

Surface Engineering of Biomaterials, Devices and Regenerative Materials: Health, Food, and Agriculture Applications

Room Town & Country B - Session MD2-2-ThM

Coatings and Sensors for Health, Food and Agriculture: Antibacterial, Bioactive, and Flexible Interfaces II

Moderators: **Valentim A.R. Barão**, University of Campinas (UNICAMP), Brazil, **Jean Geringer**, Ecole Nationale Supérieure des Mines, France

8:00am **MD2-2-ThM-1 Surface Modification of AZ31B by Oxygen-Plasma Immersion Ion Implantation to Promote Schwann Cell Interaction for Peripheral Nerve Regeneration**, **Luciana Malvestiti [luciana.malvestiti.1@ulaval.ca]**, **Carlo Paternoster**, **Francesco Copes**, LBB, CHU de Quebec research center, Laval University, Canada; **Paolo Mengucci**, **Giani Barucca**, Department SIMAU, Università Politecnica delle Marche, Ancona, Italy; **Silvia Ceré**, INTEMA-CONICET, Mar del Plata National University, Argentina; **Andranik Sarkissian**, Plasmionique Inc., Varennes, QC, Canada; **Diego Mantovani**, LBB, CHU de Quebec research center, Laval University, Canada

Their biocompatibility, electrical conductivity and biodegradability properties, make Mg-based alloys a potential biomaterial for peripheral neuropathy. Even if these alloys release Mg²⁺ cations, fundamental in neurological functions, their high corrosion rate triggers implant failure and tissue damage. To control the degradation pattern, and improve the biological response, a plasma-based technique (oxygen-plasma immersion ion implantation, O-PIII) was used on a Mg-based substrate (AZ31B) to generate a thin MgO layer. In addition, other modified surface properties such as roughness and surface energy, improved the general biological response of the material

O-PIII was performed in a PBII-300 system (Plasmionique) on the surface of chemically polished (CP) AZ31B (Al 3 wt.%, Zn 1 wt.%, Mg bal.) specimens. Pressure (5 to 10 mTorr), and pulse repetition rate (200 to 1000 Hz) were working parameters. The morphological, chemical and electrochemical characterization was performed with scanning electron microscopy (SEM), X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), static drop contact angle, and polarization curves in Hanks' solution. The cytotoxicity of the corrosion products toward Schwann cells (SC), and the interaction between the modified alloy and SC were studied through an indirect test and adhesion test, respectively.

Plasma treatment introduced different surface features depending on the used parameter values, which affected the morphology, roughness, chemical composition, and corrosion resistance. After O-PIII, XPS revealed an increase in MgO content, accounting for the ~70% total detected oxygen. The hydrophilicity of the treated surfaces decreased, from ~40° for CP to ~75°-100° after O-PIII, being within the range that promotes protein adsorption². Applying O-PIII, the corrosion rate was reduced, improving also the corrosion pattern of the material. SC exposure to 10% and 1% extracts did not show a relevant cell viability reduction. After 6 h of incubation, SC were adhered on the modified surfaces exhibiting an elongated morphology, which could be compatible with a regenerative phenotype.

O-PIII modified the AZ31B surface topography, chemical composition (Mg oxide/hydroxide related species), and wettability, enhancing its corrosion resistance. Low concentration of corrosion products did not affect SC viability, moreover, the modified surface allowed SC attachment. These results support the use of O-PIII technique as a potential surface modification for AZ31B, constituting a valid approach for peripheral nerves regeneration.

