

Thursday Afternoon, November 2, 2017

Plasma Science and Technology Division

Room: 22 - Session PS+VT-ThA

Plasma Diagnostics, Sensors and Control

Moderator: Aranka Derzsi, Wigner Research Centre for Physics, Hungarian Academy of Sciences, Hungary

2:20pm **PS+VT-ThA1 Quantitative Analysis of Composition and Temperature of Semiconductor Processing Plasmas via Terahertz Spectroscopy**, *Yaser Helal, C.F. Neese, F.C. De Lucia*, The Ohio State University, *A. Niabati, M. Johnson, B. Craver, P.J. Stout, M.D. Armacost*, Applied Materials, Inc.

Processing plasmas are at a similar pressure and temperature to the environment used to study atmospheric and astrophysical species in the terahertz (THz) spectral region. Many of the molecular neutrals, radicals, and ions present in processing plasmas have been studied in the laboratory and their absorption spectra have been cataloged or are in the literature for the purpose of astrophysical study. Recent developments in THz devices have made technology commercially available for applications outside of specialized laboratories. The methods developed over several decades in the THz spectral region for these laboratory studies are directly applicable to diagnostic measurements in the semiconductor manufacturing industry. In this work, a continuous wave, intensity calibrated THz absorption spectrometer was developed as a remote sensor of gas and plasma species. A major advantage of intensity calibrated rotational absorption spectroscopy is its ability to determine absolute concentrations and temperatures of molecular species from first principles without altering the plasma environment. An important part of this work was the design of the optical components which couple 500 – 750 GHz radiation through a commercial inductively coupled plasma (ICP) chamber. The measurement of transmission spectra was simultaneously fit for background and absorption signal. The measured absorption signal was used to calculate absolute densities and temperatures of polar species. Examples of measurements made in ICPs will be presented. Also, time resolved measurements were made and the time evolution of molecular densities will be discussed.

2:40pm **PS+VT-ThA2 In Situ Measurement of Electron Emission Yields from Plasma-Exposed Surfaces**, *Mark Sobolewski*, National Institute of Standards and Technology

Surfaces exposed to plasmas are bombarded by energetic particles which may induce electron emission. The emitted electrons may in turn influence the plasma. Accurate plasma simulations require knowledge of the flux or yields of emitted electrons. Yields can be measured directly in beam studies, but it is impractical to produce a beam of each possible energetic particle that could be produced by typical plasmas. In contrast, in-situ measurements, performed during plasma exposure, may provide useful values or bounds for effective or total electron emission yields, summed over all (or some subset) of the energetic particles present for given plasma conditions. Here, measurements were performed at 10 mTorr (1.3 Pa) in an inductively coupled plasma system equipped with an electrostatic shield and variable-frequency rf substrate bias. An insulating cap is placed on the rf-biased electrode to minimize edge effects. The cap also reduces the effective electrode size, which further limits any undesired effects of rf bias on the plasma and allows yield measurements on small substrates. The rf voltage and current across the sheath adjacent to the rf-biased electrode are measured and analyzed by detailed, numerical sheath models, which allow the current of electrons emitted from the surface to be distinguished from other mechanisms of current flow. The observed dependence on voltage and rf phase allows some discrimination between emission induced by energetic positive ions and that induced by photons and metastables. The technique is validated by comparing measurements made in argon discharges with literature results from beam studies and then is applied to plasma etching discharges in fluorocarbon gas mixtures.

3:00pm **PS+VT-ThA3 Studying Dynamic and Structured Plasma Systems Utilizing Laser-Collision Induced Fluorescence**, *Edward Barnat, A. Fierro*, Sandia National Laboratories

INVITED

Laser collision-induced fluorescence (LCIF) is a powerful diagnostic which can be used for making temporally and spatially resolved measurements of electron densities in a plasma discharge. The technique, which involves the measurement of optical emission emanating from higher energy excited states due to the redistribution of the lower energy laser-excited state by collisions with energetic plasma species, has been readily employed to study both helium and argon discharges. In this presentation, an overview of the fundamental principles and anticipated limitations of the LCIF method will be presented. Examples of the LCIF method applied to structured and

dynamic discharges generated in helium and argon will be presented to demonstrate the utility of this diagnostic technique. Finally, recent efforts used to extend the LCIF method to higher pressure (near atmospheric pressure) discharges will be discussed.

