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

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A Ion flux as an alternative deposition rate parameter

Defining Plasma Polymerization: New Insight Into What We Should Be Measuring

Andrew Michelmore et al, Mawson Institute, University of South Australia, Australia
Research School of Physics and Engineering, The Australian National University, Australia

DOI: https://doi.org/10.1021/am401484b

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

Plasma polymerization

- Depositing ultrathin functionalized films onto surfaces

Typical 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

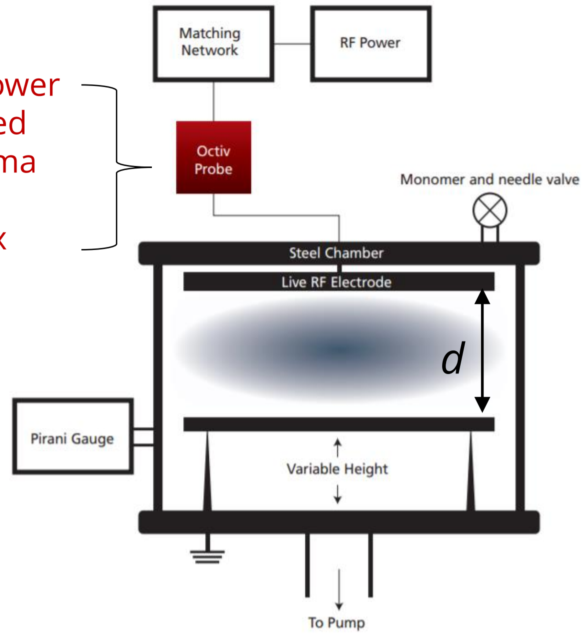
For plasma reactors with different geometries, external parameters are near useless for scale-up and process control.

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.

- RF power delivered to plasma
- Ion Flux



Experimental setup of Capacitively Coupled Plasma (CCP) system

A Research Highlights

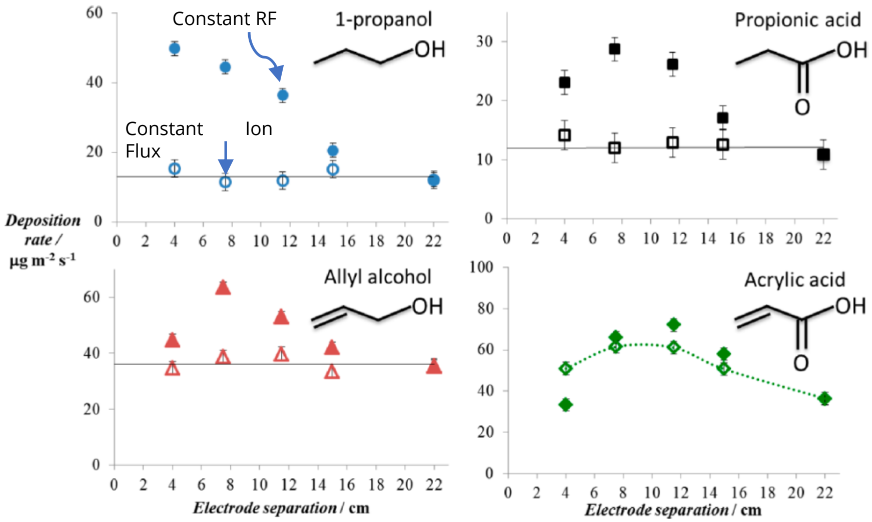
Experimental conditions: Capacitively Coupled (13.56 MHz) Plasma, 1Pa pressure, RF power 5W

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

Diagnostics used in study	Measured parameters
• Impedans – Octiv sensor	The ion flux to the RF electrode (Post-match)
• Sycon Instruments - Quartz Crystal Microbalance	Deposition rate
• SPECS SAGE spectrometer	X-ray Photoelectron Spectroscopy spectra

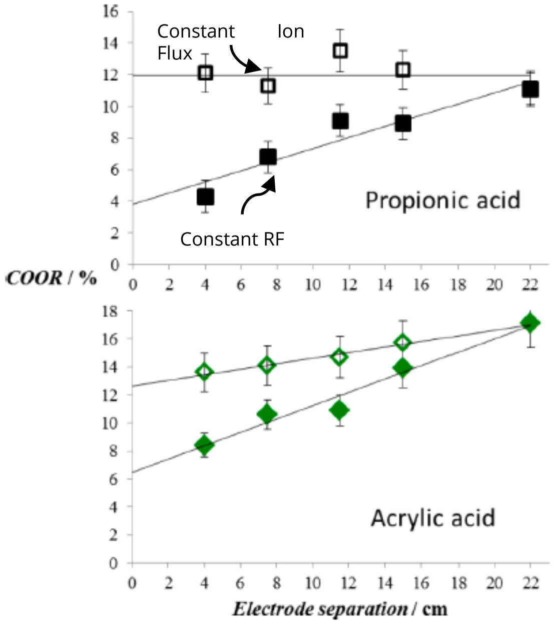
1. The deposition rate is intimately linked with the ion flux, not RF power

- ✓ 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 7.5×10^{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 7.5×10^{17} ions m^{-2} s^{-1}.

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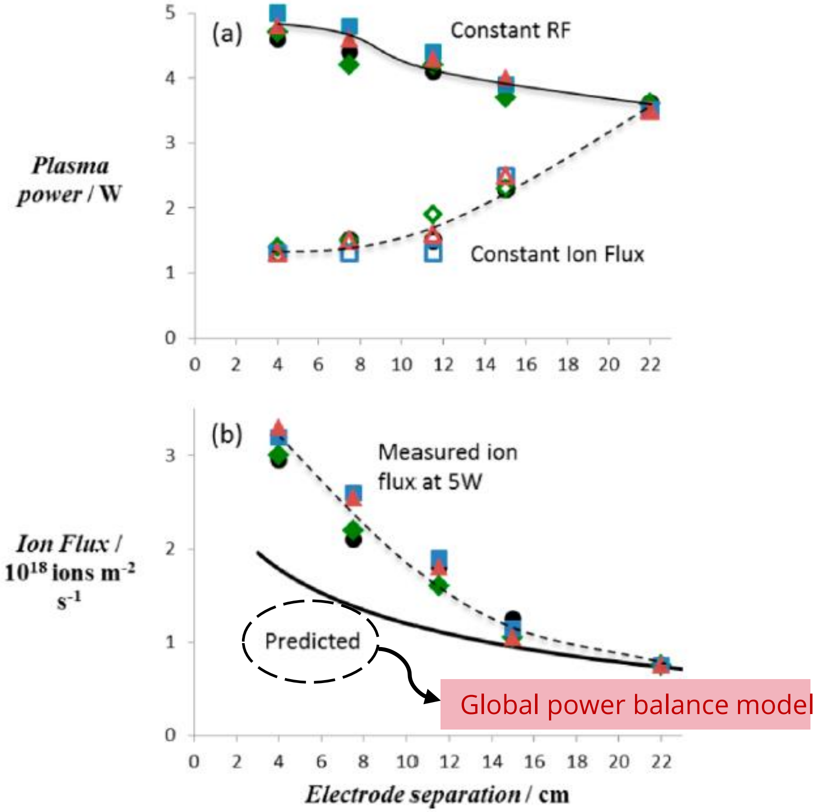
A Research Highlights

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.



Global power balance model

(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.

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

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

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.

