

## Plasma Science and Technology Division Room A106 - Session PS+SE-MoA

### Plasma Sources, Diagnostics, Sensors and Control

**Moderators:** Michael Gordon, University of California at Santa Barbara, Yohei Ishii, Hitachi High Technologies America Inc.

1:40pm **PS+SE-MoA-1 On the Influence of the Target Material on the Discharge Properties of the High Power Impulse Magnetron Sputtering Discharge**, *Jon Tomas Gudmundsson*, K. Barynova, University of Iceland; M. Rudolph, Leibniz Institute of Surface Engineering (IOM), Germany; J. Fischer, Linköping University, Sweden; S. Suresh Babu, University of Iceland; M. Raadu, N. Brenning, KTH Royal Institute of Technology, Sweden; D. Lundin, Linköping University, Sweden

High power impulse magnetron sputtering (HiPIMS) operation results in increased ionization of the sputtered species and lower deposition rate than the dc magnetron sputtering discharge, when operated at the same average power. We have applied the ionization region model (IRM) [1] to model HiPIMS discharges in argon with a number of different targets [2,3], to study various processes, such as working gas rarefaction and refill processes, the electron heating mechanisms, ionization probability and back-attraction of the sputtered species, and recycling mechanisms. The HiPIMS discharge can contain a large fraction of ionized sputtered material and often a significant fraction, of the ions involved in the sputter process are ions of the target material. This also implies that a large fraction of the ions of the sputtered species can be attracted back to the target and are not deposited on the substrate to form a film or coating. Self-sputtering and the self-sputter yield are therefore expected to play a significant role in HiPIMS operation, and have a decisive impact on the film deposition rate, at least for metal targets. We explore the relationship between the self-sputter yield and deposition rate as well as the ionization and back attraction probabilities. The back-attraction probability appears to decrease with increased self-sputter yield. The various contributions to working gas rarefaction including electron impact ionization, kick-out by the sputtered species, and diffusion, are evaluated and compared for the different target materials, over a range of discharge current densities. For all cases the working gas rarefaction is found to be significant, and to be caused by several processes, and that their relative importance varies between different target materials. In the case of a graphite target, electron impact ionization is the dominating contributor to the working gas rarefaction, with 55 - 64 % contribution, while the kick-out, or sputter wind, has negligible influence, whereas in the case of tungsten target, the kick-out dominates, with 39 - 48 % contribution. The relative role of kick-out by the sputtered species increases and the relative role of electron impact ionization decreases with increased mass of the target atoms.

[1] Huo et al., Journal of Physics D: Applied Physics 50, 354003 (2017)

[2] Gudmundsson et al., Surface and Coatings Technology 442, 128189 (2022).

[3] Babu et al., Plasma Sources Science and Technology 31, 065009 (2022)

2:00pm **PS+SE-MoA-2 Numerical Analysis of Curling Probe Designing for an Effective Electron Density Measurement in Plasma**, *Daisuke Ogawa*, S. Kato, H. Sugai, K. Nakamura, Chubu University, Japan

Electrons make a main contribution to generating reactive species in a low-temperature plasma. Optical emission is often utilized to monitor plasma, but it should be noted that the ultimate origin of the emission is due to collisions with the electrons in the plasma. This means that electron monitoring could be the primary information of the plasma. A curling probe is one of the probes that enable an electron density measurement in the plasma. The probe measures the density derived from a shift of the fundamental resonant frequency that the probe holds. Therefore, the probe measures the density even in an environment where the plasma makes a dielectric film deposition. The probe utilizes a slot antenna to make the electromagnetic resonance, which is equipped on the top surface of the probe. This antenna structure gives an advantage in directional electron density measurement. This directionality is useful, particularly when the probe desires to be embedded into a wall and/or an electrode. Recently, we have also developed a technique with the curling probe that enables one to make in-situ measurements of electron density in plasma and the film

