Probe Measurements of Electron Energy Distributions in Low Temperature Plasmas

V. A. Godyak

RF Plasma Consulting, Brookline, MA 02446, USA and University of Michigan, Ann Arbor, MI 48109, USA

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Introduction

"There is no plasma diagnostics method other than probe diagnostics where the danger of incorrect measurements and erroneous interpretation of results are so great."

L. Schott, *Plasma Diagnostics*, Amsterdam, 1968

It was true then, it is even more true today, when plasmas are more complicated and the measurement techniques are more sophisticated

The Langmuir probe is the most universal and informative plasma diagnostics tool

However, the simplicity of the probe concept frequently leads to abuse this technique resulting in erroneous results

Classical Langmuir Probe

Isotropic EVD, $a[ln(l/2a)] \ll \lambda_e \rightarrow \lambda_D$, $I_p \ll I_d$



Langmuir probe I/V characteristic (Classical Langmuir technique)



Shown in textbooks probe characteristics correspond to a large probe which gives a nice looking probe I/V, but prone to large plasma perturbation and distorted data

$$I_p = I_{es} exp(-eV/T_e) - I_i(V) \qquad I_{es} = (1/4)Senv_{Te}$$

Main problems in using Langmuir procedure:

- 1. Assumption of Maxwellian EEDF \longrightarrow errors in T_e and n
- 2. Uncertainty in plasma potential V \longrightarrow error in n

3. Arbitrariness in the ion current approximation \longrightarrow error in T_e for high energy electrons ($\varepsilon > |eV_f|$)

Basic requirements for validity of probe measurements

- Probe survives (is stable) in plasma
- No global plasma perturbation ($r_p \ll L$, $I_p \ll I_d$)
- Local perturbation is negligible, or well accounted for by a theory ($r_p \ll \lambda_e$)
- No plasma electromagnetic field and current stray interaction with probe
- Ability to make an accurate electrical measurement

Problems in Langmuir Probe Diagnostics

Majority of the published probe experiments are mediocre and of bad quality

- Bulky probes
- Low frequency noise and drift
- Rf plasma potential
- Probe contamination and sputtering
- Strong plasma rf field
- Probe circuit resistance

Low temperature plasmas in applications, space and laboratory are not Maxwellian

Electrons are not in equilibrium with ions and neutral gas $(T_e >> T_i \text{ and } T_g)$

Electrons are not in equilibrium within their own ensemble (not Maxwellian)

Electrons can be in non-equilibrium with electromagnetic field (nonlocal electron kinetics and electodynamics)

The classic Langmuir probe (and spectroscopy) in a real, mom-Maxwellian plasma can give erroneous results. Therefore, the plasma diagnostics, as well the theory and simulation must be done on kinetic level

Probe characteristics and EEDFs in Argon CCP

Godyak et al, J. Appl. Phys. 73, 3677 (1993)



Deviation from Maxwellian EEDF for high energy electrons ($\varepsilon > \varepsilon^*$, ε_i , ε_w) is typical for gas discharge plasmas. Strong non-equilibrium for bulk electrons ($\varepsilon < \varepsilon^*$, ε_i , ε_w) in DC and RF discharges in Ramsauer gases makes the <u>classical Langmuir procedure</u> inappropriate for diagnostics of such plasmas

Plasma parameters for Ar CCP at 30 and 300 mTorr

30 mTorr (bi-Maxwellian)		300 mTorr (Druyvesteyn-like)			
Langmuir	EEDF	Langmuir	EEDF		
$T_{e} = 0.73 \text{ eV}$	$T_{e} = 0.67 eV$	$T_{e} = 1.37$	$T_{e} = 3.4 \text{ eV}$		
$n=5.9 \times 10^9 \text{ cm}^{-3}$	$n=4.4 \times 10^9 \text{ cm}^{-3}$	$n=4.5 \times 10^9 \text{ cm}^{-3}$	n=2.9x10 ⁹ cm ⁻³		

The temperatures of cold and fast electrons in bi-Maxwellian plasma found from the Langmuir procedure and those found from the EEDF are different!

