Plasma Science and Technology Division
Room B130 - Session PS-TuM

Plasma Diagnostics and Sources I

Moderators: Tetsuya Tatsumi, Sony Semiconductor Solutions Corporation, Geun Young Yeom, Sungkyunkwan University, Korea

8:00am **PS-TuM-1 Optimizing Power Delivery in a Pulsed Inductively Coupled Plasma Using Set-Point Impedance Match and Frequency Tuning**, ***Chenhui Qu***, University of Michigan; *J Brandon, C Smith, S Shannon,* North Carolina State University; *D Coumou, S White,* MKS Instruments; *M Kushner,* University of Michigan

During pulsed operation of inductively coupled plasmas (ICPs) using radio frequency (RF) power, the resistance of the plasma can change by factors of 10-100 while the reactance can change sign (from negative to positive during an E-H transition). These changes in impedance add to the intrinsic impedance of the reactor. The components in the impedance matching network (IMN) hat interface the reactor to the power supply typically cannot be changed rapidly enough to track the plasma transients and maintain a match. The IMN is then tuned to match at a specified time during the plasma pulse, a process called set-point-matching. With feedback control systems and wideband amplifiers, it is possible to make a real-time adjustment of the frequency of the RF oscillator to provide a real-time impedance matching.

In this work, impedance matching to a pulsed ICP plasma was computationally and experimentally investigated using set-point-matching and frequency tuning. The Hybrid Plasma Equipment Model (HPEM) was used for the computational investigation with results compared to experiments performed on the ICAROS reactor, consisting of a four-turn solenoidal coil powering a cylindrical ICP having a 5 cm radius and 15 cm height. The ICPs were operated in Ar at pressures of 1-50 mTorr. The RF power (frequencies from 10-14 MHz) was pulsed modulated at 10 kHz pulsed power with 50 W amplitude and 50% duty cycle.

Set-point-matching early during the pulsed cycle produces more rapid rise in the plasma density while having high reflective power late in the pulse. Set-point-matching late during the pulsed cycle produces a slow rise in the plasma density while having low reflective power late in the pulse. Frequency tuning is able to functionally match during the entire pulsed cycle. For example, when the IMN was set to match in the early period during the pulse, frequency tuning within a range of ±2 MHz is able to maintain reflected power to be less than 10%, and functionally zero late in the pulse. Combinations of set-point-matching and frequency tuning are able to match pulsed-operation over a wide range of power, pressure, pulse repetition frequency and duty cycle.

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8:20am **PS-TuM-2 Compact Surface Wave Plasma Source**, *G Panici,* ***David Ruzic***, *D Qerimi, D Barlaz,* University of Illinois at Urbana-Champaign; *B Jurczyk,* Starfire Industries LLC

Surface wave plasmas have been used increasingly in industrial applications due to their high electron densities ($10^{11\text{-}12}$ cm^{-3}), high radical densities ($10^{13\text{-}14}$ cm^{-3}), and low electron temperatures (1-5 eV). Typical radical creation systems generate radicals at a distance from where they are needed. A compact surface wave plasma source can generate and deliver the radicals at the surface where etching is desired. The source is microwave driven, operating in the hundreds of megahertz. At these frequencies, the wave reflects at the plasma sheath boundary due to the cutoff frequency of electrons once the plasma reaches critical density (typically a microsecond). The power is then largely deposited in the sheath, creating a plasma along the surface. In addition to local radical delivery, they can operate from fractions of millitorr to 10 torr and utilize a variety of antenna geometries (lines, arcs, plates).

A compact surface wave plasma source was characterized over a large range of pressures. The electron densities, electron temperatures and radical densities were measured as a function of distance from the plasma source. Multiple gas species were used to investigate the influence on plasma and radical parameters. These results will be presented.

8:40am **PS-TuM-3 Overview of Linear Plasma Sources as Applied to Ribbon ion and Plasma Beam Processing of Scanned Substrates**, ***Peter Kurunczi***, Applied Materials, Varian Semiconductor Equipment **INVITED**

Ribbon beams of ions and plasmas offer unique capabilities within scanned substrate processing techniques such as ion implantation, physical and chemical surface modification, etching and deposition. This talk will cover an overview of ribbon beam technology used across a range of applications, with examples from processing of silicon, glass, and flexible plastic substrates used within semiconductor, display and roll to roll web industries. Various types of linear plasma sources and methods of ion beam generation will be discussed. Of note are the very different types of plasma ion sources used, from magnetized dc discharges driven by thermionic cathodes to rf generated plasmas. The audience is encouraged to extrapolate to how ribbon beams can be used for their specific material processing needs, for example as applied to this year's symposium theme of "Materials, Technologies and Processes for Energy Transition"

