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Laser-induced fluorescence ion diagnostics in light of plasma processing

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Abstract

A brief overview of non-perturbing light diagnostics is followed by recent examples of process plasma properties measured via laser-induced fluorescence (LIF), optical emission, and absorption spectroscopy. Examples include radical density measurement via absorption and emission spectroscopy. With LIF, examples show properties of ion beam etching sources and ion velocity angle variations in ICP sheaths near a process surface. Because of the wide range of process plasma parameters, appropriate choice of light diagnostics varies.

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1. Introduction

Process plasmas include many atomic and molecular species in neutral or charged states. Photon emission or absorption from infrared to ultraviolet may reveal plasma properties without perturbing the process. For example, process endpoint determination using spectral changes as the etched layer is removed is decades old. Total transition intensities may yield population numbers along measured chords or even local densities of individual species, such as radicals, if the density profile is determined. Finely-resolved intensity versus frequency in the transition may reveal ion velocity distribution functions and convective flow velocities via Doppler-shift analysis. Locally altering an atomic or molecular state and following that alteration in time or space may reveal plasma ion diffusion and convection coefficients.

Laser-induced fluorescence (LIF) is a method of enhancing or depleting photon emission from particular energy states. While the LIF photon may have an energy of a few eV, the state examined may be near or far from the ground state. Whether there are any electrons in high energy states depends strongly on the electron temperature and density in the plasma. As neutral

pressures rise above 10 mTorr, electron populations have decreasing tail populations at energies above 10 eV in many process plasmas. This makes visible laser LIF detection using higher energy states difficult. For example, the Ar II metastable state 611 nm line is often used for LIF. This 2 eV transition occurs between states 19 and 21 eV above ground.

Ideally, absolute radical densities may be measured using optical absorption spectroscopy, based upon a transition originating in the ground electronic state of the species. This ensures that the measured density closely represents the total amount of that chemical species, though corrections for vibrational and rotational temperature may be needed. Broad-band absorption has been used in this manner to determine CF and CF₂ in industrial etch reactors [1,2]. For some important atomic radicals, such as fluorine, no suitable absorption line is available above the vacuum limit. Instead an optical emission line intensity may be used to measure relative densities through careful calibration to a rare gas emission line (actinometry) [3]. Both methods, absorption and emission, as described [1–3], were used to measure radical densities in the present study.

2. Emission and absorption spectroscopy

2.1. Radical density measurements

Radical densities were measured in a commercial etch reactor (Lam Research Corp. Exelan[®] HP[™]) to better understand the

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influence of source gas upon plasma composition, under realistic etch conditions. Typical contact etch process conditions were utilized, based on $\text{Ar}/\text{O}_2/\text{C}_x\text{F}_y$ gases. A series of different fluorocarbon source gases were compared using flows selected to provide constant carbon equivalents. Cyclic isomers were used for C_4F_8 and C_5F_8 , and linear isomers were used for C_4F_6 and C_3F_6 . Results indicate that the F density increases with the F:C ratio of the source gas (Fig. 1). However the CF and CF_2 densities have more complex dependence on source gas. Results clearly indicate that source gas internal structure, and not just stoichiometry, influence the plasma radical composition. The F: CF_2 ratio is also shown as an indication of the overall degree of polymerization of the plasma. Results suggest that C_4F_6 and C_5F_8 are most polymerizing, followed by C_3F_6 and C_4F_8 . C_2F_6 is apparently much less polymerizing than the other cases.

2.2. Processing methyl iodide to make atomic iodine

Infrared absorption spectroscopy using a tunable diode laser provided atomic iodine production measurements. The iodine is produced via electron impact dissociation of methyl iodide carried in helium or argon in an rf CCP plasma. The number density and temperature of the atomic iodine could be determined, with a goal of finding the conditions for lowest temperature iodine produced. Fig. 2 shows the experimental arrangement, using a New Focus single mode tunable diode laser to examine the $1.315 \mu\text{m}$ 3–4 hyperfine iodine line which can be distinguished in the Zeeman splitting from the background magnetic field (6 kG). The magnetic field was uniform in the direction of the gas flow, aiding radial plasma confinement along with the Zeeman splitting for spectroscopic line separation. The methyl iodide pressure of 20–200 mTorr was carried in helium or argon at 0.2–10 Torr through a supersonic flat plate CCP geometry. RF power of 10 to a few hundred watts at 50 MHz produced the plasma. An iodine cell

