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JENION

Application Note: „Plasma Analysis with Plasma Probe Arrays and Retarding Field Analyzers"

(Version 2021)

Content:

1. Problems of Plasma Analysis in Thin Film Technology

Modern plasma and ion beam based thin film technologies like plasma etching, plasma-CVD, plasma polymerization, ion implantation, sputtering and more use plasmas to deposit, etch and modify thin films in a large variety of manners.
The produced plasma and its characteristics determines the type of thin film processes in each of these technologies. Therefore a comprehensive information about the role of plasmas in this thin film processes is needed.

There are two different regions at a low temperature plasma in thin film technology:

a) Plasma volume: with production of charge carriers by electron impact from gas phase;
b) Plasma sheaths at vacuum chamber walls and at the substrate with the plasma sheath processes, which are running at these substrate.

Table 1.1 shows an overview about the possibilities of plasma probe analysis with plasma probes, where n_e is the electron density, j_i the ion current density, $f(W_e)$ (EED) the electron energy distribution and $f(W_i)$ (IED) the ion energy distribution.

	Plasma volume	Plasma sheath / plasma interactions
Electron s	n_e and $f(W_e)$ EEV determine plasma chemistry	Production of plasma sheaths, RF-Plasma
Ions	-	Ion energy deposition into substrate j_i, $f(W_i)$ IEV
Radicals	Plasma chemistry	Surface chemistry (thin film deposition and – etching)

Tab.1.1: Types of plasma probe analysis with plasma probes

Whereas charged parts of plasmas can be analyzed easily with electrical probes, this is not possible for analysis of radicals of excited molecules and atoms.

In this case optical methods or methods of analysis from chemistry like mass spectroscopy have to be used.

Fig.1.1 shows the analysis of a plasma with two probes to determine simultaneously the spatial distribution (top) and the energy distribution of the plasma (buttom).

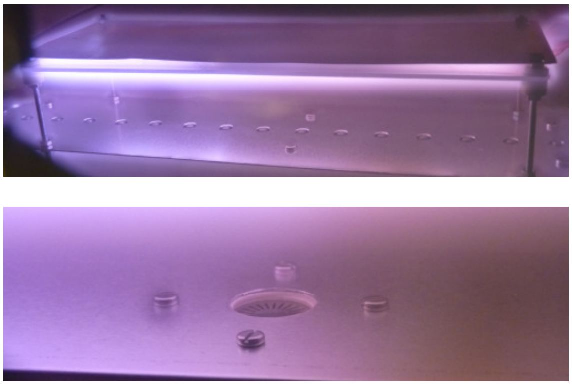

Fig. 1.1: Complex plasma analysis at a low frequency plasma with 16 plasma sheath probes (top) and Retarding Field Analyzer (bottom) integrated into the plasma electrodes.

Probe analysis at plasmas is used to determine the plasma characteristics like charge carrier density and charge carrier energies. The plasma probe has to be adapted to the type of the plasma.

An important criteria for the selection of the applied plasma probe is the Debye-Length of the measured plasma.

$$\lambda_D = \sqrt{\frac{\varepsilon_o W_e}{2e^2 n_e}} \qquad (1)$$

where is: W_e – electron energy,
n_e - electron density.

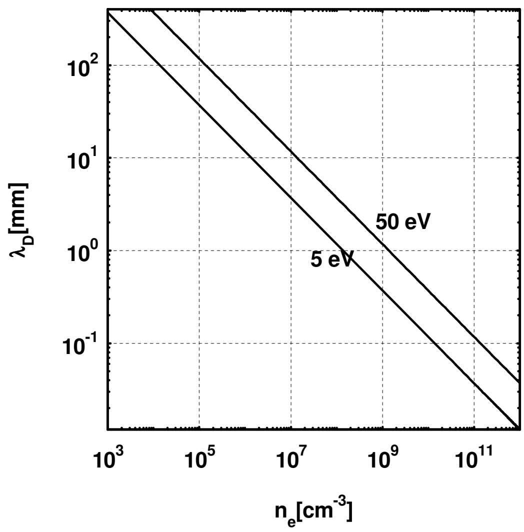

Fig.1.2: Debye-length in dependence on the electron density of plasmas with typical electron energies of 5 and 50 eV.

Plasma probes, which are used to determine the characteristics of plasma volume without producing a surrounding plasma sheath, have to be small in comparison to the Debye-Length of the plasma (see Fig.1.2).

A typical plasma probe of this type is the historically oldest plasma probe – the Langmuir probe.

Probes to determine plasma sheath characteristics must realize the production of plasma sheaths, that means they have to be large in comparison to the Debye-Length. For Langmuir-probes there are typical dimensions of 0.1 mm wire diameter tungsten, whereas plasma sheath probes require at least 3-5 mm diameter (see Fig.1.3.).

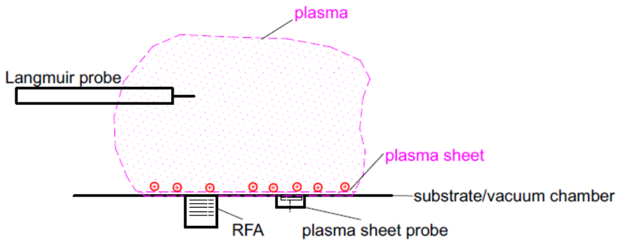

Fig.1.3: Principle of Langmuir probes for plasma volume characterization and plasma sheet probes and retarding field analyzers for plasma sheet analysis

These probes have different application fields.

a) Plasma characterisation in plasma volume by Langmuir probes for:
- Plasma chemical reactions in gas phase (as prerequisite for defined thin film processes),
- Plasma light emission,
- Afterglow- und Downstream plasmas

b) Plasma sheath characterisation by plasma sheath probes for:
- Characterisation of interactions of electrons and ions in thin film processes running on substrates;
- Determination of ion energy as a central parameter (whereas electron interactions are less important for thin film processes).

Table 1.2 shows an overview of ion energies and thin film processes.

