

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

Surface Coatings and Surface Modifications in Biological Environments I

Moderators: **Mathew T. Mathew**, University of Illinois College of Medicine at Rockford and Rush University Medical Center, USA, **Phaedra Silva-Bermudez**, Instituto Nacional de Rehabilitación Luis Guillermo Ibarra Ibarra, Mexico, **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. Cogley**, 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 ($\text{Ag}_{75}\text{Cu}_{25}$ - $\text{Ag}_{50}\text{Cu}_{50}$ - $\text{Ag}_{25}\text{Cu}_{75}$) 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 $\text{Ag}_{75}\text{Cu}_{25}$ 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 Eneider Peña 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/cm², 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 I_{corr} 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 nanostructures. The use of seeds alters the distribution of the features, and hexagonally packed nanostructure 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

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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.

Coatings for Biomedical and Healthcare Applications Room Pacific F-G - Session D1-2-MoA

Surface Coatings and Surface Modifications in Biological Environments II

Moderators: **Mathew T. Mathew**, University of Illinois College of Medicine at Rockford and Rush University Medical Center, USA, **Phaedra Silva-Bermudez**, Instituto Nacional de Rehabilitación Luis Guillermo Ibarra Ibarra, Mexico, **Kerstin Thorwarth**, Empa, Swiss Federal Laboratories for Materials Science and Technology, Switzerland

1:40pm D1-2-MoA-1 Antibacterial Performance of DLC and Ag-doped DLC coatings with a Long Term Perspective, Maneesha Rupakula, Platit AG, Switzerland; *K. Sharma*, EPFL, Switzerland; *H. Bolvardi*, *B. Paul*, *G. Wahli*, Platit AG, Switzerland

DLC display properties such as high mechanical hardness, low coefficient of friction, high chemical inertness, and good biocompatibility. Due to their amorphous structure, doping with certain elements enhances functionality still preserving structural integrity. Ag doped DLC films form an interesting class of biomaterials being bactericidal along with other favourable physical properties. Where sterile or ultra-clean conditions need to be ensured, surfaces of components, instruments, parts of tactile and environmental surfaces can benefit from such hard biomaterials. For industrial relevance, 1) anti-bacterial response to both clinically relevant gram-positive and gram-negative bacterial strains and 2) antibacterial performance over longer-term needs to be explicitly studied and not inferred.

In this work, we addressed both antibacterial behaviour and its long-term effect over months. DLC films were prepared in a hybrid vapordeposition process to obtain Ag concentrations in DLC ranging 0 to 8.5%. DLC (No Ag) films display antifouling behaviour over bare substrate with antibacterial efficiency up to 30% for both gram-positive (*S. aureus*) and gram-negative (*E. coli*) bacterial strains. Hence, DLC (No Ag) films are considered intrinsically favourable as antibacterial surfaces. This passive antibacterial effect can vary widely when tuning mechanical film strength with change in carbon content. To obtain a constant efficiency with time, an active bactericidal effect with Ag doped DLC is necessary. At an optimal Ag-concentration of 6.9 % in DLC over DLC (No Ag), very high efficiencies of up to 99.6% were obtained for both types of bacterial strains. To motivate commercial implications of Ag-DLC the long-term antibacterial performance was also investigated. In aged samples (post de-oxidising treatment), the antibacterial efficiency was recovered up to 95% of its value pre-aging, encouraging an in-depth long-term study.

2:00pm D1-2-MoA-2 TiN/NbN Superlattice Coatings Deposited by High Power Impulse Magnetron Sputtering Potential Candidate to Protect Medical Grade CoCrMo Alloys, Papken Hovsepian, Sheffield Hallam University, UK; *A. Ehasarian*, *A. Sugumaran*, Sheffield Hallam University, United Kingdom; *I. Khan*, Zimmer- Biomet UK

In recent years significant progress has been made in the application of various ceramic, namely MeN functional coatings to engineer the surfaces of medical implants utilising metal-on-metal (MoM) articulation. TiN/NbN coatings were deposited on medical grade CoCrMo alloy by High Power Impulse Magnetron Sputtering, (HIPIMS). X-ray analysis revealed that the coatings exhibit single phase fcc crystal structure and (200) texture. LA diffraction analyses revealed the superlattice coating structure with bilayer thickness of 3.0 nm further confirmed by TEM imaging.

In dry sliding pin on disc tests TiN/NbN coatings exhibited friction coefficient of $\mu = 0.7$ and wear coefficient of $K_c = 1.4 \times 10^{-14} \text{ m}^3 \text{ N}^{-1} \text{ m}^{-1}$ which were significantly lower as compared to the bare alloy.

Coating impact load fatigue resistance was studied by applying 500 N load for 1 million impacts using CemeCon impact tester which revealed coating high durability.

In the case of TiN/NbN coating deposited on CoCrMo substrate where $E_{\text{coating}} / E_{\text{substrate}}$ is as high as 1.81 indicating that the substrate does not provide the necessary load bearing support for the brittle thin film, the utilisation of the Berkovich indentation technique proved to be a potent approach to study coating material as well as structural response to applied concentrated load.

FIB/SEM analyses of the indented coatings revealed that in the hard-on-soft material systems cracks will initiate due to sub-coating substrate deformation and then propagate towards the coating surface. The FIB/SEM and low magnification XTEM analysis showed that an exceptionally strong

TiN/NbN coating substrate adhesion bonding was achieved due to the utilisation of the HIPIMS pre-treatment.

High resolution XTEM analyses revealed first time that during the indentation a collective rotation and alignment of the individual layers of the superlattice stuck takes place without compromising coatings integrity which is clear evidence for the exceptionally high coating toughness.

In potentiodynamic polarization tests in 3% NaCl and Hank's solution TiN/NbN showed several orders of magnitude lower corrosion current and higher pitting potential compared to CoCrMo which guarantees excellent protection.

Inductively Coupled Plasma Mass Spectrometry analyses of TiN/NbN coated samples corroded in Hank's solution revealed that the leaching of harmful metal ions from CoCrMo was reduced to below the detection limit.

The high toughness of the superlattice structured TiN/NbN coatings combined with their exceptionally high adhesion on medical grade CoCrMo and reliable barrier properties ranks them as a strong candidate for medical implant applications.

2:20pm D1-2-MoA-3 3D Printed Ceramics Reinforced Ti6Al4V: Structural and Nano-Mechanical Characterization, Peter Apata Olubambi, T. Tshphe, University of Johannesburg, South Africa **INVITED**

Compositional formulations and improved materials processing methodologies are innovative approaches for confronting the challenges being faced by materials and biomedical engineers in designing and producing biocompatible components and implants with longer lifetimes having enhanced wear and corrosion resistances. In this study, the structural properties of 3D additively manufactured Ti6Al4V containing ZrO₂ printed through direct metal laser sintering technique at varying process parameters were investigated and reported. Results on the relationships between the micro-scale and nano-scale mechanical properties as well as the biocorrosion behaviours of the biocompatible composites in some selected simulated human systems are presented.