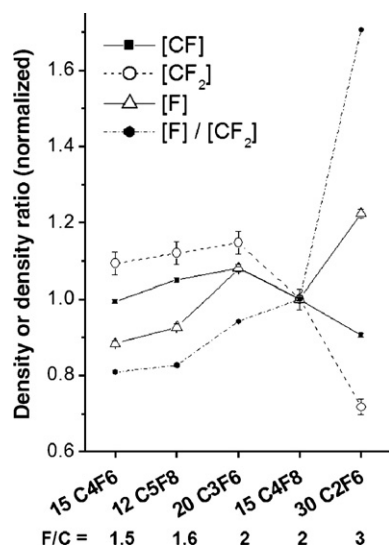


Fig. 1. Radical densities in contact etch plasma, for different fluorocarbon source gases. Also shown in F: CF_2 radical ratio and F:C ratio of source gas. Densities and F:C ratios are normalized to the C_4F_8 process.

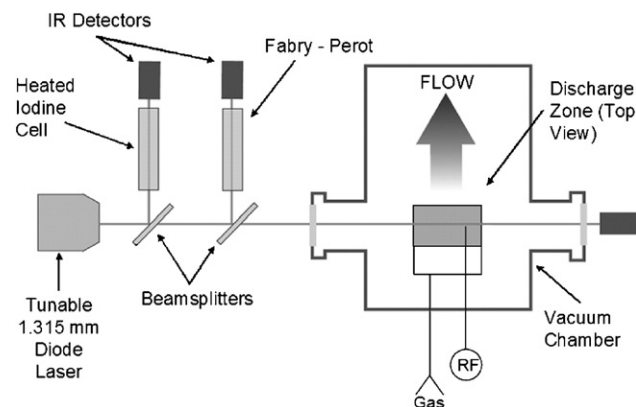


Fig. 2. Experimental arrangement for atomic iodine absorption measurement in methyl-iodide/helium mixture under rf CCP dissociation.

provided absolute velocity calibration of the laser. In a magnetic field (<6 kG), we studied the 3–4 hyperfine iodine line at $^2\text{P}_{1/2}$ ($f' = 3$) \rightarrow $^2\text{P}_{3/2}$ ($f'' = 4$) since this line has a relatively high gain.

By tuning the laser through the line, the atomic iodine temperature can be determined in addition to the absorbance (around $6\% \text{ cm}^{-1}$). Fig. 3 shows an atomic iodine absorption line, the iodine temperature typically was 300–500 K, with number densities up to 10^{15} cm^{-3} observed.

3. Laser-induced fluorescence

3.1. Kaufman-type ion beam sources

Ion beam etching/deposition sources for 0.2–1.2 kV neutralized ion beams commonly use rf, hollow cathode, or filament plasmas. LIF with diode lasers [4] can characterize the ion beam [5] and the beam source plasma. In addition, background neutrals through which the beam propagates may be ionized, causing a low temperature non-beam plasma [6].

An Ar^+ beam source plasma made with a Veeco/IonTech hollow cathode in a Kaufman-type source was measured with LIF. The ion distribution from the hollow cathode was similar to

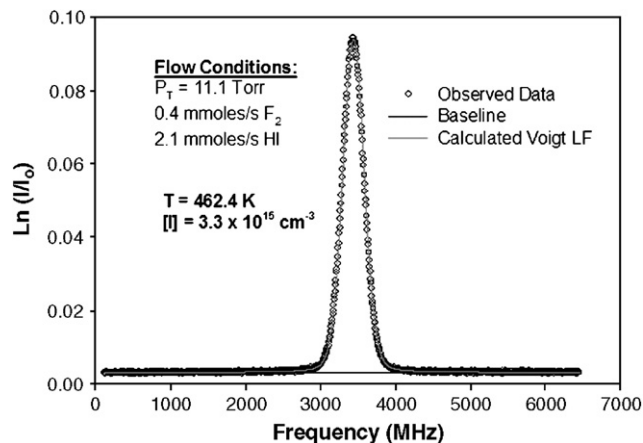


Fig. 3. Atomic iodine absorption line scanned by $1.315 \mu\text{m}$ tunable laser.

that from a filament source studied in [4], hence their interchangeability for many uses is understood.

A Veeco/Ion Tech 3 cm Ar RF Ion Source, Model #201 was studied with LIF. The ion temperature inside an RF source is anisotropic due to the polarization of the rf fields, see Fig. 3a of [7]. The directional dependence of ion temperature in an rf source ranges from 0.1 eV parallel to the acceleration direction to 0.4 eV in the perpendicular. For many purposes, such as for ion beams with energies above 200 eV, this anisotropy may be unimportant. However, LIF also has shown the ion beam energy from these rf Kaufman-type sources varies temporally by as much as 20 eV with all electrical controlling equipment held steady (accelerating grid supplies varied only within 2.5 V). We speculate this variation is due to time-dependent coating of the quartz shield interior of the plasma source thereby having an effect on the source plasma potential via small, un-noticed changes in rf coupling.

