Plasma Diagnostics for Processing Applications

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PPPL School on Plasmas for Microelectronics and Quantum Information Science, July 28 - August 1, 2025

[2025 PPPL Graduate Summer School]

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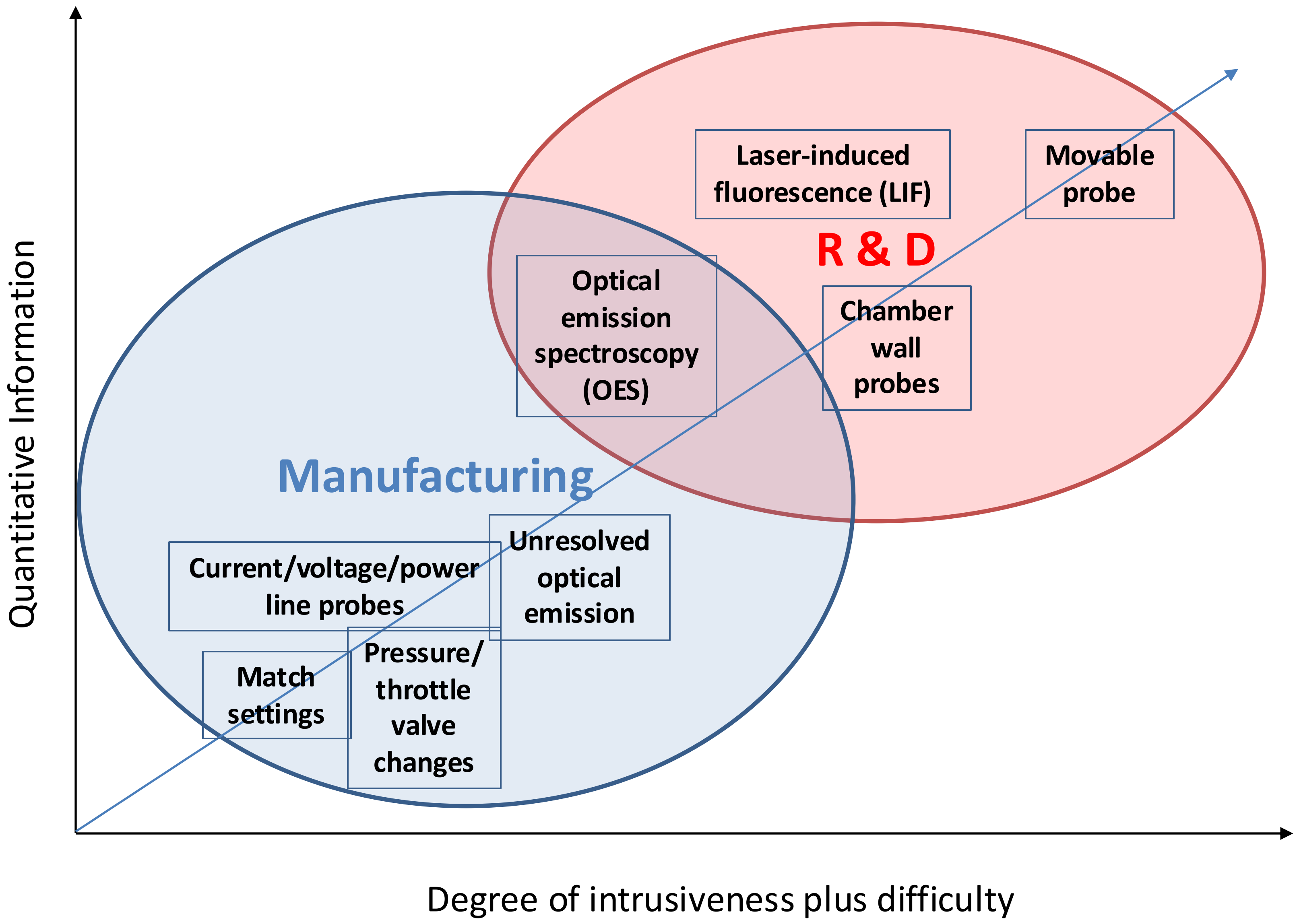
Trade-Off Between Information and Ease of Implementation



Electrical Probe Techniques

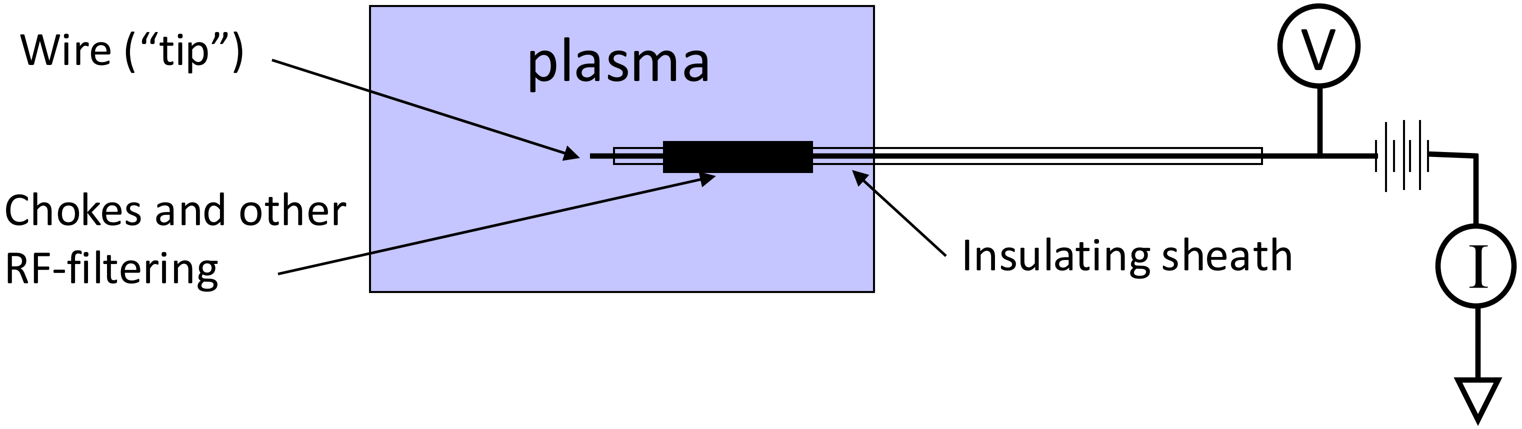
- ***Single Langmuir probe***
  - ❖ Most common probe
  - ❖ Several commercial Langmuir probes are available
  - ❖ Measures plasma potential (V_p), floating potential (V_f), electron density (n_e), total positive ion density (n_i^+), electron temperature (T_e) and electron energy distribution function (EEDF).

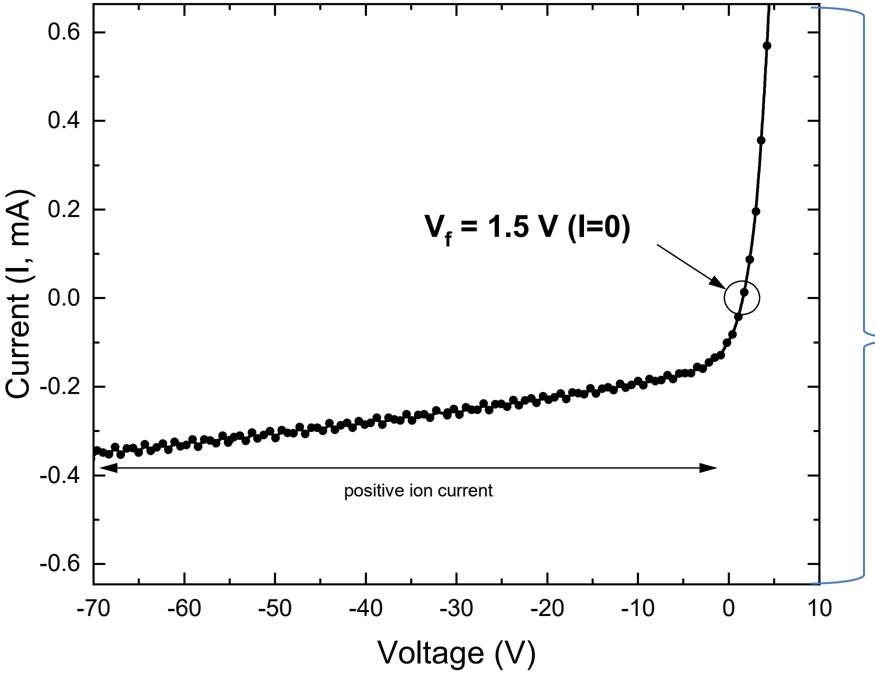
- ***Double probe***
  - ❖ Measures current as a function of voltage between two floating probes.
  - ❖ Used to obtain n_i^+ and T_e (high energy tail) in systems with no ground return path.

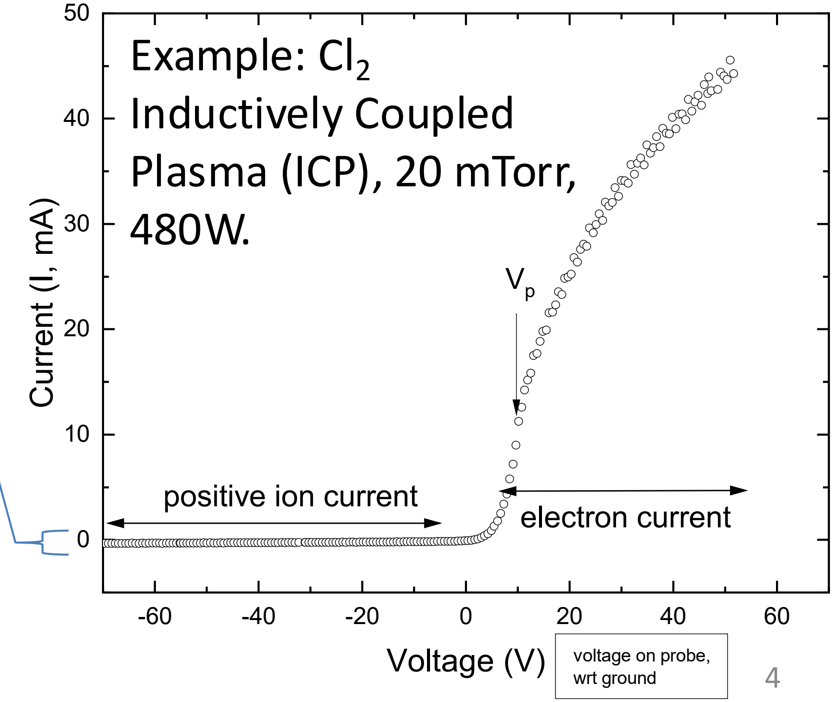
- ***Emissive probe***
  - ❖ Often used to measure V_p in systems with no ground return path.

- ***Hairpin probe***
  - ❖ Plasma-induced shift in μ-wave resonance frequency yields n_e and T_e. Can be used in systems with no ground return path.

Single Langmuir Probe Plasma Diagnostic Method



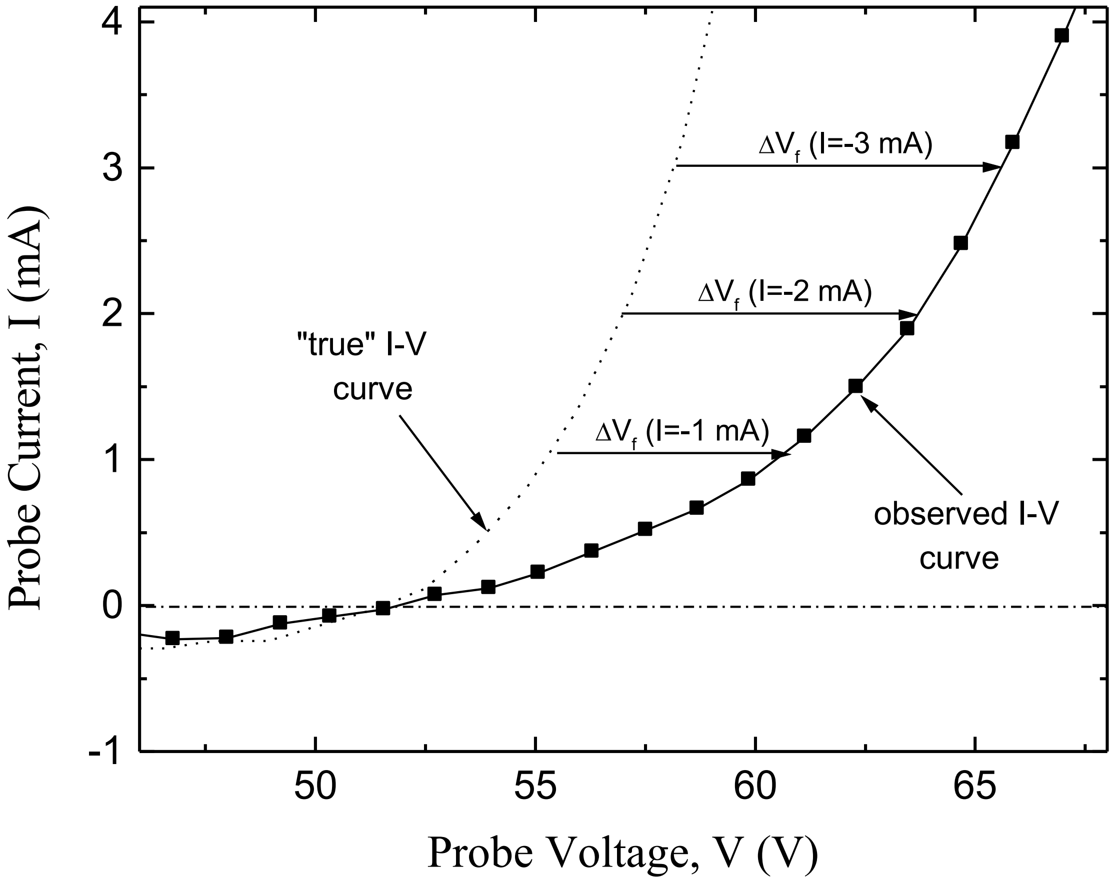




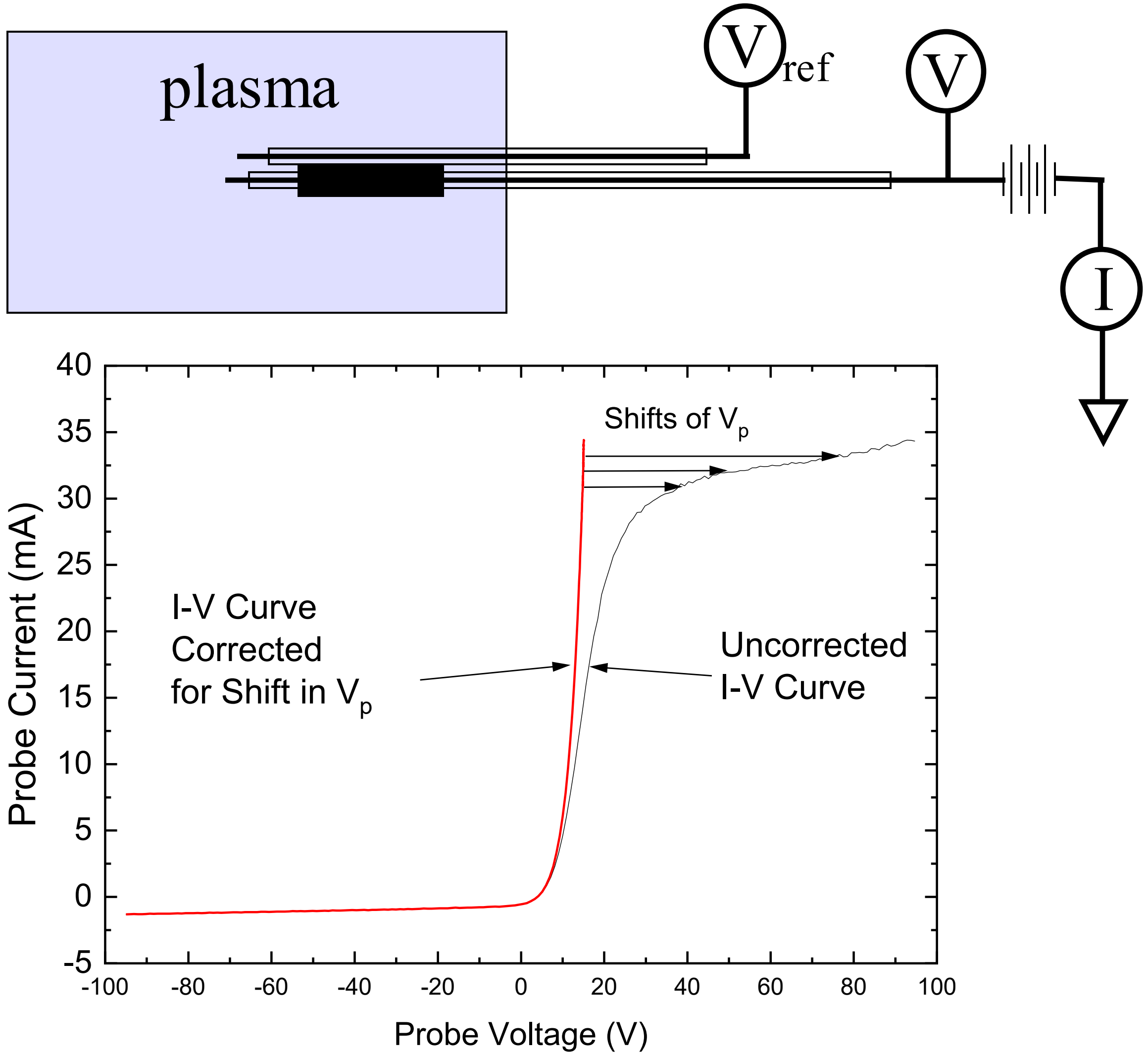
See Appendix 1 for how to extract EEDF from IV curves

Problem with Single Langmuir Probe: Shift V_p during I-V collection.

- Most commercial reactors lack a good electrical reference (i. e. ground) potential.
- Consequently, V_p increases during collection of electron currents.
- This causes a drastic overestimate of the true V_p, as well as T_e.



Solution: Insert a reference electrode into the plasma (usually attached to the Langmuir probe) to measure the shift in V_p



Why Probes are Problematic for Processing Plasmas

- Most detailed published studies are for Ar plasmas; virtually all processes use corrosive and/or depositing gases.

- Probe characteristics are distorted by contamination on probe tips.

- It is difficult to clean single and double probes in these processes in commercial reactors.

- Probes are intrusive and can perturb the process.

- Probes are feared to contaminate product wafers being processed.

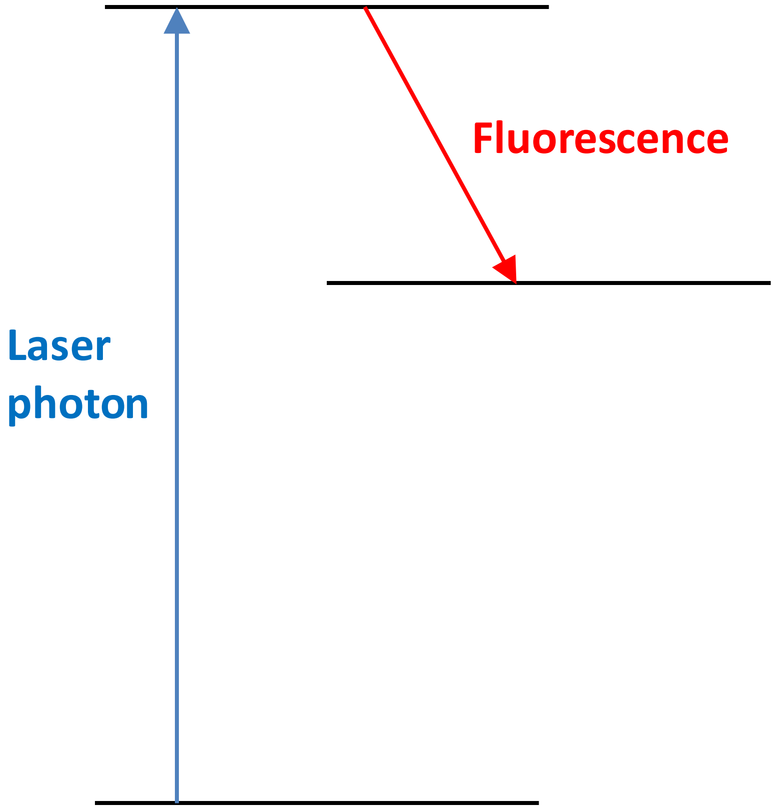
- Interpretations of measurements are complicated.

- Consequently, probes are most valuable for plasma processing research, plasma etching and deposition tool development, and some early process development.

