

Wednesday Afternoon, November 12, 2014

Plasma Science and Technology

Room: 308 - Session PS-WeA

Plasma Diagnostics, Sensors, and Control

Moderator: Jean-Paul Booth, LPP-CNRS, Ecole

Polytechnique, France

2:20pm **PS-WeA1 Diagnostics of Cl₂/O₂ Inductively-Coupled Plasmas by Ultra-High Sensitivity Broad-Band Absorption Spectroscopy**, *Mickaël Foucher*, LPP-CNRS, Ecole Polytechnique, France, *E. Carbone*, LTM - MINATEC - CEA/LETI, France, *J.-P. Booth*, LPP-CNRS, Ecole Polytechnique, France

Inductively-coupled plasmas (ICP) containing O₂ and Cl₂ (and often HBr) are widely used for etching, for example of silicon transistor gate. Simulations, including global and two-dimensional fluid models have been developed over the years, but validation against experimental measurements of absolute densities remains sparse.

Absorption spectroscopy is a powerful diagnostic for reactive plasmas, providing absolute density measurements of numerous atoms, molecules and free radicals in ground and various excited states. The sensitivity is determined in practice by the characteristics of the light source used: spectral range, stability and intensity. Previously Xe arc lamps have been used but they suffer from spatiotemporal fluctuations, limiting the sensitivity to about 10⁻³ in absorption. More recently UV light-emitting diodes have been used, giving greatly increased stability, but these have very limited spectral ranges (a few 10's of nm), necessitating the use of specific diodes for each species detected.

We have constructed a new absorption bench that overcomes these difficulties. The light from a broad-band (200-1000) nm laser-induced plasma source (Energetiq-LDLS EQ-99) is collimated and steered with reflective (achromatic) optics. After passing through the reactor the beam is analyzed with an aberration-corrected spectrograph (Acton SCT-320) equipped with a 1024 element photodiode array detector. Three gratings allow spectral ranges of 32, 63 or 250 nm to be analyzed at one time. This setup gives spectra in minutes with random noise and baseline stability better than 10⁻⁴, allowing detection of species which only show weak absorption bands over wide spectral ranges.

The gases (O₂, Cl₂ and mixtures) are flowed through a cylindrical aluminum chamber (55 cm diameter, 10 cm height). The plasma is excited at 13.56 MHz by a 4-turn planar coil through a dielectric window. We observed molecular absorption bands from ground state Cl₂, vibrationally excited O₂ and of various O_xCl_y reaction products, allowing the densities, vibrational and rotation temperatures to be determined. As far as we know this is the first time oxychlorides densities have been measured in plasmas. This data is complemented by measurements of absolute atom densities (Cl and O) by TALIF and hairpin probe measurements of electron density. The interpretation of this data set will be discussed.

2:40pm **PS-WeA2 Diagnostics in Pulsed Hydrogen Plasmas**, *Jerome Dubois*, *G. Cunge*, LTM - CEA/LETI, France, *N. Posseme*, CEA-LETI, France, *M. Darnon*, LTM - CEA/LETI, France, *L. Vallier*, CNRS-LTM, France, *O. Joubert*, LTM - CEA/LETI, France

Hydrogen plasmas have been used for decades in the microelectronic industry with applications in the fields of deposition (PECVD, Plasma Enhanced Chemical Vapor Deposition) [1], etching [2] and surface treatment (reduction of the photoresist roughness in the lithography step [3]). However hydrogen is a very peculiar element due to his low mass and his electronegative character, and the mechanisms at stake in low pressure hydrogen plasma aren't well understood yet. A complete fundamental study with reliable diagnostics would be highly valuable for many applications [4]. Moreover, hydrogen plasmas present a great potential interest for the treatment of new materials such as graphene [5] or carbon nanotubes [6]. To modify the surface of such ultrathin layers without damaging the material, very low ion energy bombardment is required (conditions such as those obtained in pulsed ICP reactor [7]). By contrast, for other applications such as etching of several nanometer thick layers, the ion energy must be very high to get a significant etch rate. To assist the development of innovative processes in H₂ plasmas, we have thus analyzed systematically CW and pulsed H₂ plasmas both with and without RF bias power. In particular, we carry out time-resolved ion flux, and time-averaged ion energy measurements in different pulsing configurations. A large variety of ion energies and shapes of IVDF are reported depending on pulsing parameters. The IVDF are typically very broad (due to the low ion transit time of low mass ion through the sheath) and either bi or tri-modal (H⁺, H₂⁺ and H₃⁺

contributions). The time variations of the ion flux in pulsed plasmas also presents peculiar features that will be discussed.

