

## Coatings for Biomedical and Healthcare Applications Room Palm 3-4 - Session MD1-1-WeA

### Surface Coatings and Surface Modifications in Biological Environments I

**Moderator: Mathew T. Mathew**, University of Illinois College of Medicine at Rockford and Rush University Medical Center, USA

**2:00pm MD1-1-WeA-1 Synergistic Antibacterial Activity and Ion Release of Ag-Cu and Ag-Cu-Mg Coatings, Serdar Sonay Ozbay (sozbay@deakin.edu.au), G. Rajmohan**, Deakin University, Australia; A. Cobley, Coventry University, UK; J. Sharp, Deakin University, Australia; G. Azar, Coventry University, UK

Silver (Ag) and Copper (Cu) thin films have been widely studied as antibacterial coatings to functionalise textile surfaces to fight antibiotic-resistant bacteria and healthcare-acquired infections (HAI) in hospitals. The release of metallic ions is considered to be the main antibacterial mechanism for both metals. Therefore, maintaining a steady ion release from the metallic coatings is necessary to achieve a sustained antibacterial activity. Despite the high effectiveness of Ag ions as antibacterial agents, Ag coatings suffer from a limited antibacterial activity due to the decreasing Ag ionisation rate caused by surface passivation. In contrast, Cu coatings exhibit rapid but brief antibacterial action due to the fast release of Cu ions. Recently, studies on combined Ag-Cu systems have reported an enhanced Ag ionisation and a synergistic antibacterial activity between Ag and Cu.

This study investigates how different compositions of Ag-Cu alloy thin film coatings can improve the limitations of pure metals to achieve a steady and long-term antibacterial efficacy. The Ag-Cu ( $\text{Ag}_{75}\text{Cu}_{25}$ - $\text{Ag}_{50}\text{Cu}_{50}$ - $\text{Ag}_{25}\text{Cu}_{75}$ ) alloys were deposited on PET textiles using magnetron sputtering technique. The growth process and microstructures of the thin films were characterised by XRD and TEM/EDS. Additionally, the galvanic relationship and antibacterial synergy between the Ag and Cu components in different alloys were investigated through the ion release studies and antibacterial tests. Finally, the effects of a more electrochemically active metal on the properties of Ag-Cu alloys were studied by co-sputtering Mg into Ag-Cu thin films.

The results showed that both  $\text{Ag}_{75}\text{Cu}_{25}$  and  $\text{Ag}_{50}\text{Cu}_{50}$  coatings improved the plateauing of Ag ion release and provided a steady Cu ion release. Antibacterial efficacy of Ag-Cu thin films followed the order:  $\text{Ag}_{50}\text{Cu}_{50} > \text{Ag}_{25}\text{Cu}_{75} > \text{Ag}_{75}\text{Cu}_{25} > \text{Ag} \approx \text{Cu}$ . Due to the sufficient release of both Ag and Cu ions in the  $\text{Ag}_{50}\text{Cu}_{50}$  coating, this sample demonstrated superior antibacterial performance compared to both other alloys and pure metal coatings. Moreover, this coating maintained a >90% bacterial reduction rate after two antibacterial test cycles, outperforming the other coatings. The ion release studies of Ag-Cu-Mg ternary alloys showed a further reduction in both Cu and Ag ion release, with the effect of less noble Mg on the Cu ion release being more significant compared to that on the Ag ion release. Overall, our results suggest that Ag-Cu and Ag-Cu-Mg thin films are promising candidates for hospital textiles that require a steady and prolonged antibacterial activity.

**2:20pm MD1-1-WeA-2 Iridium Oxide Based Electrodes for Bio-Interface Applications, Po-Chun Chen (cpc@mail.ntut.edu.tw)**, National Taipei University of Technology, Taiwan

INVITED

Iridium oxide has attracted extensive attention due to its unique advantages including excellent chemical stability and sensitivity, impressive electrochemical catalytic activity, sufficient electric conductivity, and desirable biocompatibility. To date, iridium oxide has been widely explored in applications such as anodes for water electrolysis, electrochromic layers for smart windows, and pH sensors. In addition, iridium oxide is known for its superb charge storage capacity and long-term stability renders it a desirable candidate as a bio-interface electrode for implantable bio-medical electronic devices. In this study, we developed solution processes to prepare iridium oxide film for bio-interface applications. We also characterized the electrochemical properties of the iridium oxide films and examined its stability. The iridium oxide film was found to be robust and revealed excellent charge storage capacity and charge injection capability. Additionally, the solution process allows the synthesis of iridium oxide hybrid film by combining plasma protein with enhanced electroactivity, improved cytocompatibility, and controllable electrically responsive protein release to enhance neuronal activity. Therefore, the iridium oxide film plays a multifunctional role as an electrode for bio-interface applications.

**3:00pm MD1-1-WeA-4 Improvement of Corrosion Resistance of Biodegradable Mg-Ca Alloy by Atomic Layer Deposition Technique, Hsin-Chih Lin (hclinntu@ntu.edu.tw), P. Lin, H. Chen, K. Lin**, National Taiwan University, Taiwan

Biodegradable Mg-Ca alloy has high application potential in the fields of cardiovascular stent, wound healing device and orthopedic implant. However, the degradation rate of Mg-Ca alloy is too fast to provide enough support for a long time, and it may also cause pH rise and excessive hydrogen production in vivo, which may lead to cell inflammation, vascular blockage, and other negative problems. Hence, how to precisely control the degradation rate and effectively reduce the corrosion rate is an important issue for Mg-Ca implants. Recently, we have conducted extensive studies on improving the corrosion resistance of Mg-Ca alloy by atomic layer deposition (ALD) technique. It is found that the atomic layer plasma treatment (ALPT) enhances the corrosion protection of the  $\text{ZrO}_2$  film grown by thermal-driven ALD mode (TALD- $\text{ZrO}_2$ ), further reducing the corrosion rate of Mg-Ca alloy. The ALPT effect significantly increases the crystallinity and reduces oxygen defects of the TALD- $\text{ZrO}_2$  film, effectively improving the corrosion resistance of the TALD- $\text{ZrO}_2$  film, and the maximum enhancement is observed by using 30s-ALPT time. The corrosion rate of Mg-Ca alloy is highly reduced by more than one order of magnitude, which is reflected in the reinforcement of diffusion suppression of TALD- $\text{ZrO}_2$  film processed by the ALPT treatment. ALPT technique is suggested as a potential and feasible method to prepare TALD- $\text{ZrO}_2$  films with high film quality and corrosion protection.

