12th Asia Pacific Conference on Plasma Science and Technology

including

27th Symposium on Plasma Science for Materials

31 August – 5 September 2014
<table>
<thead>
<tr>
<th>Time</th>
<th>Session</th>
<th>Speaker/Title</th>
<th>Room</th>
</tr>
</thead>
<tbody>
<tr>
<td>08:40</td>
<td>Check in Desk Open (Foyer 1)</td>
<td></td>
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</tr>
<tr>
<td>09:00 – 09:40</td>
<td>Plenary Speaker: Prof Akira Mizuno</td>
<td>Environmental application of electrostatics and non-thermal plasma</td>
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<tr>
<td>9:40-10.00</td>
<td>Prof Homero Maciel</td>
<td>Plasma-enhanced atomic layer deposition: a powerful technique to control thin film properties aimed for photoelectrochemical solar cell applications</td>
<td></td>
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<tr>
<td>10.00-10.20</td>
<td>Prof Makoto Sekine</td>
<td>Diagnostics of Hydrogen Radical-Injected SiH4/H2 Plasma Process for Microcrystalline Silicon Deposition</td>
<td></td>
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<tr>
<td>10.20-10.40</td>
<td>Mr Alexander Sibley</td>
<td>Plasma Growth of Organosilanes Films and their Attachment to Metal Oxide Surfaces</td>
<td></td>
</tr>
<tr>
<td>10.40-11.10</td>
<td>Tea Break – Served in Foyer 1</td>
<td></td>
<td></td>
</tr>
<tr>
<td>11.10-11.40</td>
<td>Invited Speaker: Prof Christine Charles</td>
<td>Radiofrequency plasma sources for space use: experiments, modelling and simulations</td>
<td></td>
</tr>
<tr>
<td>11.40-12.00</td>
<td>Dr Alexey Kondyurin</td>
<td>Curing of epoxy composites in space</td>
<td></td>
</tr>
<tr>
<td>12.00-12.20</td>
<td>Dr Tony Murphy</td>
<td>Metal Vapour in Arc Welding: Computational Modelling of the Arc, Weld and Fume Formation</td>
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<tr>
<td>12.20-12.40</td>
<td>Prof Yasunori Tanaka</td>
<td>Heat-shield Effect of Polymer Ablation from Synthetic-Fiber Fabrics due to Thermal Plasma Contact for Arc-Resistant Clothes</td>
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<tr>
<td>12.40-2.00</td>
<td>Lunch Break – Served in Foyer 1</td>
<td></td>
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<tr>
<td>2.00-2.30</td>
<td>Invited Speaker: Prof Shuyan Xu</td>
<td>High-density Plasma for Fast Etching and Surface Passivation in Silicon Photovoltaics</td>
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<tr>
<td>2.30-2.50</td>
<td>Prof David McKenzie</td>
<td>The HiPIMS Plasma Modelled as a Feedback System</td>
<td></td>
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<tr>
<td>2.50-3.10</td>
<td>Mr Behnam Akhavan</td>
<td>Plasma polymer deposition onto silica particles: A new approach to clean water</td>
<td></td>
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<tr>
<td>3.10-3.30</td>
<td>Dr Cormac Corr</td>
<td>Spatially and temporally resolved gas temperature and neutral particle dynamics in MAGPIE</td>
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<tr>
<td>3.30-4.00</td>
<td>Tea Break – Served in Foyer 1</td>
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</table>
| 09:10 – 09:40 | Invited Speaker: Dr Jun-Seok Oh  
Point-to-plate Micro Discharge Plasma for the Optical  
Sectroscopic Analysis of Aqueous Solutions |
| 9:40-10.00 | Dr Endre Szili  
Delivery of reactive oxygen species (ROS) into tissue models             |
| 10.00-10.20 | Prof Takayuki Watanabe  
Electrode Erosion Mechanism of Multi-Phase AC Arc                        |
| 10.20-10.50 | **Tea Break – Served in Foyer 1**                                         |
| 10.50-11.10 | Mr Matt Thompson  
The influence of He on damage formation and H retention in plasma exposed tungsten |
| 11.10-11.30 | Mr Edgar Wakelin  
Bio-activation of Polyether Ether Ketone using Plasma  
Immersion Ion Implantation                                               |
| 11.30-11.45 | Closing Address – Andrew Michelmore                                       |
Environmental application of electrostatics and non-thermal plasma

