Plasma Science and Technology
Room: 210A - Session PS+SE-FrM

Atmospheric Pressure Plasma Processing II
Moderator: Souvik Ghosh, Case Western Reserve University

9:00am **PS+SE-FrM3 Scaling Atmospheric Pressure Plasma Sources for Manufacturing-Scale Applications**, ***Steven Shannon***, North Carolina State University **INVITED**

Material processing using plasmas at atmospheric pressure presents one of the great areas of future growth for the Plasma Science and Technology Division of the AVS. Extension of plasma processing to atmospheric pressures (and ironically eliminating the need for "vacuum") provides two key advantages in processing. The first is the reduced cost and increased throughput that could be realized in equipment that does not require high vacuum design, load locks, etc. The second, and more significant, are the new applications that plasma science can now contribute to including water treatment, soft materials processing, processing of non-vacuum-friendly materials, and medicine.

Atmospheric plasma systems for manufacturing have the same high volume integration challenges that low pressure plasma systems do. These are scalability and throughput. These systems need to be able to process large areas (or volumes, depending on the application). This throughput scaling presents a challenge especially when some level of homogeneity in process both within a sample and from sample to sample are required. Maintaining this process uniformity over a large area is further complicated by the need to maintain a high rate of reactive specie production, especially when the standard solution of "turn up the power" results in typically undesirable instability and heating in these higher pressure discharges.

At NCSU, researchers have developed an atmospheric plasma source that seeks to address these scale-up concerns of size and reactant production. This coaxially driven source can be driven in the VHF range of frequencies (60MHz - 200MHz, with 162MHz used in the work shown here) with a plurality of feed gases not requiring noble gas dilution. The VHF heating combined with source circuit design produce a volume glow with power densities ranging from $1W/cm^3$ to $20W/cm^3$. The unique source design enables operation of an RF driven / DC grounded electrode that enables delivery of gas and liquid precursors through the electrode surface. This enables the introduction of liquid precursors into the active plasma region while maintaining a stable volume glow. Multiple sources can be run in parallel for larger volume operation, and scalability has been demonstrated. Reactive specie production has been quantified for air plasmas and water plasmas (with water delivery from the powered electrode). The water electrode configuration yields OH concentrations over $10^{15}/cm^3$ in the active plasma region.

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9:40am **PS+SE-FrM5 Improving of Harvest Period and Crop Yield of *Arabidopsis Thaliana L.* using Nonthermal Atmospheric Air Plasma**, ***Kazunori Koga***, *T. Sarinont, T. Amano, H. Seo, N. Itagaki, M. Shiratani*, Kyushu University, Japan

Nonthermal atmospheric plasmas have been widely used for biomedical applications [1-3]. Growth enhancement of plants is one of the important applications of such plasmas. Here we have studied effects of atmospheric air plasma irradiation to seeds of *Arabidopsis thaliana L.* on the harvest period and crop yield. Experiments were carried out using a scalable DBD device [2, 3]. The device consisted of 20 electrodes of a stainless rod of 1 mm in outer diameter and 60 mm in length covered with a ceramic tube of 2 mm in outer diameter. The discharge voltage and current were 9.2 kV and 0.2 A. 20 seeds of *Arabidopsis thaliana L.* were set 3 mm below the electrodes. After 3 minutes plasma irradiation, they were grown on rockwool until the harvest stage. The harvest period is defined as time from the beginning of cultivation to the first seed production. To evaluate the growth enhancement ability of the plasma irradiation, the area of two primary leaves of eight plants, namely 16 leaves, was measured every three days. The whole experiment was repeated 3 times with similar results. Data were analyzed by two-way analysis of variance. The harvest period without and with plasma irradiation are 71.31±5.63 and 66.53±3.82 days. The seed weight without and with plasma irradiation are 0.0201±0.0024, 0.0225±0.0016 mg/seed. The leaf area after 28 days from the beginning of cultivation without and with plasma irradiation are 0.049±0.011 cm^2 and 0.064±0.014 cm^2. These measured values without and with plasma irradiation are statistically significance different at $\alpha = 0.05$ ($P < 0.05$). Plasma irradiation brings about 7% shorter harvest period, 12% higher seed weights and 30% larger primary leaves, compared to those without plasma irradiation. Thus, plasma irradiation to seeds is a cost effective and environmental friendly method for improving of harvest period and crop yield of plants. The growth enhancement mechanism will be discussed in the presentation.