**3:20pm MD1-1-WeA-5 Nanospectroscopy and Nanochemical Imaging Using Photothermal AFM-IR on Biomolecular Sensors and Hydrated Self-Assembled-Monolayers, Nafiseh Samiresht (nasajobjo@gmail.com), Max-Planck Institut für Eisenforschung, Germany; G. Figueroa-Miranda, D. Mayer**, Forschungszentrum Juelich GmbH, Germany; M. Rabe, MPI für Eisenforschung GMBH, Germany

Nanostructured self-assembled-monolayers (SAMs) on solids are common platforms for functionalizing and controlling chemical and physical properties of surfaces with applications in diverse fields such as corrosion, catalysis and bio-sensors. Here Resonance enhanced AFM-IR is used to obtain nanoscale topographic information and spectroscopic, i.e. chemical information on such systems [1], in order to better understand the interplay between nanostructure and function. A tunable pulsed quantum cascade including four chips and a fast optical parametric lasers provides the IR source in the ranges of  $910\text{-}1900\text{ cm}^{-1}$  and  $2700\text{-}3900\text{ cm}^{-1}$ . Using a gold coated silicon cantilever on gold substrate provided high sensitivity to detect monolayer and single molecule.

First, results on aptamer SAMs on gold used as SARS-CoV-2 biosensors will be presented. Tapping mode AFM-IR was employed to characterize the surface after each fabrication step and after analyte binding to the receptor layer to provide insight to molecular conformational and structural variation as well as chemical composition of this system. Binding of single molecule proteins was detected (figure 1 in supplemental document).

Second, biomolecule-repellent oligoethylene glycol (OEG)-based SAMs on gold were studied. In such systems the molecular conformation of OEG and the structure of interfacial water are thought to strongly influence the repellent character [3]. Tapping mode AFM-IR investigation of water films adsorbed on nano-domains of OEG SAMs were performed under elevated relative humidity and provided structural details of the OEG moieties and adsorbed  $\text{H}_2\text{O}$ .

[1] G. Figueroa-Miranda, C. Wu, Y. Zhang, L. Nörbel, Y. Lo, J. A. Tanner, L. Elling, A. Offenhäusser, D. Mayer, *Bioelectrochemistry*, **136**, 107589, 2020.[2] P. Harder, M. Grunze, R. Dahint, G. M. Whitesides, P. E. Laibinis, J. Phys. Chem. B **102**, 426-436, 1998

**3:40pm MD1-1-WeA-6 An Electrochromic IrOx Nanofibrous Film for Multifunctional Bio-Interface Sensing Applications, Yu-Jen Tao (stephanie881111@gmail.com), P. Chen**, National Taipei University of Technology, Taiwan

Bio-interface sensing has attracted a lot of attention in recent years. For example, hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) and pH variation are essential detection targets in various fields, including clinical control and environmental protection. This present work reports a design of a non-enzymatic  $\text{H}_2\text{O}_2$  electrochemical sensor and an electrochromic pH sensor with IrOx nanofibrous film. Additionally, the electrochemical performance of the IrOx nanofibrous film can be manipulated by the annealing parameters. The resultant materials are characterized through field emission scanning electron microscopy, X-ray diffraction analysis, and X-ray absorption

spectroscopy. Furthermore, the electrochemical sensing performance of the IrOx nanofibrous film is evaluated by cyclic voltammetry, electrochemical impedance spectroscopy, and chronoamperometric (i-t) techniques. The sensitivity of the IrOx nanofibrous film is checked to investigate the effect of annealing parameters on the H<sub>2</sub>O<sub>2</sub> sensing. The performance of the nanofibers in the electro-reduction of H<sub>2</sub>O<sub>2</sub> is programmable by controlling the metallic Ir contents. The IrOx nanofibrous film annealed at 550°C with a ramping rate of 2.5 °C exhibits better electrocatalytic activity towards the electro-reduction of H<sub>2</sub>O<sub>2</sub>. Further, the broad linear range (0.1 to 1000 µM), low detection limit (LOD) of 0.16 µM with an excellent sensitivity is successfully achieved. The IrOx nanofibrous film can electrochromically detect the pH variation with a super-Nernst sensitivity of 80 mV/pH. Additionally, the IrOx nanofibrous film has appreciable selectivity in the presence of potentially interfering biological molecules, and its practical applicability is demonstrated in MCF-7 human breast cancer cells.

4:00pm **MD1-1-WeA-7 Bespoke Atmospheric Pressure Plasma Polymerization Process with an Acrylic Acid-Based Hybrid Precursor on Polylactic Acid Nonwoven for Antibacterial Scaffolds, Wei-Yu Chen (wychen@mail.mcut.edu.tw), Y. Chiang, T. Chu, L. Chang, J. Lee, Ming Chi**  
University of Technology, Taiwan

There is presently considerable interest in applying polylactic acid (PLA) nonwoven as a scaffold material in biomaterials due to its porous structure, biodegradability, favourable mechanical properties and renewable nature. However, the chemically-inert and hydrophobic surface of PLA limits its biocompatibility and poses challenges to improve its antibacterial ability through modification for inhibiting postoperative infection. In addition, PLA nonwoven is sensitive to most chemical methods for both functionalization and sterilisation. To tackle these issues without impairing the PLA nonwoven, a tailored atmospheric pressure plasma (APP) system along with a hybrid precursor of acrylic acid and silver nitrate was designed and employed for surface functionalization. In this system, electrons and reactive species created during the APP process were utilised for reducing silver nanoparticles from the hybrid precursor. By performing APP polymerization and reduction simultaneously, a silver nanoparticle-embedded and carboxyl-rich polymerized film was prepared and deposited on the PLA nonwoven surface. This study presents a comprehensive analysis of the wettability, hydrophilicity stability, surface elemental composition, biocompatibility and antibacterial efficacy of the PLA nonwoven surface functionalized using the proposed APP method. Compared with conventional methods, this process is capable of immobilising a higher percentage of carboxyl functional groups with improved efficiency on enhancing antibacterial properties.

