PROBE DIAGNOSTICS OF RF PLASMAS FOR MATERIAL PROCESSING

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Fundamental Processes, Modeling and Diagnostics of Low-Temperature Plasmas

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“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, in *Plasma Diagnostics*, ed.
W. Lochte-Holtgreven. Amsterdam,1968

45 years later when plasma and its diagnostics got more complex, that is at least as much true.

Langmuir probe diagnostics

- Langmuir probe is a powerful diagnostic tool for low pressure gas discharge plasmas
- Practically all today knowledge on gas discharge was obtained with plasma spectroscopy and Langmuir probes (both imply a Maxwellian EEDF)
- The ability of measurement of local plasma parameters and the electron energy distribution function (EEDF) makes it unique among other diagnostics
- EEDF is the most informative, universal and complete characteristic of the plasma electrons in any plasma
- Basic plasma parameters (N and T_e) and rates of plasma-chemical processes can be found as appropriate integrals of the measured (non-Maxwellian) EEDF

Non Equilibrium Gas Discharge Plasmas Diagnostics

Gas pressure, p - between fraction and hundreds mTorr
Mean electron energy, $<\varepsilon>$ – between fraction and tens eV
Plasma density, N – between 10^6-10^{14} cm^{-3}

In plasmas, electrons are often not in equilibrium with RF field, nor with ions and atoms, nor within its own ensemble, i.e. have a non-Maxwellian EEDF with $T_e >> T_i$, T_g

When we have no idea what is a real EEDF, we assume it to be a Maxwellian. Indeed, in low pressure gas discharges, it is always non-Maxwellian

Classical Langmuir probe diagnostics (based on electron and ion current), as well as many other diagnostics, assume a Maxwellian EEDF. Found this way plasma parameters and <u>especially rates of inelastic processes</u> can be in dramatic disagreement with their true values

Langmuir formula and Druyvesteyn method

$$I_e = \frac{eS_p}{2\sqrt{2m}} \int_{eV}^{\infty} (\varepsilon - eV) \frac{F(\varepsilon)}{\sqrt{\varepsilon}} d\varepsilon = \frac{eS_p}{2\sqrt{2m}} \int_{eV}^{\infty} (\varepsilon - eV) f_p(\varepsilon) d\varepsilon.$$

I. Langmuir, Gen. Electr. Rev. **25,** 1924

$$Normalization: \quad N = \int_0^\infty F(\varepsilon) d\varepsilon = \int_0^\infty \sqrt{\varepsilon} f_p(\varepsilon) d\varepsilon.$$

Druyvesteyn formula

$$\frac{d^2 I_e}{dV^2} = \frac{e^2 S_p}{4} \sqrt{\frac{2e}{mV}} \boldsymbol{F}(\varepsilon) = \frac{e^3 S_p}{2\sqrt{2}} f_p(\varepsilon)$$

M. J. Druyvesteyn, Z. Phys**.** **64**, 781, 1930

Plasma density and effective electron temperature are:

$$N = \int_0^\infty \sqrt{\varepsilon} f_p(\varepsilon) d\varepsilon, \qquad T_e = {}^2\!/_3\, N^{-1} \int_0^\infty \varepsilon F(\varepsilon) d\varepsilon.$$

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

What makes a good EEDF measurement?

• EEDF has to resolve the tail electrons ($\varepsilon > \varepsilon^*$) responsible for excitation, ionization and electron escape to the wall, as well as the low energy electrons ($\varepsilon < 2T_e$) accounting for the majority of electrons

• Due to error augmentation inherent to differentiation procedure, small (invisible) inaccuracy in $I_p(V)$ can bring enormous distortion in the inferred 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 on EEDF measurement with home made and commercial probe system.

• The constrains for the Druyvesteyn method applicability coincide with those for the classical Langmuir method

• There are techniques for EEDF measurement in collisional, magnetized and anisotropic plasmas (not considered here)

See review by Godyak and Demidov, *J. Phys. D: Appl. Phys.* ***44****, 233001, 2011*

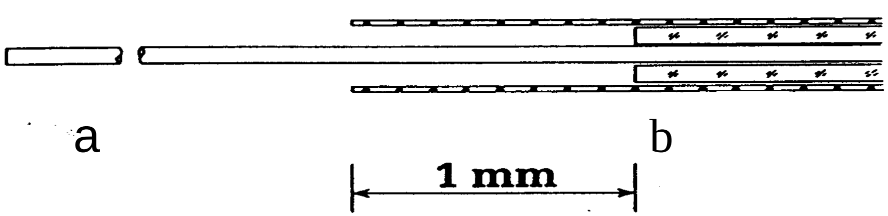
Problems in probe measurements and their mitigations.

1. Probe size: $a\ [\ln(\pi l/4a)],\ b,\ \lambda_D << l_e$ and $I_p << I_d, I_r, I_z$

$I_r \approx I_B = S_{ch} e N_s u_B$, is the current emission of the counter electrode

$u_B = (T_e/M)^{1/2}$, $I_z = e\Gamma_e$ is the current corresponding to generation rate of electrons with energy ε in the volume defined by the chamber characteristic size Λ, or by the electron energy relaxation length λ_ε

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

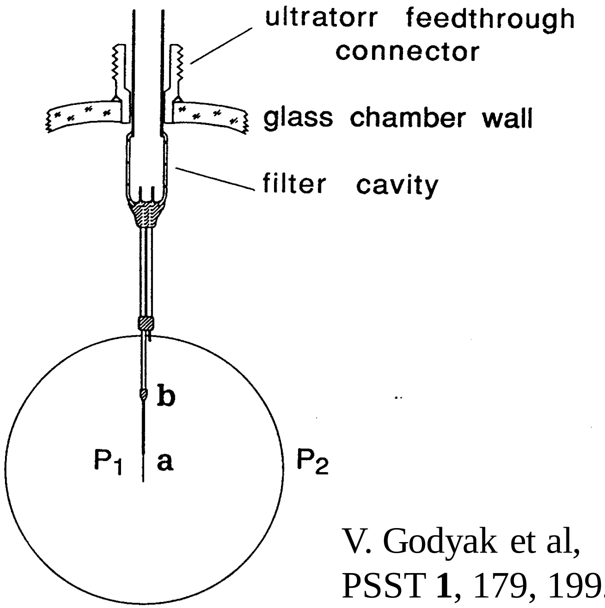


a = 38 μm

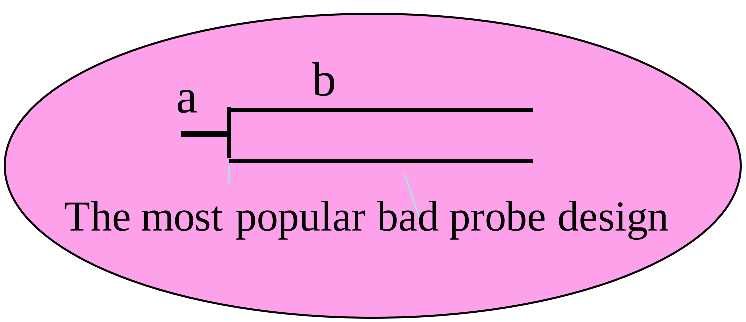
b = 175 μm

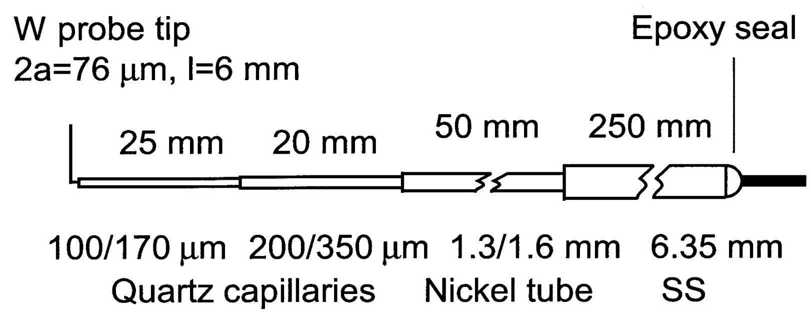
V. Godyak et al, PSST **1**, 179, 1992

Probe constructions

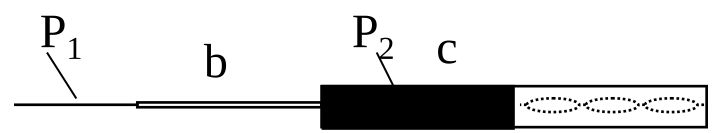


V. Godyak et al,
PSST **1**, 179, 1992





V. Godyak et al,
PSST **11**, 525, 2002



2a = 0.1 mm, 2b =1 mm, 2c = 6 mm

Plasma Sensors Probe System®
www.plasmasensors.com

RF plasma potential

Criterion for undistorted by the probe rf sheath voltage EEPF measurement is known for over 30 years

