Thursday Morning, October 23, 2008

Plasma Science and Technology
Room: 304 - Session PS1-ThM

Atmospheric Plasma Processing and Micro Plasmas

Moderator: J. Hopwood, Tufts University

8:00am **PS1-ThM1 Plasma Diagnostics in Microdischarges Using Laser Scattering**, ***S.G. Belostotskiy****, *V.M. Donnelly*, *D.J. Economou*, University of Houston, *N. Sadeghi*, Universite J. Fourier de Grenoble, France

Laser scattering experiments were performed in high pressure (100s of Torr) DC microdischarges operating in argon or nitrogen. Laser Thomson Scattering (LTS) and Rotational Raman Scattering were employed in a novel, backscattering, confocal configuration to measure important plasma parameters. LTS allows direct and simultaneous measurement of both electron density (n_e) and electron temperature (T_e). LTS experiments in microdischarges are challenging because of the low signal and excessive stray light. Measurements were performed at the center of the gap of a parallel plate slot-type microdischarge with plate separation of 600 microns. This location corresponded to the positive column of the DC microdischarge. For 50 mA current and over the pressure range of 300 – 700 Torr, measurements yielded $T_e = 0.9 \pm 0.3$ eV and $n_e = (6 \pm 3)\cdot 10^{13}$ cm^{-3}, in reasonable agreement with the predictions of a mathematical model. In order to obtain absolute values of the electron density, calibration of the Thomson scattered intensity was carried out using Raman scattering in nitrogen. This Rotational Raman spectroscopy was also employed to measure the gas temperature (T_g) in nitrogen DC microdischarges. Gas temperatures were determined by matching experimental spectra to synthetic spectra obtained by convolution of theoretical line intensities with the apparatus spectral resolution, with T_g as the adjustable parameter. Measurements were performed for a set of N_2 pressures (P = 400 – 600 Torr) and over the current range of 5 – 30 mA. In the center of the interelectrode gap, T_g changed from 450 ± 40 K at 5 mA to 740 ± 40 K at 30 mA. The gas temperature was nearly independent of pressure within the error of the experiment. Advantages and limitations of the laser scattering techniques employed will also be discussed.

8:20am **PS1-ThM2 The Effect of Excitation Frequency on Microplasmas**, ***J. Xue****, *J. Hopwood*, Tufts University

In the microwave band, higher excitation frequency is found to enhance microplasma generation. The microplasma is formed by a split-ring resonator (SRR) consisting of a half-wavelength microstrip transmission line formed into a ring with a micromachined discharge gap.[1] The SRR plasma can be operated from 0.1 to 760 Torr with less than 0.5 W of power in He and Ar. Typically, microplasmas have been generated with DC, AC, RF, and microwave power. One unanswered fundamental question, however, concerns the effect of frequency on microplasma generation. The excitation frequency of capacitively coupled plasma has been discussed by Surendra and Graves.[2] This early work suggests that plasma density scales as the square of the applied excitation frequency. That work focused on large-scale plasma at low pressure and the excitation frequency was limited to less than 120 MHz. This paper presents plasma impedance analysis of three microplasmas operating at excitation frequencies of 450 MHz, 900 MHz, and 1.8 GHz. The electron density and sheath capacitance of the microdischarges are extracted from the plasma impedance. Experimentally, these three SRR's are fabricated on microwave laminate (Rogers, RT/Duroid 6010LM) with identical microstrip widths (1 mm) and discharge gaps (200 μm). The radii of the rings are scaled by 1/f and the smallest radius is 5mm at 1.8 GHz. To determine the plasma impedance, the microwave reflection coefficient is measured as a function of frequency while maintaining a constant microwave power absorbed by the plasma. Using the method in Ref. 1, the microplasma impedance is found by fitting the theoretical microwave reflection coefficient to the measured reflection coefficient. The results show that microplasmas generated by higher frequency resonators have a lower plasma resistance. The extracted electron densities in argon microplasma at 760 Torr are estimated as 2.4, 6.0, and $8\text{x}10^{13}$ cm^{-3} for the 450 MHz, 900 MHz, and 1.8 GHz SRR, respectively. The imaginary part of the plasma impedance provides a model of the plasma sheath capacitance. This data shows a diminishing sheath impedance at high frequency which is responsible for improved electron density.

