Coatings for Biomedical and Healthcare Applications
Room Pacific F-G - Session D1-1-MoM

Surface Coatings and Surface Modifications in Biological Environments I

Moderators: Dr. Mathew T. Mathew, University of Illinois College of Medicine at Rockford and Rush University Medical Center, USA, **Dr. Kerstin Thorwarth**, Empa, Swiss Federal Laboratories for Materials Science and Technology, Switzerland

10:00am **D1-1-MoM-1 Ion Release Study of Ag-Cu and Ag-Cu-Mg Coatings Deposited by Magnetron Sputtering**, ***Serdar Sonay Ozbay,*** Deakin University, Coventry University, Australia*; G. Taghavi Pourian Azar,* Coventry University, UK*; J. Sharp, G. Rajmohan,* Deakin University, Australia*; A. Cobley,* Coventry University, UK

Ag and Cu thin film coatings are widely studied as antibacterial coatings to functionalise surfaces to fight antibiotic-resistant bacteria and healthcare-acquired infections (HAI) in hospitals. Although the precise antibacterial mechanisms of these metals are still being investigated, metal ion release has been proposed as one of the main mechanisms for their antibacterial action. In this study, we have deposited Ag-Cu ($Ag_{75}Cu_{25}$-$Ag_{50}Cu_{50}$-$Ag_{25}Cu_{75}$) alloy coatings using magnetron sputtering technique on various substrates including non-woven PET fabrics, silicon wafers and glass microscope slides. Obtained coatings were characterised using scanning electron microscopy (SEM), X-ray diffraction (XRD) and UV-visible spectroscopy techniques. As a tool to predict antibacterial activity, the coated non-woven PET fabrics were immersed in saline solution for up to 48 hours and the released metal ion concentrations were measured by inductively-coupled plasma optical emission spectroscopy (ICP-OES). Additionally, the ion release test results of the Ag-Cu alloys were compared to the pure Ag and Cu coatings, and the effect of galvanic cell formation on the metal ion release was discussed. Furthermore, Ag-Cu-Mg ternary alloy coatings were deposited to investigate the effect of Mg on the Ag and Cu ion release characteristics. According to the results, the $Ag_{75}Cu_{25}$ coating solved two critical problems which were present in the pure coatings. First, compared to the pure Ag coating, the Ag ion release was improved, and its plateauing behaviour was not observed. Second, the rapid Cu ion release, which was seen in the pure Cu coating, was changed to a steady release. Ternary Ag-Cu-Mg alloy coatings showed further reduction in both the Cu and Ag ion release. However, the effect of Mg on the Cu ion release was more significant compared to the Ag ion release. Overall, the present study suggests that Ag-Cu-Mg ternary alloys are promising coating candidates for hospital settings where long-term and steady antibacterial activity is needed.

10:20am **D1-1-MoM-2 Effect of Pulsed DC Mode on the Surface Properties of Pure Magnesium Substrates Treated with PEO**, ***Cristian Esneider Peñuela Cruz,*** *E. Hernández Rodríguez,* Universidad de Guanajuato, Mexico*; A. Herrera,* Univiversidad de Guanajuato, Campus DICIVA, Mexico

Magnesium is a promising metallic material for the manufacture of bioabsorbable orthopedic implants since in addition to being biodegradable and biocompatible, it has mechanical properties like to those of human bone. However, despite these properties, its poor resistance corrosion resistance has prevented its technological application. A solution to improve its corrosion resistance is to use a surface treatment, such as that provided by plasma electrolytic oxidation (PEO). This technique is used to apply ceramic-type coatings on metals and is widely used in biomedical applications. Therefore, in this work, we carried out a study on the effects generated by the square wave frequency of the pulsed DC mode on the surface properties of pure magnesium sheet by applying TiO_2-based coatings using the PEO technique.Anatase-TiO_2 powders were incorporated into the electrolytic solution due to their good biocompatibility and high corrosion resistance. Coatings were developed using 0.210 g of TiO_2, a current density of 200mA/cm^2, two frequencies of 2 Hz and 10 Hz were evaluated. Data acquisition of voltage transients in the PEO processes was carried out, showing that, in the first stage of the PEO process, as the frequency increases, there is a rapid growth of the voltage, indicating rapid passivation of the magnesium surface. A transversal section of the coatings was observed by optical microscope at 400x, and images showed three regions: the substrate, the substrate-coating interface, and the bulk of the coating. Through X-ray diffraction analysis (XRD), the presence of TiO_2 and MgO into the coatings was identified as a result of the PEO technique. The potentiodynamic polarization curves (Tafel) were employed to investigate the corrosion resistance of a pure Mg substrate and the coated ones, in Hanks' balanced salts. Preliminary results show that Icorr is reduced from 1.471 µA to 454.127 nA, indicating an improvement on the corrosion resistance.