Ion energies	**Thin film process**
0 – 10 eV	PECVD, remote plasma, downstream plasma
10 – 50 eV	Plasma etching, DLC-growth,
50 – 150 eV	IAD, Ion Assisted Deposition, Plasma Etching,
150 – 500 eV	RIE, Reactive Ion Etching, IAD, IBAD
500 – 2000 eV	Ion beam sputtering
> 2keV	Ion implantation

Tab.1.2: Overview of some important thin film processes in dependence on ion energies

Therefore a cost effective device for plasma analysis should use the following characteristics:

a) The devise should use different plasma probes:

- Langmuir probes (dimensions small in comparison to Debye-Length, no plasma perturbation, producing no plasma sheath),
- Plasma sheath probes (dimensions large in comparison to Debye-Length, producing a plasma sheath),
- Retarding Field Analyzer (total dimension large in comparison to Debye-Length, measuring at the plasma sheath).

b) Probe arrays (typ. 16 probes) for simultaneous measuring of local plasma distribution:

- Fit the probe arrays from 16 x 5 mm = 80 mm to 16 x 200 mm = 3200 mm to plasma dimension,
- Simultaneous measuring of plasma density distribution to estimate the homogeneity of the plasma.

c) Measuring of ion and electron energy distribution at characteristic places (border, middle):

- Retarding field analyzer (i.e. simultaneous with plasma sheath probe)

2. Langmuir Probe Arrays

Fig. 2.1 shows the principle of a simple Langmuir probe. The probe, a 5-10 mm long tungsten wire with 0.1 mm diameter, is placed in the plasma by a sufficiently long isolating tube. The probe current respective ground is recorded in form of an U-I-diagram.

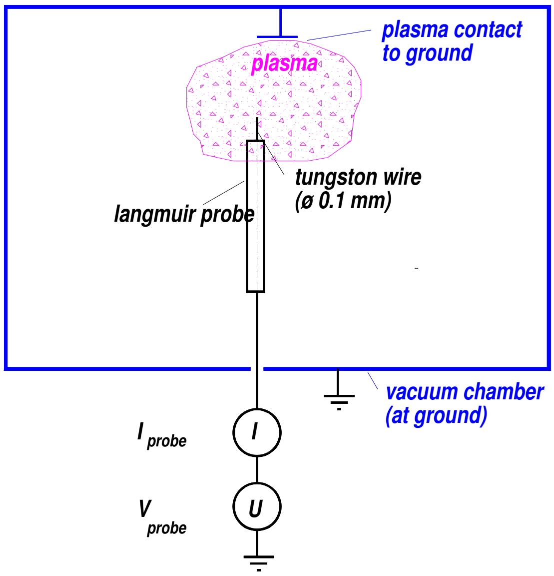

Fig.2.1: Principle of Langmuir probe

Fig. 2.2 shows in principle the arrangement of 16 Langmuir plasma probes in a line to measure plasma properties across a plasma.

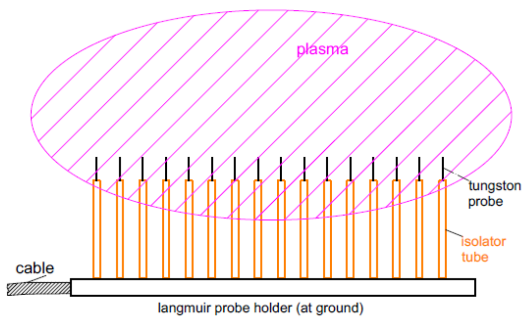

Fig.2.2: Principle of plasma analysis by 16 Langmuir probes

Fig. 2.3 shows a typical probe characteristic.

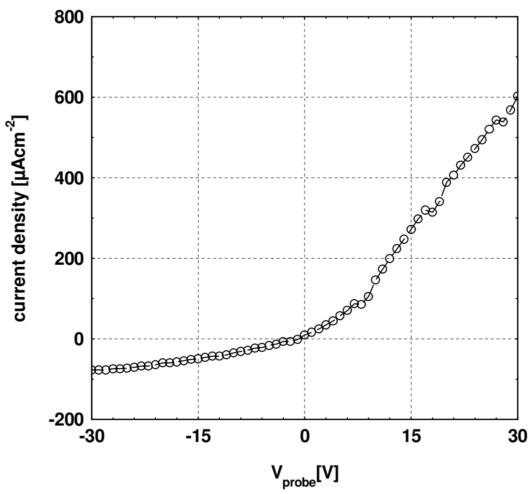

Fig.2.3.: Typical probe characteristic of a micro wave plasma (Ar, 0.1 mbar)

There are established analysing methods to analyse such probe characteristics (i.e. in [1]). All these methods are made for plasmas with isotropic charge current distributions, that means for plasmas like in the positive column of DC- or pulsed RF-plasmas.

The following qualities can be determined from a probe characteristic like in Fig.2.3. :

- Ion saturation current density,
- Floating potential,
- Plasma potential.

If the plasma potential is exactly determinable (i.e. with an exactness of < ± 1 eV) such methods will be useable to determine the

- Electron energy distribution f(W_e),
- Electron density.

Jenion offers no linear arrays of Langmuir probes, because their dimensions are mostly customer specified, but the device "PlasmMon 3" works very well also with Langmuir plasma probes. The plasma sheet probes can be direct replaced by Langmuir probes.

3. Plasma Sheath Probe Arrays

Fig. 3.1 shows the principle of plasma analysis with plasma sheath probes. A cylinder shaped probe (typical diameter of 5 mm) is embedded in a ground area, which has to be about 2 scales larger in comparison to Langmuir-probes. The probe current is recorded respective ground.

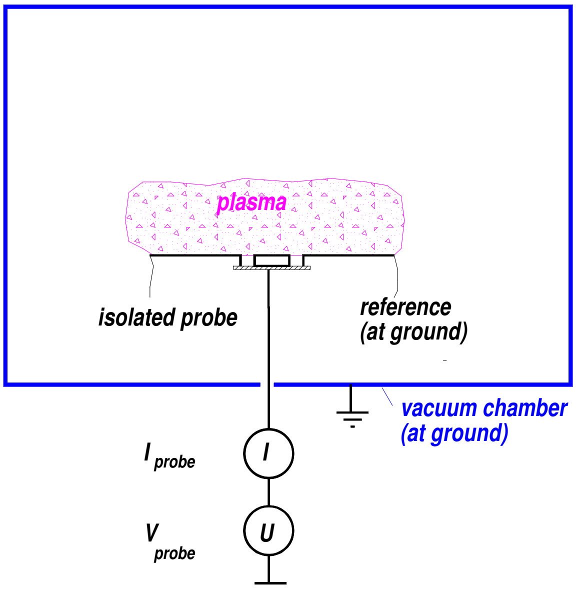

Fig.3.1.: Principle of probe measuring with plasma sheath probes

Fig. 3.2 shows a typical plasma sheath probe array of 16 probes with a distance of 20 mm.

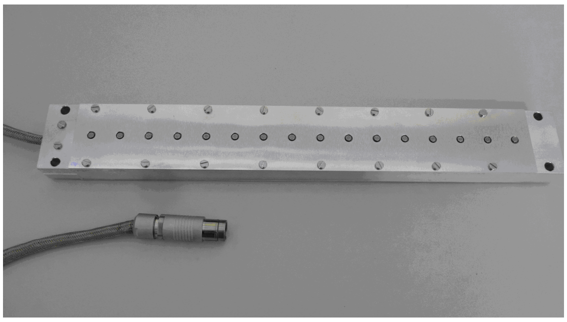

Fig. 3.2.: Typical plasma sheath probe array (16 x 20 = 320 mm)

Fig. 3.3 shows a typical probe characteristic of a plasma sheath probe, measured at a RF-plasma with variable RF-power.