[1] F. Iza and J. Hopwood, Plasma Sources Sci. Technol. 14, 397 (2005).
[2] M. Surendra and D. B. Graves, Appl. Phys. Lett. 59, 2091 (1991).

***** PSTD Coburn-Winters Student Award Finalist

8:40am **PS1-ThM3 Generations, Characterizations and Applications of Microplasmas Operated in Atmospheric Gases and Artificial Media**, ***K. Tachibana***, Kyoto University, Japan **INVITED**

Microplasmas of sub-millimeter to micrometer scales can be operated in high pressure gases or high density media with a choice of single or integrated usage. The electron density n_e of a typical microplasma lies in the range of 10^{12} to 10^{15} cm^{-3} even though the ionization degree is rather small. As for the electron temperature T_e, it shows non-equilibrium natures inherently due to the short residence time in small space or short duration of pulsed discharge in the generation. Taking the advantage of these non-equilibrium properties, microplasmas have been applied to various purposes such as material syntheses, surface treatments, environmental and biomedical issues, etc. In this talk, I would like to introduce some examples of new schemes of microplasma sources with their characterizations by various diagnostic methods; we have been applying optical emission spectroscopy (OES), laser absorption spectroscopy (LAS), and laser-induced fluorescence spectroscopy (LIF) for the measurements of excited and ionized species as well as THz time-domain spectroscopy (TDS) and infrared CO_2 laser heterodyne interferemetry for plasma parameters. In addition to the microplasma generation in usual gas phase, we have been trying to use atmospheric gases with liquid vapors (mists) and aqueous solutions with micro bubbles as artificial media of microplasmas under controlled (characterized) conditions. As one of the examples, by using a fabric electrode assembly weaved with insulated wires, we have succeeded in the generation of microplasmas in H_2 or O_2 bubbles produced underwater by electrolysis. Those results will be explained together with some examples of their potential applications.

9:20am **PS1-ThM5 Atmospheric Dielectric Barrier Glow Discharges at High Overvoltage**, ***B.D. Schultz***, *A.R. Martin, M.A. Ray, G.E. McGuire, W.M. Hooke*, International Technology Center

Atmospheric dielectric barrier plasma glow discharges in pure nitrogen gas have been generated under overvoltage conditions produced with a custom high voltage source. A voltage rise time of 25 ns at 20-30kV is readily achieved by the source and is sufficient to create overvoltage conditions in excess of three times the DC breakdown voltage of nitrogen. These large overvoltage conditions occur because the rise-times required to achieve peak voltage are shorter than the lag time between the pulse crossing the threshold voltage and the onset of a discharge. Overvoltage conditions prior to discharge have been predicted to produce significantly higher average electron energies in the discharge and to produce high instantaneous power densities. Experimentally current densities have been achieved well in excess of 10 A/cm^2 for homogeneous glow discharges of pure nitrogen gas at atmospheric pressure with total pulse currents of 1 kiloamp having been obtained. The overvoltage potential on the electrodes enables manipulation of the reduced electric field, but additional control can also be garnered through increases in the gas temperature and/or decreases in the chamber pressure along with the applied overvoltage. This paper will emphasize the correlation between the overvoltage conditions, the dielectric material properties, temperature, and small deviations in pressure to the electrical charge transfer, optical properties, and propagation mechanisms of the glow discharge. This work was supported in part by ARL and AFRL.

9:40am **PS1-ThM6 Decoloration of Organic Dyes by Bipolar Pulsed Electrical Discharge in Aqueous Solution**, ***Š. Potocký***, *N. Saito, O. Takai*, Nagoya University, Japan

During recent years plasma systems in liquid have become a topical interest. They open new possibilities in wastewater treatment. Those systems are able to produce highly active species which finally results in conversion of the organic to innocuous materials. Nevertheless, the drawback of such system meets with the requirement of very high voltage for sustaining the plasma discharge and relatively low energy efficiency. Another important process is the erosion of the electrodes that limits the operating lifetime or leads to the pollution of treated water with metal particles released from the electrodes. We demonstrate operation of solution plasma process under relatively low discharge voltage (below 4 kV) and two different plasma regimes with pulse energies in mJ range using a high frequency bipolar pulsed DC power supply. It can be operated up to the repetition frequency of 30 kHz with a pulse width range from 2 μs to 10 μs and a maximum voltage and current of ±6 kV and 7 A, respectively. The oxidative decoloration of organic dyes by the bipolar pulsed discharge plasma between needle-to-needle electrodes in water solution has been investigated in two discharge modes: (i) corona, (ii) spark/streamer mode. Ratio between H-alpha and hydroxyl radical emission line intensity differ by two orders of magnitude in those two modes even for high value of solution conductivity (1 mS/cm) which is close to typical value of a