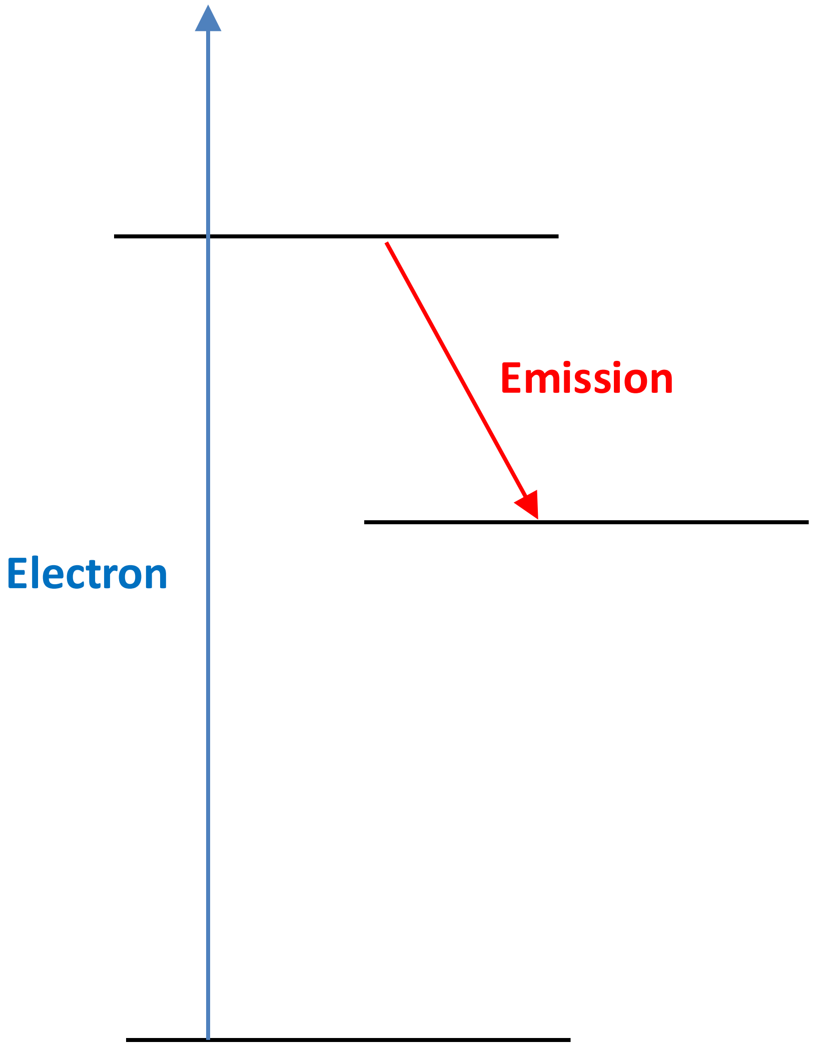
Light-Based Diagnostic Techniques

- ***Microwave Interferometry***
  - ❖ Measures line-integrated n_e, requires 2 large opposing viewports.
- ***Optical Absorption Spectroscopy***
  - ❖ UV-visible absorption (broad band and resonance lamps) for line-integrated neutral number densities.
  - ❖ Cavity ring-down spectroscopy: multi-pass laser absorption for line-integrated number densities.
- ***Laser Induced fluorescence (LIF) and scattering***
  - ❖ Simple LIF: Measures 3D relative number densities of atomic and molecular (mostly diatomics and some tri-atomics) neutrals and ions .
  - ❖ Intermediate LIF: Absolute number densities with calibrations.
  - ❖ Advanced:
    - a) Thomson scattering: measure scattering of laser photons by electrons to obtain n_e, T_e and EEDF.
    - b) Electric field measurements.
    - c) Others
- ***Optical Emission Spectroscopy (OES)***
  - ❖ Spectroscopic analysis of plasma-induced light from neutrals and ions.

Laser-Induced Fluorescence (LIF)



Optical Emission Spectroscopy (OES)



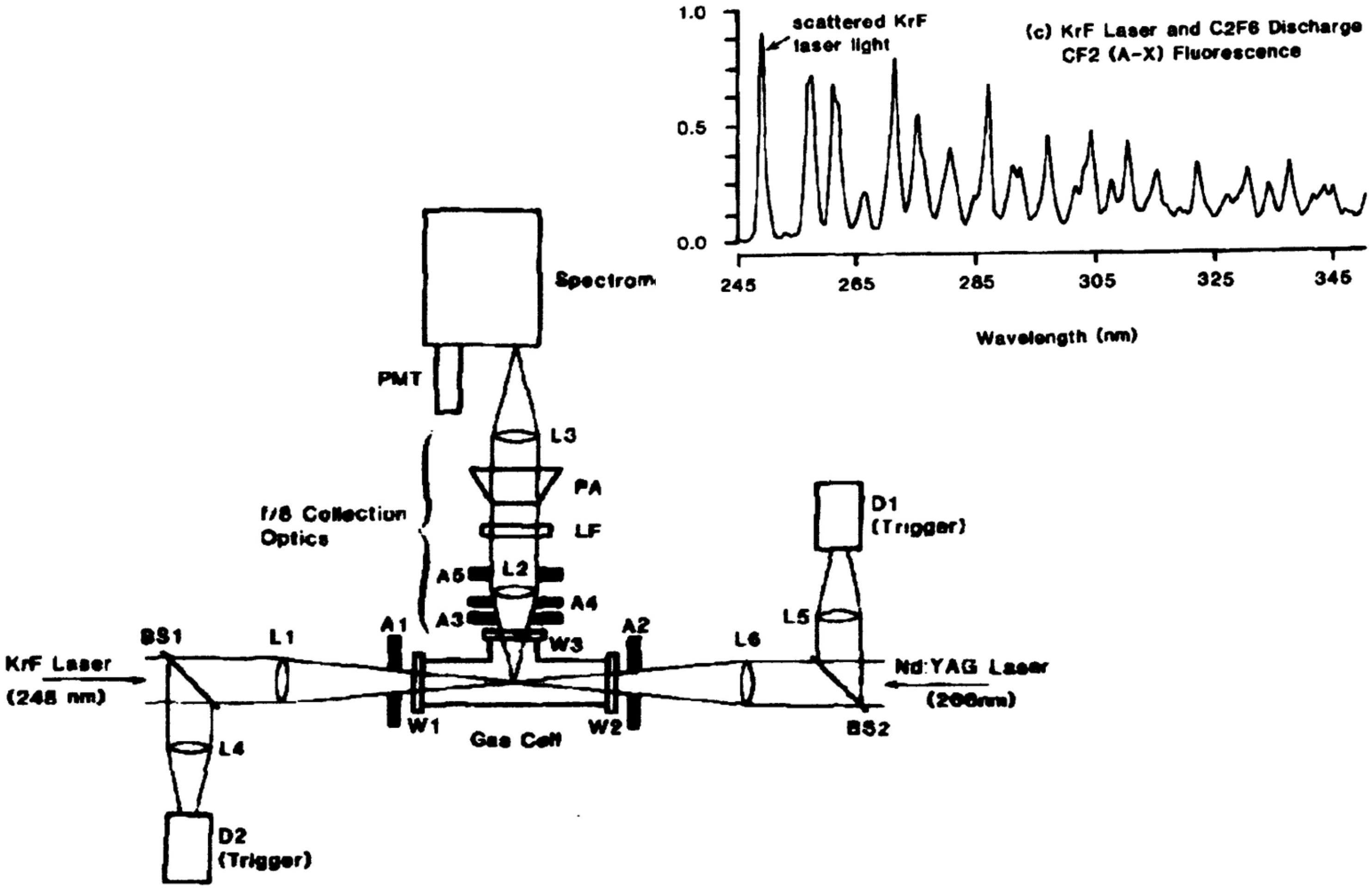
NOTE: Fluorescence = Emission

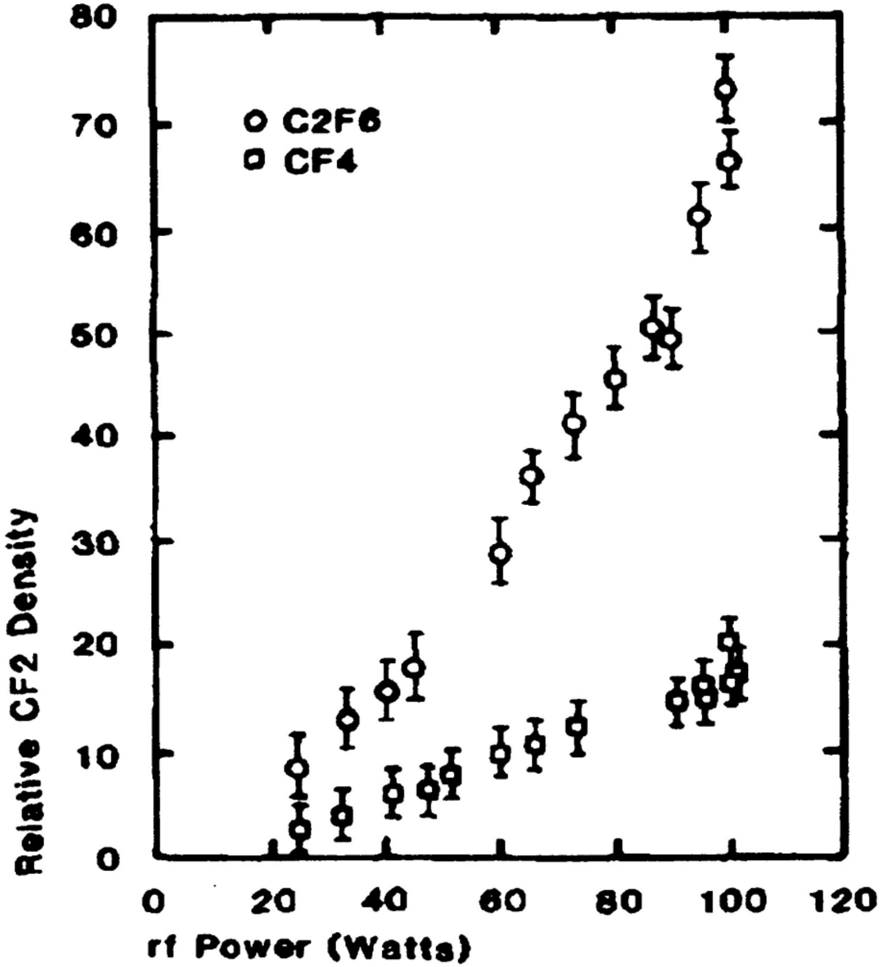
Detection of CF_2 radicals in a plasma etching reactor by laser-induced fluorescence spectroscopy

P. J. Hargis, Jr. and M. J. Kushner[a)] Appl. Phys. Lett. 40(9), 1 May 1982

Sandia National Laboratories, Albuquerque, New Mexico 87185

(Received 7 December 1981; accepted for publication 24 February 1982





Laser diagnostics of plasma etching: Measurement of Cl_2^+ in a chlorine discharge

V. M. Donnelly, D. L. Flamm, and G. Collins[a)] J. Vac. Sci. Technol. 21(3), Sept/Oct. 1982.

Bell Laboratories, Murray Hill, New Jersey

(Received 22 March 1982; accepted 11 May 1982)

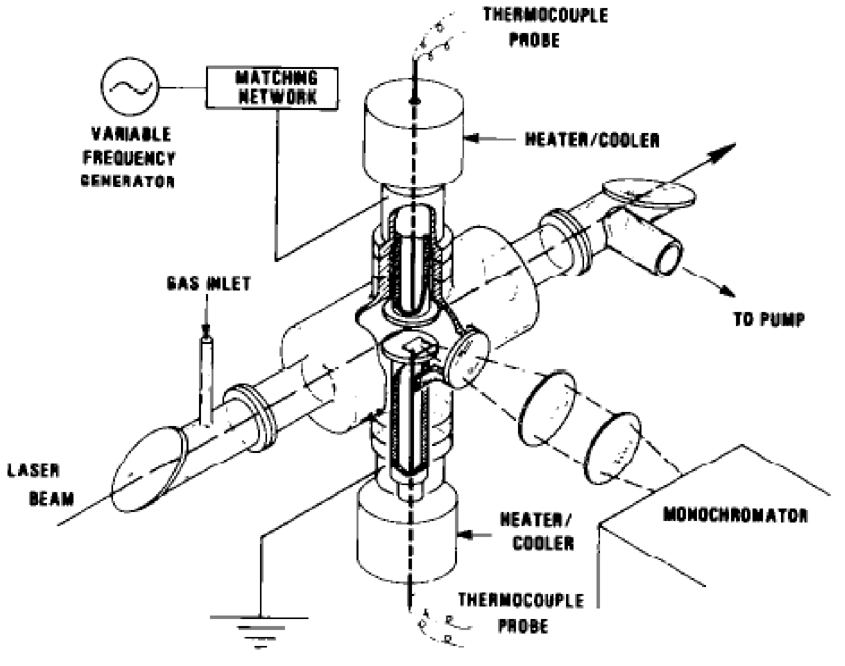
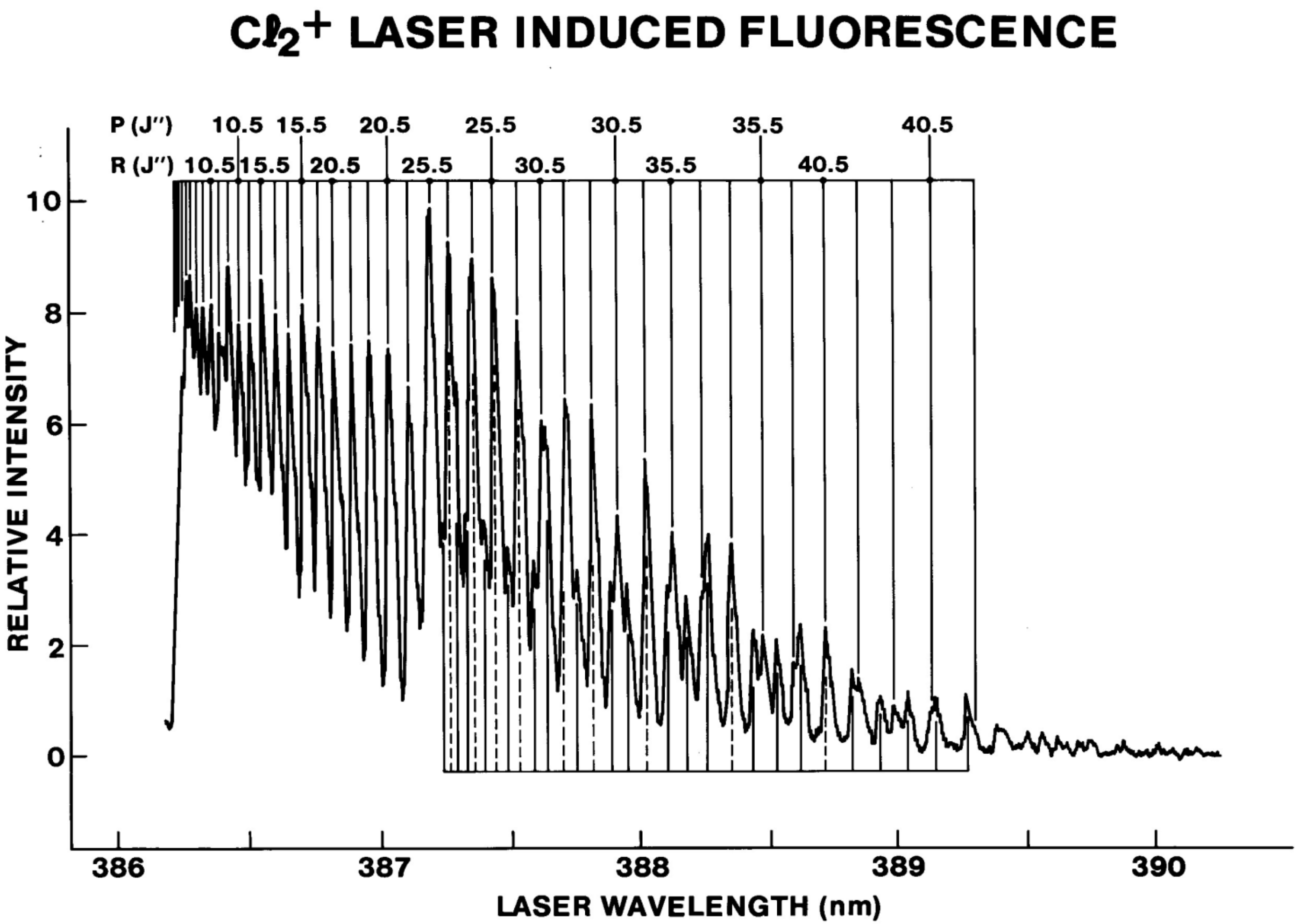


FIG. 1. Plasma reactor with variable frequency generator (0.01–25 MHz) and laser-induced fluorescence detection.



- First use of LIF – 1971 – $NO_{2(g)}$
  (K. Abe, et al. J. Molec. Spectros. 38, 552 (1971).)
- "LIF" – coined by Richard Zare –Stanford

Combining LIF and Langmuir Probe to Measure Cl^+ and Cl_2^+ Number Density vs. Power

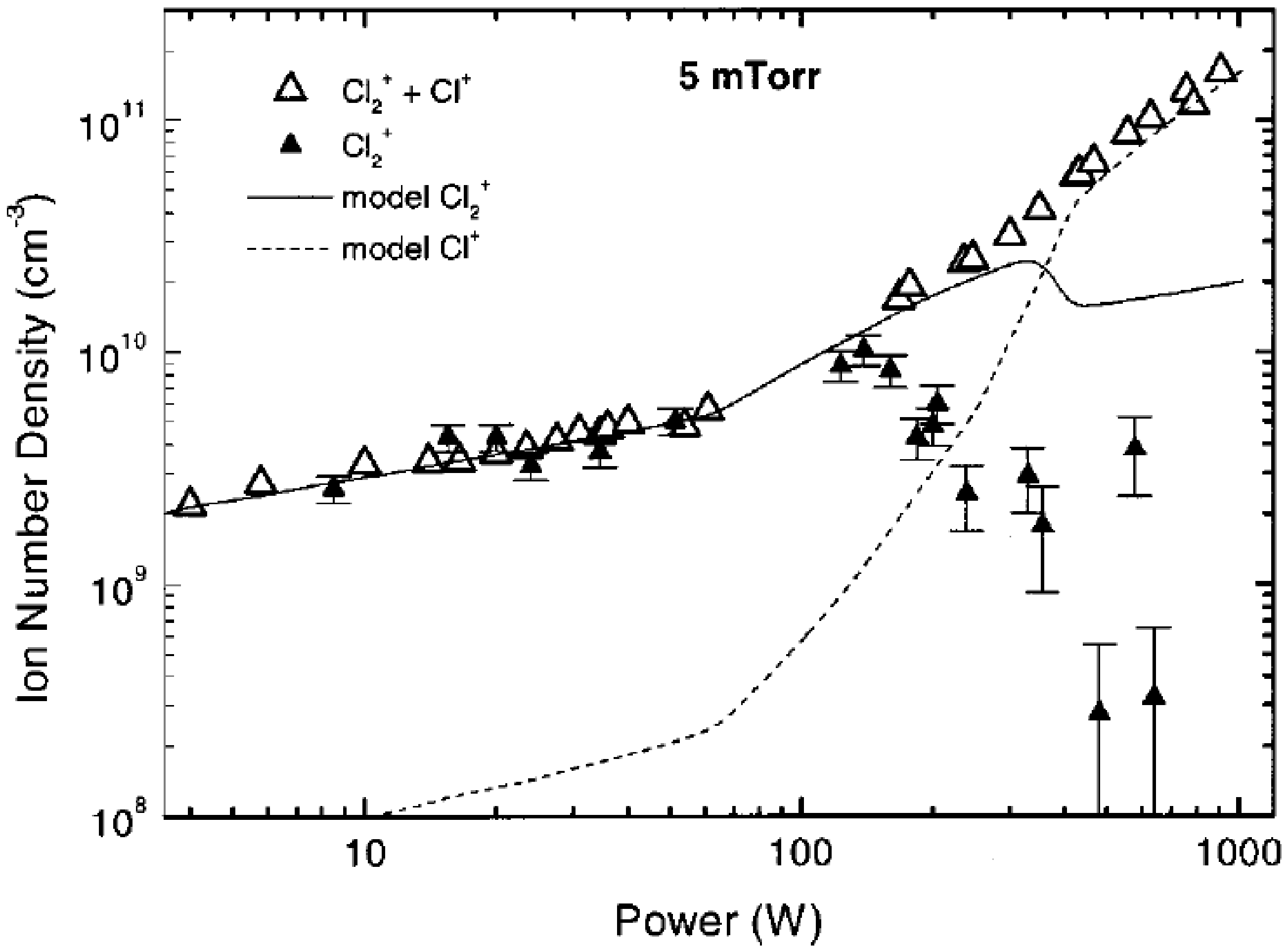
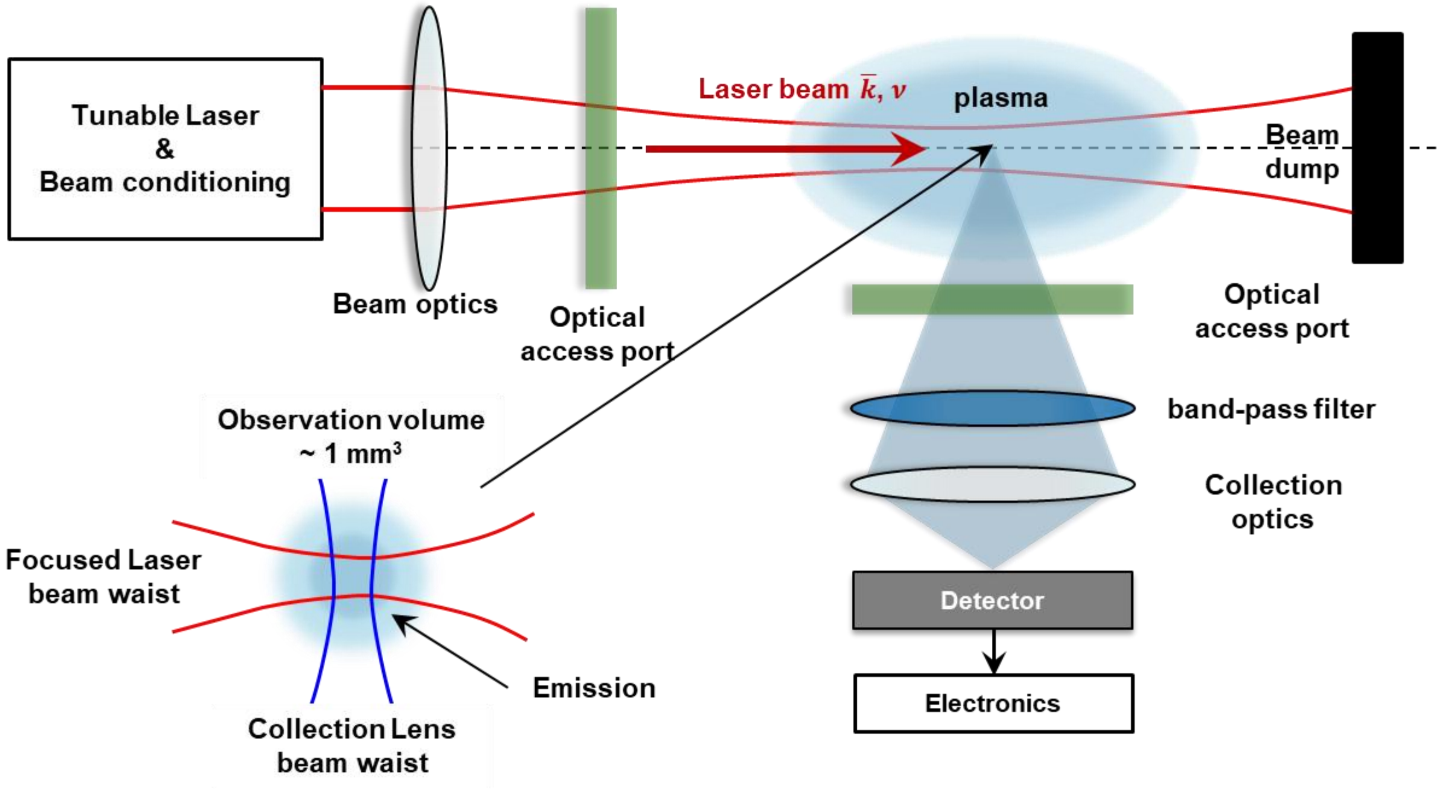


FIG. 4. Total positive-ion density, density of Cl_2^+, and modeled densities of Cl_2^+ and Cl^+ as functions of rf power for a Cl_2 plasma at 5 mTorr.

