Tuesday Afternoon, October 29, 2013

Plasma Science and Technology
Room: 102 B - Session PS1-TuA

Plasma Diagnostics, Sensors and Control
Moderator: N.St.J. Braithwaite, The Open University, UK

2:00pm **PS1-TuA1 Real Time Feedback Control of Photoresist ashing in an Electron Cyclotron Resonance Plasma Chamber**, ***B.J. Keville***, *C. Gaman, Y. Zhang, A.M. Holohan, S. Daniels, M.M. Turner*, Dublin City University, Ireland

Present practice in plasma-assisted semiconductor manufacturing specifies recipes in terms of inputs such as gas flow rates, power and pressure. However, ostensibly identical chambers running identical recipes may produce very different results. Extensive 'chamber matching', i.e. initial iterative, empirical tuning of the process recipe, which entails time-consuming, ex situ statistical analysis of process metrics, is required to ensure acceptable results. Once matched, chambers are run 'open loop' and are thus sensitive to disturbances such as actuator drift, wall seasoning and substrate loading, which may have deleterious effects on process metrics such etch depth, uniformity, anisotropy and selectivity. An alternative approach, which may reduce sensitivity to disturbances of the plasma, would be to specify a recipe in terms of quantities such as active species densities, and to regulate these in real time by adjusting the inputs with a suitable multivariable control algorithm. Multivariable closed loop control of an SF6/O2/Ar plasma in an Electron Cyclotron Resonance (ECR) etcher is the focus of a major research program in the National Centre for Plasma Science and Technology (NCPST) in Dublin City University (DCU). As an intermediate step, real time control of an O2/Ar plasma used for photoresist ashing has been implemented. More specifically, the oxygen 844 and argon 750 line intensities measured by optical emission spectrometry have been regulated by adjusting microwave power and oxygen flow rate in real time by means of a control algorithm. In order to test the efficacy of the control algorithm in reducing the sensitivity of the ashing rate to wall conditions, a wall disturbance was simulated by fluourinating the chamber walls using an SF6 plasma prior to O2/Ar ashing. In the open loop case, wall fluorination results in a large increase in the 844 line intensity, which is indicative of an increase in the density of atomic oxygen, and a concomitant increase in the ash rate is observed. However, under closed loop control, the average ash rate is unaffected by wall fluorination, thus demonstrating effective attenuation of the disturbance. Although this may not necessarily be of intrinsic interest – one generally ashes to end point without being overly concerned about tight control of the ash rate – it indicates that real time control of a plasma application – ashing, in this case - may be achieved indirectly by control of active species in the plasma. This has important implications for applications where tight control of dimensions is critical – etch profile, for example.

2:20pm **PS1-TuA2 Diagnostics of Inductively-Coupled Plasmas in Hydrogen Bromide : Bromine Atom and Electron Densities**, ***J.-P. Booth***, *N. Sirse, P. Chabert, M. Foucher*, LPP-CNRS, Ecole Polytechnique, France

Inductively-coupled plasmas (ICP) containing hydrogen bromide are widely used for conductor-etch applications, often using mixtures with Cl_2 and O_2. However, very few scientific studies (whether theoretical, simulation or experimental) have been made of HBr plasmas [1, 2] . We have studied pure HBr plasmas in an industrial-scale ICP (diameter 550mm, height 100mm, excited at 13.56MHz by a 4-turn planar coil) adapted for advanced diagnostic techniques.

We have demonstrated the first detection of Br atoms by two-photon laser-induced fluorescence (TALIF). The relative variation of Br atoms was determined as a function of HBr pressure (5-90 mTorr) and RF power (20-500W). The Br density increases with pressure over this range, although the dissociation fraction (Br density divided by the total gas pressure) decreases with pressure. The Br density also increases with RF power up to about 100W, but then progressively saturates. This saturation could be attributed to complete dissociation of the HBr, or to the onset of gas heating leading to gas density reduction. Measurements of the Br decay rate in the afterglow of a pulsed plasma allow the surface reaction coefficient of Br to be estimated.

The electron density was determined using a microwave hairpin resonator[3] . With HBr pressure the electron density peaks at 10 mTorr, and it increases with RF power. This behaviour is very similar to that observed in pure Cl_2, although the densities are about a factor 2 lower in HBr. The radial profiles of electron density are centre-peaked at low pressure, but at pressures above 50 mTorr the maximum is off-axis, peaking under the RF antenna coils.

This work was supported by Agence Nationale de la Recherche project INCLINE (ANR-09 BLAN 0019) and by the Applied Materials University Research Partnership Program. NS acknowledges the Ecole Polytechnique for a post-doctoral grant.

