Plasma Science and Technology Division
Room B131 - Session PS-ThM
Plasma Diagnostics and Sources II

Moderators: Geun Young Yeom, Sungkyunkwan University, Republic of Korea, Wei Tan, Applied Materials

8:00am **PS-ThM1 Measurement of Plasma Neutral Densities in a Very High Frequency Ar/NH$_3$ Plasma with a Line-of-sight Threshold Ionization Mass Spectrometry**, ***Jianping Zhao***, *P.L.G. Ventzek, C. Schlechte, M. Burtner,* Tokyo Electron America, Inc.*; D. Li, J.G. Ekerdt,* The University of Texas at Austin*; T. Iwao, K. Ishibashi,* Tokyo Electron Technology Solutions Limited, Japan

Atomic precision plasma processes for logic and memory fabrication are in increasing demand due to the shrink of critical dimensions to near physical limits and increase in stack complexity. Meeting dimensionality requirements is not enough. Infinite selectivity and damage-free process with sub-angstrom control are sought to deliver high quality films with productivity worthy yields. Plasma enhanced processes, particularly plasma enhanced atomic layer deposition (ALD) rely on plasma generated radicals for much of their perceived benefit. Furthermore, low energy ions are required to mitigate damage. 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. Depending on the film and process, benefits are reduced ion energy and high radical and ion fluxes. It remains the case that it is difficult to relate the role of the combination of species flux and energy exactly to film growth mechanisms or material properties. Ideally, both species flux and film properties would be measured simultaneously (and in real time) as the surface state from ALD is changing continuously. A significant challenge is that it is difficult to measure the absolute density of neutral species in industrially relevant plasmas reliably especially at high pressure. In order to understand the fundamental plasma chemistry property of VHF plasma, we present here the measurement of the neutral species with a line-of-sight threshold ionization mass spectrometry (LS-TIMS) technology in VHF Ar/NH$_3$ plasmas. Ar/NH$_3$ plasma has been widely used in plasma enhanced ALD and CVD. Systematic measurements were performed in a 100 MHz plasma source with a wide RF power, pressure, Ar to NH$_3$ flow ratio range. Plasma chemistry properties of Ar/NH$_3$ plasma are derived after a careful background subtraction and mass-to-charge ratio dependent sensitivity calibration. Density of NH$_3$ and various amine dissociation products are determined as a function of plasma discharge conditions. The LS-TIMS results are also compared to those from other optical based neutral diagnostics.

8:20am **PS-ThM2 Radical Probe System for In-Situ Measurements of Hydrogen, Oxygen and Nitrogen Radical Densities**, ***Dren Qerimi***, *G.A. Panici, A.J. Jain,* University of Illinois at Urbana-Champaign*; J.W. Wagner,* Colorado State University*; D.N. Ruzic,* University of Illinois at Urbana-Champaign

The current state-of-the-art methods to identify presence of radical species in vacuum chambers are optical methods, which suffer from the lack of spatial resolution and require expensive optical equipment. Center for Plasma Material Interactions (CPMI) at the University of Illinois developed a catalytic radical probe array to measure concentrations of reactive species in low temperature plasma with high spatial resolution. Radical probes as plasma diagnostic tool can be used to determine radical densities of hydrogen, nitrogen and oxygen in any continuous plasma source in vacuum environment. The basic principle and advantage of a probe array is the capability to distinguish between different gas species due to several sensitive elements acting as recombination catalysts [1]. The catalytic coatings cover an area of several square millimeters on the tip of a sheathed thermocouple. The catalytic probe surface provides efficient recombination of active species with subsequent energy release as a heat. All the probes are exposed to the same background plasma heating/cooling mechanisms, but the temperatures are not the same due to the fact that different catalytic materials have different recombination coefficients, therefore a temperature difference between probes is generated. The system consists of two additional probes, first to obtain the overall heat flux on probe array, and the second is a reference probe with surface chemically active to all gases.

Lifetime of radical probes, specifically catalytic surface degradation, depends highly on vacuum conditions, chamber contamination and the fact that radical species cause surface properties to change. Lifetime of radical probe system is usually three hours. However, probe surface cleaning has been achieved by applying a bias which causes contamination layer on the probe tip to sputter via ion bombardment. Argon gas is used to sputter clean probe tip. Additionally, if probe shows signs of high contamination then a long sputtering process is used to remove all the catalytic material from the probe tip and then magnetron sputtering is used to redeposit new catalytic coating. The array of several probes is capable to distinguish between different gas species with sub centimeter spatial resolution. The probes give accurate results in a broad range of reactive species concentrations from about 10^{12} to 10^{14} cm^{-3}.

