Wednesday Morning, November 11, 2009

Plasma Science and Technology Room: A1 - Session PS1-WeM

Plasma Diagnostics, Sensors, and Control I

Moderator: G. Upadhyaya, Lam Research

8:00am PS1-WeM1 A New Diagnostic Tool of Electron Energy Distribution Function in Capacitive Mode Plasmas in a Variety of Frequencies, H. Shindo, Y. Nakazaki, Tokai University, Japan

A new diagnostic tool to measure Electron Energy Distribution Function (EEDF) by an emissive probe has been proposed[1] and applied to radiofrequency (RF) plasmas. In particular, the measurements are made, focused on the condition in which the mode transition from the capacitive to the inductive is occurred at the frequencies of 2 to 60 MHz. It is generally difficult for a conventional probe method to measure EEDF in RF plasmas, because of the plasma potential fluctuation, particularly in the capacitive mode. On the contrary, one of the advantages of the present method is that the measurements are free from the high frequency potential fluctuation.

The method is based on measurement of the functional relationship between the floating potential change ΔV_F and the heating voltage V_H of emissive probe. If the Maxwellian plasma is concerned, a practical and useful equation for ΔV_F can be obtained as in [1]. It is important to know that the value of ΔV_F contains information of electron energy distribution with several electron volt interval along the floating potential V_F , because ΔV_F is determined only by the current of plasma electrons with an energy interval.

In the experiments, the values of ΔV_F were measured in the Ar plasmas which were produced by a single-loop antenna[2] in the frequencies of 2 to 60 MHz and the gas pressures of 5 to 100 mTorr. The values of ΔV_F behave quite differently, depending on the frequency and the gas pressure, hence the plasma mode. It is found that in the inductive mode appeared at the pressures above 20 mTorr at 2 MHz, 30 mTorr at 13 MHz, the value of ΔV_F is consistent with the above-cited equation, enabling to determine the electron temperature, while in the capacitive mode appeared below abovementioned pressures and at 60 MHz, the behavior of floating potential change ΔV_F is fairly complicated, hence non-Maxwellian plasma. In all capacitive modes, from the data set of ΔV_F and V_F , the electron energy probability function (EEPF) is calculated, and the EEPF thus obtained reveals a bi-Maxwellian with the two electron temperatures depending on the frequencies. It should be emphasized that the present diagnostic method becomes powerful in observation of the plasma mode transition in a variety of frequencies.

References:

[1] K.Kusaba and H.Shindo, Review of Scientific Instruments, **78**, 123503-1(2007).

[2] Y.Jinbo and H.Shindo, Applied Physics Express, 2, 016001-1(2009).

8:20am PS1-WeM2 A Diagnostic Investigation of Pulsed PECVD for Thin Film Deposition, C. Lange, C.A. Wolden, Colorado School of Mines Low frequency (~1 Hz) pulsed PECVD is an alternative approach for selflimiting growth of high quality thin films at high rate and with good conformality. This technique raises a number of new questions with respect plasma ignition and dynamics. Critical questions include the role of metal precursors in the gas phase as well as those that adsorb to chamber surfaces when the plasma is off. To gain a more fundamental understanding of this process we have built a reactor with a diagnostics suite that includes I-V measurements, Langmuir probe, optical emission spectroscopy (OES), quadrupole mass spectrometry (QMS). In this paper we will present transient measurements from these techniques that are acquired in registry with the plasma pulse waveform. Relevant time scales in this system range from microseconds for application of a stable voltage waveform to seconds for mass transfer and chemical reactions. These critical time scales in the process are experimentally determined. Results will be compared to detail computational models. To decouple the complexities of this process comparisons are made among systems of increasing complexity. These include a baseline O2/Ar plasma, continuous wave PECVD, plasmaenhanced ALD, and finally pulsed PECVD.

8:40am PS1-WeM3 Characterization and Active Stabilization of Plasma and Generator Interactions, V.L. Brouk, D. Carter, Advanced Energy Industries, Inc., J. Roberg, Advanced Energy Industries, Inc

Plasma instabilities are often seen in low power, low pressure, electronegative discharges. Instabilities affecting particle density, optical emission and coil voltage have been observed with oscillation frequencies ranging from a few hundred hertz to well over one hundred kilohertz [1,2].

