left[\frac{F_{Ar}}{(1 + d)F_{Cl_{2}} + F_{Ar} - 3F_{Si}}\right] n_{g} \\ I_{Cl} &= K_{Cl}n_{Cl}n_{e} \qquad I_{Ar} = K_{Ar}n_{Ar}n_{e} \\ \text{Measured Actinometry Ratio:} \\ \left[\frac{I_{Cl}}{I_{Ar}}\right]_{m} &= A_{m} = \frac{K_{Cl}S_{Cl}n_{Cl}n_{e}}{K_{Ar}S_{Ar}n_{Ar}n_{e}} = \frac{1}{\alpha_{Cl}}\left[\frac{2dF_{Cl_{2}} - 4F_{Si}}{F_{Ar}}\right] \end{split}$$

Cl Actinometry Signal Suppressed by Etch/Loading

$$PV = n_g RT_g \to n_g = \frac{PV}{RT_g}$$

Assume T_g is constant & $n_e = C \omega_{BB}^2$ where C is a portionality constant fixed during the etch run.

$$I_{Cl} = P\omega_{BB}^{2} \left[\frac{K_{Cl}^{'}\alpha_{Cl}^{'}A_{m}F_{Ar}}{F_{Cl_{2}} + \left(1 + \frac{1}{2}\alpha_{Cl}^{'}A_{m}\right)F_{Ar} - F_{Si}} \right]$$
$$= P\omega_{BB}^{2} \left[\frac{K_{Ar}^{'}A_{m}F_{Ar}}{F_{Cl_{2}} + \left(1 + \frac{1}{2}\alpha_{Cl}^{'}A_{m}\right)F_{Ar} - F_{Si}} \right]$$
$$I_{Ar} = P\omega_{BB}^{2} \left[\frac{K_{Ar}^{'}F_{Ar}}{F_{Cl_{2}} + \left(1 + \frac{1}{2}\alpha_{Cl}^{'}A_{m}\right)F_{Ar} - F_{Si}} \right]$$

 K_{Ar} & α_{Cl} are the only unknowns They can be extracted if there is sufficient variation in $I_{Cl}(t)$ & $I_{Ar}(t)$

Ar OES Intensity & Fit: SiCl₄ Included from RTSE



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CI OES Intensity & Fit: SiCl₄ Included from RTSE



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Ar Fraction : SiCl₄ Included from RTSE



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Dissociation Fraction : SiCl₄ Included from RTSE



Dissociation Fractions: SiCl₄ Included from RTSE



- Net Dissociation Fraction (d) Is Increased by Higher TCP Power in Closed Loop Run
- Net d is higher than estimated from procedure ignoring SiCl₄
- Wall Recombination Still Suppresses Cl, d

T_e (EEDF) Issue



With some assumptions which we believe are justified:

$$\left[\frac{\omega_n^4}{I_a}\right] = f(T_e \text{ only})$$

T_e for open loop case appears ~constant

 T_e is increased initially for closed loop case (constant α_{Cl} ' assumption may not be accurate)

Wall-State Effects Model

- n_{cl} reduced due to recombination on F-cleaned walls.
- n_{CI+} reduced due to lower availability of n_{CI} precursor. ER decreases due to lower ion bombardment.
- Real-time feedback control corrects for n_e≈n_{Cl+} losses by increasing T_e, but does not fully recover n_{Cl}.
- Model supports ion dominated etch of Si w/ Cl₂;
 n_{Cl+}⇔ER ≠ n_{Cl}. High n_{Cl} keeps surface Cl-saturated.
 ∴ ion bombardment is rate limiting step.
- Extracted d varies significantly, causing constant I_{Ar}.

HBr/Cl₂ Etches

- HCl Is Formed In Mixing Manifold By HBr/Cl₂ Reaction
- Collaboration With Stanford Group Shows Similar Plasma/Gas Chemistry Trends To Cl₂ Only Cases
 - HCl absolute concentration was measured by laser diode absorption
 - HCI Dissociation follows BB-RF/plasma density trends
 - Chamber cleaning suppresses dissociation of HCI & increases plasma density variation
- Open Loop Etch Rates Become More Constant With Increasing HBr & Show Less Sensitivity to Chamber Wall Condition
- Closed Loop Plasma Density Control Causes More Time Variation In Etch Rate for High HBr Concentration Cases
- HBr/Cl₂ Etch Rates Are Not Directly Ion Limited & Future Work is Needed
 - Wafer Surface Temperature?

HBr/Cl₂ Etch (80/20)

Open Loop

Closed Loop



Future Work

<u>Modeling of BB Signals to extract more from the</u> <u>shape of the data</u>

- <u>collision parameters</u>
- Possible T_e/EEDF Information
- Improved antenna designs for BB System
- Lower-cost electronics for BB reflectometry
- Apply density control to topography & profile variations.
- Expand to other ion-dominated etches besides Cl₂ etching of Poly-Si.
- Larger scale, multi-wafer tests to verify control improvements.
- Ion density control most effective when etch is ion dominated. Chemically dominated etches do not show same effects.
- Combine ion density control with ion energy control.

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