

Active and passive spectroscopy in plasma deposition and plasma etching

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ACTIVE AND PASSIVE SPECTROSCOPY IN PLASMA DEPOSITION AND PLASMA ETCHING

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The advance of new plasma based techniques as deposition and etching has proceded by step to step research in the past decades. In the beginning this advance was achieved by phenomological research and as a first step relations between discharge parameters as pressure, power density, and flow and the deposition or etching results was sought for. At the same time a more fundamental approach started in which the plasma was simulated by ion— and radical—beams [1—2]. The etched gaseous products were analysed and so relations between etching result and kind and magnitude of fluxes could be established. In these experiments it was found that plasma surface modification has to be regarded as a synergetic process, i.e. the effect of the combined fluxes is more than the sum of the effects of ion and radical beams separately.

The last decennium the investigations were more and more directed to the study of relationships between deposition or etching result and plasma quantities as densities of ions and radicals and temperatures [3-6]. Only in this way one can find information on the fluxes and in relating these findings to the above mentioned beam studies one can

obtain a picture of the plasma modification process.

In relation with radicals a specific remark needs to be made: radical densities will not only be determined by volume production and diffusion, but also by wall processes as association and desorption, adsorption and etching. In many instances radical densities are enhanced significantly above the value which would result from the volume production and outward diffusion balance. As a consequence radical densities may give

more information about processes at the wall than about the plasma.

Active and passive spectroscopy can be used as a way to unravel the mechanisms of plasma surface modification, and to improve and optimize deposition and etching of thin layers. There are strong indications that plasma deposition differs from chemical vapour deposition in an essential way. First, ions and electrons contribute to the deposition process, not only radicals. Recombination of positive and negative charge may also lead to polymerization and space charge and magnetic fields may infuence these processes. Second, plasma deposition is in fact a balance of deposition and etching in which deposition overweights. Therefore in plasma deposition recycling through the plasma may assist the surface migration, as may be deduced from crystalline diamond deposition with high rates. In this framework also the study of recirculation in the deposition chamber close to the substrate should be pursued. Spectroscopy should be directed to the measurement of densities of electrons, ions (also negative), radicals, of convection and diffusion velocities, of electron and heavy particle temperatures (or energy distributions) and vibrational and rotational distributions.

Some words need to be said about plasma characterization. In ref.[7] several plasma types are characterized by their values for electron densities and ionization degree (here defined as the ratio of electron density, n_e and neutral density, n_o): n_e/n_o . This characterization has to be preferred as the electron temperature is a logarithmic quantity which is always close to $T_e \simeq E_i/4$ at low pressure to $T_e \simeq E_i/15$ at atmospheric pressure (E_i is the ionization potential). Nevertheless we will also pay attention to temperature

measurements, besides the determination of densities.

We will start the discussion with emission and absorption spectroscopy, very classical

but very powerful techniques. At the same time we will introduce some concepts on excitation equilibria, which are needed for our discussion.

EMISSION AND ABSORPTION SPECTROSCOPY

Emission spectroscopy is the most widely used diagnostics in plasma etching and deposition. It gives information on the density of excited states of atoms, molecules and ions. Ground state densities can be derived from it and also information on a (effective) temperature can be obtained. The system can be calibrated by placing a tungsten ribbon lamp at the location of the plasma, or at an equivalent position. Note that the small additional complication of absolute calibration (compared to relative) is well worth taking. If the plasma is observed sidewards then an Abel inversion is needed to convert a measured lateral profile to a radial profile; this inversion has to be performed at all wavelength points to obtain a λ -profiles at all radii. For non-cylindrically symmetric plasmas more general inversion procedures exist. If the apparatus profile is known, information on integrated line emission and continuum emission can be obtained. If the apparatus profile is smaller than the line profile additional information can be obtained

from the line broadening and shift.

The most important and used line broadening effects are Doppler broadening and Stark broadening. Doppler broadening and Doppler shift as measured by a high resolution monochromator or an additional Fabry Pérot gives direct information on the temperatures and velocity (the component along the line of sight) of the species observed. For species excited by electron impact the temperature and velocity is equal to those of the ground state particle. If the excitation arises from a dissociative channel as dissociative recombination, than some of the excitation energy will be transformed to kinetic energy. This gives rise to a pedestal in the line broadening superimposed on the smaller electron excitation feature. In the sheath of the RF-capacitively coupled discharges substantial potentials may arise. These accelerates ions, and excited neutral species which result from charge exchange or dissociative recombination in the sheath may show this velocity as a large shift when observed through the RF-electrode. [4],[6]. In ref. [8] an example is given of the velocity profile of an expanding arc plasma used for plasma deposition, which gives a good example of the possibilities of Doppler shift and broadening measurements. If performed cautiously, typical minimum detectable velocities of 10-50 m/s and thermal velocities of a few 100 m/s (room temperature) can