3.2. ICP sheaths

In cases for an ICP source without beam accelerating grids but only the plasma sheath near the wafer surface, LIF can show the spread in velocity angles of the ions in the sheath as they approach the wafer. For the conditions of Fig. 2d in [7] we see in Fig. 4 the velocity spread of the ion distribution function in the sheath. These experiments were done using a six-turn 3.8 cm diameter ICP RF solenoid (similar to designs in ion beam etch sources) driven at 18.62 MHz. A bias-able (75 V below ground in the case shown) conducting substrate was placed in the plasma. A background magnetic field of 130 G ran normal to the substrate surface to the ICP source above. The contour lines of the figure show the levels of constant phase space ion density. We see the bulk of the ions in the sheath have accelerated to 6 km/s towards the substrate. However, we also see additional features in this velocity-space tomograph. First, there is considerable perpendicular spread to the incoming ion velocity distribution function due to the ion heating process of the ICP source (here at 18.62 MHz). Second, there are also ions coming

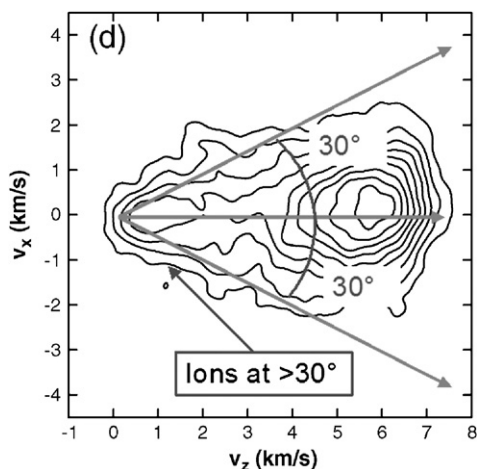


Fig. 4. Velocity space tomograph showing spread of ion velocities and associated incidence angles for ions in the sheath near a substrate.

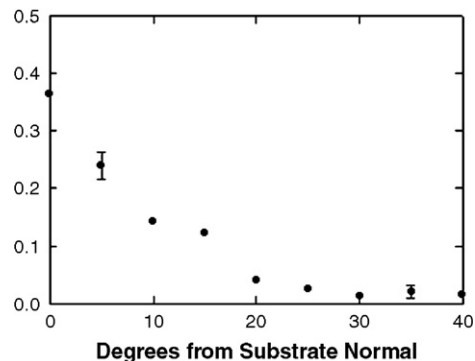


Fig. 5. Fraction of ions with incidence angle in the xz -plane in the sheath near a substrate. 5° sampling bins.

towards the substrate at much lower speeds than 6 km/s, all the way down to stationary. Charge exchange with neutrals can produce new ions within the sheath which have not been accelerated over the same regions as those ions born in the bulk plasma.

For the velocity space tomograph shown in Fig. 4, we may calculate the fraction of ions approaching the substrate for any given angle of incidence. This is interesting to know for etch and deposition processes which are sensitive to incoming ion incidence angle. The tomograph of Fig. 4 reveals the incidence angles in the $v_x v_z$ -plane. The fraction of ions as a function of incidence angle in this plane, displayed in Fig. 5, is found by integrating the phase space density as a function of angle. It is important to note that the velocity space plane projection of the tomograph of Fig. 4 shows just the angle for the ions in the xz -directions. Ions also have a yz -angular dependence. For example, included in the ions which show zero degrees in the xz -plane are all those ions which have that zero angle but include all possible angles in the yz -plane. The fraction of ions with incidence angles greater than some specified figure thus is greater than that shown in Fig. 5 assessment for the xz -plane. The direction of ion flow, not just in the average of the distribution but for each ion, may play a role in processes near surfaces. LIF can help in quantifying the importance of ion velocity distribution angular variations.

4. Conclusions

This brief overview with current examples describes a wide range of spectroscopy techniques for non-perturbing diagnosis of process plasmas. Whether laser-induced fluorescence, optical emission, or absorption spectroscopy is best depends on the energy states available and the populations in those states. Current applications of these diagnostics included radical density measurements, products in supersonic process flow regimes, etching ion beam properties, and ICP sheath ion properties.

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