[1] Cunge G., Fouchier M., Brihoum M., et al., *J Phys D Appl Phys,* 2011, **44**,

[2] Sasic O., Dujko S., Makabe T., et al., *Chemical Physics,* 2012, **398**, 154-159

[3] Piejak R., Al-Khuzee J. and Braithwaite N. S., *Plasma Sources Sci. Technol.,* 2005, **14**, 734

2:40pm **PS1-TuA3 Ion Energy Distribution Measurements at the Substrate Location in Continuous-wave and Pulse Modulated Plasmas**, *D. Gahan, P. Scullin, D. O'Sullivan,* ***M.B. Hopkins***, Impedans Ltd., Ireland **INVITED**

Low pressure plasmas are used extensively in modern industry to process substrates. Both etching and deposition processes are controlled by energetic ion bombardment of the substrate. As substrates become larger, feature sizes smaller and film properties more complex there is an increasing demand for accurate monitoring and control of the ion energy distribution. Ion energy measurements have been reported in the literature for many decades. The two most common instruments that have been used are the quadrupole mass spectrometer and the retarding field energy analyzer (RFEA). The simplicity of the RFEA has led to its widespread use in various applications. The focus of this talk will be on the recent advances in RFEA technology for ion energy measurements in different types of plasma processes. In most cases the substrate is processed using an electrical bias. The substrate holder may be powered with radio-frequency (RF), pulsed-RF, direct current (DC) or pulsed-DC electrical signals. This complicates the use of RFEAs for ion energy measurement at the substrate location. The presence of the RFEA must not disturb the electrical bias and the electrical bias must not impair the RFEA data acquisition. Here we present an electrically filtered RFEA technology that allows for deployment at the biased substrate location. The filtering allows the RFEA sensor to float at the electrical bias voltage. We will discuss a number of filter configurations that cover the many frequencies, waveform shapes and bias levels that can be encountered. We also discuss a special configuration that permits time resolved measurements of the ion energy at the biased substrates under certain conditions. A number of novel extensions to the standard technology will also be presented. Spatially resolved measurements of the ion energy at multiple locations (simultaneously) across 300mm and 450mm substrate holders, used for plasma etching, will be presented. This RFEA design gives important information about the spatial uniformity of the plasma process under various plasma conditions. An embodiment of the RFEA design which can be used to determine the ion angular distribution is presented. This technique uses an additional orifice with variable aspect ratio to scan the angular distribution of the in coming ions across the energy range. Measurement of the ion angular distribution is particularly important near the edge of substrates, where yield is often poor due to sheath curvature in this region. Finally, the first results of a novel RFEA configuration that enables discrimination of the different ion species in the bombarding energy distribution will be presented and discussed.

4:00pm **PS1-TuA7 Time-resolved Optical and Electrical Diagnostics of Pulsed Plasmas Etching Processes**, ***G. Cunge***, *M. Brihoum, M. Darnon, E. Despiau-Pujo, A. Davydova, M. Haass, R. Blanc,* Cnrs/ujf/ Cea - Ltm, France*, N.St.J. Braithwaite,* The Open University, UK*, D. Gahan,* Impedans Ltd, Ireland*, S. Banna,* AMAT*, O. Joubert, N. Sadeghi*, Cnrs/ujf/ Cea - Ltm, France **INVITED**

Pulsed ICP plasmas are a promising solution to several issues related to IC fabrication. Recent results are indicating that pulsing the ICP power and/or the RF biasing power allows to increase the etch selectivity, to reduce plasma induced damages and to minimize ARDE. However, the reasons for these improvements remain unclear. In particular, the impact of plasma pulsing on the radicals flux, on the ion flux and on the ion energy in electronegative plasmas has not been studied in details. We have used a capacitively-coupled planar ion flux probe to monitor the time variations of the ions flux in an industrial ICP etch reactor from AMAT. At the same time, the time-averaged IEDF are measured by an RFA analyzer. Finally the radicals' densities are measured with a 10 µs time resolution by combining several diagnostic techniques: small polyatomic radicals are detected by broad band absorption spectroscopy (BBAS) in UV with highly stable LEDs as a light source, large closed shell molecules are detected by BBAS in the VUV and atomic species are monitored by threshold ionization