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B 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

Andrew Michelmore et al, Mawson Institute, University of South Australia, Australia
Research School of Physics and Engineering, The Australian National University, Australia

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.

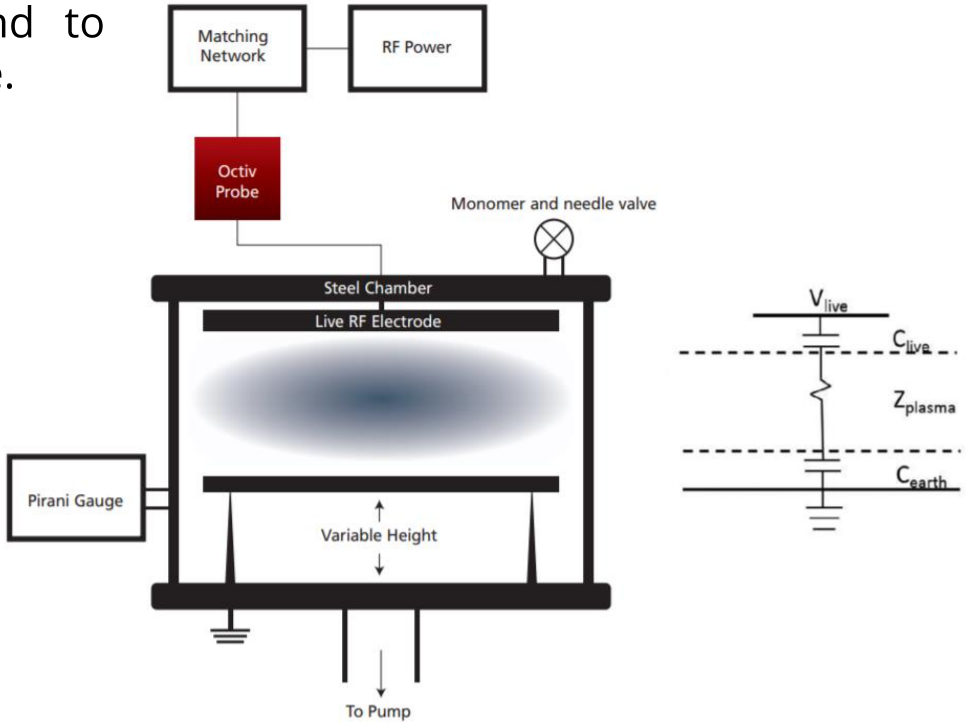
Challenge: TRIAL-AND-ERROR IN PROCESS SCALE-UP

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

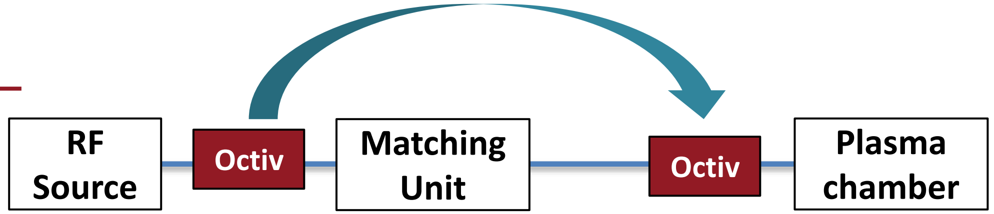


Experimental setup and electrical model of plasma.

B Overall scheme of Octiv Application in CCP

Input Parameters

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



Pre-match condition

U_{rms}, I_{rms}, θ, P_{rms} and $Z = \frac{U^2_{rms}}{P_{rms}}$

Post-match condition

$V_{live} = V_{dc} + V_{RF} = V_{self\text{-}bias} + V_{RF}$, Ion flux, Ion energy flux

Power balance equations

STEP 1. Determine the sheath edge plasma density n_{sheath} from energy balance:

Total power absorbed P_{rms} = Total power lost $(e n_{sheath} u_B A_{eff} E_T)$

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

STEP 2. Determine Ion Bohm flux per unit area $\quad \mu_i = n_{sheath} u_B$

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

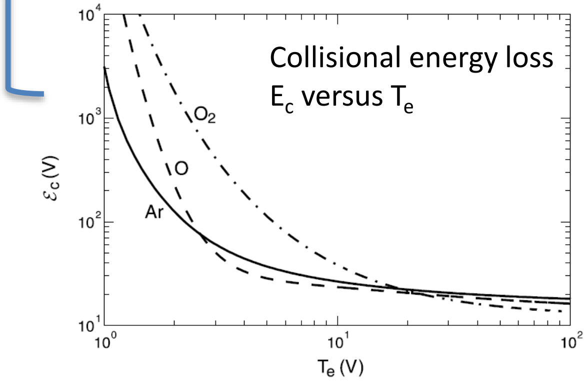
$$P_{A_{live}} = e n_{sheath} u_B \left(V_{pdc} - V_{self-bias}\right) \qquad P_{A_{earth}} = e n_{sheath} u_B V_{pdc}$$

STEP 4. Model estimate of $V_{self\text{-}bias}$

$$V_{self-bias} = -[0.84 \times 1.4 \times \frac{V_{live}}{U_{rms}} \times \sqrt{Z P_{rms}} + 5kT_e]$$

$$Bohm\ velocity\ u_B = \sqrt{\frac{kT_e}{M}}$$

$E_e = 2\,kT_e$



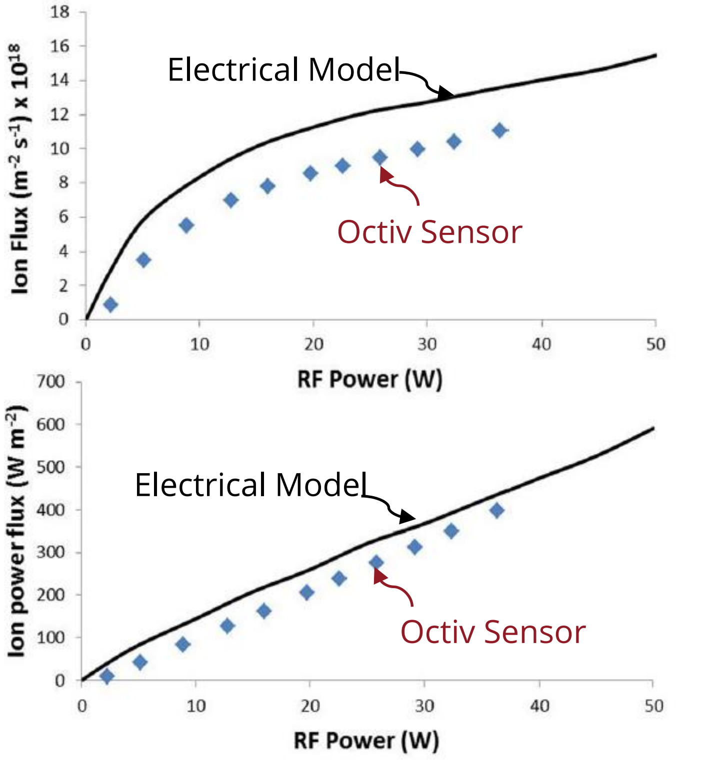
Collisional energy loss
E_c versus T_e

E_T: Total energy lost per electron–ion pair lost from the system

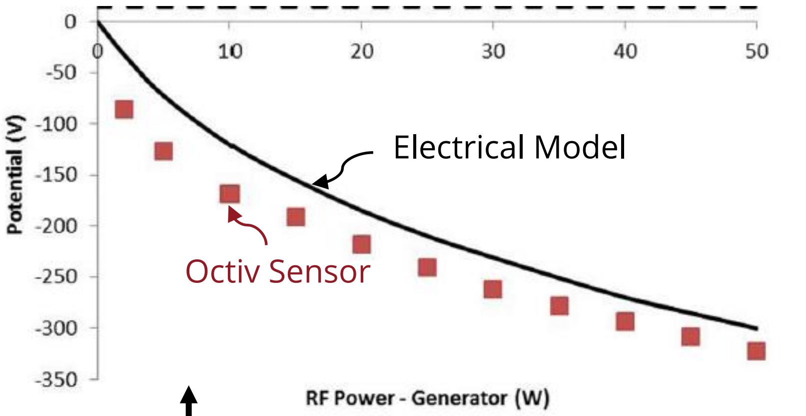
$E_T = E_c + E_e + E_i$

Lieberman *et al.* (2005)