Reference:

[1] M Mozetic, M Kveder, M Drobnic, A Paulin, and A Zalar. Determination of atomic

hydrogen density with catalytic probes. Vacuum, 45(10-11):1095-1097, 1994.

8:40am **PS-ThM3 Post Charge Separation Grid Ion Flux Evaluation in Inductive Coupled Plasma Source Downstream Asher**, ***Luke Zhang***, *S. Ma,* Mattson Technology, Inc.

With semiconductor device shrink and gate dielectric thickness decrease, the potential for device damage at the photoresist strip level increase. It is desired to develop the downstream plasma asher producing high active radical concentration with low ion concentration while still maintaining excellent ash rate for different strip application. Therefore, it is critical to understand the population of charge species that can reach the wafer surface. In this study, an inductive coupled plasma (ICP) source with patented grounded Faraday shields is used [1], which offers superior resist strip capability to leading edge memory, logic and foundry applications. Faraday shield is used to reduce ion energy and electron temperature from plasma generation to obtain the better plasma damage performance. To further reduce the ion concentration on the wafer surface, charge separation conductive grid [2] is also added between the top plasma source and heated pedestal. By optimizing grid pattern, uniform gas and radical distribution can be obtained, thus the wafer uniformity can be improved. Ion flux underneath the grid are evaluated with different diagnostic tools to evaluate the grids effect including Langmuir probe and Retarding Field Energy Analyzer. The Langmuir probe with plasma detect limit 10^8 cm-3 is inserted plasma at 1cm above the pedestal is used to detect the ion flux underneath the grids. RFEA (Retarding Field Energy Analyzer) is equipped on the pedestal, which measures ion energy and ion flux directly. Both the diagnostics tools show that ion density is below the detection limit after charge separation grid. To further characterize the grid effect, one self-made thick probe with length 10mm and diameter 3.8mm inserted to the plasma with biased at negative voltage to measure ion saturation current, Pico amp accuracy ammeter is used to measure the collected current. It is found that grid dramatically reduce the ion saturation current, one thousandth of ion saturation current at Oxygen plasma detected under double grids compared to no grid condition. Different plasma chemistry and different grid are also evaluated.

[1] Stephen E. Savas, Brad S. Mattson, Martin L. Hammond, Steven C. Selbrede, Patent US 6143129

[2] Stephen E. Savas, Brad S. Mattson, Patent US 5811022

9:00am **PS-ThM4 Development of a Novel Langmuir Probe for the Investigation of Dusty Non-thermal Plasmas**, ***Austin Woodard***[1], *L. Mangolini, K. Shojaei, C. Berrospe,* University of California, Riverside

Dusty plasmas are characterized as plasmas containing micro- to nano-sized particles. Probing the plasma physics inherent in these systems is a daunting, but critical, task necessary for the engineering design and optimization of many common-place industrial manufacturing processes utilizing plasma, such as thin film etching and fabrication. We present the development of a test-bed for the characterization of dusty plasmas via a simple Langmuir probe. This diagnostic tool allows for the precise determination of the electron energy distribution function (EEDF) and subsequent plasma parameters but is notoriously difficult to use in dust-forming chemistries due to the inevitability of an insulating coating. To combat this, we have designed a two-stage reactor scheme that overcomes this limitation. In the first plasma reactor, the particle production cell, we synthesize graphitic carbon nanoparticles from the complete dissociation