Conventional LIF configuration

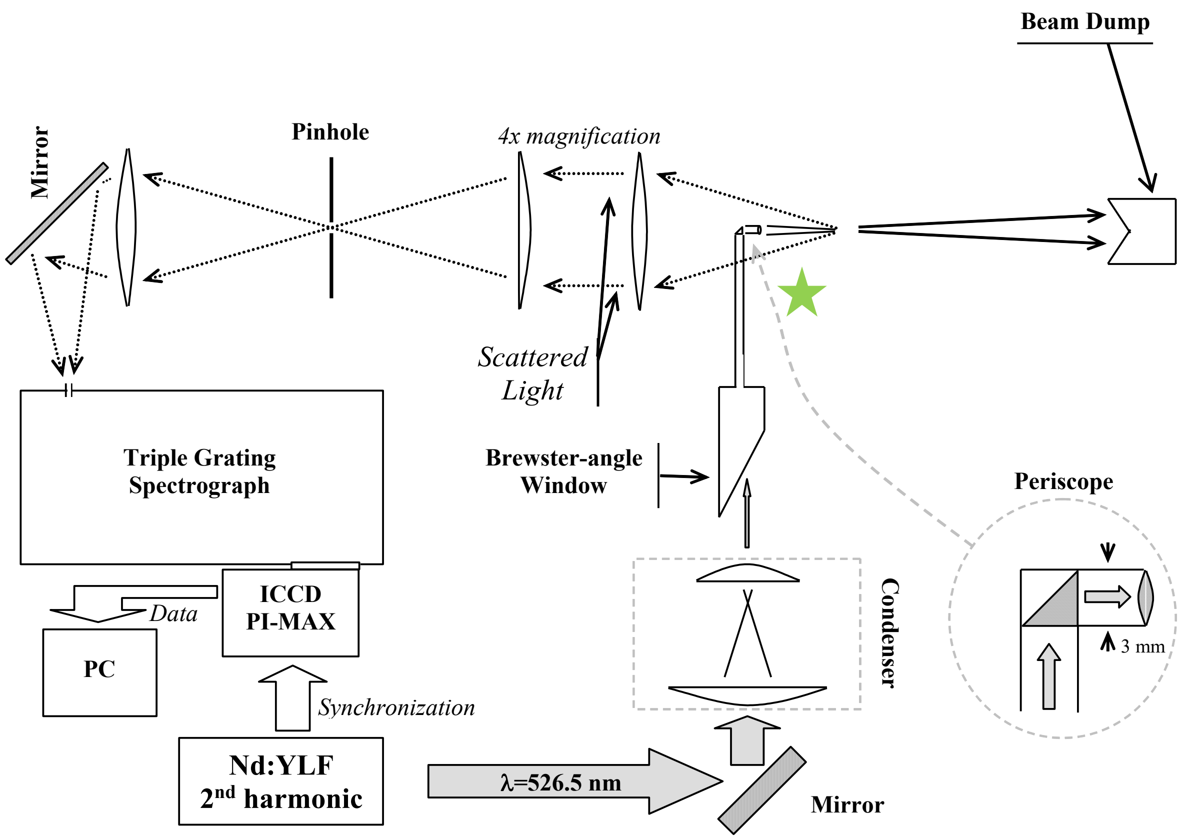
Slide courtesy of Yevgeny Raitses



- Best spatial resolution (sub-mm range) at $\pi/2$ fluorescence collection
- **Minimum of two optical ports for operation**

Confocal – need only 1 viewport

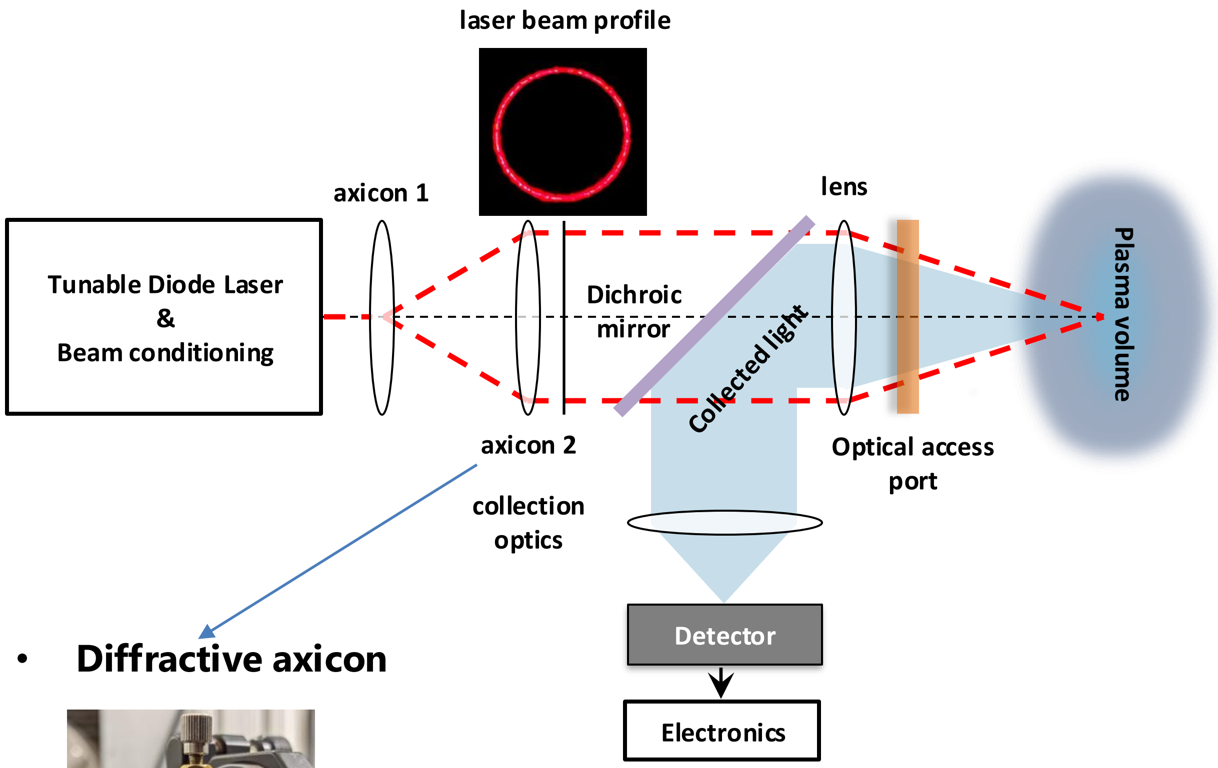
Confocal Raman/Thomson Scattering



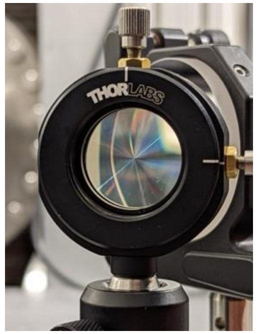
Belostotskiy, et. al., Appl. Phys. Lett. ***92****, 221507 (2008).*

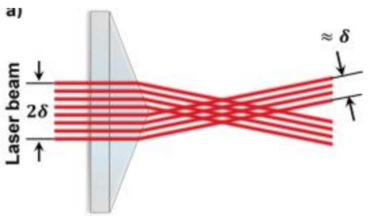
Confocal LIF

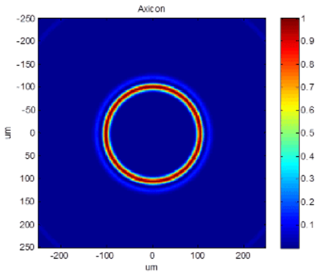
Slide courtesy of Yevgeny Raitses



- **Diffractive axicon**

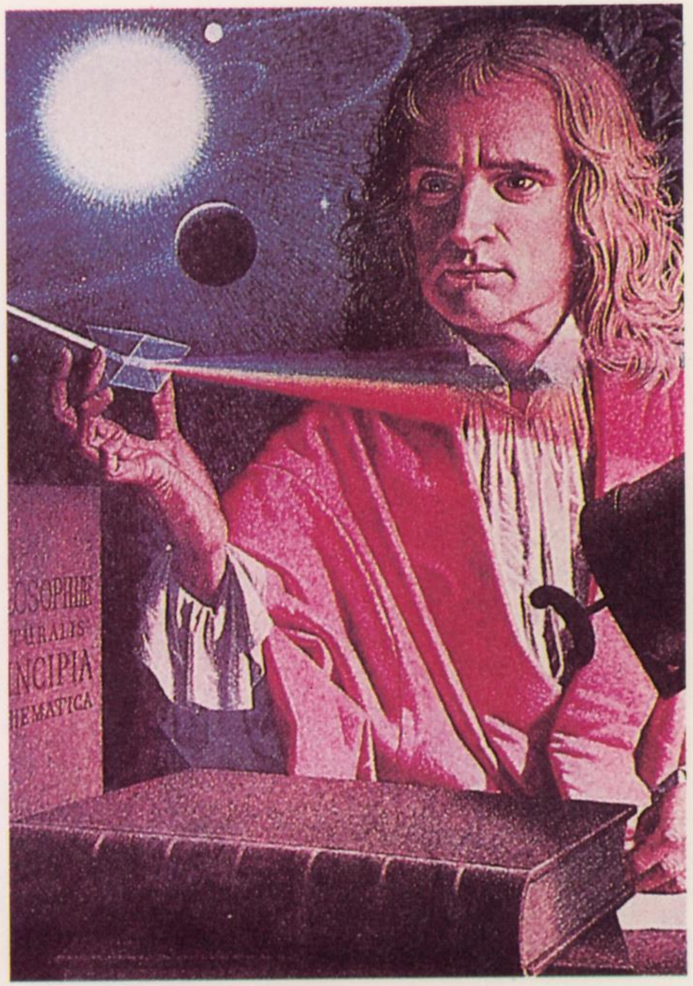






I. Romadanov & Y. Raitses, Rev. Sci. Instrum. ***94*** *(2023)*

Optical Emission Spectroscopy (OES)



Isaac Newton. Painting by Jean-Leon Huens, © National Geographic Society.

Plasma Optical Emission Spectroscopy

• Optical emission spectroscopy (OES) is one of the most widely used diagnostic technique in plasma processing, including in manufacturing for ***end-point detection***, first used by Harshbarger, *et al.* in 1977 to study a CF_4/O_2 plasma during Si etching.

• Optical emission in plasmas is a mostly from electron-impact excitation.

• Most atoms and diatomics can be monitored, as well as some triatomics (e.g. CF_2, $SiCl_2$, CO_2^+).

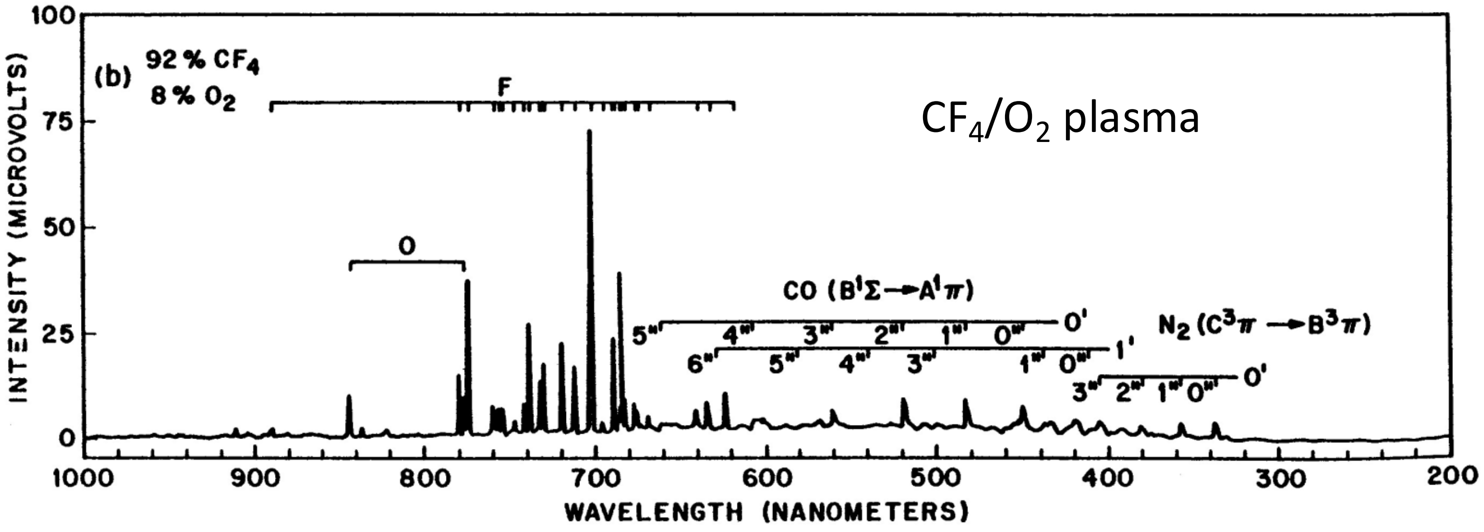
• Emission from larger molecules is either lacking, or broad and featureless.

• The complex excitation mechanism, makes it is difficult (but not impossible, see “actinometry” below) to derive relative and absolute species number densities from OES, hence it is generally qualitative.

"A Study of the Optical Emission from an rf Plasma during Semiconductor Etching"

Applied Optics, 31, 201 (1977). **W. R. HARSHBARAR, R. A. PORTER, T. A. MILLER, and P. NORTON**

Bell Telephone Laboratories Inc. Allentown, PA and Murray Hill, NJ



CF_4/O_2 plasma

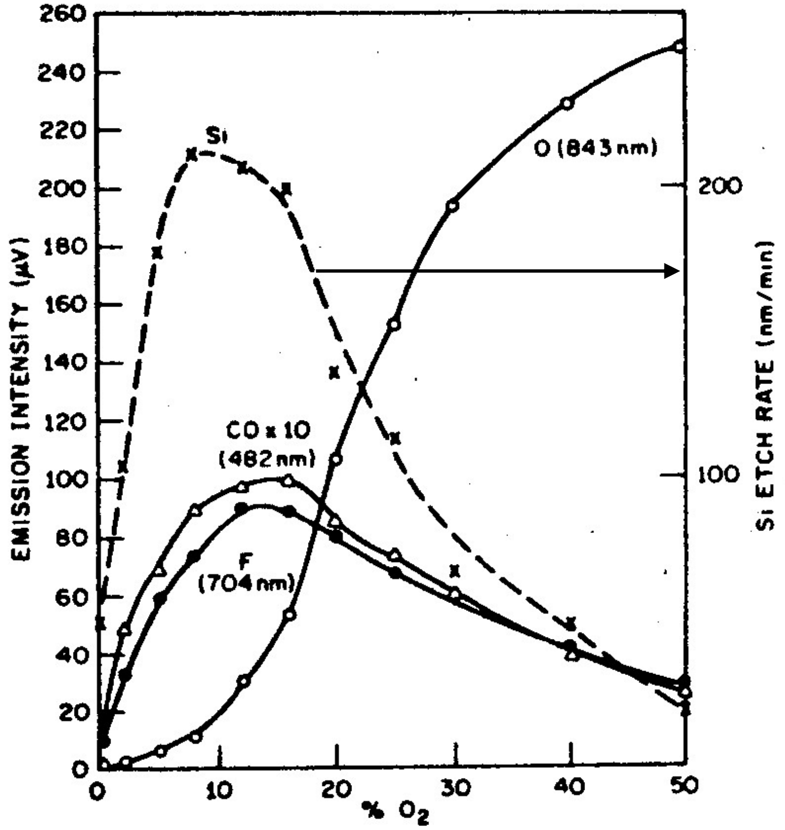


FIG. 3. Emission intensity and silicon etch rate as a function of percent O_2 concentration.

- Adding O_2 to CF_4 plasma greatly increases F atom density.
- Si etching rate also increases and peaks before F peaks.
- Provided great insights into plasma chemistry and etching mechanism.

Basic Requirements for OES

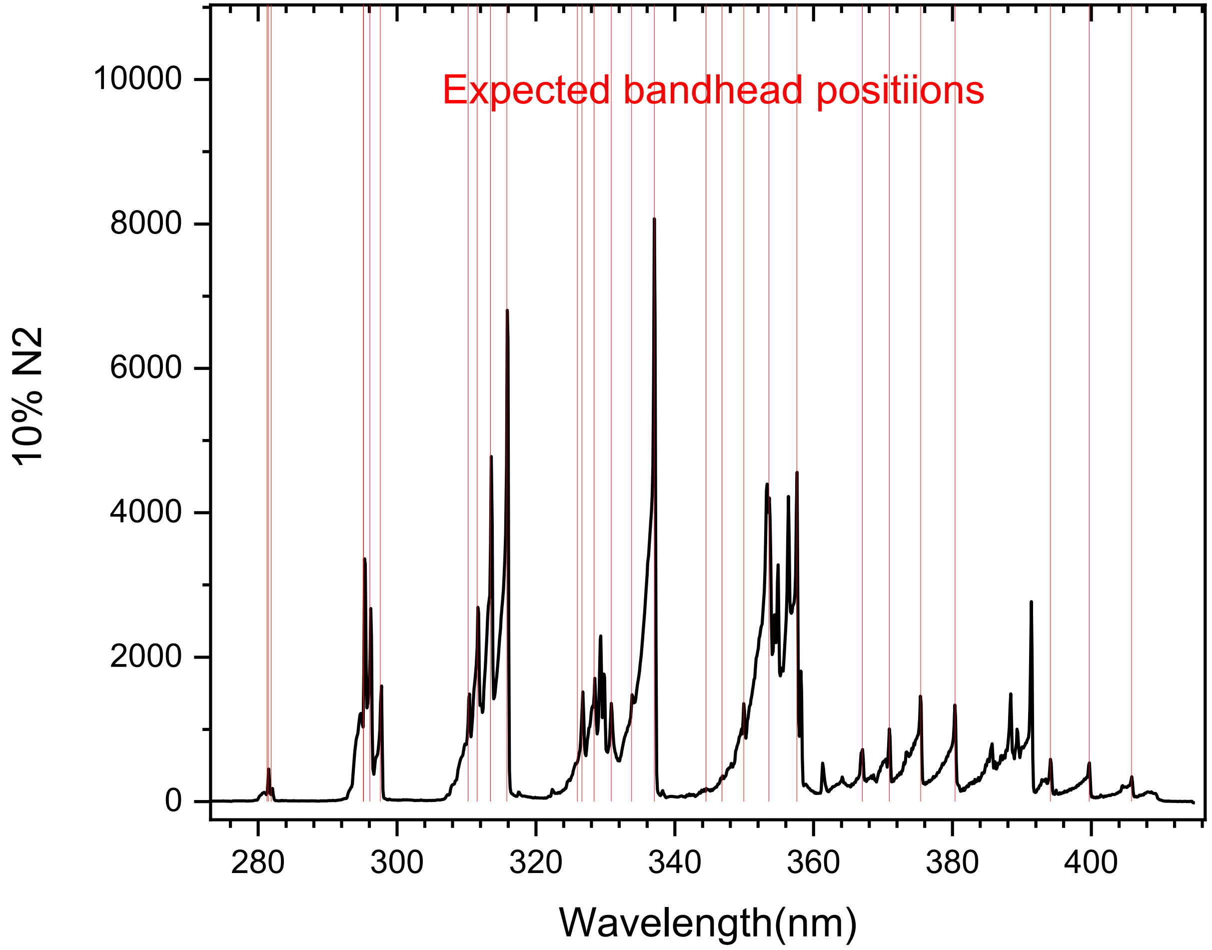
- ***Spectrometer***
  - Decent resolution (0.5 nm or better)
  - Spectral range: 200 to 900 nm.
  - Detector: CCD is sensitive enough, and can follow changes as fast as 0.05 s. Intensified CCD (ICCD) provides ns resolution and higher sensitivity, if needed.
- ***Reactor***
  - **Single UV-grade fused silica or sapphire viewport is adequate.**
- ***Reference Data***
  - Atomics: NIST Atomic Spectra Data base. https://www.nist.gov/pml/atomic-spectra-database
  - Diatomics: "Molecular Spectra and Molecular Structure: Constants of Diatomic Molecules", Huber and Herzberg, Van Nostrand, New York, 1979; "Spectroscopic Data" Part 1: Heteronuclear Part 2: Homonuclear Diatomics, Suchard and Melzer, Springer 1976; "The Identification of Molecular Spectra", Pearce and Gaydon, 4th Edition, Chapman and Hall, London/Wiley, New York, 1976.
- ***Expertise***
  - Considerable experience is necessary. Many assignments in the plasma processing literature are questionable.