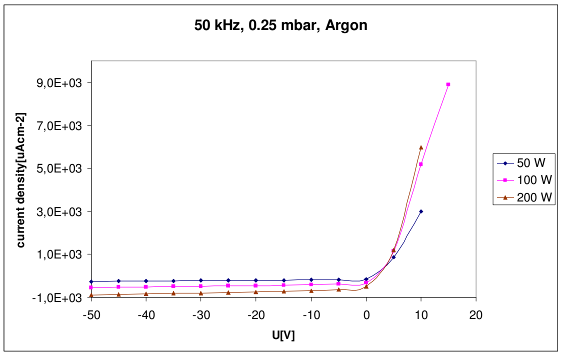

Fig.3.3: U-I curve of a plasma sheath probe at a low frequency plasma (50 kHz) with variable plasma power

The ion saturation current density and the floating potential can be determined from the probe characteristic like in the analysis of Langmuir probes.

The necessary probe voltage for measuring the ion saturation current density is well determinable (about – 30 V in Fig.3.3). Ion density profiles like in Fig.3.4. can be simultaneously recorded at this probe voltage with plasma sheath probes.

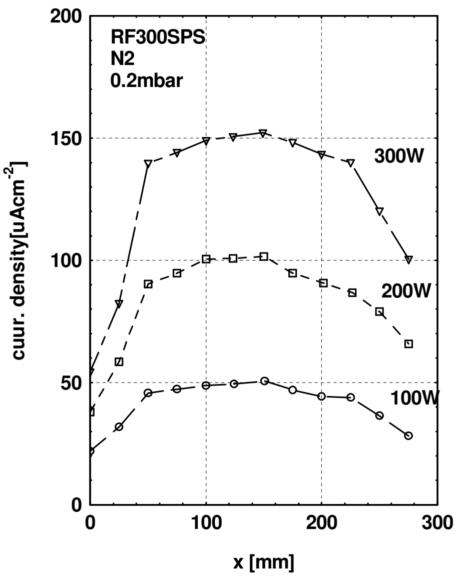

Fig. 3.4: Ion current density profile of a RF-plasma at different RF-powers

The ion density can be estimated from the ion saturation current density, if the mean ion energy is given. The mean ion energy can be measured e.g. by a Retarding Field Analyzer (see chapter 4).

$$n_+ = \frac{4 * j_+}{e * \sqrt{\frac{2 * W_+}{M_+ * m_p}}} \qquad (2)$$

where is:

j_+ – Ion saturation current density,
W_+ – Ion energy,
M_+ – relative ion mass (in amu),
m_p – Proton mass,
e – Electron charge.

When the plasma is quasi neutral (i.e. $n_+ = n_e = n$), it is possible to determine the ion density and the plasma charge carrier density by using equ. (2).
Fig.3.5 shows the corresponding plasma density in dependence on ion saturation current density according to (2).

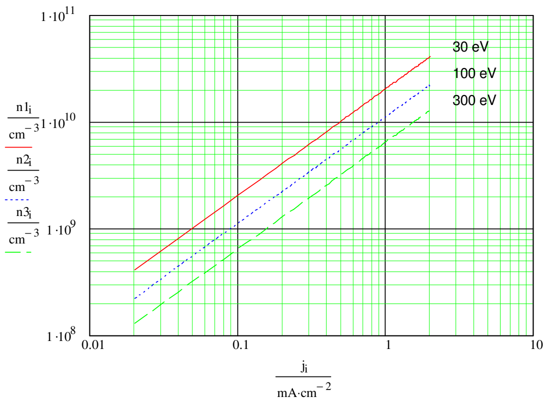

Fig.3.5: Plasma density in plasma sheath in dependence on ion saturation current density for different ion energies (for Argon, M= 40).

4. Retarding Field Analyzer

Fig.4.1. shows the principle of a Retarding Field Analyser [2,3].

A hole grid system of four grids extracts charge carriers from the plasma. The extraction grid can have a positive potential (measuring electron energy distribution or negative ions) or a negative one (measuring positive ion energy distribution).

The third grid is the retarding grid, which only ions can overcome, having an energy higher than the retarding voltage. Finally a collector plate together with a secondary electron suppression grid measures this current in dependence from the retarding voltage. The energy distribution of the analyzed particles (electrons, positive or negative ions) is then the derivative of the $I(U_{ret})$-curve.

With a Retarding Field Analyzer with an array of holes it is possible to extract particles from an area of about some cm^2, giving a much larger current then one hole. The Retarding Field Analyzer is made of a laser cutted hole grid system with holes of 0.5 mm diameter and 0.25 mm grid distance. Therefore two limits arise:

- Gas pressure has to be smaller than 10^{-1} mbar (mean free path has to be larger than free flight length in the analyzer of about 1mm),
- Plasma charge density has to be in maximum up to 10^{11} cm^{-3} (Debye-Length has to be bigger than hole diameter of 0.5 mm).

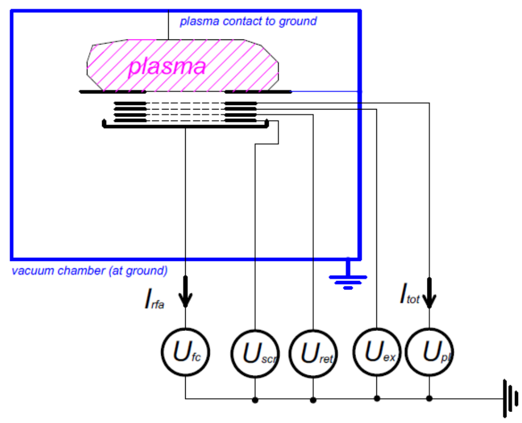

Fig.4.1.: Principle of retarding field analysis

Fig.4.2 shows a Retarding Field Analyzer with laser cutted holes of 0.5 mm diameter.

Fig.4.2: Retarding Field Analyzer

Fig. 4.3. shows the ion energy distribution of a RF-plasma (13.56 MHz) measured at the ground electrode.