thickness deposited on the probe surface. The technique requires two different-sized curling probes, so we named it the double curling probe method. This technique is potentially powerful in a plasma-processing reactor with electron density monitoring. However, we noticed that the technique requires further improvement in their measurement resolution; the frequency shift is not always noticeable, especially when the deposited film thickness is small. The frequency resolution depends on the quality factor of an inverted peak in the reflectance spectrum. According to the circuit theory, the factor depends on antenna configuration, such as the antenna's resistance, inductance and capacitance. These parameters depend on the antenna design, so we have researched how curling probe design affects the factor with an electromagnetic wave simulator, CST microwave studio. Our recent result showed that the factor depends on the antenna material, the antenna length, and the antenna thickness. In particular, the long antenna helps stabilize the factor even when increasing electron density in plasma. In this presentation, we will show our recent analysis to suggest what antenna design a curling probe ought to have to improve electron density measurement with a curling probe.

2:20pm **PS+SE-MoA-3 Annular Beam Confocal Laser-Induced Fluorescence Diagnostic for Measurements of Ion Velocity Distribution Function in Industrial Plasmas**, *Ivan Romadanov*, Y. Raitses, Princeton Plasma Physics Laboratory

Laser-Induced Fluorescence (LIF) is a powerful diagnostic tool for analyzing ion velocity distribution functions (VDFs) in plasma [1]. However, the requirement for two-sided access to plasma for beam injection and fluorescence collection in conventional LIF configuration is not always practical. Confocal LIF configurations, which are widely used in various fields such as biology and medicine, have been developed for several plasma diagnostic applications [2]. The primary advantage of confocal LIF configurations is the coincidence of the laser beam injection and fluorescence collection branches, enabling measurements in systems with limited optical access or complex geometries. This study introduces a novel variation of confocal LIF - Annular Beam Confocal Laser-Induced Fluorescence (ABC-LIF) configuration [3]. The proposed LIF configuration utilizes a structured Laguerre-Gaussian laser beam with an annular intensity profile, generated by diffractive axicons. This approach facilitates LIF signal collection along the main optical axis within the ring region while controlling spatial resolution through laser beam parameters, such as annulus thickness and beam diameter. Consequently, all enclosed fluorescence light is collected, maximizing the signal-to-noise ratio (SNR). This method achieves a spatial resolution of approximately 5 mm at a 300 mm focal distance, with the potential for 1 mm resolution, comparable to conventional LIF. The ABC-LIF configuration benefits from a small depth of field (DOF), typically achieved by Gaussian beams of large diameter, while the Laguerre-Gaussian beam allows for maintaining spatial separation between fluorescence and laser lights at comparable DOF values. Additionally, the configuration circumvents issues with beam back reflection. The ABC-LIF configuration was experimentally verified in industrial DC plasma source measurements of argon ion VDFs. Comparisons between confocal and conventional LIF revealed good agreement in determining plasma parameters, such as ion temperature, flow velocities, and ion density profiles. Applicable to various plasma processing equipment and sources, including hollow cathodes, microplasmas, and electric propulsion, the ABC-LIF configuration presents a promising diagnostic tool for industrial plasmas.

#### References

[1] Bachet G et al 1998 Phys. Rev. Lett. 80 3260

[2] Thompson D et al 2017 Rev. Sci. Instrum. 88 103506

[3] I. Romadanov, Y. Raitses, arXiv preprint arXiv:2303.12580. (2023)

Funding Acknowledgement: This work was performed under the U.S. Department of Energy through contract DE-AC02-09CH11466.

2:40pm **PS+SE-MoA-4 Control of Electron Energy Distribution Function in Electron Beam Generated ExB Plasma**, *Nirbhav Chopra*, Y. Raitses, Princeton Plasma Physics Laboratory

Electron beam (e-beam) generated plasmas are promising for low pressure, low damage threshold material processing applications requiring efficient generation of ions and radical species [1,2]. The production of reactive species generated by electron impact is controlled by the electron energy distribution function (EEDF). In this work, we investigate the EEDF and plasma parameters of a partially magnetized plasma generated by e-beam in low pressure (0.1-10 mTorr) argon and nitrogen. The e-beam (energy < 100 eV) is extracted from a negatively biased thermionic filament and

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injected into a cylindrical vacuum chamber with applied axial magnetic field. The EEDF is measured using electrostatic probes. Results show the presence of e-beam electrons with energies comparable with the applied cathode voltage and a group of warm electrons (10-30 eV). Mechanisms of the formation of this intermediate group of electrons will be discussed. In addition, we will present and discuss the effect of the addition of nitrogen gas to the argon plasma on the EEDF.