wastewater. The current-voltage characteristics of the system together with an optical emission spectroscopy of plasma discharge were used to characterize regimes of plasma discharge operation. Analysis of generated hydrogen peroxide concentration by colorimetric method using titanium reagent and the absorption spectroscopy was performed. Measurement of electrodes erosion and metal concentration (by inductively coupled plasma mass spectroscopy) due to solution plasma process was also carried out indicating obvious difference in two modes of plasma discharge operation.

10:40am **PS1-ThM9 Surface Modification of Ultra High Molecular Weight (UHMW) Polyethylene Films Using Atmospheric Pressure Dielectric Barrier Discharges**, ***D.D. Pappas***, *K.E. Strawhecker, A.A. Bujanda*, United States Army Research Laboratory

In this work, dielectric barrier discharge (DBD) plasmas, operating in nitrogen, air and helium-oxygen at atmospheric pressure, are used to modify the surface properties of ultra high molecular weight polyethylene films. The imposed changes of the hydrophilicity, chemical composition and roughness of the surface appear to have a dependence on the DBD operating parameters such as processing duration and discharge power as well as the nature of the gas being used for the plasma treatment. Contact angle and X-ray photoelectron spectroscopy (XPS) data reveal that in all cases the plasma exposed surfaces exhibit improved wettability that can be attributed to the mild oxidation of polyethylene as confirmed by XPS analysis. Atomic force microscopy (AFM) results show that longer processing duration and higher oxygen concentration are key for increased surface roughness, a factor affecting the adhesion properties of the film. Standard lap-shear evaluations reveal that plasma treatments may lead to significant increases in the bond strength of polymer films and metallic/polymeric substrates and changes in modes (adhesive vs. cohesive) and loci of failure. The plasma treatments increase the mechanical interlocking and frictional energy dissipation effects when bonded to a substrate. Changes in other mechanical properties are also investigated. Most importantly, this uniform modification occurs within a few seconds of exposure, time comparable to continuous on-line industrial processing.

11:00am **PS1-ThM10 The Discharge Characteristics of an Industrial Scale Atmospheric Pressure Uniform Plasma Processing System**, ***W. Graham***, *D. Della Croce, L. Schaper,* Queens University Belfast, Northern Ireland, *L. O'Neill, A.M. Hynes*, Dow Corning Plasma Solutions, Ireland

Time and space-resolved electrical, optical and imaging characterisation of a commercial, 1800cm^2 atmospheric pressure plasma system, operating with polymer film is reported. . The system is based on a dielectric barrier discharge operated in air with flowing helium. The system is optimized for plasma treatment, rather than the physical appearance of the plasma. The Dow Corning Plasma Solutions LabLine™ system establishes discharges in two back to back, identical 340mm x 300mm transparent, electrode structures each with an inter-electrode gap of 5mm. The driving power supply produces a sinusoidal voltage, of up to 20 kV peak to peak, at frequencies of around 20 kHz. This is applied to the two internal electrodes. The outer electrodes are grounded. Helium is introduced from the top of the electrodes. Polyethylene Terephthalate (PET) polymer film could be suspended in the centre of the electrode gap, parallel to the glass dielectrics. Standard high voltage and current measurement techniques were used monitor the voltage applied to the electrodes and the current (Id) drawn through the power cable to the electrode assembly. A fast photomultiplier tube was used to measure the temporally resolved emission from the discharge while the spatially and temporally resolved behaviour of the discharge was studied by imaging the electrodes and the electrode gap onto a gated ICCD. The imaging indicates that with or without polymer film present, static or moving, radially uniform discharges, persisting for a few microseconds, are consistently created at the same phases of the applied voltage. The number of discharges increases with increasing input power and hence applied voltage. The structure of the discharge emission is suggestive of that of an atmospheric pressure being most intense at the cathode and showing evidence of a dark space and much less intense emission beyond that. At higher input powers, when the applied voltage considerably exceeds the initial breakdown voltage, these discharges occur so frequently that they sustain and enhance these structured discharges for periods of up to 25 μs. In the presence of the polymer film the discharge was generally more intense in the region occupied by the film and always produced emission between the polymer and the cathode. The authors wish to thank Dr. D. Dowling and B. Twomey of U.C. D. for their support and assistance and gratefully acknowledge EPSRC and Dow Corning support for of D. D. C.