Keywords: atmospheric pressure plasma, textile functionalization, biodegradable polymers, polylactic acid, scaffolds, acrylic acid, silver nitrate, antibacterial property

## Coatings for Biomedical and Healthcare Applications Room Palm 3-4 - Session MD1-2-ThM

### Surface Coatings and Surface Modifications in Biological Environments II

**Moderator: Mathew T. Mathew**, University of Illinois College of Medicine at Rockford and Rush University Medical Center, USA

**8:40am MD1-2-ThM-3 The Biocompatibility of Thermal Sprayed Bioactive Glass Hydroxyapatite Compositized Coatings, Pin-Jie Chen (mtou335@gmail.com), C. Wu, R. Chung, Y. Yang**, National Taipei University of Technology, Taiwan

In recent years, with the trend of demographic changes and aging, the demand for medical care has continued to rise, and the demand for orthopedic medical devices has increased day by day. In order to maximize the benefits of biomedical composite materials. This experiment uses flame spraying (FS) to coat the surface of Ti-6Al-4V to study the effects of modified hydroxyapatite composite coatings prepared with different proportions of bioactive glass powder on bone healing in rats. help. The experiment will be divided into two parts for discussion. The first part is the microstructure observation, phase composition analysis, mechanical properties and bonding strength test of the modified hydroxyapatite composite coating. The acetylene flow rate will be 1.60 Nm<sup>3</sup>/hr, the spray gun speed is 250 mm/s, the spraying distance is 125 mm, and the number of flame spray (FS) sprays is 2 times for animal experiments; the second part is the observation of the bone healing situation of the modified hydroxyapatite composite coating. The coated round rods were implanted into the femurs of 6-week-old SD rats for 2 weeks and 4 weeks. Finally, microstructure and through-compression tests were conducted to test the implant bonding strength.

**9:00am MD1-2-ThM-4 Mineralization Ability and Inflammatory Reaction of hFOB 1.19 and THP-1 Cells on the Surface of the Borided AISI 316 L Steel, Erick Japhet Hernandez-Ramirez (japhet\_hdez@hotmail.com), R. Perez Pasten Borja, Y. Marquez-Flores, N. Hernandez-Delgado**, Instituto Politecnico Nacional, Mexico; **I. Mejia-Caballero**, Universidad Autonoma Metropolitana, Mexico; **I. Campos-Silva**, Instituto Politecnico Nacional, Mexico

Metal-based implants, including stainless steel, titanium-based alloys, and cobalt-based alloys, are the most commonly used materials in orthopaedic implants. They are favored for their cost-effectiveness, exceptional durability, and advantageous mechanical properties. Stainless steel, in particular, offers an economical alternative that can help reduce costs in public health services. However, its wear and corrosion behavior in body fluid environments can lead to various corrosion-related failures, exacerbated by the release of metal ions, such as chromium and nickel, or fretting debris from the steel.

To enhance the performance of stainless steel, a FeB-Fe<sub>2</sub>B layer developed through the boriding process has been applied to 316 L stainless steel. This boride layer generally improves wear and tribocorrosion resistance on the stainless surface. In addition, cytotoxicity results indicate satisfactory properties with regard to the effects on the survival and proliferative activity of human fibroblasts, osteoblasts, and macrophage cells on the surface of the borided AISI 316 L steel.

The hFOB 1.19 (osteoblast cells) mineralization and the evaluation of nitric oxide formation in THP-1 (macrophage cells) on the surface of borided AISI 316 L steel were obtained in this study. The FeB-Fe<sub>2</sub>B layer on the stainless steel was developed using a novel method called the pulsed-DC powder pack boriding process at 900°C for 3600 s, with a 5 A current intensity and cyclic polarity changes of 10 s, resulting in a total boride layer thickness of 20 µm. The mineralization ability of hFOB 1.19 adhered to the borided surface during 1 – 28 days of exposure was analyzed with the alizarin red S assay, while the inflammatory reaction of THP-1 cells to borided AISI 316 L steel was estimated by the Griess reaction after 10 days of exposure. Both assays were also replicated in non-borided AISI 316 L steel.

Notably, for mineralization, the Gompertz and Michaelis-Menten models were used to describe the evolution of the calcium compounds and alkaline phosphatase over the borided and non-borided surfaces.

Based on the mineralization results, it was evident that the FeB-Fe<sub>2</sub>B layer promoted osteoconductive and osteoinductive properties, rendering it a promising bioactive layer. Consequently, the THP-1 cells in contact with the

borided surface exhibited a controlled inflammatory reaction, with NaNO<sub>2</sub> values measuring below 3µM.

**9:20am MD1-2-ThM-5 Tribological Composite Coatings Prepared by Cold Spray, Sima Alidokht (saalidokht@mun.ca)**, Department of Mechanical and Mechatronics Engineering, Memorial University of Newfoundland, Canada **INVITED**

The cold spray process stands out as a cutting-edge coating technology within the thermal spray family. Its versatility spans from component repair to additive manufacturing. Initially, cold spray research predominantly focused on metallic materials within the first decade since its inception. Cold spray suitability is confined to materials exhibiting ductility at high strain rates. While metals remain primary in cold spray, recent research explores developing metal matrix composites, particularly for tribological purposes. This presentation touches upon a number of tribology fundamentals and principles underpinning the deposition of metal matrix composites via cold spray. It provides an overview of advancements in fabricating tribological coatings through this method. Strategies involving powder characteristics and process parameters optimization to enhance coating quality and retain ceramic or solid lubricant will be covered. Despite successful measures in controlling the retention, these changes might unintentionally impact the coatings' tribological properties. The focus will be on the tribology aspects of composite coatings manufactured through cold spray. The presentation also covers structural, chemical, and mechanical changes in third bodies during sliding, offering insights into wear processes influencing cold-sprayed composite coating's wear resistance and friction characteristics. Lastly, we will discuss potential future trajectories for metal matrix composites produced via cold spray and their suitability for tribological applications.