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Electrostatic precipitation has been used to remove fine particles because of high efficiency with low pressure drop. Recently, submicron particles in flue gas or exhaust from vehicles have been of concern. Significant improvement of this old technology can be made by utilizing ionic wind, as shown in Fig.1[1]. Ionic wind associated by corona discharge is effective to drive suspended particles towards collection electrode. If backward wind is reduced, collection efficiency can be improved. Collection electrode covered with fine fibres is effective to decelerate the backward wind. These fibres also keep collected particles because electric field converges to the fibres to generate gradient force. Due to this mechanism, abnormal re-entrainment of dusts can be reduced, and collection efficiency can be improved.

Non-thermal plasmas (NTP) have been studied for environmental applications, especially since pulse energization has been used in electrostatic precipitation to cope with back corona problem[2, 3]. NTP generate reactive radicals, and promote chemical reactions at low temperature condition. For practical applications, selectivity and energy efficiency of NTP process should be improved. For this purpose, combination of NTP and catalysts is an effective method[4]. The first generation of the combination of NTP and catalysts was a pulsed streamer corona with a TiO2 catalyst. This combination has been commercialized successfully as an indoor air cleaner attached to an air conditioner. A packed bed is another effective way to combine NTP and catalyst. A packed bed uses a layer of dielectric pellets of catalysts. The pellet layer is sandwiched by the electrode energized with AC or pulsed voltages. Each contacting point of the pellets is ionized. The packed bed has been used for testing effectiveness of the combination, and also for plasma assisted combustion of VOCs with lean-burn condition. Recently, discharges in honeycomb catalysts have been developed. As shown in Fig.2, one end of a honeycomb catalyst is attached to the surface dielectric barrier discharge. When a DC electric field is applied across the honeycomb, ionization proceeds along fine channels of a ceramic honeycomb. This honeycomb discharge could be used for various NTP processes combined with catalysts.

In this presentation, electrostatics and NTP in cleaning of gases, fuel reforming, sterilization and other biological applications will be presented.
Fig. 1  Utilization of Ionic Wind to improve performance of Electrostatic Precipitation

Fig. 2  Combination of Honeycomb Catalyst and Non-thermal Plasma for improvement of Energy Efficiency

References
Plasma-enhanced atomic layer deposition: a powerful technique to control thin film properties aimed for photoelectrochemical solar cell applications

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Plasma-enhanced atomic layer deposition (PEALD) is an energy-enhanced method for the synthesis of ultra-thin films with angstrom-level resolution in which a plasma is employed during one step of the cyclic deposition process. The use of plasma species as reactants allows for more freedom in processing conditions and for a wider range of material properties compared with the conventional thermally-driven ALD method [1]. For example, it is possible to control the material crystallinity only by changing the flux of neutral species through the substrate surface [2]. Also, the film roughness can be improved by changing the substrate temperature and plasma power, so allowing the increase of surface area for example. These properties are main requirements for photoelectrochemical solar cells in order to improve its efficiency. In this work, some results of titanium dioxide (TiO₂) thin films grown by PEALD using a metal precursor (titanium tetrachloride) and an oxygen precursor (O₂ plasma) are presented. The effects of discharge power and substrate temperature are investigated concerning the film structure and morphology. GIXRD, Raman spectroscopy and AFM techniques were used for the films characterization. Results show a high RMS roughness for high plasma power, and that this roughness is directly related to film crystallinity. It is remarkable that pure anatase TiO₂ film can be synthetized by this technique.