11:20am **PS1-ThM11 Influence of Air and Water Vapour Contaminations on the Atmospheric Pressure PECVD of Fluorocarbon Thin Films**, ***F. Fanelli***, Univeristy of Bari, Italy, *R. d'Agostino, F. Fracassi*, University of Bari, Italy

Low pressure plasma-enhanced chemical vapour deposition (PECVD) of fluorocarbon films has been extensively studied in the last decades. Very recently atmospheric pressure dielectric barrier discharges (DBDs) have been addressed as an attractive route towards the deposition of fluoropolymers, nevertheless, the utilization of this approach is still a challenge. Research efforts should be devoted to evaluate if DBDs can actually be advantageous compared to low pressure plasmas; for this purpose, besides the fundamental investigation of fluorocarbons fed DBDs, it is also important to gain insights into the influence of contaminants such as air and water vapour. The presence of these contaminants into the atmospheric pressure reactor could have, in fact, serious detrimental effects on the overall deposition process because it might result in a drastic decrease of the F/C ratio of the films, in the uptake of oxygen and nitrogen as well as in deposition rate reduction. On the other hand, the knowledge of the highest level of contamination compatible with an acceptable process performance and consequently the possibility of depositing fluoropolymers in "contaminated" environments could allow to reduce the cost of plasma processes and reactors. For these reasons we decided to evaluate the influence of air and water vapour contaminations on the PECVD of fluoropolymers in atmospheric pressure cold plasmas. Controlled amounts of air and water vapour have been added to a DBD fed with argon-hexafluoropropene (Ar-C_3F_6). The discharge regime has been clarified by electrical measurements, while film characteristics have been studied by FTIR, XPS, WCA measurements and SEM. Gas phase has been investigated by optical emission spectroscopy and the stable species contained in the gas effluent have been analyzed using gas chromatography coupled with mass spectrometry, in order to have indications on the reactive fragments generated inside the discharge. The results obtained in this work show that Ar-C_3F_6 DBDs allow to deposit coatings with a deposition rate of 56 nm/min and a XPS F/C ratio of 1.7. Contaminants addition causes a slight variation of the F/C ratio and a decrease of the deposition rate. In particular, if the [Air]/[C_3F_6] and [H_2O]/[C_3F_6] ratios in the feed are kept below 0.25 and 0.125, respectively, the variation of the F/C ratio is negligible and the deposition rate remains higher than 45 nm/min.

11:40am **PS1-ThM12 Characterization of Atmospheric Pressure Plasma with Arc-free and Antistatic Plate for Flat Panel Displays, Solar Cells, Semiconductor, and Nano-biology Processing**, *K.H. Lee, H.R. Lee, Y.J. Park,* ***S.I. Jun***, PSM America Inc.

We have characterized the parametric and functional properties of Atmospheric Pressure (AP) Plasma System with newly designed Arc-free and Antistatic Plate to provide uniform and arc-free AP plasma on substrates such as glass, wafers, and any flexible substrates for improving surface treatments. The AP Plasma System showed very highly efficient capabilities of removing organic contaminants for flat panel displays, semiconductor, and solar cell processing with very high throughputs and very low running coat provided by in-line layouts and usage of CDA (cold dry air) not using expensive gas such as helium. The AP treated substrates and thin films showed drastic lowering of the contact angles; 43 degree to 5 degree for bare glass substrate and 70 degree to 8 degree for ITO thin films. The etch rate non-uniformity of ITO films on 730x920mm glass substrate was improved from 8.8% to 7.5% after AP Plasma treatment with the Arc-free and Antistatic Plate. Besides these conventional applications of AP Plasma, we have proposed newly emerging applications, nanobiotechnologies. The AP Plasma System showed much improved performances in (1) direct contact with living tissue, (2) wound treatment with living tissue sterilization, (3) promoting blood coagulation, (4) skin flora sterilization, and (5) treatment of human melanoma, macrophages, and leishmania major promastigote cell line.

Authors Index

Bold page numbers indicate the presenter