8:20am **MD2-2-ThM-2 Multifunctional PEO-PPy/Zn Coatings Combined with Electrical Stimulation for Enhanced Antimicrobial and Osteogenic Titanium Surfaces**, **Valentim A. R. Barão [vbarao@unicamp.br]**, **Maria Helena R. Borges**, **Samuel Santana Malheiros**, **Julia M. Teodoro**, University of Campinas (UNICAMP), Brazil; **João Gabriel S. Souza**, Guarulhos University (UNG), Brazil; **Elidiane C. Rangeli**, Sao Paulo State University (UNESP), Brazil; **Ana Paula Souza**, **Bruna Egumi Nagay**, University of Campinas (UNICAMP), Brazil

Dental implant failure often results from polymicrobial biofilm infections and poor osseointegration, underscoring the need for multifunctional surface modifications of titanium (Ti). Here, we developed a plasma electrolytic oxidation (PEO) coating followed by the electrodeposition of the conductive polymer polypyrrole (PPy) and the antimicrobial agent zinc (Zn),

combined with osteogenic stimulation through electrical therapy (ES). We systematically investigated the surface, mechanical, physicochemical, tribological, electrochemical, antimicrobial (mono- and polymicrobial biofilms), and biological properties, as well as osteogenic activity using MC3T3-E1 cells. Ti discs were prepared as four groups: (1) machined, (2) PEO, (3) PEO+PPy, and (4) PEO+PPy/Zn, each evaluated with or without ES. The PEO+PPy and PEO+PPy/Zn surfaces exhibited enhanced mechanical strength, tribocorrosion resistance, reduced wear, and increased hardness ($p < 0.05$). Zn incorporation imparted pronounced antimicrobial effects, significantly decreasing biofilm viability and metabolic activity ($p < 0.05$), while promoting protein adsorption ($p < 0.05$). Moreover, ES further improved cell proliferation, osteogenic differentiation, and mineralized nodule formation ($p < 0.05$). Collectively, the multifunctional PEO+PPy/Zn coating, particularly when combined with ES, shows strong potential to enhance implant longevity by strengthening substrate resistance, preventing microbial colonization, and stimulating osteogenesis.

8:40am **MD2-2-ThM-3 Effect of Zirconium Addition on Zn- and Mg-Based Thin Film Properties Deposited by Magnetron Sputtering for Intravascular Biodegradable Materials**, **Fatiha Challali [fatiha.challali@univ-paris13.fr]**, **Cristiano Poltronieri**, Laboratoire des Sciences des Procédés et des Matériaux (LSPM) – CNRS, France; **Vinicius De Oliveira F. Sales**, **Carlos Henrique Michelin Beraldo**, **Carlo Paternoster**, Université Laval, Canada; **Frédéric Chaubet**, Université Sorbonne Paris Nord, France; **Philippe Djemia**, Laboratoire des Sciences des Procédés et des Matériaux (LSPM) – CNRS, France; **Diego Mantovani**, Université Laval, Canada

Intravascular medical devices allow the treatment of internal vessel-related diseases circumventing open-surgery, in daily-hospital treatment, and with great benefits for patients. The thickness of the devices is inversely proportional to how far in vascular bed diseased sites can be accessed, especially for cerebrovascular applications. Unhappily, the fabrication of thin and ultrathin (hundreds to tens of microns) metallic implants remains a key challenge for these applications. Moreover, biodegradable metals are now a reality for adding the degradability components to these devices. For thin intravascular applications, zinc-based alloys are promising candidates due to their moderate degradation rates compared to magnesium-based materials, despite mechanical properties and corrosion rate still need to be investigated. The uniformity of the expected degradation also constitutes a main bottleneck, and metallic glasses, being exempted by surface defects like grain joints, provide a new insight, as recently shown on amorphous Zn–Mg–Ca. Thin film metallic glasses (TFMGs), characterized by a disordered atomic structure, exhibit exceptional mechanical properties, including a large elastic limit (>2%), high hardness, and yield strength (>2 GPa). Their homogeneous atomic arrangement promotes uniform corrosion with tunable rates depending on alloy composition.

