Octiv VI Probe

RF Measurement and Plasma Control Sensors

https://impedans.com/octiv-mono-rf-wattmeter

https://impedans.com/octiv-poly-vi-probe

https://impedans.com/octiv-suite-vi-probe
Ion flux as an alternative deposition rate parameter

Defining Plasma Polymerization: New Insight Into What We Should Be Measuring
DOI: https://doi.org/10.1021/am401484b

Plasma polymerization - Depositing ultrathin functionalized films onto surfaces

Typically external parameters (RF power and precursor flow rate) are quoted by researchers/industrialists to define plasma polymerization experiments so that other researchers can replicate.

LIMITATION OF RELYING ON EXTERNAL PARAMETERS
For plasma reactors with different geometries, external parameters are near useless for scale-up and process control.

So, the important question is which parameters can provide a better prediction of plasma polymerization processes.

Best solution: Monitor the intrinsic parameters, particularly the ion flux.

The results indicate that ion flux is a better predictor of deposition rate than the often quoted RF power and are highlight in next slides.

Varying ‘d’ simulate low pressure RF plasma reactors of different geometry.

- RF power delivered to plasma
- Ion Flux

Experimental setup of Capacitively Coupled Plasma (CCP) system
Research Highlights

Experimental conditions: Capacitively Coupled (13.56 MHz) Plasma, 1Pa pressure, RF power 5W

Diagnostics used in study
- Impedans – Octiv sensor
- Sycon Instruments - Quartz Crystal Microbalance
- SPECS SAGE spectrometer

Measured parameters
- The ion flux to the RF electrode (Post-match)
- Deposition rate
- X-ray Photoelectron Spectroscopy spectra

Constant Ion Flux 7.5 x 10^{17} ions m^{-2} s^{-1} is maintained by adjusting the RF power.

1. The deposition rate is intimately linked with the ion flux, not RF power

✓ The deposition rate remains constant when the ion flux is kept constant, but increases dramatically for smaller electrode separations at constant RF power.

![Deposition rate versus electrode separation constant RF power of 5W (closed symbols) and constant ion flux of 7.5 x 10^{17} ions m^{-2} s^{-1} (open symbols).]

2. Functional group retention link with the ion flux

✓ Functional group retention is an important parameter in many plasma polymer applications.

✓ Measured by XPS.

✓ Functional group retention also remains relatively constant at constant ion flux compared to constant RF power.

Functional group (COOR) retention versus electrode separation constant RF power of 5W and constant ion flux of 7.5 x 10^{17} ions m^{-2} s^{-1}.

![Graphs showing functional group retention versus electrode separation for different ions and RF powers.]
3. RF power coupling efficiency and Ion Flux with electrode separation variation

Measuring power coupling efficiency confirm that intrinsic plasma properties vary greatly with reactor geometry at constant applied RF power.

Ion flux and RF power coupling efficiency confirm that intrinsic plasma properties vary greatly with reactor geometry at constant applied RF power.

One can also predict the Ion Flux (as shown in Figure) from the external parameters using a simple global power balance model in CCP systems.

The ability to predict plasma parameters from external inputs for a range of gases would be extremely useful in a wide range of applications.

These results are applicable to multiple types of plasma (pulsed plasma, downstream processing); and not just continuous plasma.

(a) Plasma power actually delivered to plasma (OctIV sensor) and (b) Ion flux versus electrode separation for 4 precursors.

The ion flux offers a more widely applicable method of defining plasma polymerization processes.
Global model for Capacitively Coupled Plasma – Validation using Octiv probe

An Experimental and Analytical Study of an Asymmetric Capacitively Coupled Plasma Used for Plasma Polymerization
DOI: https://doi.org/10.1002/ppap.201400026

The objective of this paper was to develop a simple model of an asymmetric capacitively coupled plasma (CCP) used for polymer processing and to validate this model using a simple, non-invasive and low-cost Octiv probe.

Challenge
TRIAL-AND-ERROR IN PROCESS SCALE-UP

Solution
ANALYTICAL GLOBAL MODEL FOR CCP
Predicts plasma parameters (Ion flux, Ion energy flux) from external inputs (Voltage, Current, Power)

Input Parameters
• RF Power
• Electrode area
• Self Bias voltage
• Energy loss terms

Power Balance
Global Model

Output Parameters
• Plasma density
• Ion Flux
• Ion Energy Flux

Experimental setup and electrical model of plasma.
**Input Parameters**

- \( P_{\text{rms}} = U_{\text{rms}} \ I_{\text{rms}} \cos \theta \) [Using Impedans Octiv]
- Electrode areas \( A_{\text{live}} \) and \( A_{\text{earth}} \)
- Electron temperature (Approximate ~ \( T_e = 3 \text{eV} \))
- Average plasma potential \( V_{pdc} \sim 5 \ kT_e \) [Argon]
- DC self bias potential \( V_{\text{self-bias}} \) [Using Impedans Octiv]

**Power balance equations**

**STEP 1.** Determine the sheath edge plasma density \( n_{\text{sheath}} \) from energy balance:

\[
\text{Total power absorbed } P_{\text{rms}} = \text{Total power lost } (e n_{\text{sheath}} u_B A_{\text{eff}} E_T)
\]

\[
n_{\text{sheath}} = \frac{P_{\text{rms}}}{e u_B [A_{\text{live}} (E_c + E_e + (V_{pdc} - V_{\text{self-bias}})) + A_{\text{earth}} (E_c + E_e + V_{pdc})]}
\]