Diagnostics of Hydrogen Radical-Injected SiH₄/H₂ Plasma Process for Microcrystalline Silicon Deposition


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Plasma-enhanced chemical vapor deposition system with hydrogen radical-injection (RI) is proposed for the fabrication of hydrogenated microcrystalline silicon (μc-Si:H) thin films. The plasma parameters and resultant growth characteristics obtained with the RI-capacitively coupled plasma (RI-CCP) system excited with 60-MHz power were compared with those obtained using a conventional CCP (C-CCP) system. The absolute density of hydrogen (H) radicals was measured by vacuum ultraviolet laser absorption spectroscopy (VUVLAS) to evaluate the effect of RI for controlling the H radical density. A higher density of H radicals was achieved with RI-CCP than with C-CCP by hydrogen radical injection. The crystallinity factor, preferential orientation, defect density, microstructure, and post-deposition oxidation of Si thin films deposited using C-CCP and RI-CCP were investigated. Crystallinity factor of 0.6 was realized with high deposition rate of around 2 nm/s even under a low plasma density using RI-CCP. The defect density of μc-Si:H thin films prepared using RI-CCP was lower than that in thin films prepared using C-CCP. In addition, post-deposition oxidation of the films with RI-CCP was lower than that with C-CCP. The high performance of RI-CCP for the fabrication of μc-Si:H thin films for solar cell devices is also demonstrated.

Plasma Growth of Organosilanes Films and their Attachment to Metal Oxide Surfaces

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It has been well-established in literature that organosilane coatings exhibit much potential as a more environmentally benign replacement for inorganic anti-corrosive treatments. Wet chemistry is the most common approach for forming these coatings but has several disadvantages. For instance, organic solvents are often required and the process can be time consuming. Plasma-based methods present a promising alternative due to their ability to clean and pre-treat the substrate in clean vacuum conditions followed immediately with a coating step using the pure precursor molecule. The challenge (but potential strength) of plasma based coating comes from the many different experimental variables present and the effect that they have on the final structure and properties of the coating.

This study involves a series of experiments designed to improve the understanding of how varying plasma conditions such as power, pressure, gas composition and exposure time affect the deposition of propyl-trimethoxysilane (PTMS) coatings on the native oxides of magnesium and aluminium. Samples are cleaned, pre-treated and coated under vacuum using a custom built inductively coupled plasma chamber operating at a frequency of 13.56MHz. Elemental composition and coverage of the coatings is determined through X-ray Photoelectron Spectroscopy and Scanning Auger Microscopy respectively, while information about the structure of the films is obtained through the use of Infrared Spectroscopy and Neutral Impact Ion Scattering Spectroscopy.
Radiofrequency plasma sources for space use: experiments, modelling and simulations

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Commercial space companies and space agencies are responding to society’s growing interest in access to space, i.e. satellites on low or geostationary Earth orbits and long term exploratory missions to moons, planets or asteroids. Radiofrequency plasma engines are good candidates for providing extended lifetime, power scaling and broad choice of propellant (including use of spacecraft systems’ residues and of ‘green’ and safe propellants). Here the physics behind the development of new-generation low-cost radiofrequency (rf) plasma thrusters such as the high-power Helicon Double Layer Thruster (HDLT) [1] and the low-power Pocket Rocket [2] will be presented. New technological challenges such as the design of low-weight rf matching networks and rf generators need to be addressed to determine the thrusters’ performance (thrust, specific impulse, efficiencies) [3]. Testing of our plasma sources is carried out in various size vacuum chambers (IRUKANDJI, WOMBAT and WOMBAT XL at Mount Stromlo) using a range of diagnostics (thrust balance, optical and electrostatic probes) and the results are used to develop analytical models and computer simulations aiming at a better understanding and control of the thrust generation mechanisms.

References:
Curing of epoxy composites in space

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Future space exploration requires large light-weight structures for antennas, reflectors, radiation shields, habitats, greenhouses, space bases, space factories and space mining. A new approach enabling large-scale constructions in space that relies on curing in the free space environment of fiber-filled composites with a curable polymer matrix will be presented. In free space, the material is exposed to high vacuum, dramatic temperature changes, plasma that is generated by cosmic rays, sun irradiation and atomic oxygen (in low Earth orbit), micrometeorite fluences, electric charging and microgravitation. The development of appropriate polymer matrix composites requires an understanding of the chemical processes of polymer matrix curing under the specific free space conditions to be encountered.