V. Godyak and S. Oks, Sov. Phys. -Tech. Phys. **24**, 784, 1979

$$V_{shrf} \leq (0.3\text{-}0.5)T_e/e$$

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:

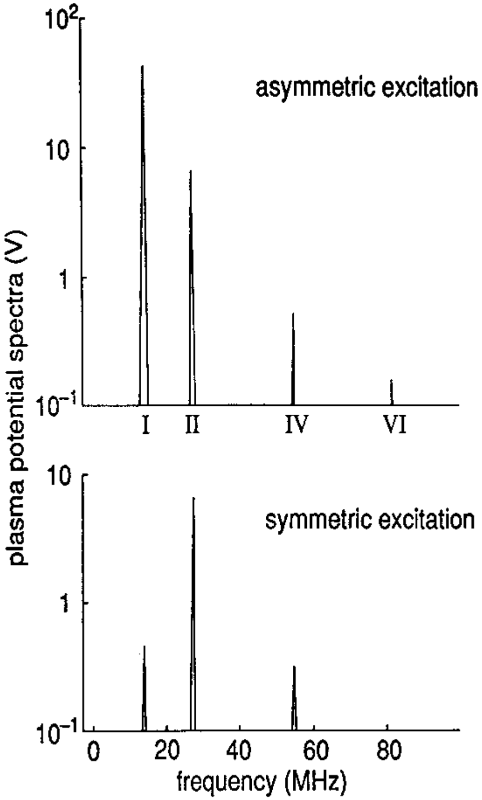
$$Z_f \geq (2\text{-}3)Z_{pr}eV_{plrf}/T_e$$

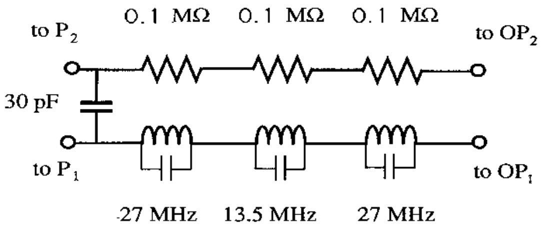
For filter design one needs to know (measure, calculate) V_{plrf} and minimize Z_{pr}

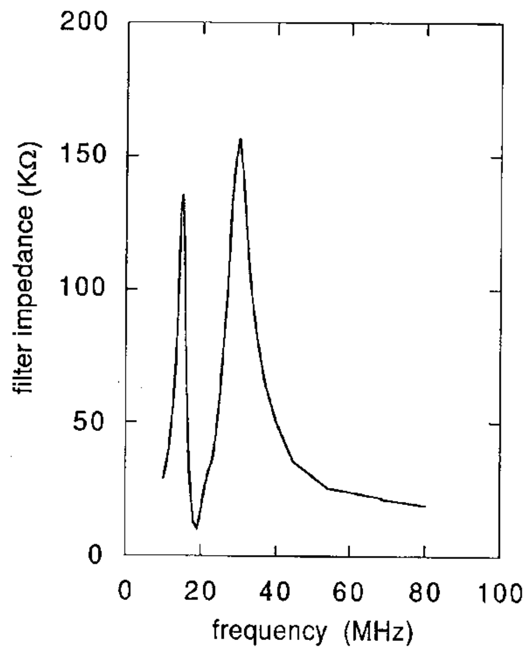
Z_{pr} is the impedance between the probe and plasma (Z_{pr} is defined by its sheath capacitance at floating potential, Z_f is the filter impedance, V_{plrf} is the rf plasma potential reference to ground, V_{shrf} is the rf Voltage across the probe sheath, and T is electron temperature

Filter design procedure

CCP at 13.65 MHz, V = 100 V



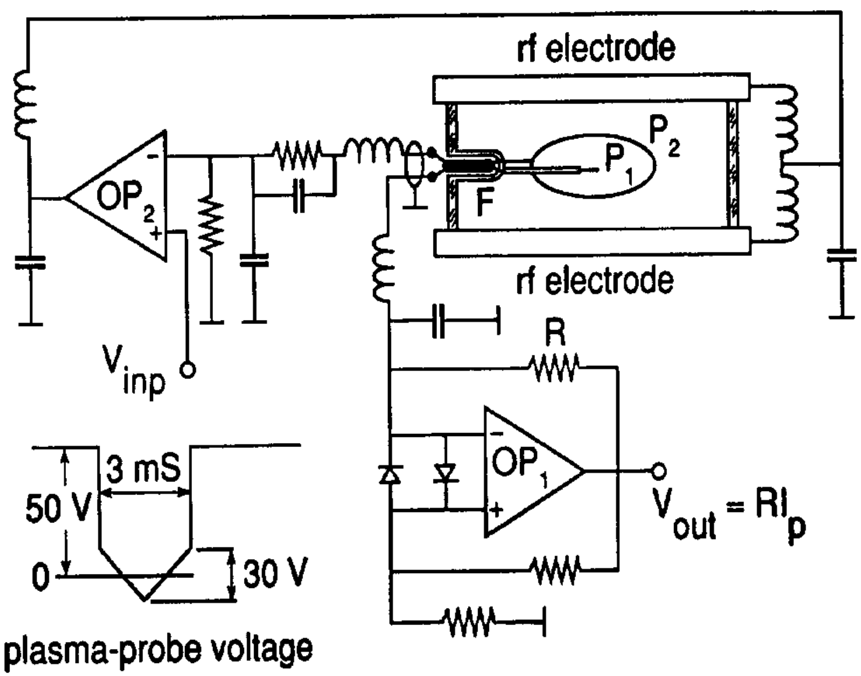


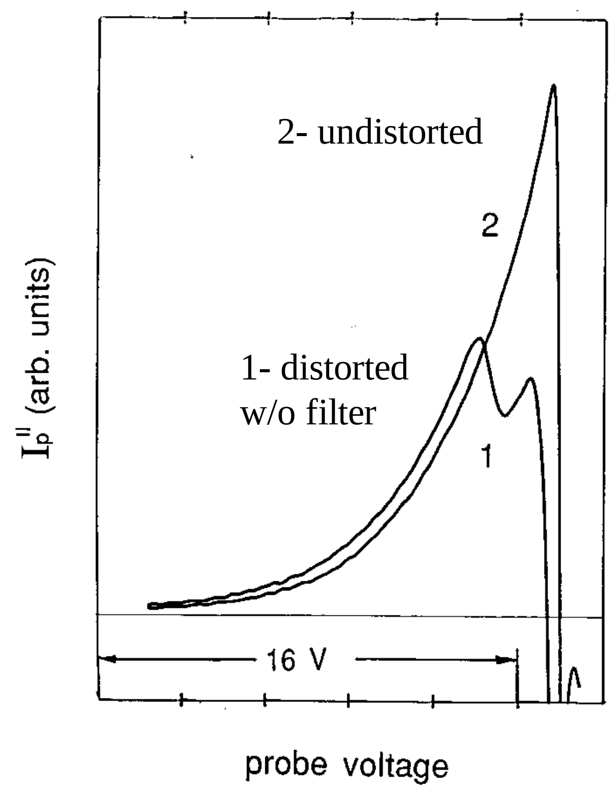


The filter has to be designed after the measurements of the rf plasma potential spectrum!

