

Plasma Science and Technology Division Room 609 - Session PS-MoA

Plasma Diagnostics I

Moderator: H.H. Sawin, Massachusetts Institute of Technology

2:00pm PS-MoA1 Diagnostics for Insight into Fluorocarbon Plasma Chemistry, *H. Sugai*, Nagoya University, Japan **INVITED**

Understanding and controlling high-density fluorocarbon plasmas for dielectric etching has been requiring innovative tools for plasma diagnostics. First, I present a novel and simple probe called plasma absorption probe (PAP), which enables one to measure the local electron density even when the probe surface is soiled with processing plasmas.¹ The PAP is based on the resonant absorption of surface wave excited in a "cavity" at the probe head. Second, I describe a new technique for measuring electron energy distribution function (EEDF), i.e., RF-biased optical probe (RF-BOP).² These new tools reveal a clear difference between high-density ICP and surface wave plasma in their EEDFs, radical and ionic compositions. In the latter half of presentation, I focus on plasma wall interactions. Steady-state radical density profiles near the wall suggests that radical production on surfaces often exceeds the production by electron-impact dissociation of parent molecule. To give an insight into the mechanism, the time/space resolved measurement of radical density is made in a source gas puffed out repeatedly. This experiment allows us to separate the gas phase from the surface processes in time space. Another approach of ion-beam surface experiment is challenged: an ion beam of fluorocarbon species is injected to a substrate at the energies from 10 to 200 eV, and the radicals produced on the surface are detected as a function of the ion energy. For example, SiF₂, CF₂ and CF with less CF₃ are observed desorbing from silicon surface bombarded with CF₃⁺ ion. The relevant fluorocarbon chemistry will be discussed combining the beam study with the plasma experiment. ¹H. Kokura, K. Nakamura, I. Ghanashev and H. Sugai, to be published in Jpn. J. Appl. Phys. ²H. Toyoda et al., Extended Abstract of Int. Conf. Reactive Plasmas / GEC (Maui, 1998) p.27.

2:40pm PS-MoA3 Comparison of Actinometric and Diode-laser Absorption Measurements of [CF] and [CF₂] in an Inductively Coupled Plasma Reactor, *T.M. Bauer, A. Inoue, P.-T. Ton-Nu, J.L. Cecchi*, University of New Mexico

CF and CF₂ radicals have been shown to be significant precursors for selective oxide etching. Consequently, diagnostics that measure concentrations of these radicals are of interest for process development, monitoring, and control. We compare two such diagnostics: actinometry and wavelength-modulated diode laser absorption spectroscopy. Actinometry is a relatively simple technique. However, it relies on excited state species measurements and may suffer from unwanted influence from other plasma parameters. The diode laser is a more complicated technique, but it provides absolute ground state concentrations. To carry out these comparisons, we have made actinometric and diode laser measurements contemporaneously on an ICP reactor over a pressure range of 10 to 30 mTorr and rf power range of 200 to 1000 W. Gas feedstocks included C₂F₆, CHF₃, and CF₄, each with 10% Ar added as an actinometer. Over these ranges, the CF concentration varied from 8x10¹² to 4x10¹³ cm⁻³, as determined by the diode laser. The CF₂ concentrations ranged from 5x10¹³ to 9x10¹⁴ cm⁻³. Our results indicate that for CF₂ concentrations greater than 8x10¹³ cm⁻³, the actinometric measurements are proportional to the absolute concentrations measured with the diode laser system, with the same constant of proportionality for all feedstocks and over the entire operating range. For CF₂ below 8x10¹³ cm⁻³, we observe some deviation of the actinometric data, possibly due to interference from other features in the spectrum. For CF, we find that actinometric measurements are proportional to the absolute concentrations measured by the diode laser as a function of power, however, the constant of proportionality depends strongly on pressure and to a lesser extent on the feedstock. We believe that this reflects an underlying dependence of the CF actinometric signal on electron temperature.

