Plasma Science and Technology
Room 124 - Session PS-ThM

Plasma Sources, Diagnostics and Control I

Moderators: John Arnold, IBM Research Division, Albany, NY, **Michael Gordon**, University of California at Santa Barbara

8:00am **PS-ThM-1 Radio-frequency Hollow Cathode Discharge: Modeling and Experimental Diagnostics***, K. Bera,* Applied Materials, Inc.*; H. Luo,* Applied Materials, Inc., Canada*; X. Shi,* Applied Materials, Inc.*; I. Korolov,* Ruhr Universität Bochum, Germany*; **Abhishek Verma**, S. Rauf,* Applied Materials, Inc.*; J. Guttmann, J. Schulze,* Ruhr Universität Bochum, Germany

Low to intermediate pressure radio-frequency (RF) hollow cathode discharges (HCDs) have gained significance for advanced plasma processing in the semiconductor industry. HCDs form in the cavities in the cathode that is separated from the anode by a dielectric. In the HCD, RF sheath heating as well as secondary electron acceleration can lead to plasma production. Complementary modeling and experimental studies are performed for slotted hollow cathode discharge to validate plasma model as well as to elucidate discharge physics behavior at various operating conditions in several designs. The design parameters include the slot width (10 - 20 mm), angle (0 - 10°) and shape (linear and curvilinear), while the process parameters are pressure (2.5 – 100 Pa), RF voltage (200 – 500 V) and gas mixture. Electropositive Ar, electronegative O_2, and their mixtures are considered. For the experiments, slot structures are constructed in the cathode, and Phase Resolved Optical Emission Spectroscopy (PROES) is performed for Ar I emission. From the emission spectrum, spatio-temporal distribution of excitation rate is obtained. A hairpin probe is utilized to diagnose plasma density in the transverse direction. For plasma modeling, a 2-dimensional planar computational domain in transverse plane to the slot is considered. At low-pressure (tens of mTorr), where kinetic effects are important, particle-in-cell Monte Carlo collision (PIC-MCC) modeling scheme is used.The PIC-MCC model includes evolution of charge densities from charged particles and electrostatic field and charged particle collisions with each other and with neutral fluid using a Monte Carlo model. At intermediate pressure (several hundreds of mTorr), due to high collisionality, the PIC-MCC modeling becomes computationally prohibitive. Therefore, hybrid-fluid plasma model is used that includes fluid plasma equations for charged and neutral species, and a Monte Carlo model for secondary electrons, coupled with Poisson's equation for self-consistent electrostatic plasma simulation. At low pressure the behavior of excitation rate using PIC-MCC simulation matches reasonably well with experimental data. However, excitation rate distribution at moderate pressure using the hybrid-fluid model has some discrepancies. The plasma density profiles from PIC-MCC and fluid simulations are consistent with the experiments. Based on our analysis, the hybrid-fluid plasma model is being improved to better describe RF hollow cathode characteristics.

8:15am **PS-ThM-2 Advancing In situ Transmission Electron Microscopy to Study Plasma-Nanomaterials Interactions***, **Jae Hyun Nam**,* University of Minnesota, USA*; D. Alsem,* Hummingbird Scientific*; P. Bruggeman,* University of Minnesota, USA

Non-thermal plasma (NTP) has been increasingly recognized as a promising alternative to conventional technologies in diverse fields due to its high reactivity and non-equilibrium characteristics. These properties make NTP particularly effective for material interactions, such as materials synthesis, etching, deposition, and chemical redox processes. Although NTP is extensively employed for the synthesis and functionalization of nanomaterials, the direct observation and mechanistic understanding of these interactions have been limited due to the significant technical challenges associated with the development of *in situ* diagnostic capabilities.

Significant advances have been made in *operando* environmental Transmission Electron Microscopy (TEM) enabling the monitoring of nanoparticle growth in solutions in an environmental TEM configuration. Despite these advancements, applications of *in situ* TEM in the NTP field have been sparse, primarily due to the challenges of integrating the necessary vacuum conditions and microscale requirements of TEM with the gaseous environment and high-voltage requirements of NTP. A decade ago, an integration of microplasma within a TEM was performed by demonstrating gold sputtering by argon plasma, albeit with a spatial resolution restricted to approximately 100 nm. Our recent new developments have successfully overcome these limitations, establishing an *in-situ* plasma TEM capability with a spatial resolution of less than 1 nm. This was achieved through the introduction of micron-sized electrodes in a gas environmental TEM cell enabling atmospheric micro-plasmas directly inside an electron microscope. The new device allows us to sustain atmospheric plasma steadily for several hours.