3:00pm D1-2-MoA-5 A Remote Atmospheric Pressure Plasma-Assisted Textile Functionalization Process on Polymeric Scaffolds for Bone Tissue Engineering, Wei-Yu Chen, J. Lee, T. An, Taiwan Textile Research Institute, Taiwan; *A. Matthews*, University of Manchester, UK

The porous textile structure of nonwoven polylactic acid (PLA) possesses the potential of being applied to tissue engineering scaffolds. However, when adopting biodegradable polymers, including PLA, for bone tissue engineering scaffolds, the inert and hydrophobic surface properties of these materials not only lead to poor biocompatibility and osteoconductivity but also limit the efficiency of surface modification treatments, which include hydroxyapatite (HAp) deposition via alternate soaking processes. To develop a rapid, environmentally friendly and polymeric textile-suitable process to tackle these issues, a remote atmospheric pressure plasma (APP) system using a bespoke Pyrex chamber and acrylic acid monomer was utilised to deposit carboxylic acid functional groups onto the PLA surface, which are beneficial for performing the following alternate soaking process for HAp deposition. Being compared with the neat PLA, the plasma functionalized PLA exhibited a 16% improvement in hydrophilicity and showed better biocompatibility after HAp deposition. The stability of hydrophilicity and surface elemental composition of the APP-functionalized surface are also reported and the HAp-deposited PLA was examined by a scanning electron microscopy and energy dispersive X-ray analysis.

3:20pm D1-2-MoA-6 Development of Modified Hydroxyapatite Composite Coating Prepared by the Thermal Spray, Jo-Han Yu, National Taipei University of Technology, Taipei Tech, Taiwan; *K. Feng*, Ming Chi University of Technology, Taiwan; *Y. Yang*, National Taipei University of Technology, Taipei Tech, Taiwan

This study shows the modified hydroxyapatite composite coating formed under different spray conditions by Flame Spray (FS). Pure crystalline HA has a low dissolution rate, which slows down the bone integration. However, bioactive glasses are a family of special glasses which are able to bond to bones and, if the glass composition is properly designed, even to soft tissues. In this experiment, the hydroxyapatite and bioactive glass will used Flame Spray (FS) to coating on 304 stainless steel surface. This experiment will be divide into two different parts for discussion. In the first part will analysis the microstructure, phase composition, mechanical properties and bonding test of the modified hydroxyapatite composite coating. Based on the results, the acetylene flow rate of 1.60 Nm³/hr and the speed of spray gun is 250 mm/s, the spray times of Flame Spray (FS)

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using 2 times to conducting the experiments; The second part is the biocompatibility test of the modified hydroxyapatite composite coating, cell attachment morphology observation and human simulated body fluid immersion test.

3:40pm **D1-2-MoA-7 New Generation of Thin Films for Protection of Stainless Steel Against Corrosion and Bacterial Contamination, Akram ALHUSSEIN, A. BELGROUNE, E. KAADY**, University of Technology of Troyes, France; **L. AISSANI**, University of Khenchela, Algeria; **R. HABCHI**, Lebanese University, Lebanon; **S. Rtimi**, Federal Polytechnic School of Lausanne, Switzerland

Coatings present a great solution to protect a material and give it multifunctional properties. The objective of our research is the development of new generation of protective and multifunctional coatings. In our studies, the deposition rate is controlled to obtain a uniform film of 1-3 μm thickness. The influence of process parameters on the coating physicochemical properties (morphology, microstructure ...) and functional performance (corrosion resistance, biomedical properties...) is evaluated.

In this work, thin films were developed to protect stainless steel widely used in different severe environments in particular for maritime and biomedical applications. We present many strategies used for enhancing the coating efficiency based on the substrate used, the doped elements and coating architecture.

Acknowledgements: The authors would like to thank the European Union (FEDER - Fond Européen de Développement Régional), the GIP52 (Groupement d'Interet Public Haute-Marne), the PROFAS B+ Franco-Algerian program and the Doctoral School in Sciences and Technology at the Lebanese University (Réseau UT/INSAUL) for their financial supports.

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- [2] E. Kaady, R. Habchi, M. Bechelany, E. Zgheib, A. Alhussein, Effect of Al₂O₃, ZnO and TiO₂ Atomic Layer Deposition Grown Thin Films on the Electrochemical and Mechanical Properties of Sputtered Al-Zr Coating, *Coatings* 13 (1), 65, 2023.
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- [4] A. Belgroune, L. Aissani, A. Alhussein, M. Zaatat, J. Kiwi, S. Rtimi, Bacterial inactivation on sputtered TiO₂ and TiO₂-Ag thin films under solar simulated light, *Chemical Engineering Journal*, 141590, 2023.

4:00pm **D1-2-MoA-8 Effects of Silver Acetate Additives on Antimicrobial and Corrosion Behaviors of Plasma Electrolytic Oxidation Coatings on AZ31B Magnesium Alloy, Yu-Tse Sung**, Department of Materials Engineering, Ming Chi University of Technology, Taiwan; **C. Tseng**, Department of Materials Engineering & Center for Plasma and Thin Film Technologies, Ming Chi University of Technology, Taiwan

In this study, the bioceramic composite coatings on AZ31B magnesium alloy were prepared by using plasma electrolytic oxidation (PEO) in alkaline solutions with sodium phosphate, sodium silicate, potassium fluorotitanate and silver acetate additions. The effect of silver acetate content on antimicrobial and corrosion behaviors of PEO coatings on AZ31B magnesium alloy was investigated. The antimicrobial properties of PEO coatings were carried out by measuring the numbers of *Escherichia coli* bacterial colony after various incubation durations. The potentiodynamic polarization measurements were conducted to evaluate the corrosion behaviors of PEO coatings in simulated body fluid (SBF) solutions. The experimental results show that the antimicrobial effect of the PEO coatings is significantly improved with increasing silver acetate additive. More interestingly, the PEO coating with 0.2 g/L silver acetate addition exhibits a 100% antibacterial efficiency to *Escherichia coli* after incubation in 60 minutes. However, the potentiodynamic polarization curves display that the PEO coatings, as compared to AZ31 magnesium alloy, exhibit higher corrosion resistances in SBF solutions. Furthermore, the PEO coating with 0.15 g/L silver acetate addition shows the optimal corrosion resistance due to its lowest corrosion current density (i_{corr}), highest passivation breakdown potential (E_b) and largest polarization resistance value (R_p). In summary, the antimicrobial and corrosion behaviors of PEO coatings on AZ31B magnesium alloy can be pronouncedly improved by silver acetate additions.

Coatings for Biomedical and Healthcare Applications Room Pacific F-G - Session D2-TuM

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

Moderators: Hamdy Ibrahim, University of Tennessee at Chattanooga, USA, Margaret Stack, University of Strathclyde, UK

8:00am **D2-TuM-1 Empowering PVD-Coatings to Control the Time Dependent Chemical and Microstructural Coating Properties in Aqueous Electrolytes**, Holger Hoche, Center for Structural Materials, TU-Darmstadt, Germany; T. Ulrich, Center for Structural Materials, TU Darmstadt, Germany; P. Polcik, Plansee Composite Materials, Germany; M. Oechsner, Center for Structural Materials, TU-Darmstadt, Germany **INVITED**

Intelligent PVD coatings, which can change their chemical composition and their surface structure in aqueous electrolytes have a great potential for various medical applications. Therefore the coating material must be able to maintain the corrosion resistance and simultaneously release specific chemical species.