3:00pm PS-MoA4 Ion Energy Distribution Functions (IEDFs) in NF₃-based Discharges with Various Diluents, *H. Hsueh*, The Pennsylvania State University; *B.S. Felker*, Air Products and Chemicals, Inc.; *R.T. McGrath*, The Pennsylvania State University; *J.G. Langan*, Air Products and Chemicals, Inc. NF₃-based discharges are commonly used for cleaning residual SiO₂ and Si₃N₄ films formed on the walls of PECVD chambers. In order to find a balance between fast chamber cleaning and overly aggressive cleaning chemistries, which can lead to premature hardware failure, a fundamental understanding of the physical and chemical characteristics of the discharge is required. For this reason, we have measured the relative concentration of the ionic and neutral species, ion energy distribution functions (IEDFs), and the optical emission spectra (OES) present within capacitively coupled parallel plate discharges operated with NF₃ diluted with either Ar, He, Ne, or O₂. For reactor operation at a fixed power density of 1.35 W/cm² and various NF₃ mole fractions, we found that when Ar was used as the diluent, the principal ion present was Ar⁺ for all pressures investigated (0.5-1.5 Torr) and the IEDFs exhibit the highest average energies for all gas mixtures studied. In contrast, for similar reactor operating conditions using helium dilution, He⁺ concentration was relatively low, with NF₂⁺, F⁺, F₂⁺, and N₂⁺ all having larger concentrations and the IEDFs for all species have lower energy peaks. When oxygen is used as the diluent, NO⁺ is the major dominant positive ion species. The IEDFs are also lowered when oxygen is used. A summary of how changes in pressure and gas mixture affect IEDFs for all of the diluent options mentioned above will be presented. Optical emission spectra were also recorded as a reference for all of the discharge conditions investigated.

3:20pm PS-MoA5 Optical Emission Thermometry Applied to the Measurement of Neutral Gas Temperature within a High-density, Inductively-coupled Plasma Abatement Device, *D.B. Graves, E.J. Tonnis, M.W. Kiehlebauch*, University of California, Berkeley

Flowing plasma reactors have proven important as downstream sources of reactive species and show promise for applications such as abatement of perfluorocompounds and other environmentally harmful effluents produced by the semiconductor industry. Recent spectroscopic measurements and simulation predictions in an inductively-coupled plasma source indicate that at high input powers, the neutral gas temperature within the plasma zone can be many times higher than ambient (> 1500 K) depending upon the plasma chemistry. High neutral-gas temperature could alter the dominant chemical mechanisms in the plasma and it is therefore important to measure and compare these results to model predictions. In this work, the neutral temperature of flowing CF₄/O₂, C₂F₆/O₂, and CF₄/H₂/O₂ plasmas was measured at varying input conditions using optical emission thermometry techniques. It was found that the rotational temperature, which was assumed to thermalize with translational temperature, is sensitive to the chemical nature of the plasma. In particular, C₂-derived (516.5 nm) emission temperatures measured within a CF₄/O₂ plasma were estimated at greater than 2000 K, which is several times higher than N₂-derived (399.8 nm) temperatures (~500 K) observed within a pure N₂ discharge over the same range of pressures and powers. In addition, the rotational temperatures were found to be relatively insensitive to the input power as long as a high-density discharge was sustained.

3:40pm PS-MoA6 Using Optical Emission Spectroscopy (OES) to Monitor Different Parameters for a Contact Hole Etch Process between Wet Clean, *D. Knobloch*, Infineon Technologies Dresden GmbH & Co. OHG, Germany; *F.H. Bell*, Infineon Technologies AG, Munich, Germany; *J. Zimpel, K. Voigtlaender*, Fraunhofer Institute, Germany

Oxide etch processes in IC-fabrication is gaining more and more importance, since the open area of contact hole processes still decreases and metal etch processes become partly replaced by the dual damascene technology. However, process development and stability issues in a manufacturing environment is still handled by trying to adapt a well known base line process on new applications. A typical example is the use of design of experiments in order to determine the robustness of the process window. We established a plasma monitor module for oxide etch processes to simplify process development, characterize process drifts, investigate process mix, and optimize endpoint detection. The monitor module is based on an optical multi-channel analyzer system that allows simultaneous detection of wavelength ranges between 200-950 nm. The