While instabilities can be inherent to plasma conditions it is well known that power delivery plays an important role in promoting or propagating the behavior. A study of the mutual interaction between RF amplifier and plasma impedance shows the alignment between impedance trajectory and the power profile contours of the generator is critical in determining a system's sensitivity to instabilities. Reactive elements in the delivery path can be used to rotate impedance trajectories but the recent advent of variable frequency RF supplies has provided a more convenient means for trajectory rotation and active stabilization. In this work we empirically evaluate these behaviors and demonstrate the utility of RF frequency as a controllable parameter for plasma stabilization. In defining an active stability control system, we demonstrate measurements for detecting and quantifying instabilities. Instability oscillation frequency offers insight into the nature of parasitic feedback from mutual generator-plasma interaction and thus we show how discriminating instability frequency is also useful in defining an intelligent stability control system.

1) A. M. Marakhtanov, et. al., J. Vac. Sci. Technol. A 21 (6), Nov/Dec 2003

2) A. Descoeudres, et. al., Plasma Sources Sci. Technol. 12 (2003) 152–157

9:00am **PS1-WeM4 Plasma Characterization of a 200-mm Hollow Cathode Magnetron for the Deposition of Metallic and Compound Thin Films**, *L. Meng*, *R.E. Flauta*, *M.J. Neumann*, *D.N. Ruzic*, University of Illinois at Urbana-Champaign

The hollow cathode magnetron (HCM) is a high density plasma tool developed for ionized physical vapor deposition (I-PVD) used for highaspect ratio thin film interconnects. To better understand the fundamental mechanisms of the HCM device performance and consequently obtain the control to ensure highly conformal and uniform thin film deposition, it is necessary to study the plasma conditions and correlate them to the resultant thin film properties. A commercial high power 200-mm INOVA HCM deposition tool from Novellus was characterized using a 3-D scanning Langmuir probe that was specifically engineered for the intense metal plasma present. This yielded a spatial resolution of both electron density (ne) and temperature (Te). In addition, a gridded energy analyzer (GEA) was integrated with quartz crystal microbalance (QCM) to determine the ionization fraction of the metal flux reaching the substrate. With an increasing input power in the range of 0-16 kW, Te at the substrate decreased from 3 to 1 eV while n_e increased from 6×10^{10} to 2×10^{12} cm⁻³. A decreasing pressure also increased the electron density. The 3-D spatial distribution of n_e and T_e in the HCM tool revealed a higher n_e and lower T_e at the center of the plasma than at the edge. These results strongly correlated to the resultant film deposition quality and uniformity on the substrate. The deposition rate of metal flux was recorded with QCM, while the GEA was adjusted to repel or admit the metal ions to allow for an ionization fraction of the metal atoms to be calculated. This fraction varied from less than 10% to over 90% depending on the input power and pressure conditions. Lower HCM power increased the ionization fraction due to the corresponding higher T_e and thus higher ionization cross section. At higher pressures, the ionization was enhanced because of the greater residence time of atoms in the plasma. The ion energy distribution was also studied using the GEA/QCM tool. These plasma diagnostics measured the resultant mechanisms of the HCM and provided a matrix of parameters such as Te, ne, metal ionization fraction, ion energy and deposition rate to allow for optimization of the deposition process. Ta and TaN thin films were then formed on Si substrates using Ar or Ar/N₂ sputtering plasmas, respectively. These films were characterized through the use of scanning electron microscopy (SEM), X-ray diffraction (XRD) and X-ray photoelectron spectroscopy (XPS) to determine the microstructure, crystal quality, and stoichiometry of the deposited film. The film properties were found to be affected by the HCM power, pressure and the sample locations, and correlated with the plasma parameters.

9:20am PS1-WeM5 Probe Measurements in a Very High Frequency CCP Discharge, L. Dorf, S. Rauf, K. Ramaswamy, K. Collins, Applied Materials INVITED

Langmuir probe (LP) measurements in a realistic very high frequency (VHF) capacitively coupled plasma (CCP) discharge are complicated by a number of factors, such as absence of a well-defined DC ground reference and unpredictable behavior of standard electronic components at VHF. The amplitude of RF potential in a VHF CCP discharge can be large (few tens of volts), especially compared to that in an ICP discharge with similar parameters. RF potential distorts both electron and ion parts of the measured probe V-I characteristic, resulting in unrealistic plasma parameters, and therefore needs to be compensated for. Here, we present results of measurements performed in a 300 mm 162 MHz dielectric plasma etcher using a compensated LP (CLP) and a floating double probe (DP). Probe designs employ a number of previously developed techniques. The