**10:20am MD1-2-ThM-8 Bio-Tribocorrosion Performance of AISI 316 L Steel Enhanced by Pulsed-DC Powder-Pack Boriding, Alan Daniel Contla-Pacheco (aldani.90@hotmail.com)**, TecNM/Tecnologico de Estudios Superiores de Jocotitlan, Mexico; **I. Mejia-Caballero**, Universidad Autonoma Metropolitana-Azcapotzalco, Mexico; **A. Delgado-Brito, V. Castrejon-Sanchez**, TecNM/Tecnologico de Estudios Superiores de Jocotitlan, Mexico; **R. Perez Pasten-Borja, I. Campos-Silva**, Instituto Politecnico Nacional, Mexico

Stainless steel, titanium and CoCrMo alloys are commonly employed in the fabrication of biomedical implants, particularly for artificial replacements like hip and knee joints, owing to their mechanical strength and corrosion resistance. Among these materials, AISI 316 L steel stands out for its corrosion resistance, biocompatibility, affordability, and nonmagnetic properties, making it a frequently utilized biomaterial. However, low hardness and poor tribological properties are the important disadvantages of AISI 316 L steel. In this sense, boriding process is known to increase the surface properties of AISI 316 L steel; the resulting boride layer has excellent wear resistance due to its high hardness, thermal and chemical stability, and adhesion to the substrate material.

This study presents novel insights into the bio-tribocorrosion resistance of borided AISI 316 L steel when immersed in calf serum. Initially, the boride layer (FeB-Fe<sub>2</sub>B) on the steel surface was formed through pulsed-DC powder pack boriding at 950 °C for 0.5 h, using a 10 A of current intensity, and inverse polarity cycle changes every 10 s. Subsequently, a diffusion annealing process, at 1000°C during 3 h in an inert atmosphere, was conducted on the borided AISI 316 L steel to induce a microstructural change in the layer, resulting in the development of a monophasic Fe<sub>2</sub>B layer. In both experimental conditions, the bio-tribocorrosion experiments were performed following the procedure disclosed in the ASTM G119-09 standard; a ball-on-flat tribometer connected with a three-electrode-chemical cell was used for this purpose. The experiments were also replicated in the AISI 316 L steel (reference material).

The results indicated a wear-corrosion regime for both borided AISI 316 L (FeB-Fe<sub>2</sub>B) steel and borided steel exposed to diffusion annealing (Fe<sub>2</sub>B), characterized by a material loss ratio of corrosion to wear (C/W) approximately 0.2. The regime was significantly influenced by the boride layer, enhancing the bio-tribocorrosion resistance on the material surface by approximately 2 times compared to the reference material. In contrast, the reference material exhibited a wear-dominated regime (C/W= 0.095) attributed to passive film removal and high contact pressure in the tribopair, resulting in an increased total material loss rate due to wear.

10:40am **MD1-2-ThM-9 SERS Substrates Based on Self-Organized Dimple Nanostructures on Polyethylene Naphthalate Films Produced via Oxygen Ion Beam Sputtering**, S. Lee, KIMS, Republic of Korea; **Jun-Yeong Yang** ([yjy8184@kims.re.kr](mailto:yjy8184@kims.re.kr)), Korea institute of materials science, Republic of Korea

Surface-enhanced Raman spectroscopy (SERS) utilizes metal nanostructures to enhance the intensity of Raman signals. Although many methods have been developed for fabricating SERS nanostructures, most involve multiple steps. Herein, we employed oxygen ion beam sputtering (IBS), a one-step technique suitable for processing flexible substrates in roll-to-roll processes for mass production. Specifically, one-step oxygen IBS was used to fabricate self-organized dimple nanostructures, whose area and roughness could be controlled using the ion irradiation [<https://www.sciencedirect.com/topics/physics-and-astronomy/ion-irradiation>] energy density, on the surfaces of polyethylene naphthalate films. Gold nano-tips for SERS were subsequently obtained by evaporating gold onto the dimple nanostructures. Finite-difference time-domain (FDTD) simulations revealed that nano-tip structures with spacings of less than 10 nm increased the localized E-field enhancement, which improved the SERS signal. Fabrication at a low energy density ( $5.8 \text{ J/cm}^2$ ) produced more nano-tips with spacings of less than 10 nm, corresponding to a density of  $61.4 \text{ nano-tips}/\mu\text{m}^2$ . SERS analysis conducted with methylene blue at 638 nm and 785 nm demonstrated that the Raman signal intensity was stronger for SERS substrates fabricated with low energy density ( $5.8 \text{ J/cm}^2$ ) than for substrates fabricated with high energy density ( $17.3 \text{ J/cm}^2$ ), because of the high density of nano-tips on the former substrate.

11:00am **MD1-2-ThM-10 Design and Fabrication of a Hybrid IrOx/Polydopamine Thin Film via a Co-Electrodeposition Process as a Bendable Bio-Interface Microelectrode Array**, **Hung-Yu Chen** ([bryan950396@gmail.com](mailto:bryan950396@gmail.com)), M. Tsou, National Taipei University of Technology, Taiwan; K. Tso, K. Sasagawa, J. Ohta, Nara Institute of Science and Technology, Japan; P. CHEN, National Taipei University of Technology, Taiwan

Iridium oxide (IrOx) is a promising electrode material for implantable neural therapeutic devices owing to its remarkable performance on bio-interfaces. We demonstrate a unique chemical formula to co-electrodeposit hybrid iridium oxide thin film with polydopamine (PDA) on a flexible Parylene C substrate. In a mild alkaline solution, electrochemical deposition facilitated the formation of inorganic/organic nanoparticle (NP) corona structures. The NP-corona with an order atomic structure enabled enhanced electrochemical stability and bioactivity. The incorporated PDA contributed to nanorough surface structures that led to higher current storage capacity (CSC) and lower impedance than that of pristine IrOx. The IrOx/PDA microelectrode array also demonstrates an excellent sensitivity to dopamine which is an important neurotransmitter related to brain diseases. In addition, the hybrid IrOx/PDA thin film reveals an impressive mechanical property. In a bending test of 15,000 cycles, the hybrid IrOx/PDA thin film retains 90% of its initial CSC without any physical crack or delamination. Our results provide solid evidence of fabricating a robust flexible electrode for neural interfaces for potential use in implantable electronic devices.