[1] **Coburn & Winters Student Award Finalist**

of acetylene, confirmed by a residual gas analyzer, which are then directly injected into the primary chamber volume. The quality of the measurement is minimally affected by the presence of a graphitic nanoparticle coating on the probe tip due to its high electrical conductivity, thus creating a more forgiving environment in which to employ this technique. Additionally, the approach has the advantage of decoupling the nucleation and growth-phase kinetics of the nanoparticles from the primary chamber discharge thus allowing us to study the plasma properties when varying processing parameters such as primary plasma power and chamber pressure. Due to the particle trapping induced in the primary plasma, a continues wave laser (532 nm) was used to investigate the actual particle density in the primary chamber volume, and from this, the average charge per particle. The analysis of the EEDF as a function of the plasma parameters highlights the onset of unexpected trends in plasma the properties which are not predicted by traditional OML theory; we observe secondary peaks in the EEDF that change with the processing parameters, indicating not an electronic transition, but a phenomenon directly related to the presence of dust. To investigate this theoretically, we performed simulations with a Boltzmann-solver modified to account for the effect of secondary electron emissions. These simulations also exhibited a secondary peak, at the same energy levels observed experimentally; thus, we tentatively attribute this observation to secondary emission processes directly tied to the floating potential of the particles.

9:20am **PS-ThM5 Historical Review of Microwave Plasma Diagnostics using Plasma Cutoff Phenomenon***, **Shin-Jae You**, S.J. Kim,* Chungnam National University, Republic of Korea*; Dw. Kim,* KIMM, Republic of Korea **INVITED**

Though this paper, we present historial review of our cutoff probe research which has been performed for almost 2 decade. This paper focus on the whole progress for the cutoff probe including how to start to develop the cutoff probe in the initial period, what idea has been included during the development, how to evolve the probe during 17 years. The cutoff probe is most simple diagnostics among the plasma diagnostics tools which was made by simple intuition for the cutoff phenomenon of the plasma wave. However, the cutoff probe has been used for a long time without test of validation of probe itself. Later, EM waver simulation supported the validation for the cutoff frequency determination. Recently, by supposing the circuit modeling, the physics behind for the cut off probe spectrum (S21) was revealed and the accuracy and the application window of the probe were established. Very recently, as an extended version of the circuit model, we makes transmission line modeling to explain the cutoff spectrum in high density plasma as well as low density plasma.

Based on recent developments we also introduce a novel methodology to interpret the probe spectrum that eliminates the sheath and collisional effects and enables the use of this precise diagnostic technique in a broad range of practical processing conditions.

11:00am **PS-ThM10 Characterization of Inductive Coupled Plasma Source RF Power Pulsing for Advanced Surface Treatment Applications***, **Shawming Ma**, L. Zhang, D. Kohl,* Mattson Technology, Inc.

As device dimensions continue to shrink into the sub-10 nm regime, low electron temperature plasma and radicle energy control become very important factors in the fabrication of microelectronics device. A pulsing plasma reactor [1] is an efficient way to bring down the electron temperature and improve the process window by adding an additional tuning knob. Pulsed plasmas have been widely used in plasma etch tools, as high-density plasmas at low pressure demonstrate excellent plasma charge damage reduction, improved microloading and reduced mask erosion compared to traditional etchers. However, no results have thus far been reported for a high pressure, downstream pulsed plasma reactor for surface treatment and plasma ashing. For advanced surface treatment applications, radical energy control becomes necessary to control either selectivity to underlying films during resist ashing, surface film growth or surface property change. Therefore, it is desired to have radical energy control capability to improve the process window.

In this work, we explore pulsing plasma in a high pressure, downstream, grounded Faraday Shielded ICP source[2]. Source RF power of 13.56MHz frequency with pulsing frequency up to 100kHz and a vacuum capacitor automatch were used in the experiment. The pulsing window was mapped with maximum pulsing frequency 30KHz and duty cycle from 10% to 90%. A Langmuir probe is used to measure the electron energy distribution function (EEDF) developed by Plasma Sensor [3], which is inserted in the reactor 1cm above the wafer pedestal. Due to the limitations of plasma density measurement, (Langmuir probe requires plasma density above 10^8 cm-3 detection limit) the grid [4] which is used to separate high density and low density plasma, is removed from the reactor to make the measurement possible. Pulsed plasma program performs time resolved measurements of the probe V-I, the plasma parameters and EEDF. Both electropositive (e.g. Ar) and electronegative (O2/N2) plasma are used to study the pulsing plasma. The plasma impedance for the different plasma are read from the matching networks internal I-V probe. Electron temperature, density and afterglow temporal evolution at different pulsing conditions are also discussed.