modulated beam mass spectrometry. We discuss in detail the experimental set-up that we have designed to carry out such time resolved measurements. Results are presented for various electronegative (Cl2, BCl3, SiCl4, SF6, CF4) plasmas, that are synchronously pulsed (ICP and rf bias pulsed in phase) or in which only the rf bias power is pulsed. We will show that the duty cycle is the predominant parameter to control the ion flux, the ion energy and the plasma chemistry. These results are in good agreement with global models of electronegative Cl2 plasmas. In particular, we show that in chlorine based plasmas, both the radical densities and the ion flux drops dramatically when the duty cycle is reduced. As a result for the same rf biasing power, the ion energy is much higher in pulsed plasma than in a CW plasma. By contrast, in a pulsed ICP without bias power the time averaged ion energy is bimodal, with the predominance of a very low energy peak (1-5 eV). Therefore, plasma pulsing allows to reach new domains of ion energy and radical fluxes, thereby extending the operating range of ICP reactor to that of CCP plasmas and downstream plasmas. This has interesting applications for ultrathin layer etching, ranging from graphene cleaning to metal gate and nitride spacer etching processes. Finally, both radical and ions flux oscillations in the kHz range are observed by time resolved diagnostics in pulsed plasmas. We will show that they are due to the propagation of acoustic waves in the reactor, with potentially interesting effect on the process uniformity.

4:40pm **PS1-TuA9 Optical Emission and Langmuir Probe Diagnostics of CH_3F-O_2 Inductively Coupled Plasmas**, *E. Karakas, V.M. Donnelly,* ***D.J. Economou***, University of Houston

CH_3F plasmas, mostly with added O_2, are used in selective Si_3N_4 etching over Si or SiO_2. Despite their use, fundamental plasma studies in these gas mixtures are very scarce. In this work, optical and Langmuir probe diagnostics were employed to study inductively couple plasmas in CH_3F/O_2 gas mixtures. In 50% CH_3F/50% O_2 plasmas, the electron density increased linearly ($0.7x10^{11}$→$2.7x10^{11}$ cm^{-3}) as power was increased 150-400W at 10 mTorr, but only weakly ($1.7x10^{11}$→$2.7x10^{11}$ cm^{-3}) within the pressure range of 10-40 mTorr at 300W. The effective electron temperature representing the high energy tail of bi-Maxwellian EEPFs was nearly independent of power and pressure. The gas temperature increased from 400-900 K as a function of inductive mode power between 75 and 400 W at 10 mTorr. For a constant feed gas flow rate and composition, the absolute H, F and O atom densities, estimated by optical emission rare gas actinometry, increased linearly with power. The feedstock gas was highly dissociated and most of the fluorine and oxygen was contained in reaction products HF, CO, CO_2, H_2O and OH. Reaction mechanisms were proposed to explain the observed behavior of the relative density of F and HF *vs*. power and pressure. Measured relative densities as a function of O_2 addition to CH_3F/O_2 changed abruptly for H, O, and particularly F atoms (factor of 4) at 48% O_2. A corresponding transition was also observed in electron density, effective electron temperature and gas temperature, as well as in C, CF and CH optical emission. These abrupt transitions were attributed to the reactor wall reactivity, changing from a polymer-coated surface to a polymer-free surface, and vice-versa, as the O_2 content in the feed gas crossed 48%.

*Work supported by Lam Research Corp.

5:00pm **PS1-TuA10 Non-contact Measurements of Substrate-Temperature by Frequency-Domain Low-Coherence Interferometry**, ***T. Tsutsumi***, Nagoya University, Japan, *T. Ohta,* Meijo University, Japan, *K. Ishikawa, K. Takeda, H. Kondo, M. Sekine, M. Hori,* Nagoya University, Japan, *M. Ito*, Meijo University, Japan

High-precision, rapid temperature measurements on wafers such as silicon, sapphire, etc. are required for plasma processing. A method is demonstrated here that uses a frequency-domain low-coherence interferometer (FD-LCI).

In order to realize the plasma processing with high precision, the temperature control of wafer or chamber-wall is one of important factors. Especially the wafer temperature influences on etched profile and etching rates. Moreover, a spatial distribution of radicals in gas phase or plasma-surface interactions on the surface are affected by temperatures of the chambar-wall. Therefore, the precisely non-contact temperature-measurement technology is needed for the fabrication of electric devices.

In this study, we have developed a highly precise and non-contact temperature monitor using a Fourier domain low-coherence interferometer (FD-LCI) and a super luminescent diode (SLD: center wavelength: 1330 nm, spectral width: 37.6 nm) as a low coherence light source. The FD-LCI can measure an optical path length of wafer on the basis of auto-correlation signals, which are generated by interferences between the light reflected from the front and back surfaces, and analyzed by the inverse Fourier transform of spectral interferogram. Since the optical path is changed by thermal expansion and refractive-index, the wafer-temperatures can be analyzed. By this method, we have successfully achieved to estimate a temperature of various commercial wafers such as silicon, sapphire during plasma processes.