V. Godyak et al,
PSST **1**, 179, 1992

Probe measurement circuit for EEPF measurement in Ar CCP, incorporating, dc voltage and low frequency noise suppressions, rf compensation and rf filter dc resistance compensation with I/V - converter having a negative input resistance (gyrator)



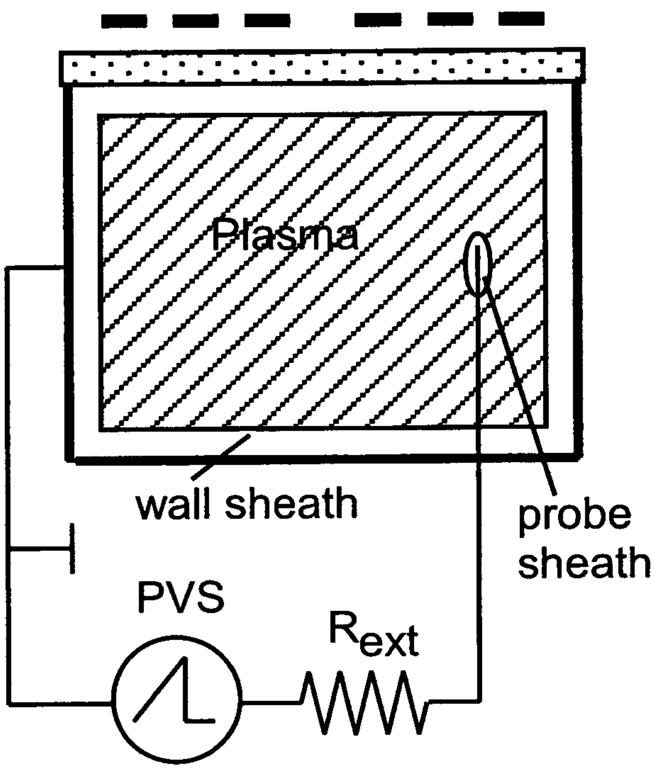


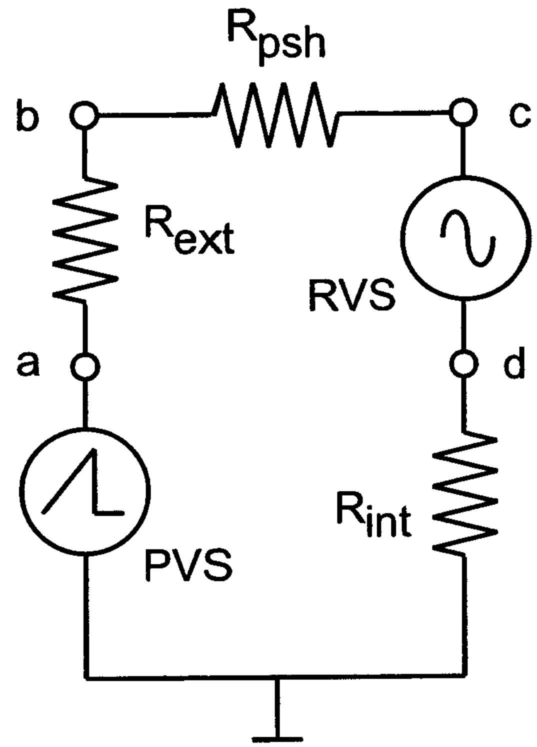
V. Godyak et al, PSST **1**, 179, 1992

Probe circuit resistance R_c (the most common problem)

$$R_c = R_{ext} + R_{psh} + R_{int}$$

The voltage V applied to the probe is distributed along the probe circuit elements (R_{ext}, R_{psh}, R_{int}), thus, $V_{psh} < V$!

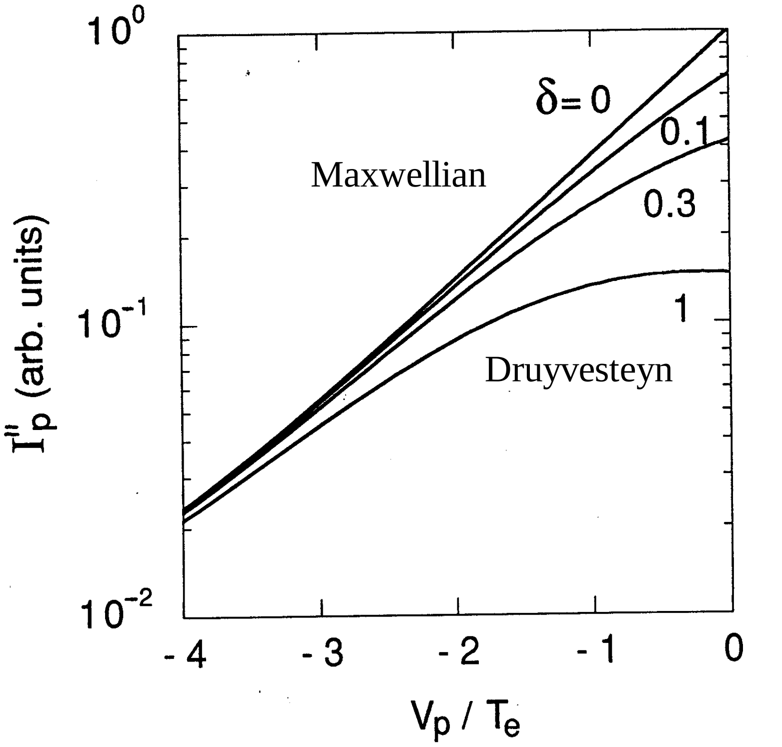




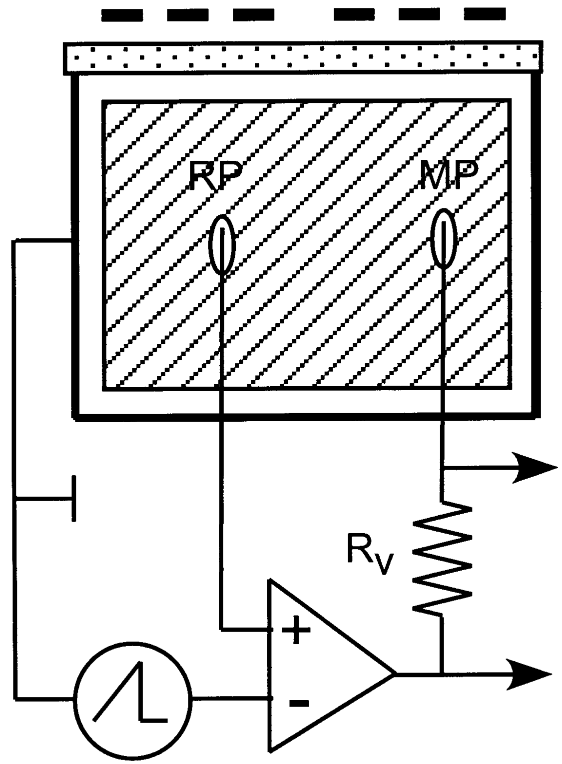
V. Godyak et al,
PSST **11**, 525, 2002

EEPF Druyvesteynization due to circuit resistance R_c

$\delta = R_c/R_{pmin}$ $\quad R_{pmin} = T_e/eI_{esat}$



R_c and LF noise compensation



Error in EEPF less than 3% requires $R_c/R_{pmin} < 0.01$!