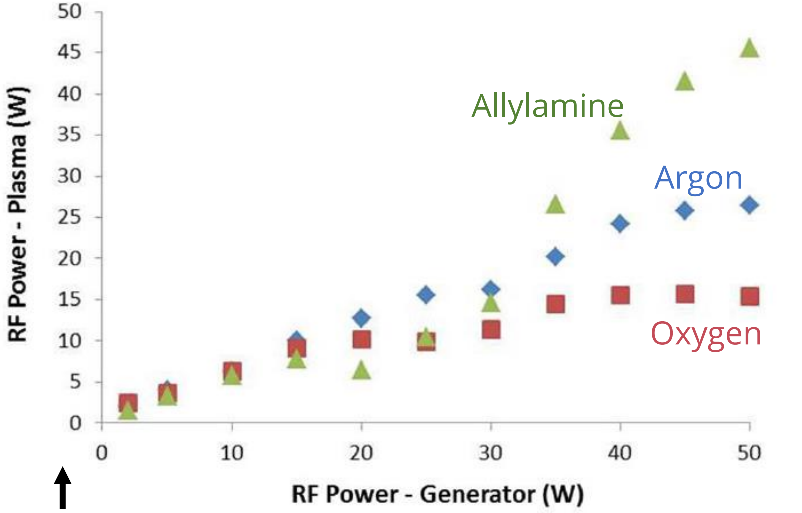
B Results: Comparison of Octiv measurements and electrical model



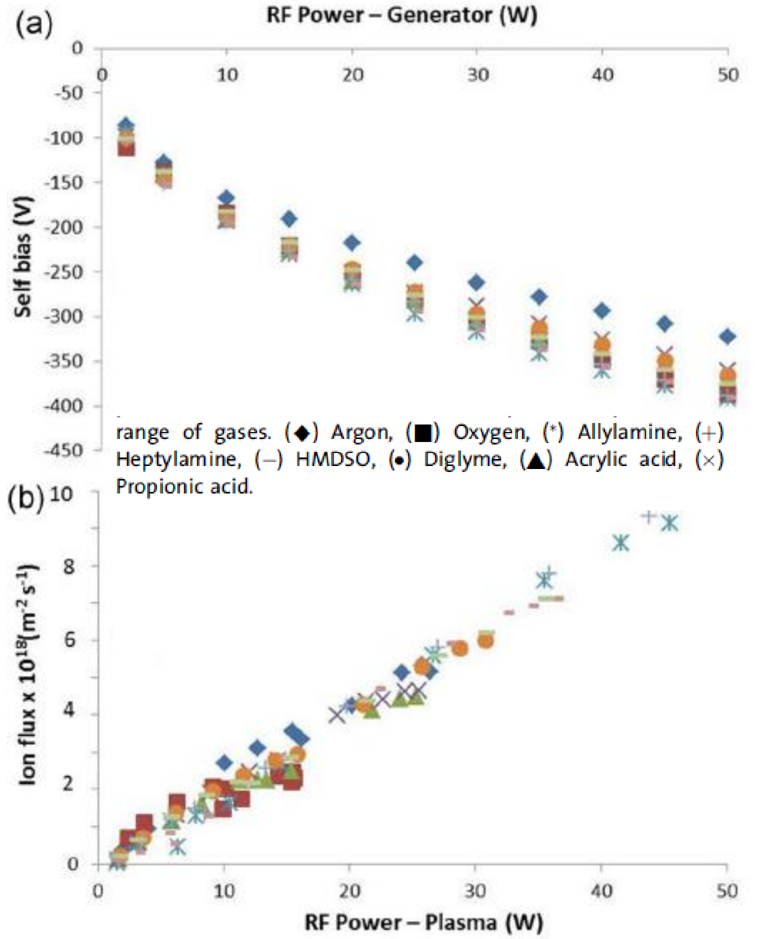
Ion flux and ion energy flux as a function of RF plasma power (CCP-Argon plasma)



Self-bias potential as a function of RF generator



RF power supplied versus power deposited to plasma.



range of gases. (◆) Argon, (■) Oxygen, (*) Allylamine, (+) Heptylamine, (–) HMDSO, (●) Diglyme, (▲) Acrylic acid, (×) Propionic acid.

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.

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Octiv applications continued

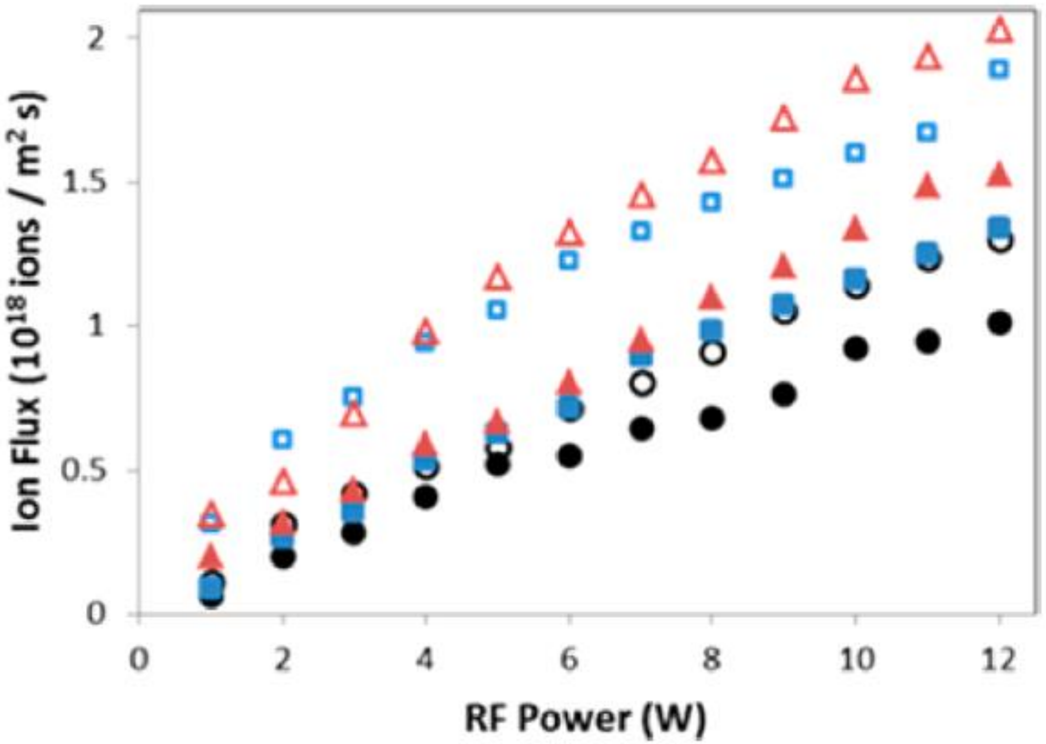
Contribution of ions and neutral species to plasma polymer growth

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

Andrew Michelmore et al, Mawson Institute, University of South Australia, Australia
School of Applied Chemistry, Reutlingen University, Germany

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.