We investigated the curing process of suitable epoxy compositions in a simulated free space environment. Uncured epoxy resin was spun onto a silicon wafer and treated by plasma and plasma immersion ion implantation (PIII) with argon ions of energy up to 20 keV. Ellipsometry, FTIR spectroscopy and optical microscopy methods were used to study the curing processes. Etching, carbonization, oxidation and crosslinking effects were observed. Curing reactions in the modified epoxy resin were observed in the absence of a hardening agent. A model of structural transformations in the epoxy resin under plasma and ion irradiation is proposed and discussed in relation to processes in a space environment. Our laboratory investigations were supported by a subsequent stratospheric flight experiment with an uncured, epoxy composite prepreg. For the first time, the epoxy matrix was cured successfully in the stratosphere. These results can be used for predicting curing processes of polymer composites in free space environments during orbital space flights.

These investigations were supported by grants from the Alexander von Humboldt Foundation, ESA, NASA and RFBR (05-08-18277, 12-08-00970 and 14-08-96011).
Metal Vapour in Arc Welding: Computational Modelling of the Arc, Weld and Fume Formation

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Arc welding is arguably the most widely-used plasma application. An electric arc is struck between two metal electrodes, one or both of which are partially melted. This melting, as well as being integral to the welding process, promotes the formation of metal vapour.

Metal vapour is important in arc welding for two reasons [1], both of which are addressed in this paper.

First, the presence of metal vapour alters the properties of the arc, due to increased radiative cooling and increased electrical conductivity at lower temperatures. Both of these changes decrease the heat flux to the welded metal, and therefore the depth of the weld.

Second, metal vapour nucleates and condenses to form welding fume (chains of metal oxide nanoparticles), which is a significant occupational health hazard for welders.

We have developed sophisticated computational models of the arc welding process that allow the formation and influence of metal vapour to be predicted self-consistently [2]. These models have been coupled to a model of nanoparticle nucleation and growth to allow the size and morphology of fume particles to be calculated [3]. The models will be described, and their predictions compared with available experimental data.

Heat-shield Effect of Polymer Ablation from Synthetic-Fiber Fabrics due to Thermal Plasma Contact for Arc-Resistant Clothes

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Heat-shield effect of polymer ablation due to thermal plasma contact was studied from synthetic fabrics of arc-resistant clothes. Effective arc-resistant clothes are greatly desired for workers nearby electrical devices especially in the United States, because there have been reported 5-10 arc flash incidents in a day according to the statistics by CapShell, Chicago. The arc plasma is a thermal plasma established between high-voltage conductors, having a high temperature above 5000 K. The arc-flash incident thus can result in serious injuries to nearby workers. To enhance the arc-resistant ability of fabrics, we have focused polymer ablation phenomena. Polymer ablation from synthetic fabrics produces ablated vapour covering the surface of the fabrics, which can involve heat-shield effects. To study the heat-shield effect of polymer ablation from fabrics, two-layered fabrics were prepared; One layer is ablative fabric made of modacrylic (MA) fiber, and the other is non-ablative and shape-stable fabric made of para-aramid (p-Aramid) fiber. Backside temperature of the fabrics was measured with a thermocouple during the Ar thermal plasma irradiation to the two-layered fabrics. Figure 1 shows the time variations in the backside temperatures for p-Aramid/p-Aramid, p-Aramid/MA and MA/p-Aramid in two-layered fabrics. The backside temperatures for the three conditions increase rapidly just after initiation of Ar thermal plasma irradiation. The MA was ablated from 1.6 s after Ar thermal plasma irradiation, which results in a decrease in the rise rate of the backside temperature. This decrease in the temperature-rise rate is found to arise from the heat-shield effect of the polymer ablation.