Stark broadening is the broadening caused by the microfields of the charged particle motion. For most atoms this Lorentzian broadening becomes only measurable at higher n_e ($n_e > 10^{22}/m3$), unless Doppler free measured (see LIF). Hydrogen lines show first order Stark broadening and it can be used to substantially lower electron densities. Recently much attention has been paid to Stark broadening at lower $n_e \ (\lesssim \ 10^{20}/m3)$ for

hydrogen Balmer lines [9],[10].

A third broadening effect, important only for Doppler free measurements at higher pressures is collisional broadening. If a line is partially self absorbed the absorption will be λ -dependent, which may lead to a deformation of the line profile: first flattening of the top, and at higher absorptions in the periphery of the plasma, even line reversal. In general, in absorption measurements care has to be taken about emission line profile and absorption line profile which may be different. Situations with sources with either a very small λ -profile (dye-lasers, diode lasers) or very broad (continuum sources) can most

If self absorption can be neglected, line emission experiments give direct information on absolute densities of the upper state of the transition. If absorption is not small it is possible to correct for it; usually it is better to perform an absorption experiment, as it is

more precise and gives information on the density of the lower state.

Some remarks on excited state distributions in several excitation equilibria are in order. Let us first discuss a simple atomic system with a large energy gap from the ground state to the first excited sate (e.g. noble gases, fluor, hydrogen). For ionizing plasmas with low electron densities $n_e \lesssim 10^{18}/m3$, the lower part of the atomic system is in corona equilibrium; i.e. it is populated by electron excitation and depopulated by radiation. The densities of these lower states are proportional to neno, and depend on the electron temperature through an exponential factor. Note, that in particular for ionizing plasmas, at these low electron densities the electron distribution function may differ appreciable from a Maxwellian one. If so, an effective Teeff can be defined which is related to the production efficiency of electrons.

In this corona range the use of actinometry has become quite popular: a line from a radical with unknown density (e.g. fluor) is compared with an argon line with similar excitation energies. Then the exponential factor and the electron density drop out in the ratio of the two lines; so this ratio can be used to obtain the ratio of the ground states, apart from a constant, provided that both levels are in the corona part of the system. The density of fluor atoms can then be deduced from the known density of the

actinometer admixture (usually argon).

For higher states, with quantum number $p > p_{cr1}$, the equilibrium is governed by excitation-deexcitation by electrons. These higher levels have populations governed by the excitation saturation phase and are only proportional to n_0 (or more precisely by the density of the lowest level of the ESP, p_{cr1}); as now also the deexcitation is proportional to n_e , the n_e -proportionality cancels out. Also in the ESP-range actinometry can be

Above a second critical level, p_{cr2} , the ionisation, dielectronic recombination dominates (PLTE). The level densities of these high lying states are in Saha equilibrium with the continuum: $n_p^s/g_p = \frac{n_e}{2} \frac{n^+}{g^+} \left[\frac{h_2}{2\pi m_e k T_e} \right]^{3/2} \exp\left[E_{pi}/k_{e}T_e\right]$, $p > p_{cr2}$ (1) Level density measurements in this range give information on n_e and/or T_e and not on n_o anymore. Conclusions on the electron temperature from the slope of the relative densities (the so-called Boltzmann plot) are possible but usually dengerous (Small densities (the so called Boltzmann plot) are possible but usually dangerous. Small influences may disturb the level distribution such that large changes in the apparent slope and thus apparent Te result.

For higher electron densities the critical levels p_{cr1} and p_{cr2} shift downward. For $n_e > n_{ecr1} \sim 10^{20}/m3$, the first excited level is already in the ESP-phase. At even higher densities ($n_e > n_{ecr2} \sim 10^{22}/m3$ typically) this level comes in equilibrium and the PLTE-plasma state is established. Full LTE is only arrived at very high densities and is

not discussed here.

The relationship between level densities and plasma parameters $(n_e, T_e, \text{ and } n_o)$ is usually discussed in terms of collisional radiative models. In these models the excited state density is related to ground state density and ion density through so called collisional radiative coefficients, r_p^0 and r_p^1 : [11,12]. $\frac{n_p}{g_p} = r_p^1 \cdot n_o + r_p^0 n^+$

Extensive work on cr coefficients has been performed on e.g. hydrogen and argon. Care has to be given to the cut off procedure, as was demonstrated recently by e.g. Van der Mullen et al. [12], [13].