To achieve this, the authors developed a novel alloying concept for PVD coatings, where TiN-based coatings were alloyed with Mg and MgGd, respectively. Thereby, the corrosion protection capability of corrosive substrate materials, e.g. magnesium or mild steel, can be improved significantly [1]. Depending on the chemical composition of the coating, an adjustment of the corrosion resistance in NaCl electrolytes between 24h and 1000h is possible. Moreover, the developed alloying concept enables to get control of the time dependent chemical and structural properties in aqueous electrolytes: The chemical stability of the coating is determined the proportion of Mg and Gd in the entire TiN matrix: Under a corrosive load, the coating depletes of Mg and consequently, the chemical composition and the surface microstructure changes.

For the present study, TiN+Mg and TiN+MgGd coatings, respectively, are deposited using standard PVD magnetron sputtering technology. Therefore, TiMg and TiMgGd targets were produced using spark plasma sintering. The effect of the mentioned alloying concept on the time dependent chemical and structural changes during exposure of the specimens in NaCl electrolytes is comprehensively investigated. The underlying mechanisms and the resulting properties will be discussed.

[1] T. Ulrich, C. Pusch, H. Hoche, P. Polcik, M. Oechsner, Surface and Coatings Technology 422 (2021) 127496.

8:40am **D2-TuM-3 Early Detection of Fretting-Corrosion at the Hip Modular Junction Interface by Acoustic Emission Non-Invasive Technique**, Bill Keaty, Y. Sun, University of Illinois at Chicago, USA; M. Mathew, University of Illinois - Chicago, USA; D. Ozevin, J. Eapen, T. Zhang, University of Illinois at Chicago, USA

Total hip replacement (THR) is becoming an increasingly common surgical procedure globally, as it is an end-stage treatment for patients suffering from osteoarthritis or trauma¹. Early failure is a common occurrence in THRs due to tribocorrosion processes. As a result, a diagnostic method is necessary to catch early failure of THR before irreversible damage occurs. It was reported that the possible feasibility of acoustic emission (AE) technique can detect material degradation at the THR head-cup interfaces². However, no investigation has been done at the modular junction interface, where fretting-corrosion occurs. It was hypothesized that the acoustic emission data could be correlated to the varying level of THR damage at the modular junctions.

A series of fretting-corrosion experiments were conducted with a custom-made fretting rig capable of collecting simultaneous mechanical, electrochemical, and AE data. ZrO₂-Ti₆Al₄V couples were chosen to represent THR femoral head-neck interfaces for the fretting test. To investigate the efficacy of acoustic emission of detecting fretting-corrosion damages, the samples were subject to three different potentials to simulate (i) new implant: no corrosion at Ecathodic (ii) well function implant: normal corrosion at Eoc (iii) and damaged implant: accelerated corrosion at Eanodic. Bovine calf serum was selected as the electrolyte to simulate the synovial fluids. The standard electrochemical protocol was followed in the fretting under potentiostatic conditions.

The results showed that anodic potential conditions had the most significant increase in current during fretting of ±0.03 mA compared to Eoc conditions with a current increase of ±0.005 mA. This data agrees with the

expected trend that the samples exposed to accelerated Eanodic corrosion will have a significant current increase while fretting. Mechanical data was obtained, expressed in the form of force vs. displacement as a friction loop, with an energy ratio of <0.2 for the entire testing (3600 cycles), indicating the partial slip fretting region³. The trend in-situ AE data displayed a pulse-like acoustic emission fluctuation, indicating a strong correlation between the fretting-corrosion processes and acoustic emission results. This is an ongoing investigation; the study will focus on linking the AE data and THR fretting-corrosion damages associated with the mechanistic transitions. This could be extremely helpful in the early prediction of THR failure in orthopedic implant patients and clinical practices.

[1]J. Geringer et al., *Metals and Surface Engineering*, 2011 [2]C. Lee et al., *J of the Mech Beh of Bio Mat*, 2019 [3]Fouvry et al, *Wear*, 1997

9:00am **D2-TuM-4 Corrosion Evaluation of Plasma Electrolytic Oxidation Coatings on Titanium Alloys For Biomedical Implant Application**, E. Sondgeroth, K. Cheng, Y. Sun, UIC School of Medicine at Rockford, USA; C. Takoudies, UIC School of Medicine, USA; E. Vries, Faculty of Engineering Technology, University of Twente, The Netherlands, USA; D. Matthews, N. Bolink, Faculty of Engineering Technology, University of Twente, The Netherlands; A. Yerokhin, Department of Materials, University of Manchester, United Kingdom; Mathew Mathew, UIC school of medicine at Rockford, USA

Titanium is a widely used biomaterial in biomedical implants partly due to its good corrosion resistance which is chiefly attributed to the nano-scale passive layer on its surface(1). While the rates of corrosion for titanium implants currently seem to be low, the incorporation of mechanical wear in a corrosion environment such as the fretting-corrosion seen in a human femoroacetabular joint can result in devastating damage to titanium implants(2). Thus, coating technology may enhance the resistance to tribocorrosion damage. The objective of this study was to test the efficacy of Plasma Electrolytic Oxidation (PEO) coatings on titanium implants compared to bare titanium.

PEO was conducted under different conditions to produce six sample coating groups of varying electrolyte concentrations and thicknesses: NaAlO₂ + Na₃PO₄, NaAlO₂ + Na₃PO₄ (18 um thickness), NaAlO₂ + Na₃PO₄ + Al₂O₃, NaAlO₂ + Na₃PO₄ + PTFE NP₅, NaAlO₂ + Na₃PO₄ + PTFE NP₅ (thick), and NaAlO₂ + Na₃PO₄ + Al. The samples were washed for 10 mins each in isopropanol and DI water. Corrosion testing was conducted in Bovine calf serum 30 g/L (pH 7.4) to simulate synovial fluid and CP and EIS were collected. CP data was graphed as Potential versus Current Density and interpolated to find I_{corr} and E_{corr} for each test group. EIS data was plotted as Nyquist and Bode plots and fitted via a Rapid Electrochemical Assessment of Paint electrical circuit model to estimate each sample's corrosion resistance and coating capacitance.

The I_{corr} was 1.002 ±0.55µA/cm², 2.38±0.005 µA/cm², 0.88 ±0.48 µA/cm², 0.91±0.1 µA/cm², 0.41 ±0.31 µA/cm², and 0.069±0.0007 µA/cm² and E_{corr} was -0.39±0.17 V, -0.40±0.03 V, -0.36±0.15 V, -0.4±0.08 V, -0.47±0.03 V, -0.51±0.04 V, -0.53±0.02 V for each group, respectively. The data shows mild partial improvement in corrosion, especially corrosion potential, in the coated samples as compared to bare Ti. Overall, the results indicate that the coating assists in improving corrosion resistance. EIS data analysis also partially agrees with the improved corrosion resistance. It is worth noting that, the chemistry of the surface and structure of the coating will be influencing the parameters of the corrosion resistance. Particularly in the current study, the coatings displayed a severe porous nature, which may adversely affect the corrosion and wear properties. The hypothesis is partially validated, as the coating enhanced the corrosion resistance of the implant surface. The studies will continue to evaluate the surface properties and underlying mechanisms.