probes were used to study the effects of magnetic field, input power, pressure, and chemistry on plasma density radial profiles. The electron part of the CLP V-I characteristic was also used to study the effect of the input power on the electron energy distribution function (EEDF). For all operating conditions - input power of 100 - 1400 W and neutral pressure of 10 - 300 mT - the measured electron temperature was found to lie in the range of 2 - 4 eV, and the plasma density in the range of a few 10^{10} to a few 10^{11} cm⁻³, increasing with power. In Argon, at 10 - 50 mT, the density was found to increase with pressure (due to higher ionization rate). At higher pressure, 50 - 100 mT, the density profile was found to become more uniform, but no significant change in maximum density was observed; further increase in pressure (100 - 300 mT) leads to a decrease in plasma density. Applying a magnetic field of a few tens of Gauss (generated by solenoidal coils placed above the top electrode) was confirmed to have a significant effect on the radial density distribution. Due to a difference in electron residence time caused by the difference in field lines geometry, edge density increases with magnetic field, whereas center density decreases. In electronegative chemistries, the effects of pressure and magnetic field are different. Namely, in O_2 at 15 – 100 mT: (a) positive molecular ion density decreases with pressure (possibly due to higher attachment), and (b) center density decreases with magnetic field, but edge density remains largely unchanged. In CF4, the effect of pressure is similar to that in O2, whereas the effect of magnetic field is like that in Ar at low pressure (15 mT), and like that in O_2 at high pressure (100 mT). Experimental results were found to be in general agreement with results of applicable simulations.

10:40am PS1-WeM9 Macroscopic Diagnostics for In-Situ Measurement of Sidewall Charging During Plasma Etching, E. Ritz, J.A. Hoban, M.J. Neumann, D.N. Ruzic, University of Illinois at Urbana-Champaign

In plasma etching processes, especially those with high aspect ratios, the leading type of manufacturing defects that occur are trenching, bowing and twisting. These defects cause failures in semiconductor devices such as processors and DRAM. In order to investigate the role of feature sidewall charging on these defects, a series of macroscopic diagnostics were implemented which are capable of measuring time-resolved charge buildup at several points along a feature profile. This in-situ diagnostic consists of alternating conducting and insulating layers made of copper and teflon, respectively, with an axial hole that acts as the feature via. The insulating layers create discrete measurement layers, provided by the copper electrodes, which can be independently monitored inside of a commercial etching tool in real time to determine how the incident current from the plasma varies along the feature depth. By measuring the current reaching the bottom of the feature, as compared to the mid-plane or the top, one can determine the influence of sidewall charging. To determine the effect of geometry on charging, several aspect ratios were used by maintaining the same device height but varying the diameter of the via. The entire diagnostic is 19.2mm tall with aspect ratios from 5:1 to 10:1. Plasma and charging experiments were conducted in a commercial silicon dioxide etch chamber with three available frequencies (2.0 MHz, 2.2 MHz, and 13.56 MHz) thus allowing study of frequency-dependent charging, as well. Typical powers ranged from 300W to 1000W resulting in sidewall current measurements on the order of milliamps. Typical plasma densities are $2*10^{12} \pm 5*10^{11}$ cm⁻³ and electron temperatures are 3 ± 0.3 eV. Results from the diagnostics are shown for various plasma conditions and compositions.

11:00am PS1-WeM10 Study on Relation between CFx Radicals and Plasma Parameters in ICP Plasmas with Laser-Induced Fluorescence and Wave Cutoff Probe, *J.-H. Kim*, Korea Research Institute of Standards and Science (KRISS), *K. Rho*, KAIST, South Korea, *Y.-S. Yoo*, *S.-J. You*, *D.-J. Seong*, *Y.-H. Shin*, KRISS, South Korea

The behaviors of CF and CF2 radicals were studied in CF4 inductively coupled plasma. CF and CF2 radicals were measured using a laser-induced fluorescence method [1,2]. Absolute electron density was measured using a cutoff probe [3], and the electron temperature was measured using a Langmuir probe to study relation between the electron property and radicals. CF and CF2 densities are drastically changed by variations of operating pressure, ratio of mixed gases and RF source power. To examine the relation between electron density and CF and CF2 radicals, CF, CF2 radical and electron density were measured as varying the RF power which is a major external parameter influencing to the electron density. As the RF power was increased, CF and CF2 radical density increased in the range of low electron density and then decreased over a critical electron density. Dependence of CF and CF2 radical density on the electron density was theoretically analyzed with rate equations. The theoretically analyzed relation between the electron density and the radical density was in good agreement with the experimental result.