In ionizing systems usually the first term of the RHS of eq. (2) dominates; provided that ne and Te are known, the neutral density can be determined if the r¹_p coefficient is known.

For electron densities above n_{ecr2}, the system is in PLTE and another procedure is preferred. Absolute density measurement of an excited state density by emission analysis can then directly be related to n_e and T_e through eq. 1. Even more is possible if the deviation from equilibrium of the ground state is not too large. This deviation is characterised by an overpopulation factor ,b₁, defined as the ratio of the actual neutral density as, n_1 , and the Saha density, n_1^s , as given by eq. 1 for p=1. $b_1 = \frac{n_1}{n_1^s}$

in which n_1^c is given by eq. 1 for p=1. If the pressure is known (e.g. 1 atm., as in normal pressure ICP's or arcs) one can investigate the sensitivity of the absolute level density to the deviation of b_1 from 1. This is shown in ref. [14] in which the level density of the 5d variations of $1/10 < b_1 < 10$ the resulting variation in T_e is only 20%, which indicates the possibilities of absolute level density measurements once more.

So far we have treated an atomic noble gas system only. In etching and deposition experiments usually molecular kinetics play a role. Also other systems like C, Si, H, etc. more ignore and some considerations on other systems are in order. Let us for one step and silicon. They differ in one respect from systems as noble gases, hydrogen and fluor in that they lack the large energy gap between the ground state and the first excited state systems as e.g. Ar and H, i.e. the carrier gas.

Let us first consider the possibilities emission and spectroscopy absorption here. Assuming quartz optics are used, absolute line emission experiments can now give information on levels which are more close to the ground state. If absorption can be measured, it is even possible to obtain information on the state. Hence, measurements in the visible and UV a can be obtained population distribution of the neutral and ion states. Absolute densities of lying excited states information on ne n+ if Te is known by using eq. 1. Of the latter an example is given from recent measurements in expanding arc deposition experiment [15]. In fig. 3 absolute level densities are given of H, Si, and systems. From the ratios knowing the electron temperature, the ion composition can be deduced; in this particular case it was Ar+; Si+:H+ : 10^{-3} $\mathbf{B}\mathbf{y}$ using quasi-neutrality $(n_e = \sum_{i=1}^{n} n_i^+)$ also the

electron density is obtained from eq. 1 giving $n_e \sim 3.10^{18}/m3$.

In the above atomic systems are discussed in the framework of the Quasi Steady State Solution as in common in CRM-work. However, for fast changes in time and/or space it has to be verified that these changes are slow compared to excitation/de-excitation processes. If

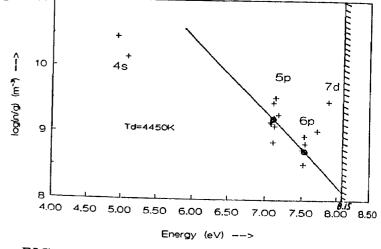


FIG. 1: The ecited state density of Si I vs. the energy from the groundstate [15]

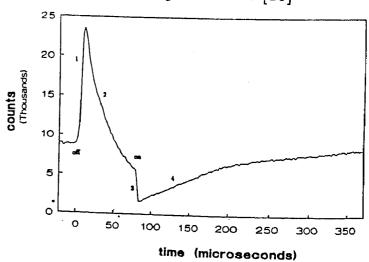


FIG. 2: Response of Ar(6d) level to an interruption of the RF power of an ICP plasma [16]

it is not the case, in repetitive pulsed off experiments, new information can be obtained from the time constants and the changes. As an example fig. 2 shows such an experiment in a ICP [16]. The level density of a high lying state *increases* at the sudden switch off of

the RF-power, with a time constant $\tau \sim \mu s$ which reflects the diffusion time in excitation space. The jump reflects the relaxation of T_e to the somewhat lower heavy particle temperature, T_h . In this way by measuring the reactions of several levels, both T_e and T_h can be deduced [16].

In the above we have ignored molecular processes, molecular intermediates, and heavy particle interactions. These processes can sometimes influence the distribution of excited states.

The first is near resonant asymmetric charge transfer. Taking again the case mentioned above the following process might be possible: $Ar^+ + Si \rightarrow Si^{+*} + Ar$. It is clear that such a process (which is known to be fast) will influence the state distribution of the Si II system: it will lead to a higher population of the with Ar^+ resonant Si II level. Other examples are to be found in ICP work, where a similar mechanism populates analyte ion lines (like Mg II).