9:20am **D2-TuM-5 Large-Scale Metallic Nanotubes Array (MeNTA) with Plasmonic Nanoparticles for SERS Application**, Alfreda Krisna Altama, J. Chu, National Taiwan University of Science and Technology, Taiwan; P. Yiu, Ming Chi University of Technology, Taiwan; W. Chiang, National Taiwan University of Science and Technology, Taiwan

The limitations in reproducing large-scale and highly-sensitive surface-enhanced Raman scattering (SERS) substrates are a major problem for practical applications. In this work, we report on our works about a periodic three-dimensional (3D) metallic nanostructure array, referred to as MeNTA fabricated using PVD processes developed for large-scale semiconductor engineering. Specifically, the MeNTAs were fabricated using metallic glass alloy, providing high strength, ductility, and good biocompatibility. For further development, over the MeNTAs deposited a uniform coating of gold nanoparticles (NPs) to form a high-sensitivity

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AuNP@MeNTAs 3D-SERS substrate. Using crystal violet (CV) as a probe molecule, the performance of an AuNP@MeNTA 3D-SERS substrate can be provided. Here we found a very low detection limit with a comparable enhancement factor. The SERS performance did not degrade for more than a week after installation, thereby demonstrating high stability and repeatability. This research provides useful guidelines for the low-cost fabrication of SERS substrates of high sensitivity.

9:40am **D2-TuM-6 Carbide-derived Carbon (CDC) for Implant Application: Tribocorrosion Kinetics and Mechanisms, Kyle Kinnerk**, Department of Biomedical Engineering, University of Illinois at Chicago, USA; *Y. Sun, M. Daly*, Department of Civil, materials, and Environmental Engineering, University of Illinois at Chicago, USA; *M. Wimmer*, Department of Orthopedic Surgery, Rush University Medical Center, USA; *M. McNallan*, Department of Civil, materials, and Environmental Engineering, University of Illinois at Chicago, USA; *M. Mathew*, Department of Biomedical Sciences, UIC College of Medicine at Rockford, USA

Between 2000 and 2014, the estimated annual incidence of primary total hip replacement per 100,000 in the U.S. in the 55-70 year age range increased by 194.3% [1]. Previously, we fabricated carbide-derived carbon (CDC) on Ti6Al4V, and our finding indicated that CDC can provide superior protection to the substrate in a tribocorrosive environment. The objective of this work is to test CDC under three different electrochemical conditions, cyclic polarization (CP), OCP, and potentiostatic (PS), with the surface characterized by SEM-EDS and 3D profiler.

CDC was prepared on a Ti6Al4V substrate via the electrolysis approach[2]. Ti6Al4V was polished until the surface had a mirror-like finish ($R_a < 25$ nm). Alumina was used as the pin to simulate a ceramic counter body, and bovine calf serum (BCS) was selected to emulate biological fluids. Two groups of samples were tested, alumina (Al_2O_3)-Ti6Al4V (control group) and alumina-CDC, using a custom tribometer with reciprocal sliding for 3600 cycles. A standard electrochemical protocol was followed, and CP, OCP, PS were used for the sliding stages. Electrochemical impedance spectroscopy (EIS) was conducted before and after the testing stage to analyze the local corrosion kinetics. SEM/EDS was used to observe the wear scar and obtain the surface composition, 3D profiler will be used to measure the wear volume, and ICPMS will be used to measure the metal ions concentration released into the BCS solution.

Based on the potentiodynamic results under sliding conditions, the CDC coated sample showed approximately 126-fold smaller carbon density variation (6.301×10^{-6} A/cm²) than the bare titanium alloy (7.577×10^{-4} A/cm²), implying that CDC has a smaller corrosion weight loss (K_c). The OCP results agree with the potentiodynamic results, where the potential drop of CDC (0.194V) is smaller than Ti6Al4V (0.7V), meaning that CDC can provide protection to the substrate material under tribocorrosive environment. Furthermore, SEM-EDS results indicate that CDC still remained on the surface after tribocorrosion testing for both electrochemical conditions. Therefore, it is possible that CDC can be smeared over the surface by mechanical motion and act as a solid lubricant at the interface. Potentiostatic condition will be included to fully evaluate CDC's tribocorrosion performance, and more characterization techniques will be employed to reveal the protection mechanism of CDC. In this work, we found that CDC can significantly improve the substrate's tribocorrosive resistance, showing that CDC is promising in further implant applications.

[1] M. Sloan et al., *J. Bone Jt. Surg.*, 2018 [2] Y. Sun et al., *Surf. Coat. Technol.*, 2021

10:00am **D2-TuM-7 PEKK as Biomaterials Under Fretting Corrosion Solicitations: May This Biopolymer Be Considered as New Hip Implant Component?, Jean Geringer**, *J. Monnatte*, Mines Saint-Etienne, France; *G. Planche*, EPIC sarl, France; *J. Porteus*, Oxford Polymers, USA

Some materials dedicated to orthopaedic implants are well used from more than 40 years. About hip implants some PAEK, poly Aryl Ether Ketone, materials may have a right impact on the lifetime. PEEK, poly Ether Ether Ketone actually is under investigations. However PEKK, poly Ether Ketone, needs some improvements concerning Friction/Fretting corrosion resistance. From the fundamental point of view, PEKK was under investigations through this study about fretting corrosion investigations. Some fundamental investigations under these tribological conditions are not quite well described. This work aims at providing some fundamental results on tribology/tribocorrosion.

A typical fretting corrosion machine, Fig 1, allowed to manage 16 hours of experiments. Titanium alloy, Ti-6Al-4V, was the counter part in bovine serum. The displacement amplitude was sinusoidal with the amplitude of ± 40 μ m.

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This study aimed at establishing the fretting map and at drawing thanks to OCP, Open Circuit Potential, evolution the A ratio (Dissipated energy over the total energy) vs. OCP drop. This approach is well improved and the suggested ranking of contact was well achieved in accordance with previous results [1].

Under hip implants mechanical constraints PEKK material showed good performance under fretting corrosion solicitations compared to UHMWPE for example. Further investigations need to be performed in order to assess the capability of using as implants.

Figure 1

10:20am **D2-TuM-8 Fretting-corrosion (<5 μ m) Performance of Carbide-derived Carbon (CDC) Surface Modification for Hip Implants, Yani Sun, M. Daly, M. McNallan**, Department of Civil, Materials and Environmental Engineering, University of Illinois at Chicago, USA; *M. Mathew*, Department of Biomedical Sciences, UIC College of Medicine at Rockford, USA

Fretting-corrosion at the taper junction is one of the main causes of early failure of total hip replacement (THR)¹. Previously, we have proved that carbide-derived carbon (CDC) can protect Ti6Al4V from tribocorrosive damages². However, the fretting-corrosion behavior of CDC still remains unknown. Also, experimental fretting setups used in the literature simulate motions of 50 μ m or higher³, which might not simulate micromotions at the taper junction that leads to partial-slip fretting. Therefore, we aim to develop a device simulating a motion < 5 μ m while simultaneously collecting electrochemical data, and test CDC's fretting-corrosion behavior.

The fretting-corrosion apparatus mainly consists of a stepper motor, a force sensor, and a corrosion cell connected to a potentiostat. Two groups were designed as (i) ZrO₂ pin on the Ti6Al4V as the control group and (ii) ZrO₂ pin on the CDC. Bovine calf serum (BCS) with protein content of 30 g/L was selected as the electrolyte at $37 \pm 1^\circ$ C. A normal load of 83 N was applied, and the pin was controlled to move with an amplitude of 2 μ m at 1 Hz for 3600 cycles. A typical protocol was followed to monitor the evolution of open-circuit potential (OCP). After the fretting-corrosion testing, the exposed area was characterized by SEM/EDS.