References

1. W. A. Lanford, and M. J. Rand, *Journal of Applied Physics*, **49**, 2473-2477 (1978)
2. A. Efremov, N. K. Min, J. Jeong, Y. Kim, and K. H. Kwon, *Plasma Sources Science and Technology*, **19** (2010)
3. E. Pargon, L. Azarnouche, M. Fouchier, K. Menguelti, R. Tiron, C. Sourd, and O. Joubert, *Plasma Processes and Polymers*, **8**, 1184-1195 (2011)
4. M. Sode, T. Schwarz-Selinger, and W. Jacob, *Journal of Applied Physics*, **113** (2013)
5. E. Despiau-Pujo, A. Davydova, G. Cunge, L. Delfour, L. Magaud, and D. B. Graves, *Journal of Applied Physics*, **113** (2013)
6. A. Hassanien, M. Tokumoto, P. Umek, D. Vrbancic, M. Mozetic, D. Mihailovic, P. Venturini, and S. Pejovnik, *Nanotechnology*, **16**, 278 (2005)
7. C. Petit-Etienne, M. Darnon, P. Bodart, M. Fouchier, G. Cunge, E. Pargon, L. Vallier, O. Joubert, and S. Banna, *Journal of Vacuum Science & Technology B*, **31** (2013)

3:00pm **PS-WeA3 The Role of Diagnostics in Plasma Etch Reactors in Enabling the Information Age**, *Alex Paterson*, *J. Holland*, *S. Sriraman*, *E. Hudson*, *H. Singh*, *V. Vahedi*, Lam Research Corp **INVITED**

Over the last decade, semiconductor industry growth has been driven chiefly by the demand for consumer electronics: the move to mobile smart devices such as phones and tablet PC's. It is now common place for hand-held mobile devices to have 32 Gb of memory and processor speeds of over 1 GHz, a truly remarkable feat that would have been unthinkable 10 years ago. This capability has been enabled by the continuation of IC scaling to smaller and smaller features sizes with the present technology being mass produced by 28 nm node technology and smaller nodes down to 10 nm currently being developed by IC manufacturers. The limitations of lithography to keep up with the decrease in dimensions required for these smaller nodes has resulted in new challenges for plasma etch to enable patterning at these small feature sizes. Device performance requirements also drive critical dimension (CD) non-uniformity to less than one nanometer across the entire 300 mm wafer for sub-20 nm features and yield requirements extend this patterning region to within 1.5 mm of the wafer edge. Wafer fabrication production also relies on plasma etch solutions to be stable at these levels across long periods of time and capable of flexibility in multiple applications. The realization of all of these goals has been greatly facilitated by a much better understanding of the basic chemical, physical, and electromagnetic processes that occur during the plasma etch of semiconductor devices.

In this paper we will discuss the crucial role diagnostics play in achieving this understanding and in the development of state of the art plasma etch chamber technology that allow the continuation of Moore's Law. Diagnostics are essential not only to understand etch mechanisms and chamber characteristics but to also accelerate hardware development in order to meet customer time critical needs. We will review the different types of diagnostics commonly used in plasma etch chamber development with reference to findings from literature and augment this with diagnostic work undertaken at Lam Research. Finally, we will discuss the suitability of diagnostics in main stream production and give some thoughts on future diagnostics that may be required for production enhancement and also angstrom level etching.

4:20pm **PS-WeA7 Ion Angular Distributions Measured with a Planar Retarding Field Analyzer**, *Shailesh Sharma*, Impedans Ltd., Ireland

In microelectronics fabrication the angular distribution of the bombarding ions can impact the process outcome. The ion energy distribution as a function of ion angle at specific locations on the substrate or wafer surface need to be controlled in certain anisotropic etching and conformal deposition plasma processes. We report a novel method for the measurement of ion energy distributions as a function of ion angle, at the substrate location, using a planar retarding field analyser.

Planar retarding field analyzers are commonly used to measure ion energy distributions but provide no information about the angular distribution of ions bombarding the substrate surface. Here, we report on a novel planar retarding field analyser design capable of resolving the angular distribution of the energetic ions. The design has three active grids, a collector plate and an aperture with variable aspect ratio (height / diameter) to control the angular spread of the ions allowed through the device for detection. First, the potential of the ion energy discriminating grid is modulated to select

ions with a specified energy resolution for analysis. Then, the aspect ratio of the aperture is varied from large acceptance angle to narrow acceptance angle with specified angle resolution - predetermined from the aperture geometry. The ion current is recorded for each acceptance angle to give an integral form of the ion angular distribution at a given ion energy where the angular distribution can be recovered by taking a first derivative. The procedure is then repeated for each ion energy. Once the angular distribution is determined as a function of ion energy, the energy distribution as a function of ion angle is easily calculated.