4:00pm **PS+VT-ThA6 Effect of Ion Inertia on Ion Energy Broadness on Biased Electrode in Dual Frequency Capacitively Coupled Argon Plasma**, *Yunchang Jang, H.-J. Roh, N.-K. Kim, S. Ryu, G.-H. Kim*, Seoul National University, Republic of Korea

Ion response time to RF sheath voltage is important to control the energy spread of ion energy distribution (IED) in the dual frequency capacitively coupled argon plasma. IED is known as being governed by the dynamics of ion in RF sheath and the magnitude of RF voltage peak. In previous study, semi-analytic models to determine IED were derived from concept of ion response time (τ_i). Ion energy broadness (ΔE_i) was represented in terms of the sheath voltage oscillation (V_{pp}) and τ_i/τ_{rf} . Ion response time was assumed as ion transit time across the sheath, τ_{ion} by adopting correction factor without thorough understanding. In this study, we investigate the underlying physics of the correction factor, consequently defining the ion response time τ_i with RF sheath voltage oscillation. Experiment were performed in dual frequency CCP at 20 mTorr of argon gas which has the ratio of maximum sheath size to ion mean free path ~ 2 . Various ranges of RF bias (from $\tau_{ion}/\tau_{rf} \sim 0.05$ to $\tau_{ion}/\tau_{rf} \sim 10$) were applied to bottom electrode to enhance the incident ion energy with very high frequency (VHF, $\tau_i/\tau_{rf} \sim 10$) applied on the top electrode (showerhead) to sustain plasma. A commercial retarding field analyzer (Impedans, Vertex V4.0.10) was employed to measure IED. Plasma density, electron temperature and plasma potential were measured by using RF compensated Langmuir probe. Experimental results of ΔE_i to V_{pp} were compared with models under assumptions that ion response time is ion transit time across the sheath (τ_{ion}) or one of ion plasma frequency ($1/\omega_{pi}$). Experiment results revealed that the time scale of ion response time is determined by $1/\omega_{pi}$ rather than τ_{ion} in this high-density plasma. This result implies that ion response time is governed by the ion inertia at the sheath boundary to RF sheath oscillation. Ion inertia becomes the initial condition of ion acceleration and govern the ion energy arriving at surfaces.

4:20pm **PS+VT-ThA7 Collision Frequency Estimation using Microwave Hairpin Resonator Probes**, *D. Peterson, Steven Shannon*, North Carolina State University

Microwave hairpin resonator probes have become convenient alternatives to Langmuir probes to measure electron density in low temperature plasmas. The impact of electron collisions with neutrals with regard to the analysis of the resonant frequency shift from which this density is determined has been well established.[1] In this work, a method for extracting the electron neutral collision frequency by measuring resonance broadening due to collisions is presented. By using both the resonance frequency and collision-broadened resonance width, the electron density and electron neutral collision frequency can be measured. Measurements are made in argon, oxygen, Ar/O₂, and helium plasmas sustained in an RF driven capacitive coupled parallel plate system operating in the 100's of mTorr to Torr range. Comparisons to calculated and modeled collision frequencies in single component background gases (Ar and He) are made; experimental results agree well with these conditions. Collision frequency measurements in more challenging regimes including molecular gases and gas mixes are also presented to demonstrate applicability across a broad range of pressures and gasses. Probe design, analysis methodology, and parametric trends in capacitive systems with regard to gas density, electron density, power density, and gas composition will be presented. This work is supported through a generous gift by Applied Materials Inc.

[1] *Plasma Sources Science and Technology* 16, no. 4 (2007): 716

4:40pm **PS+VT-ThA8 In-Situ Diagnostics of Processing Plasma and Semiconductor Films for High-Efficiency Silicon Hetero-Junction Solar Cells**, *Shota Nunomura*, National Institute of Advanced Industrial Science and Technology (AIST), Japan

INVITED

The plasma processing is a key technology for fabricating semiconductor devices such as solar cells, light-emitting diodes and transistors. In those devices, the semiconductor films are often prepared and/or post-processed by various plasma processes. During the processes, the films are exposed into the UV, radicals, and ions, and thereby the electronic property of the films is often degraded. So, the investigation of the plasma-material interaction is important for understanding the degradation mechanism and also for further developing the plasma processing technology.