We demonstrate our *in situ* TEM capability through the real-time observation of morphological changes in iron oxide nanoparticles (magnetite, Fe_3O_4), likely induced by reduction processes, under DC hydrogen microplasma (He/H_2, 0.5 %) exposure with a temporal resolution of 1 s. This novel technique not only deepens our understanding of plasma and material interactions on a small scale, but also significantly expands the potential for *in situ* TEM studies in the NTP field.

Acknowledgement: This material was also based upon work supported by the Army Research Office accomplished under Grants No. W911NF-20-1-0322 and W911NF-20-1-0105.

8:30am **PS-ThM-3 Sensing and Control of Radio-Frequency Driven Plasmas***, **Timo Gans**,* Dublin City University, Ireland **INVITED**

Sensing and control of radio-frequency driven plasmas is crucial for next-generation plasma manufacturing. Surface interactions and negative ions play key roles in the properties of reactive molecular processing plasmas. Radio-frequency driven oxygen and hydrogen plasmas are ideal test-beds for investigations into the role of surface interactions for the chemical kinetics of the plasma and associated negative ion formation. Oxygen containing plasmas can exhibit either mostly electro-negative or mostly electro-positive characters [1, 2]. Oxygen negative ions can be efficiently destroyed by singlet oxygen which in turn is itself strongly influenced by surface reactions and surface properties [3]. Atomic oxygen, as a key reactive species, can also be dependent on surface properties [4]. In hydrogen plasmas, negative ions can be produced through surface processes as well as volume processes [5]. Nitrogen doped diamond surfaces are promising candidates for enhanced surface production of negative hydrogen ions [6,7,8]. Volume processes are determined by dissociative attachment involving vibrationally exited hydrogen molecules. These in turn are also dependent on surface properties [5]. This interplay between surface properties and the plasma chemical kinetics as well as plasma dynamics will be discussed for the examples of single [1, 3, 4, 5, 6, 7, 11] and multi-frequency [2, 9, 10] driven oxygen and hydrogen plasmas.

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9:00am **PS-ThM-5 Dorothy M. and Earl S. Hoffman Scholarship Awardee Talk: Time Resolved Diagnostics of HiPIMS Discharges With Positive Cathode Reversal***, **Zachary Jeckell**[1],* University of Illinois at Urbana Champaign*; T. Choi, N. Connolly,* University of Illinois Urbana-Champaign*; S. Das,* University of Illinois at Urbana Champaign, India*; M. Hossain,* University of Illinois Urbana-Champaign, Bangladesh*; D. Kapelyan, N. Vishnoi, R. Pickering,* University of Illinois at Urbana Champaign*; D. Qerimi,* University of Illinois Urbana-Champaign*; D. Ruzic,* University of Illinois at Urbana-Champaign

This work investigates the temporal evolution of a high-power impulse magnetron sputtering (HiPIMS), with a positive cathode reversal, discharge by using a number of different diagnostics such as gated fast cameras, time resolved Langmuir probes, and time and energy resolved mass spectrums acquired using the plasma sampling mass spectrometer (PSM). Using time resolved Langmuir probes we have studied that the rise time of the plasma potential is 1-2 µs which correlates to the time scale over which the ion energy distribution function (IEDF) is seen to increase. This, paired with PSM data collected where the magnetron and PSM are 90 degrees off axis