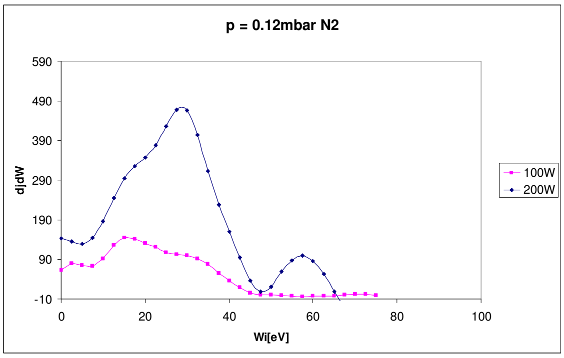

Fig.4.3.: Example of an ion energy distribution from a RF-plasma, measured at the ground electrode

5. Application

5.1. PlasmaMon – a Compact and Allround Device

Fig. 5.1 shows the device „PlasmaMon-3" which was developed by JENION in 2020. There is an electronic unit, operating with Langmuir- or plasma sheath probes and a Retarding Field Analyzer in a compact rack. The device is PC-controlled via serial port.

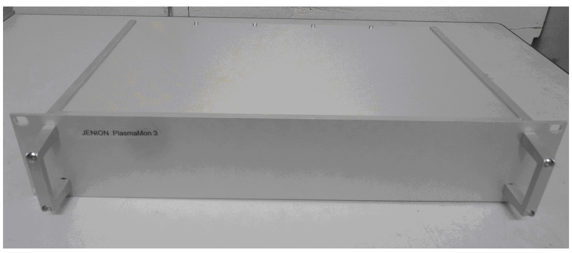

Fig.5.1: Electronic Control Unit (ECU) of „PlasmMon-3"

Table 5.1 shows the technical data.

Parameter	Typical values	Maximum value
Measuring time per measuring point	1-30 s	>= 1s, max. 1000 s
V_{probe}	-50 ... + 50 V	-100 V, resp. +100 V max. 50 mA
$V_{ret\ field}$	-400 V resp. + 400 V	-800 V, resp. +800 V max. 5 mA
Measring range probe current:	0.01 – 300µA	+- 2.5mA

Tab.5.1: Technical Data of PlasmaMon 3

Fig.5.2.shows the principle arrangement of the complete device at a vacuum chamber for plasma analysis.

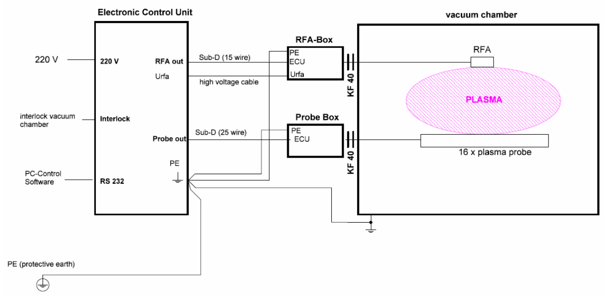

Fig.5.2: Arrangement of PlasmaMon 3 with plasma probes and Retarding Field Analyzer at a vacuum chamber for plasma analysis.

There are three types of operations (see Fig. 5.3 to 5.5):

a) **„Plasmaprofile":** Measuring of ion current densitiy profiles,

b) **„Plasmaprobe":** Measuring of probe characteristics,

c) **"Ret. Field Analyzer":** Retarding Field Analysis.

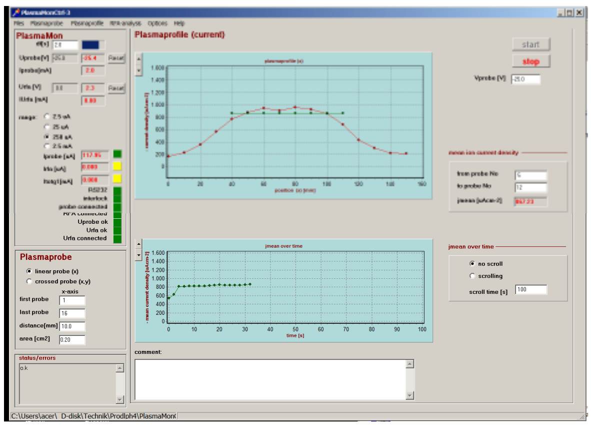

Fig.5.3: Screenshot in operation type „Plasmaprofile"

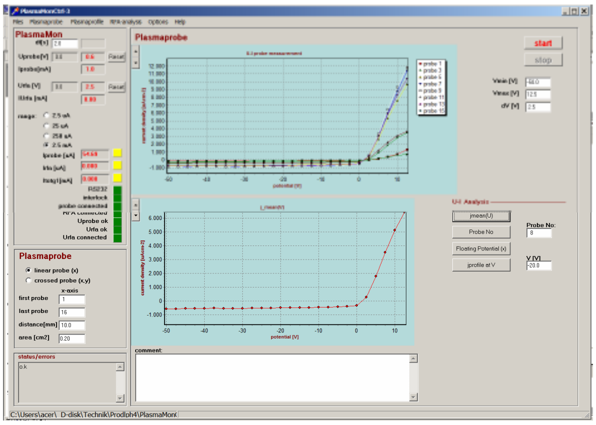

Fig.5.4: Screenshot in operation type „Plasmaprobe“

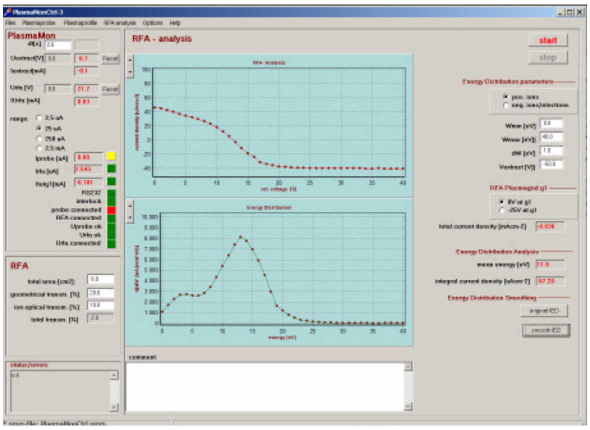

Fig.5.5: Screenshot in operation type „RFA – Ret. Field Analyzer“

All measured diagrams can be stored as ASCII-file and used for further processing.

5.2. Plasma analysis at a simple electrode plasma (50 kHz)

A simple electrode plasma, generated by a 50 kHz plasma generator like shown at Fig.5.2.1. was analyzed.

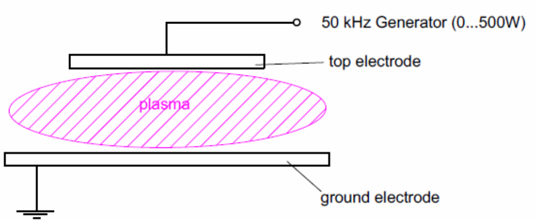

Fig.5.2.1.: Principle of a low frequency electrode plasma (50 kHz)

A multiple plasma sheet with 16 probes at a distance of 20 mm was integrated into the ground electrode like shown at Fig.5.2.1. So the plasma homogeneity over a length of more than 300 mm could be observed by measuring the ion saturation current density at a probe voltage of -25 V.