This work aimed to enhance the glass-forming ability (GFA) and achieve tunable corrosion rates in Zn- and Mg-based thin films through the addition of Zr. Incorporating Zr as a glass-forming element is an effective strategy to extend the compositional ranges of amorphous or nanocrystalline alloys. The addition of Zr enables precise control over the microstructure and crystallinity, facilitating the design of biodegradable materials with improved performance. Thin films of Zn–Zr and Mg–Zr binary alloys were synthesized by magnetron co-sputtering onto silicon substrates using pure metallic targets, with thicknesses ranging from 300 to 900 nm, covering a wide composition range. The film microstructure and chemical composition were analyzed by XRD and SEM/EDS, respectively. Corrosion behavior was evaluated through electrochemical measurements performed at room temperature, while biodegradability was assessed by immersion tests in simulated body fluid at 37 °C for up to eight weeks. XRD results showed that Mg–Zr films exhibited a nanocrystalline structure, while amorphous Zn–Zr films were obtained for Zn contents between 26 and 88 at.%. Immersion tests revealed premature cracking and delamination in Zn- and Mg-rich films after one week, whereas Zr-rich films remained adherent even after eight weeks of immersion.

9:00am **MD2-2-ThM-4 Swelling Effect on Uhmwpe Cup: Long Term Consequences on Malfunctioning the Junction Head-Cup-Metal Back**, **Jean Geringer [geringer@emse.fr]**, **Albert Boyer**, Mines Saint-Etienne, France; **Bertrand Boyer**, Hopital Edouard Herriot - Hospices Civils de Lyon, France; **Frederic Farizon**, CHU Saint-Etienne, France

Objectives

Hip arthroplasty is nowadays a successful surgical operation. 90% of patients, generally speaking, are satisfied and with time (after 10 years) this success rate is decreasing to 80% and less. Some factors are responsible for this decrease: infection, impingement, some pain due to joint blocking. The

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swelling effect of the cup polymer, UHMWPE, should be one of the factors. 0,2% is not a big deal...

In any case, swelling effect should have a minor effect or no effect. Long term action should have an impact and has an effect. On 10cm, we are talking about 2mm, roughly, of dimension change. Let's imagine a quotation change of 2mm on any dimension, radius of sphere, clearance between cup and metal back... The manufacturing lab of any organisation should fire any member if he is saying: 'don't care, pay attention on next...'

Materials and Methods

The cup sample was issued from the SERF™, now Stryker™, It was manufactured in 2019 with traditional manner. The material was UHMWPE polymer, polyethylene was the based material. UHMWPE was real cup. PEEK material was used, under disc. The first weight measurement was in 2008, less than 20 years. A balance with 5 digits was used. The control and the maintenance were always under survey.

New results

UHMWPE is gaining mass with time, after more than 8000 weeks. It means that adsorption is increasing with time. About PEEK material, one may say that stabilisation does occur after 100 weeks, i.e a bit less than 2 years. Thus the mass is stabilized after 2 years.

Conclusions & significance

UHMWPE is constantly increasing his mass with time. PEEK material was maintaining the mass after 2 years. Cup dimensions were changing with time when the material was UHMWPE. On the contrary, PEEK was constant after 15 years.

Acknowledgements

The authors are grateful to Mr. J-L. Aurelle for providing cup samples, ex. Serf company.

9:20am **MD2-2-ThM-5 Effect of Thermal Evaporation Deposited Silver Nanoparticles on the Antibacterial Behavior of Plasma Electrolytic Oxidation Coated AZ31B Magnesium Alloy, Ming-Hsuan Chang [tom0935802018@gmail.com], Chuan-Ming Tseng, Ming Chi University of Technology, Taiwan**