The analytical theory used to define ion current as a function of incident ion angle, ion energy and aperture aspect ratio is presented. The method used to vary the aspect ratio of the additional aperture is also discussed. This novel method allows ion angular distributions to be determined using a compact planar retarding field analyser.

With the modified retarding field analyzer design and advanced analytical technique, ion angular distributions with angle resolution as low as 3° have been measured and resolution up to 1° can be achieved. This technique adds important functionality to the retarding field analyser technology - which has become one of the most important technologies in the field of plasma diagnostics in recent years.

4:40pm PS-WeA8 Quantitative Analysis of Neutral Species Generated in Styrene Low Pressure RF Plasma, as a Function of Plasma Power, X. Gillon, J.-J. Jean-Jacques, Laurent Houssiau, University of Namur, Belgium

Plasma polymerization processes enable unique polymer coatings unattainable with conventional wet chemistry. Among them, plasma polystyrene (pPS) deposition has been intensively studied, but very few studies report on plasma diagnostics of styrene discharges, which is however a necessary step to understand the fundamental mechanisms of plasma polymerization. In this work, pPS was produced from pure styrene vapor injected in a vacuum chamber at 50 mTorr (6.7 Pa) and 28 sccm. An inductively coupled plasma was ignited by a planar coil delivering a continuous wave RF power ranging from 30 W to 210 W. Plasma diagnostics was achieved by means of a mass spectrometer (MS) located in the post-discharge region, enabling only the detection of neutral species. A problem associated with electron impact MS is the cracking of organic molecules, which hampers species identification and quantification in the plasma phase. However, by reducing the electron energy as low as 12 eV, much below the standard 70 eV energy, we were able to suppress most of the molecular cracking, so that species measured by MS could be unambiguously assigned to neutral species existing in the plasma. The assignment was further confirmed by measuring the appearance potentials of all ions in the MS. This procedure revealed the existence of 55 neutral species in the styrene plasma, which is five times more than what has been reported so far. Their relative intensities in the MS spectrum help understanding the formation pathways of these species, either by direct fragmentation of the styrene molecule, or by recombination of small molecules. The most abundant species generated in the styrene plasma were H_2 , methane, acetylene, ethylene, benzene, toluene and naphthalene. In order to quantify the partial pressure of those species in the plasma, we determined their sensitivity factors by injecting them pure in the plasma reactor and measuring the molecular ion peak intensity at 50 mTorr pressure. The MS intensities measured in the styrene plasma were subsequently converted into partial pressures. The sum of partial pressures from the main species present in the plasma, including styrene, was found to match remarkably well with the measured pressure in the reactor, confirming the quantitiveness of the procedure. The main features observed in the plasma chemistry when the power was raised are: a decrease of styrene pressure (monomer consumption) along with a strong production of acetylene and hydrogen. A very sharp drop of the plasma pressure was measured around 185 W, corresponding to the disappearance of the monomer and a sudden increase of the deposition rate.

5:00pm PS-WeA9 Comparison of Commercial Plasma Probe Systems, Valery Godyak, RF Plasma Consulting, *B.M. Alexandrovich,* Plasma Sensors

Electrostatic (Langmuir) probes are powerful instruments for diagnostics of non-equilibrium plasmas in experimental and industrial plasma reactors. There are three levels of the probe diagnostics comprising different equipment complexity and having different accuracy of obtained the plasma parameters. These three approaches are based on inferring the plasma parameters from: *i* – the ion part of the probe characteristic, *ii* – the electron part of the probe characteristic (classical Langmuir probe method), and *iii* – by differentiation of the probe characteristic to obtain an Electron Energy Distribution Function, EEDF. Then, the electron temperature T_e and plasma density N , as well as, the rates of collisional processes and transport coefficients are found as corresponding integrals of the measured EEDF. Methods *i* and *ii* assume Maxwellian EEDF which is not valid for most cases of non-equilibrium plasmas. This and many others questionable

assumptions in methods *i* and *ii* make actual EEDF measurements the only reliable contemporary probe diagnostics. Langmuir probes in plasma processing reactors are often subjected to the probe surface contamination and high level of rf and low frequency noise. Reliable EEDF measurements require the probe system capable of efficient mitigation of these environmental distortions. The presentation main subject is comparison of EEDF measurement results obtained with different commercial probe systems. It is shown that the measurement accuracy of the plasma parameters in many commercial probe systems is compromised by heavily distorted EEDF in low and high energy region. Low energy distortions make impossible to detect low energy electrons (comprising the majority of electron population), thus leading to underestimation of the plasma density, while high energy distortions make impossible detection of fast electrons producing excitation and ionization. Therefore, some commercial probe systems yielding distorted EEDFs are unable to reveal any additional valid information to that obtained with classical Langmuir procedure.