[1] J. Raiser and M. Zenker, J. Phys. D, **39**, 3520 (2006).

[2] T. Sarinont, et al., JPS Conf. Proc. **1**, 015078 (2014).

[3] S. Kitazaki, et al., Curr. Appl. Phys., **14**, S149 (2014).

10:00am **PS+SE-FrM6 Numerical Modelling of Atmospheric Pulsed Streamers over Water; Electrodynamics at the Interface**, ***Alex Lindsay***, *S. Shannon*, North Carolina State University, *D.B. Graves*, University of California at Berkeley

There is significant interest in characterizing interactions between atmospheric plasmas and water for applications in medicine, water decontamination, distributed farming, etc. In one particular example members of the community are investigating replacement of invasive electroporation for drug delivery and gene therapy with low-power atmospheric plasma devices. Although the mechanism by which electric fields create conductive pathways for drug delivery into cells is generally known, the mechanism by which plasmas create those liquid-phase electric fields is an active area of research. Pioneering work in [1] has done much to advance our understanding, but more work remains. We wish to present modeling tools that are open to the community in the hope that this will enhance development of the tools, scrutiny and reproducibility of numerical results, and the pace at which plasma-liquid research is conducted. By developing open community tools, we hope to reduce the time-waste that comes from different groups re-inventing the wheel to study similar phenomena. With those motivations, we consider both finite-volume and finite-element discretizations of the Poisson and continuity equations governing electrodynamics in the gas and liquid phases. For a first pass, a local-field approximation is used to study streamer propagation in a point-to-plane configuration with water serving as the planar electrode. Different methods for stabilization (e.g. inconsistent vs. consistent, streamline and/or crosswind) as well as markers for mesh adaption (potential, electron density curvatures) are considered.

[1] Babaeva et. al. J. Phys. D: Appl. Phys. 47 (2014) 235201

10:20am **PS+SE-FrM7 Application of Atmospheric Pressure Plasma treatment on Carbon Fiber Reinforced Plastics for Adhesive Bonding**, ***Timo Hofmann***, *J. Schäfer*, Bundeswehr Research Institute for Material, Fuels and Lubricants, Germany, *T. Löbel*, German Aerospace Center (DLR), *T. Meer*, Airbus Group Innovations, *J. Rehbein, J. Holtmannspötter*, Bundeswehr Research Institute for Material, Fuels and Lubricants, Germany

The demand for environmentally friendly means of transportation has led to a strong increase in the use of carbon fiber reinforced plastics (CFRPs). Joining of CFRP structures is currently performed using rivets and bolts. In order to exploit further weight-saving potential, the usage of adhesive bonding is investigated.

An important key factor for the success of adhesive bonding is the surface pre-treatment of the adherents. In this contribution, CFRP surfaces were treated using Atmospheric pressure plasma jet (APPJ) as a method to clean the samples and to improve adhesion by creating surface functional groups.

We present a detailed investigation of the surface morphology and the composition of CFRPs before and after treatment with APPJ. The CFRP surfaces were examined using a combination of Field-Emission Scanning Electron Microscopy (FE-SEM), Atomic Force Microscopy (AFM), Energy-Dispersive X-ray spectroscopy (EDX), and X-Ray Photoelectron Spectroscopy (XPS). Destructive tests were carried out to determine the adhesive strength and the failure mode as a function of APPJ.

We demonstrate that APPJ-processes can be used to form structural and long term stable bonds. Our results show that through the combination of analytical techniques and destructive tests it is possible to develop an understanding of the processes at the surface and to optimize the plasma treatment process.

