

Wednesday Afternoon, November 2, 2011

Advanced Surface Engineering Division

Room: 104 - Session SE+PS-WeA

Atmospheric Pressure Plasmas

Moderator: H. Barankova, Uppsala University, Sweden

2:00pm **SE+PS-WeA1 An Investigation of the Influence of Hybrid Current Modes on the Plasma Behavior during Plasma Electrolytic Oxidation (PEO) Coating Process on Mg and Mg-Alloys, R.O. Hussein, D.O. Northwood, X. Nie, University of Windsor, Canada**

The increased use of magnesium alloys is considered one of the more promising methods for light-weighting in the automotive industry since, for a given strength level, Mg represents a 57% weight reduction over steel and 8% weight reduction over aluminum. However, due to its high chemical and electrochemical activity, magnesium has poor corrosion resistance in aqueous and other environments. In order for Mg and its alloys to find increased usage, there is a need to surface engineer these materials for improved corrosion and wear resistance. Plasma Electrolytic Oxidation (PEO) is an electrochemical process working at atmospheric pressure that uses an environmentally-friendly aqueous electrolyte to oxidize the metal surfaces to form ceramic oxide coatings which impart a high corrosion and wear resistance. The properties and structure of PEO coatings are dependent on parameters such as substrate metallurgy, composition of the electrolyte and the process conditions including current density, current mode and processing time. In this study we investigated the effect of current mode on plasma temperature and coating properties of PEO coatings formed on pure magnesium and an AM60B magnesium alloy (mass fraction: Al 5.6–6.4%, Mn 0.26–0.4%, Zn \leq 0.2%, balance Mg). Unipolar, bipolar and hybrid (combination of both) current modes were used in this work. Optical Emission Spectroscopy (OES) was employed to study the plasma species, and electron temperature of the plasma. The morphology and microstructure of the coatings were investigated using Scanning Electron Microscopy (SEM). Potentiodynamic polarization in a 3.5% NaCl solution was used for the corrosion investigations.

2:20pm **SE+PS-WeA2 Aging Mechanism of the Hydrophilic Silicon (100) Native Oxide Surface, T.S. Williams, R.F. Hicks, University of California Los Angeles**

The wetting behavior of surfaces is important in many applications, such as for example, microfluidic devices that are fabricated on silicon wafers. In this study, silicon native oxide surfaces were cleaned with a radio frequency, atmospheric pressure helium and oxygen plasma and with ammonium hydroxide, hydrogen peroxide, and deionized water in a 1:1:5 ratio (RCA SC-1). Both processes created a hydrophilic state with water contact angles of $<5^\circ$ and $16.2 \pm 1.7^\circ$, respectively. During subsequent storage in a chamber purged with boil off from a liquid nitrogen tank, the water contact angle increased over several days at a rate dependant on the cleaning method used. Internal reflection infrared spectroscopy revealed that the change in water contact angle was due to the adsorption of organic molecules with an average hydrocarbon chain length of 10 ± 2 . The rate of the adsorption process decreased with the fraction of hydrogen-bonded hydroxyl groups on the surface relative to those groups that were isolated. On Si (100) surfaces that were cleaned by RCA SC-1 and the plasma, 96% of the silanol groups were hydrogen bonded. The first-order rate constant for adsorption of the organic contaminant on this surface was $0.182 \pm 0.008 \text{ hr}^{-1}$. Several methods have been explored for keeping the silicon dioxide surface in a hydrophilic state for extended periods of time, and these will be presented at the meeting.

2:40pm **SE+PS-WeA3 Polyimide Surface Treatment to Hydrophobic Surface with Self Assembled Mask Layer for Direct Inkjet Patterning Process, J.B. Park, G.Y. Yeom, Sungkyunkwan University, Republic of Korea**

The inkjet printing technology is emerging as one of the important process technologies for electronics, because it can significantly reduce the manufacturing process cost, materials waste, and number of process steps. Especially, many researchers investigating the inkjet-printed process have focused their attention on the printing of conductive films on a polymer film. The technique is not suitable for applying to organic transistors yet, because its pattern width is limited to several dozens μm by statistical variations of the flight direction of droplets and their spreading on the substrate.