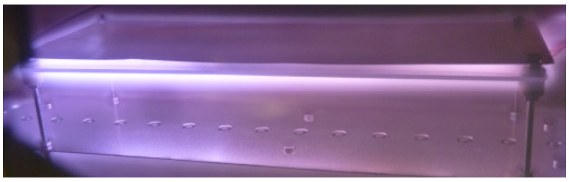

Fig.5.2.2.: Integrated plasma sheet probe (16 x 20 = 320 mm, probe see Fig.5.1.3) at the ground electrode of a 50 kHz electrode plasma

Fig.5.2.3. shows the ion saturation current density profiles for different pressures at an 200W argon discharge.

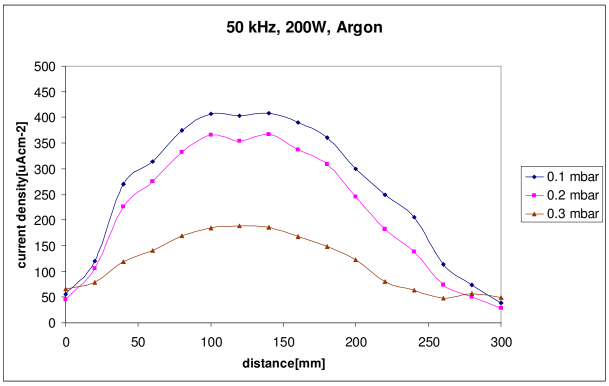

Fig.5.2.3.: Ion saturation current density profiles at different pressures (200 W argon)

The ion current density and therefore the plasma density (see e.g. equ. (2)) grows with decreasing pressure. The plasma is only at the mid of the electrodes homogeneous.

Fig.5.2.4. shows a typical U-I curve of the mid range (probe No. 8) of the plasma. The U-I curve shows at negative voltages a clear ion saturation current behaviour. For positive probe voltages the electron part of the U-I curve appears.

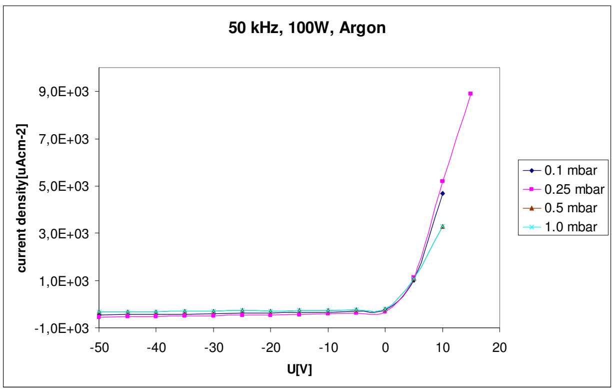

Fig.5.2.4.: U-I curves (Argon, 200 W) of probe No 8 (at the mid of the plasma) in dependence from the pressure

To analyze the ion energy distribution of the plasma at the ground electrode a Retarding Field Analyzer was integrated into the ground electrode instead of the multiple plasma probe of Fig.5.2.2 (see Fig.5.2.5.).

Fig.5.2.5.: Retarding Field Analyzer integrated into the ground electrode (0.1 mbar N_2, 100 W electrode distance 40 mm)

Fig.5.2.6. shows the ion energy distributions measured at a nitrogen plasma of 100 W at different pressures. Like known from literature, the 50 kHz plasma is very similar to a DC-plasma in its behaviour. That means at every second half cycle the electrode with the RFA acts as cathode with the typical cathode fall.

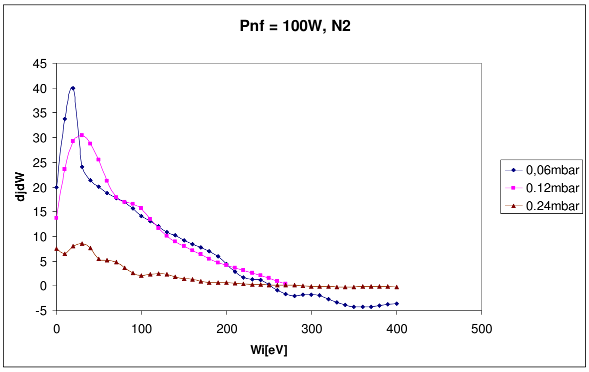

Fig.5.2.6.: Ion energy distribution for different pressures at RF power of 100 W,

Fig.5.2.6. shows the ion energy distribution with the long linear decay up to the cathode fall voltage of approx. 350 V at a small pressure of 0.06 mbar very clear. At high pressure of 0.24 mbar occur to much scattering of ions with neutral gas inside the RFA, so that the measured ion energy distributions is scattered down

5.3. Ion energy analysis at a simple electrode plasma (13.56 MHz)

Now a simple electrode plasma generated by a radio frequency generator with 13.56 MHz was analyzed by a Retarding Field Analyzer integrated into the ground electrode (like shown at Fig.5.2.5.

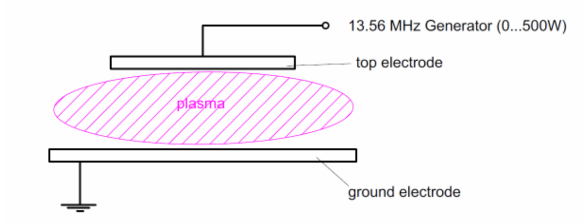

Fig.5.3.1.: Principle of a radio frequency electrode plasma (13.56 MHz)

Fig.5.3.2. shows the ion energy distribution at different pressure for a nitrogen discharge, powered by 100 W. While at a pressure below 0.12 mbar good results are measured, the IED curve for 0.24 mbar is not acceptable because of to much ion-neutral scattering inside the RFA.

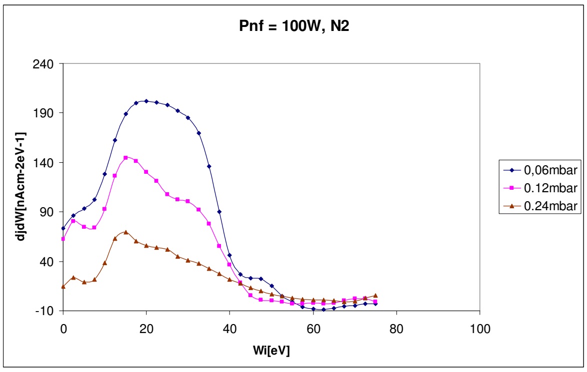

Fig.5.3.2.: Ion energy distribution for different pressures at RF power of 100 W

Fig.5.3.3. finally shows the ion energy distribution at 0.12 mbar for two different plasma powers.