How to Correctly Identify Peak in Optical Emission Spectra

(*necessary but not sufficient)*

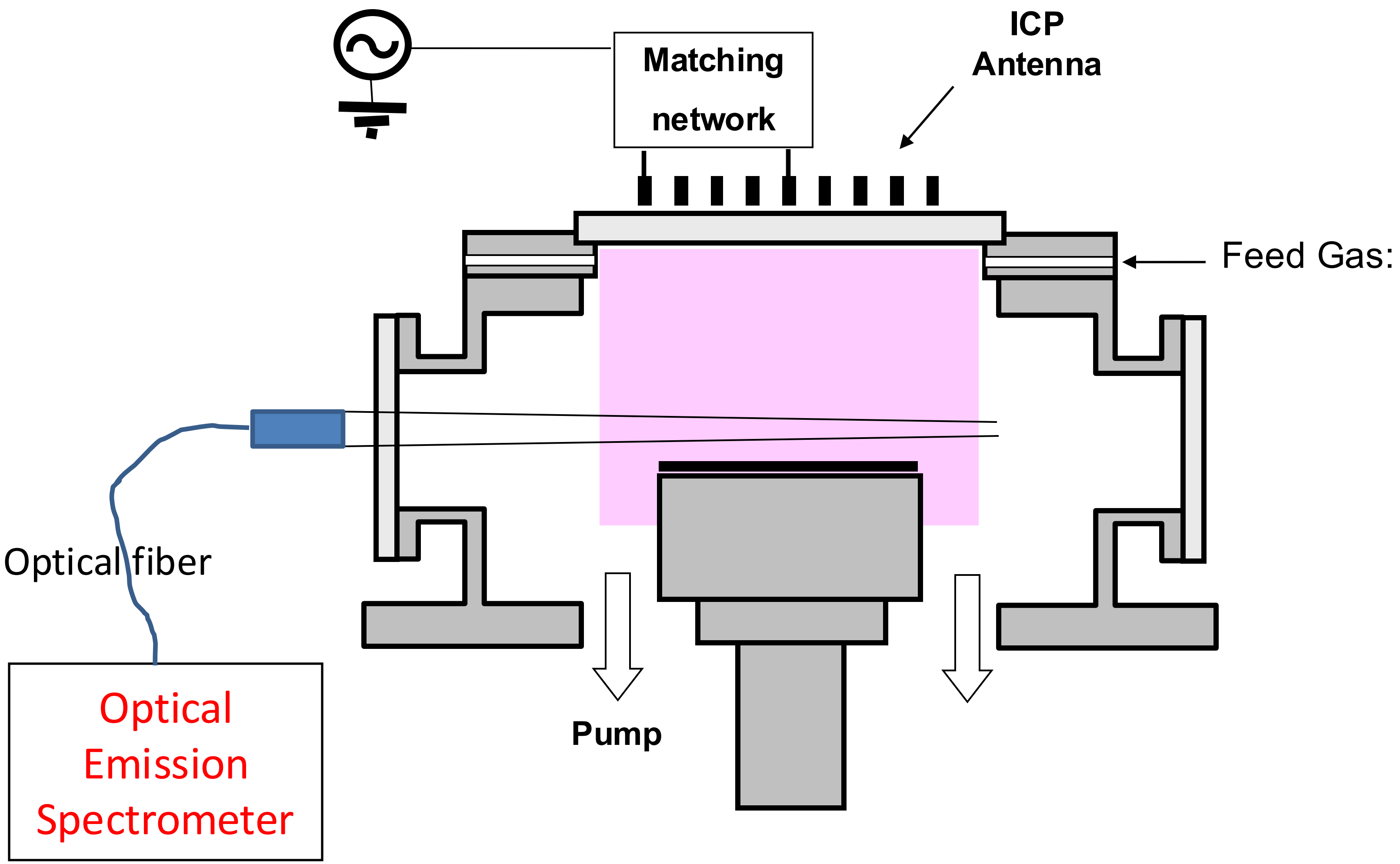
- Calibrate the wavelengths of your spectrometer as accurately as possible.
- Go to the primary source for known emission wavelengths (see previous slide).
- Atomic lines are narrow and symmetric (only as good as the spectrometer).
- Molecular lines are almost always asymmetric, degraded either toward the red or the violet direction.
- If one feature is assigned to a species, then other emissions from that species must also be present with relative intensities similar to tabulated values.

Example 1: $N_2(C^3\Pi_u \rightarrow B^3\Pi_g)$ Emission in N_2 Plasma



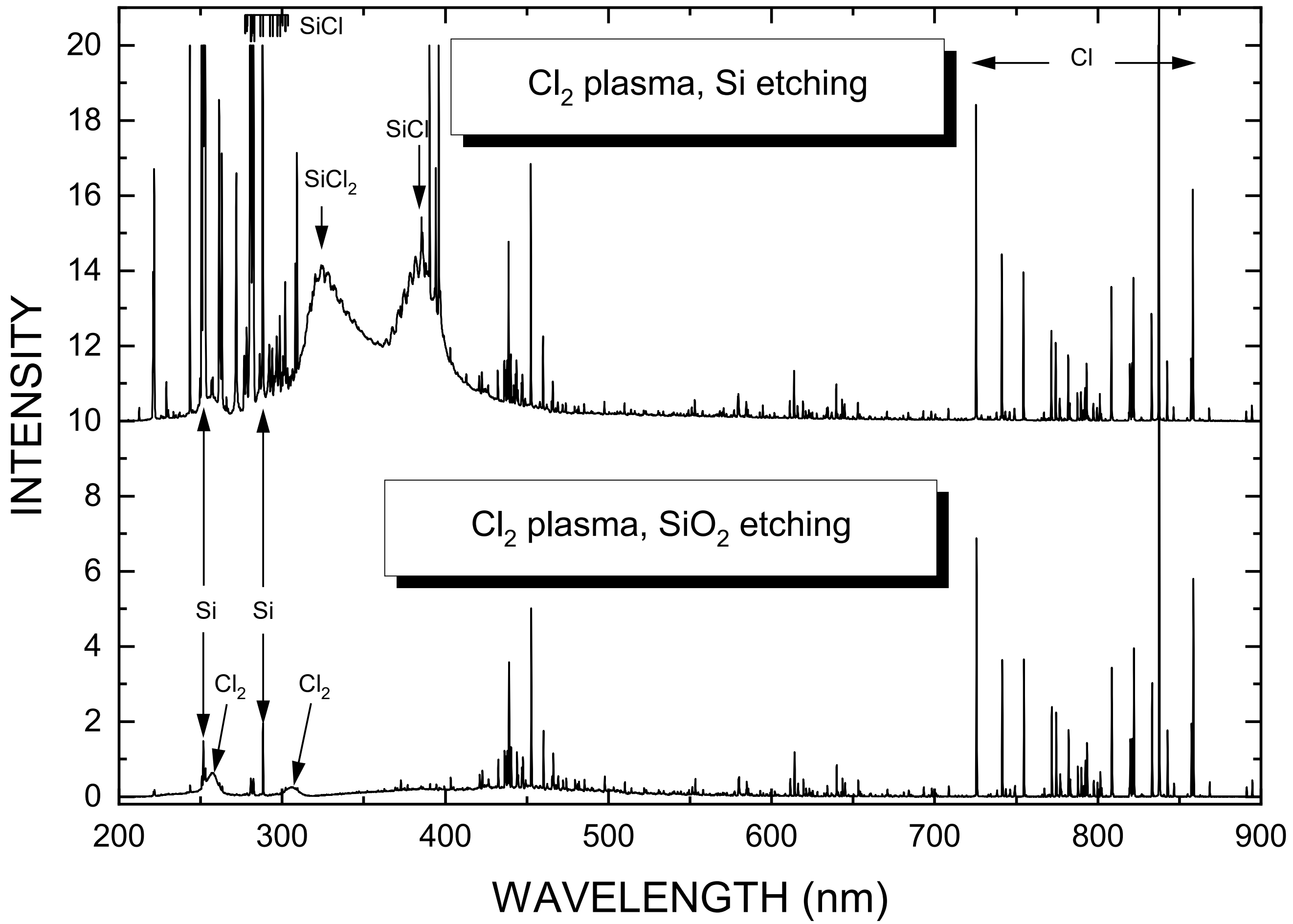
- Bands are violet-degraded.
- Band progressions due to combinations of transitions between vibrational levels of the upper and lower electronic states.

Optical Emission Spectroscopy Setup



Optical Emission Spectra of a Cl_2 Plasma Etching of Si and SiO_2

- Spectra are dominated by emission from Cl.
- Etching Si with substrate stage RF-bias, strong Si, SiCl, and $SiCl_2$ emissions are observed.
- Emission from Cl_2 is also apparent in the spectrum recorded during slow etching of SiO_2.



OES with Actinometry

(see Appendix 2 for details)

• Add a rare gas, A, (usually Ar) to the feed gases, at a known number density n_A.

• If the electron impact excitations cross sections, $\sigma_X(\varepsilon)$ *and* $\sigma_A(\varepsilon)$ for the species of interest, X, and A have similar relative energy, ε, dependences, then

$$n_X = a_{X,A} \, n_A \, (I_{X,i,j,k} / I_{A,i,j,k})$$

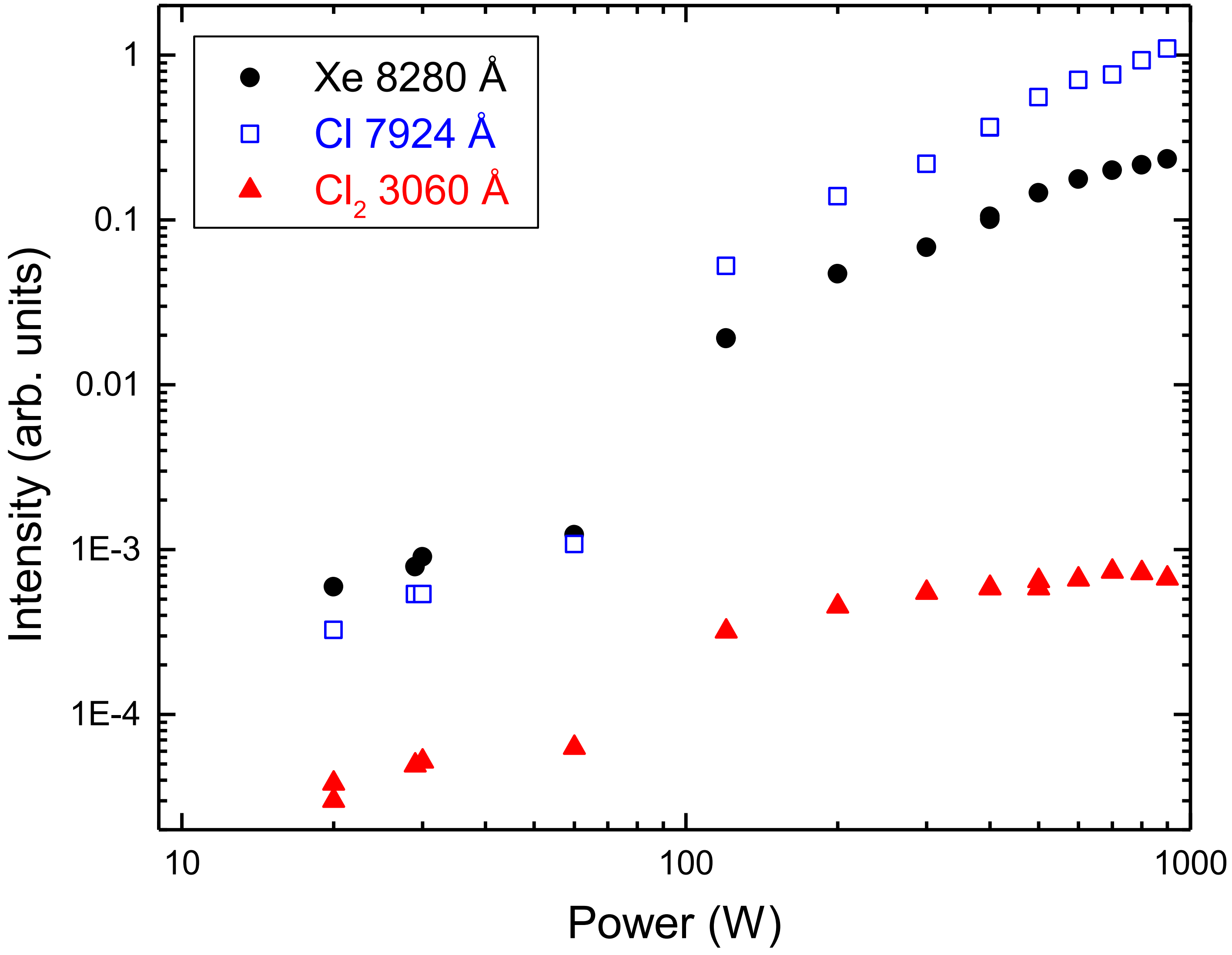
where $a_{X,A}$ is a constant, proportional to $\sigma_X(\varepsilon)$ *and* $\sigma_A(\varepsilon)$ and other factors.

• Relative densities of atoms (F, Cl, Br, H, O) and small molecules (Cl_2, CF, CF_2, BCl, N_2, CO) have been determined by this method, with varying degrees of accuracy.

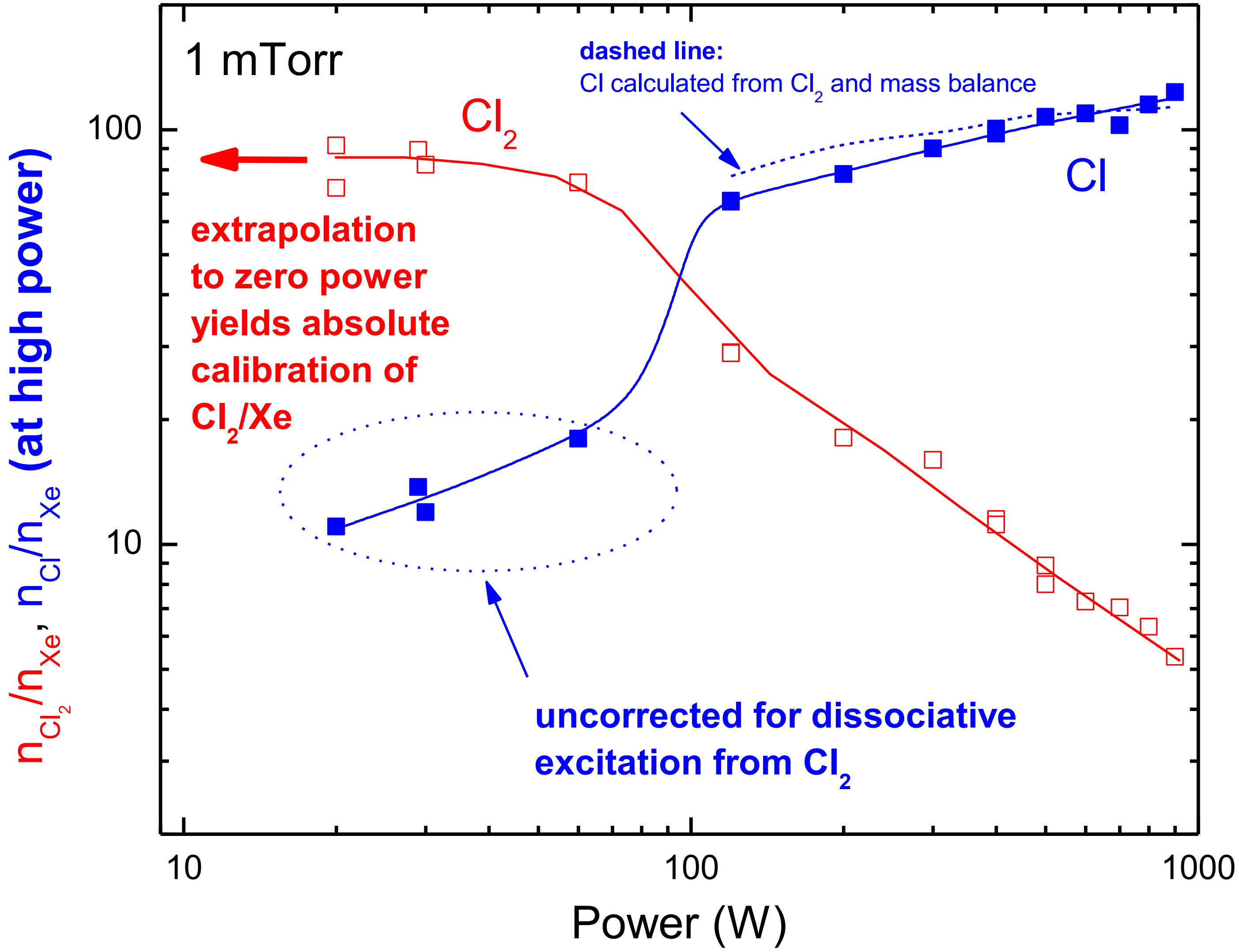
• In some cases, absolute number densities have also been measured with calibration methods.

• Lets look at an example.

