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Status and challenges in electrical diagnostics of processing plasmas

Eugen Stamate

Department of Energy Conversion and Storage, Technical University of Denmark, Frederiksborgvej 399, Roskilde 4000, Denmark

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ABSTRACT

Dry processing based on reactive plasmas was the main driven force for micro- and recently nano-electronic industry. Once with the increasing in plasma complexity new diagnostics methods have been developed to ensure a proper process control during etching, thin film deposition, ion implantation or other steps in device fabrication. This work reviews some of the unconventional methods developed in the last two decays to measure the parameters of reactive plasmas including, the test function method, thermal probes, and plasma-sheath-lens probes. The negative ion detection and surface contamination in plasmas with a high degree of contamination are also addressed.

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1. Introduction

Reactive plasmas produced in oxygen, nitrogen, hydrogen and other complex gas mixtures are used for various applications including deposition of thin films [1–3], etching [4,5], ion implantation [6,7], particle growth [8], oxidation and other surface functionalization processes [9]. Most of the reactive gases are also electronegative so that, the role of negative ions cannot be neglected [10]. The continuous decrease of the feature size in micro- and nanoelectronic industry requires a precise control of plasma parameters including the negative ions [4]. Despite a good progress in plasma diagnostics [11–26], yet more is to be done for developing techniques compatible with the strict requirements for device-making plasma sources. Moreover the properties and possibilities to control the electronegative discharges are not completely understood [10,27,28]. Electrostatic probes have been used to diagnose electronegative plasma since 70s [11-13]. While this technique can give good results for density ratios of negative ion to electron higher than 10 its applicability for lower density ratios is questionable [29-35]. In this context it was demonstrated that the double hump structures observed in the electron energy probability function (EEPF) close to plasma potential cannot be associated with negative ion parameters because those structures are produced by a particular change in the work function over the probe surface due to discrete ion focusing [29,30]. Another way to detect the plasma parameters in the presence of negative ions is to use the high sensibility of the test function in the mid and low energy part of the distribution function [36,37]. The presence of negative ions is also associated with a lower heat flux to the probe, a fact that resulted in the development of a thermal probe that allows to record at the same time not only the current bias, but also a temperature bias characteristic [38-43]. The recent discovery of the discrete and

http://dx.doi.org/10.1016/j.surfcoat.2014.09.070 0257-8972/© 2014 Elsevier B.V. All rights reserved. modal focusing effects [44,45], associated with three-dimensional plasma-sheath-lenses [46], has created the possibility to detect even low densities of negative ions using the sheath-lens probe [47]. The positive ion extraction from reactive plasmas is rather easy. However, this is not the case for negative ions [48]. The influence of biased electrodes, of small or large dimensions on plasma parameters in electronegative discharges can give more information about the possibility to control and use these plasmas for processing, so that proper negative ion diagnostics is essential [27,28,49].

The aim of this work is to review some of the new developments in electrical probes for reactive plasmas including the test function method, thermal probes, plasma-sheath-lens probe and issues related to surface contamination.

2. Electrical probes

A single electrical probe (double or triple probes are also available) is an electrode of certain geometry (planar, cylindrical or spherical) immersed in plasma that collects a probe current, I_{p} , for a given applied voltage, V [11,12,50]. By sweeping V for negative and positive values with respect to plasma potential, V_{pl} , one can obtain the probe characteristic, $I_{\rm p}(V)$. Under the assumptions of isotropic plasma (Maxwellian distribution functions for electrons and ions with electron temperature, $T_{\rm e}$, and ion temperature $T_{\rm i}$, with $T_{\rm e} > T_{\rm i}$), no charge reflection or emission at the surface, a collisionless sheath thinner than the probe dimensions, and using a model for ion collection in the range of $V < < V_{pl}$ it is rather easy to extract the main plasma parameters. A typical $I_p(V)$ for a plasma with Maxwellian electrons is shown in Fig. 1, including the first and second derivative of I_p with respect to V (noted herein as I_p ' and I_p "), the electronic current, I_e , and the ionic current I_i . The cross point of $I_p = 0$ corresponds to the floating potential V_f . For plasma composed only of Maxwellian electrons and a single ion species,

E-mail address: eust@dtu.dk.

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Fig. 1. Typical $I_p(V)$ for a plasma with Maxwellian electrons.

collected by the probe under the orbital motion limited model, we have: $I_{\rm p} = I_{\rm e} + I_{\rm i}$ with,

$$I_e(V) = en_e S_v \sqrt{\frac{kT_e}{2\pi m_e}} \exp\left(\frac{e\left(V - V_{pl}\right)}{kT_e}\right)$$
(1)

$$I_{i}(V) = -en_{i}S_{\sqrt{\frac{kT_{e}}{2\pi m_{i}}}}\alpha \left(1 + \frac{e\left(V_{pl} - V\right)}{kT_{e}}\right)^{\beta},$$
(2)

where n_i is plasma density corresponding to I_i , S is the probe surface of radius r_p , m_e and m_i are the electron and ion masses, and α and β are two coefficients dependent on the probe geometry with typical values of $\infty = \frac{2}{\sqrt{\pi}}$ and $\beta = \frac{1}{2}$ for cylindrical probes and $\beta = 1$ for spherical probes [51,52]. Due to the edge effects the I_i of a planar probe for $V < V_{pl}$ is not constant and one can use the correction proposed by Johnson and Holmes [53] to calculate $I_i(V)$ as,

$$I_{i}(V) = -0.61eSn_{i}\sqrt{\frac{eT_{e}}{m_{i}}}\left(1 + I_{\delta}(V)\sqrt{\frac{1}{n_{i}}}\right)$$
(3a)

where,

$$I_{\delta}(V) = \frac{2\pi}{r_p} \sqrt{\frac{\varepsilon_0 T_e}{e}} 1.018 \left(\frac{\left(V_{pl} - V\right)}{T_e}\right)^{\frac{3}{4}}$$
(3b)