A charge exchange with a molecule could lead to the following sequence:

 $Ar^+ + Si H \rightarrow SiH^{+*} + Ar$ followed immediately by dissociative recombination: $SiH^+ + e^- \rightarrow Si^* + H$. Again these processes will disturb the excitation characteristics of the molecular ion and of the Si-atom systems. At the other hand this means that in such circumstances the resulting emissions can be used to monitor the above given processes. The Doppler pedestals in the line profile can, as discussed before, help to obtain

information on these dissociative processes.

Again, it should be noted that these molecular influences will only be apparent in the low and intermediate n_e range $n_e \leq 10^{20}/m3$. At higher densities usually electron dynamics dominate and the earlier atomic case applies.

Note also that in some systems like F in Ar such a charge transfer is not possible. And

the actinometric determination of F concentrations is relatively safe.

In this section also some words should be said about molecular spectroscopy. Molecular spectra of diatomic molecules can be simulated and from comparison with measured spectra, rotational and vibrational temperatures can be deduced [17]. As above given examples processes like charge transfer may leave the product molecule also vibrationally and rotationally excited, it is not guaranteed that temperatures of electronically excited molecules are equal to those of the ground state. Then again absorption measurements are more powerfull.

Absorption spectroscopy is also an important tool to obtain densities of electronic ground state molecules. Vibrational excitation can be extremely important in chemical kinetics, [5] as has become clear from the thoroughly investigated negative ion production in H₂-gasdischarges [18]. Fourier transform infrared absorption spectroscopy (FTIAS), infrared diode laser absorption, can be employed to observe molecules in the ground electronic state and to investigate the vibrational and rotational population [19]. These distributions can be compared with the vibrational and rotational distribution of electronically excited states. Differences can then be attributed to dissociative channels which populate these states. In the thoroughly investigated H₂-discharge also the vibrational population of the ground state was found to be non thermally populated by another scheme: multiphoton ionization and ion detection [20].

Continuum analysis is another rewarding method to obtain the electron density in the higher density range. Free-bound and e.i. free-free transitions give a spectrum proportional to n_e^2 . The spectrum is flat to $h\nu=kT_e$ and then decays with an exponential factor. Measurements at long and short wavelengths give thus the possibility of measuring n_e and T_e provided $n_e > 10^{19}/m3$ and $n_e/n_0 > 10^{-2}$. For lower ionization degrees $n_e/n_0 < 10^{-2}$, the e-o-contribution dominates the continuum [21].

Let us conclude once more that, if performed with accuracy and common sense classical emission and absorption experiments give very rich information, in particular when they are done absolutely.

ACTIVE SPECTROSCOPY

1. Interferometry

A relatively old method to measure densities in the higher density range is interferometry [22]. In its basic form it consists of a source, an interferometer, and a detector. It is based on the fact that electrons influence the propagation of e.m. waves through an index of refraction which depends on the electron density and at higher pressures and high frequencies also on the neutral density.

 $n = \epsilon_r^{\frac{1}{2}} = (1 - \omega_p^2/\omega^2)^{\frac{1}{2}} = \text{with } \omega_p^2 = n_e e^2/m_e \epsilon_0$. Usually the applied frequency is much larger than the plasma frequency and the detected path change is proportional to change of electron density. The minimum detectable electron phase change determines (with the path length) the minimum detectable electron density. In practice this means FIR for electron densities around 10¹⁹/m3, IR (CO₂-laser) for the 10²¹/m3 range and visible light (He-Ne laser) for even higher densities. The demands on stability increase with decreasing wavelength.

In the high density range (thermal arcs, shock tubes) also the Schlieren arrangement has been used. It measures gradients of densities rather than densities itself. Reflectometry is another related method used in fusion research which would be applicable also in the higher ne plasma chemistry. For deposition and etching experiments FIR measurements would be feasible to determine electron density in schemes with higher ne. In the lower density range 8 mm wave interferometry or microwave detuning are methods to

determine n_e.

2. Thomson and Rayleigh scattering

Thomson scattering is the first active method used to determine local plasma properties: ne and Te can be determined with this method. The main problem is the very small Thomson scattering cross section which makes strong reduction of stray light essential. Present day arrangements make use of doubled Nd-Yag lasers and with care detection limits of 1018/m3 are possible. In fig. 3 an example is shown of a recent Thomson scattering on an expanding arc in which the shock is clearly visible [23]. For $n_e > 10^{18}/m3$ this method, though complicated, is very fruitful as it delivers two basic plasma parameters locally resolved. Also neutral densities can be measured in the central Rayleigh scattering channel if the stray light is sufficiently reduced.