In this study, porous MgO-Mg₂SiO₄ bioceramic composite coatings were successfully fabricated on AZ31B magnesium alloy by using plasma electrolytic oxidation (PEO) under a bipolar pulsed power mode in alkaline sodium phosphate (Na₃PO₄×12H₂O), sodium silicate (Na₂SiO₃×5H₂O) and potassium titanium fluoride (K₂TiF₆) solutions. The MgO-Mg₂SiO₄ composite coatings exhibited higher hardness and better adhesion, serving as stable scaffolds for subsequent functionalization. In order to enhance antibacterial efficiency, silver nanoparticles (Ag NPs) deposited onto PEO coatings were employed via thermal evaporation deposition with various deposition durations (5~15 min). The effect of silver particle size on the antibacterial behaviors of Ag NPs deposited PEO coatings was also investigated in this study. The microstructural characteristics of Ag NPs deposited PEO coatings using XRD, SEM-EDS and EPMA show that the particle size of Ag NPs is increased parabolically from ~56 nm to ~220 nm as increasing deposition duration from 5 min to 15min, respectively. However, the prolonged evaporation gives rise to the aggregation of Ag NPs and the formation of larger island-like structures on PEO coatings. The antibacterial properties of Ag NPs deposited PEO coatings were carried out by measuring the numbers of *Escherichia coli* bacterial colony after various incubation durations (30~90 hr). The experimental results show that the antibacterial effect of the Ag NPs deposited PEO coatings is significantly improved with decreasing particle size of Ag NPs on PEO coating. Moreover, the Ag NPs deposited PEO coating after deposition duration with 5 min exhibits a 100% antibacterial efficiency to *Escherichia coli* after incubation in 90 min. The PEO coating with smaller size of Ag NPs (~56 nm) deposited exhibits a significantly larger specific surface area, and 100% antibacterial efficiency after incubation in 90 min can be achieved due to the facilitated rapid silver ion release.

Keywords: Plasma electrolytic oxidation (PEO), Thermal evaporation deposition, AZ31B magnesium alloy, Silver nanoparticles (Ag NPs), Antibacterial behavior.

10:20am **MD2-2-ThM-8 Dislocation-Mediated Plasticity and Strain Localization in Transition Metal Nitrides: Insights from Micropillar Compression, Rainer Hahn [rainer.hahn@tuwien.ac.at], CDL-SEC, TU Wien, Austria; Peter Polcik, Szilard Kolozsvari, Plansee Composite Materials GmbH, Germany; Klaus Boebel, Oerlikon Surface Solutions AG, Liechtenstein; Helmut Riedl, CDL-SEC, TU Wien, Austria** **INVITED**
Transition metal nitrides are key materials in advanced protective and functional coatings, yet their mechanical response is often constrained by

intrinsic brittleness. Recent studies show that defect engineering and electronic structure control can fundamentally alter this behavior, enabling metallic-like plasticity in selected compounds. In this work, we employ in-situ micropillar compression to investigate the deformation mechanisms of epitaxial TiN, CrN, and WN thin films. Despite their structural similarity, these nitrides exhibit strikingly different responses under load. TiN and CrN deform through slip-band formation and early strain localization, indicating limited dislocation mobility and a strong tendency toward brittle failure. In contrast, WN displays pronounced metal-like plasticity, sustaining large plastic strains through dislocation glide and interaction processes more typical of metallic systems. These findings demonstrate how compositional tuning and bonding character influence the transition from brittle to ductile behavior in refractory nitrides. The results establish in-situ micropillar compression as a powerful tool to uncover intrinsic deformation pathways and identify WN as a key model system bridging metallic and ceramic mechanical responses.

11:00am **MD2-2-ThM-10 Biofunctional Zinc Phosphate-Loaded Membranes as a Potential Anti-Biofilm and Remineralizing Approach for Caries Management, Gina Prado-Prone [ggradoprone@comunidad.unam.mx], Lorena Reyes-Carmona, Lizeth A. González-Vargas, Laboratorio de Biointerfases, DEPEL, Facultad de Odontología, Universidad Nacional Autónoma de México; Phaedra S. Silva-Bermudez, Unidad de Ingeniería de Tejidos, Terapia Celular y Medicina Regenerativa; Instituto Nacional de Rehabilitación Luis Guillermo Ibarra Ibarra, Mexico; Sandra E. Rodil, Instituto de Investigaciones en Materiales, Universidad Nacional Autónoma de México; Nicola Cioffi, Dipartimento di Chimica, Università degli Studi di Bari Aldo Moro, Italy; Camila A. Zamperini Zamperini, Department of Restorative Dentistry, College of Dentistry, University of Illinois Chicago, USA; Argelia Almaguer-Flores, Laboratorio de Biointerfases, DEPEL, Facultad de Odontología, Universidad Nacional Autónoma de México**

Introduction: Dental caries is the most common oral disease, mainly caused by persistent cariogenic biofilm on dental surfaces. The acidic environment created by acid-producing and acid-tolerant bacteria leads to dental demineralization and, ultimately, caries formation. The most important strategies in cariology currently focus on preventing caries and treating early-stage lesions by inhibiting biofilm formation and optimizing tooth health remineralization. Therefore, developing novel antibiofilm and remineralizing anti-caries biomaterials has great potential to enhance caries prevention and treatment.