OES Emission Intensities: 1mTorr Cl_2/5% rare gases plasma



Absolute Number Density Ratios of Cl_2/Xe at low power and Cl/Xe at high power



Added Problems in Quantifying OES: Dissociative excitation and metastables, e.g. Cl_2 Plasma

Cl_2 emission:

$$Cl_2 + e \rightarrow Cl_2(4s\sigma_g{}^1\Pi_g) + e \qquad E_{th} = 8.4eV$$

$$Cl_2(4s\sigma_g{}^1\Pi_g) \rightarrow Cl_2({}^1\Pi_u) + h\nu, 3060\mathring{A}$$

Cl emission:

$$Cl + e \rightarrow Cl(4p^2P^0, J = 3/2) + e \qquad E_{th} = 10.59eV$$

$$Cl_2 + e \rightarrow Cl + Cl(4p^2P^0, J = 3/2) + e \qquad E_{th} = 13.0eV$$

$$Cl(4p^2P^0, J = 3/2) \rightarrow Cl(4s^4P, J = 1/2) + h\nu, 7924\mathring{A}$$

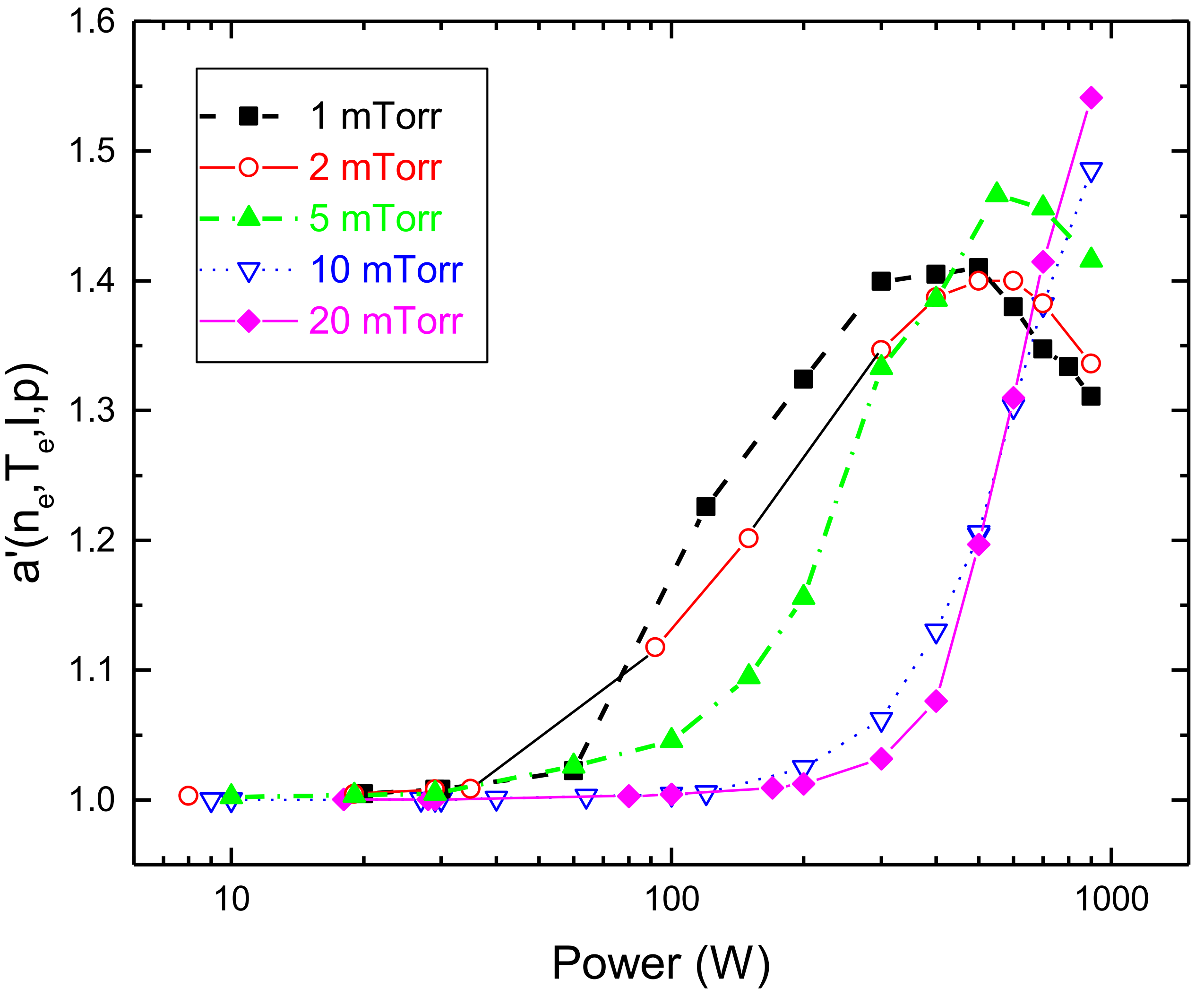
Xe emission:

$$Xe(1S^0) + e \rightarrow Xe(2p_5) \qquad E_{th} = 9.94eV$$

$$Xe(1s_{3,5}) + e \rightarrow Xe(2p_5) \qquad E_{th} = 1.5eV$$

$$Xe(2p_5) + e \rightarrow Xe(1s_5) + h\nu, \quad 8280\mathring{A}$$

EFFECT OF Xe METASTABLES ON ACTINOMETRY PROPORTIONALITY CONSTANT $a'(n_e,T_e,l,p)$



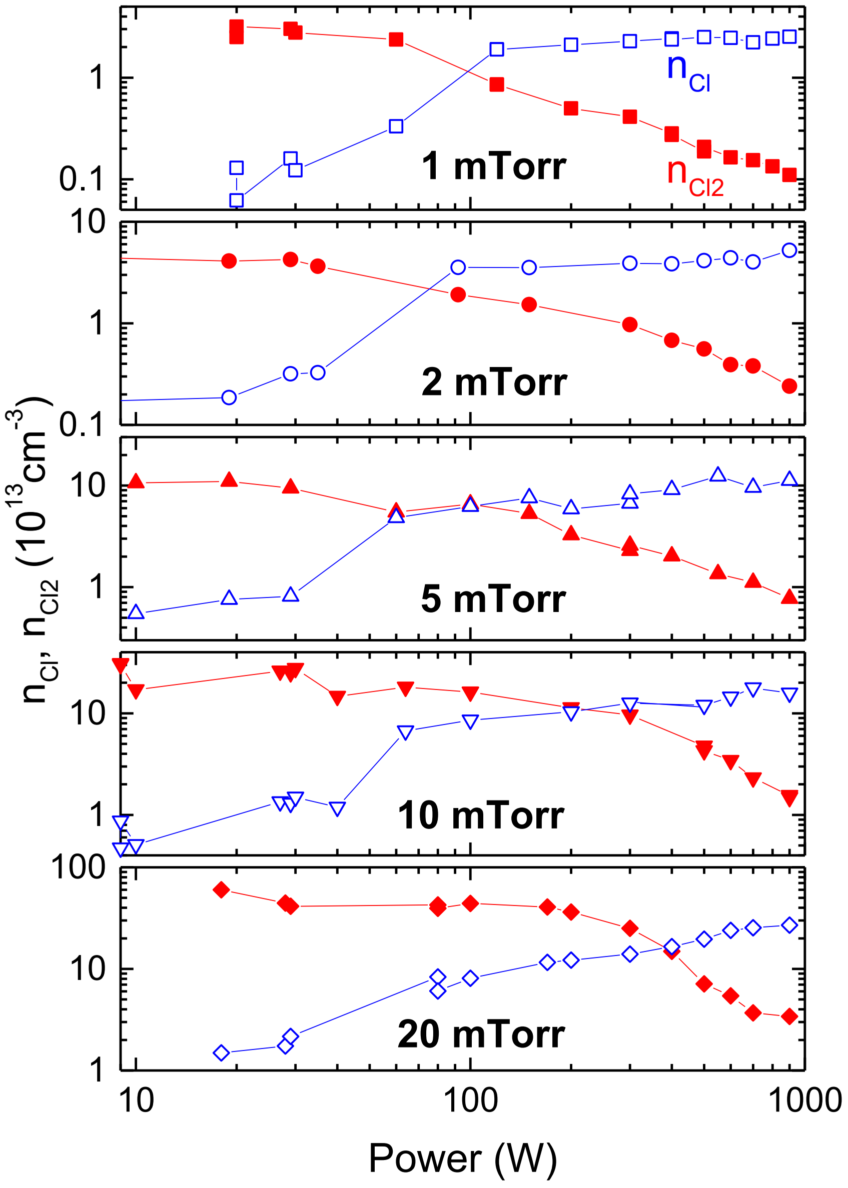
Advanced Actinometry: Cl and Cl_2 Number Densities in a Cl_2 Plasma

- Include electron impact excitation from rare gas metastables.

- Include conditions-dependence proportionality constant.

- Include dissociative excitation:

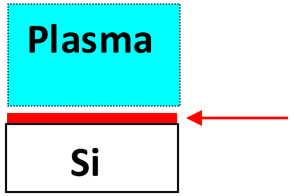
$$\frac{I_{Cl}}{I_{Xe}} = a(T_e, n_e, l, p)\frac{n_{Cl}}{n_{Xe}} + b(T_e, n_e, l, p)\frac{n_{Cl2}}{n_{Xe}}$$



Plasma-Surface Interactions

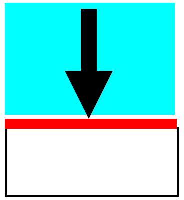
Ion-Stimulated Desorption – Plasma Induced Emssion

1)



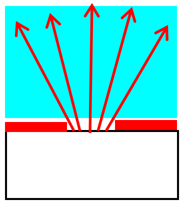
$SiCl_x$ layer

2)

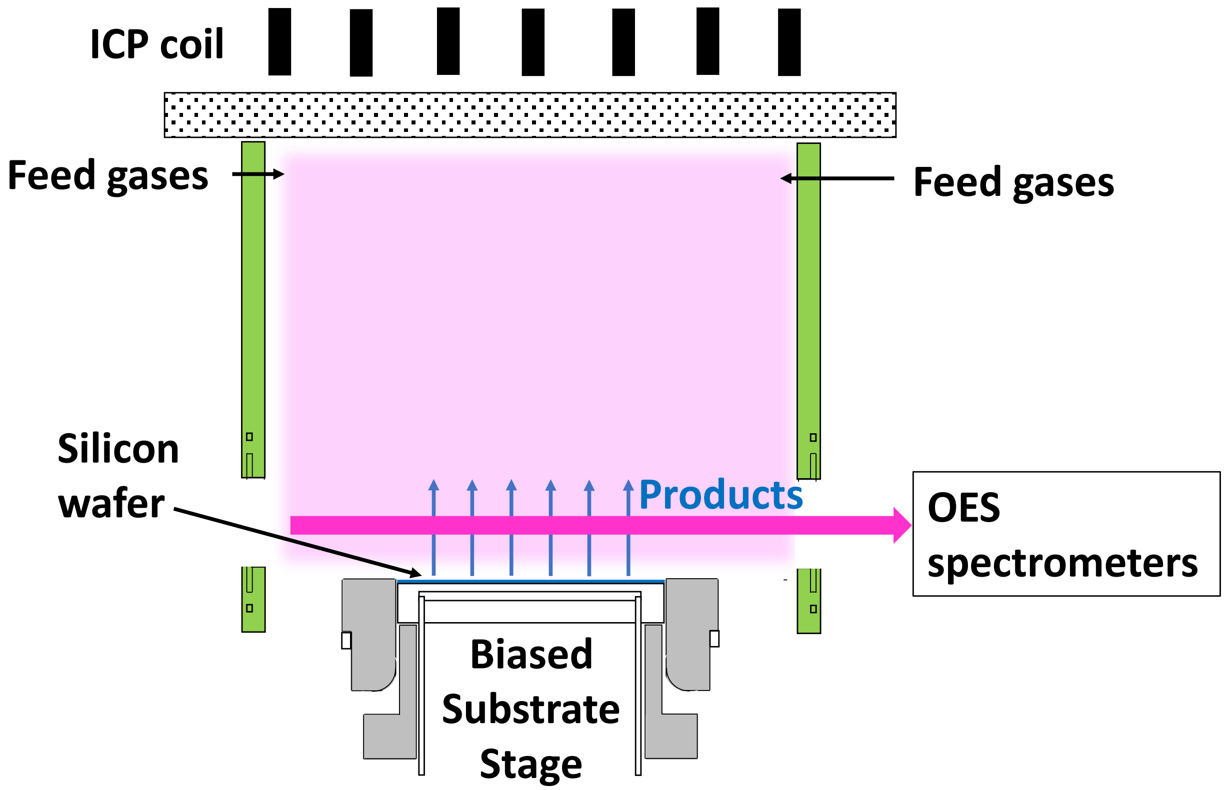


Energetic Ar ion pulse

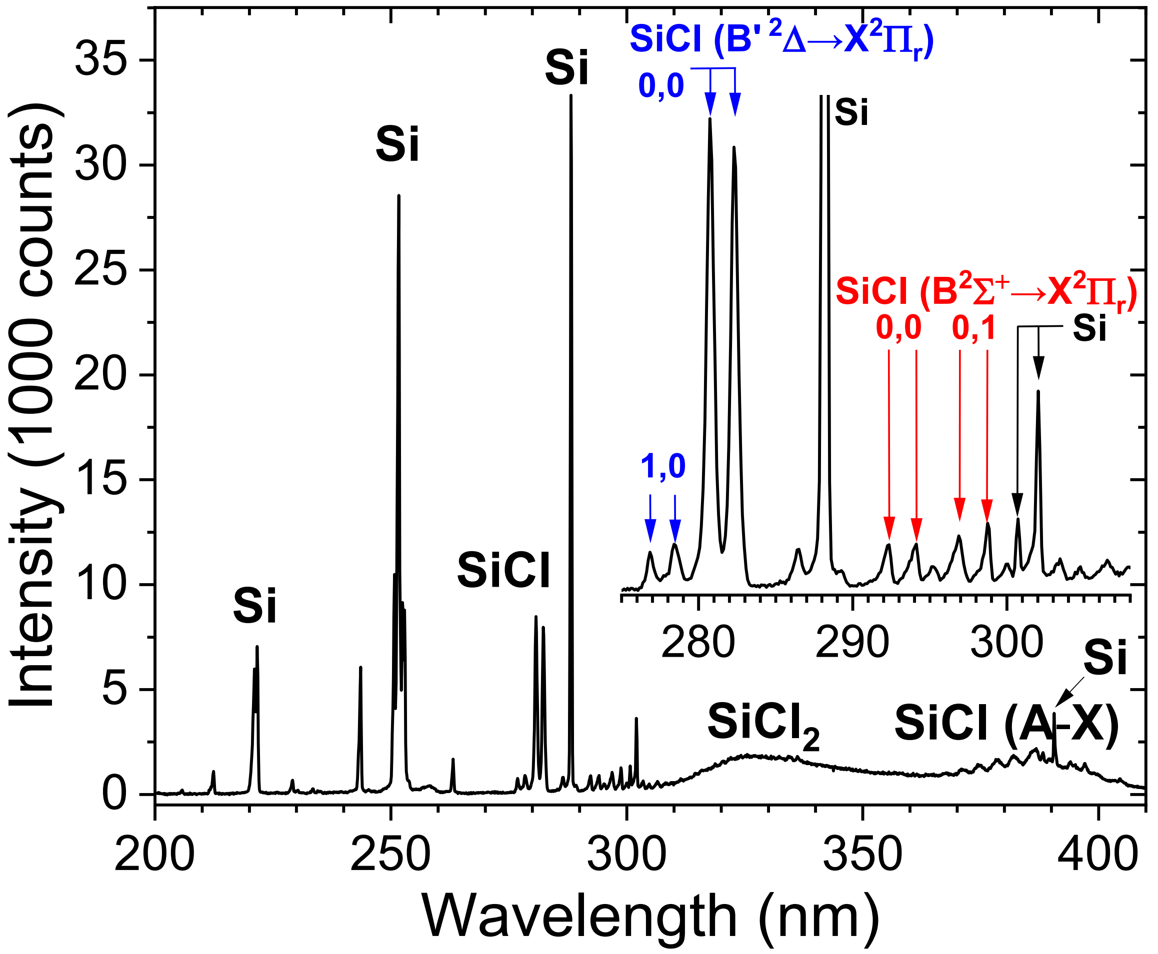
3)



SiCl excitation by plasma (later and at some distance from the substrate)

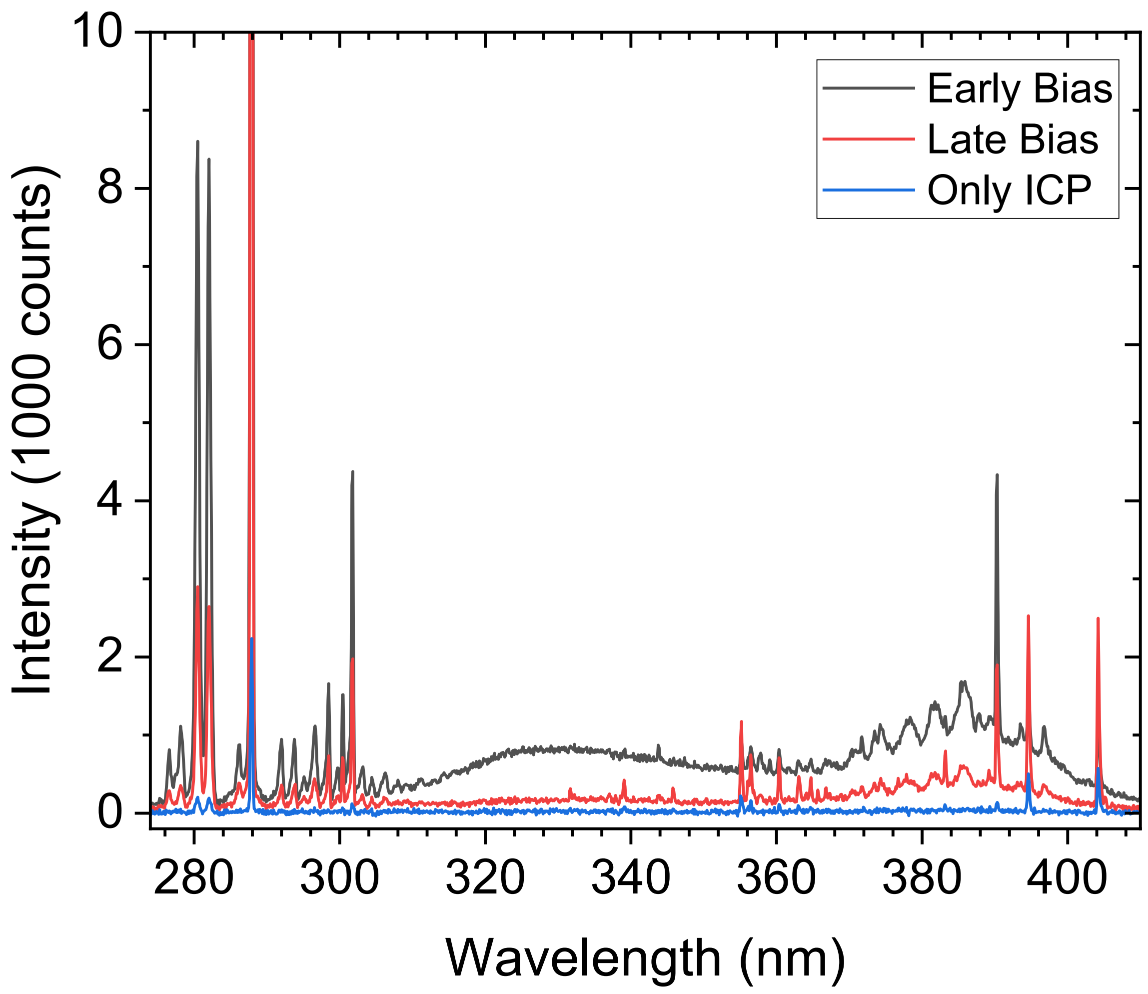


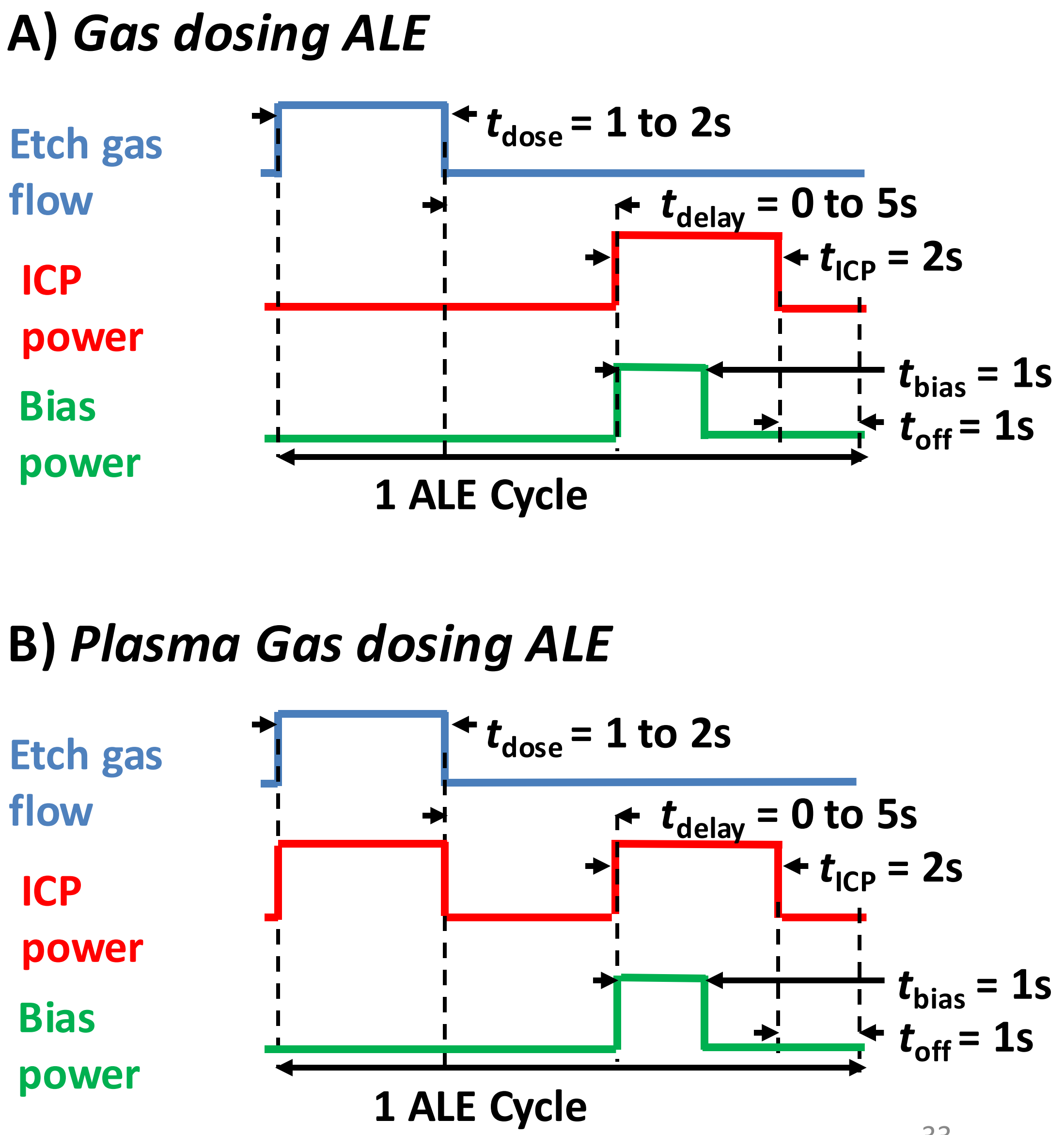
Spectrum Recorded with Continuous Cl_2 Flows and Powers



- 10 sccm Cl_2, 50 sccm Ar, Pressure = 20 mTorr, ICP power = 267 W, self-bias VDC = -59 V.
- Emission from Si, SiCl and $SiCl_2$.