As a result, friction loops of both groups show a narrow elliptical shape, and the energy ratios of both groups remained below 0.2 throughout the entire stage, indicating a fretting regime of partial slip according to Fouvry et al.⁴. Moreover, CDC possesses higher OCP (-0.076 V) than Ti6Al4V (-0.541 V) during the fretting stage, suggesting that CDC has a higher resistance to corrosion. The fluctuations of OCP caused by fretting motions of CDC is approximately 0.005 mV, which is smaller than that of Ti6Al4V (0.03 mV), suggesting that CDC can provide protection to the substrate under the fretting-corrosion conditions. Based on the SEM images, the main damage showing on the Ti6Al4V disk is abrasive wear with typical ploughing features, which might be because of the higher hardness of ZrO₂ than the Ti alloy. Less damages were observed on the CDC surface and CDC still presents on the surface after the testing, implying that CDC might act as a solid lubricant and get smeared over the surface by the mechanical wear and thus protect the substrate from fretting-corrosion damages. Also, TEM will be utilized in the following study to reveal detailed mechanism and structural information.

[1] S. Yu et al. *J. Clin. Orthop. Trauma* 2020 [2] Sun, Y et al. *Surf. Coat. Technol.*, 2021. [3] J. Geringer et al., *Tribocorrosion Passive Met. Coat.*, 2011 [4] S. Fouvry et al., *Wear*, 1995

10:40am **D2-TuM-9 Roll-to-Roll Sputtering and Laser Patterning for Mass-Produced 2D Electronic Biosensors, Christopher Muratore**, University of Dayton, USA; *N. Glavin*, Air Force Research Laboratory, USA

Materials with high surface-to-volume ratios demonstrate exquisite sensitivity and detection limits in diverse molecular sensing applications. Integration of nanowires, nanotubes, and two-dimensional (2D) semiconductors into sensing devices, however, presents challenges inhibiting product development. For example, thousands of trials are required to obtain government approval for point of care diagnostics, yet producing a suitable number of 2D devices via conventional synthesis and fabrication techniques to meet this is not feasible. To realize applications of 2D transducers in ubiquitous low-cost diagnostic devices, new fabrication approaches are required. Processes for high-rate ($> 10^6$ per day) mass-production of low-cost two-dimensional electronic medical diagnostic devices with limits of detection rivaling PCR will be presented. Rapid and inexpensive sensor chip fabrication relies upon laser patterning and crystallization processes in a roll-to-roll physical vapor deposition system. In addition to electronic properties responsive to molecular attachment, the composition of 2D MoS₂ with tunable sulfur vacancy density facilitates

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direct functionalization for highly selective analyte binding with antibody fragments or even smaller molecules for enhanced sensitivity. A large supply of unfunctionalized diagnostic devices may therefore be stored to be functionalized with any selective binding agent on-demand to immediately reduce the impact of future disease outbreaks. Materials for large-scale device fabrication were selected to ensure recyclability of the devices for reduced waste stream impact in anticipation of large numbers of devices are consumed daily. A combination of fundamental studies to highlight the role of defects in 2D MoS₂ on the sensitivity and dynamic range of the 2D devices is shown via extensive testing of thousands of mass-produced device measurements of biologically significant concentrations as low as 10 fg/mL of viral proteins and/or other biomarkers as low 10 fg/mL in the presence of interference protein concentrations >10¹² times higher than those of the target analytes. Response times for most analytes are 2 minutes or less. Detection of multiple analytes on an array of sensors fabricated on a single flexible chip is straightforward as the laser patterning steps dictating device architecture are adjustable by directing the automated patterning laser with a modified electronic drawing possessing the desired features. Integration of other processes to facilitate production exceeding a million devices daily, such as *in situ* device quality control via high throughput Raman spectroscopy and chip singulation tools is shown for high-yield, low-cost production of devices performance meeting stringent requirements for quantitative analysis of biological samples.

Coatings for Biomedical and Healthcare Applications Room Pacific D - Session D3-TuA

Biointerfaces: Coatings to Promote Cell Adhesion while Inhibiting Microbial Growth

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

1:40pm D3-TuA-1 Chemical Vapor Deposition of Tantalum for Enhanced Cell Adhesion, Jessica DeBerardinis, Ultramet, USA **INVITED**

Tantalum is a refractory metal with demonstrated strength, ductility, resistance to corrosion and oxidation, and biocompatibility. It has long been used as a biomaterial for medical devices such as pacemaker electrodes, contrast media and markers, and cell growth matrices, and for decades Ultramet's open-cell tantalum foam has been used in orthopedic implants. The use of chemical vapor deposition (CVD) has enabled these biomedical applications of tantalum. Biocompatibility research has demonstrated the following:

1. CVD tantalum coatings can enhance the binding of collagen and proteoglycans because of their surface roughness and tension [1,2].
2. CVD tantalum has antioxidant properties, which limit the negative effects of reactive oxygen species on osteogenic differentiation [1,3].
3. CVD tantalum can up-regulate integrins, which are transmembrane adhesive proteins that enhance cell adhesion and reduce apoptosis [1,4].
4. In the presence of CVD tantalum, cells demonstrate enhanced proliferation and autophagy, promoting cell survival in the presence of an implant [1,5].
5. Porous tantalum increases cell proliferation and enhances osseointegration in comparison with porous titanium substrates [6].

CVD allows small amounts of metallic material, such as tantalum, to be evenly applied over three-dimensional structures. CVD is not line-of-sight limited, so it can be used to uniformly coat and infiltrate extremely complex three-dimensional structures, such as Ultramet's reticulated (porous) carbon foam (Figure 1). A thin film of CVD tantalum can be applied over less expensive and/or less biocompatible materials to create a biointerface that promotes cell adhesion. Furthermore, Ultramet can modify its CVD processing to deposit tantalum such that it forms a diffusion bond, which occurs at the atomic level, so the tantalum cannot peel or delaminate from the substrate (Figure 2). Ultramet's CVD processing can also be modified to grow a textured tantalum surface (Figure 3), which has been demonstrated to affect the adhesion of human bone marrow stromal osteoprogenitor cells. Examination of the microscale topography of textured tantalum showed increased cell spreading with significant growth of actin fibers, and metabolic activity was also notably higher than with the bare substrate and untextured tantalum.

Ongoing research of textured CVD tantalum coatings at Ultramet involves modifying the texture according to tissue type and incorporating other bioactive layers. Process development continues to improve the application of CVD tantalum coatings to various medical grade substrates to create more economical orthopedic and dental implants.

2:20pm D3-TuA-3 The Functionalization of N95 Masks Using Atomic Layer Deposited Silver Nano-Islands to Induce Antimicrobial Activity, Harshdeep Bhatia, C. Takoudis, University of Illinois, Chicago, USA

Due to the recent COVID-19 pandemic, the demand and use for antimicrobial textiles has increased. This demand saw a similar surge in these textiles during the SARS-COV-1 outbreak, resulting in many patents, after which the use of Silver as a potential material to give a material some antibacterial/antiviral properties was popularized. Additionally, the use of disposable N95 masks has become popularized as the safest and easily available barrier against these viral outbreaks. In this study, a novel strategy to deposit nano-islands of Silver on N95 masks using ALD is developed to give it some antibacterial properties. X-ray Photoelectron Spectroscopy (XPS) and X-ray adsorption fine structure (XAFS) were used to characterize the as-deposited silver nano-islands. The size and roughness of the nano-islands was calculating using atomic force microscopy (AFM).