5:20pm **PS1-TuA11 Detection of Vacuum Ultraviolet in Argon-containing Inductively Coupled Plasmas**, *S.B. Radovanov,* Varian Semiconductor Equipment, Silicon Systems Group, Applied Materials Inc., *H.M. Persing,* Applied Materials Inc. Varian Semiconductor Equipment, Silicon Systems Group, Applied Materials Inc., *J.B. Boffard, C.L. Culver, S. Wang, C.C. Lin,* ***A.E. Wendt***, University of Wisconsin-Madison

The spectrum of light emitted by plasmas used in materials processing applications includes vacuum ultraviolet (VUV) photons, which are known to play a significant role in critical surface reactions under certain process conditions. Monitoring of the surface flux of VUV photons emitted from the inductively coupled plasma (ICP) and its dependence on discharge parameters is thus highly desirable. However, non-invasive direct detection of VUV photons is generally difficult, as few window materials transmit in the VUV. We thus examine the argon resonance level atom concentration as a prospective proxy for VUV emission, as 106.7 and 104.8 nm VUV photons are produced in the spontaneous radiative decay from Ar resonance levels to the ground state. Argon resonance level concentrations have been measured in the center of an ICP with a planar spiral induction antenna through "branching fraction" analysis of the visible optical emission (OES) spectrum.* Measured concentrations are subsequently used as inputs to a VUV radiation transport model developed to determine the corresponding axial VUV photon flux. Reabsorption of VUV photons by ground state atoms is significant even at pressures as low as 1 mTorr, and a proper accounting in the model thus requires accurate representation of the gas temperature. Model results based on the resonance level concentrations over a range of pressures (1-25 mTorr) and RF (13.56 MHz) power (up to 1000 W) compare favorably with the axial VUV photon flux sampled directly through a small hole at the center of an electrode located opposite the ICP antenna. Absolute VUV fluxes were measured with a windowless aluminum oxide photodiode sensitive to wavelengths below ~110 nm. Additionally, relative VUV fluxes were also obtained using a sodium salicylate coating on the inside of a side port vacuum window. The sodium salicylate converts VUV into a detectable visible light signal through fluorescence, and, unlike the photodiode, is sensitive in the wavelength range of H atom VUV emissions (122 nm). Preliminary results suggest that a combination of photodiode and sodium salicylate signals thus allows discrimination between hydrogen and argon contributions to the VUV flux in Ar/H_2 gas mixtures.

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5:40pm **PS1-TuA12 Time-resolved *In Situ* Quantum Cascade Laser Diagnostics Applied to Transient Molecular Plasmas**, ***S. Welzel***, Eindhoven University of Technology, Netherlands, *F. Brehmer,* Eindhoven University of Technology, Netherlands; AFS GmbH, Germany, *M.C.M. van de Sanden,* Eindhoven University of Technology; DIFFER, Netherlands, *R. Engeln*, Eindhoven University of Technology, Netherlands

The detection of stable and transient species along with gas temperature measurements remains a challenge for the majority of molecular (complex) plasmas. Considering particularly plasmas at atmospheric pressure with inherently small discharge volumes, (optical) access to the active plasma is often hampered. On the other hand, phase- and time-resolved *in-situ* measurements are a valuable tool (i) to establish heavy particle temperatures, (ii) to identify excitation mechanisms in the plasma, (iii) to discriminate gas phase and surface reactions as they occur on significantly different time scales, and (iv) to unravel particularly temperature-dependent reaction mechanisms. Modern mid-infrared laser sources, known as quantum cascade lasers (QCLs), provide a means for highly time-resolved absorption spectroscopy in the molecular "fingerprint" region. The time-resolution can be thereby as good as a few tens of nanoseconds. Although continuous-wave QCLs are increasingly being applied for conventional monitoring purposes, pulsed distributed feedback QCLs are perfectly suited for diagnostic studies on transient plasmas.

CO_2 containing dielectric barrier discharges (DBDs) operated in the mid-frequency (kHz) range were studied by means of *in-situ* time-resolved QCL absorption spectroscopy. Special beam shaping optics was used to accommodate the laser beam diameter to typical gap widths of ~ 1 mm in single and multiple-pass configuration. Different synchronisation schemes were applied to achieve phase-resolved measurements during individual AC cycles as well as to monitor molecular absorption signals during pulsed discharge operation. Mixing ratios of CO in its electronic and vibrational ground state were of the order of a few percent and thus confirmed earlier *ex-situ* studies of the effluent. More importantly, the concentrations levels were changing only slowly in time, i.e. of the order of the residence time. A direct CO_2-to-CO dissociation through electron impact appears very unlikely under these conditions. The kinetics of low-lying ro-vibrational states of CO_2 along with the evolution of the CO concentration were studied on a sub-millisecond time-scale to (i) establish (rotational) gas

temperatures, and (ii) to estimate the influence of vibrational-vibrational and vibrational-translational energy transfer processes.

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