![Fig.1 Backside temperature variations for three two-layered fabrics during Ar thermal plasma irradiation.](image-url)
High-density Plasma for Fast Etching and Surface Passivation in Silicon Photovoltaics

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This talk focuses on the plasma aided fabrication of silicon solar cells by high-density plasmas through fast dry etching and surface passivation. Simple processes of silicon texturing and passivation have been developed by means of chemically active plasma etching, atomization and dielectric passivation using low-frequency, high density inductively coupled plasmas. The processes feature a high level of deterministic control over production of radical species, cluster fragmentation, particle transport, and radical and ions interaction with silicon surfaces. Through manipulation of electron energy distribution, ion transport and suppression of plasma surface heating, the effect of a-Si:H passivation on c-Si is significantly enhanced. The minority carrier lifetime of up to 3 ms can be routinely obtained by depositing only a few nm of a-Si:H on c-Si wafer. Furthermore, remote deposition of a-Si:H drastically enhances the overall absorption coefficient of the active layer and decreases the dark conductivities of the Si films without deterioration of photo-conductivities and photo-responses, leading to substantial improvement of the solar cell performance.
The HiPIMS Plasma Modelled as a Feedback System

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Models are useful in the control and diagnostics of time dependent plasmas, especially in the initiation of pulses and in the selection of pulse lengths. Feedback models are well known in mathematics and have been used with success in many areas of science. We present a 1-D feedback model that captures the essential elements of the current time relation in pulsed sputtering discharges. Our model falls into the class of single-species population models with recruitment and time delay, which show no oscillatory behaviour. We include the external circuit and we focus on the time evolution of the current as a function of the applied voltage and the plasma parameters. The model reproduces experimental data on time-dependent plasma properties and predicts the key parameters affecting the evolution of the discharge. We find the necessity of a nonlinear loss term to ensure a stable discharge, and we show that a higher secondary electron emission coefficient reduces the time delay for current initiation. We report that I-V characteristics in the plateau region, where it exists, fit a power curve of the form \( I = k V^n \), where \( n \) is influenced most strongly by the nonlinear loss term.

Left: A schematic of the model

(a) the effect of secondary electron emission coefficient on the time delay and (b) an example in HiPIMS where an oxide coverage of the target increases the secondary electron emission coefficient and reduces the time delay for current initiation.
Plasma polymer deposition onto silica particles: A new approach to clean water

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In recent years, functionalized hydrophobic materials have attracted considerable interest as petroleum hydrocarbon removal agents. This investigation has applied plasma polymerization as a novel, green and one-stage process [1] to develop nanostructured hydrophobic and oleophilic films for water purification. 1,7-Octadiene was plasma polymerized onto silica particles using a radio frequency inductively coupled reactor fitted with a rotating chamber. Plasma polymerized 1,7-octadiene (ppOD) films were deposited using a varied range of plasma power, 1,7-Octadiene flow rate and deposition time. The surface chemistry of ppOD coated particles was investigated via X-ray photoelectron spectroscopy and time-of-flight secondary ion mass spectroscopy, while Washburn capillary rise measurements were applied to evaluate the hydrophobicity and oleophilicity of the particles. The effectiveness of ppOD coated particles for the removal of hydrophobic matter from water was demonstrated by adsorption of motor oil, kerosene, and crude oil. Petroleum hydrocarbon removal was examined by varying removal time and particle mass. The morphology of oil loaded ppOD coated particles was examined via environmental scanning electron microscopy observations. Increasing the polymerization time increased the concentration of hydrocarbon functionalities on the surface (Fig. 1), thus also increasing the hydrophobicity and oil removal efficiency (ORE) [2,3]. The ppOD coated particles have shown to have excellent ORE. These particles were capable of removing 99.0–99.5% of high viscosity motor oil in 10 min, while more than 99.5% of low viscosity crude oil and kerosene was adsorbed in less than 30s. Plasma polymerization has shown to be a promising approach to produce a new class of materials for a fast, facile, and efficient hydrophobic matter removal.