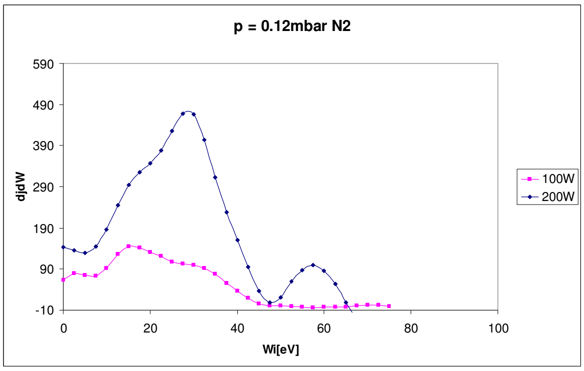

Fig.5.3.3.: Ion energy distribution for different RF power at a pressure of 0.12 mbar

5.4. Broad ion beam analysis of a 40 mm Kaufman ion source

Fig.5.4.1. shows in principle the arrangement of a RFA and a multiple plasma probe with 16 probes at a distance of 10 mm on a Kaufman type broad beam ion source. The extraction grids of the ion source generate a broad ion beam of 40 mm diameter. The source is operated at $4x10^{-4}$ mbar argon with an ion beam current between 5 and 15 mA. By means of a moveable substrate holder the RFA or the multiple plasma probe could be moved into the beam.

A filament neutralizer was used to neutralize the ion beam if necessary.

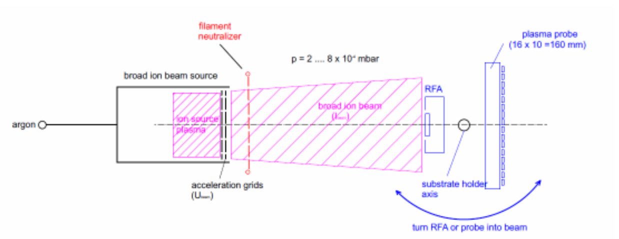

Fig.5.4.1.: Principle arrangement of a linear plasma sheet probe and a RFA at a broad ion beam source

Fig.5.4.2. shows the ion current beam profile across the beam at a distance of 250 mm from the Kaufman ion source in dependence from the generated ion beam current. Current densities up to 50 uA cm^{-2} are measured.

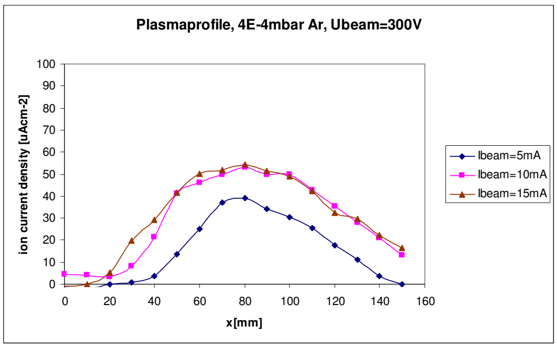

Fig.5.4.2.: Broad ion beam profile at different ion beam currents

Fig.5.4.3. shows the ion beam profile for a low extraction voltage of only 100 V, where the broad ion beam is normally very divergent. Only a small ion beam current density of some $uAcm^{-2}$ is measured. If the ion beam is neutralized, most of the positive space charge inside the beam is compensated and higher ion beam current densities up to 10 uA cm^{-2} are measured.

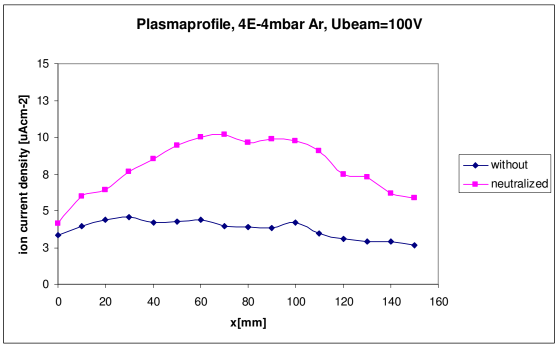

Fig.5.4.3.: Broad ion beam profile at 100 eV beam energy without and with neutralizer

To demonstrate more over the function of the neutralizer Fig.5.4.4. shows the floating potential profile across the ion beam. Without neutralizer floating voltages between +50 and +75 V are measured. If the neutralizer is on, the floating potential is around -4V overall across the ion beam.

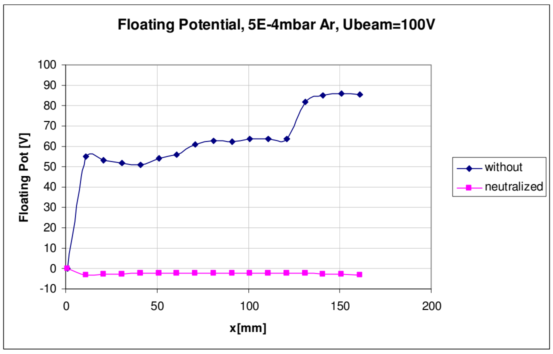

Fig.5.4.4.: Floating potential profile with and without neutralizer:

Finally the RFA was moved into the ion beam and the ion energy distribution was measured for Extraction voltages of 100, 200, 300 and 400 V (see Fig.5.4.5).

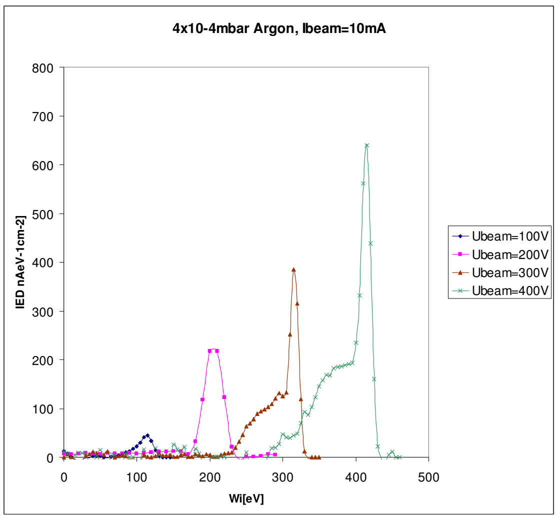

Fig.5.4.5.: Ion Energy distributions measured by Retarding Field Analyzer for different beam extraction voltages

The RFA has a ion energy resolution of +- 1%. So the nearly mono energetic broad ion beam of the Kaufman ion source could be demonstrated at Fig.5.4.5. . Moreover some scattering phenomena of the comparatively long broad ion beam are shown.

5.5. Positive and negative ion energy analysis at ITO sputtering with argon

With PlasmaMon also sputter plasmas can be analyzed, if they deposit conductive layers. But the measuring time at running deposition is then limited. More than approx. 10 um deposited layers at the plasma sheet probes or the RFA create parasitic layers, which can split away, causing short cuts into the sensors. Plasma sheet probes can very easy cleaned from parasitic layers at customer side. The RFA also can be cleaned, but that need some experience or a refurbished grid system.