3. Light Induced Fluorescence (LIF)

In this method photons are irradiated which are resonant with an appropriate transition. This irradiation leads to a disturbance of the (excited) state densities which can be monitored in another direction by observing the resulting change in emission at the same line or another line [6]. As also this method involves a crossed beam system it again gives locally resolved information. If the power is large enough, the pumped transition is saturated; the upper level density becomes equal with the lower densities (normalized for the statistical weights). Therefore absolute measurements of the change in level density and time constants do give useful information, not only on the original lower state density but also on plasma parameters as ne. Problems are similar as in Thomson scattering; now the cross section is resonantly enhanced, but the number of scatterers is lower which points to the achievable low detection limits. Also available pumped dye lasers have a wavelength range which is limited at the small wavelength side. This limits the applicability as regards to atomic systems, but in these impressive results are obtained [24]. In Rf excited capacitively coupled discharges the densities can be strongly dependent on location and on phase of the RF field. Then LIF has the advantage of very good spatial and temporal resolution, which can display such refined kinetics.

With the common, narrow line tunable dye lasers excitation also in LIF Doppler broadening and shift can be measured and give locally resolved information on

temperature and velocity, with, if needed, also temporal resolution.

On an even more refined scale Doppler free measurements can yield the Stark broadening. If the latter is determined by electron broadening the electron density can be determined in this way. Even more important is the determination of the electric field [25], [26]. In these experiments Stark mixing and Stark shifting are used to detect

spatially and temporally resolved electric fields. The findings helped the development of appropriate modelling enormously. This is the great potential of LIF; to obtain detailed measurements on densities and fields and velocities spectroscopically accessible systems. The resulting modelling can then be applied to other systems, also those in

which LIF is not possible.

Another detection method is optogalvanic detection. (LIF-OGE). [27]. A pumped transition will depopulate the lower (usual metastable) level. As a consequence metastable ionization decreases, but, higher n_e, stepwise ionization increases. Both effects will lead to (inverse) changes in the conductivity, in which determined is electron-neutral collisions at ionization degrees 1%. < optogalvanic detection is extremely sensitive and may offer advantages to common LIF disadvantage is the fact that in this case the measurement is not as local as in the common scheme.

4. Photodetachment

Negative ions pose a specific case in plasma chemical spectroscopy. They are very difficult to observe in mass

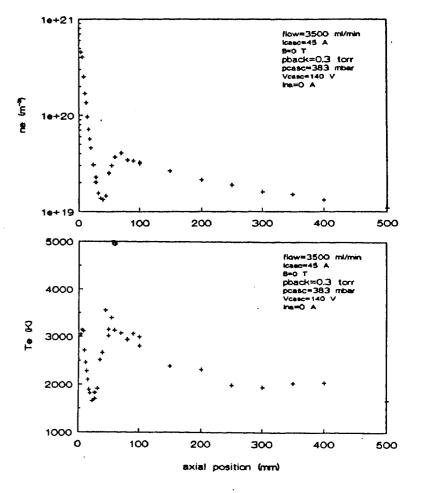


FIG.3: Electron densitutemperature vs. axial distance of an expanding thermal arc plasma [23]

spectrometry, because of the usual negative bias potential around the plasma. A good way to detect negative ions is the use of photodetachment and monitoring the liberated electrons by probes [28] or by microwave detuning techniques [29]. Time dependent studies have been performed [30].

Negative ions form an important (and unexplored) item in plasma chemistry as, without

exception, electronegative gases are involved.

5. Raman scattering and CARS

Raman scattering is scattering of e.m. light, in which the scattered light frequency is shifted from the incident light frequency by an amount which corresponds to an energy difference between rotational, vibrational or electronic levels. Like Thomson and Rayleigh scattering the main problem is the small cross section. This limits sensitivity and leads to the necessity of extremely good wavelength resolution with good rejection. The reward is the possibility to measure ground state densities of otherwise inaccessible systems, as H2. CARS (Coherent anti Raman scattering) involves a three wave mixing process. The scattering is in a specific direction which improves the yield. The necessity of coherence is however a complicating factor.

In this paper we have concentrated on volume spectroscopic methods and have ignored surface analysis methods, as ellipsometry and second harmonic generation. However, techniques which could give in situ information on surface processes are likely to become extremely important as the essentials of the plasma-surface interaction are still being discussed, in particular for deposition. With passive and active spectroscopy thus a complete picture of the etching and deposition mechanism will be finally obtainable.

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