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system can be coupled to etch tools via the host net to collect data on a run to run basis. Intelligent data analyses software, such as principal component analysis (PCA) and partial least square (PLS) algorithms, is implemented to extract process and equipment relevant parameters. A typical manufacturing issue is the mean time between clean (MTBC) of oxide etch equipment. In general, wet cleans are conducted in case particles or the etch rate non-uniformity exceed certain specifications. Indeed, for the etch chamber under investigation, the decrease of the oxide etch rate at the wafer edge determines the wet clean cycle. We analyzed oxide and silicon surfaces after etching as a function of rf-hours and on different spots of the sample using x-ray photoelectron spectroscopy (XPS). The results show that the etch rate non-uniformity can be correlated to the polymer composition. Moreover, the optical emission data show that analysis of suitable wavelength ranges can be used for physical interpretation of the non-uniformity phenomenon. Furthermore, the influence of process mix on the cleanliness of the etch chamber will be discussed.

4:00pm PS-MoA7 Radical Detection using Appearance Potential Mass Spectrometry, H. Singh, J.W. Coburn, D.B. Graves, University of California, Berkeley

Appearance potential mass spectrometry (APMS) has recently gained importance for quantitative measurements of reactive radical species in plasmas. We have characterized the contributions to the APMS signal from the line-of-sight "beam" component and the background component of the species in the ionizer of the mass spectrometer. The beam signal is proportional to the number density of the species in the plasma, while the background component of the signal depends on various factors like the vacuum system design and pump speeds. Single stage differential pumping of the mass spectrometer is generally inadequate as the background signal usually dominates the beam signal for both radical and stable species. This necessitates implementation of modulated beam mass spectrometry using a mechanical chopper in the beam path. With one stage of differential pumping, the uncertainty in the beam component measurements is found to be as large as $\pm 180\%$. High beam to background signal ratio (>1.0) is achieved using three stages of differential pumping, and this vastly reduces the uncertainty in the beam component measurement to less than $\pm 10\%$. Another source of error in the APMS measurements is due to the lower extraction efficiency of the hot fragment ions produced by dissociative ionization in the ionizer of the mass spectrometer. The collection efficiency of the hot fragments is found to be up to 15 times smaller than that of products of direct ionization. The use of the dissociative ionization signal under plasma-off conditions to calibrate the radical direct ionization signal thus leads to overestimation of the radical number density, and we recommend an argon reference signal to avoid the discrimination effects due to dissociative ionization. The combination of multiple stages of differential pumping, background subtraction, and use of a direct ionization reference signal allows accurate quantitative measurements of radical density at the beam sampling point using APMS.

4:20pm PS-MoA8 C@sub 4@F@sub 8@ Dissociation Rate Control for Oxide Etch Process Plasma by Changing EEDF, S. Noda, T. Tatsumi, N. Ozawa, K. Adachi, M. Okigawa, M. Sekine, Association of Super-Advanced Electronics Technologies (ASET), Japan

In a dry etching process of SiO₂, optimizing the fluxes of chemical species and ion energy is essential to achieve high etch performances. The dissociation rate of reactive gas is one of the important parameters that can be used to control the density of the chemical species, i.e. radicals and ions, and their composition in the gas phase. In Ar based C₄F₈ plasma in a dual frequency parallel plate reactor system, we showed that the radical density ($[CF_x]$) and the composition ($[F]/[CF_x]$) were well correlated with a relation " $n_e \propto \tau_{res}^{-1}$ " (n_e : gas residence time, n_e : electron density, and $\langle \sigma \rangle$: dissociation rate coefficient (a function of electron impact dissociation cross section and electron energy distribution function (EEDF)).
1@ The dissociation rate could be easily controlled by choosing a gas flow rate (τ_{res}) and a source rf power (n_e) at a constant $\langle \sigma \rangle$ value. For further control, we need to know the effects of the $\langle \sigma \rangle$ which is varied by the EEDF depending on discharge conditions and types of plasma sources. We investigated if the control of dissociation rate and radical density in the gas phase would be possible by changing the EEDF. We employed a C₄F₈ gas dilution with Xe gas that has a lower ionization threshold energy than Ar. The EEDF profile was determined by the relative intensity method using optical emission spectroscopy (OES).
2@ In the Xe based plasma, the EEDF profile was much different from that in the Ar based plasma. The density of