Fig.5.5.1. shows in principle the application of a RFA at sputtering of ITO from a rotary target. The RFA is embedded into the substrate holder at ground (see Fig.5.5.2) and mostly the first grid of the RFA will be also deposited by ITO.

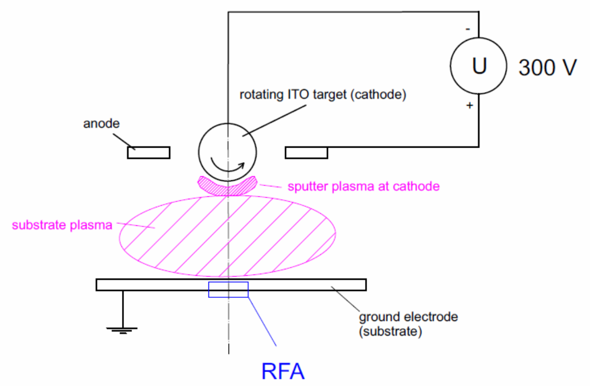

Fig.5.5.1.: RFA analysis at ITO sputtering

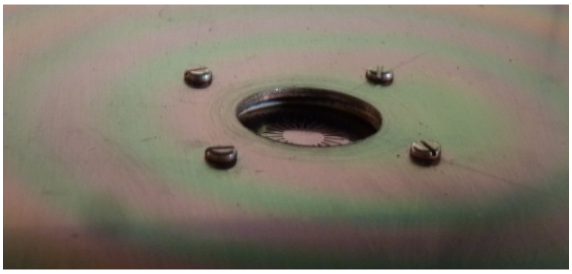

Fig.5.5.2.: Retarding Field Analyzer embedded into a substrate holder at sputtering of ITO with argon

With the arrangement shown at Fig.5.5.1. the RFA measures the ion energy distribution at the substrate, which interacts with the growing ITO layer. While sputtering with argon that are mostly positive argon ions, but from literature it is well known, that also oxide ions like InO_x^- or O^- generated at the target surface can reach the substrate. Because of the potential difference between cathode and substrate at ground they can be accelerated up to energies of 300 eV (nearly the sputter generator voltage).

Fig.5.5.3. shows the ion energy distribution (IED) of the positive ions measured with the RFA. The mean ion energy is around 15 eV, which is typical for plasma sheets against ground in DC-sputtering.

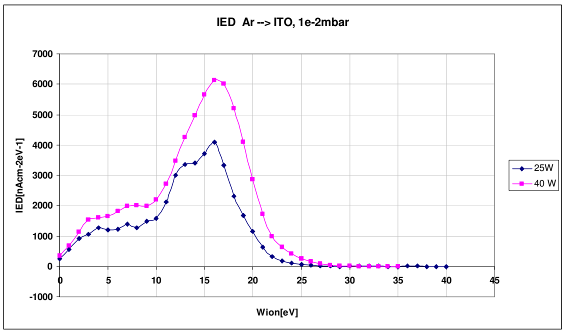

Fig.5.5.3.: Positive ion energy distribution at substrate for sputtering ITO with argon

PlasmaMon 3 also enables the energy analysis of electrons and negative ions with the RFA. That was done in two steps: first the electron energy distribution (EED) was measured in the energy range between 0 and 20 eV with a fine energy resolution of 1 eV (see Fig.5.5.4.). Fig.5.5.4. shows the typical EED with a mean electron energy at approx. 3 eV.

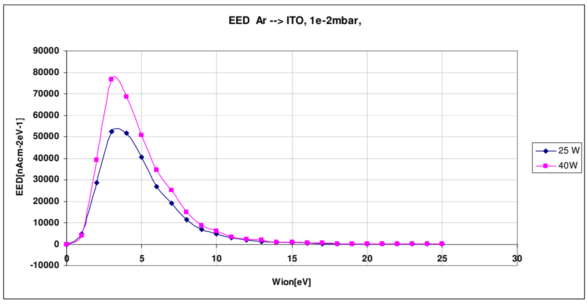

Fig.5.5.4.: Electron energy distribution at substrate for sputtering ITO with argon

The peaks of the EED should be some orders of magnitude bigger than the energy distribution of the negative ions (nIED). Therefore the nIED was analyzed with a separate scan at the energy range of 50 to 400 eV to avoid overlapping of the big EED with the smaller nIED. Fig.5.5.5. shows the energy distribution of the negative ions, with a mean peek at around 300 eV.

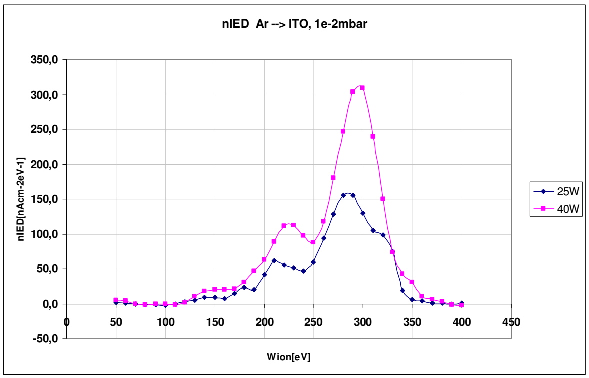

Fig.5.5.5.: Negative ion energy distribution at substrate for sputtering ITO with argon

Fig.5.5.6. shows together the positive and negative ion energy distribution on logarithmic scale. The contribution of the negative ions to the total ion current density is between 5 and 10%.

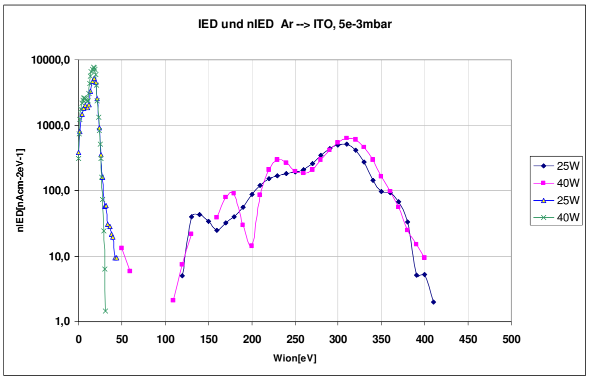

Fig.5.5.6.: Positive and negative ion energy distribution at substrate for sputtering ITO with argon

5.6. Plasma homogeneity analysis at a large scale linear plasma source

The multiple plasma sheet probes with 16 probes can be manufactured with lengths from 100 mm to more than 1000 mm. So they are very good applicable to analyze the homogeneity of large industrial plasmas, where not only the plasma parameters are of interest, but mostly the homogeneity of the plasma process.