[1] Zhao F et al C G 2021 *Carbon* **177** 244–51

[2] Walton S G et al 2015 *ECS J. Solid State Sci. Technol.* **4** N5033–40

3:00pm **PS+SE-MoA-5 Expanding the Capabilities of Microwave Hairpin Resonator Probes**, *Steven Shannon*, North Carolina State University  
**INVITED**

Microwave hairpin resonator probes are a common diagnostic for measuring electron density in plasmas. They are particularly effective in low temperature plasmas, RF driven plasmas, reactive (particularly depositing) plasma chemistries, and other plasma environments that can challenge the effective use of comparable probe diagnostics such as Langmuir probes or emissive probes. Efforts to increase the utility of these probes through both innovative probe design (such as biasing and curling probe design) and combination of the hairpin probe with other diagnostic techniques (such as laser photodetachment studies in electronegative discharges) have increased the utility of hairpin probes in the field of experimental plasma science. This work presents efforts to expand on the measurement capabilities of these probes in two ways. First, the analysis of resonance data is expanded to account for plasma contributions to the Q-factor of the loaded resonance circuit. From this, additional plasma parameters such as electron neutral collision frequency can be estimated. Second, the time resolution of these probes are expanded to provide insight into the time modulation of plasma discharges including pulsed RF discharges, and can be extended to time resolved measurements within the period of an RF driven system, complimenting the growing area of phase resolved plasma characterization. The methods for expanding the capabilities of these probes are presented in this talk as well as examples of where this extension of probe capability has provided insight into basic plasma phenomena including moderate pressure operation of RF discharges, sheath heating, probe perturbation effects on density measurement, electronegative plasma instabilities, and the role of plasma edge uniformity on power coupling in inductively coupled plasma reactors. This work has been supported by the National Science Foundation, U.S. Department of Energy, Samsung Electronics, Applied Materials Inc. MKS Instruments and the state of North Carolina.

4:00pm **PS+SE-MoA-8 Time-Resolved Electron Energy Distribution in a Multi-Frequency Capacitively Coupled Plasma Reactor**, *C. Kelly, Md. Amzad Hossain, D. Kapelyan, D. Ruzic*, University of Illinois at Urbana-Champaign

This work uses a time-resolved Langmuir probe to measure the electron energy distribution function (EEDF) in a capacitively-coupled parallel-plate (CCP) plasma reactor. The EEDF completely determines the plasma chemistry in a low-temperature plasma, and that is why it is so important to obtain. By seeing how the EEDF changes throughout an RF cycle, both as a function of time and position, one then knows the extent by which altering the RF waveform can affect the energy of the electrons. Often industry mixes RF frequencies to alter the plasma -- particularly the ion energy distribution at the substrate. Here we add a second frequency in a systematic manner and examine the changes in the instantaneous EEDF. We also examine the turn on and turn off times of the RF generator itself.

Specialized circuits were designed for this work to ensure high frequency fidelity so digitization at 1.5 GHz is possible and accurate. A set of experiments were conducted to show how only altering circuit parameters affect the results, and steps were taken to eliminate those effects. Spatial variations of the resulting EEDFs were investigated, especially near the edge of the CCP reactor, to see which aspects change the most with radius.

4:20pm **PS+SE-MoA-9 Mass Spectral Characterization and Control of Plasma Etch Processes**, *L. Shoer, P. Heil, S. Pursel*, Intel Corporation; *N. Salovich*, Edwards Vacuum; *David Shykind*, Intel Corporation

As semiconductor critical dimensions have reached the single-digit nanometer scale, reproducible control of etch processes has become critically dependent on consistent wafer-to-wafer processing. Nanometer feature sizes and atomically thin layers have led to a regime where traditional bulk plasma characterization techniques no longer give insight into the chemical processes occurring on the wafer. Furthermore, the number of moles of reactants on the walls of an etch chamber are greater

than or equal to the quantity of reactants intended to be etched on a wafer itself. Uncontrolled, this situation complicates etch processes, introducing hysteretic behavior even assuming an ideal input stream of identical wafers, and exacerbates actual wafer-to-wafer variation effects. We show how high-speed (subsecond time resolution), non-invasive mass spectrometry of plasma cleaning, seasoning and actual etch steps themselves leads to improved performance and enhanced mechanistic understanding of plasma etch processes.