In this study, polyimide film was treated to hydrophobic surface using modified atmospheric pressure plasma system (double discharge system). Especially, for keeping the surface characteristics permanently, surface of

the polymer film was etch/textured with self assembled mask layer to form the textured polymer surface. The texturing process is progressed for 4 process step. The mask layer (HMDS) is deposited and agglomeration/oxidation by using atmospheric discharge of HMDS/He/O₂ gas mixture through 1st step and 2nd step. And Etch and texturing process is progressed with atmospheric discharge of He/O₂/Ar gas mixture in 3rd step. And the mask layer is removed in 4th step using NF₃/N₂ gas mixture of remote atmospheric plasma. In the 2nd process, the HMDSO mask layer is formed specific tissue layer as the input power was increased. And the morphology is also varied with the whole size of the HMDSO mask layer. And this whole process is possible because of perfect etch selectivity between the polyimide film and HMDSO layer. The contact angle of textured polyimide film was measured over the 100 degree and varied with textured surface morphology. And keeping the pattern width of inkjet printing was clearly improved compare with normal polymer surface.

3:00pm **SE+PS-WeA4 In Situ Fabricating Blue Ceramic Coatings on Al Alloy by Plasma Electrolytic Oxidation, Z.J. Wang, R.O. Hussein, X. Nie, H. Hu, University of Windsor, Canada**

In-situ formation of novel blue ceramic coatings on Al alloy with a controllable blue color was successfully achieved using a plasma electrolytic oxidation (PEO) process working at atmospheric pressure. This novel blue ceramic coating overcomes the shortcomings of surface treatments resulting from traditional dyeing process by depositing organic dyes into the porous structure of anodic film, such as poor resistance of abrasion and rapid fading when exposed to light. X-ray diffraction, scanning electron microscopy and energy dispersive spectroscopy were employed to characterize the microstructure of the blue ceramic coating. The main compositions of the coating are CoAl₂O₄ and Al₂O₃. This work shows that the working current density plays significant roles on CoAl₂O₄ phase in the ceramic coating which has a controllable influence on the coating color. Low current density reduces the percentage of CoAl₂O₄ component in the coating, and light blue ceramic is produced. On the other hand, higher current density increases CoAl₂O₄ component in the coating, hence deep blue ceramic is fabricated. Electrochemical test, ball-on-plate sliding wear tester and thermal shock method were utilized for the corrosion, wear and thermal shock resistance analysis of the fabricated ceramic coatings. The results indicate that the developed blue coating via PEO process superiorly improves the tribological property, anti-corrosion property and thermal shock resistance. The fabricated blue ceramic coating tends to be applied to colored light cast alloys for applications used in relatively harsh and severe working environments.

4:00pm **SE+PS-WeA7 Cold Atmospheric Plasma Sources for Treatment of Cell-Containing Surfaces, M.G. Kong, Loughborough University, UK**

INVITED

Low-temperature gas discharges generated at atmospheric pressure, commonly known as cold atmospheric plasmas, are a relatively new member of the processing plasma family. Without the need for a vacuum chamber, they offer a much more cost-effective route to material processing and open up opportunities for chamber-less processes and vacuum-incompatible materials such as moist objects. With a mean electron energy at a few eV and a gas temperature close to room temperature, cold atmospheric plasmas are ideally placed for effective chemical dissociation and hence for a wide range of materials processing applications. In this contribution, a review of the current cold atmospheric plasma sources will be presented in terms of their underpinning science and their current technology capability. These include the traditional dielectric barrier discharges, radio-frequency glow discharges, and cold atmospheric plasma jets and jet arrays. Through discussion of their plasma characteristics, their applications are exemplified through treatment of cell-containing surfaces, including living tissues. The interaction of cold atmospheric plasmas with individual microorganisms and indeed microbial communities will be discussed, and its implications to treatment of skin diseases and wounds will be presented. This will also be supported with data of plasma interaction with mammalian cells.

4:40pm **SE+PS-WeA9 High Performance of 60-Hz Atmospheric Pressure Plasma: Basic Characteristics and Applications, F. Jia, K. Takeda, K. Ishikawa, H. Inui, S. Iseki, Nagoya University, Japan, H. Kano, NU Eco-Engineering Co., Ltd., Japan, H. Kondo, M. Sekine, M. Hori, Nagoya University, Japan**

In this paper, the spatial distribution of atomic oxygen density in a 60-Hz non-equilibrium atmospheric pressure plasma[1] is diagnosed by two-photon absorption laser induced fluorescence (TALIF)[2]. The plasma unit is made of ceramics comprised three regions: gas diffusion region, main discharge region, and plasma jet in the open air. The discharge gases were