Furthermore, inductively coupled plasma mass spectrometry (ICP-MS) was used to study the leaching of these Silver nano-islands in standard 1x phosphate buffer over predetermined times to simulate the effect of biological fluids such as saliva or mucus. A microbiological assay was also conducted to study the effect Ag- coated N95 had on *Staphylococcus Aureus*. The films grown at the two temperatures, 90 °C and 120 °C, were stable in ambient conditions. The deposited silver nano-islands were stable on the N95 filter media against washing. A comparison of the characteristics of the films grown at different temperatures has been made. The functionalization of materials using ALD of silver provides a repeatable method to impregnate textiles and induce antimicrobial activity which releases silver at a slow rate.

2:40pm D3-TuA-4 Cold Atmospheric Plasma Jets Generated from Flexible Sources, Carles Corbella, S. Portal, H. Solomon, M. McCraw, M. Keidar, S. Solares, George Washington University, USA **INVITED**

Atmospheric pressure plasma jets (APPJs) are excellent resources for a myriad of applications in energy and healthcare industries, being cancer therapy a major breakthrough. Their working principle is based on the generation of a rich plasma chemistry and photon emission in open air, which can gently modify surfaces of different materials at room temperature. APPJs originated from a flexible source are able to adapt to complex topologies and to treat delicate samples, like soft matter and organic tissues. Stable and reproducible operation of a flexible multi-jet array has been proven in sources set with planar (concave and convex modes) and radial (cylindrical source) configurations. Also, plasma plumes with different shapes have been achieved by conveniently modifying the nozzle geometry, thereby demonstrating the wide control over plasma performance. The participation of reactive oxygen and nitrogen species (RONS) in combination with energetic UV photons emitted by flexible APPJs as a function of the nozzle-sample distance are crucial aspects that need thorough characterization. Indeed, the action of these plasma species must be considered for the sake of APPJ safety in applications that require proximity of sensitive samples, especially in medical practices. Here, we demonstrate methods to estimate macroscopic parameters of relevance at the plasma-surface interaction region, namely local temperature, relative humidity (RH), pH variations, electric field (jet potential), and UV dose irradiation including shortest vacuum-UV wavelengths (VUV<100 nm). Portable platforms of flexible APPJ arrays exhibiting specific configurations and nozzle geometries will constitute the next generation of plasma devices aimed at biomedical applications.

4:00pm D3-TuA-8 Multifunctional Coating Approach Integrating Visible-Light Driven Photodynamic Therapy and Photocatalytic Activity for Controlling Biofilm Accumulation and Reinforcing Wear Protection, Bruna Nagay, C. Dini, R. Costa, A. Santos, University of Campinas (UNICAMP), Brazil; J. Cordeiro, Centro Universitário das Faculdades Associadas de Ensino, Brazil; B. Gomes, University of Campinas (UNICAMP), Brazil; E. Rangel, N. Cruz, Sao Paulo State University, Brazil; J. van den Beucken, Radboud University Medical Center, Netherlands; V. Barão, University of Campinas (UNICAMP), Brazil

The accumulation of biofilm and further establishment of infections on implant devices is one of the major concerns in the biomedical industry and clinical management. Among current therapies, although antimicrobial photodynamic therapy (aPDT) has been considered effective, inconsistencies regarding its outstanding performance in battling the burden of growing peri-implantitis prevalence have raised concerns towards the development of novel strategies. To overcome these drawbacks, we proposed an integrated coating strategy combining visible-light-driven photocatalytic activity and aPDT to simultaneously face titanium implant infections and optimize the implant biointerface for biomedical and dental implant applications. A multifunctional bismuth (Bi)-doped TiO₂ coating was synthesized upon titanium (Ti) substrate using plasma electrolytic oxidation (PEO). Polished Ti and pure TiO₂ coating were used as controls. PEO produced a crystalline, rough coating on Ti surface with superhydrophilicity features. The incorporation of Bi into the TiO₂ matrix was confirmed by X-ray photoelectron spectroscopy. UV-vis diffuse reflectance spectroscopy revealed that Bi effectively narrowed the band gap of TiO₂, making Bi-TiO₂ promising to exhibit photocatalytic activity under visible-light irradiation, which was confirmed by the methylene blue (MB) degradation assay. In addition, because the visible light used herein has a wavelength compatible with the MB used in aPDT, we hypothesized that the combination of reactive oxygen species generated by the photocatalysis mechanism along with MB-mediated aPDT could potentiate

¹ Graduate Student Award Finalist

the eradication of microorganisms. As such, *in vitro* experiments using human saliva as inoculum revealed that Bi-TiO₂ potentiated the polymicrobial biofilm reduction mediated by aPDT, and that 1 min of light exposure had similar antimicrobial effects when compared to 5 min. Furthermore, the Bi-TiO₂ coating was not cytotoxic to human bone mesenchymal stem cells and human gingival fibroblasts, even under light exposure. Finally, PEO coatings presented higher wear resistance, hardness, and albumin adsorption than control groups, indicating their outstanding properties to optimize the implant biointerface. The proposed proof-of-concept research holds great promise to face peri-implant infections by using a visible-light-driven photocatalytic coating and aPDT in a smart and safe manner to reduce biofilm while maintaining the properties that affect the longevity of biomedical devices (e.g., wear resistance, cell-material interactions). This opens a new perspective for the development of effective antimicrobial surfaces by the implant industry.

4:20pm **D3-TuA-9 ZnO_x Nanolayers as Antimicrobial Surfaces**, *L. Reyes-Carmona*, Universidad Nacional Autónoma de México; *O. Sepulveda-Robles*, Instituto Mexicano del Seguro Social, Mexico; *A. Almaguer-Flores*, *C. Ramos-Vilchis*, **Sandra E. Rodil**, Universidad Nacional Autónoma de México
The regular use of disinfectants is not an ecologically friendly solution to control the transmission of viruses and bacteria, and it could promote antibacterial resistance. Antimicrobial surfaces are a plausible solution applicable to rigid and flexible surfaces, as well as to protective equipment for healthcare personnel.

In this work, we investigated the antiviral and antimicrobial properties of sputtered ZnO_x nanolayers deposited on polypropylene (PP) fabrics for their use in respiratory protection equipment. Because of the fast traveling of respiratory drops containing viruses and bacteria, our study emphasizes the effect of contact time on the antimicrobial response. Since the substrate cannot be heated, the films were substoichiometric and amorphous.

Two methods were developed to test the antimicrobial response as a function of contact time. The shortest contact time was simulated using a bactericidal/virucidal filtration system where the material was exposed to an aerosol loaded with microorganisms (bacteria and surrogate viruses). Larger contact times between 0.5 and 24 hr. for the viruses and 24 hr. for the bacteria were tested by placing a drop containing the microorganisms on the surface for the specified contacting time. After such time, the virus was recovered, and the infectivity was evaluated by counting the plaque-forming units. Colony-forming unit determination was used for the bactericidal tests to evaluate the survival of aerobic and anaerobic bacteria.