V. Godyak et al,
PSST **11**, 525, 1002

Specifics of probe diagnostics in plasma processing chambers

- High plasma RF and dc voltage with wide RF spectrum (multi-frequency)
- High low-frequency noise typical for molecular and electronegative gases
- Bead electrical contact between plasma and grounded chamber
- Probe contamination with reaction products

Probe measurements in processing RF plasma are usually distorted. The problem is not recognized when one just measures the probe I/V characteristic, since distorted and undistorted probe characteristics look very similar. But the problem becomes apparent after differentiation the I/V characteristic to get the EEDF

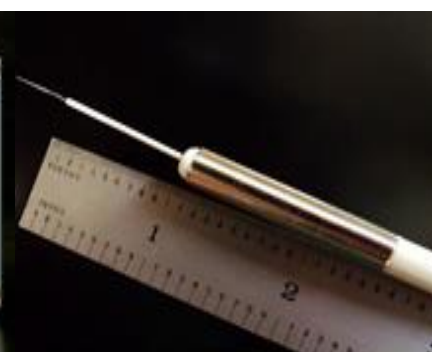
REMEDIES:

- Feedback with reference probe to <u>compensate R_c and LF noise</u>
- <u>Continuous</u> <u>probe cleaning</u> with ion bombardment, electron heating together with <u>fast</u> probe scan (mS)
- <u>Adequate</u> <u>RF filtering</u> for all relevant RF plasma frequencies and potentials

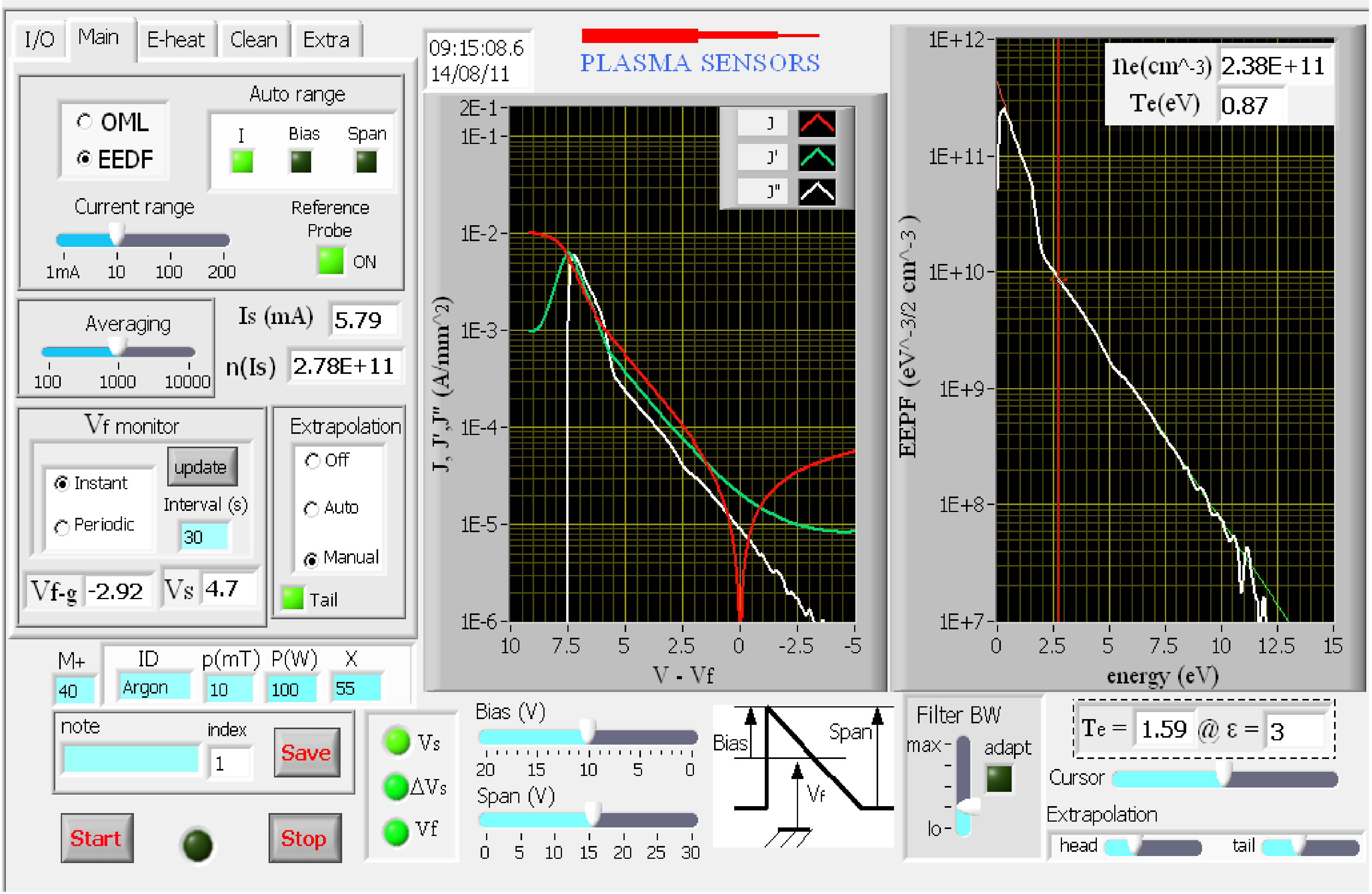
Plasma Sensors Probe System MFPA

The aforementioned problems has been addressed in MFPA instruments

- Reliable high quality EEDF and plasma parameter measurements in research and industrial CW and pulsed plasmas in noble and reactive gases, having high DC (up to 1 kV) and RF voltages and multi-frequency drive (2-200 MHz)
- User friendly interface, automatic ranging, real time processing, display, and data output of the plasma parameters
- High energy resolution and dynamic range of MFPA enables precise analyses of low and high energy parts of EEDF, correspondingly representing the bulk plasma behavior and the electron impacted inelastic processes
- MFPA eliminates errors common to existing commercial probe systems, which are caused by the probe work function variation, the plasma potential drift, and the plasma potential bias by the probe current
- Control of peripheral devices such as motor drives, electrostatic energy analyzers, probe array multiplexors can be seamlessly plugged into MFPA software routine. The MFPA real-time output data can be used for the endpoint detection and deposition rate assessment
- MFPA employs automatic probe cleaning by the ion bombardment and heats it to a preset temperature (500 -1500°C) with the electron current
- MFPA allows for measurements of the ion current with probes that are contaminated or chambers coated with non-conductive materials



