Tuesday Morning, November 5, 2002

Plasma Science Room: C-105 - Session PS+MS-TuM

Plasma Diagnostics and Sensors

Moderator: R.J. Shul, Sandia National Laboratories

8:40am **PS+MS-TuM2 High-energy EEDF Tail Detection in Highfrequency Discharges**, *J. Kudela*, *K Suzuki*, *Y. Nakagawa*, *Y. Numasawa*, ANELVA Corporation, Japan, *T. Beppu*, RITE, Japan

It is well-known that the enhanced high-frequency (HF) fields in the oscillating plasma sheaths can lead to generation of high-energy electrons. At sufficiently low gas pressures, this phenomenon is crucial for the discharge maintenance. However, the phenomenon may also cause the discharge instabilities. The detection of the high-energy tail in the electron energy distribution function (EEDF) in HF discharges is, therefore, of a particular interest from the scientific point of view. It is also important from the technological point of view. The high-energy electrons are determining factor in processing plasmas affecting the discharge chemistry in the plasma volume, and on the processing surface as well. Moreover, on the processing surface, the high-energy electrons may also be responsible for undesired physical processes like charge damage. In our work, we illustrate the detection of high-energy electrons by electrostatic probes in HF discharges at two different frequencies, 2.45GHz and 60MHz. In the microwave band (2.45GHz), the detection of the high-energy electrons is rather simple and it requires only direction-sensitive probes.¹ Similar technique is applied to the VHF band (60 MHz) discharges. In these discharges, however, a proper probe compensation is necessary. The conventional compensation methods, which are based on sensing floating potential fluctuations around the probe tip, lose information about the EEDF tail. We propose a method that can lead to detection of high-energy electrons, as well as to the diagnostics of the VHF discharges. This work is supported by NEDO.

¹ J. Kudela, T. Terebessy, and M. Kando: Hot electrons and EEDF-anisotropy in large-area surfacewave discharges; Proc. IV Int. Workshop Microwave Discharges: Fundamentals and Applications, Sept. 18-22, 2000, Zvenigorod, Russia; ed. Yu.A. Lebedev (Yanus-K, Moscow, 2001), p.63.

9:00am **PS+MS-TuM3 Coupled Diagnostic Studies of Plasma Etch Byproducts**, *M.T. Radtke*, *D.B. Graves*, *J.W. Coburn*, University of California Berkeley

Plasmas used for etching invariably include species that originate at surfaces. Etch byproducts commonly play a major role in plasma composition, in addition to influencing etch rate, anisotropy, critical dimension control, and selectivity. Etch byproducts often deposit on chamber walls, altering wall chemistry such as radical recombination reactions, and leading to the formation of particles. Chamber wall cleaning and conditioning protocols can play an important role in etch tool cost-ofownership. For new high-k and low-k dielectrics and metal gate electrode materials, the etch characteristics and etch byproducts are usually not known. In addition to posing a challenge for feature critical dimension control and other etch objectives, the unknown etch byproducts may pose environmental, health and safety hazards. We reports studies using an inductively coupled plasma reactor equipped with a cooled, rf-biased chuck, a downstream FTIR spectrometer, a quartz crystal microbalance, a Langmuir probe, an ion flux wall probe, an ion mass spectrometer, a separate threshold ionization mass spectrometer for neutral radical detection, and optical emission spectroscopy. We have employed this system to measure etch byproducts and etch byproduct transport for a range of new high-k and low-k dielectric materials as well as candidates for metal gate electrodes. We illustrate the use of coupled plasma diagnostics for ZrO₂/Cl₂, SiO₂/CF₄, RuO₂/O₂, HfO₂/Cl₂, and Si/Cl₂/O₂. In particular, detection and identification of low volatility byproducts can be challenging, and often require combining information from the ion mass spectrometer, the neutral mass spectrometer, film composition measurements on the quartz microbalance, optical emission spectroscopy, and the downstream gas composition.