**STEP 2.** Determine Ion Bohm flux per unit area \( \mu_i = n_{\text{sheath}} u_B \)

**STEP 3.** Ion energy flux per unit area for live and grounded electrode

\[
P_{A_{\text{live}}} = e n_{\text{sheath}} u_B (V_{pdc} - V_{\text{self-bias}})
\]

\[
P_{A_{\text{earth}}} = e n_{\text{sheath}} u_B V_{pdc}
\]

**STEP 4.** Model estimate of \( V_{\text{self-bias}} \)

\[
V_{\text{self-bias}} = -[0.84 \times 1.4 \times \frac{V_{live}}{U_{\text{rms}}} \times \sqrt{Z P_{\text{rms}}} + 5kT_e]
\]
Results: Comparison of Octiv measurements and electrical model

Ion flux and ion energy flux as a function of RF plasma power (CCP-Argon plasma)

Self-bias potential as a function of RF generator power.

RF power supplied versus power deposited to plasma.

Measured (a) self-bias potential and (b) Ion flux versus RF generator power for range of gases, using Octiv sensor.

Estimates from model are in very good agreement with Octiv measurements.
Contribution of ions and neutral species to plasma polymer growth

On the Effect of Monomer Chemistry on Growth Mechanisms of Nonfouling PEG-like Plasma Polymers

DOI: https://doi.org/10.1021/la304713b

In this work, it is shown that the deposition of saturated monomers diglyme and triglyme are intimately linked to the ion flux to the surface. In contrast, the deposition of unsaturated monomer diethylene glycol divinyl ether (DEGDVE) is strongly dependent on neutral species.

Some example data is shown to the right.

Ion flux of diglyme (●), triglyme (▲), and diethylene glycol divinyl ether (■) at 0.5 Pa (open symbols) and 1 Pa (solid symbols).
Plasma polymer films growth: Link between deposition mechanism and physical properties

The link between mechanisms of deposition and the physico-chemical properties of plasma polymer films

DOI: https://doi.org/10.1039/c3sm51039e

This paper focusses on the link between deposition mechanism and physical properties (like density, solubility and mechanical properties etc.) of the plasma polymer films. In this regard, thin films from three classes of commonly used plasma polymers (hydrocarbons, glymes and carboxylic acids) were deposited.

Some example data is shown to the right.

Ion flux and deposition rate of saturated (open symbols) and unsaturated (closed symbols) analogues of (a) acids (b) glymes and (c) hydrocarbons at 1 Pa as a function of RF power.
To investigate the role of ions in plasma polymerization of γ-Terpinene

Structural Characterization of γ-Terpinene Thin Films Using Mass Spectroscopy and X-Ray Photoelectron Spectroscopy

DOI: https://doi.org/10.1002/ppap.201400220

In this work, plasma polymerization of γ-Terpinene has been investigated to study the influence of the precursor chemical structure on the process of polymerization. Ion flux is measured as a function of input power. Ion energy, neutral and positive ion mass spectra and film deposition rate are simultaneously monitored.

Some example data is shown to the right.

Variation of Mass deposition rate, ion flux and deposition rate per ion from a plasma of γ-Terpinene as a function of plasma Power.
Relationship between plasma parameters and film stability of aminated plasma polymers

Plasma Parameter Aspects in the Fabrication of Stable Amine Functionalized Plasma Polymer Films

DOI: https://doi.org/10.1002/ppap.201400215

This study focuses on the link between plasma parameters, monomer structure, and the stability of aminated plasma polymers. Plasma polymers were grown from allylamine (AA) and ethylenediamine (EDA) over a range of RF powers. The ion flux, ion energy, and mass deposition rates were measured and used to calculate the energy per deposited mass.

Some example data is shown to the right.
Visual inspection of the plasma reveals a change from intense homogeneous plasma at low pressure to lower intensity bulk plasma at high pressure, but with increased intensity near the walls of the chamber.

In this work, it is demonstrated that this occurs at the transition from a collision-less to a collisional plasma sheath, which in turn increases ion and energy flux to surfaces at constant RF power.
Investigation of plasma polymer deposition under low pressure conditions

The chemistry of organophosphate thin film coatings from low pressure plasma and the effect of the substrate on adhesion

DOI: https://doi.org/10.1002/ppap.201700037

The objective of this paper was to investigate the deposition of organophosphate thin films under low pressure plasma. Using triethyl phosphate as the precursor, the effect of power on the chemistry of the plasma phase and the deposited thin film is studied at constant pressure in the absence of atmospheric nitrogen.

Some example data is shown to the right.
Plasma polymerization: Deposition of organic thin films derived from sandalwood oil

Fabrication and characterization of bio-renewable plasma polymer films using sandalwood oil precursor

DOI: [https://doi.org/10.1002/app.49288](https://doi.org/10.1002/app.49288)

In this work, organic thin films derived from sandalwood oil were deposited using plasma polymerization over a range of radio-frequency (RF) powers, with the aim of minimizing degradation of the precursor.

Some example data is shown to the right.