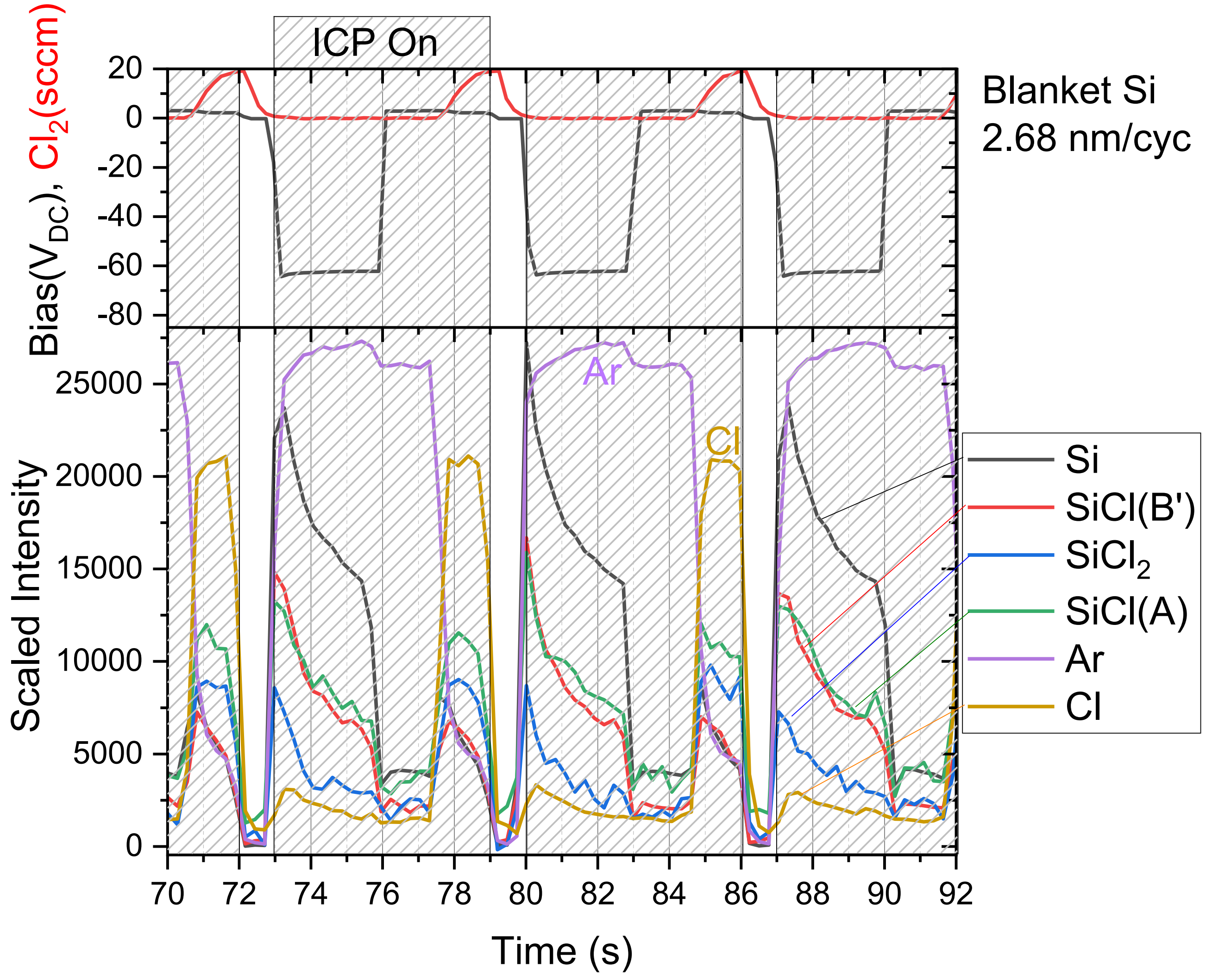
Spectra Recorded Under the Atomic Layer Etching ALE Conditions





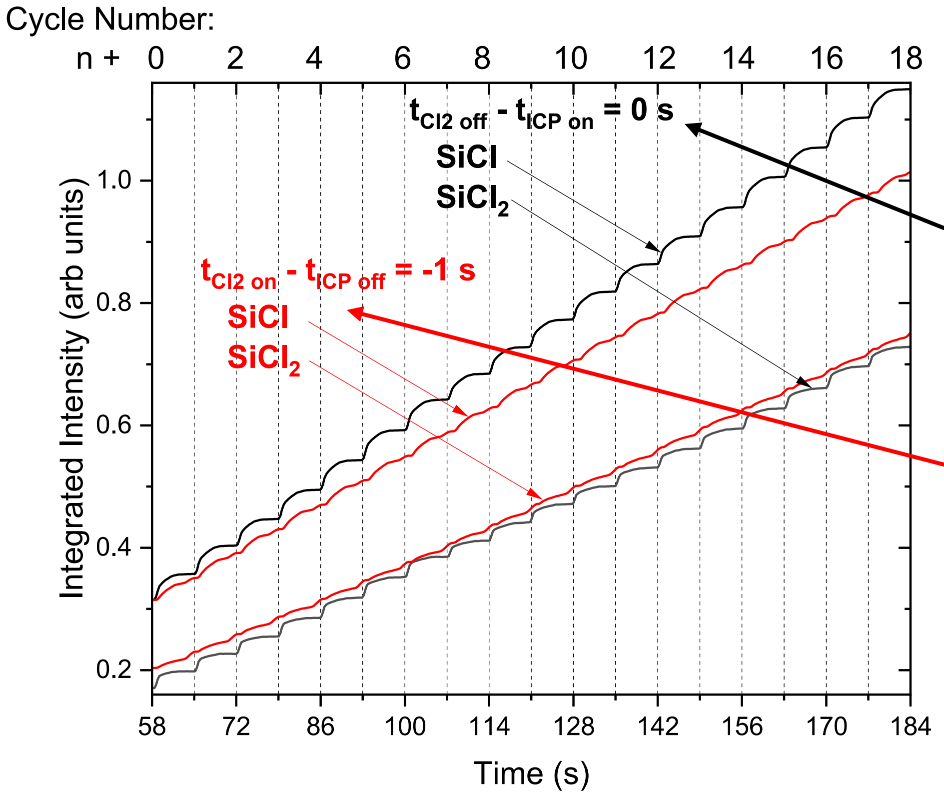
- 160 sccm continuous Ar, 20 sccm, 1s Cl_2 doses with ICP off, then ICP(280W), -60 V_{DC} bias.
- $SiCl_x$ emission intensities decay during the bias period.

Time-Dependent OES During Si ALE with Cl_2/Ar Plasma



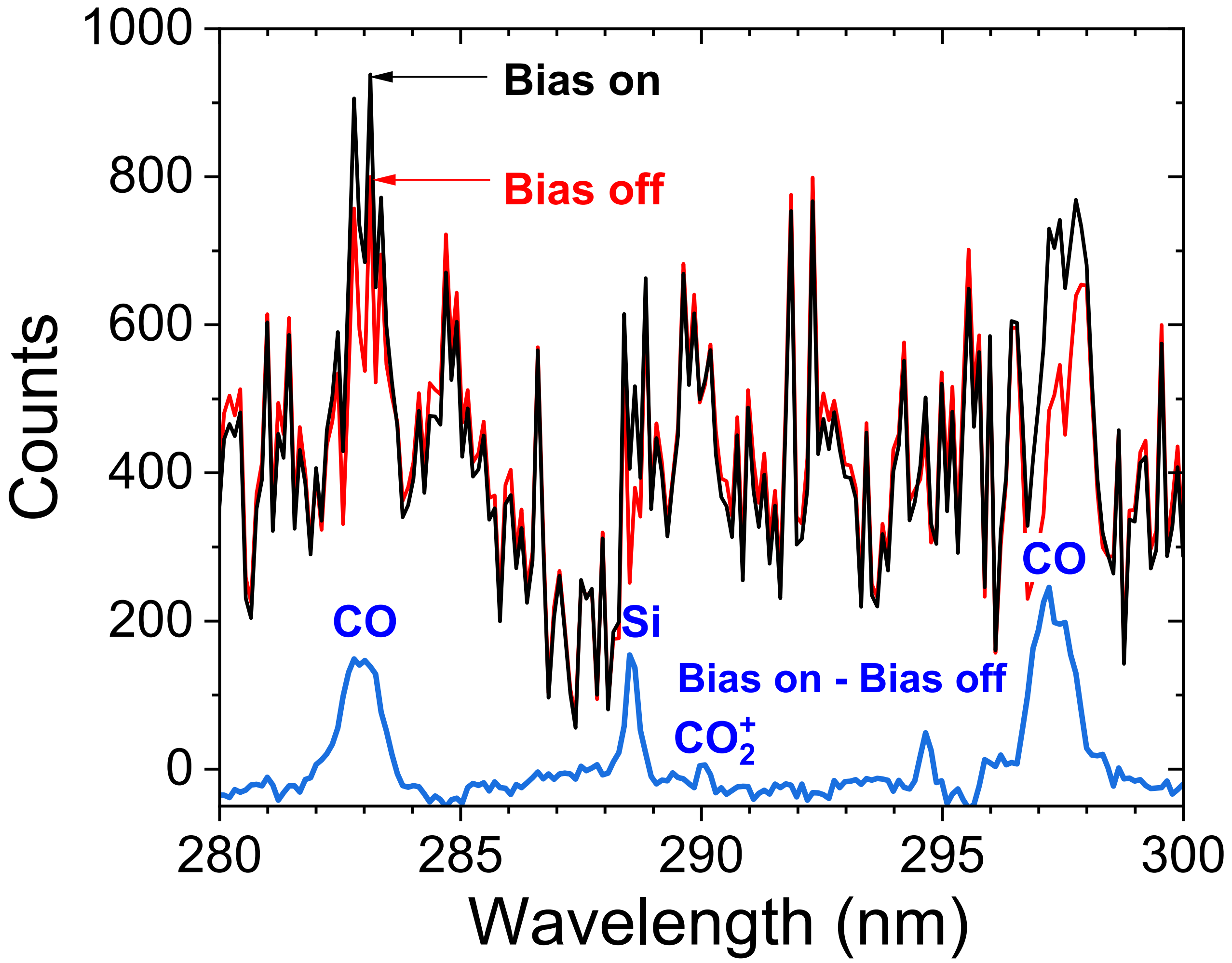
- Emissions primarily from primary Si, SiCl and $SiCl_2$ desorption products (see Appendix 3).
- Roughly 2s is required to sputter away the $SiCl_x$ layer formed during each dosing step.

Using OES to Follow Cl-uptake and Si Etching During Atomic Layer Etching

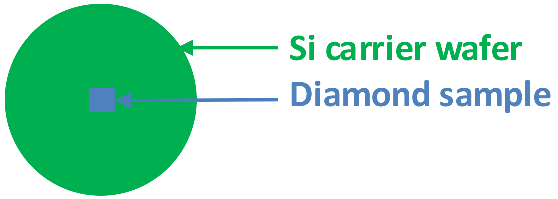


- The slope of integrated SiCl and $SiCl_2$ emissions track the per-cycle etching rates of 2.86 and 2.68 nm/cycle.
- Only a small overlap in time between Cl_2 pulse and ICP power pulse. ALE behavior because most of the Cl_2 dosing occurs without etching.
- Partial overlap between Cl_2 pulse and ICP power pulse. Non-ALE behavior because nearly half the etching occurs during the Cl_2/ICP dosing step.

UV Spectra During O_2/Ar Plasma Etching of Diamond (100)

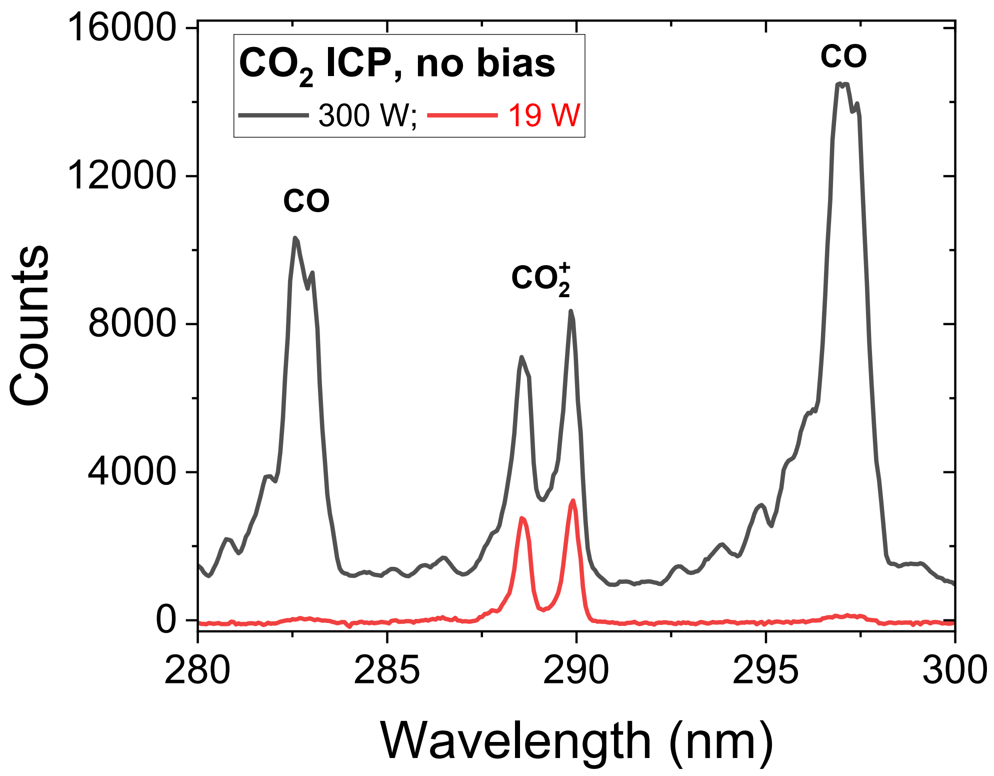


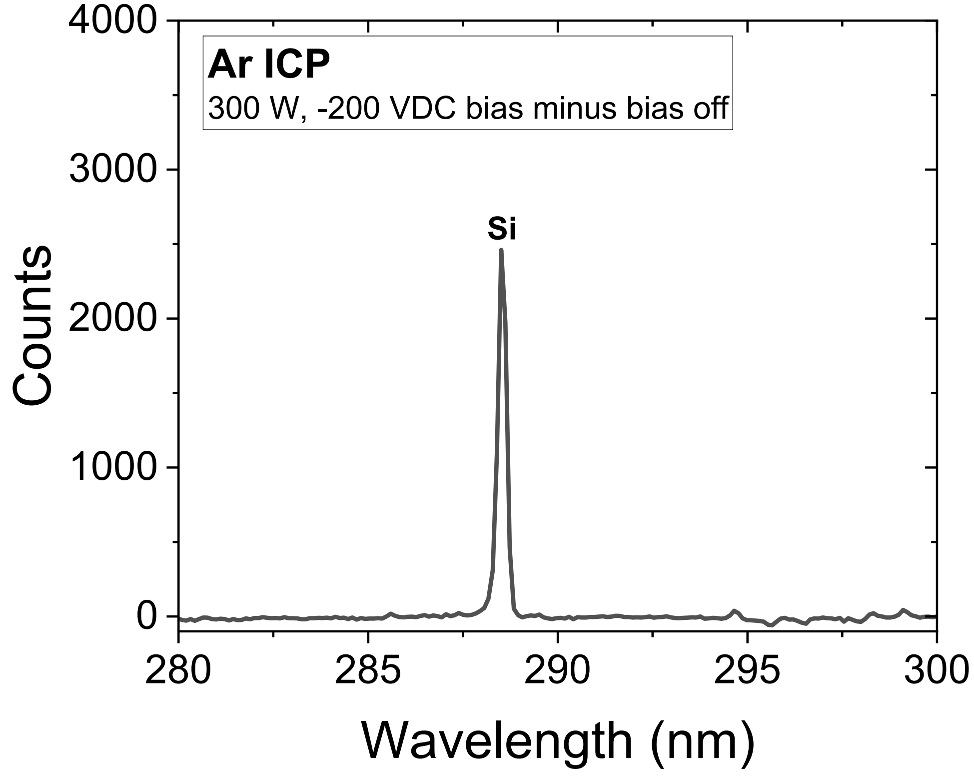
- Flow rates (sccm): Ar = 8, O_2 = 2
- Pressure = 10 mTorr
- ICP power = 300 W
- Magnet current = 55 A
- Bias = 0 or -200 V_{DC} self bias



- Bias-on and bias-off spectra dominated by complex emission spectra of O_2^+,
- Subtracting bias-off from bias on reveals emissions from CO, CO_2^+ and Si.

How Can We Be Sure of the Product Assignments?

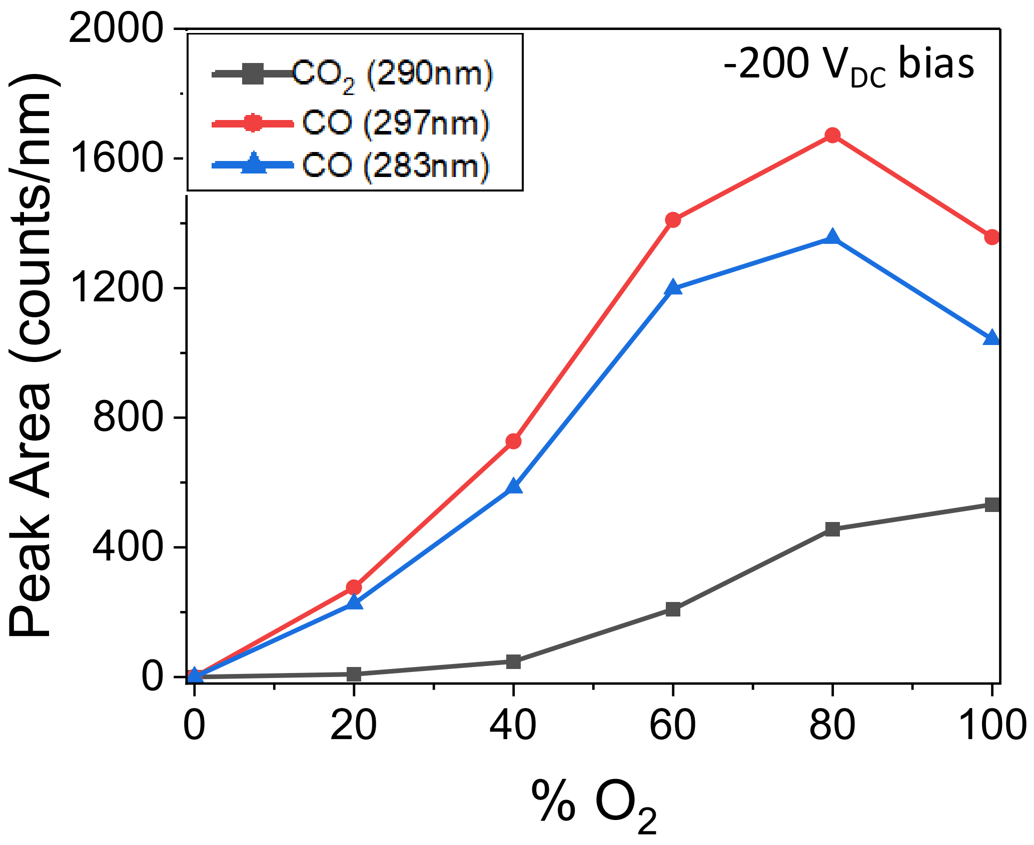


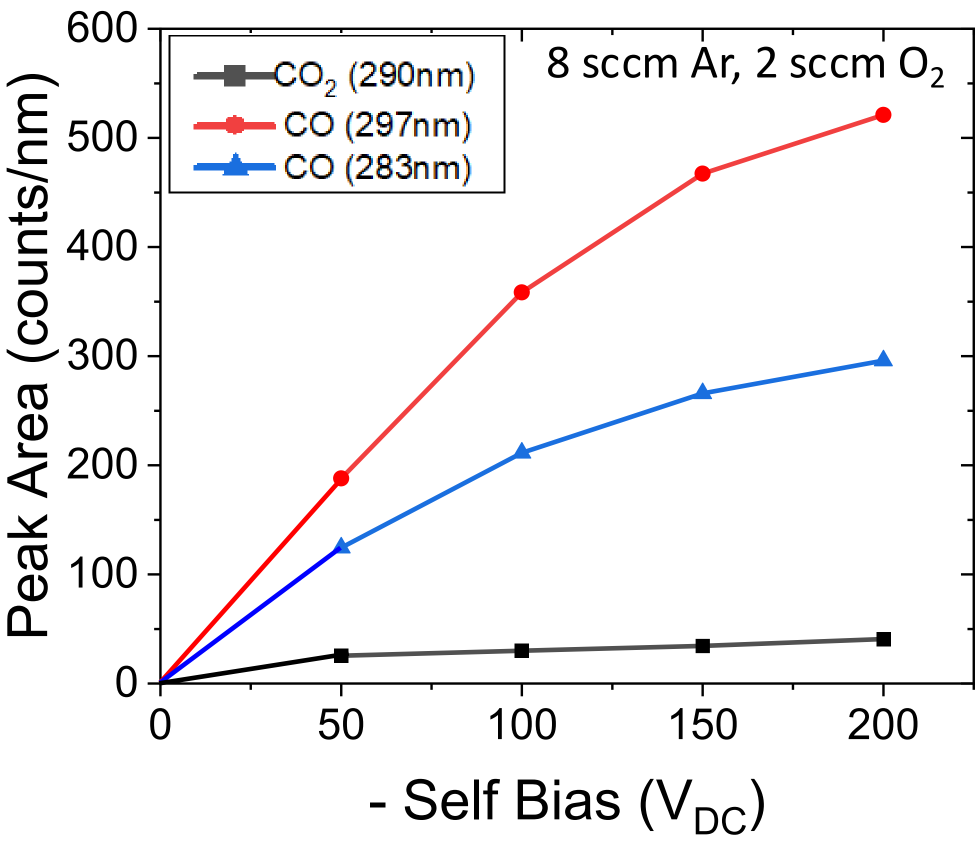


- CO_2 plasma confirms the assignments of CO and CO_2^+.
- Note that CO emissions are very weak relative to CO_2^+ at low power because very little CO_2 dissociation occurs.