10:40am **PS+SE-FrM8 Atmospheric Plasma Deposition of Transparent Organosilicate Multifunctional Coatings on Plastics in Air**, ***Siming Dong**, Z. Zhao, R.H. Dauskardt*, Stanford University

Atmospheric plasma deposition is a versatile coating process that enables deposition on large and/or complex shapes in air. The low temperature plasma and solvent free process allows deposition on, and simultaneous functionalization of plastic substrates in a single step. Building on our previous studies, we demonstrate a highly efficient deposition method using two precursors, an inorganic tetraethoxysilane (TEOS) and an organic 1, 5-cyclooctadiene (CYC), to deposit multi-layer organosilicate transparent coatings on poly methyl methacrylate (PMMA) and silicon (Si) substrates with atmospheric plasma deposition in air. The coatings deposition rate, transparency, chemical composition and adhesion with the PMMA substrate were investigated. Using only the TEOS precursor, high density and elastic modulus coatings were deposited on PMMA but with poor adhesion. The addition of the organic CYC precursor allowed controlled incorporation of organic components into the coating molecular network which significantly improved adhesion. The deposition rate increased from ~65 nm/min for the single precursor to ~130 nm/min for the two precursor process. The coatings exhibited ~100% transmittance in the visible wavelength range. FTIR and Raman spectroscopy of the coatings showed that the organic component $(-C-C-)_n$ in the coatings can be incorporated to form an organosilicate molecular network. This incorporation increased the coating deposition rate and also resulted in mechanical plasticity in the coatings. The adhesion of coatings with PMMA increased from ~2 J/m^2 to ~10 J/m^2 and the Young's modulus ranged from 22GPa to 34GPa. Coatings structures including composition and coating thickness to achieve optimized hardness and adhesive properties are reported.

11:00am **PS+SE-FrM9 Atmospheric Plasma Deposition of Anti-Reflection Coatings on Silicon in Open Air**, ***Michael Hovish**, R.H. Dauskardt*, Stanford University

For many modern energy and sensing applications, multilayer optical coatings are an effective way to dramatically improve light collection. Traditionally, such multilayer coatings are deposited on hard substrates using vacuum depositions. Furthermore, traditional vacuum techniques are not easily scalable, due to high costs and poor integration into the manufacturing scheme. Atmospheric plasma deposition has received attention in materials processing due to the ability to deposit functional coatings at room temperature and in open air. Room temperature operation allows for a dynamic range of substrates, both organic and inorganic. In addition to these qualities, atmospheric plasma deposition is a solvent free technique, making it a competitive alternative to sol-gel methods. In our research program, we have successfully shown several material systems which are amenable to atmospheric plasma deposition, including multifunctional organosilicate and metal oxide films. In particular, the solvent-free deposition of metal oxide films at atmospheric pressure and near room temperature provides an attractive platform for the design and fabrication of optical coatings.

We have employed atmospheric plasma to deposit thin, anti-reflection coatings on silicon. Both TaO_x and TiO_x films were investigated as candidates for single layer anti-reflection coatings. Films were optimized for low reflection within the visible wavelengths of light. High purity helium gas was used to transport either tantalum ethoxide or titanium ethoxide vapor into the afterglow of a helium-nitrogen plasma. A high temperature precursor delivery system was used to prevent the condensation of precursor vapors en route to the afterglow. Within the afterglow, the metal-organic compounds undergo molecular fragmentation and redistribution onto the substrate. Deposition rates, chemical compositions, optical properties, and adhesion energies to the substrate were investigated as a function of plasma power and gas composition. Spectral reflectance at 10° from normal was measured to determine the anti-reflection properties of the coatings. Atmospheric plasma deposited films on silicon show excellent anti-reflection properties, with less than 3% reflection loss near 550 nm.