Keywords: Magnesium, corrosion, PEO, surface treatment.

10:40am **D1-1-MoM-3 Non-Stick Thin-Film Metallic Glasse (Tfmg) Coating for Reducing Trauma**, ***Helmi Son Haji,*** *J. P. Chu,* National Taiwan University of Science and Technology, Taiwan*; P. Yiu,* Ming Chi University of Technology, Taiwan

Thin Film Metallic Glasse (TFMG) that refers to a specific group of amorphous multi-component metallic alloys, has special characteristic such as smooth surface, high hydrophobicity, high strength, low friction, good ductility, low surface free energy and good thermal stability exceeding those of other ceramics and alloys coatings. The non-stick characteristic of hydrophobic materials makes them ideal for medical devices such as cutting blade, tattoo needle and syringe needle. This presentation reports on the coating of the medical devices with Zr-based (ZrCuAlTa) TFMG to reduce trauma to the skin. Preclinical study proved that TFMG-coated needle has 33% less endothelial damage than bare needle, showed some clinical benefits such as anti-adhesion, and reduced invasion. Confirm the good biocompatibility and hemocompatibility. Furthermore, clinical study showed TFMG coating could preventing the adhesion of platelet by up to 77% and cancer cells by up to ~87%. Analysis on porcine tissue and polyurethane rubber shows TFMG-coated needles has lower cutting force by up to ~24%, frictional forces by up to ~24%, and resistance to insertion by up to ~44%. TFMG also proved to enhance the sharpness of cutting blades, when tested repeatedly on hairless skin, the surface roughness increases only 8.6% far below the bare that reach 70%, preserving the cutting quality of surgical instruments. Experimentation on live pig and pig skins demonstrated that TFMG could reduce the spread of the pigment to the surface of surrounding skin by up to 57%, resulted narrower tattoo lines of higher density indicating that could be useful in high-resolution tattooing, reducing trauma in skin indicating with no secretion of fluids immediately after tattooing, has faster wound closing and faster healing compared with bare needle.

11:00am **D1-1-MoM-4 Synthesis of Antimicrobial Surfaces by Glancing Angle Deposition with Natural Seeds**, ***Chuang Qu,*** *J. Rozsa, M. Running, S. McNamara, K. Walsh,* University of Louisville, USA

This research focuses on the synthesis and characterization of bio-inspired artificial antimicrobial surfaces. The antimicrobial surfaces studied in this research are surfaces that are covered with nanoscale protrusions, which are observed in nature such as cicada wings and dragonfly wings [1]. These nanoscale protrusions, mostly nanopillars and nanocones, achieve antimicrobial by mechano-bactericidal: the bacteria are deformed by being punctured into the protrusions and eventually killed [2]. Even though research groups have published papers on mechano-bactericidal surfaces [1, 3, 4], there are still some challenges in this research: 1) the synthesis, scaling-up and fine control of the artificial nanoscale protrusions are still challenging; 2) the mechano-bactericidal mechanism is not universal. The challenge of the synthesis comes from the fact that the three dimensional protrusion features for puncturing the bacteria have to be much smaller than bacteria, as small as only a couple nanometers. The proper spacing of the protrusion features is also required to allow the deformation of the bacteria. However, the sub-100 nm, high aspect-ratio, three-dimensional (3D) features are challenging to obtain through traditional nanofabrication methods, especially by top-down nanofabrication techniques.