Ar and a small amount of O₂. The plasma could offer electron density as high as 10¹⁵ cm⁻³ with a low gas temperature[1,3], and have been successfully used to clean glass surface[1]and inactivate the spores of *Penicillium digitatum*[4]. In the above applications, we find that atomic oxygen plays an important role[1,4], and the samples are usually treated in the open air; therefore it is necessary to investigate the behavior of atomic oxygen, especially in the open air, in order to achieve high performance. A dye laser pumped by an excimer laser is used to generate nanosecond UV laser pulses at around $\lambda = 226$ nm for the two-photon excitation of atomic oxygen ($2p\ 3P-3p\ 3P$). The laser power is adjusted to 0.1 mJ/pulse to make sure that the effect of photo dissociation of ozone can be negligible in the experiment. The results showed that the effect of O₂ admixture variation on the atomic oxygen density is totally different in the main discharge region and in the plasma jet. In the main discharge region where the discharge gas are only Ar and O₂, the density of atomic oxygen increased quickly with adding only 0.25% O₂, became saturated with adding 1% O₂, and reduced quickly when adding 1.5% O₂. The discharge was stable until adding 2.5% O₂. However, in the plasma jet that was in the open air, the density of atomic oxygen remained almost same while adding O₂ from 0% to 2.5%. This is because the mechanisms of generation and recombination of atomic oxygen are different in the main discharge region and plasma jet. More work will be done to study the behavior of atomic oxygen in the plasma jet, the data and results will be very useful to understand the behavior of atomic oxygen and improve the applications of non-equilibrium atmospheric pressure plasma. [1] M. Iwasaki, H. Inui, Y. Matsudaira, H. Kano, N. Yoshida, M. Ito, and M. Hori, *Appl. Phys. Lett.* **92**, 081503 (2008). [2] K. Niemi, V. Schulz-von der Gathen, and H. F. Dobeles, *Plasma Sources Sci. Technol.* **14**, 375 (2005). [3] Fengdong Jia, Naoya Sumi, Kenji Ishikawa, Hiroyuki Kano, Hirotoshi Inui, Jagath Kularatne, Keigo Takeda, Hiroki Kondo, Makoto Sekine, Akihiro Kono, and Masaru Hori, *Appl. Phys. Express*, **4**, 026101 (2011). [4] S. Iseki, T. Ohta, A. Aomatsu, M. Ito, H. Kano, Y. Higashijima, and M. Hori, *Appl. Phys. Lett.* **96**, 153704 (2010).

5:00pm **SE+PS-WeA10 Dense Atmospheric Pressure Discharges for Surface and Gas Treatment**, *M.J. Kelly, B.D. Schultz, W.M. Hooke*, International Technology Center

Dielectric barrier discharge (DBD) plasmas have been formed in atmospheric pressure gases exhibiting peak currents in excess of 100 amperes. Power densities during the pulse routinely exceed 100 kilowatts per cubic centimeter for moderately sized electrodes (>100 square centimeters) with ionization densities of 10¹⁴ per cubic centimeter. Charge transfer of 100 microcoulombs per pulse has been repeatedly generated at frequencies up to 100 hertz, and the charge delivery is found to scale in proportion to the electrode area for a given dielectric consistent with a homogeneous discharge. Diffuse discharges have been formed over larger areas (exceeding 1 meter in length and 500 square centimeters) as well. These results were obtained using a custom high voltage driving source and in the absence of helium, argon, or any other easily ionized gas. The source readily achieves an overvoltage in excess of the DC breakdown voltage prior to the onset of breakdown in which 20-30 kV is delivered with rise times shorter than the lag time between the pulse crossing the threshold voltage and the onset of a discharge. Electrical modeling of the discharge characteristics has produced correlations relating power and charge transfer to various electrical and geometrical parameters of the system which will be discussed in this paper.

5:20pm **SE+PS-WeA11 Investigation of Discharge Modes of Cylindrical Dielectric Barrier Discharge Configuration for Surface Treatment at Atmospheric-Pressure**, *T.S. Cho, Y.L. Wu, J.M. Hong, Z. Ouyang, D.N. Ruzic*, University of Illinois at Urbana Champaign

To date, various structural concepts of atmospheric-pressure dielectric barrier discharge had been studied and some of the concepts have already been commercialized for surface treatment processes because of its simplicity and scalability. In this study, cylindrical dielectric barrier discharge configurations for treating the powder particles or controlling the air pollutants at atmospheric-pressure have been investigated. The electrical characteristics of the cylindrical dielectric barrier discharge structure as a capacitive load have been experimentally measured for the mixture of helium and nitrogen and its flow rate with voltage-charge lissajous analysis method. Also, it has been compared with remote plasmas from the commercial planar dielectric barrier discharge system of which capacitance has been 280pF for driving conditions of 5kV and 30 kHz. Emissions from the atmospheric-pressure dielectric barrier discharge plasma have been analyzed for varied gas conditions with the optical emission spectroscopy. For comparison purposes, the glass substrates treated with commercial planar and cylindrical dielectric barrier discharge plasmas have been analyzed. In addition, the sugar-alcohol particles have been treated with atmospheric-pressure plasma from the cylindrical dielectric barrier discharge system, and compared with the untreated particles.

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