- Strong Si emission in pure Ar plasma with bias due to Si sputtering.

Diamond Etching Product Emission Intensities as a Function of %O_2 and DC Self-Bias

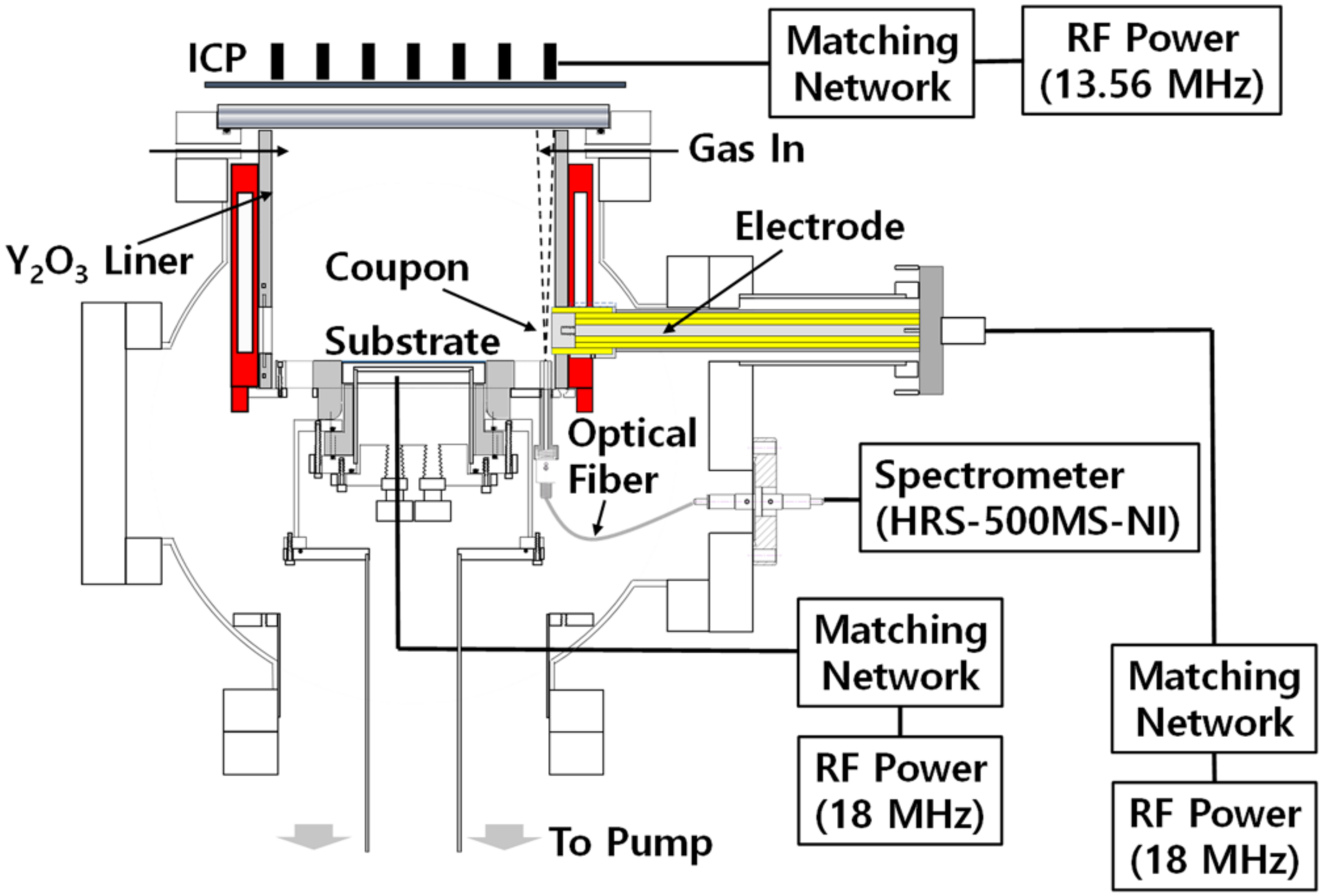




- Product emissions (and presumably etching rates) increase with %O_2 in the feed gas.
- CO_2 emission increases strongly with increasing %O2, while CO appears to peak near 80%O_2.

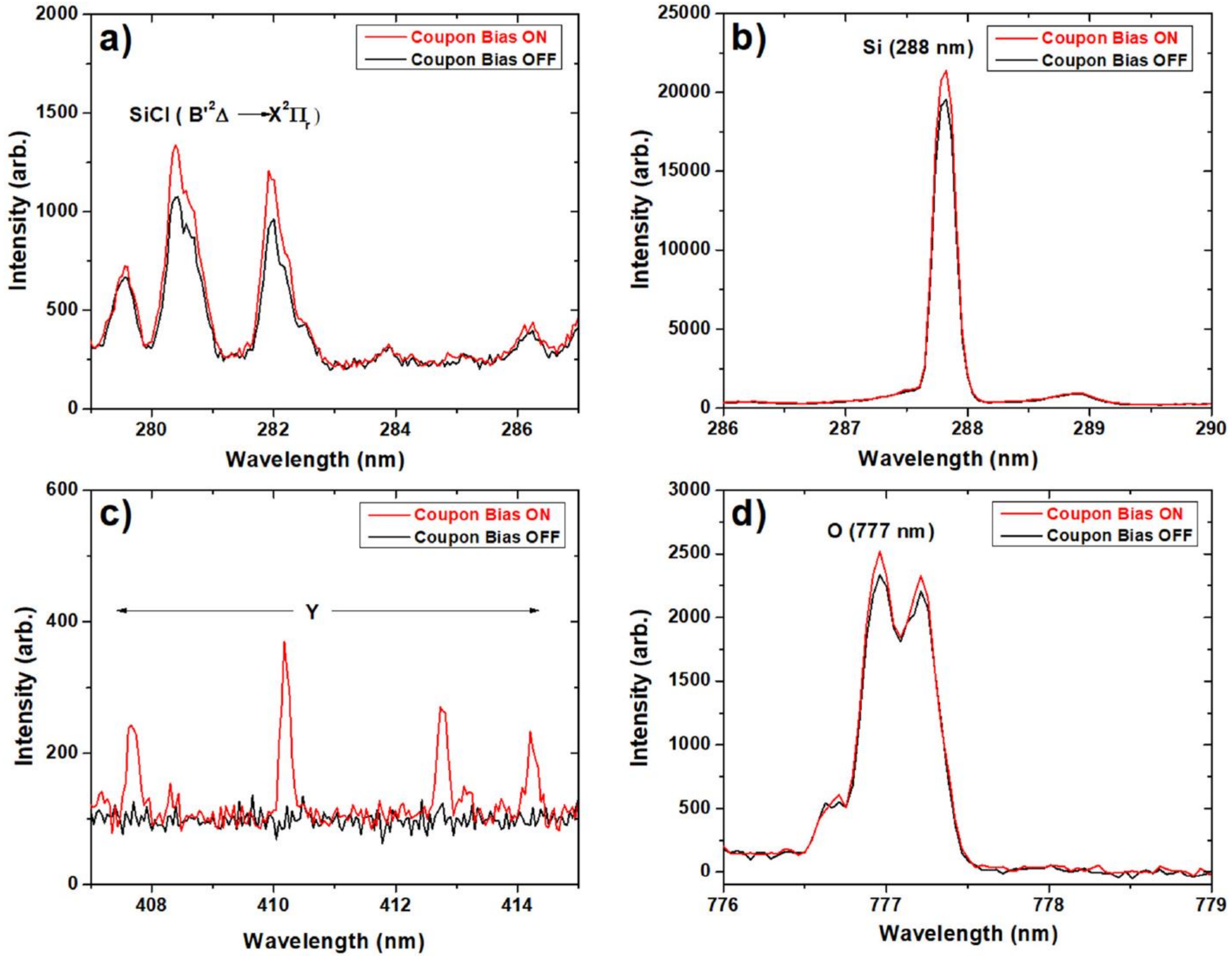
- CO emissions increase with bias much more strongly than does CO_2.

Chemical Wall Probe: Ion-Induced Desorption, Optical Emission Spectroscopy

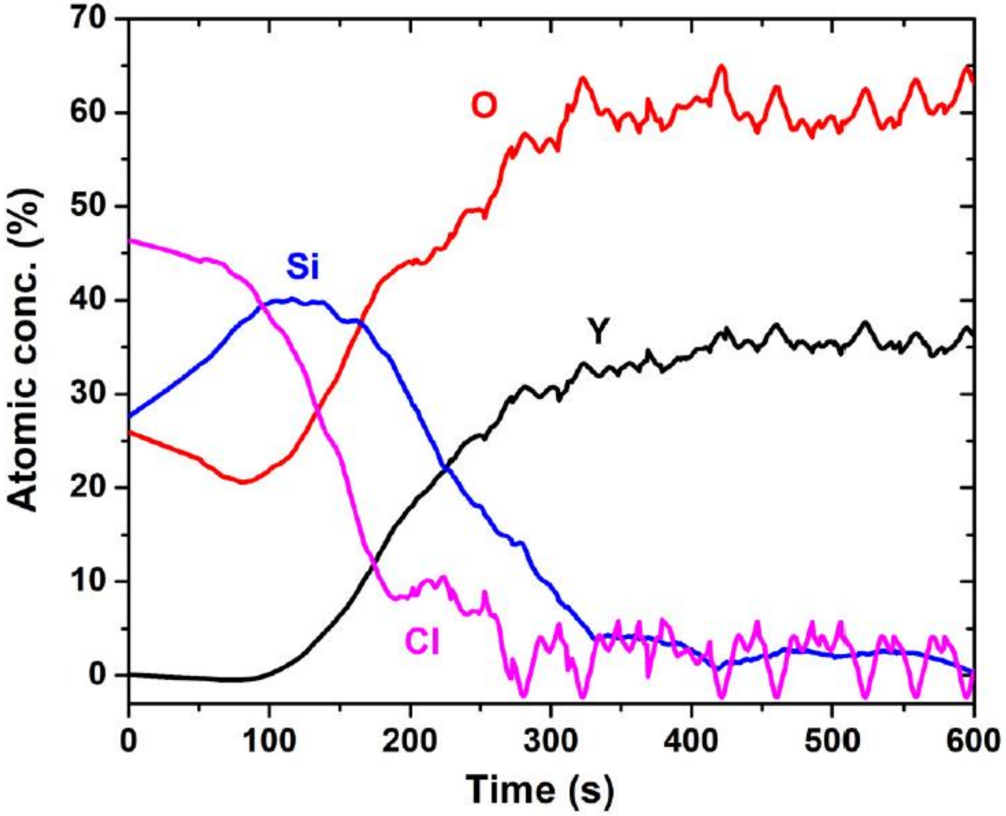


- Coupon piece in chamber wall.
- Periodic RF bias applied to coupon.
- Line-of-sight OES isolates region above coupon.
- Very small perturbation to plasma.
- Record OES with and without coupon bias - Difference comes from products desorbed from the coupon.

Optical Emission Spectra with the Coupon Bias On and Off During (a) and (b) or After (c) and (d) Sputter Removal of a $SiCl_xO_y$ Layer that Forms on the Yttria Walls During Si Etching in a Cl_2/O_2 Plasma.



In-Situ Sputter Depth Profile of $SiCl_xO_y$ Layer Deposited on Yttria in a Cl_2/5%O_2 Plasma During Silicon Etching



- With further calibrations, it is possible to convert sputter time to depth.

Questions?

<u>Appendix 1</u>: Determination of electron energy distributions (EEDF) from Langmuir probe measurements (see Lieberman, Lichtenberg, 2nd ed. p 185-195)

- Cylindrical probe of area, A
- Isotropic electron velocity distribution
- Probe operated with electron-retarding voltage V_B (electron-retarding means $V = \Phi_p - V_B > 0$, where Φ_p is plasma potential)
- In spherical polar coordinates, electron current is:

$$I_e = eA \int_{v_{\min}}^{\infty} dv \int_{0}^{\theta_{\min}} d\theta \int_{0}^{2\pi} d\phi \, v \cos\theta \, v^2 \sin\theta \; f_e(v)$$

- v is electron speed, $\theta_{min} = cos^{-1}(v_{min}/v)$ and $v_{min} = [2e(\Phi_p - V_B)/m_e]^{1/2}$
- Integrate over θ and ϕ

$$I_e = \pi e A \int_{v_{\min}}^{\infty} dv^3 \left(1 - \frac{v_{\min}^2}{v^2}\right) f_e(v)$$

Determination of electron energy distributions (EEDF) from Langmuir probe measurements (cont.)

- Change from electron speed to energy, $\varepsilon = mv^2/2e$:

$$I_e = \frac{2\pi e^3}{m^2} A \int_V^\infty d\varepsilon \; \varepsilon \left(1 - \frac{V}{\varepsilon}\right) f_e[v(\varepsilon)]$$

- $V = \Phi_p - V_B$
- Differentiate twice with respect to V

$$\frac{d^2 I_e}{dV^2} = \frac{2\pi e^3 A}{m^2} f_e[v(V)]$$

- EEDF, $g_e(\varepsilon)$, is related to f_e by $g_e(\varepsilon)d\varepsilon = 4\pi v^2 f_e(v)dv$, therefore

$$g_e(\varepsilon) = \frac{2m}{e^2 A}\sqrt{\frac{2e\varepsilon}{m}} \frac{d^2 I_e}{dV^2}$$

Determination of electron energy distributions (EEDF) from Langmuir probe measurements (cont.)

- In terms of $V = \Phi_p - V_B = \varepsilon$

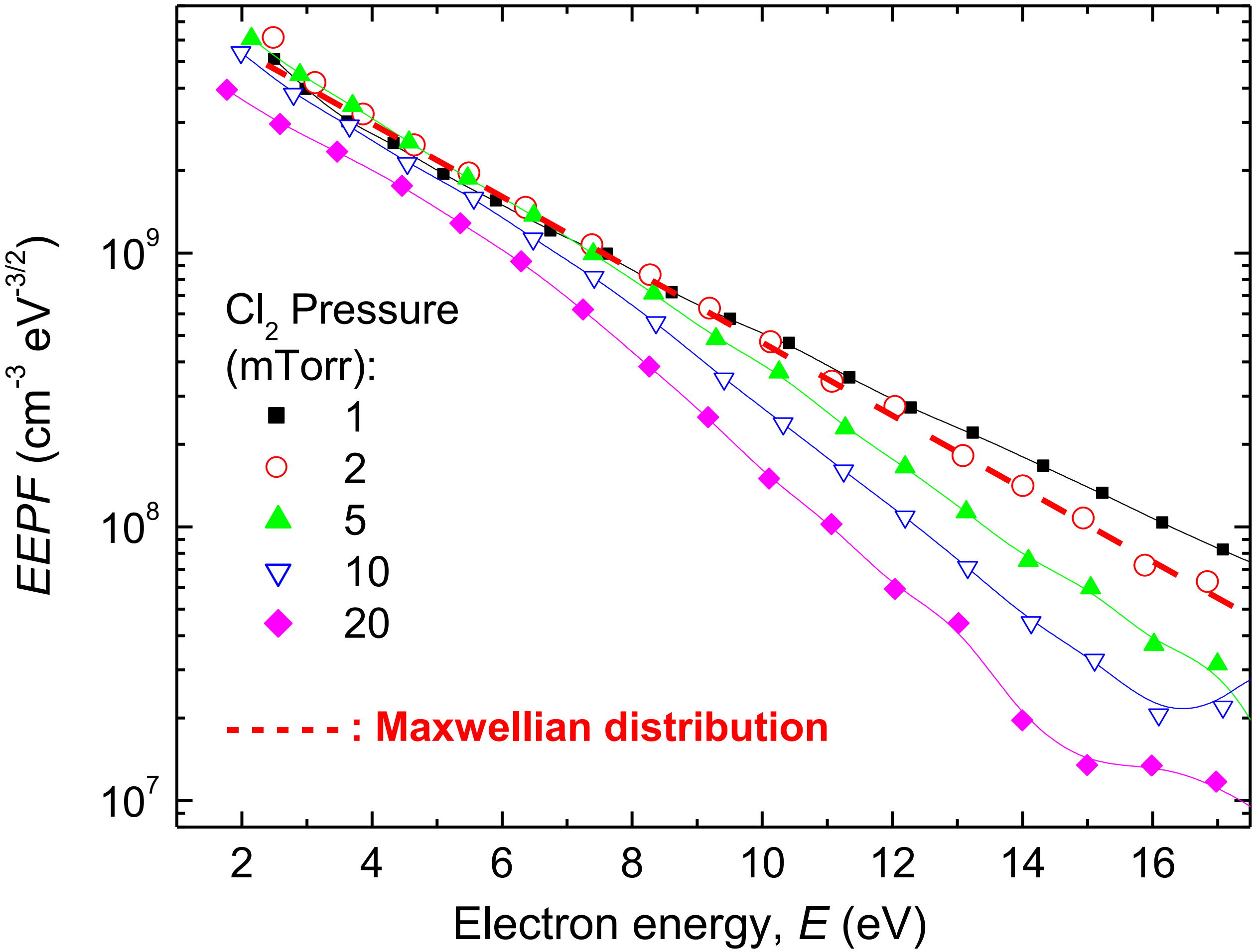
$$g_e(V) = \frac{2m}{e^2 A}\sqrt{\frac{2eV}{m}}\frac{d^2 I_e}{dV^2}$$

- More common to plot electron energy probability function (EEPF),

$$g_p(\varepsilon) = \varepsilon^{-1/2} g_e(\varepsilon)$$

- For Maxwellian EEPF

$$g_p(\varepsilon) = \frac{2n_e}{\sqrt{\pi}}\frac{1}{T_e^{3/2}}\exp\left(\frac{-\varepsilon}{T_e}\right)$$



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• Allen-Boyd-Reynolds Theory: J. E. Allen, R. L. F. Boyd, and P. Reynolds, Proc. Phys, Soc. London, Sect. B **70**, 297 (1957); C. M. C. Nairn, B. M. Annaratone, and J. E. Allen, Plasma Sources Sci. Technol. **4**, 416 (1995).

• F. F. Chen, in *Plasma Diagnostics Techniques*, ed. R. H. Huddlestone, and S. L. Leonard (Academic, New York, 1965), pp. 113-200; Plasma Phys. **7**, 47 (1965).

• M. A. Lieberman and A. J. Lichtenberg, *Principles of Plasma Discharges and Materials Processing*, 3rd edition (Wiley, New York, 2024).

• *Plasma Diagnostics* (ed. Auciello and Flamm) Academic Press, 1989, Ch. 3 (N. Hershkowitz)

• Comparative analyses of plasma probe diagnostics techniques, V. A. Godyak ; B. M. Alexandrovich, J. Appl. Phys. **118**, 233302 (2015).

• Other works, too numerous to list by: J. E. Allen, F. F. Chen, V. A. Godyak, N. Hershkowitz, I. Langmuir, and others.

Appendix 2: Actinometry

• Assume excited state (*k*) of species X is populated solely by e-impact from its ground state (*i*).