![Fig. 1. ToF-SIMS CH⁺ ion maps for uncoated and coated particles for different times](image)

References
Spatially and temporally resolved gas temperature and neutral particle dynamics in MAGPIE

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In this work a range of spectroscopic diagnostics are employed to investigate the interaction of the plasma with fusion-relevant materials, under high flux conditions (<10^{23} \text{ m}^{-2}\text{s}^{-1}). Experiments were performed in the MAGnetized Plasma Interaction Experiment (MAGPIE) plasma reactor: a linear magnetically enhanced Helicon reactor designed to investigate the interaction between plasmas and material surfaces in a fusion-relevant environment. In hydrogen plasma discharge, MAGPIE achieves densities up to 10^{19} \text{ m}^{-3} with an electron temperature of 3-6 eV.

The atomic loss probability of hydrogen on tungsten and graphite is measured using pulsed induced fluorescence to provide information of fuel retention and is shown to be dependent on the ion flux at the surface. Tuneable diode laser absorption spectroscopy (TDLAS) provides information of atomic hydrogen densities and gas temperature, while spatially and temporally resolved optical emission spectroscopy provides information of atomic and molecular particle fluxes at the plasma-surface interface as well as atomic and molecular temperatures. The experimental results are complimented with modelling of the plasma discharge.
Point-to-plate Micro Discharge Plasma for the Optical Spectroscopic Analysis of Aqueous Solutions

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Underwater discharge plasma research has a very long history, it has been developed for treatment of polluted water simultaneously developing industry and increasing population since the mid-1900s. The discharge phenomena have been well studied with developing of high voltage pulse power technology.\textsuperscript{[1-3]} A point-to-plate (or point-to-point) is a well-known electrode configuration for the electrical discharge in liquid. In the early studies, electrode gap is typically increased up to a few cm and the amplitude of the applied voltages are from several ten to hundred kilovolts. The large electrode gap and high voltage (or current) provide large volume streamer discharge. The discharge activates the liquid physically and chemically, for examples, an intense uv emission, overpressure shock waves and sufficient active species (\textit{H}•, \textit{O}•, \textit{OH}•, \textit{O}_2•, \textit{HO}_2•, \textit{H}_2\textit{O}_2 and \textit{O}_3) were induced as results.\textsuperscript{[4]} The method aimed massive productions of the physical and chemical effects and suggested to solve environmental issues such as a water treatment at the early stage of this kind of researches as mentioned above.

On the other view point of chemical analysis of liquid, the underwater discharge can be a good analytical method for investigation of the composition of aqueous solutions. There is an example of analytical approach by Ichiki et al.,\textsuperscript{[5]} demonstrated the optical emission spectroscopic analysis of injection of the liquid sample into microplasma jet as the analytical view point. However, the microplasma jet is a dynamic method which is easily influenced by carrier gas condition. We here developed a point-to-plate underwater microplasma which can be operated several hundred volts and the plasma source aiming on-site analysis of chemical composition of liquid. Our recent achievements of electrical and optical behaviours of the microplasma working in various liquids will be discussed.

This work supported by KAKEHI Grant Numbers 26600129.

Delivery of reactive oxygen species (ROS) into tissue models

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The direct application of ionised gas (plasma) to living tissue has been successfully applied to the treatment of wounds, cancers, dental, decays and many other medical indications [1]. The successful application of plasma to disease treatment, including randomised clinical trials has opened up a whole new field of research termed ‘plasma medicine’.

Biomedical plasma sources that interact with the surrounding air, before reaching the biological target, produce a complex mixture of reactive oxygen species (ROS) and reactive nitrogen species (RNS) in abundance. Many of these ROS/RNS are also produced in vivo within cells and are known to regulate signalling processes. Consequently, plausible links have been made between the ROS/RNS generated by plasma to the observed end biomedical outcome.