[1] **AVS National Student Awardee**

from each other still showing an increase in the Ag^+ energy suggests that the plasma potential commuting throughout the volume is one of the primary causes of increased ion energy after the positive cathode reversal. The Langmuir probe setup also allows for the creation of time resolved electron energy distribution functions (EEDF) both near the substrate and near the target which offers valuable insight into the discharge physics. Using the PSM we have shown that at early stages of the positive cathode reversal there is an elevated population of metal ions and that the overall fraction of metal to working gas fraction is at its highest. Time and energy resolved mass spec data was collected on this system for a variety of conditions such as pulse lengths, pressures, and target material with the objective of developing a better understanding for the energetics at play. Additionally, it was discovered that for the reactive sputtering of TiN that the IEDF of the N^+ species has a high energy tail of 40 eV at a pressure of 4 mTorr. This distribution mirrors that of Ti^+which indicates that the target poisoning is responsible for an increase in the energy of the nitrogen species and that target poisoning could be the source of nitrogen in the film. Gated fast camera measurements taken with the PI-MAX 4 camera have been performed to investigate the formation of hot spots and observed that the formation of hot spots occurs above the critical current density for silver. Fast camera measurements are also taken on and off axis to visualize the 3-D structure of spokes, and to visualize how the discharge changes when the positive cathode reversal occurs. Additionally, it was found that for cases with current four times higher than the critical current threshold that the hot spots seem to disappear, however, coupling PSM measurements with the fast camera we can see linear correlation between discharge current and counts of Ag^{+2} which suggests that the hot spots have disappeared due to rarefaction of the working gas.

9:15am **PS-ThM-6 Understanding Plasma Surface Dynamics Through Time Resolved Ion Energy Analysis for Deposition/Etch Processes***, **Angus McCarter,*** Impedans Ltd., Ireland*; A. Verma,* Impedans Ltd., India

As semiconductor critical dimensions are decreasing rapidly, reproducible control of etching and deposition processes has become crucial. These processes are heavily dependent on the chemical/physical processes occurring on the wafer surface. Like any other surface in contact with plasma, a sheath usually develops on the wafer which pulls down the ions out of bulk plasma necessary to complete the process on the wafer. Furthermore, external RF/DC/tailored waveform biases are applied to the wafer to modify the ion behaviors as it can affect the chemical composition, microstructure and the associated electrical properties of the thin films during plasma assisted deposition processes as well as the selectivity and anisotropy of high aspect ratio trenches in etching processes. Therefore, the characterization of only bulk plasma is not sufficient in providing insights necessary to understand the plasma surface interactions. A high-speed monitoring of the ion energy distribution function and ion flux can lead to enhanced understanding of the plasma surface interactions and improved process performance.

We will highlight the successful measurements done by the *Semion* RFEA diagnostic under different chamber and bias conditions. Such applications enabling accurate and precise control of etching profiles on different materials and various plasma chemistries. The *Semion* measures the ion energies hitting a surface, the ion flux, negative ions and bias voltage at any position inside a plasma chamber using an array of integrated sensors. On the other hand, the *Semion pDC system* measures these parameters in real time over an energy range up to 2000 eV (process dependent). It can do sub-microsecond time resolved measurements, for studying pulsed ICPs, or pulsed DC biases, as well as floating and grounded substrate conditions. The Semion Pulsed DC system is the key instrument used to measure the temporal evolution of the ion energy and flux at different times through the pulse period of a pulsed DC plasma process.These measurements are essential for establishing the correlation between the plasma inputs and the ion energy/flux which, in-turn, determines the effectiveness of the surface treatment.

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9:30am **PS-ThM-7 The Plasma Dynamics of Dual Frequency Capacitively Coupled Argon Discharge using Tailored Voltage Waveforms***, **Syed M Zulqarnain**, A. Lietz,* North Carolina State University*; J. Prager, T. Ziemba, J. Perry, P. Melnik,* EHT Semi

Ion energy and flux represent the primary factors of significance for etching and deposition plasmas in the semiconductor industry. The independent control over ion energy and flux to some degree can be achieved by harnessing the dual-frequency capacitively coupled plasmas. Tailoring the voltage waveform shape beyond simple sinusoids has unlocked a regime where ion energy distributions can be controlled. In this work, we simulate a dual-frequency capacitively coupled argon discharge employing a high-frequency (60 MHz) sinusoidal voltage waveform at the upper electrode and low-frequency (400 kHz) tailored triangular-shaped waveform at the lower electrode using a Monte-Carlo collision-based, particle-in-cell simulation (EDIPIC[1]). This exploration entailed a comprehensive assessment of plasma dynamics in response to alterations in the shape (including peak width and slope of the negative and positive regions) of the tailored voltage waveform. The temporal control of ion energies, synchronized with the shape of the tailored voltage waveform, was observed, offering potential benefits for regulating the chemistry during processing. The ion energies exhibited a correlation with the cumulative effects of the two applied waveforms. The low-frequency waveform dominated ion acceleration and temporal variations of ion energies. However, the energetic ions were slightly influenced by the high-frequency component leading to the emergence of two energetic peaks. The most power was delivered to the ions primarily during the negative cycle, especially after the positive pulse ended, while the electrons gained power mostly in the sheath edge with most power delivered during the positive pulse. The rate of rise of the positive portion of the waveform had minimal impact on ion distribution, but it did result in an increase in the number of electrons (of energies between 20-40 eV) reaching the lower electrode. This outcome could potentially aid in addressing charging concerns in the process of semiconductor etching of high aspect ratio features.