Fig.5.6.1. shows the principle arrangement of a linear plasma source (here a 50 kHz driven electrode source) over a moved carrier. If the carrier is moved at constant velocity in y-direction and the linear plasma source generates a homogeneous process in x-direction, than the process (here an plasma etching process with CF_4) is homogenous.

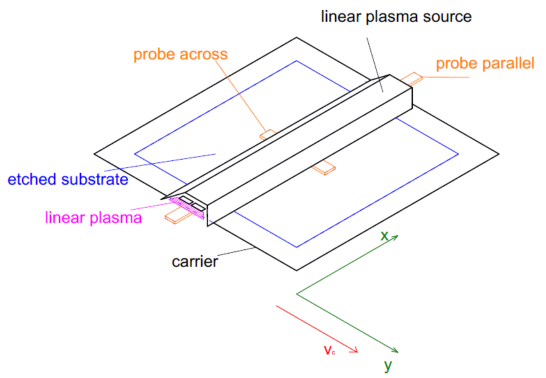

Fig.5.6.1.: Principle of a large scale linear plasma source for etching of large moved substrates together with the used plasma probe positions

To analyze the plasma homogeneity a large multiple plasma sheet probe (16 probes at 65 mm distance) was mountet parallel to the plasma source at carrier position and a short multiple probe (16 x 20 mm) was mounted across the linear plasma source. By this way the plasma homogeneity, measured by ion current saturation profiles could be analyzed for all etching areas.

Fig.5.6.2. shows the ion current density profiles in y-direction (across the source) and Fig.5.6.3.shows the ion current density profiles in x-direction.

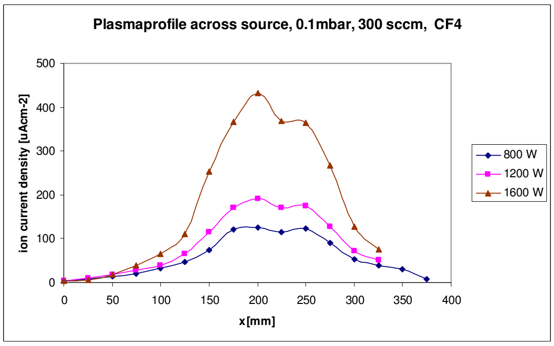

Fig.5.6.2.: Ion current density profile across the source (in carrier movement direction)

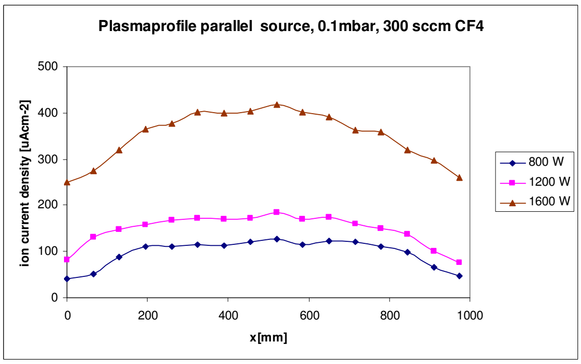

Fig.5.6.3.: Ion current density profile parallel to the source (across carrier movement direction)

The plasma profile in y-direction (carrier movement) can have nearly any shape. Best is of course a Gaussian type shape. So the source is like shown at Fig.5.6.2. ok in y-direction. At best case in x-direction the ion current density profile should be constant over the whole length of the linear plasma source. Then every carrier position should have the same plasma process. That is like Fig.5.6.3 shows not complete the case. The ion current density at the outer sides of the carrier drops down up to 30%.

PlasmaMon 3 measures such plasma profiles direct every 2 seconds, so that an optimization of parameters like pressure, power, gas flow, temperature, can be easy done at short time to achieve good process homogeneity.

Finally a RFA was placed at mid position under the linear plasma source instead of the linear plasma probes and the ion energy distribution was measured (Fig.5.6.4.).

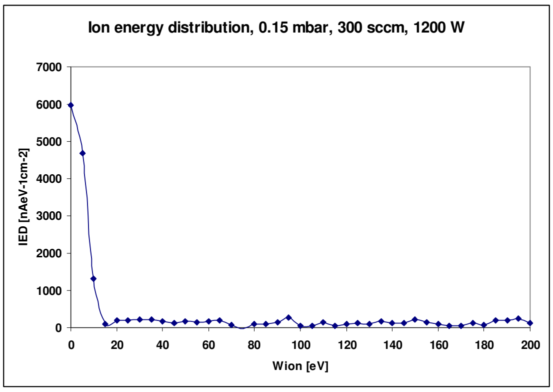

Fig.5.6.4.: Ion energy distribution

The linear plasma source, designed for low energy plasma etching, has a maximum ion energy of 20 eV.

6. Some more customer spezified optional features of PlasmaMon

A variety of customer specified solutions can be developed on the basis of PlasmaMon:

- Development and production of customer specified probes, integrated into substrate holders or other components
- Probes which are embedded in insulators (simulation of plasma sheath processes on insulating substrates),
- PlasmaMon Devices with more than 16 measuring inputs (i.e.. 32 or 64 probes),
- Plasma sheath probes for plasmas at higher pressures up to atmospheric pressure.

7. Conclusions

- Like shown, a variety of investigations about electrical plasma properties at low temperature plasma can be done with the cost effective plasma analysis tool PlasmaMon. This contains the classical part of plasma analysis by Langmuir probes and the modern and innovative part of plasma sheath or Retarding Field Analysis.
- Local properties of laboratory and industrial plasmas of any dimension can be determined by simultaneous measuring with 16 probes of a probe array by using appropriate probe dimensions from centimeters up to meters.
- The application range of PlasmaMon contains all conventional low temperature plasmas with excitation by DC-to middle frequency and radio frequency up to micro wave generated plasmas.
- Whereas the application range of Langmuir probes is limited for pressures smaller than 10 mbar, the plasma sheath probes can be used for higher pressures up to atmospheric plasmas. Retarding field analyzers are limited for pressures below 0.2 mbar. Fig.6.1. shows the pressure ranges of the probes.

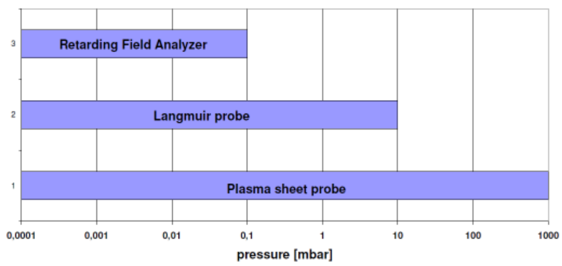

Fig.6.1.: Application pressure ranges of different probes

8. References

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