Considering that plasma usually only modifies the uppermost surface of organic materials (e.g. polymers), it is difficult to reconcile how plasma can have effects on many hundreds to thousands of micrometres into the tissue subsurface (e.g. destruction of solid tumours and deactivation of biofilms on wounds). In order to address this issue, we developed ‘tissue models’ to study plasma interactions with phospholipid vesicles and tissue. I will highlight the relevance of these tissue models for plasma medicine and surprising results that show plasma is capable of directly accessing a tissue’s subsurface, as opposed to other proposed mechanisms, which involve stimulating surface reactions that trigger a cascade of biomolecular signalling events (into the tissue) [2-3].

Electrode Erosion Mechanism of Multi-Phase AC Arc

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The multi-phase AC arc has been applied to the innovative in-flight glass melting technology due to the following advantages; high energy efficiency, large plasma volume, and low gas velocity [1]. The electrode erosion is one of the most important issues to be understood because it determines the electrode lifetime and the purity of the products. The purpose of this work is to investigate electrode erosion mechanism of the multi-phase AC arc.

The multi-phase AC arc system consists of 12 electrodes, 12 sets of AC power supplies in 60Hz, and main chamber [2]. The electrodes were made of 2wt%-thoriated tungsten with diameter of 6.0 mm. Argon with purity of 99.99% was injected around each electrodes to prevent it from oxidation.

The combination of the high-speed camera and the appropriate band-pass filters system was used to observe the radiation from the electrodes even during arc discharge with strong emission from the arc [3]. Then, the surface temperature of the electrode was estimated. Moreover, the dynamic behaviour of the droplet ejection from the molten electrode was observed. Two wavelengths, 785 nm and 880 nm, were selected in a region free of line emission from the plasma. Therefore, only the thermal radiation from the electrode can be obtained using the high-speed video camera with the band-pass filter system. The effects of number of the phases, argon shield gas, arc current, and electrode tip angle on electrode erosion were investigated.

The obtained results indicate both the evaporation of the electrode material and droplet ejection from the molten electrode contributes to the electrode erosion in the multi-phase AC arc. Especially, the droplet ejections mainly occur at the cathodic period, while electrode material mainly evaporated at the anodic period. Electrode erosion mechanism of the multi-phase AC arc was proposed from the experimental results.

The influence of He on damage formation and H retention in plasma exposed tungsten

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He-induced damage in W has emerged as a major concern for the next-step ITER and future DEMO tokamak fusion reactor experiments, owing to its tendency to precipitate into small bubbles, and its potential to degrade or erode W through the formation of complex surface structures [1-3]. To develop a more detailed understanding of the role He plays in W degradation, experiments have been conducted on the Magnum-PSI and MAGPIE linear plasma devices using H-He plasmas of various compositions to simulate the fusion reactor environment. H and He retention has been measured through elastic recoil detection analysis and secondary ion mass spectroscopy, revealing a complex relationship between plasma He composition, temperature, and H retention. For sample temperatures above 700°C, near-surface (<100nm) retention of H was found to increase somewhat for mixed plasmas, while total retention in the first 10μm decreased, an effect believed to result from the formation of He nano-bubbles near the surface acting as a diffusion barrier [2]. Positron annihilation lifetime spectroscopy and small angle x-ray scattering have also been performed on He-exposed samples to measure He nano-bubble formation, with the ultimate goal of developing a better understanding of the formation mechanisms of He damage structures.

Bio-activation of Polyether Ether Ketone using Plasma Immersion Ion Implantation

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Polyether Ether Ketone (PEEK) orthopaedic implants have significant benefits compared to metallic implants due to their mechanical similarity to bone [1], however use is limited due to its poor biocompatibility. Plasma Immersion Ion Implantation (PIII) is used to improve the surface biocompatibility of PEEK. The kinetic properties of the treated surface are characterised here, allowing for tuning of the PIII process in order to produce PEEK with optimised bioactivity. The optimum PIII ion fluence for PEEK is determined to be $1 \times 10^{16}$ ions/cm².

A model, based on the premise that there is a distribution of local chemical environments produced by PIII, is proposed. The model produces good fits for the kinetics of the decay of radicals, nitrogen loss, oxygen incorporation and changes in surface energy. The distribution of chemical environments results in a bioactive surface that has a long shelf life, compatible with clinical needs.

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