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9:45am **PS-ThM-8 A Real-Time 2-D Gas Temperature Monitoring Sensor Based on Molecular Emission Spectroscopy***, D. Patel,* University of Illinois at Urbana-Champaign*; D. Jacobson,* Lyten*; **Dren Qerimi,*** University of Illinois at Urbana-Champaign*; M. Stowell,* Lyten*; D. Ruzic,* University of Illinois at Urbana-Champaign

Recent trends have indicated a growth in the usage of thermal and non-thermal plasmas for material processing applications. In these processes, gas temperature is a critical thermodynamic state variable, but sensors suitable for monitoring its fluctuations in plasmas are few and often not applicable. The most straightforward method is to estimate the gas temperature based on the rotational temperatures obtained from molecular transitions in plasma. Not only does this require access to a spectrometer, but analysis can often be time-consuming. This study aims to address this by designing and testing a stand-alone 2-D optical sensor capable of simultaneous *in-operando* measurements of rotational and vibrational temperatures.

The proposed optical sensor monitors the emission intensity of specific rovibronic transitions of the Δv = 0 and +1 sequences of C_2 swan system. The light collected from a point source is split into three different color channels using beam-splitters and carefully selected bandpass filters. The net intensity of light from each channel is monitored with photodiodes/CCD sensors. The rotational and vibrational temperatures are inferred by comparing relative intensity of light collected from each channel to those predicted by simulations. The simulations used in this work assume that the rotational and vibrational states follow Boltzmann statistics to minimize the number of channels required in this device. The applicability of this sensor is limited to cases which satisfy this assumption. As a result, it is necessary to test the feasibility of this sensor in various discharge conditions on a case-by-case basis. For the scope of this work, we limit ourselves surface wave microwave discharges, operated at low and high pressures.

The accuracy in temperature measurements is evaluated by comparing them to those obtained using the well-known Boltzmann analysis technique. A high-resolution Czerny-Turner spectrometer is employed to obtain well-resolved ro-vibronic transitions of the Δv= +1 sequence. Boltzmann analysis of these transitions is not only used to estimate the rotational and vibrational temperatures, but also monitor any departure from expected density distributions. In addition, rotational temperatures

are also compared to gas temperature measurements obtained from the doppler broadening of the H_β line. Combined these results will demonstrate the viability of this sensor for its usage as a gas temperature monitoring device.

11:00am **PS-ThM-13 Fiber PROES: Phase-Resolved Optical Emission Spectroscopy by Time-Correlated Photon Counting Through Optical Fibers***, **Florian Beckfeld**, I. Korolov,* Ruhr University Bochum, Germany*; M. Höfner,* SENTECH Instruments GmbH, Germany*; J. Schulze,* Ruhr University Bochum, Germany

Phase-resolved optical emission spectroscopy (PROES) is a powerful, non-intrusive plasma diagnostic tool that can give useful insight into plasma parameters like the electron density or surface coefficients by visualizing the dynamic of the highly energetic electrons in the plasma. Furthermore, PROES may also be used to calculate the energy distribution function of these highly energetic electrons by applying a rate equation model. Despite these benefits, PROES has not been able to make a transition into the industry, as it requires optical access to wide regions of the discharge, which is not given in industrial plasma chambers. Additionally, the intensified charged-coupled device (ICCD) cameras commonly used for the diagnostic require expensive investments. To address these issues, we propose a technique for PROES based on photon counting. This brings several benefits: photon counting can be realized with a photomultiplier tube (PMT), which has fractions of the cost of an ICCD camera. This PMT can then be connected to the plasma chamber with an optical fiber, making the diagnostic easy to integrate into existing structures. In this work, measurements of the heating mode in a geometrically symmetric capacitively coupled plasma will be shown to demonstrate the capability of the approach to PROES with a PMT compared to measurements with an ICCD camera.