• Then its absolute ground state number density (n_X) is related to the intensity ($I_{X,i,j,k}$) of emission at wavelength $\lambda_{j,k}$ accompanying the transition $X_k \rightarrow X_j$ by

$$I_{X,i,j,k} = 4\pi a_X(\lambda_{A,j,k}) n_X Q_{A,k} b_{A,j,k} \int_0^\infty \sigma_X(v) v^3 f_e(v) dv$$

$a(\lambda,j,k)$ = spectrometer sensitivity at $\lambda_{A,j,k}$

$\sigma_{X,i,k}(v)$ = cross section at electron speed *v* for e + X_i → X_k + e

$f_e(v)$ is the electron speed distribution function ($4\pi v^2 f_e(v)dv$ *is* the number of electrons with speeds between *v* and *v*+*dv*)

$Q_k = \tau^{-1} / (\tau^{-1} + k_q P)$ is the quantum yield for emission by X_k where τ and k_q are the radiative lifetime and quenching rate constant for A_k by all species at total pressure *P*

$b_{A,j,k} = i_{X_{j,k}} / \sum i_{X_k}$ *is the branching ratio for the transition* $X_k \rightarrow X_j$

Actinometry (cont.)

• The electron speed distribution and the proportionality constant are difficult to determine.

• Consequently rare gas actinometry is often used to convert emission intensities into quantitative, relative number densities.

• Technique was first applied in plasmas by Coburn and Chen.

• Add n_A amount of a rare gas, A, with an excited state A_k at energy close to that of X_k.

• Assuming that rare gas emissions are caused solely by e-impact excitation of the ground state, then it emission intensity is

$$I_{A,i,j,k} = 4\pi a_A(\lambda_{A,j,k}) n_{A_i} Q_{A,k} b_{A,j,k} \int_0^\infty \sigma_A(v) v^3 f_e(v) dv$$

Actinometry (cont.)

- If $\sigma_A(v) \propto \sigma_X(v)$ at any *v, then* n_X can be simply expressed as

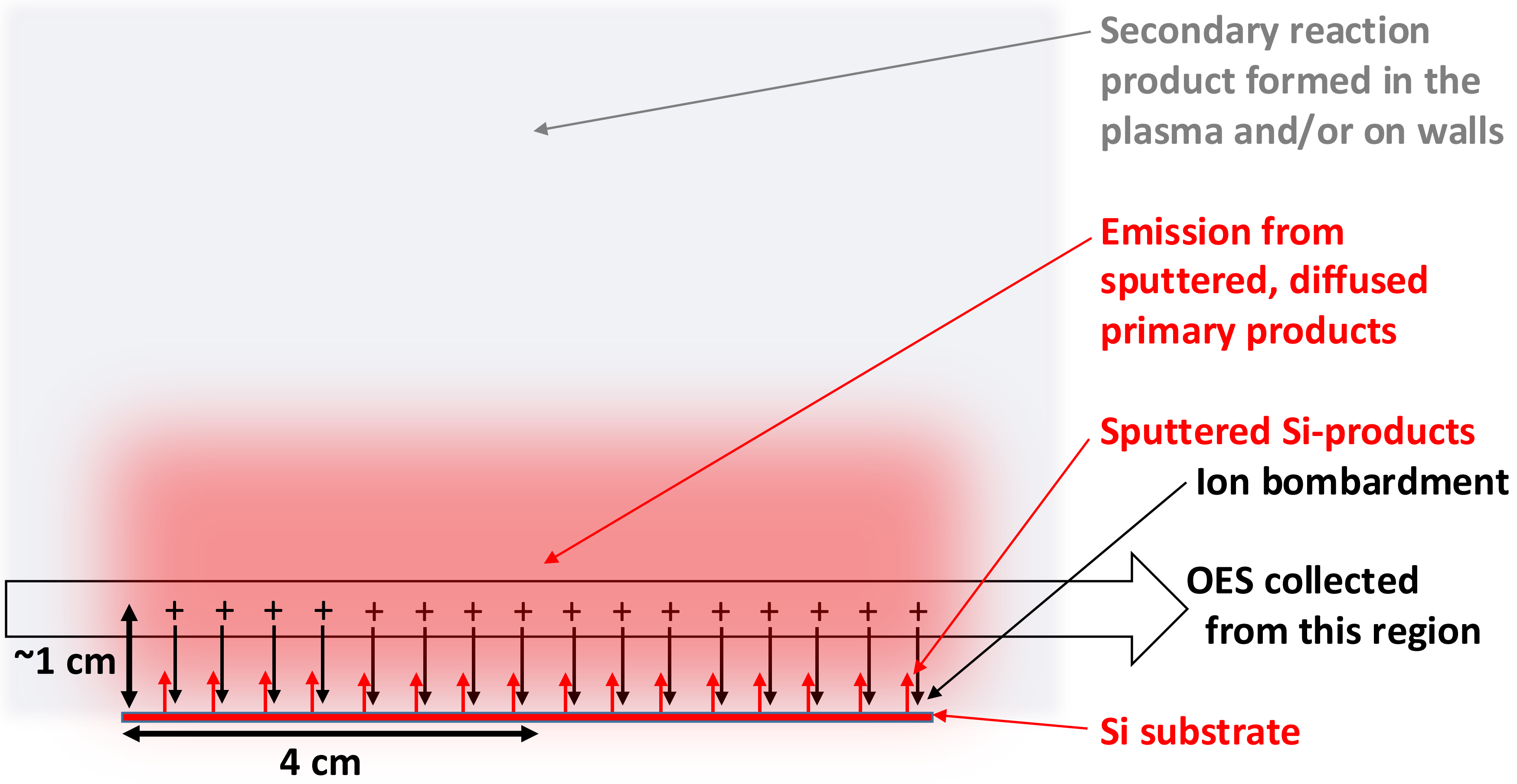
$$n_X = a_{X,A}\, n_A\, (I_{X,i,j,k} / I_{A,i,j,k})$$

where $a_{X,A}$ is a proportionality constant.

- Relative densities of atoms (F, Cl, H, O) and small molecules (Cl_2, CF, CF_2, BCl, N_2, CO) have been determined by this method.

- In a few cases, absolute number densities have also been measured through several calibration methods.

Appendix 3: Excitation of Primary Desorption vs. Secondary Etch Products?

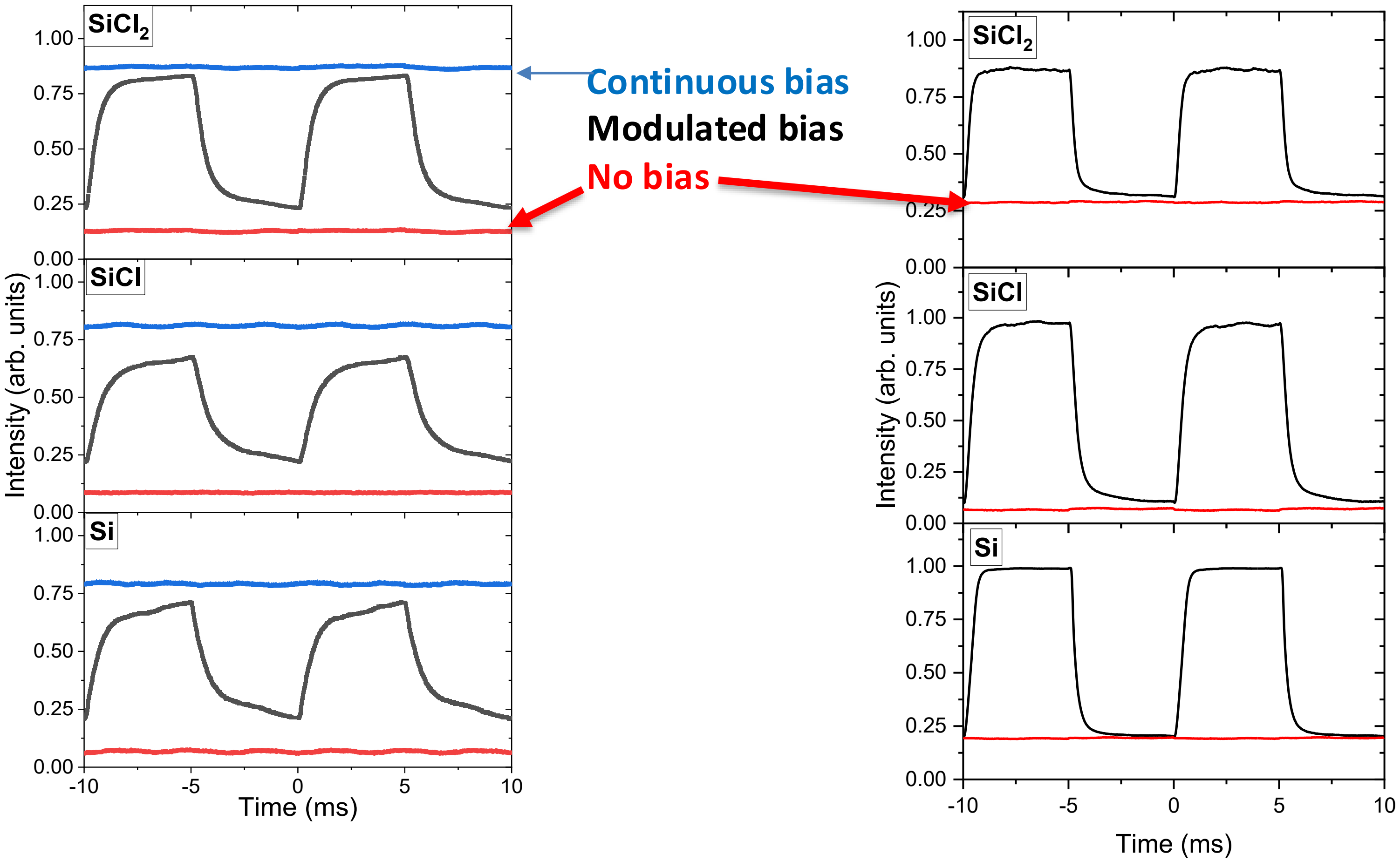


- Emissions from primary desorption products have rise and fall times of ~0.7 to 4 ms, corresponding to diffusion into and out of the observation region.

- Emissions from secondary products will rise and fall times on the order of gas residence time of ~30 ms.

Time-Resolved Emission with Modulated Bias (5 ms on/5 ms off)

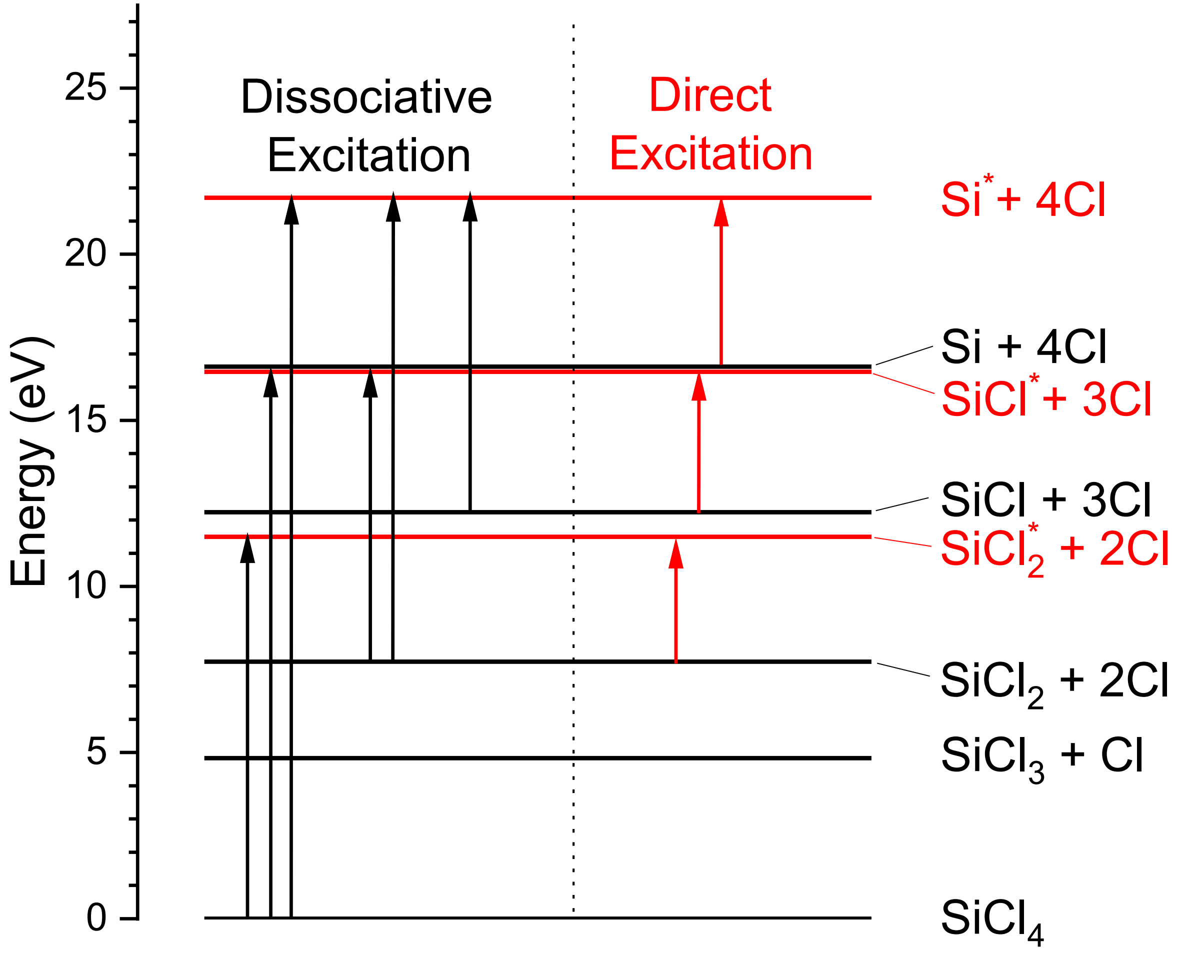
Continuous Powers and Flows | ALE Conditions



- Emissions are highly modulated, with rise and fall times of ~0.4 to 2 ms, indicating that they are predominately from the primary desorption products.

- Could be from electron impact directly of emitting species, or by dissociative excitation of higher chloride.

Energetics of Direct and Dissociative Excitation



- Conclude SiCl and $SiCl_2$ emissions are from SiCl and $SiCl_2$ primary desorption products.
- Si emission late in the ALE cycle is from sputtered Si; Si emission early in the cycle could be partially from the dissociative excitation of SiCl.

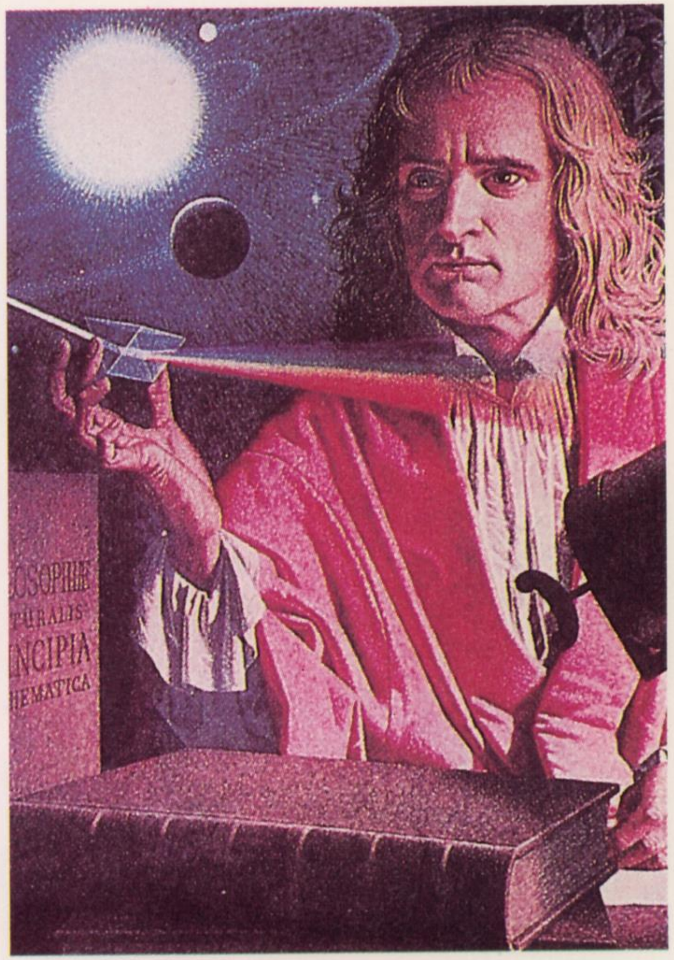
Extras

THE SECOND PLASMA OPTICAL EMISSION SPECTROSCOPY EXPERIMENT- 1666

Isaac Newton – Cambridge University

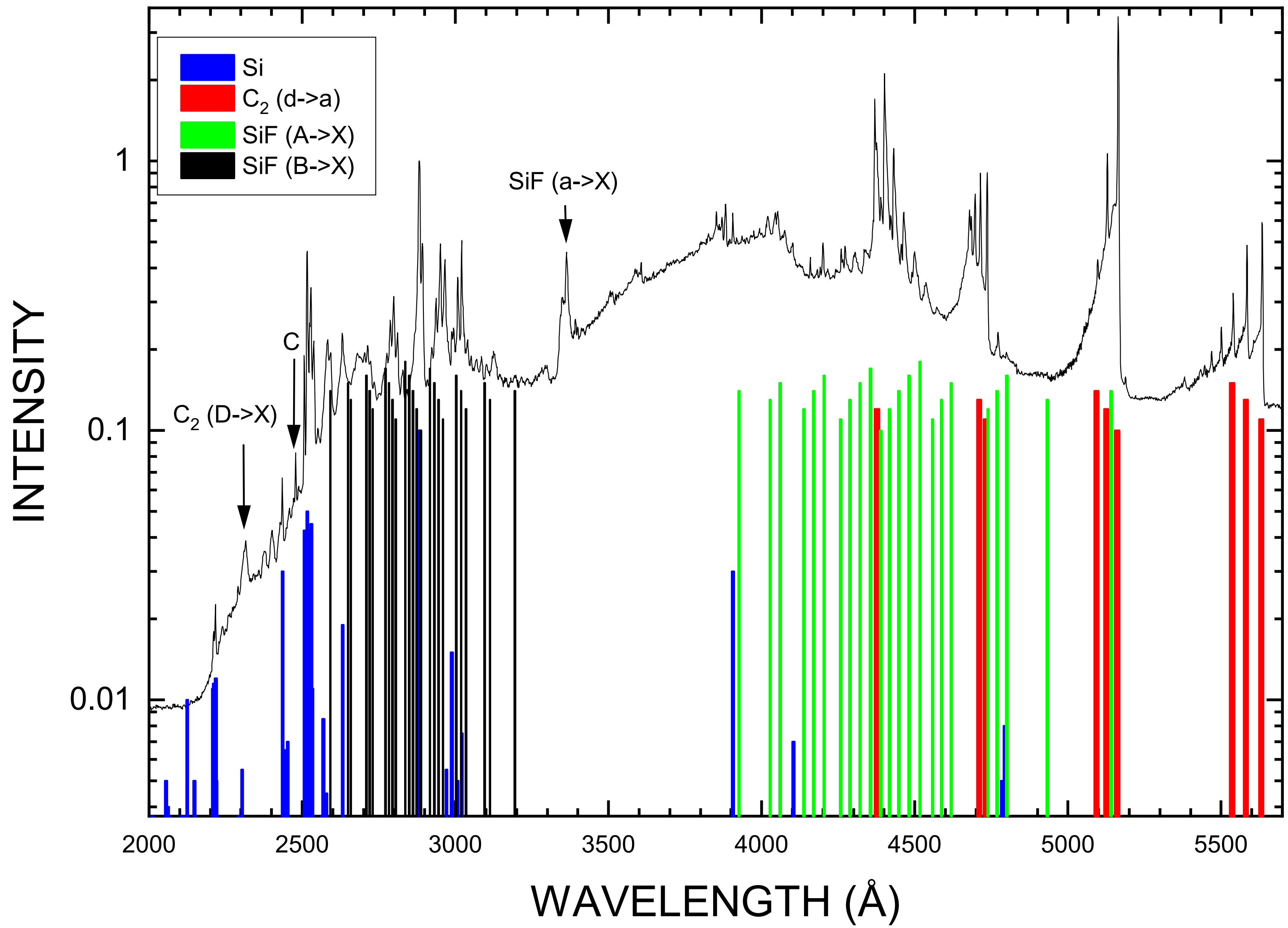
The first plasma optical emission experiment was less than a total success:

In a few hours I had brought my eyes to such a pass that I could look upon no bright object with neither eye but I saw the Sun before me, so that I durst neither write nor read but to recover the use of my eyes shut my self up in my chamber made dark three days together & used all means to divert my imagination from the Sun. For if I thought upon him I presently saw his picture though I was in the dark.



Isaac Newton. Painting by Jean-Leon Huens, © National Geographic Society.

Example 2: Emission spectrum of a C_2F_6 inductively-coupled plasma during etching of SiO_2 and Si



Coupon Bias On Minus Coupon Bias Off Optical Emission Intensites for Si, SiCl, Y and O During Sputter Removal of a $SiCl_xO_y$ Layer that Forms on the Yttria Walls During Si Etching in a Cl_2 or $Cl_2/5\%O_2$ Plasma.

- With calibrations, these measurements can be converted into sputter depth profiles.