## Coatings for Biomedical and Healthcare Applications Room Palm 3-4 - Session MD2-ThA

### Medical Devices: Bio-Tribo-Corrosion, Diagnostics, 3D Printing

**Moderators:** Steve Bull, Newcastle University, UK, Hamdy Ibrahim, University of Tennessee at Chattanooga, USA

**2:00pm MD2-ThA-3 Corrosion Risk Analysis of CoCrMo alloy as a Function of Microstructure: Biomedical Applications, Maansi Thapa (mthapa3@uic.edu),** University of Illinois at Chicago, USA; Y. Sun, B. Keaty, M. Mathew, C. Takoudis, M. Daly, D. Ozevin, University of Illinois - Chicago, USA

CoCrMo alloys are widely used in orthopedic implants and various biomedical applications, exhibit excellent corrosion resistance and mechanical properties. However, it has raised concerns about inferior corrosion behavior and subsequent side effects due to metal ion release. While the electrochemical nature of this alloy is well studied, the microstructure's effect needs further research. The objective of this study is to evaluate corrosion behavior of CoCrMo alloys in two microstructures: unbanded (transverse) and banded (longitudinal).

Six CoCrMo disks (11x7mm) were prepared and polished following metallographical protocol for a surface finish of <50 nm. The unbanded CoCrMo rod was cut perpendicular to the axis, while the banded CoCrMo rod was cut parallel to the axis. The electrolyte used was bovine calf serum (30 g/L proteins) with a pH of 7.6 to simulate the joint environment. The electrochemical test followed ASTM G61 standard using a three-electrode system: the sample as the working electrode, a saturated calomel electrode (SCE) as a reference electrode, and a graphite rod as the counter electrode. The typical protocol involved: open circuit potential (OCP), electrochemical impedance spectroscopy (EIS), and cyclic polarization curve. Using Tafel's estimation, the corrosion potential ( $E_{corr}$ ) and corrosion current ( $I_{corr}$ ) were determined. EIS data was utilized to generate Bode and Nyquist plots and construct an equivalent electric circuit to determine polarization resistance ( $R_p$ ) and double layer capacitance ( $C_{dl}$ ). The corroded surfaces were characterized by white light microscopy, SEM, and EBSD.

Our study showed that the CoCrMo specimens with unbanded microstructures exhibited increased corrosion resistance ( $E_{corr}$ : -0.678 V vs SCE,  $I_{corr}$ : 1.85E-06 A/cm<sup>2</sup>) compared to banded microstructures ( $E_{corr}$ : -0.736 V vs SCE,  $I_{corr}$ : 5.05E-06 A/cm<sup>2</sup>). The EIS data supported this observation, revealing higher  $R_p$  and lower capacitance. SEM observations revealed larger pitting in the banded microstructure compared to unbanded. Previously, Jacob et al.<sup>1</sup> reported superior fretting-corrosion behavior of unbanded microstructures and potential risk of banded microstructures under an infected environment.

The banded microstructure, with increased grain boundary exposure, heightens the risk of intergranular and galvanic corrosion. Further exploration is needed to understand microstructural mechanisms and develop strategies to inhibit increased corrosion risk. Our investigation emphasizes the vital role of material composition and configuration in microstructural and corrosion behavior.

(1)Manthe, J *Mech. Behavior of Biomed Materials*. 2022.

**2:20pm MD2-ThA-4 Comparative Study of Composite Coatings on Magnesium for Biomedical Devices, V. Patil,** University of Tennessee at Chattanooga, USA; B. Williams, University of Arkansas, USA; J. Rich, University of Tennessee at Chattanooga, USA; M. Elsaadany, University of Arkansas, USA; **Hamdy Ibrahim (hamdy-ibrahim@utc.edu),** University of Tennessee at Chattanooga, USA

Magnesium (Mg) and its alloys exhibit a biodegradable nature in aqueous environments, rendering them appealing for diverse biomedical applications where permanent existence in the body is not advisable. However, the utilization of Mg for bone fracture repair faces notable challenges, primarily stemming from its constrained mechanical strength and rapid corrosion rates. To address these issues, extensive research has been directed towards the development of biocompatible coatings that can offer Mg the necessary protection in vivo. In this study, we have formulated various composite coatings and conducted a comprehensive assessment of their properties, including corrosion rates, biocompatibility, adhesion strength, and surface morphology. The evaluated composite coatings were derived from three distinct processes: Plasma Electrolyte Oxidation (PEO), sol-gel coating, and polymer dip coating. Results indicated a substantial

enhancement in corrosion resistance within the sol-gel coated composite group. Moreover, the polymer-coated groups demonstrated superior osseointegration and biocompatibility. This investigation underscores the feasibility of producing biocompatible magnesium-based implants with enhanced strength and controlled corrosion properties through the application of diverse composite coatings.

**2:40pm MD2-ThA-5 Laser-Induced Graphene Coatings on Polymers for Biomedical Devices, Mostafa Bedewy (mbedewy@pitt.edu),** University of Pittsburgh, USA

**INVITED**

Nanocarbons like graphene and related materials are promising for various biomedical applications; however, major manufacturing challenges still hinder our ability to scalably produce graphene with tailored morphology and surface chemistry, especially on flexible and polymeric substrates. While chemical vapor deposition (CVD) processes enable the synthesis of high-quality graphene, the typically high temperatures in such reactors limit the choice of substrates to silicon, quartz, metals or other temperature-resistant materials. On the other hand, emerging flexible devices, such as implantable surgical meshes and biosensors require the fabrication of such nanocarbon coatings and electrodes directly on polymers. Unlike different transfer techniques of CVD-grown nanocarbons, or printing methods from inks, this talk will focus on a bottom-up approach for directly growing different types of graphenic nanocarbons on aromatic polymers by laser irradiation. The speaker will present an approach that leverages this direct-write process, often referred to as laser-induced graphene (LIG), for creating spatially-varying morphologies and chemical compositions of LIG electrodes, by leveraging gradients of laser fluence. Three distinct morphologies are identified, and process control map is generated for maximizing the electrical conductivity of these porous graphene for biomedical devices. Moreover, this talk will introduce a method for controlling heteroatom doping of LIG based on controlling the molecular structure of the polymer being lased, i.e. by introducing sulfur- and fluorine- containing backbones. We demonstrate superhydrophobic and parahydrophobic surface properties for the fluorine-doped LIG patterns. We also show antibacterial properties of LIG coated surgical devices. Finally, a demonstration of these functional doped LIG electrodes as electrochemical biosensors will be presented for the detection of the neurotransmitter dopamine with nanomolar sensitivity.