11:15am **PS-ThM-14 Atomic Hydrogen Density, Electron Density and Ion Flux Energy Distribution of an Ar/H2 Remote CCP Plasma Source for Atomic Scale Processing***, M. van Gurp, **Thomas van den Biggelaar**, J. Wubs, A. Salden, H. Knoops, E. Kessels,* Eindhoven University of Technology, Netherlands

A remote CCP source (Oxford Instruments PlasmaPro ASP) has been developed for higher throughputs for atomic layer deposition (ALD) processes, by generating high radical fluxes combined with controlled ion bombardment. In a previous study [1], it has been shown that this plasma source is well suited for Al_2O_3 deposition on GaN when operated with Ar/O_2 plasmas. This work uses a testbed setup containing the remote CCP source. The setup has optical access that allows extensive studies on plasma-surface interaction (PSI), relevant for ALD, atomic layer etching (ALE) and 2D material modification. Ar/H_2 plasmas can generate hydrogen atoms that, combined with ions, could modify materials synergistically. In this study, an Ar/H_2 (95%/5%) plasma is characterized by three diagnostics as a starting point for PSI studies. First, two-photon absorption laser induced fluorescence (TALIF) has been employed to measure the absolute atomic hydrogen density as a function of distance to the substrate surface for different pressures (38-375 mTorr). In this pressure range, a constant dissociation degree of 10% has been found, with $n_H = 10^{12} - 10^{13}$ cm^{-3}. Second, a Langmuir probe has been used to spatially measure the electron density, which is in the order of 10^{10} cm^{-3}. Third, a retarding field energy analyzer (RFEA) probe has been employed to determine the ion flux energy distribution, ion energies and ion fluxes at the substrate table. The measured ion flux energy distribution has been found to be bimodal, with ion energies and fluxes that are dependent on pressure and power. Both the Langmuir probe and the RFEA probe show that the electron density at the substrate table rapidly drops at pressures greater than 263 mTorr. Since the hydrogen dissociation degree remains constant in this region, it is hypothesized that the plasma source becomes remote at pressures above 263 mTorr, i.e. the plasma becomes confined near the electrodes instead of filling the whole reactor. Given the outcomes, PSI for atomic scale processing will be further investigated specifically for hydrogen atoms and ions.

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11:30am **PS-ThM-15 Spatially Resolved Measurement of Fluorine Radical Density Using Reactive Radical Probes***, **Jeremy Mettler**,* University of Illinois at Urbana-Champaign*; T. Tohara,* Tokyo Electron*; D. Qerimi, D. Ruzic,* University of Illinois at Urbana-Champaign

Plasma etching kinetics and reaction mechanisms often involve complex interactions between radical and charged species. Characterization of these mechanisms requires accurate measurement of reactant concentrations, which is often achieved using spectroscopic techniques. While nonintrusive, these techniques have significant limitations in their spatial resolution and ability to produce absolute measurements. For complex plasma and reactor geometries, spatial distribution of radical species can play a key role in the observed etching characteristics.

In this work we discuss the development of etching based, nonequilibrium radical probes for measuring fluorine radical densities across industry relevant pressures, powers, and radical densities. Tungsten pellets are used as the active probe surface and to preserve probe lifetime a nonequilibrium analysis method was developed. The tungsten etching rate is determined in-situ by measuring the rate of energy deposition to the probe via temperature response. Fluorine radical density is determined from the etching rate, independently of ion exposure, due to the purely chemical nature of tungsten etching at high temperatures. Results from the radical probe are compared against actinometry in spatially uniform and nonuniform plasmas to validate the probe technique and highlight its spatial resolution. Fluorine density profiles are then characterized for SF_6, NF_3, and CF_4 plasmas to demonstrate the generality of the technique to multiple fluorine process gases. Measured fluorine densities ranged from 1.4×10^20 ± .2×10^20 (#/m^3) to 3.9×10^21 ± .6×10^21 (#/m^3).