11:20am **PS+SE-FrM10 Polymer Thin Film Deposition using Atmospheric Pressure Single Plasma Jet or Plasma Jet Array from a Plasma Gun Device**, ***Céline Vivien***, IEMN CNRS/Université Lille 1, France, *E. Robert, J.-M. Pouvesle*, GREMI CNRS/Université d'Orléans, France

Plasma Enhanced Chemical Vapour Deposition (PECVD) processes have been used for decades for surface processing in a wide range of industrial applications like semiconductor films, low-k films, barrier diffusion. Thin film deposition is especially of high interest for biomedical applications for the production of protective coatings, adhesion layers, hydrophilic or hydrophobic layers. Up to now, most of used processes are usually realized under low pressure. Actually, there is a great and increasing interest in the development of plasma sources operating at atmospheric pressure. The present work deals with plasma polymerisation of TMDSO and HMDSO with a Dielectric Barrier Discharge plasma jet at atmospheric pressure, the Plasma Gun developed in GREMI. Depending on parameters like voltage, frequency, carrier gas and monomer injection, the deposited polymer appears either as a gel-like coating or a transparent film with fringes. Deposits are characterized by Fourier Transformed IRspectroscopy and contact angle measurements. The precursor used was introduced in both liquid or gaseous state, in case TMDSO and only gaseous state in case of HMDSO. The liquid flow rate was regulated by a peristaltic pump (Ismatec) while the vapour flow was ensured by bubbling nitrogen or oxygen with a fixed flow rate of 10 sccm. Microscope slides and polished Silicon wafer (100) were used as substrates. The polymerized coatings have been obtained at frequencies between 500Hz and 4 kHz, for applied voltages between 14 and 20 kV and exposure times from 1 to 10 minutes. The deposition were realized with plasma tube edge-to-substrate distances ranging from 3 to 12 mm. Profilometer measurements revealed thicknesses comprised between 500 nm and 1.5 µm at the middle of the deposit. Deposited films analyses clearly show the efficiency of this atmospheric plasma-type TMDSO and HMDSO polymerisation and their similarity with those usually realized under low pressure RPECVD. The most interesting deposited films are obtained when the monomer is introduced under gaseous state, the samples clearly showing a better homogeneity. The influence of the transport gas is not evidenced. More experiments and analyses need to be achieved to complete these preliminary results. It must be stressed that multi-spot deposition has been obtained from plasma multi-jet delivered by a single Plasma Gun.

11:40am **PS+SE-FrM11 XPS to Investigating Spatial and Temporal Modification of Polymeric Platforms for Micro-Fluidic Devices**, ***Marshal Dhayal***, CSIR Centre for Cellular and Molecular Biology (CCMB), India

Spatial and temporal changes in surface chemical composition silicon (Si), carbon (C) and oxygen (O) of polydimethyl siloxane surfaces before and after plasma treatment were estimated from quantitative elemental analysis of X-ray photoelectron spectroscopy (XPS) wide scan spectra. Theoretical ratio of Si/C/O in repeating unit $(–[Si-(CH_3)_2-O]_n–)$ of polydimethyl siloxane were calculated and were compared to experimentally obtained ratio for Si/C/O obtained from untreated and plasma treated surfaces used for micro-fluidic devices. The contact angle measurements have shown that surfaces treated by air plasma can recover up to about 50% of its hydrophobic nature in less than 30 min of air exposure. These plasma modified surfaces were functionalized with poly(ethylene glycol) (PEG) silane to obtained polydimethyl siloxane surface as hydrophilic in nature for micro fluidic application. The surface chemistry of PEG-functionalized polydimethyl siloxane substrate has been studied using XPS. These different types of surfaces were used fabricate micro-fluidic devices and effects of surface nature of micro channels on fluid velocity were observed in PEG grafted micro channel in polydimethyl siloxane base micro fluidic devices. The effect of different pH of the fluids on the fluid velocity in polydimethyl siloxane -based micro channel was also studied.

Authors Index

Bold page numbers indicate the presenter