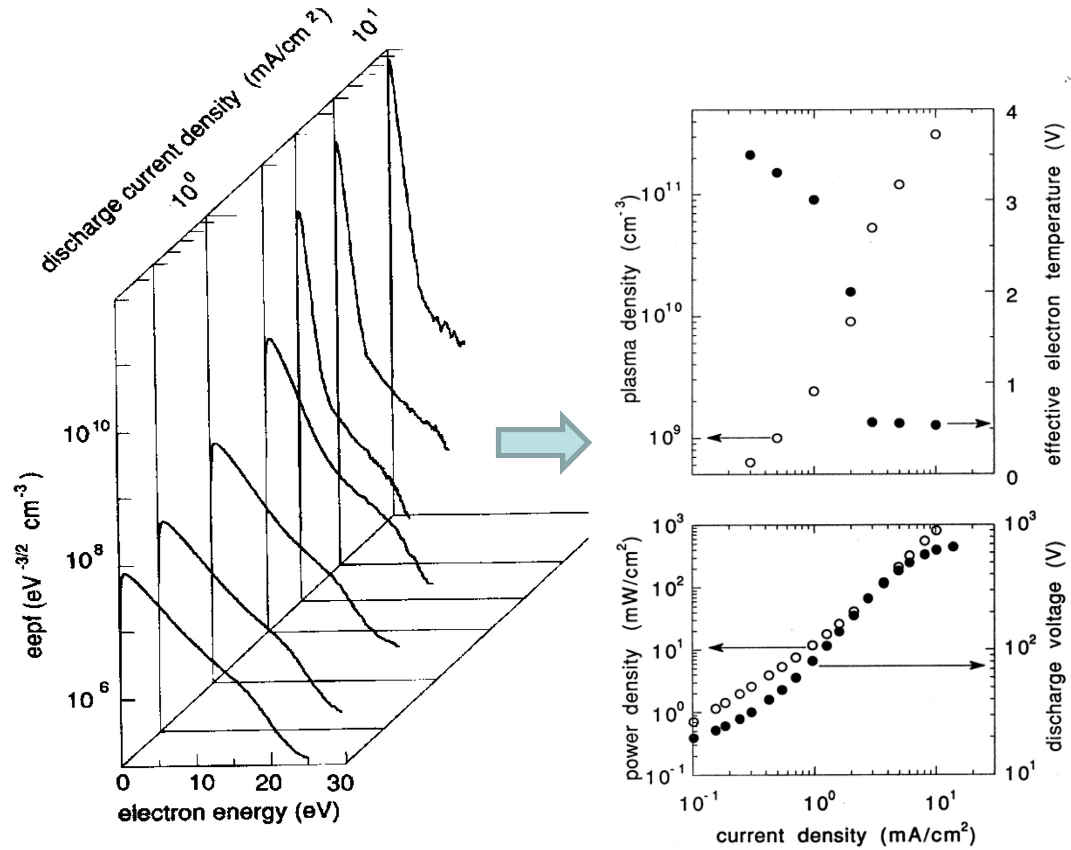

MFPA Display



Examples of EEPF evolution measurements in CCP at 13.56 MHz

Heating mode transition in Ar

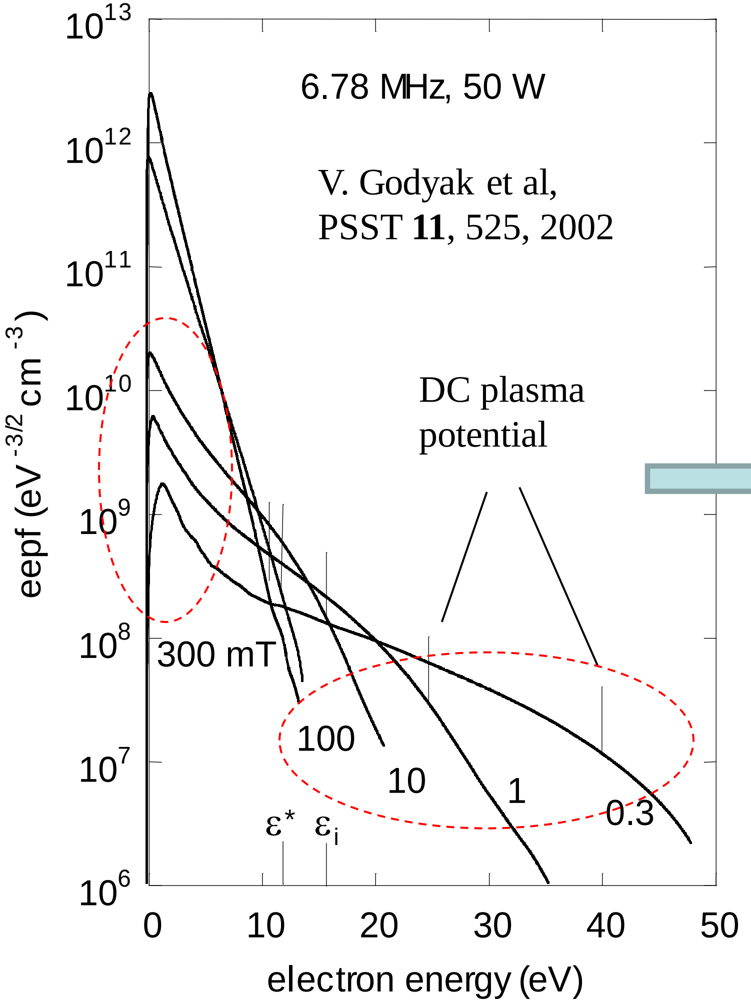
Transition from the α to the γ - mode



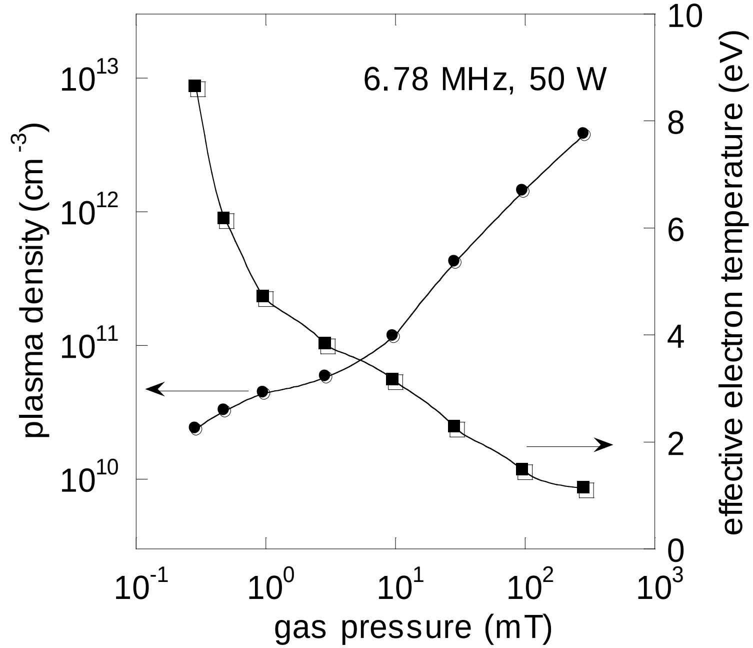
V. Godyak et al, Phys. Rev. Lett. **65**, 996, 1990

V. Godyak et al, Phys. Rev. Lett. **68**, 49, 1992

Example of EEPF measurement in argon ICP with a probe having adequate R_c and RF noise compensation



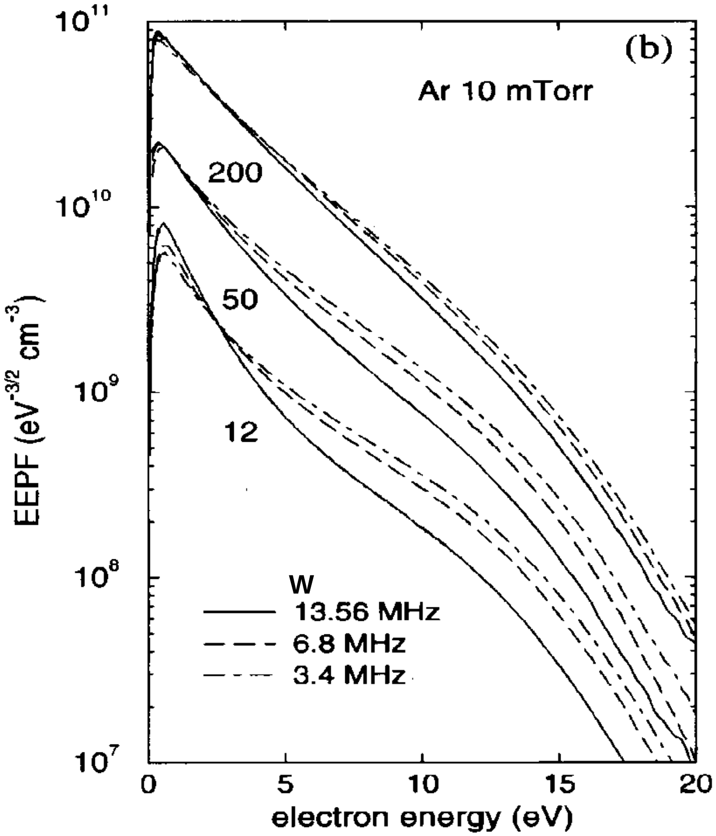
The maximal argon pressure, was limited by the chamber surface when $I_{ich} > I_{esat}$