Our research group proposed to use glancing angle deposition (GLAD) to recreate cicada-wing-mimicry antimicrobial surfaces [5]. GLAD is a bottom-up process for achieving complex 3D nanofeatures. The use of seeds alters the distribution of the features, and hexagonally packed nanofeature arrays are recreated. However, the requirement of the nanosphere seeds adds complexity to the process: the preparation of a monolayer of nanospheres is challenging, and the area of the seeds can be limited if seeding is not properly conducted. In the current research, we discuss the possibility of synthesizing the mechano-bactericidal antimicrobial surfaces by GLAD without predetermined seeds. The design and control of the process for synthesizing the antimicrobial surfaces are addressed in the study. Multiple materials are used for creating nanoscale protrusions for uncovering the mechano-bactericidal mechanism. The characterization of the surfaces, including the morphology, the superhydrophobicity, and the effectiveness of the antimicrobial property are presented. Finally, potential integration methods of the surfaces with nanoscale protrusions and biomedical implants are discussed in the study.

11:20am **D1-1-MoM-5 FDA Regulatory Considerations for Performance Evaluation of Coatings in Medical Devices*, Nandini Duraiswamy,*** U.S. Food and Drug Administration , USA **INVITED**

Coated medical devices are used in millions of interventional and diagnostic procedures, and as permanent implants for treatment of intended patient populations throughout the world. The surgical implants can vary from those used for acute treatments such as interventional catheters to the more complex chronic implants such as drug-eluting coronary stents. The intended uses for the coatings on such implants can also vary such as additively manufactured porous metal surfaces for biological fixation (or osteointegration), non-degradable or degradable polymer with drug mixture for reducing cell proliferation and maintain vessel patency, or metallic coating on endosseous dental implants for biological fixation. These coated medical devices are regulated by Food and Drug Administration (FDA), Center for Devices and Radiological Health (CDRH) (Center for Devices and Radiological Health | FDA). The FDA-recognized international device-specific standards (Recognized Consensus Standards (fda.gov)) are used to assess performance, however the presence of thin coatings on medical devices often lack appropriate test methods for performance assessments of coatings. For example, coating are commonly used on guidewires and catheters, which are used for percutaneous cardiovascular interventions. Seven out of eleven Class I recalls and majority of MDRs in 2013-2014 were related to the "Wire, Guide, Catheter" device category (ProCode DQX). Some of those recalls resulted due to manufacturing changes to the coatings because the Environmental Protection Agency (EPA) wanted manufacturers to eliminate a surfactant, a carcinogen used in Teflon coatings, by 2015 [U.S. Environmental Protection Agency | US EPA]. There is a growing public health need to be able to identify any bad formulations of coatings early on through in vitro testing for the safety of the patients. In addition, the use of varied (or new) formulations of coatings further adds to the complexity of assessing risk of delamination in the patients and testing requirements, and expectations for minimum performance criteria for FDA regulatory clearance or approval. Results from prior research at the Office of Science and Engineering Labs (OSEL) (Office of Science and Engineering Laboratories | FDA) will also be shared as examples.

12:00pm **D1-1-MoM-7 Post-Anodization and Additives Role Towards the Effective Control of Bio-Degradation of Mg Alloys*, Zeeshan Ur Rehman,*** *B. Koo,* Changwon National University, Republic of Korea

Mg and its alloys are widely seen as potential candidates for orthopedic implants and devices, due to their excellent biocompatibility and optimum mechanical properties. However, the major issue with Mg is its high corrosion rate in human body fluid/Chloride containing environment. This would make the implant failure possibility very high, before the bone tissues recovery, thus a sustainable surface treatment is mandatory before promoting the widespread use of Mg in biomedical implants. In this study, the Pre-coated Mg samples using plasma electrolytic oxidation techniques were further processed through sol-gel and dip coating technique. Various additives such as chitosan, Melamine, EDTA and PVA, with varying concentration were used in the sol-gel matrix. The finally obtained samples were characterized using SEM, XRD, EDS, FTIR, XPS for surface molecular and microstructural observations. Furthermore, biodegradation properties were analyzed using EIS, Potentiodynamic polarization and weight loss method to study the degradation rate of the coated samples. The result showed that post-anodization treatment under optimized concentration of the stated additives has slowed down the degradation significantly.

Keywords: AZ31, Orthopedic implant, Post PEO, biodegradation, SEM

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