11:45am **PS-ThM-16 The Applicability of a Microwave Resonant Probe to the Plasma Processing of Silicon Oxide***, D. White, G. Hassall, **James Ellis**,* Oxford Instruments Plasma Technology, UK

In-situ diagnostics for industrial scale plasma processing represents a sizeable challenge for multiple semiconductor hardware companies. Simple, robust, and inexpensive sensors are required both to understand process development challenges and for real-time monitoring of the plasma process; the advent of machine learning only enhances the importance of such sensor development. A microwave enhanced resonance probe with a spiral-shaped slot design, also known as a curling probe[1-3], has been used to measure relative electron densities in the edge region of a Plasma Pro 100 Oxford Instruments Plasma Technology reactor. Etch tests were run across a broad range of plasmas processing parameters centred around a CF_4/O_2 plasma chemistry to ascertain the potential impact of the curling probe on the process. It was confirmed through both global and localised etch rate measurements that the curling probe was not observed to have any impact on the wafer outcomes. The sensitivity of the curling probe enabled changes in the plasma conditions to be tracked in real time with failure modes clearly visible from the measured shift in the resonant frequency.

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[2] I. Liang, K. Nakamura, and H. Sugai, *Appl. Phys. Express*, **4**, 066101 (2011)

[3] F. Boni *et al., Rev. Sci. Instrum.*, **92**, 033507 (2021)

12:00pm **PS-ThM-17 Terahertz Absorption Spectroscopy for Measuring Atomic Oxygen Densities: A Comparison with ps-TALIF and CRDS***, **Jente Wubs**, U. Macherius, A. Nave,* Leibniz Institute for Plasma Science and Technology (INP), Germany*; L. Invernizzi, K. Gazeli, G. Lombardi,* Laboratoire des Sciences des Procédés et des Matériaux (LSPM), CNRS, France*; X. Lü, L. Schrottke,* Paul-Drude-Institut für Festkörperelektronik Leibniz-Institut im Forschungsverbund Berlin, Germany*; K. Weltmann, J. van Helden,* Leibniz Institute for Plasma Science and Technology (INP), Germany

Oxygen-containing plasmas are widely used in industry for a variety of applications, including etching, thin-film deposition, and other surface modification processes. Atomic oxygen is a key species in most of these applications. An accurate method for determining atomic oxygen densities is therefore of great importance, not only to gain a fundamental understanding of the plasma chemistry, but also to improve industrial processes. However, existing techniques, such as two-photon laser induced fluorescence (TALIF), vacuum ultraviolet (VUV) absorption spectroscopy, cavity ring-down spectroscopy (CRDS), and optical emission spectroscopy (OES), are all either bulky and expensive, experimentally challenging, or indirect and relying on a multitude of assumptions.

Terahertz (THz) absorption spectroscopy with quantum cascade lasers (QCLs) has recently been developed and implemented as a novel diagnostic technique for determining atomic oxygen densities. It is based on detecting the $^3P_1 \leftarrow {}^3P_2$ fine structure transition of ground-state atomic oxygen at approximately 4.75 THz (i.e. approximately 63 μm). THz absorption spectroscopy allows for direct measurements (i.e. no calibration is required) of absolute ground-state atomic oxygen densities, and its accuracy depends almost exclusively on the accuracy to which the line

strength of the transition is known. Furthermore, the narrow laser linewidth of QCLs makes it possible to determine the temperature from the detected absorption profiles as well. In addition, the experimental setup for THz absorption spectroscopy is relatively compact (especially compared to TALIF setups that typically involve bulky laser systems), vacuum conditions are not essential (as opposed to when working in the VUV), and the requirements for the optical alignment are not as strict as for CRDS. These features make THz absorption spectroscopy an attractive alternative to existing diagnostic techniques.

To confirm the accuracy of THz absorption spectroscopy, we performed picosecond TALIF (ps-TALIF) and CRDS measurements of atomic oxygen densities on the same capacitively coupled radio frequency (CCRF) oxygen discharge, for a variation of the applied power (20 to 100 W) and the gas pressure (0.7 and 1.3 mbar). The obtained atomic oxygen densities (all of the order of 10^{14} cm^{-3}) were found to be in excellent agreement, both qualitatively and quantitatively. This demonstrates that the three different diagnostic techniques all allow for accurate measurements and can be used interchangeably, provided that no spatial resolution is required.

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