V. Godyak et al,
PSST **11**, 525, 2002

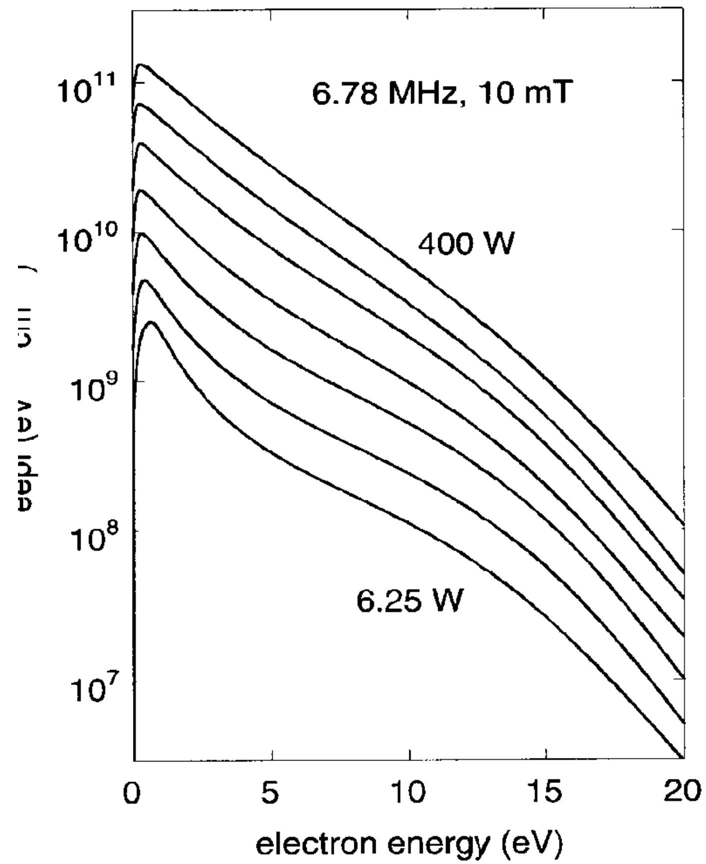
Examples of EEPF measurements in Ar ICP

Frequency dependence



V. Godyak & V. Kolobov, Phys. Rev. Lett., **81**, 369, 1998

Power dependence

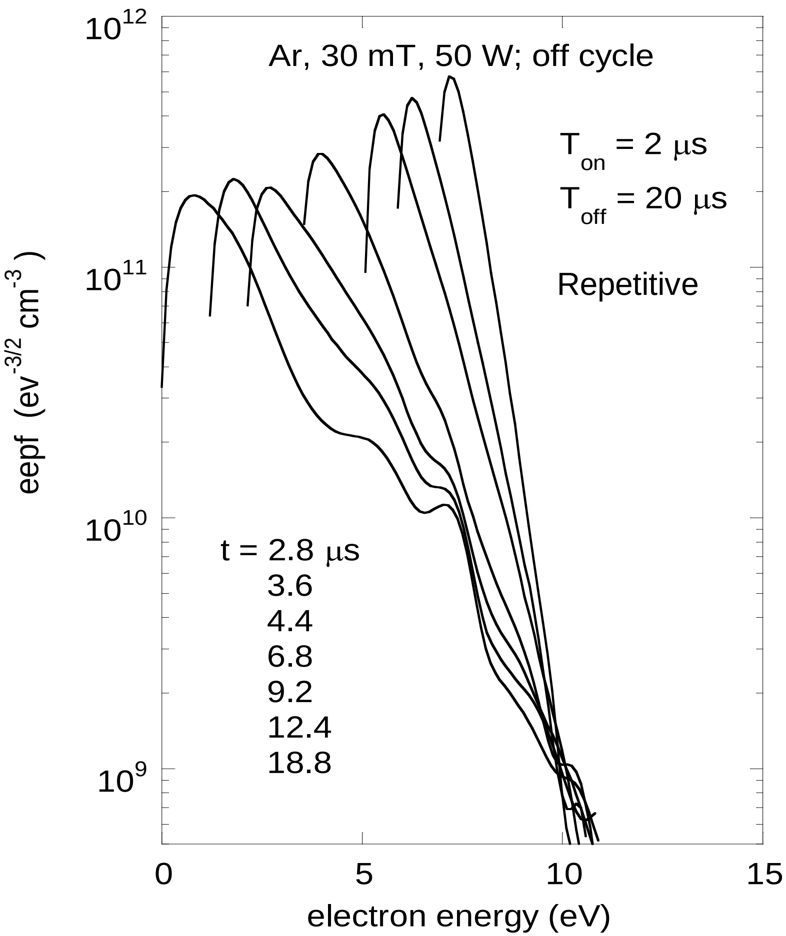


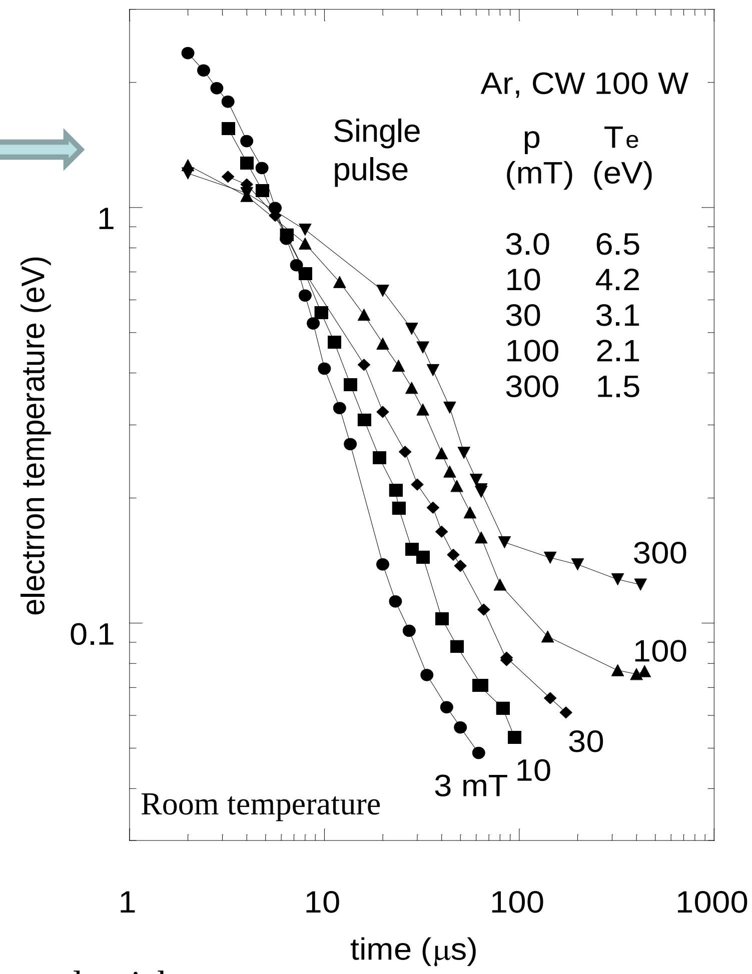
V. Godyak et al, PSST **11**, 525, 1002

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

Time resolved EEPF measurements

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





V. Godyak & B. Alexandrovich,
XXVII ICPIG, vol. 1, p. 221, 2005

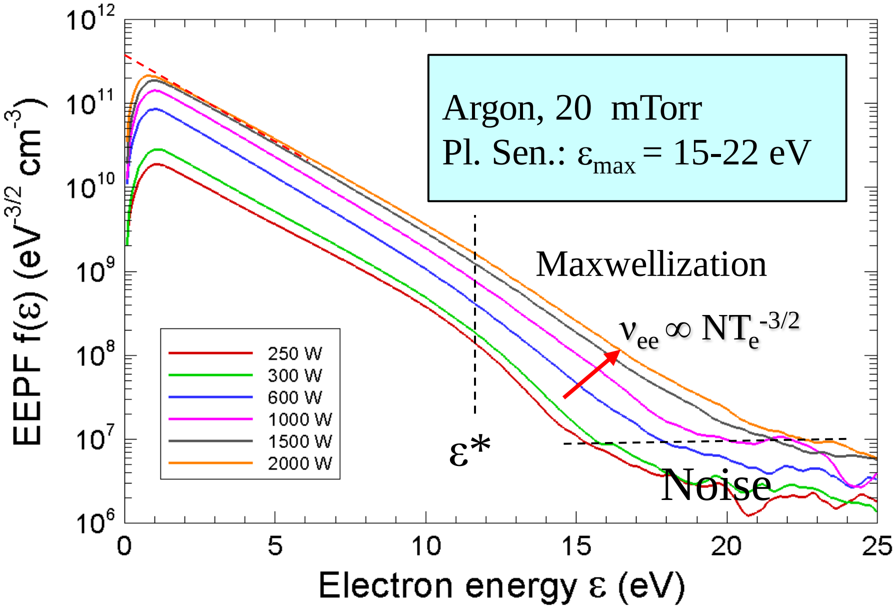
EEDF measurements with commercial probe systems

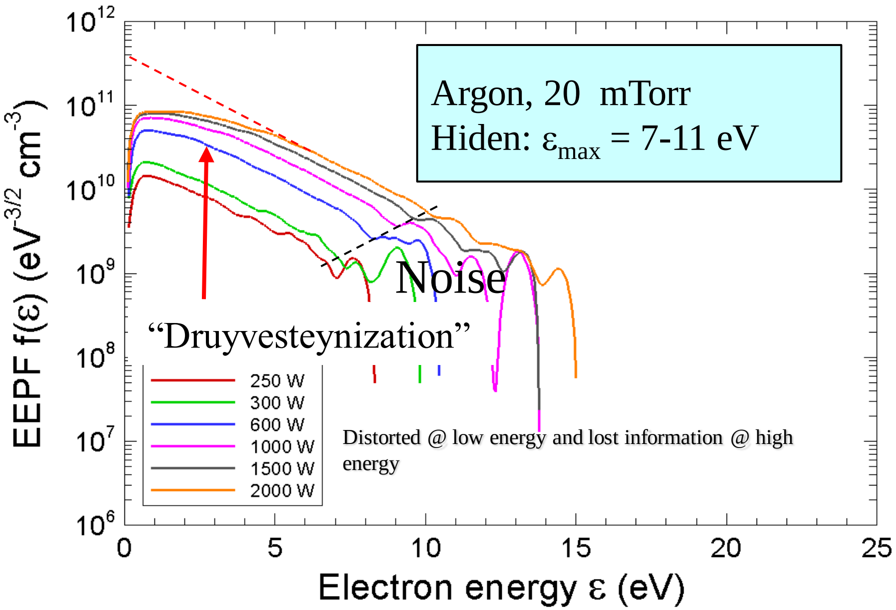