$$T_{ec} = 0.73 \text{ eV}$$
 and $T_{he} = 4.2 \text{ eV}$ are found as $T_e^{-1} = dlnI_e/dV$

 $T_{ec} = 0.50 \text{ eV}$ and $T_{he} = 3.4 \text{ eV}$ are found as $T_e^{-1} = dlnf_e/dV$

Error factor n/n_{EEDF} found by different authors

TABLE I.	Plasma	density	error	factor	n/n_0 .
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Source	Gas/pressure	EPPC	Ion OMT	Ion RMT	Cut-off pr.	Hairpin pr.
Ref. 10	Ar, 30 mTorr	1.34	2.5	0.16		
Ref. 10	Ar, 0.3 Torr	0.38/0.07	3.3	1.4		
Ref. 18	He, 40 mTorr	0.85	9	0.25		
Ref. 21	Ar, 7–22 mTorr		2.6-3.25		1.1	1.5
Ref. 19	Ar, 1 mTorr		≈ 3	0.3		
Ref. 19	Ar, 10 mTorr		≈ 4	0.45		
Ref. 19	Ar, 0.1 Torr		≈ 2	0.14		

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Langmuir formula and Druyvesteyn method

$$I_{e} = \frac{2\pi e S_{p}}{m^{2}} \int_{eV}^{\infty} (\varepsilon - eV) f(\varepsilon) d\varepsilon = \frac{eS_{p}}{2\sqrt{2m}} \int_{eV}^{\infty} (\varepsilon - eV) \frac{F(\varepsilon)}{\sqrt{\varepsilon}} d\varepsilon = \frac{eS_{p}}{2\sqrt{2m}} \int_{eV}^{\infty} (\varepsilon - eV) f_{p}(\varepsilon) d\varepsilon$$
$$\frac{d^{2}I_{e}}{dV^{2}} = \frac{2\pi e^{2}S_{p}}{m^{2}} f(eV) = \frac{e^{2}S_{p}}{4} \sqrt{\frac{2e}{mV}} F(\varepsilon) = \frac{e^{2}S_{p}\varepsilon}{4} \sqrt{\frac{2e}{mV}} f_{p}(\varepsilon).$$
(7)
$$N = \frac{2\sqrt{2m}}{e^{\frac{3}{2}}S_{p}} \int_{0}^{-\infty} I_{e}^{\prime\prime}(V) \sqrt{V} dv \qquad T_{e} = \frac{4\sqrt{2m}}{3e^{\frac{3}{2}}NS_{p}} \int_{0}^{-\infty} I_{e}^{\prime\prime}(V) V^{1.5} dv.$$

Similarly, all plasma parameters (T_{esk} , λ_D , J_B) and rates of plasmachemical processes (v_{ea} , v_{ee} , v^* , v^i ,) can be found as appropriate integrals of the measured EEPF.

What makes a good EEDF measurement?

- EEDF has to resolve tail electrons ($\varepsilon > \varepsilon^*$) responsible for excitation, ionization and electron escape to the wall, as well as low energy electrons ($\varepsilon < T_e$) accounting the majority of electrons. In the majority of published EEDF, the both groups of electrons are practically absent.
- Due to error augmentation inherent to differentiation procedure, small (invisible) inaccuracy in $I_p(V)$ can bring enormous distortion in the measured EEDF.
- It is important to realize the source of the possible errors and to be able to mitigate them.
- The sources of the errors are well elucidated in the literature, but are insistently ignored in the majority of published papers.

Problems in probe measurements and their mitigations.

- 1. Probe size: $a[ln(\pi l/4a)]$, b is probe holder radius
- 2. No collisions in the probe sheath: $\lambda_D \ll \lambda_e$ and
- 3. To neglect the voltage drop across the wall sheath, the following requirement has to be satisfied:

 $(S_p N_0 / S_{ch} N_s) (M / 2\pi m)^{1/2} << 1$

- 4. Probe surface has be clean
- 5. No RF and LF plasma potential oscillations



Probe constructions

Telescopic probe construction



Probe circuit resistance (the most frequent problem)





EEPF Druyvesteynization due to R_c



Error in EEPF less than 3% requires $R_c/R_{pmin} < 1\%$!