Ion flux of diglyme (●), triglyme (▲), and diethylene glycol divinyl ether (■) at 0.5 Pa (open symbols) and 1 Pa (solid symbols).

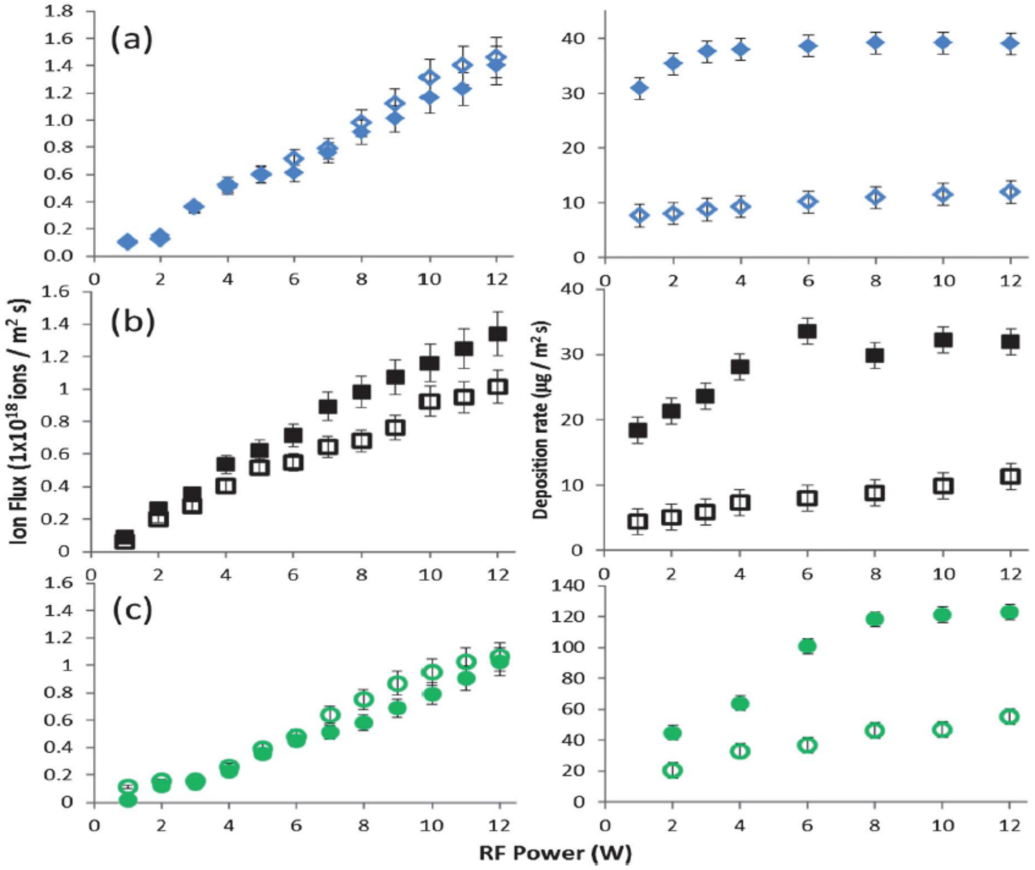
Plasma polymer films growth: Link between deposition mechanism and physical properties

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

Andrew Michelmore et al, Mawson Institute, University of South Australia, Australia

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.



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

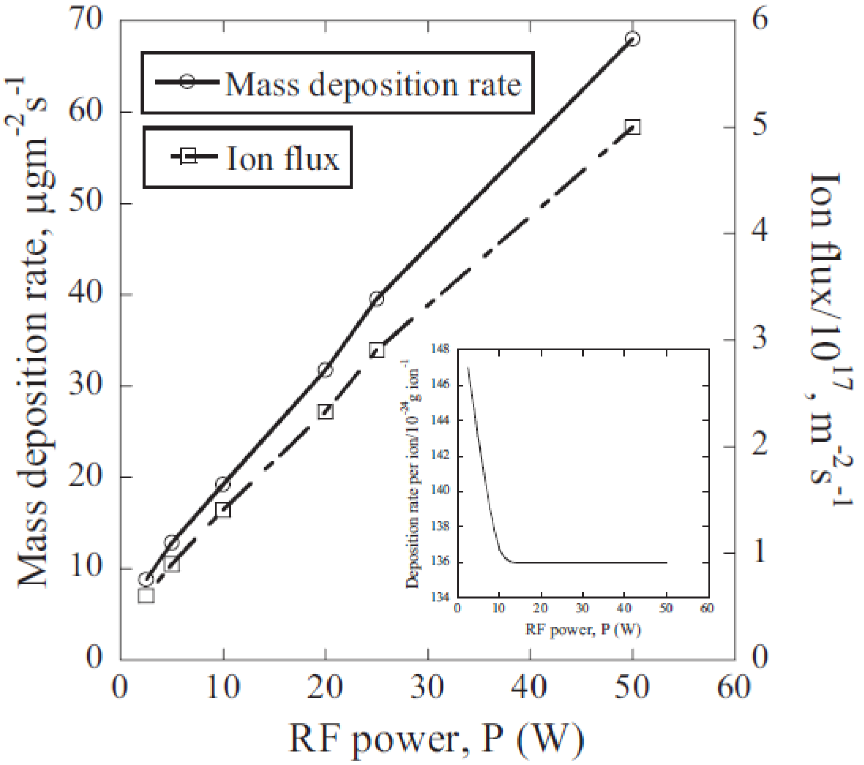
C To investigate the role of ions in plasma polymerization of γ-Terpinene

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

Jakaria Ahmad et al, College of Science, Technology and
Engineering, James Cook University, Australia
Mawson Institute, University of South Australia, Australia

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.



Variation of Mass deposition rate, ion flux and deposition rate per ion from a plasma of γ-Terpinene as a function of plasma Power.