[1] Pulsed plasma etching for semiconductor manufacturing, Demetre J Economou, J. Phys. D Appl. Phy. 47(2014)

[2] Stephen E. Savas, Brad S. Mattson, Martin L. Hammond, Steven C. Selbrede, Patent US 6143129

[3] Comparative analyses of plasma diagnostics techniques, V. A. Godyak and B. M. Alexandrovich, Journal of Applied Physics 118, 23302 (2015)

[4] Stephen E. Savas, Brad S. Mattson, Patent US 5811022

11:20am **PS-ThM11 In-situ Measurement of Deposited Tilm Thickness and Electron Density by Double Curling Probe***, **Daisuke Ogawa**,* Chubu University, Japan*; Y. Sakiyama,* Lam Research Corporation*; K. Nakamura,* Chubu University, Japan*; H. Sugai,* Nagoya Industrial Science Research Institute, Japan

We developed a technique for measuring the thickness of a deposited film and electron density of a processing plasma simultaneously during a process with the use of two curling probes (CPs). As might be already known, CP is one of the microwave resonators which has a spiral-shaped slot antenna to make resonance. The resonance frequency (RF) depends on the geometry of the probe (the length and diameter of the antenna) and material of the antenna cover, etc., and the RF shifts to a higher frequency as electron density increases. Also, our recent research showed that CP can find the thickness of the film deposited after a plasma processing through the observed shift of RF when a dielectric constant of the film is known. In a deposition process operated in semiconductor industries, a film deposits not only on the processing substrate but also on a CP. The deposition shifts the RF lower, but the plasma shifts the RF higher. As a result, the observed shift in RF corresponds to a summation of the two shifts. This summation makes it difficult for us to discriminate the two shifts, but a pair of different-sized CPs (*double CP*) enables discrimination of the two shifts caused in the same plasma with the identical film thickness. We so far derived the equation giving the two shifts based on the previous theory. In order to confirm the solution of the equation, we performed a simple model experiment of deposition in an argon plasma, inserting two CPs covered with a polyimide film of known thickness (55-micrometer). We first measured the original RFs of the two probes with no polyimide film and no plasma exposure. And then, we measured the RF of each CP with a layer of the polyimide film and with plasma exposure. Finally, we derived the film thickness and electron density from the four RFs: the result revealed almost the same film thickness as the 55-micrometer polyimide film and the electron density of 2×10 cm^{-3} which decently matches with the Langmuir probe data. In this presentation, we will show our latest results using the double CPs with the industrial application in mind.

11:40am **PS-ThM12 Study of Selective PECVD of Silicon on Silicon Nitride and Aluminum Oxide***, **Ghewa Akiki**, E.V. Johnson, P. Bulkin,* LPICM, CNRS, Ecole Polytechnique, Institut Polytechnique de Paris, France*; D. Daineka,* LPICM, CNRS, Ecole Polytechnique, Institut Polytechnique de Paris

Research in the field of area selective deposition currently focuses on the use of Atomic Layer Deposition (ALD) technique, and requires an initial nucleation delay between two different substrates, as well as a "passivation" step, namely a plasma etching step that resets the nucleation delay for one surface [1]. In analogy, we aim to demonstrate a Plasma Enhanced Chemical Vapor Deposition (PECVD) based approach using a non-sinusoidal voltage waveform [2] to excite an $Ar/SiF_4/H_2$ plasma. This plasma chemistry is believed to be a key ingredient to creating a varying nucleation delay as the surface processes depend on the deposition/etching balance controlled by the H_2 flow rate [3]. As a building block for our PECVD based approach, we report on the observation of a nucleation delay for a PECVD process for microcrystalline silicon films on two different substrates, first using a standard 13.56 MHz radio frequency excitation source. The deposition selectivity on a patterned chip containing both SiN_x and AlO_x areas as well as the influence of the plasma parameters, will be presented. The analysis is performed by comparing ex-situ ellipsometry spectra before and after deposition and by Scanning Electron Microscopy (SEM) micrographs.

[1] R. Vallat, R. Gassilloud, B. Eychenne, and C. Vallée, **J. Vac. Sci. Technol. A 35,** 01B104 (2017)

[2] J. Wang and E.V. Johnson, **Plasma Sources Sci. Technol. 26** (2017) 01LT01

[3] Dornstetter JC, Bruneau B, Bulkin P, Johnson EV, Roca i Cabarrocas P, **J. Chem. Phys. 140**, 234706 (2014).

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