**3:20pm MD2-ThA-7 Microfluidic Device for the Isolation, Detection, and Purification of Exosomes Based on Metallic Nanostructure Arrays, Alfreda Krisna Altama (alfredakrisna@gmail.com), Y. Hsiao, C. Chen,** National Taiwan University of Science and Technology, Taiwan; R. Haliq, National Taiwan University of Science and Technology, Indonesia; P. Yiu, Ming Chi University of Technology, Taiwan; P. Wu, J. Chu, National Taiwan University of Science and Technology, Taiwan

Due to their low cost, rapid processing, and ability to analyze even minuscule samples, microfluidic devices are widely used in disease detection and specimen separation. In this study, metallic nanostructure arrays (MeNTAs) with tube-like features are embedded into microfluidic devices for immunoaffinity-based detection and efficient exosome isolation. MeNTA candidates were evaluated based on their ability to withstand mechanical stress during microfluidic operations, X-ray diffraction, zeta potential, and electrostatic interactions. The Zr<sub>60</sub>Cu<sub>25</sub>Al<sub>10</sub>Ni<sub>5</sub> thin film metallic glass (Zr-TFMG) exhibited superior mechanical properties and a negative zeta potential compared to other materials. (e.g., Cu, Bronze, Ag, 7075Al, Ti64, 718Ni, SS316, Cu-TFMG, W-TFMG, and Al-TFMG). The resultant microfluidic device featured Zr-based MeNTAs with an interdigital electrode in a microchannel. In testing with derived exosomes (liquid biopsy of 500 µL), the device achieved a 95.3% exosome recovery rate within 1 hour while resisting nonspecific binding to HeLa-derived exosomes (recovery rate < 0.1%). The device facilitated the isolation of 1 x 10<sup>8</sup> exosome particles per mL for electrochemical impedance spectroscopy detection and allowed efficient release of captured exosomes via cyclic voltammetry operations. The proposed Zr-MeNTA microfluidic device holds significant potential for the isolation, detection, and purification of exosomes in liquid biopsy samples for cancer diagnosis, as mentioned by Hsiao et al. (2023).

## Coatings for Biomedical and Healthcare Applications Room Golden State Ballroom - Session MD-ThP

### Coatings for Biomedical and Healthcare Applications (Symposium MD) Poster Session

**MD-ThP-1 Investigation of Silver/Copper Diffusions in the Matrix of Amorphous Carbon Thin Films Produced by Magnetron Sputtering, Hailin Sun (hailin.sun@teercoatings.co.uk), Teer Coatings Ltd, UK**

The environment inside a spacecraft is ideal not only for the members of the crew onboard, but also for bacteria and fungi to grow. The proliferation of harmful microorganisms can become a hazard for the human crew as well as for the safe running of equipment. In our previous work, we used magnetron sputtering to develop amorphous carbon coatings doped with silver and copper for antimicrobial application in space stations, and the benefits of the bactericidal properties added by silver- and copper-doping were shown under both terrestrial gravity and micro-gravity conditions [1]. In addition, these thin films are scratch-resistant and wear-resistant with high hardness, providing a long lifetime which is critical for the applications in a space station.

The prepared Ag- and Cu-doped amorphous carbon coatings showed a slow diffusion of Ag from the carbon matrix to the surface, eventually replenishing the Ag at the surface lost due to daily wear and tear. Such diffusion process is a key factor in the coating performance: if too fast, the antimicrobial lifetime of the coating would be shorter, if too slow the bactericidal efficiency of the coating would be affected. Therefore, it becomes apparent and critical to identify the key factors that influence the Ag diffusion rate in a carbon matrix, and also to understand how they influence it.

In this work we report the latest study on Ag and Cu diffusion in Ag- and Ag/Cu-doped amorphous carbon coatings. Samples with the same concentration of Ag and different concentrations of Cu have been prepared and annealed in oven at 100 °C, 150 °C and 200 °C to speed up the diffusion process of the metals. With the combination of RBS (Rutherford Backscattering Spectrometry) and ToF-ERD (time of flight elastic recoil detection) the elemental depth profile is accurately measured, which is supported by XPS data to investigate the chemical state of the species at the surface. Preliminary results have shown that higher temperature causes a higher diffusion rate, and the addition of copper has slowed down the diffusion rate of silver, which is confirmed also by cross-section SEM images. Interestingly, XPS data show how Ag retains always its metal state and does not oxidize, while Cu bonds with carbon, oxygen and hydrogen to form more complex molecules such as Cu(II) carbonate dihydroxide.