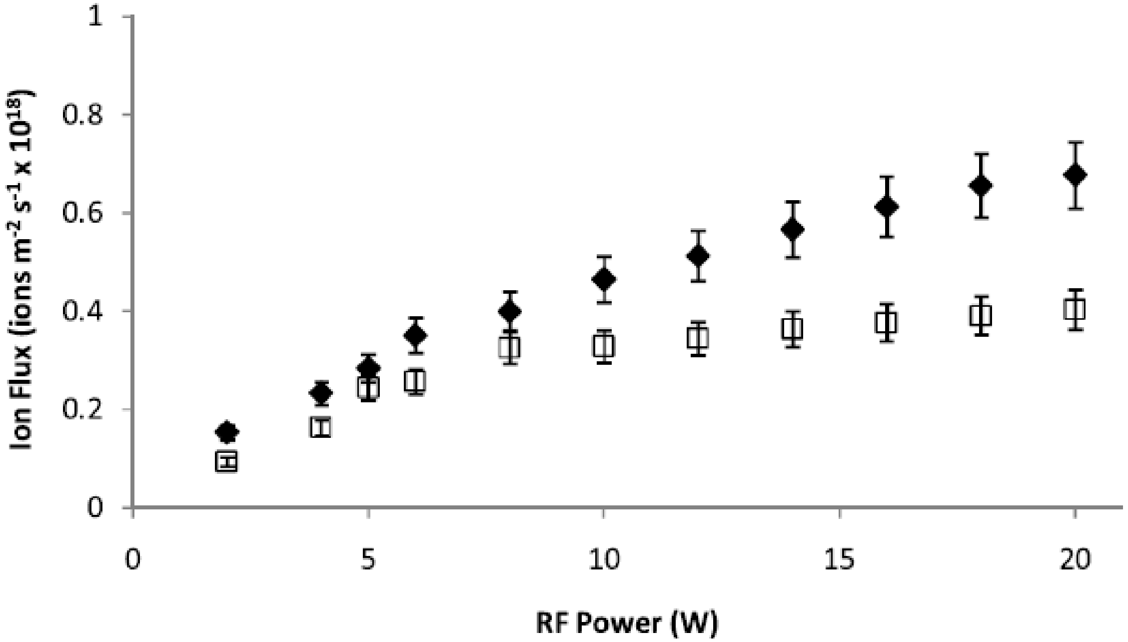
C Relationship between plasma parameters and film stability of aminated plasma polymers

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

Daunton C et al, Mawson Institute, University of South Australia, Australia

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.



Variation of ion flux of allylamine (◆) and ethylenediamine (☐) plasma at 4 sccm as a function of RF power.

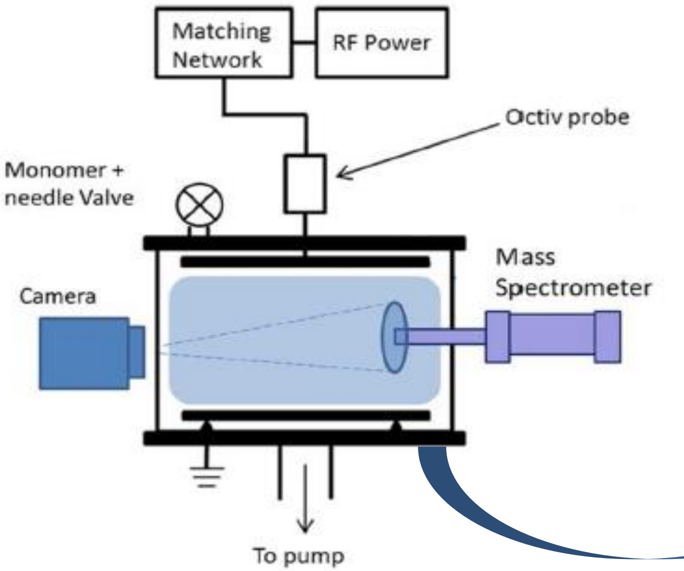
Effect of pressure on the chemical and physical processes of plasma polymerization

Comparison of Plasma Polymerization under Collisional and Collision-Less Pressure Regimes

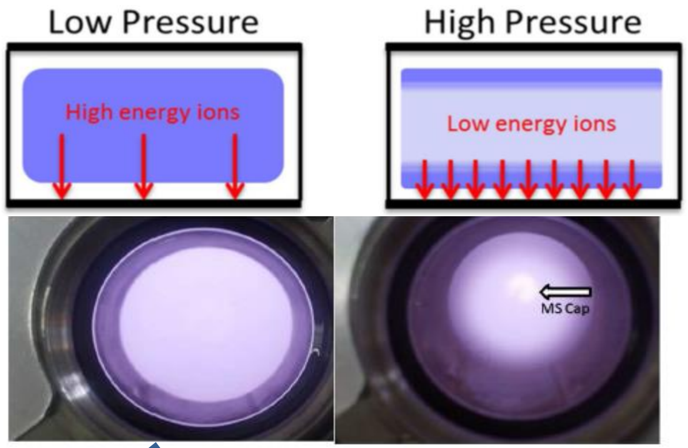
Saboohi S et al, Mawson Institute, University of South Australia, Australia

DOI: https://doi.org/10.1021/acs.jpcb.5b07309

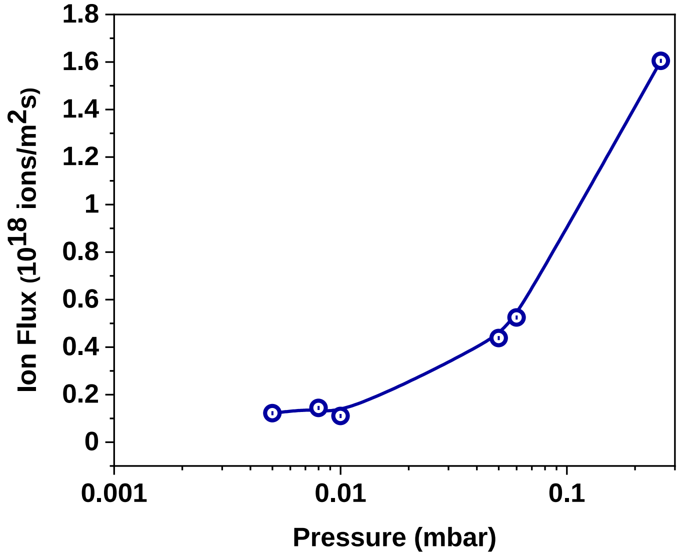
- ✓ Visual inspection of the plasma reveals a change from intense homogeneous plasma at low pressure to lower intensity bulk plasma at high pressure, but with increased intensity near the walls of the chamber.
- ✓ In this work, it is demonstrated that this occurs at the transition from a collision-less to a collisional plasma sheath, which in turn increases ion and energy flux to surfaces at constant RF power.



Experimental setup and diagnostics



Glow of 0.01 mbar (left) and 0.07 mbar (right) plasmas of ethanol at 15 W. Arrow indicates the position of the cap of the plasma mass spectrometer.



Ion flux measured using Octiv at fixed plasma power (15W) over the pressure range.