9:20am **PS-TuM-5 Online Diagnostics of Non-Thermal Plasma Nanoparticle-Laden Systems by Ion Mobility Spectrometry**, ***Xiaoshuang Chen***, *S Ghosh, D Buckley,* University of Minnesota, Minneapolis; *M Sankaran,* Case Western Reserve University; *T Seto,* Kanazawa University, Japan; *U Kortshagen, C Hogan,* University of Minnesota, Minneapolis

Non-thermal plasmas (NTPs) have been shown to be capable of producing chemically-pure, size-controlled, low polydispersity, crystalline nanoparticles (NPs). Vapor-phase precursors are dissociated in the NTP reactor, typically by high electron energy impact, leading to nucleation and NP growth in the gas phase. A prevailing thought is that the NPs are unipolarly charged negative in the plasma, which mitigates coagulation and promotes surface growth, yielding low polydispersity NPs. In this study, we apply ion mobility spectrometry (IMS) as an online diagnostic to NTPs to address particle charging and coagulation.

In the first part of the study, we present an IMS system developed for low pressure to characterize Si nanocrystals synthesized in a radio-frequency (RF), capacitively-coupled NTP operating at 2 Torr. A uniquely designed low-pressure differential mobility analyzer (LPDMA) coupled with an electrical detector was utilized to measure NP size distributions in real time. Via the Twomey-Markowsi inversion approach, we have demonstrated, for the first time, that DMAs can be utilized to analyze NPs synthesized in low-pressure NTPs. Excellent agreement was found between the size distributions measured and inverted by LPDMA system and inferred from TEM images. Importantly, we found that at the outlet of the NTP flow tube reactor, Si NPs are bipolarly charged with nearly identical size distribution functions for both negatively and positively charged NPs. Furthermore, NPs are modestly aggregated, implying that the decharging of NPs exiting the plasma reactor from highly negative charge states to a bipolar charge distribution likely drives aggregation on the plasma boundary.

In the second part, ion-mobility mass-spectrometry (IM-MS) method was implemented to study the morphology of as-synthesized carbon-coated Ni NPs generated in an atmospheric-pressure DC microplasma. Sequentially, NPs were sampled by a DMA that classifies NPs by their mobilities, and an aerosol particle mass analyzer (APM), which separates NPs by masses. The concentration of size-mass classified NPs was measured by a condensation particle counter (CPC) downstream of the DMA-APM system, yielding two-dimensional (2D) size-mass distribution function. The shape and location of the sampled NPs on the 2D contour plot reveal their morphologies and extent of aggregation. Utilizing fractal theory to describe particle mobility, particle morphologies were described quantitatively. Our results demonstrate that Ni NPs leaving the plasma reactor are aggregated in chain-like, low fractal dimension aggregates, which are also verified by TEM images.

9:40am **PS-TuM-6 Experiment-Model Comparisons in Capacitively Coupled Plasmas at Moderate Pressures for Argon, Helium and Nitrogen**, ***David J. Peterson***, North Carolina State University; *T Koh, T Chua, W Tian, K Bera, S Rauf, P Kraus,* Applied Materials, Inc.; *S Shannon,* North Carolina State University

Discharge parameters including electron density, effective collision frequency, effective electron temperature, voltage & current characteristics and sheath thickness around the probe are measured over different pressures and powers ranging from 0.01-4.0 Torr and 10-100 W in Ar, He and N2 plasmas. Fully floating hairpin resonator probes are used in a parallel plate capacitively coupled radiofrequency (rf) discharge driven at 13.56 MHz with a gap distance of 1 inch. Probe measurements are made in the axial and radial directions. Effective collision frequency is measured

using the resonance full width half max. Effective electron temperature can determined from the effective collision frequency through the plasma conductivity equation but must assume an electron energy distribution function (EEDF). Probe sheath thickness is measured using a time resolved measurement system capable of ~5 ns time resolution. High temporal resolution is utilized to measure rf phase resolved electron density and effective collision frequency. Measurements indicate the presence of enhanced ionization rates near the powered sheath edge in the collisional regime. Electron density peaks at the discharge center at lower pressures, <100 mTorr, and begins to shift towards the powered electrode at higher pressures due to a measurable DC self bias that is known to occur for geometrically asymmetric discharges. The influence of axial probe spatial resolution and approaches for providing sufficient probe isolation from ground are discussed. Spatial profiles of plasma parameters along with voltage & current characteristics are compared with 2-dimensional fluid plasma simulation results. Detailed model-experiment comparisons play an important role in understanding plasma chemistry mechanisms for these low temperature plasmas at moderately high pressure. All analysis and data acquisition is done with python scripts which are freely available to the public.