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[2] S. Hayashi, K. Kawashima, M. Ozawa, H. Tsuboi, T. Tatsumi, and M. Sekime, Sci. and Tech. Adv. Mat. V2, p555 (2001)

[3] J.H.Kim, D.J.Seong, J.Y.Lim, and K.H.Chung, Appl. Phys. Lett. V83, p4725 (2003)

[4] J.H.Kim, K.H. Chung and Y-S Yoo, J. Korean Phys. Society. V47, p249 (2005)

11:20am PS1-WeM11 Development of High Density Radical Source and the Behaviors of Radicals in N_2 -H₂ Mixture Plasma, S. Chen, Nagoya University, Japan

Dry processes using nitrogen atoms are essential to nitride semiconductor device fabrications such as nitridation, etching damage restoration or nitrogen doping technologies. To reduce the processing time and improve the film quality, the high density radical source with high efficiency and stability is strongly required. So far, some kinds of radical sources have been evaluated and characterized qualitatively using optical emission spectroscopy (OES). However, the absolute density could not be measured by the OES. In this study, we have developed a new high density radical source (HDRS) and measured the absolute density of atomic radicals by using vuacuum ultraviolet absorption spectroscopy (VUVAS).

The HDRS was designed by optimize the number of antenna coil turns in ICP. The ICP with 4 turns coil antenna enabled us to obtain the highest N atomic radical density. It was found that the radical density was significantly dependent on the power density, plasma density and gas temperature. N radical density was increasing from 7.3×10^{11} to 3.6×10^{12} cm⁻³ with pressure increases from 0.025 to 0.5Pa. These results show the N radical density was one order magnitude higher than traditional source. In the power dependence of radical density, the radical density was increased with increase the powers up to 400W and saturated.

The HDRS was also characterized using N2-H2 gas mixture. Relative changes of N, H and NH3 densities were measured as a function of the N2 flow rate ratio. NH3 was measured by Quadrupole Mass Spectroscopy (QMS). In this experiment, the total pressure N₂/H₂ was fixed at 0.5Pa. When N_2/H_2 ratio increased from 10% to 33.3%, the absolute density of H radical was increased from 2.3x10¹² to 4.1x10¹² cm⁻³. Absolute density of N radical increased from 2.3x10¹¹ to 1.7 x10¹² cm⁻³. At the N₂/H₂ ratios beyond 33.3%, the N radical density increased to $2.1 \times 10^{12} \text{ cm}^{-3}$, but H density decreased to 3.2x10¹¹ cm⁻³. In this experiment, the behaviors of NH₃ relative density agreed with those of H radical. When the N_2 flow rate ratio of 33% was fixed and the pressure was varied from 0.025 to 0.35Pa, it was found that the H radical density was higher than N radical density, but at pressures of above 0.35Pa the N radical density increased rapidly to 5.1x10¹¹cm⁻³ and H radical density increased to 3.4x10¹¹cm⁻³. As a result, the behaviors of radicals in N2-H2 mixture plasma were investigated and the mechanism of radical kinetics in HDRS was discussed.

Authors Index Bold page numbers indicate the presenter $|-\kappa -$ | Ritz, E.: R

- K —
Kim, J.-H.: PS1-WeM10, 2
- L —
Lange, C.: PS1-WeM2, 1
- M —
Meng, L.: PS1-WeM4, 1
- N —
Nakazaki, Y.: PS1-WeM1, 1
Neumann, M.J.: PS1-WeM4, 1; PS1-WeM9, 2
- R —
Ramaswamy, K.: PS1-WeM5, 1
Rauf, S.: PS1-WeM5, 1
Rho, K.: PS1-WeM10, 2

Ritz, E.: PS1-WeM9, **2** Roberg, J.: PS1-WeM3, 1 Ruzic, D.N.: PS1-WeM4, 1; PS1-WeM9, 2 — **S** —

Seong, D.-J.: PS1-WeM10, 2 Shin, Y.-H.: PS1-WeM10, 2 Shindo, H.: PS1-WeM1, 1

Wolden, C.A.: PS1-WeM2, 1

Yoo, Y.-S.: PS1-WeM10, 2 You, S.-J.: PS1-WeM10, 2