Here, we show in-situ characterization of the electronic property of semiconductor films as well as the gas-phase plasma diagnostics during the plasma process. The process we diagnosed was PECVD of hydrogenated

amorphous silicon (a-Si:H) for the passivation of silicon heterojunction solar cells. The plasma parameters and gas-phase species, such as ions, radicals, and precursors are measured by using conventional techniques of Langmuir probe, quadrupole mass spectroscopy and optical emission spectroscopy [1-3]. Together with this gas-phase diagnostics, we measured the transport properties of the growing a-Si:H films such as carrier transport and trapping, by using a recently developed optical pump probe technique [4,5]. The optical property such as the bandgap, refractive index and extinction coefficient, was also characterized by real-time spectroscopic ellipsometry.

We found that transport property of the a-Si:H films was strongly limited by the defects generated during the PECVD process, and improved by post-deposition annealing process. The generated defects were distributed near the film surface; the defect rich surface layer was estimated to be less than approximately 10 nm. As for defect annihilation, the post deposition annealing was very efficient. The annealing temperature and period strongly influence the defect relaxation, inducing the improved carrier transport. The relation between the plasma process and transport property will be described in the presentation.

[1] S. Nunomura, I. Yoshida, and M. Kondo, *Appl. Phys. Lett.* **94**, 071502 (2009). [2] S. Nunomura and M. Kondo, *J. Appl. Phys.* **102**, 093306 (2007). [3] S. Nunomura, H. Katayama, I. Yoshida, *Plasma Sources Sci. Technol.* **26**, 055018 (2017). [4] S. Nunomura, I. Sakata, and M. Kondo, *Appl. Phys. Express* **6**, 126201 (2013). [5] S. Nunomura and I. Sakata, *AIP Advances* **4**, 097110 (2014).

5:20pm **PS+VT-ThA10 Towards *In Situ* Microwave Imaging in Plasmas**, A. Tselev, University of Aveiro, Portugal, *J. Fagan*, NIST, **Andrei Kolmakov**, CNST/NIST

There exists a great need for *in situ* nanoscale characterization of surface/interface morphologies during plasma treatments. These include plasma induced growth, surface modification, sputtering and other processes relevant to semiconductor and aerospace industries, environmental remediation and biomedical applications. To address these needs, the current approaches rely on either “post mortem” sample microscopy or *in situ* optical analytical methods. The latter, however, lack required nanoscale spatial resolution.

In this communication, we propose to use near-field microwave imaging known as scanning Microwave Impedance Microscopy (sMIM) to image processes in plasma. Different to optical microscopy, the sMIM is sensitive to variations of local permittivity and conductivity of the material under a scanning probe. We demonstrate applicability of the sMIM to monitor plasma-assisted processes with a submicron spatial resolution. In our approach, a plasma environment with an object of interest is separated from the sMIM probe and the rest of the microscope by a SiN membrane of a few-10s nm thickness, and the imaging is performed through this membrane. As a proof of concept, we were able to image carbon nanotube films drop-casted onto the SiN membranes and their transformations in the process of plasma-induced oxidation by a low-pressure air plasma. To the best of our knowledge this is the first report on application of an SPM for *in situ* imaging of plasma processing. The experiential limitations such as electromechanical and thermal stability of the membranes will be discussed.

5:40pm **PS+VT-ThA11 Probe System for Radical Species Characterization in Vacuum with Centimeter Spatial Resolution**, **Ivan Shchelkanov**, D. Qerimi, A. Hayes, J.T. Wegner, D.N. Ruzic, University of Illinois at Urbana-Champaign

Among plasma diagnostics one of the most difficult tasks is getting an estimate of radical gas species concentration in the ground state without plasma presence in the diagnosed volume. This is probably the major task for characterisation of downstream plasma composition in various areas of applied plasma technology and the requirements for characterisation tool are very strict. The ultimate device should have a good spatial resolution, relatively high response time, operate in highly reactive plasmas and in presence of sputtering flux materials, should be capable to characterize species of unknown geometrical distribution and composition.