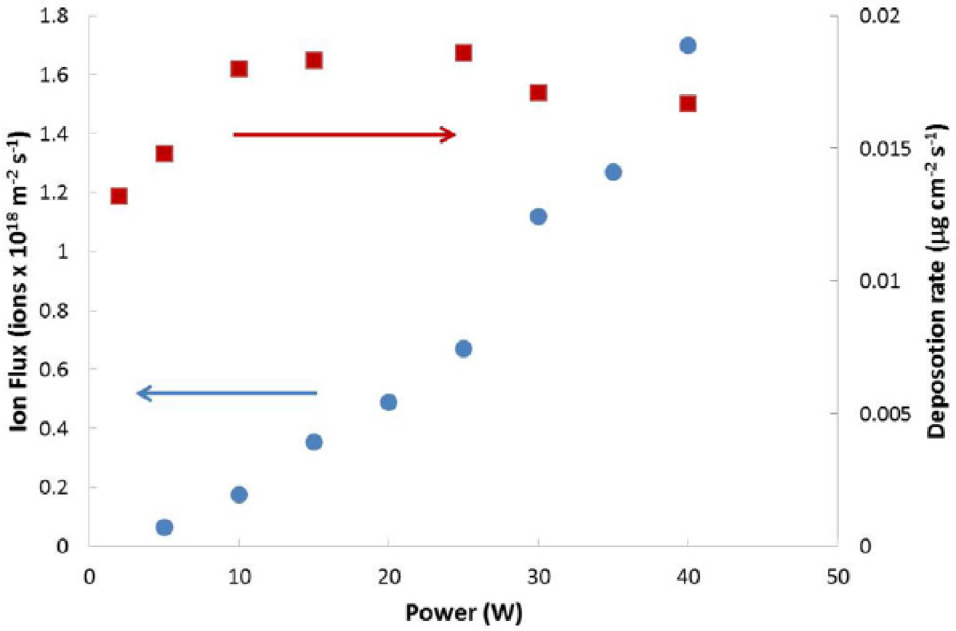
c Investigation of plasma polymer deposition under low pressure conditions

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

Paulino R V et al, Pontifícia Universidade Católica de Minas Gerais, Brazil
Future Industries Institute, University of South Australia, Australia

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.



Ion flux and deposition rate as a function of applied RF power for 1 Pa triethyl phosphate plasma.

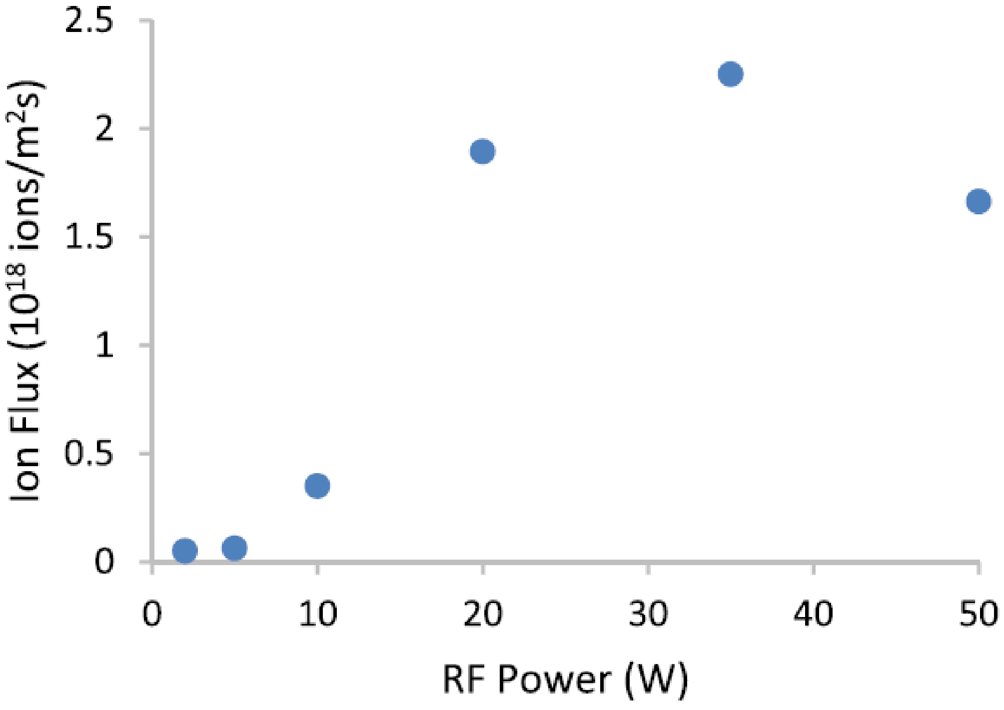
C Plasma polymerization: Deposition of organic thin films derived from sandalwood oil

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

Hennekam B E et al, School of Natural Built Environments, University of South Australia, Australia
Future Industries Institute, University of South Australia, Australia

DOI: 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.



Ion flux versus RF power for sandalwood oil plasmas

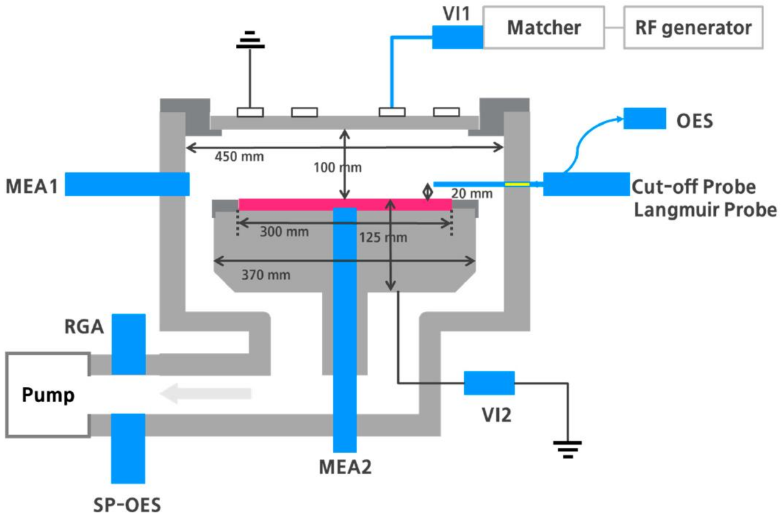
c Plasma Nitriding process: Nitriding of Silicon Oxide film

Comprehensive Data Collection Device for Plasma Equipment Intelligence Studies

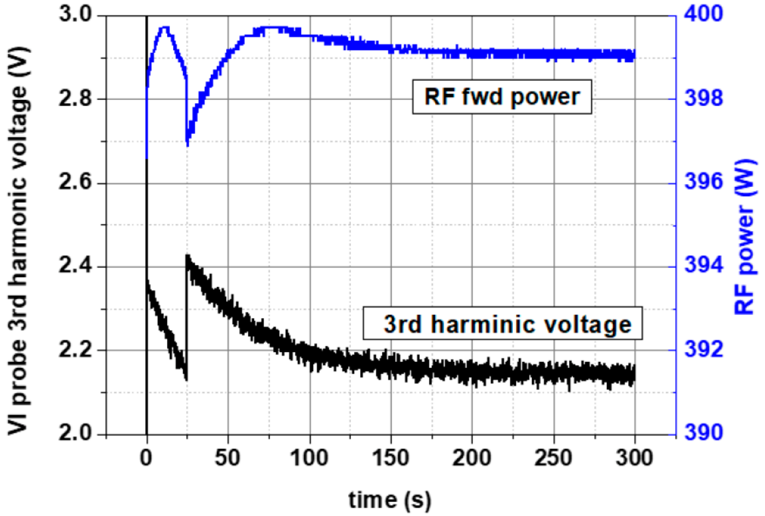
Kim Y H et al, Fundamental Technology Research Division, Institute of Plasma Technology, Korea Institute of Fusion Energy, Korea
Plasma E.I. Convergence Research Center, Korea Institute of Fusion Energy, Korea

DOI: https://doi.org/10.3390/coatings11091025

In this work, plasma parameters were measured and analyzed for the intelligence evaluation of plasma process equipment. The correlation between the measured data was investigated using regression analysis. The association of VI probe specific harmonics with the RF power changes indicated that VI probe specific harmonics might be associated with plasma parameters or process results.



Schematic of the experimental device showing VI probe (VI1, VI2)



RF forward power and 3rd harmonic voltage of VI probe (VI1, antenna), RF power 400 W and 10 mTorr pressure.