#### References

[1] G. Sanzone et al., "Antimicrobial and aging properties of Ag-, Ag/Cu- and Ag cluster-doped amorphous carbon coatings produced by magnetron sputtering for space applications", *ACS Appl. Mater. Interfaces* 14 (2022) 10154–10166 (doi.org/10.1021/acsami.2c00263)

**MD-ThP-2 Enhanced Biomedical Implant Surfaces: Stainless Steel Modification Through Hipims-Coated Titanium and Peo Treatment, Bruno Pereira (brnlp7@gmail.com), Pontifícia Universidade Católica do Paraná, Luxembourg; L. Fontana, Universidade do Estado de Santa Catarina, Brazil; C. Lepienski, Universidade Federal do Paraná, Brazil; P. Soares, Pontifícia Universidade Católica do Paraná, Brazil**

The main causes of failures in implantable devices are often attributed to bone reabsorption, due to a mismatch in elastic modulus at the implant-bone interface, as well as and bacterial infections. Plasma electrolytic oxidation (PEO) is a versatile surface modification technique for metals, such as Titanium (Ti), and is capable of producing coatings that exhibit a reduced elastic modulus. Moreover, this method can incorporate bactericidal elements, such as Copper (Cu), achieving a durable antibacterial effect. However, PEO is not directly applicable to stainless steel, which is frequently employed as biomaterial. In this study, austenitic stainless steel was coated with titanium using High Power Impulse Magnetron Sputtering (HIPIMS). The Ti-coated steel (SS-Ti) was subsequently subjected to the PEO process to improve the surface properties essential for implantable devices. The PEO process involved an electrolyte mixture of calcium acetate, calcium glycerophosphate, and copper sulphate. Post-PEO surfaces were examined by Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS). Crystalline structures were characterized using X-ray diffraction (XRD), while the

thickness of the layers was measured through cross-sectional analysis. Nanoindentation tests were employed to measure the hardness (H) and elastic modulus (E) of the layer, while nano-scratch tests were performed to evaluate the layer adhesion. The resulting coating presented a homogeneous, porous oxide structure containing calcium (Ca), phosphorus (P), sulphur (S), copper (Cu), and crystalline anatase (TiO<sub>2</sub>). The modified oxide layer was approximately 4 µm thick. Nanoindentation tests indicated a considerable reduction in the elastic modulus (~50%) compared to SS, while the scratch tests showed strong adhesion of the Ti+oxide layer to the SS substrate, with no exposure of the SS substrate in the scratched regions. The resulting coating, due its properties, displayed potential for use in biomedical applications.

**MD-ThP-3 Nanospectroscopy and Nanochemical Imaging Using Photothermal AFM-IR on Biomolecular Sensors and Hydrated Self-Assembled-Monolayers, Nafiseh Samisereht (nasajobjo@gmail.com), MPI für Eisenforschung GMBH, Germany; G. Figueroa-Miranda, D. Mayer, Forschungszentrum Juelich GmbH, Germany; M. Rabe, MPI für Eisenforschung GMBH, Germany**

Nanostructured self-assembled-monolayers (SAMs) on solids are common platforms for functionalizing and controlling chemical and physical properties of surfaces with applications in diverse fields such as corrosion, catalysis and bio-sensors. Here Resonance enhanced AFM-IR is used to obtain nanoscale topographic information and spectroscopic, i.e. chemical information on such systems [1], in order to better understand the interplay between nanostructure and function. A tunable pulsed quantum cascade including four chips and a fast optical parametric lasers provides the IR source in the ranges of 910-1900 cm<sup>-1</sup> and 2700-3900 cm<sup>-1</sup>. Using a gold coated silicon cantilever on gold substrate provided high sensitivity to detect monolayer and single molecule.

First, results on aptamer SAMs on gold used as SARS-CoV-2 biosensors will be presented. Tapping mode AFM-IR was employed to characterize the surface after each fabrication step and after analyte binding to the receptor layer to provide insight to molecular conformational and structural variation as well as chemical composition of this system. Binding of single molecule proteins was detected.

Second, biomolecule-repellent oligoethylene glycol (OEG)-based SAMs on gold were studied. In such systems the molecular conformation of OEG and the structure of interfacial water are thought to strongly influence the repellent character [3]. Tapping mode AFM-IR investigation of water films adsorbed on nano-domains of OEG SAMs were performed under elevated relative humidity and provided structural details of the OEG moieties and adsorbed H<sub>2</sub>O.

[1] G. Figueroa-Miranda, C. Wu, Y. Zhang, L. Nörbel, Y. Lo, J. A. Tanner, L. Elling, A. Offenhäusser, D. Mayer, *Bioelectrochemistry*, **136**, 107589, 2020.[2] P. Harder, M. Grunze, R. Dahint, G. M. Whitesides, P. E. Laibinis, *J. Phys. Chem. B* **102**, 426-436, 1998

**MD-ThP-4 Development of Hierarchical Surfaces Coated with Zinc Nanoparticle-Doped Polycaprolactone on 316LVM Stainless Steel Substrate for Biomedical Applications, Tarciana Dieb Toscano (tarcianadieb@gmail.com), Pontifícia Universidade Católica do Paraná (PUCPR), Brazil; A. Bhattacharjee, Colorado State University, USA; K. C. Popat, George Mason University, USA; P. Soares, Pontifícia Universidade Católica do Paraná (PUCPR), Brazil**

The use of 316LVM stainless steel in medical implants, while advantageous due to its mechanical strength and biocompatibility, poses a significant challenge in the form of bacterial infections. These infections occur when bacteria adhere to the implant surface, forming biofilms that are resistant to antibiotics and immune responses. This can lead to persistent infections, causing complications such as implant failure, the need for surgical revision, and prolonged patient suffering. It is known that nanostructured and biomimetically textured surfaces have demonstrated superiority over smooth surfaces in bacterial inactivation and reduction of bacterial adhesion. Bacterial inactivation through surface morphology can occur through a physical-mechanical mechanism. By integrating bactericidal elements into these nanostructured surfaces, it is possible to create a route for physical-mechanical and chemical inactivation. Thus, the surface of 316LVM steel was modified using the cathodic plasma electrolytic oxidation process, followed by acid etching to promote surface nanostructuring. This nanostructured surface was subsequently coated with polycaprolactone doped with zinc oxide nanoparticles (nP-ZnO/PCL). The objective of this study is to evaluate the morphological, topographical, and chemical properties, along with the wettability and electrochemical response of the textured surfaces. Additionally, the effect of surface texturing on the

adhesion of the nP-ZnO/PCL coating was investigated. The bactericidal effect of the nanotextured and nP-ZnO/PCL-coated surfaces was also assessed using *Staphylococcus aureus* bacteria. For this purpose, scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS), X-ray diffraction (XRD), contact angle measurements using a sessile drop goniometer, and evaluation of the electrochemical response through potentiodynamic polarization tests were employed. Coating adhesion to the substrate was assessed through scratch tests. The bactericidal effect was evaluated using colorimetric assays and live/dead bacterial quantification. The results showed that the application of cathodic plasma electrolytic processes in conjunction with acid treatment successfully induced surface nanostructuring. There was an improvement in corrosion resistance properties and an increase in surface contact angle for the textured and coated samples, along with a significant reduction in bacterial adhesion. It was also observed that the zinc oxide-doped polymeric coating exhibited better adhesion on textured surfaces compared to polished material.