The idea of a tool, which could satisfy most of the mentioned requirements, was proposed more than ten years ago [1] but only recently the Center for Plasma Materials Interaction was able to develop a complex system which can measure composition and density of radical species with 1 cm spatial-resolution and response time of 15 seconds in the presence of high intensity RF fields and flux of sputtered material. The system can measure density of oxygen, nitrogen, and hydrogen radicals, when different species present in the chamber at the same time. For vacuum chamber of 13 inch in diameter and 46 inches tall, which is equipped with 1 kW Helicon plasma source, the measured density at ~75 mTorr, 1kW power and 10 inch from the source, the density of radical species of hydrogen was $0.7 [\pm 0.5] * 10^{21} \text{ m}^{-3}$ and of nitrogen radicals it was $1.1 [\pm 0.7] * 10^{20} \text{ m}^{-3}$. Additional comparison with zero dimensional model showed a match within an errorbar between an experiment and the model.[2]

The principle of the radical species concentration measurement is the following. The thermocouple tip is coated with a particular catalytic metal. Once the probe is exposed to the gas atoms, recombination of gas atoms occurs on the surface of the probe tip. The catalytic surface provides efficient recombination thus more energy is delivered to the surface from the recombination reaction [3] compared to a probe tip without the catalytic surface. By measuring the temperature of the probe it becomes possible to quantify the amount of gas atoms in the probe vicinity. Different radical species can be distinguished by using catalytic surfaces particular to the species in question. Current work is focused on radical probe system capabilities, physical limitations, and examples of characterized plasmas.

References:

1. M. Mozetic / *Vacuum* **V.47 #6-8** pages 943 to 945 (1996)
2. D.T. Elg / *J. Micro/Nanolith. MEMS MOEMS*, **16**, 023501 (2017)
3. M. Mozetic / *Surface & Coatings Technology* **201 (2007)** 4837-484

6:00pm **PS+VT-ThA12 Spatiotemporal Evolution of RF Magnetic Field and Plasma Current in a Very High Frequency Plasma Source**, **Jianping Zhao**, P.L.G. Ventzek, B. Lane, C. Campbell, Tokyo Electron America, T. Iwao, K. Ishibashi, Tokyo Electron Limited

Large-area plasma processing systems capacitively driven at very high frequencies (VHF, e.g. 100MHz) have attracted much interest for semiconductor device and flat panel display processing. VHF has the advantage of generating plasma with more efficiency as power is coupled more into electrons and less into ions in the sheath. Benefits are seen for processes requiring reduced ion bombardment energy, ostensibly to minimize damage, high ion and radical flux to the substrate. The benefits of VHF are accompanied by challenges. The short wavelength associated with VHF source power is reduced even further in the presence of high density plasma. The wavelengths are comparable to the RF electrode dimension. High plasma densities can also lead to skin effects that screen the electromagnetic fields from parts of the plasma. As a result, spatial variations in plasma density and sheath voltage can arise and lead to undesired non-uniformities in process parameters such as etch or deposition rate. E to H transitions and plasma-sheath local resonances are other potential destroyers of plasma uniformity. Resonances and mode jumping can potentially prevent smooth plasma property control through adjustable process parameter changes. In order to understand these fundamental electromagnetic effects on VHF plasma non-uniformity to achieve a better design of plasma source, it is desired to have a detailed investigation on the spatial and temporal evolution of RF magnetic field and plasma current spanning a large RF power, pressure, and chemistry range. We present here a time and phase resolved measurements of the spatial structure of the electromagnetic waves in a 100MHz plasma source performed with a magnetic field probe (B-dot loop). The probe was translated across the diameter of the VHF plasma, measuring the magnitude and phase of the fundamental and harmonics of the plasma excitation frequency as a function of radial position. The measured magnetic fields displayed a transition from simple to complex behaviors depending on plasma conditions. The spatiotemporal resolved magnetic field exhibits a series of fast current reversal and subsequent circulation driven by inward wave propagation that are electromagnetic in nature. We show how the onset, frequency and amplitude of the current reversal and subsequent circulation are strongly related to applied plasma conditions (e.g., density, sheath thickness). We also show that plasma current derived from the magnetic field distribution is closely correlated to the plasma density profile measured by a plasma absorption probe.

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