C ICP E-H mode transition: Studies on power transfer efficiency

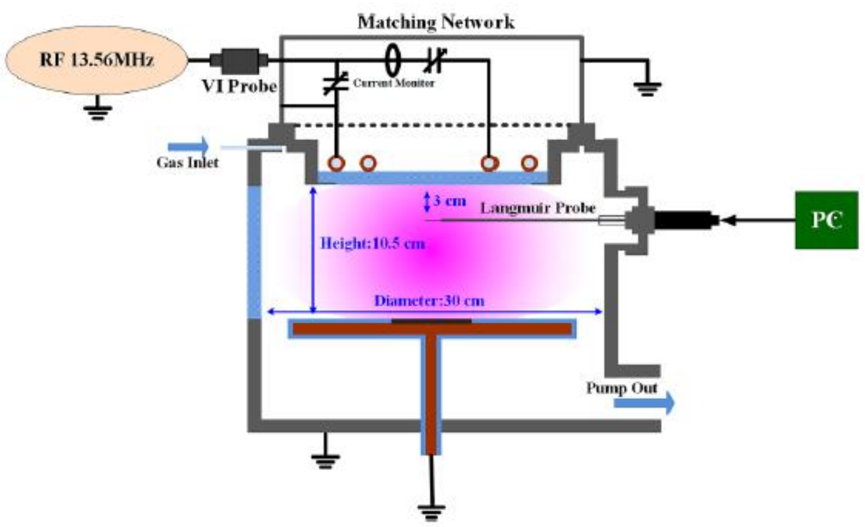
Power transfer efficiency and the power threshold for E–H mode transition in inductively coupled Plasmas

Du P C et al, Key Laboratory of Materials Modification by Laser, Ion, and Electron Beams, Dalian University of Technology, People's Republic of China
College of Physical Science and Technology, Dalian University, People's Republic of China

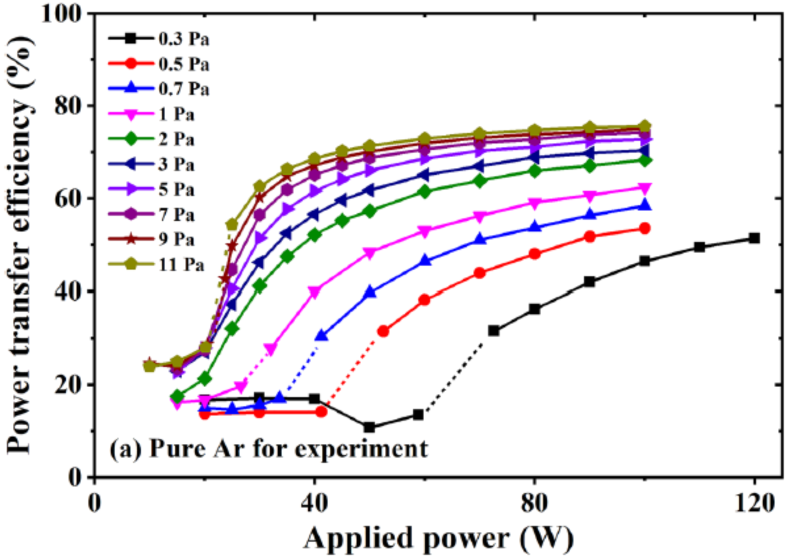
DOI: https://doi.org/10.1063/5.0077769

In this work, the effects of gas pressure and gas component on the power transfer efficiency η and transition power threshold P_{th} during the E–H mode transition in inductively coupled plasmas are studied. Experiments were performed using pure Ar and Ar/O_2 discharges.

$$\eta = \frac{P_{abs}}{P_{app}} = \frac{P_{app} - I_{rms}^2 r}{P_{app}},$$



Schematic diagram of the ICP chamber.

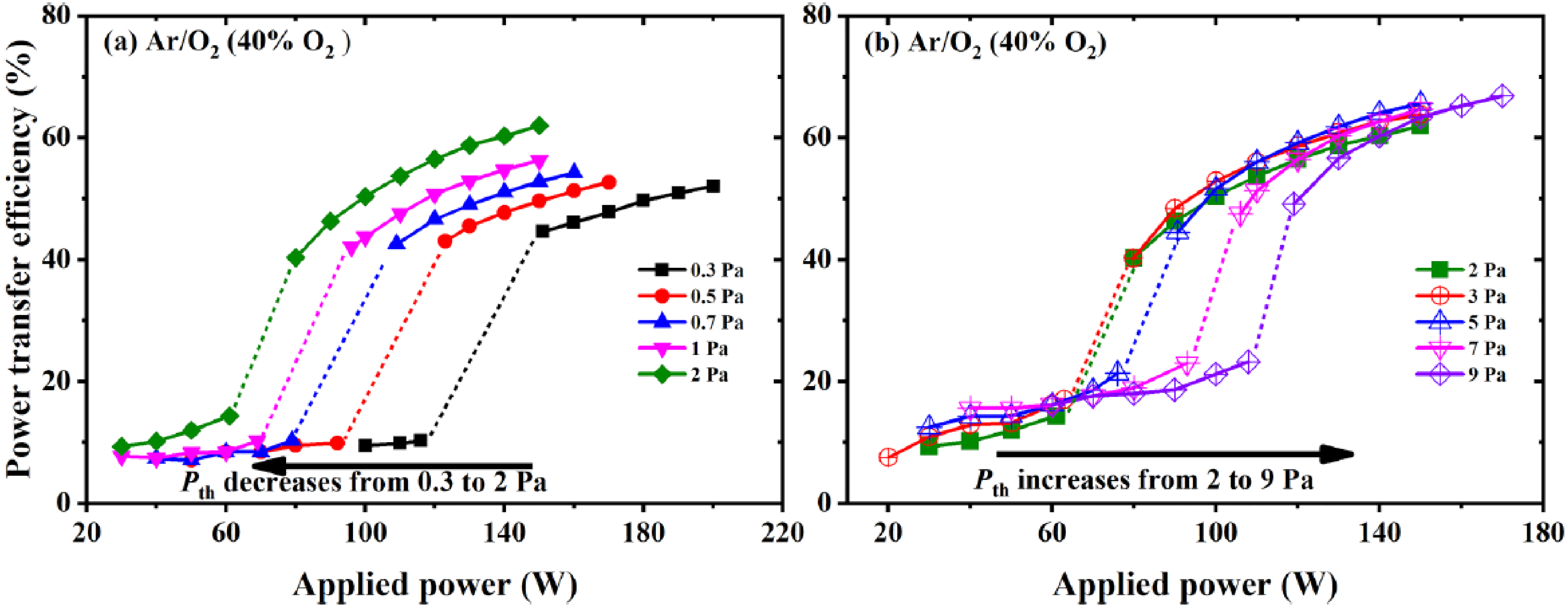


The power transfer efficiency obtained by experiment vs the applied power during the E–H mode transition in pure Ar discharge at different pressures.