Virus viability assays were used to study the survival of PaMx54, PaMx60, PaMx61 (ssRNA, Leviviridae), and PhiX174 (ssDNA, Microviridae) as surrogates for non-enveloped viruses. An approximate 40% PFUs reduction was obtained after 12 hr. for the RNA viruses, but only 12% was achieved for the DNA virus compared to the uncoated PP. For larger contacting times, the RNA viruses were completely reduced after 12 hr. in the ZnO-coated fabric, but no reduction was observed for the DNA virus or the uncoated PP.

For the aerosols containing the anaerobic bacteria, which are typically found in the oral environment, inhibition ratios between 53-96% were obtained, depending on the strain. Similarly, for the aerobic bacteria aerosols, the inhibition was between 26 and 90%, being slightly more resistant. However, after 24 hr of direct contact between the bacteria and the ZnO-coated surface, most strains were inhibited (80-90%).

These results suggest that ZnO_x nanolayers deposited by magnetron sputtering reduce the infectivity of non-enveloped RNA respiratory viruses and inhibit the growth of anaerobic and aerobic pathogen bacteria.

4:40pm **D3-TuA-10 Cytocompatibility of Chitosan-Silver Coated Titanium Coupons**, *E. Coleman Montgomery*, **J. Amber Jennings**, *M. Atwill*, *J. Bumgardner*, University of Memphis, USA

Introduction

Titanium is commonly used in orthopedics due to its strength, resistance to corrosion, and bone-like mechanical properties. Silver ions affect microbials by blocking transport in and out of the cell, inhibiting the production of energy, and interacting with DNA to prevent replication. These characteristics lead to broad spectrum antimicrobial properties against bacteria and fungi and therefore support the advantage of silver ions as an implant coating using chitosan biopolymer as a complexing agent and coating to localize silver.

Methods

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Treated Coupons: Titanium coupons were polished with 400, 600, 800, and 1200 grit sandpaper before being sonicated in soapy water, acetone, and ethanol to remove oil and residue for 10 minutes each. The coupons were then soaked in 5M NaOH for 24h to allow accumulation of hydroxide reactive groups on the titanium surface and rinsed with deionized (DI) water twice. The coupons were treated with a linking agent and dried for 10 minutes in a 110°C oven. Chitosan-silver solution (Chitozan Health) was added and left to dry overnight. The coated coupons were immersed in phosphate buffer for 1 hour, rinsed with DI water, and dried fully.

Untreated Coupons: Titanium coupons were polished with 400, 600, 800, and 1200 grit sandpaper before being sonicated in soapy water, acetone, and ethanol to remove oil and residue for 10 minutes each. The uncoated coupons were rinsed with DI water and dried fully.

Cytocompatibility: Coupons were UV-sterilized for 20 minutes and washed in cell medium. Saos-2 cells were seeded at 90,000 cells/well in a 12-well plate before exposure to 3 test groups: treated coupons, untreated coupons, and tissue culture plastic (TCP) control. After 24 hours, cell viability was determined using CellTiter-Glo Viability Assay (n=3), and cell morphology was determined using Live/Dead staining (n=1).

Results

The Saos-2 cell viability for treated coupons was not statistically different than untreated coupons and was about 70 percent of the TCP control. Live/Dead staining also produced similar results, with mostly living cells in all groups.

5:00pm **D3-TuA-11 Nonsurgical Decontamination Protocols for 3D-Printed Implant Surfaces**, **Valentim Barão**, *R. Costa*, *T. Takeda*, *C. Dini*, University of Campinas (UNICAMP), Brazil; *M. Bertolini*, University of Pittsburgh, USA; *M. Feres*, *J. Shibli*, *J. Souza*, Guarulhos University, Brazil

There is still a lack of a predictable nonsurgical protocol for effective dental implant decontamination, including for 3D-printed surfaces. Therefore, this study aimed to scrutinize the deleterious effects of different mechanical and chemical decontamination treatments on titanium (Ti) surface, electrochemical properties, biofilm cleaning potential, and cell behavior. 3D-printed Ti discs obtained by direct metal laser sintering were used. These samples were coated with polymicrobial biofilm from human saliva *in vitro*. Biofilm-covered surfaces were decontaminated with mechanical [Ti curette, plastic curette, Ti brush, water-air jet device, and Er:YAG laser] and chemical [amoxicillin; minocycline; tetracycline; H₂O₂ 3%; chlorhexidine 0.2%; NaOCl 0.95%; and hydro-carbon-oxo-borate-based formula antiseptic] protocols isolated. Negative control using 0.9% NaCl was adopted and PVPI 0.2% as a biofilm matrix-degrading agent applied before all chemical protocols. Surface deterioration and corrosion were analyzed before and after mechanical instrumentation as well as fibroblast adhesion on these degraded surfaces. The best *in vitro* mechanical/chemical protocol was tested in combination using *in situ* biofilm model. Er:YAG laser treatment displayed optimum surface cleaning by biofilm removal with minimal deleterious changes on the surface, smaller Ti release, anti-corrosion performance, and improved cell spreading. NaOCl 0.95% was the most effective chemical agent to reduce *in vitro* and *in situ* biofilms when applied isolated and more prominent results when associated with PVPI as pre-treatment to disrupt biofilm matrix. The combination of mechanical and chemical treatments promoted an optimum mechanical cleaning ability with biofilm matrix disruption and killing remnants *in situ* biofilms (~99% biofilm eradication). We conclude that Er:YAG laser + PVPI 0.2% + NaOCl 0.95% is considered an optimized decontamination protocol by demonstrating a potential to eliminate *in vitro* and *in situ* biofilms with minimum deleterious effects on 3D-printed Ti surfaces, opening new perspectives to improve implant-related infection therapies.

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

Coatings for Biomedical and Healthcare Applications (Symposium D) Poster Session

DP-ThP-1 Antibacterial Properties of Ag Doped Tetrahedral Amorphous Carbon Coatings Synthesized Using Hybrid Filtered Cathodic Vacuum Arc and Magnetron Sputtering System, *SangYul Lee, K. Oh, J. Park*, Korea Aerospace University, Republic of Korea; *D. Kim, J. Kim*, KIMS, Republic of Korea

Tetrahedral amorphous carbon (ta-C) coating is a hydrogen-free carbon coating with the remarkable properties comparable with those of diamond film, such as high hardness, optical transparency and chemical inertness. In this study, as silver (Ag) is known to be a potent antibacterial agent, silver (Ag) doped ta-C coatings with different Ag concentrations were synthesized on the AISI 316L and Ti64 coupons using an hybrid filtered cathodic vacuum arc (FCVA) and magnetron sputtering system. The doping effects of Ag concentration in ta-C coatings on the bacteria biofilms formed on the surface of medical products were examined. Surface condition and its physicochemical properties were investigated using SEM, AFM and XPS and also studied as their antibiofilm efficacy against a multi-drug resistant nosocomial pathogen *Pseudomonas aeruginosa*. In addition the antibacterial activity of Ag doped ta-C was evaluated by bacterial eradication tests with *Escherichia coli* (*E. coli*) at different incubation times. The result revealed that the synthesized Ag doped ta-C coatings inhibited the biofilm formation of *P. aeruginosa* and the antibacterial activity against *E. coli* in a concentration-dependent manner. Hence, this study demonstrated the possible use of Ag doped ta-C coatings against the biofilm-related infections out of *P. aeruginosa* and antibacterial activity against *E. coli*. Experimental details will be discussed.