9:20am **PS+MS-TuM4 Two-Dimensional Ion Flux Distributions on the Wafer Surface in Inductively Coupled Plasma Reactors**, *E.S. Aydil*, *T.W. Kim*, University of California, Santa Barbara

A two-dimensional array of planar Langmuir probes manufactured on a 200 mm diameter silicon wafer was used to measure the radial and azimuthal variation of ion flux impinging on the wafer surface in various mixtures of electropositive and electronegative gases maintained in an inductively coupled plasma etching reactor. The spatial variation of ion flux in a pure Ar discharge is radially symmetric and peaks at the center of the wafer for pressures between 10 and 60 mTorr. Addition of small amounts of

electronegative gases to an Ar discharge flattens the radial and azimuthal ion flux distribution and accentuates azimuthal variation due to subtle asymmetries in the reactor geometry such as pumping ports. At fixed power, pressure, and flow rate, the spatially averaged ion current density decreases with increasing mole fraction of the electronegative gases in the feed gas. In conjunction with experimental data, we developed a simple theoretical framework within which the spatial variation of ion flux in gas mixtures can be understood. Ion Flux uniformity in various binary mixtures of Cl2, He, Ar, HBr, O2, and SF6 will be discussed. Spatiotemporal variation of ion flux in presence of instabilities in SF6 discharges will be presented.

9:40am PS+MS-TuM5 Surface Dependent Effects at the Plasma-Surface Interface, G.A. Hebner, Sandia National Laboratories INVITED In a typical etching application, a number of different materials from the common silicon, and silicon oxide to more exotic nitrides and low-k materials can be located in very close proximity to each other. The interaction of these different materials through changes in the plasma chemistry, non-equilibrium surface layers and local electric field is of fundamental interest since the local chemistry and plasma properties determine the characteristics of the resulting etch profile. A number of techniques have been used to characterize etching plasmas as a function of the surface material. Plasma species such as CFx, SIFx and BCl radials have been measured as functions of the surface material and radial position using laser induced fluorescence. Those measurements show significant changes in radical species concentration for silicon, silicon oxide and ceramic surfaces. Measurements of the electron and negative ion density using a microwave interferometer and laser photodetachment also show surface dependent changes in the bulk plasma chemistry. In addition to the plasma chemistry, the sheath electric field is of interest since it's magnitude and vector guide the ion species. Of particular interest is the measurement of the material dependent surface charging, a task that is challenging considering the required spatial, temporal and electric field sensitivity. An atomic beam system combined with pulsed laser spectroscopy has been used to directly calibrate the electric field induced Stark shift of high lying energy levels. Measurements of the electric field within an inductively driven argon discharge will be discussed. The possibility of using this system to calibrate energy level shifts in other gases of technological interest to the microelectronics and lighting industry will be discussed. This work was supported by the United States Department of Energy (DE-AC04-94AL85000).

10:20am PS+MS-TuM7 Monitoring Sheath Voltages and Ion Energies in High-Density Plasmas using Radio-Frequency Current and Voltage Measurements, *M.A. Sobolewski*, National Institute of Standards and Technology

The bombardment of substrate surfaces by energetic ions plays an important role in plasma etching and other plasma processing applications. To obtain optimal results, ion kinetic energies must be carefully controlled. However, measuring ion energy distributions in situ, at a wafer surface during plasma processing, is difficult or impossible. A method for indirectly monitoring ion bombardment energies would thus be useful, both as a source of information to guide process development and as a tool for process monitoring and control in manufacturing. Accurate ion energy distributions can be calculated by models of plasma sheaths if one knows the sheath voltage, the electron temperature, and the total ion flux. These parameters are in turn related to radio-frequency (rf) current and voltage signals that can be measured outside a plasma reactor, without perturbing the plasma or the process. Indeed, several different model-based methods have been proposed for using rf current and voltage measurements to determine sheath voltages and ion energies. In this study, three such methods were tested. Tests were performed in argon and \check{CF}_4 discharges at 10 mTorr, in an inductively coupled, high-density plasma reactor. All the methods were able to successfully detect changes in sheath voltages and total ion flux, and to infer the effect of these changes on ion energy distributions. However, the rf measurements are relatively insensitive to changes in the electron temperature. To obtain the most accurate sheath voltages and ion energies from rf measurements, the electron temperature should be known ahead of time, or monitored by some independent measurement technique.