Objectives: To develop zinc phosphate (ZnP)-loaded membranes and evaluate their capacity to prevent dental caries-related biofilms and promote dental remineralization.

Methods: ZnP microparticles were synthesized via chemical precipitation. Membranes were synthesized via electrospinning a polycaprolactone-gelatin blend (1:1) incorporating ZnP microparticles at 1%, 2%, and 5% (w/w) concentrations. The micro-morphology, chemical composition, wettability, and thermal properties were analyzed using SEM, EDS, FTIR, WCA, TGA, and DSC. The *in vitro* anti-biofilm effect was evaluated by turbidity and Alamar Blue assays using four bacteria associated with caries: *Streptococcus mutans* (25175™ATCC®), *Streptococcus sanguinis* (110556™ATCC®), *Lactobacillus acidophilus* (4356™ATCC®), and *Veillonella parvula* (17745™ATCC®). To assess the remineralization potential of the experimental membranes in *in vitro* root caries lesions, human root dentin specimens were slightly demineralized and exposed to the membranes under remineralization cycling. The mineral density, depth, and porosity of the final root caries lesions were assessed by computer microtomography and confocal scanning laser microscopy after rhodamine infiltration.

Results: Membranes exhibited a microfibrillar structure with interconnected porosity and desirable physico-chemical properties for clinical applications. Antibacterial testing showed 44-80% inhibition of biofilm formation of the four bacterial strains on the ZnP-membrane surfaces; the antibiofilm effect appears to depend on the ZnP concentration. The 5% ZnP-loaded membranes exhibited a more favorable pattern of remineralization for *in vitro* root caries treatment, but there were no statistically significant differences in caries depth and porosity among groups (p≥0.05).

Conclusions: ZnP-loaded membranes can potentially be used as an anti-biofilm and remineralizing approach for dental caries management.

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11:20am **MD2-2-ThM-11 Growth Mechanism and Cellular Response to Film Thickness Variations of Nanoporous Alkaline Titanate-Converted, Magnetron Sputtered Ti Thin Films**, **Matthew Wadge** [m.wadge@mmu.ac.uk], Manchester Metropolitan University, UK; *Kozim Midkhatov*, University of Manchester, UK; *Jonathan Wilson*, *Louise Briggs*, *Timothy Cooper*, *Zakhar Kudrynskiy*, University of Nottingham, UK; *Reda Felfel*, University of Strathclyde, UK; *Ifty Ahmed*, *Colin Scotchford*, *David Grant*, University of Nottingham, UK; *Justyna Kulczyk-Malecka*, Manchester Metropolitan University, UK; *Mahetab Amer*, University of Manchester, UK; *Peter Kelly*, Manchester Metropolitan University, UK

The standard process for improving bioactivity of implant surfaces for natural fixation is reliant on high temperature (>1500 K) plasma spraying of hydroxyapatite (HA) [1]. However, these surfaces have been shown to spall due to their brittle nature, high internal stresses, and weak mechanical adhesion [1]. Bioactive titanate surfaces have been developed as a low-temperature, more simplistic alternative, however, their applicability is limited to titanium (Ti) and its alloys only via chemical conversion routes [1]. The present authors previously demonstrated the applicability of titanate surfaces generated from PVD Ti coatings [2], however, assessment of thickness variation on cellular performance is still required, due to potential unwanted effects such as poor cellular proliferation. This paper highlights for the first time the cellular performance of titanate films generated on various thicknesses of Ti coating. By varying the thickness of the PVD deposited Ti coating, one can influence the formation mechanism of the wet-chemically derived titanate surface produced, since the mechanism is diffusion dependant and material limited.