4:40pm **PS+SE-MoA-10 Development of a Catalytic Probe for the Detection of Fluorine Radicals with Applications to Semiconductor Manufacturing**, *Nicholas Connolly, J. Mettler, R. Garza, R. Sankaran, D. Ruzic*, University of Illinois Urbana-Champaign

Plasma processing is an essential part of integrated circuit manufacturing, with plasma etching, plasma strip, and chamber cleaning being three critical steps. All of these steps rely on radicals, highly reactive neutral species created in the plasma, to drive the desired etching reactions. Because of the importance of radical species in etching reactions and rates, quantification of the densities of these species is important for understanding plasma etching dynamics. Additionally, spatial resolution of radical densities allows specific knowledge of etch dynamics at a substrate or a chamber component of interest.

One technique that has been developed to detect and quantify radical species is a catalytic probe, which consists of two thermocouples each coated with a different metal. The different metals catalyze the recombination of radical species at different rates, leading to a temperature difference between the thermocouples. This temperature difference is proportional to the density of radical species, and so a radical density can be determined. The catalytic probe technique provides in-situ, spatially resolved radical densities. This has advantages over techniques which gather a line-averaged signal, such as optical emission spectroscopy (OES), and measurement methods that require ex-situ analysis, such as coupon etch rates.

Previous studies have applied catalytic probes to the detection of hydrogen (H), oxygen (O), and nitrogen (N) radicals.<sup>1</sup> To our knowledge, a catalytic probe for fluorine (F) has yet to be reported. Here, we present a thermocouple-based catalytic probe to determine spatially resolved fluorine radical densities in SF<sub>6</sub>/Ar plasmas. The catalytic activity of zinc, copper, and gold is reported. The radical densities determined from the radical probes are compared to those determined via actinometry and coupon etch rates. These methods also provide verification of the recombination coefficient of the probe material and thereby confirm the quantitative results of the radical probe.

[1] D. Qerimi, I. Shchelkanov, G. Panici, A. Jain, J. Wagner, and D.N. Ruzic. *J. Vac. Sci. Technol. A* **39**, 023003 (2021).

5:00pm **PS+SE-MoA-11 Multi-Diagnostic Investigation of Etching Plasma Species in an Industry-Grade Inductively-Coupled Plasma Etcher**, *Jeremy Mettler<sup>1</sup>, N. Connolly, S. Dubowsky, D. Ruzic*, University of Illinois at Urbana-Champaign

Plasma etching kinetics and reaction mechanisms often involve complex interactions between radical, neutral, and charged species. Optimization of etch rate and selectivity for a given process can be tedious without a detailed mechanistic understanding of the etching mechanisms, which in turn can be difficult to determine without accurate measurements of all relevant plasma species. Many diagnostics exist which are able to measure some of these species, but each has their own tradeoffs, and none are able to measure all species under all conditions.

In this work we discuss the development of a suite of plasma diagnostics for measuring the environment in an etching system, including neutral, charged, and radical species. To accurately measure each component of the etch process, results from appearance energy mass spectroscopy, optical emission spectroscopy, fluorine radical probe analysis, and Langmuir probe analysis are combined, with overlap in the sensing capabilities of each diagnostic used for cross-validation. The use of multiple independent diagnostics with different spatial resolutions and species sensitivities provides flexibility and increased confidence in quantitative results. This work will present a comparison of results obtained by the individual diagnostics across several CF<sub>4</sub> based etching conditions in an industry-grade inductively-coupled plasma etching tool. Further comparison will be made between experimental etching results and 0-D plasma modeling of the etching system.

<sup>1</sup> PSTD Coburn & Winters Student Award Finalist

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