high energy-electron (>10 eV) was about ten times lower than that of Ar based plasma at the same n_e (4×10^{11} cm⁻³) and τ_{res} (6 msec). It was also confirmed that the dissociation rate of both plasmas were followed by the relation " $n_e \propto \tau_{res}^{-1}$ ". We conclude that the dissociation rate of reactant gas can be controlled by EEDF and the EEDF monitor may be a preferable way to control the radical composition in CF plasmas. This work was supported by NEDO.
1@ Tatsumi et al., Jpn. J. Appl. Phys., 37 (1998) 2394.
2@ S.Noda et al., Proc. 20th Symp. on Dry Process, Tokyo (1998) p.235

4:40pm PS-MoA9 Plasma and Surface Diagnostics in Cl@sub 2@/O@sub 2@ Discharges in Transformer Coupled Plasma Reactors, E. Edelberg, Lam Research Corporation; S. Ullal, A. Godfrey, University of California, Santa Barbara; V. Vahedi, J.E. Daugherty, N. Benjamin, A. Perry, D. Cooperberg, R. Gottscho, Lam Research Corporation; E.S. Aydil, University of California, Santa Barbara

Plasma etching with high density transformer coupled plasma (TCP) reactors has become a ubiquitous process in microelectronics because of its ability to transfer patterns from a mask onto an underlying film with precision unequalled by any of the other etching methods. TCP reactors are operated at low pressures where the mean free paths of species are comparable to reactor dimensions. Thus, the role of walls and surface reactions has increased over the role of gas phase reactions in determining the plasma properties and etching behavior. This is most apparent when a stack of thin films of different materials must be etched sequentially in the same reactor using different gases. Chemicals used for etching one material adsorb onto the walls and contaminate the plasma in the following steps and may have deleterious effects on etching of other materials in the stack. In this study, we have explored the effects of wall adsorbates on the plasma properties. The objective is to understand how chemicals adsorbed on walls during one etching step affect the plasma properties in the following steps. Silicon trench isolation using Cl₂/O₂ plasmas is taken as a model process and plasma properties and their dependence on the wall conditions were studied through multiple plasma and surface diagnostics including, downstream Fourier transform infrared spectroscopy, in situ multiple total internal reflection Fourier transform infrared spectroscopy, optical emission actinometry, and Langmuir probe.

5:00pm PS-MoA10 Oxide Etch Studies in an Inductively Coupled GEC Reference Cell C@sub 2@F@sub 6@ Discharge using Diode Laser Spectroscopy, W.L. Perry, H.M. Anderson, University of New Mexico

An inductively coupled GEC Reference Cell has been modified to allow etching of patterned oxide wafers under conditions typical of commercial high density plasma reactors. This study reports on the oxide and photoresist characteristics of the tool as a function of reactor source power, bias power, pressure and heated silicon ring temperature. Diode laser absorption spectroscopy (DLAS), optical emission spectroscopy (OES) and Langmuir probe measurements were made at the same time. These measurements are used to construct response surface models of the tool's plasma chemistry behavior versus oxide and photoresist etch rate behavior. The oxide and photoresist etch rate behavior was found to also be profoundly influenced by the temperature of the cooling wafer chuck, so this became a fifth variable in the study. In a C₂F₆ discharge, CF and CF₂ radical concentrations measured by DLAS were also found to be highly dependent on wafer chuck temperature. In a 6 mTorr C₂F₆ discharge at 350 W source power and 75 W bias power, typical CF and CF₂ concentrations were in the range of 1×10^{12} cm⁻³ and 3×10^{13} cm⁻³ range. However, if the wafer temperature is allowed to rise toward 100 C, the CF₂ concentration increases dramatically apparently due to greater photoresist interaction with the plasma. If the wafer is properly cooled, the oxide and photoresist etch rates appear to be dominated by bias power and at low bias power, net deposition of fluorocarbon polymer prevails over net etching. However, with increased wafer temperature, both power and bias exert a strong influence on film etch rates and etching occurs at even zero applied bias power. The data is expected to provide an important database for models of oxide etching in inductively coupled plasma tools. This project was funded by SEMATECH.

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