Magnetron sputtering was employed to generate the Ti coatings (ca. 50, 100, 200, 500 nm) owing to its excellent step coverage, relatively quick deposition rate, ability to coat onto, and from, a wide variety of materials. In the conversion process, Ti coatings are treated in NaOH (5 M; 60 °C; 24 h) to generate sodium titanate structures [1]. The resultant materials were characterised using SEM, EDX, XPS, XRD, as well as cellular assessments, in order to understand the formation mechanism, the resultant morphological (Fig.1&2), structural (Fig.3) and chemical (Fig.4) properties, as well as influence on cellular response. It was clear that the Ti coatings exhibited good step coverage. Following titanate formation, only the 200 and 500 nm coatings produced the characteristic nanoporous 'webbed' titanate structures, due to the lack of free Ti in the coating, as opposed to the conventional diffusion limitation (Na and O) of the titanate mechanism. Both XPS and XRD analyses confirmed the formation of titanate on all of the coatings tested, despite the morphological differences and irrespective of thickness. Through utilising sputtering, the applicability of these titanate materials in a biomedical context can be significantly improved due to its ability to coat most materials and matching the subsequent wet-chemical temperature conditions.

References

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- [2] M. Wadge, et al., *Journal of Colloid and Interface Science*, 566, 271-283, 2020

12:00pm **MD2-2-ThM-13 Low-Pressure Plasma Processes for the Deposition of Adherent Diamond-Like Carbon Coatings on Titanium Alloys for Biomedical Applications**, *Chloé Audet*, **Pascale Chevallier** [pascale.chevallier@crchudequebec.ulaval.ca], Laboratory for Biomaterials and Bioengineering, (CRC-Tier I), Dept Min-Met-Materials Eng., & Regenerative Medicine, CHU de Quebec, Laval University, Canada; *Sandra Rubio*, Laboratoire Interdisciplinaire de Spectroscopie Electronique, Namur Institute of Structured Matter, University of Namur, Belgium; *Andranik Sarkissian*, Plasmionique Inc, Canada; *Laurent Houssiau*, Laboratoire Interdisciplinaire de Spectroscopie Electronique, Namur Institute of Structured Matter, University of Namur, Belgium; *Diego Mantovani*, Laboratory for Biomaterials and Bioengineering, (CRC-Tier I), Dept Min-Met-Materials Eng., & Regenerative Medicine, CHU de Quebec, Laval University, Canada

Titanium and its alloys are widely recognized as the gold standard for bone contact implants due to their suitable mechanical properties and biological performances. However, their long-term clinical performance remains impaired, mainly due to insufficient integration with surrounding tissues and the risk of infection. In order to enhance their performance, surface modification through coatings are explored. Among these coatings, diamond-like carbon (DLC) has emerged as a promising material due to its superior mechanical properties, chemical inertness, and stability, as well as its ability to integrate antibacterial agents such as Ag, ZnO, etc., resulting in a multifunctional coating. However, due to the high intrinsic stresses of DLC

compared to the native oxide layer, the adhesion of DLC to metallic surfaces remains rather low. Therefore, this work focuses on improving DLC adhesion to the Ti alloy surface using plasma-assisted chemical vapor deposition (PECVD), by optimizing different surface pretreatments prior to coating. The results showed that the nature and duration of the pretreatment significantly influenced the chemical composition and topography of the substrate prior to deposition, which in turn had an impact on the thickness, structure and morphology of the DLC. Short methane carburizing, particularly for 10 min, appeared to be the most effective pretreatment, as it removed the native oxide layer, enhanced carbon implantation, led to thick, adherent DLC coatings with a diamond-like structure, and stable even after 7 days of aging in pseudo-physiological conditions. In contrast, argon etching alone was ineffective, and combining both treatments yielded thinner films.

Controlled plasma carburization prior to DLC deposition significantly improves coating adhesion and stability on Ti alloys, paving the way for improved implant performance. The addition of antibacterial agents within the DLC matrix could further improve clinical outcomes of implants and reduce implant-associated infections.

Keywords: Surface modification, plasma-enhanced chemical vapor deposition, diamond-like carbon, Ti-alloy medical implant.

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