Taking into account an isotropic electron energy distribution function (EEDF), f_e , one can find integrating the electrons collected by probe the following relation [50],

$$f_e(V) = \frac{2m_e}{e^2S} \sqrt{\frac{2eV}{m_e}} \frac{d^2I_e}{dV^2}$$
(4)

which establishes a direct correlation between the EEDF and $I_{e''}(V)$. If $\varepsilon = eV$, then EEPF, f_p , can also be introduced as,

$$f_p(\varepsilon) = \frac{f_e(\varepsilon)}{\sqrt{\varepsilon}} \tag{5}$$

Additionally, n_e can be obtained by integrating $f_e(\varepsilon)$,

$$n_e = \int_0^\infty f_e(\varepsilon) \mathrm{d}\varepsilon \tag{6}$$

and the effective electron temperature, $T_{\rm eff}$ as,

$$T_{eff} = \frac{2}{3n_e} \int_{0}^{\infty} \varepsilon f_e(\varepsilon) d\varepsilon.$$
⁽⁷⁾

Using this set of equations the data processing steps to obtain the main plasma parameters, considering the $I_p(V)$ as a set of minimum 1000 points in a range that captures both ionic and electronic saturation regions, can be [36,54],

- 1) Calculate $I_{p'}$ and $I_{p''}$ and obtain V_{pl} as V giving the maximum value of $I_{p'}$ or zero crossing for $I_{p''}$.
- 2) Ions are entering the sheath with Bohm velocities so that their collection is controlled by $T_{\rm e}$. For $V < < V_{\rm pl}$, $I_{\rm p}(V) \approx I_{\rm i}(V)$ and also $I_{\rm i}'' < < I_{\rm e}''$ for V slightly below $V_{\rm pl}$, so it is a good approximation to obtain $T_{\rm e}$ from the logarithmic plot of $I_{\rm p}''$ or $T_{\rm eff}$ by Eq. (7) and use it to fit $n_{\rm i}$, under the condition $I_{\rm p} I_{\rm i} \approx 0$. Having $n_{\rm i}$ and $T_{\rm e}$, is possible to calculate $I_{\rm i}(V)$ and then subtract it from $I_{\rm p}$ as to obtain $I_{\rm e}(V)$.
- 3) Calculate EEDF and EEPF using $I_{e''}(V)$ and correct the T_{e} and T_{eff} values.
- 4) Calculate n_e from $I_e(V_{pl})$ and also using Eq. (6). The values should be reasonable close to n_i from $I_i(V)$.

3. Test function method

Electrical probes are now one hundred years old and yet there are new models describing the ion collection, additional effects resulted when investigating multi ion species plasmas, collisional sheath, secondary electron emission at the surface, and presence of additional groups of negative charges [11,26]. A large volume of work was reported in the last three decays and extensive reviews are available [11,12]. The combination of digital to analog and analog to convertors allows very fast data acquisition and several commercial system are available including compensated probes for RF discharges. However, the increasing complexity of plasma processing requires even more sensitive ways to detected deviations of EEDF from Maxwellian, if possible of fractions below 5%. Of particular interest are the electronegative discharges with applications for ion sources used for space propulsion, and low, or even charge free, etching by negative ions or neutral beams [15,49]. Thin films deposited in reactive plasmas produced in O₂, N₂, H₂ and other gas mixture are also of high interest for solar cells, thin film batteries and thin film fuel cells, thermoelectric materials, protective and surface functionalization coatings [50]. In order to address the diagnostics of such plasmas by probes better approaches are needed. One of the most common deviations of EEPF from Maxwellian is the bi-Maxwellian distribution, detected in DC and RF discharges where the electrons can be separated in two groups [20,54]. See for example the DC plasma produced by filaments where three distinct groups of electrons contributing to I_p have been identified [54]. The directive primary electrons emitted by the filament and accelerated in the cathode sheath to energies for tens of eV, the hot isotropic electrons resulted by thermalization of primary electrons after successive reflections by the magnetic confinement at the walls and the low temperature (or bulk) electrons resulted after ionization by primary and hot electrons. For certain plasma parameters (pressure, discharge current, injected power) it is easy to notice deviations from a Maxwellian EEDF, but this is not always the case. Using the EEPF representation helps us to assess the linear dependence of the logarithmic representation that can give a fast estimation of T_{e} . The problem arises when the deviation is not large and appears at higher energies where the logarithm is more sensitive, so the useful information is covered by noise. Such an example is shown in Fig. 2 where the logarithm of I_{p} " and $(I_{p} - I_{i})$ " is presented for $I_p = I_e + I_{eh} + I_i$, corresponding to two groups of electrons, of $T_{\rm e} = 1.5 \text{ eV}$ (bulk) and $T_{\rm eh} = 5 \text{ eV}$ (hot) with a density ratio of bulk to hot electrons, $n_{\rm e}/n_{\rm eh} = 50$. As presented above the $I_{\rm i}$ contribution can

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