Comparison of EEPF measured with different commercial probe stations, Espion of Hiden and VGPS of Plasma Sensors.

At maximal discharge power of 2 kW, N ≈ 10^{12} cm^{-3}, due to e-e collisions, the EEPF @ ε < ε* has to be a Maxwellian one.

"Druyvesteynization" effect is found in many publications of EEDF measurements made with home-made and commercial probe systems.

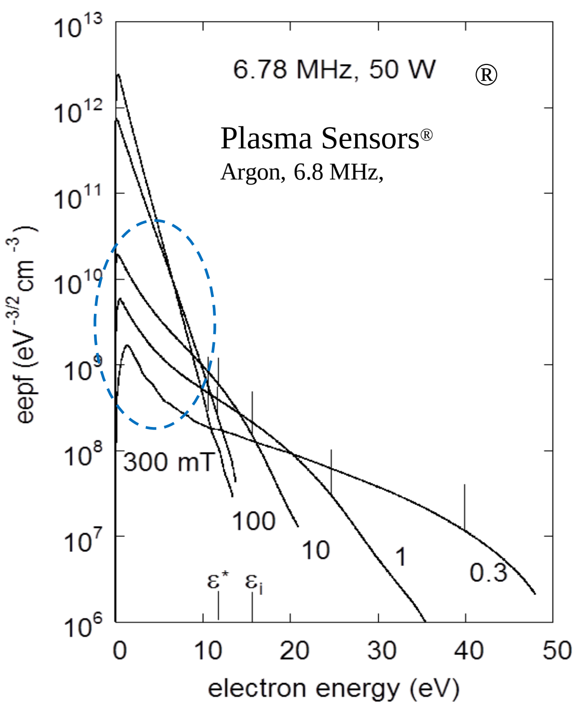
V. Godyak et al, GEC 2009, Saratoga Springs, NY, USA

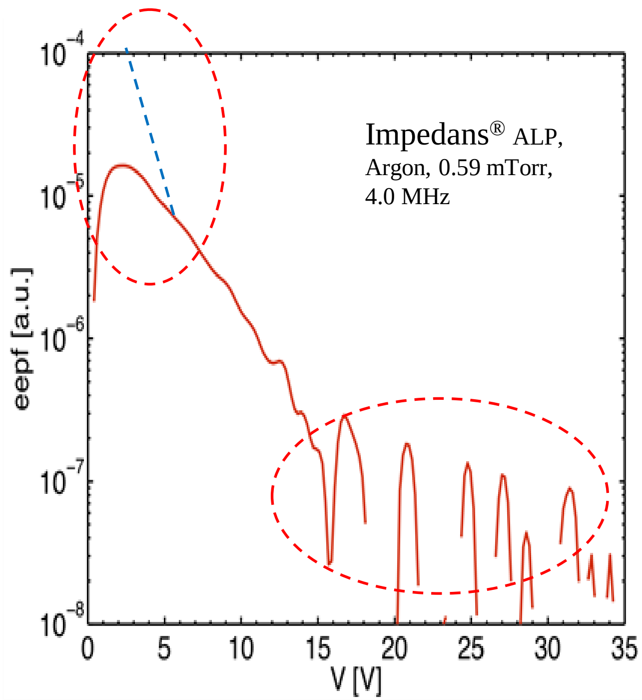


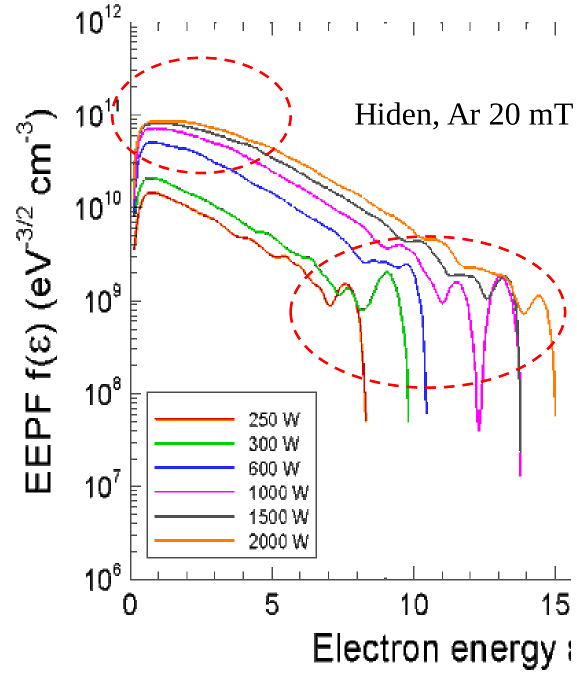


EEDF measurements in a commercial ICP reactors

Many probe system manufacturers claim the superiority of their product, but the real criterion of the instrument merit is the quality of the results obtained with this instrument