Example of EEPF measurement in argon ICP with a low disturbance probe having R_c and noise compensation



Maximal argon pressure, where measurement was possible, was limited by the chamber surface



In a high density plasmas, EEPF at low energy must be Maxwellian



RF plasma potential

Criterion for undistorted by rf sheath voltage EEPF measurement is known for over 30 years (1979)

$$Z_{pr}/Z_f \leq (0.3-0.5)T_e/eV_{plrf}$$

A presence of a filter in the probe circuit does not guarantee undistorted EEPF measurement. To do the job, the filter has to satisfy the following condition for all relevant harmonics:

$$Z_{f} \geq (2-3)Z_{pr}eV_{plrf}/T_{e}$$

For filter design we need to know (measure!) V_{plrf} and minimize Z_{pr}

Filter design procedure





0.1 MΩ

MAX

27 MHz 13.5 MHz

to P₂

to P₃

200

30 pF

 $0.1 M\Omega$ $0.1 M\Omega$

27 MHz

to OP₂

to OP,

100

Probe measurement circuit incorporating, dc voltage and low frequency noise suppression, rf compensation and rf filter dc resistance compensation with I/V converter having a negative input resistance



probe voltage

Example of EEPF measurement demonstrating a paradoxical T_e distribution in argon 13.56 MHz CCP

 $30 \text{ mTorr, }_{Teo} < T_{es}$

 $300 \text{ mTorr}, T_{eo} > T_{es}$



EEPF and plasma parameters evolution during CCP transition to the γ - mode



Time resolved EEPF measurements

EEPF measured in afterglow stage of ICP with internal ferrite core inductor



EEPF and T_e measured along rf period in an induction lamp operated at 300 mTorr Ar and 7 mTorr Hg



Probe sheath capacitance to plasma is the main limiting factor in EEPF measurement speed. The minimal time resolution, $\delta t > (10-30)\omega_{pi}$

EEDF in running striations, argon, p = 10 mTorr, $I_d = 1$ A



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Ion current effect

$$I_p = I_e + I_i$$
 and $I''_p = I_e'' + I_i''$

Ion current effect occurs in light gases, low density plasmas and small probes, and is maximal at orbital ion motion ($a/\lambda_D < 1$), according to:

$$I_{e}"/I_{i}" = (4\pi M/m)^{1/2}\eta^{3/2}exp(-\eta) \qquad \eta = eV/T_{e}$$

independently on a/λ_{D}

In practice, I_i " effect is essentially smaller and can be neglected Ratio I_e "/ I_i " depends on EEPF and a/λ_D

Relative I_i" effect for orbital and radial ion motion for Maxwellian and Druyvesteyn distributions



Probe surface effects:

- Probe surface work function (changes during probe scan)
- Non conductive layer of reaction product \rightarrow (R_c)
- Sputtering of electrode and probe and deposition conductive layer on the probe holder $\rightarrow S_p$
- Strong temperature dependence of all above

REMEDIES

- <u>Continuous</u> probe cleaning with fast probe scan (mS)
- Ion bombardment, electron heating and rf biasing
- Probe cleaning before or after probe scan is ineffective

EEPF measurements with MFPA in Plasma reactors

(Wide specter with large amplitude of rf plasma potential and high rate of probe contamination are the major problems)

EEPF in commercial two-inductor ICP in different processing mixtures at 15 mTorr. *Mattson Technology, by V. Nagorny* EEPF measured in ICP reactor, H_2/CF_4 at 30 mTorr with a polymer layer deposition. University of Maryland by N. Fox-Lyon







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A remark

Today, plasma simulation codes are practically main tool for study plasma electrodynamics, plasma transport and plasma kinetics in commercial plasma reactors, space and lab plasmas. These codes applied to complicated processing gas mixture are missing many cross sections for variety of (accounted and not) plasma-chemical reactions.

They also are missing effects of nonlocal and nonlinear plasma electrodynamics that has been proved can be important and even dominant in rf plasmas at low gas pressure.

In such situation, a feasibility of EEDF measurement in low temperature plasma would give a valuable experimental data for understanding variety of kinetic process in such plasmas and validation of existing numerical codes.