5:20pm PS-WeA10 Systematic Diagnostic Approach for Fabricating High Quality SiNx:H Film using UHF Assisted Capacitively Coupled Plasma Source, J.G. Han, B.B. Sahu, Kyung S. Shin, Sungkyunkwan University, Republic of Korea, *K. Ishikawa, M. Hori,* Nagoya University, Japan

Silicon nitride thin films have shown many useful applications in microelectronic and optoelectronic industries. Fabrication, of these films at low temperature, is done typically by PECVD using a mixture of silane (SiH_4) and ammonia (NH_3). Recent trend shows that the most practical deposition of low-hydrogen-content-silicon-nitride-films ($SiNx:H$) is to use N_2 instead of NH_3 as the main nitrogen source. However, N_2 has an inherently much higher bonding energy than NH_3 , which makes N_2 more difficult to dissociate into free nitrogen active species, thus nitrogen deposition rate is significantly reduced. But if low hydrogen nitride films can be obtained, which may give better device performance, the deposition rate may not be an important factor and PECVD of silicon nitride by little addition of SiH_4 and NH_3 to the N_2 is still an attractive process. Moreover, the important deposition parameters for any PECVD process are RF power, working pressure, substrate temperature, the gas flow ratio of the reactant gases and the electrode spacing (for CCD or parallel plate system). All these parameters have significant role on the deposition and etch rates along with other physical and optical properties of film depending on device applications.

Although PECVD processes have shown as an emerging method for achieving good quality $SiNx:H$ films for the industry, still there are lack of understanding in correlation between the properties of the plasmas and the characteristics of the synthesized films. In the present work, a deposition parameter matrix is constructed for $N_2-SiH_4-NH_3$ PECVD process and the effect of variation of above parameters on deposition is studied. The present study investigates PECVD process with different plasma processing conditions by utilization of different plasma sources, e.g., RF, which is capacitively coupled plasma (CCP) source at 13.56 MHz and UHF, a 320 MHz very high frequency (VHF) RF source. The goal of the UHF source is to assist and enhance the dissociation of nitrogen radicals along with the RF. One of the major goals of this work is also to investigate dissociation of the nitrogen radicals, which controls the $SiNx:H$ film deposition process. To understand the fundamental plasma surface interactions in this process, basic plasma diagnostics such as Langmuir probe (LP), optical emission spectroscopy (OES), and vacuum ultraviolet absorption spectroscopy (VUVAS), etc., are used. Thus, the investigations, of high quality $SiNx:H$ film synthesis described in this paper, focus predominantly on the plasma diagnostics and film synthesis. This also reports about high quality film having transmittance about 90 %.

5:40pm PS-WeA11 Electron Beam Generated Plasmas in Fluorine Chemistries, David Boris, R.F. Fernsler, G.M. Petrov, Tz.B. Petrova, S.G. Walton, Naval Research Laboratory

Electron beam generated plasmas are characterized by high plasma densities and very low electron temperatures, making them well-suited for next-generation processing techniques where high fluxes of low energy ions are desirable. In this work, we focus on plasma generation in fluorine containing gas backgrounds due to their relevance to a number of industrial plasma applications. In particular, we focus on the effect of fluorine-containing gas dilution on the plasma properties of electron beam generated plasmas including electron density, total plasma density, electronegativity, and electron temperature. These parameters are measured through a combination of Langmuir probe, and RF impedance spectroscopy techniques.

6:00pm **PS-WeA12 Characterization of Hydrogen Recombination at the Wall and its Effect on Hydrogen Source Performance, *Shaun Smith*, MKS Instruments, Inc.**

There is ongoing interest in using remote plasma sources for on wafer processing with hydrogen radical based chemistries. Yet there has been limited availability of reliable measurements for recombination rates, as they pertain to semiconductor processing. Presented here are diagnostics and some insight to understating of how process responds to material choice or the surface condition of the plasma facing wall. The impact that surface recombination of radical species has on the discharge is discussed and the impact that surface material choice and condition has on source operation and process performance is examined. As well as a discussion of factors that can effect that recombination rate. These parameters are explored in the 1-10torr 1-5slm and 1-10kW regime

Atomic hydrogen recombination rate is measured for a range of materials. Toroidal plasma sources are then built with these materials as the plasma facing wall and are characterized for their discharge parameters and atomic hydrogen output. The discussion will include a description of the diagnostic tools used in this study; a comparison of modeled source discharge parameters running in Ar and H₂ with experiment along with a brief comparison of the impact of volumetric and surface recombination of radical species is presented.

This work was specifically targeted for the use of radicals produced by a toroidal remote plasma sources for semiconductor applications but is generalizable to discharges in hydrogen independent of excitation or application.

Surface recombination of the hydrogen radical is shown to be a dominant mechanism in determining process parameters for semiconductor applications.

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