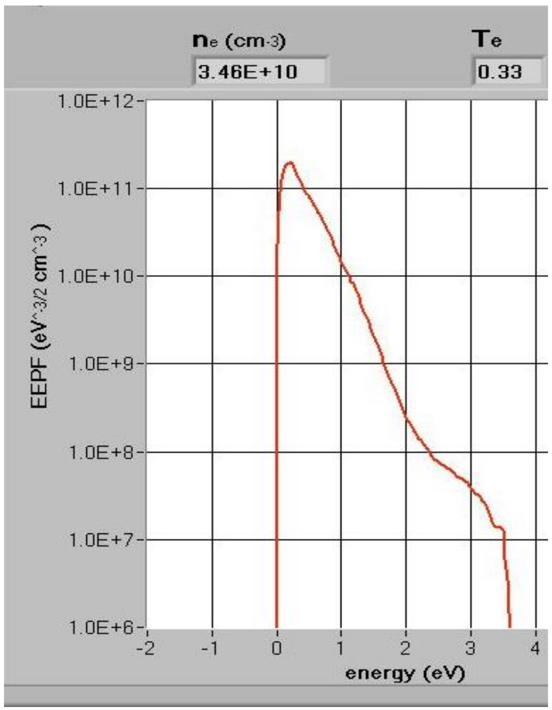


In ICP at mTorr pressure range and low plasma density, due to selective electron heating typical for anomalous skin effect, the EEDF has a low energy peak followed with a strong tail of high energy electrons.

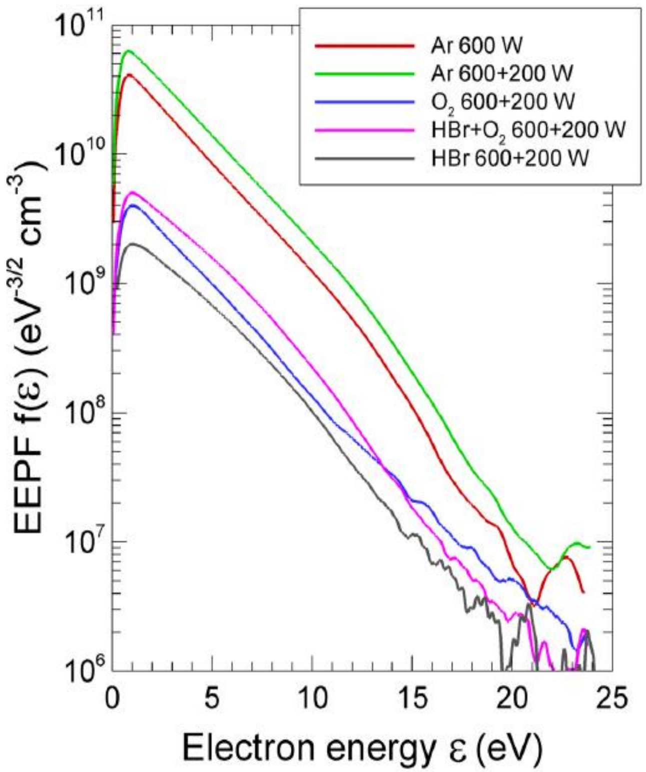
To our knowledge, no one commercial probe system (except by Plasma Sensors) was able to demonstrate such feature of EEDF in ICP at low gas pressure

EEPF measurements in plasma reactors

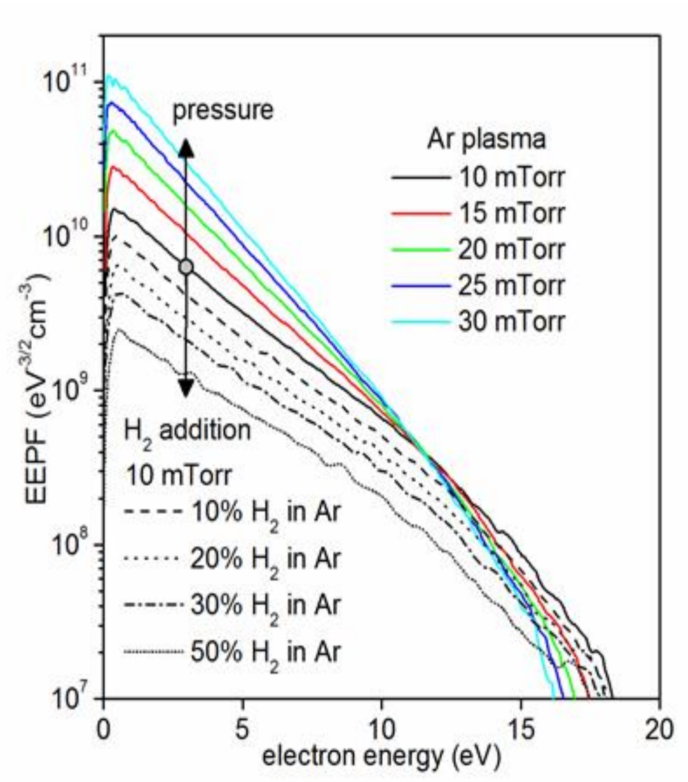
Wide specter and large amplitude of rf plasma potential and high rate of probe contamination are the major problems making even classic Langmuir probe diagnostic impossible



EEPF measured in ECR array reactor, Ar/SiF_4 at10 mTorr with crystalline silicon deposition.
Ecole Polytechnique, France



EEPF measured in commercial two-inductor ICP etcher with different processing mixtures at 15 mTorr.
Mattson Technology, USA



EEPF measured in ICP reactor, Ar and Ar/H_2 at different argon pressure and hydrogen addition.
University of Maryland, USA

Concluding Remarks

- Today, plasma simulation codes are practically main tool for study plasma in industrial plasma sources. These codes applied to plasma with complicated processing gas mixture are missing many cross sections for variety of plasma-chemical reactions.
- They also are missing effects of nonlocal and nonlinear plasma electrodynamics that has been proved are important and even dominant in rf plasmas at low gas pressure.
- In such situation, a reliable measurement of EEDF and plasma parameters would give a valuable experimental data for understanding variety of electrodynamics, transport and kinetic process in such plasmas and for validation of existing theoretical models and numerical codes.

For more complete information see:

1. V. Godyak, *Measuring EEDF in Gas Discharge Plasmas*, review in NATO ASI Series, E. Appl. Sci., V. 176, *Plasma Surface Interaction and Processing of Materials*, pp. 95-134, Kluwer, Acad. Publisher, 1990

2. V. Godyak and V. Demidov, *Probe Measuring of Electron Energy Distribution in Plasmas: What Can We Measure and How Can We Achieve Reliable Results?*, J. Phys. D: Appl. Phys. **44**, 233001, 2011

3. *Plasma Sensors Probe System*, www.plasmasensors.com, GEC2013 exhibition

PLASMA SENSORS