C

ICP E-H mode transition: Studies on power transfer efficiency

Power transfer efficiency and the power threshold for E–H mode transition in inductively coupled plasmas



The power transfer efficiency measured by the experiment vs the applied power during the E–H mode transition at (a) 0.3–2 and (b) 2–9 Pa for 40% O_2 content in Ar/O_2 discharge, respectively

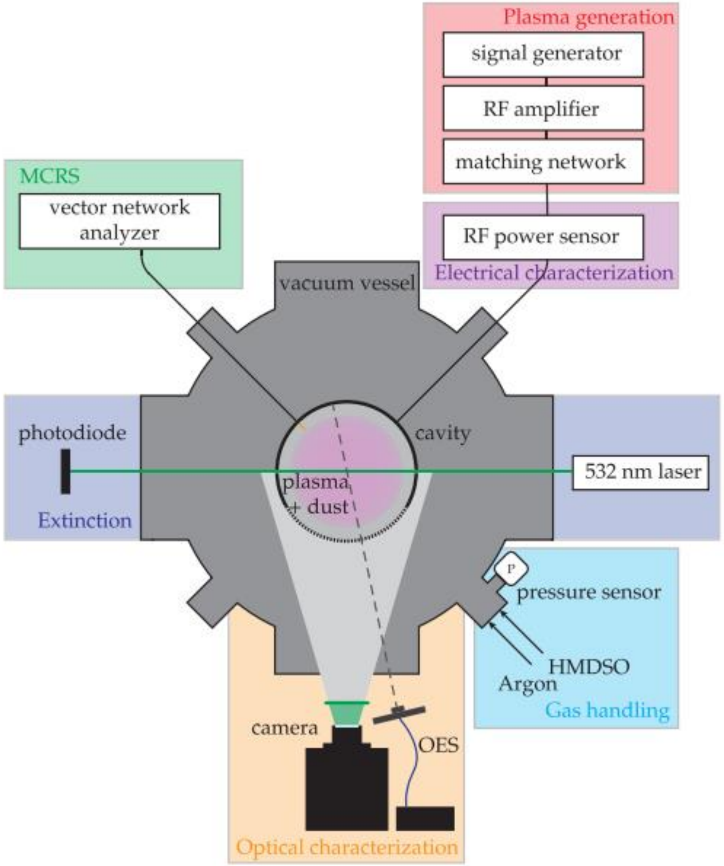
C Investigations on the cyclic growth of dust particles

Characterization of cyclic dust growth in a low-pressure, radio-frequency driven argon-hexamethyldisiloxane plasma

T J M Donders et al, Department of Applied Physics, Eindhoven University of Technology, Eindhoven, The Netherlands

DOI:https://iopscience.iop.org/article/10.1088/1361-6463/ac802a

Study reports the variation of several discharge parameters such as plasma power and hexamethyldisiloxane content related to the fundamental understanding of the mechanisms behind dust growth in low-pressure plasmas.



Schematic top view of the experimental setup

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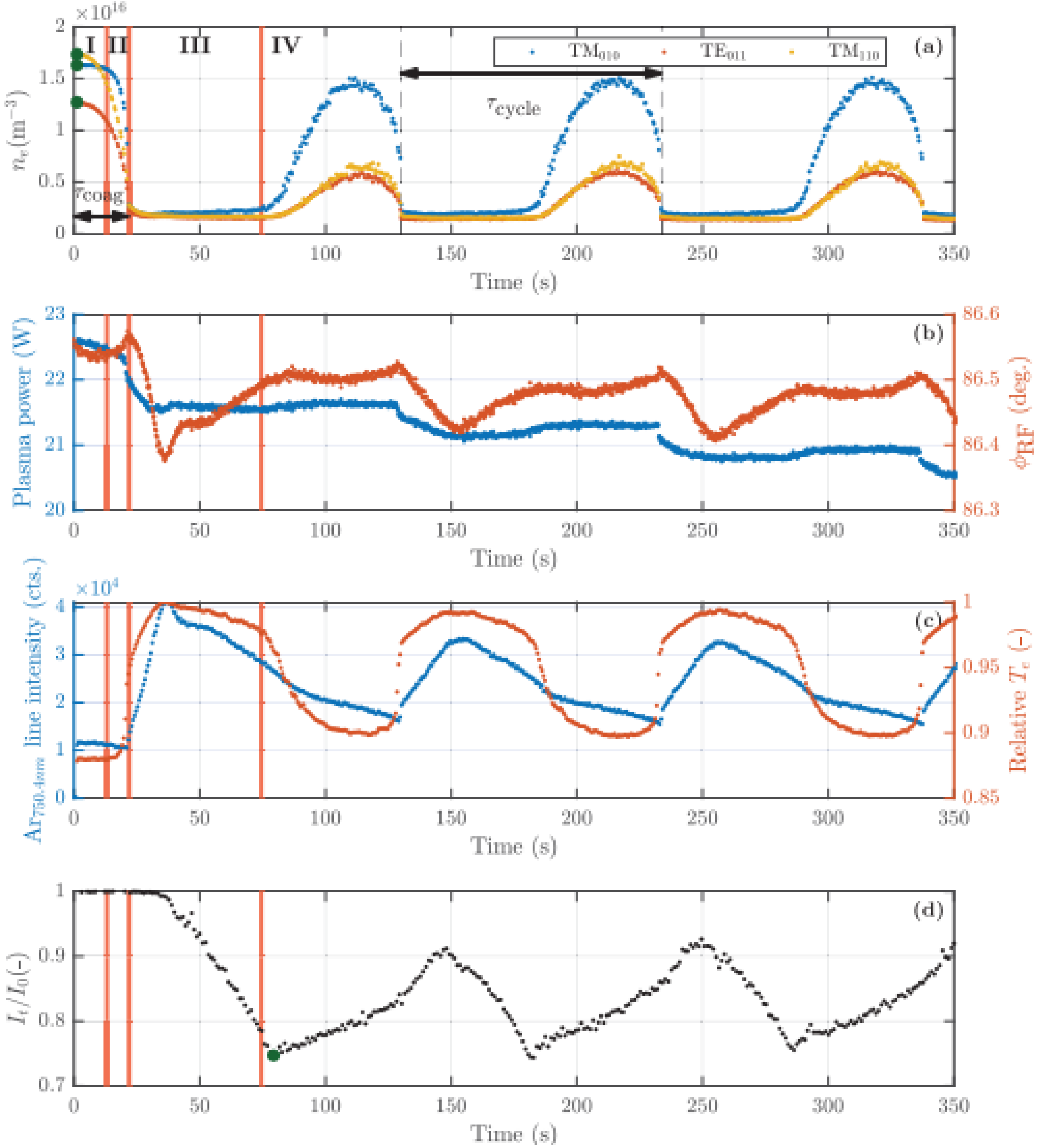
C Investigations on the cyclic growth of dust particles

Characterization of cyclic dust growth in a low-pressure, radio-frequency driven argon-hexamethyldisiloxane plasma



Overview of the results of a typical dust growth monitoring experiment. Panel (a) shows the electric-field-squared-weighted volume-averaged free electron density in the plasma derived from multimode MCRS. Panel (b) shows the results of the electrical characterization, indicating the plasma power in blue and the phase angle in red. Panel (c) shows the results of optical emission spectroscopy, in the form of the line intensity of the most intense argon line (λ = 750.4 nm) and the relative behavior of the electron temperature. Panel (d) shows the results of the laser light extinction measurements, by showing the transmittance ratio I_t/I_0*. For this measurement, operating pressures were* $p_{total} = 6.5$ *Pa,* $p_{Ar} = 5.4$ *Pa and* $p_{HMDSO} = 1.1$ *Pa*

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