11:00am **PS-TuM-10 Optical and Mass Spectrometric Measurements of O_2 and NF_3 Dissociation in a Low Frequency, High Density, Remote Plasma**, ***Hanyang Li***, *Y Zhou, V Donnelly,* University of Houston*; J Chiu, X Chen,* MKS Plasma & Reactive Gas Solutions

Remote plasma sources are widely used in many applications, such as chamber cleaning and flowable chemical vapor deposition (FCVD). Processes using remote plasmas are purely chemical in nature, since there are no ions present. In such processes, it is desirable that the dissociation rate of the feed gases in the plasma source be as high as possible, while recombination rates of reactive species on the way to a downstream chamber should be minimized. Only a few studies have been reported on low frequency, high pressure, very high density remote plasma sources. In this paper we present results on radical densities and gas dissociation fractions for a 400 kHz toroidal transformer-coupled plasma source (MKS Instruments), operating at a power density of 5 – 50 W/cm^3 with feed gases mixtures of O_2 or NF_3 with Ar, and pressures of 0.4 or 2.0 Torr. The radical densities and feed gas dissociation percentages in the plasma were measured by optical emission spectroscopy (OES), combined with Ar actinometry. Plasma products flow into an anodized Al downstream chamber that is probed by vacuum ultraviolet (VUV) absorption spectroscopy and line-of-sight molecular beam mass spectrometry, allowing radical and stable species number densities to be determined in the plasma source as well as in the downstream chamber. The dissociation of O_2 and NF_3 was found to be roughly from 60% to 10% with the rise O_2% in plasma and >95% in the plasma source (via Ar actinometry with O and F) and not very dependent of flow rate. Midway across the downstream chamber, substantial recombination of O to form O_2 (via VUV O_2 absorption) occurred; the O/O_2 ratio was a strongly increasing function of increasing flow rate. At the back wall of the downstream chamber, O has nearly completely recombined to O_2 (mass spectrometry), even at the highest flow rate. NF_3 is completely dissociated and does not reform in the downstream chamber; no NF or NF_2 was detected. F was found to be mostly recombined to for F_2 at the back of the downstream chamber. The F_2, F and N_2 product absolute number densities confirmed the 3:1 F:N mass balance of the NF_3 feed gas. The gas temperature at the back downstream chamber was also measured by mass spectrometry, and was found to be 450K for 95% NF_3/Ar at a flow rate from 200 sccm to 600 sccm and 2.0 Torr.

11:20am **PS-TuM-11 A Combined Experimental and Modeling Study of Reactive Vapor-nanoparticle-plasma Interactions in a Dusty Atmospheric-pressure Plasma**, ***Nabiel Abuyazid***, Case Western Reserve University*; X Chen,* University of Minnesota, Minneapolis*; D Mariotti, P Maguire,* University of Ulster, UK*; C Hogan,* University of Minnesota, Minneapolis*; M Sankaran,* Case Western Reserve University

Low-temperature (non-thermal), atmospheric pressure plasmas are characterized by several important fundamental and technological advantages for the gas-phase synthesis of nanoparticle materials. However, the effect of the particles on these plasmas remains poorly understood. It is generally accepted that nanoparticles acquire charge, typically negative, which leads to a reduction in the electron (plasma) density. The degree of reduction is not known and experimental measurements are challenged by several issues. One, plasmas operated at atmospheric pressure are small in size (~1 mm) and probes cannot be easily introduced into the plasma volume. Two, there are strong gradients in the plasma volume as the precursor vapor is dissociated and nanoparticles nucleate and grow, and the effect of particles on the plasma must be decoupled. Three, the material could have other effects on the plasma, for example by undergoing further reactions or by vaporizing after particle formation, that must also be isolated or avoided.

We present a tandem, atmospheric-pressure plasma system that separates a first “reactive” plasma, where the precursor vapor is dissociated leading to particle growth, from a second “dusty” plasma, where the effect of particles on the plasma can be studied. Two non-contact methods, an external electrical conductivity probe (Impedans Octiv Poly) and spectroscopy are applied on the dusty plasma to monitor changes in the electron density. We focused our study on carbon which has a relatively high boiling point and should be chemically stable within the plasma environment. The measurements show that electron densities decrease as expected upon the introduction of the nanoparticles into the second plasma at all powers. For example, at a power of 50 W, the electron density decreased from 4.0 x 10^{14} cm^{-3} for a pure Ar plasma to 3.6 x 10^{14} cm^{-3} for a dusty Ar plasma with a total particle concentration of about 4.0 x 10^6 particles/cm^3. Monte Carlo simulations were carried out in support of experiments and showed that by preferential negative charging, particles in plasmas can reduce bulk electron concentrations. We will also discuss the effect of residual hexane vapor and possible particle evaporation on plasma properties.

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