**MD-ThP-5 Catastrophic Corrosion in Metal Guitar Strings with or Without DLC Films Using Artificial Sweat**, *C. Andrés Velásquez Andrade*, Universidade do Vale do Paraíba, Brazil; *N. Pereira Alves Granado*, IGT PAN, Brazil; *Lucia Vieira (lvs.lucia@gmail.com)*, Universidade do Vale do Paraíba - Univap, Brazil

Diamond-like carbon (DLC) film was deposited on electric guitar strings to evaluate corrosion under the impact of artificial sweat at a constant temperature of 37 degrees Celsius. The objective was to assess the effect of corrosion on the change in string mass over time and the string tonality variation. The process involves the application of a DLC by plasma-enhanced chemical vapor deposition (PECVD), recognized for its corrosion-resistant film. The strings' mass changes due to exposure to artificial sweat were measured over 240 hours, focusing on mass variation to obtain information on the strings' durability and corrosion resistance. The results show that all strings suffered corrosion, and the tonality (frequency variation of the notes) varied due to the film of the strings. The results can help design electric guitar strings with better tonal qualities and corrosion resistance. Understanding how external elements such as sweat affect the integrity of the DLC film contributes to the sound quality and longevity of the strings.

**Keywords:** (mass variation; Corrosion; artificial sweat; nickel plated steel; Raman; guitar strings; DLC; PECVD)

## Coatings for Biomedical and Healthcare Applications Room Palm 3-4 - Session MD3-FrM

### Bioactive Surfaces

**Moderators:** Valentim A.R. Barão, University of Campinas (UNICAMP), Brazil, Sandra E. Rodil, Universidad Nacional Autónoma de México

8:20am **MD3-FrM-2 Electrochemical Aspects of Interaction between Surface Engineered Metal Implants and Biological Environment, Aleksey Yerokhin (Aleksey.Yerokhin@manchester.ac.uk), University of Manchester, UK** **INVITED**

Interfacial redox reactions involving charge transfer between metallic biomaterials and biological environment are of particular interest for development of new generation of biomedical implant devices. Controlling reaction kinetics can help achieving the targeted biological functionality such as osteogenic and biocidal activity, or drug-release ability of surface engineered smart and multifunctional implants; this is also important when minimising corrosion rates to enhance long-term performance of permanent metal implants or controlling degradation behaviour of bioabsorbable implants. Extensive exploratory research at the interface of biomedical and materials engineering often takes advantage of lab-scale electrochemical methods for express assessment of relevant implant properties. However interpreting results of the tests that were originally designed to evaluate aqueous corrosion of metals in engineering applications and are now being adopted to study more complex interactions between implants and biological environment is not straight forward. This talk will revisit electrochemical fundamentals of most common corrosion tests based on potentiodynamic polarisation and frequency response analysis, with a focus on implications for the assessment of implant material degradation in vitro and relevance of obtained characteristics to the implant performance in vivo. Discussions will be provided into effects that composition of simulated biological media, implant surface state and test conditions may have on basic kinetic parameters of anodic and cathodic processes, including exchange current densities, Tafel slopes and limiting current densities, and how these are reflected in polarisation curves. Attention will further be drawn to the multistage nature of the interfacial charge transfer process and correct evaluation of contribution from different stages to the overall corrosion kinetics that inform the strategies for environmental degradation control of biomedical implant materials.

9:00am **MD3-FrM-4 New Approach for Controlling Peri-Implant Infections Integrates Multifunctional Photocatalytic Coating and Photodynamic Therapy – an in Vitro and in Vivo Study, Valentim A. R. Barão (vbarao@unicamp.br), B. Nagay, R. Costa, C. Dini, A. Santos, University of Campinas (UNICAMP), Brazil; L. Cintra, Sao Paulo State University (UNESP), Brazil; N. da Cruz, L. Faverani, São Paulo State University (UNESP), Brazil; J. van den Beucken, Radboudumc, Netherlands**

Although peri-implant infections reduce the longevity of dental implants, there is still no gold-standard therapeutic strategy. Therefore, here we developed a visible light-responsive multifunctional bismuth (Bi)-TiO<sub>2</sub> coating on titanium (Ti) surface to optimize the properties of dental implants and enhance antimicrobial photodynamic therapy (aPDT)-mediated microbial reduction. Bi-TiO<sub>2</sub> experimental coating was synthesized via plasma electrolytic oxidation (PEO). TiO<sub>2</sub> and polished Ti were controls. Topographic, physicochemical, tribological, structural and photocatalytic properties were analyzed. In vitro microbiological assays were performed under different light times (0, 1 and 5 min). In vitro cytocompatibility was evaluated in mesenchymal cells and gingival fibroblasts. The antimicrobial activity and inflammatory response were investigated in vivo (in a subcutaneous tissue of rats). PEO created rough, superhydrophilic and crystalline surfaces, with higher hardness values and tribological performance compared to Ti control ( $p < 0.05$ ). Bi-TiO<sub>2</sub> was not cytotoxic and enhanced the microbial reduction mediated by aPTD ( $p < 0.05$ ) by presenting photocatalytic activity under visible light. The combination of Bi-TiO<sub>2</sub> and aPDT reduced microbial viability and modulated the inflammatory response in vivo ( $p < 0.05$ ). The Bi-TiO<sub>2</sub> coating is a promising strategy for rehabilitation with dental implants as it presents optimized surface properties and enhances microbial reduction and inflammatory modulation mediated by aPDT.

Note: This study was supported by the Sao Paulo Research Foundation (FAPESP, Brazil) (grant numbers: 2019/17238-6 and 2022/16267-5).



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