10:40am **PS+MS-TuM8 Gas Temperature Effects on CF_x Kinetics in a CF₄ Inductively Coupled Plasma**, *H. Abada*, **J.P. Booth**, *P. Chabert*, Ecole Polytechnique, France

We have used Laser Induced Fluorescence to determine CF and CF₂ radical concentrations in steady state and pulse-modulated inductively-coupled plasmas in CF₄ at 5 and 33 mTorr. The rotationally-resolved LIF excitation

spectra of the CF radical were used to determine the space and time resolved gas temperature. Strong temperature gradients were observed, with the temperature reaching 800 K in the reactor center at 33 mTorr, 250 W RF power. These measurements were used to correct the LIF measurements for the dependence of the partition function on the gas temperature, providing the first reliable measurements of CF and CF2 kinetics in this system. The concentration profiles can be used to deduce the net flux of these species from or to the reactor walls, using Fick's law but also allowing for thermodiffusion. The steady-state CF2 profiles showed that this species is produced predominantly at the reactor walls by CFx+ ion bombardment. Surprisingly, in the post-discharge the CF₂ density increases markedly for several milliseconds, before decaying slowly. We will explore the possible origins of this phenomenon, which include convection induced by gas cooling, vibrational relaxation and conversion of CF to CF₂ by chemical reaction. In contrast, the CF radical appears to be both produced and destroyed in the gas phase, and it's concentration decays monotonically and rapidly in the post-discharge.

11:00am PS+MS-TuM9 Electron Energy Distribution in GF₄/CF₃I Ultrahigh-Frequency and Inductively Coupled Plasmas, *T. Nakano*, National Defense Academy, Japan, *S. Samukawa*, Tohoku University, Japan INVITED

The electron energy distribution function (eedf) is an important factor in determining radical compositions in plasmas for nanometer-scale device fabrication. In this presentation, the electron energy distribution in plasma through a C₂F₄/CF₃I mixture, which is a novel chemistry proposed for lowdamaged, fine structure etching of SiO₂, is studied by trace rare gas optical emission spectroscopy (TRG-OES) and probe measurements. The integrated eedf above 13.5 eV (Seedf) is evaluated from the Ar emission at 750.4 nm. The Steedf exhibits a weaker dependence on the gas composition for the C2F4/CF3I mixture than for the C4F8/Ar mixture which is conventional chemistry for SiO₂ etching. For practical etching conditions, the S_{eedf} for the C_2F_4/CF_3I mixture becomes smaller than 1/3 of that for the C₄F₈/Ar mixture in both ultrahigh-frequency (UHF) plasmas and inductively coupled plasmas (ICP). Thus, using the C₂F₄/CF₃I chemistry, low charging damage in SiO₂ etching is expected. The probe-measured electron temperature (T_e), which indicates the degree of the exponential eedf decay in the low energy, is 2.5 eV in UHF plasma through the C_2F_4/CF_3I mixture and 4.1 eV in the ICP, while S_{eedf} is twice as large in the UHF plasma as in the ICP. This suggests an eedf enhancement in the middle energy region (5-10 eV) for the ICP, which prompts the dissociation of the feedstock gases. A quantitative estimation of the eedf using a bi-Maxwellian-like function, which is crucial to understanding the relationship between the eedf and feedstock gas dissociation, is in progress. The preliminary results also support the eedf enhancement in the middle energy region for the ICP.

11:40am **PS+MS-TuM11 Dependence of Radical Densities on Fluorocarbon Feed Gases in a Dielectric Etch Plasma**, *E.A. Hudson*, *J. Luque*, Lam Research Corp., *N. Bulcourt, J.P. Booth*, Ecole Polytechnique, France

Unsaturated fluorocarbon gases are increasingly important for critical dielectric etch applications. Under typical plasma etch conditions, these feed gases promote the deposition of fluorocarbon polymer films. Using process parameters to tune the polymer deposition characteristics, one can control the critical dimension and profile of the etched feature, and minimize the loss of the photoresist mask. Different unsaturated fluorocarbon gases produce different process results, for reasons which are poorly understood. In an effort to better understand these differences, the plasma radical composition has been analyzed for a range of feed gases including the unsaturated fluorocarbons octofluorocyclobutane(C_4F_8) and octafluorocyclopentene (C5F8), and also a saturated compound, perfluoroethane (C2F6). Optical emission spectroscopy (OES) and broadband UV absorption spectroscopy (UVAS) have been used to measure radical densities in a dual-frequency, capacitively-coupled, dielectric etch reactor. Species detected include CF, CF_2 , and F. Notable variations in radical densities were observed for a series of processes based on Ar, O2, and one of the fluorocarbon feed gases. $\mbox{\rm CF}_2$ density, in particular, showed a strong dependence on fluorocarbon feed gas. The F/CF2 density ratio increased by more than a factor of 2 when C₂F₆ was substituted for C₅F₈. For each fluorocarbon feed gas, the sensitivity to changes in the O2 flow has been evaluated. Results suggest that the role of oxygen in controlling polymer film thickness in and around etched features is related to polymer formation as well as polymer removal.

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