DP-ThP-2 Adhesion, Corrosion Resistance, and Blood Compatibility of Mao- Pretreated Magnesium Alloy Coated with Graphene Oxide and Pyrolytic 1,8-Diaminooctane-Incorporated Oxidized Polydopamine, *Chau-Chang Chou, S. Chang, H. Lee*, National Taiwan Ocean University, Taiwan; *W. Chen*, Cheng Gung Memorial Hospital, Keelung, Taiwan

Surface treatment and functional coating can effectively reduce the degrading status and promote the blood compatibility of magnesium implants. This study applied micro-arc oxidation (MAO) treatment to improve the corrosion resistance of magnesium alloy. The electrolyte was prepared by using sodium silicate, sodium hydroxide, and sodium citrate. The MAO process was performed by adjusting voltage which was determined by observing the completeness of the ceramic films. Then, layer-by-layer assembly technique was implemented by dipping the substrates with graphene oxide, oxidized polydopamine mixed with pyrolytic 1,8-diaminooctane sequentially to provide their corrosion resistance, adhesive strength, and anti-clotting capability. The composition of substrate was confirmed by inductively coupled plasma mass spectrometry. Microstructural morphologies and crystallography of the MAO coatings were characterized using X-ray diffraction. Optical microscopy was implemented to observe the topographies of coatings. The samples' surface transition was revealed by water contact angle. Their surface topographies and element were observed by scanning electron microscopy and energy-dispersive X-ray spectroscopy. The cross-sectional morphology and the film thickness were evaluated by dual-beam focused ion beam microscopy. The function groups contained in composite multilayers were examined by Fourier transform infrared spectroscopy. The degradation resistance was measured by corrosion polarization curve. The combination of the multilayers was investigated by conducting scratch tests. The hemocompatibility of composite films for original, 7 days, and 15 days were studied according to the activated partial thromboplastin time and the platelet adhesion experiment. The results indicated that the coatings with multilayers of graphene oxide, oxidized polydopamine mixed with pyrolytic 1,8-diaminooctane layers after micro-arc oxidation had the best adhesion strength, corrosion resistance, and anticoagulant ability which can sustain more than 7 days under orbital shaking tests.

DP-ThP-3 Surface Alloying for Antibacterial Martensitic Stainless Steel Fabrication, *Z. Chen, B. Liu, Wen-Ta Tsai*, National Cheng Kung University (NCKU), Taiwan; *C. Huang*, Tung Mung Development Co., Ltd., Taiwan
Stainless steels (SSs) containing antibacterial elements, such as copper element, could be used as antibacterial structural materials. However, the traditional metallurgical processes for the fabrication of copper-containing

SSs include melting, casting, rolling and many other steps, which are highly costive and inconvenient for final product manufacturing. Furthermore, for ferritic and martensitic stainless steels, the low copper solubility in these grades of stainless steels makes copper alloying more difficult. Therefore, this study aims to develop a novel method for preparing copper-containing martensitic SS (namely SS 440C) on its surface by incorporating electrochemical plating and thermal diffusion process.

Successful surface alloying of copper onto SS 440C surface was achieved with unique processes consisting proprietary copper electroplating and subsequent thermal diffusion treatment. It generally accepted that stainless steels (normally SS 304) containing 3 wt% copper exhibits effective antibacterial activity. The SEM images and the associated EDS results of the above treated samples reveal that a surface layer with copper concentration greater than 3 wt% and a thickness as high as tens of micrometer could be produced on SS 440C surface by employing the novel technique invented in this investigation, which meets the requirement for antibacterial applications. The effective antibacterial performance of the above mentioned product was confirmed by employing the standard test (JIS-Z2801) for antibacterial activity examination.

DP-ThP-4 Enhancing the Surface Properties of Polymethylmethacrylate (PMMA) by Functionalizing with Atomic Layer Deposited Titanium(Iv) Dioxide, *Harshdeep Bhatia, C. Takoudis*, University of Illinois, Chicago, USA

Polymethyl methacrylate (PMMA) is a widely used polymer in applications such as engineering structural plastics, energy storage materials, and biomaterials. However, its poor surface properties lead to fracture and deformation. Functionalization of the PMMA surface can make it more resistant to aggressive environments and prevent it from biodegradation, discoloration, and increased surface roughness. Here, atomic layer deposition (ALD) was used to deposit TiO₂ thin films from tetrakis(dimethylamido)titanium (TDMAT) and ozone on PMMA substrates to improve its surface properties without the need for plasma assistance or another interlayer. Spectroscopic ellipsometry was used for the first time to measure the metal oxide film thickness on thick PMMA substrates. Two different growth regimes were observed, one for initial and the other for later ALD cycles. Initially, the growth rate on PMMA was 1.39 Å²/cycle, which is *3.5 times higher than that on stand-alone silicon (0.4 Å²/cycle); this is attributed to cyclic chemical vapor deposition of TDMAT and moisture within PMMA concomitant with TiO₂ ALD from TDMAT and ozone. However, after the formation of about 30-nm-thick film on PMMA, the TiO₂ growth rate became similar to that on silicon. Moreover, our results revealed that the presence of PMMA in the deposition reactor affects the TiO₂ growth rate on silicon substrates as well. These findings are discussed and corroborated with residual gas analyzer and X-ray absorption near-edge structure data. The thermal stability of the PMMA samples was examined by thermogravimetric analysis. Chemical composition and surface roughness of coated PMMA were studied by X-ray photoelectron spectroscopy and optical profilometry, respectively. The TiO₂ coating increased wettability by ~70% and surface hardness by 60%

DP-ThP-5 Diffusion-Based Plasma Nitriding for Surgical Needle, *Takao Yamauchi, P. Abraha*, Meijo University, Japan

Surgical instruments require medical grade and biocompatible materials with good corrosion resistance and tensile strength. Austenitic stainless steels are appropriate materials that can provide a sharp edge for repetitive use in medical procedures. These surgical tools may be cutting and holding tools or closure and hemostatic tools such as suture needles.

This research aims to enhance suture needles for improved ease of use in the repetitive suturing of large incisions. The shape of the needle body and the sharpness of the needle tip determines the force acting on suture needles. Therefore, better ease of use in terms of a smaller resistance force, a smaller body diameter, and a sharper needle tip are preferable. However, as we reduce the body diameter and sharpen the needle tip, the needle strength tends to decrease, and bending of the needle tip and possibly the needle body occurs, resulting in a short useful life span. In this study, surgical suture needles are plasma nitrided to suppress the bending of the needle tip and body for applications in large incisions.

Currently, silicone-coated or metallic glass-coated modified surfaces are the available methods that enhance the durability of surgical tools. However, delamination of the coating while in use may cause an undesirable medical problem for the subject and compromise the sharpness of the needle tip. This study aims to determine the insertion-retraction characteristics of untreated, silicone-coated, and plasma-nitrided suture needles. The results of our experiments show the measured

insertion-retraction characteristics curves of plasma nitrided suture needles were superior to those of the silicon-coated and untreated samples tested. Furthermore, since plasma nitriding is a diffusion process, the dimensional changes are minimal and no danger of delamination. The presentation will show the performance of plasma-nitrided stainless steel surgical suture needles.

DP-ThP-6 Development of TiO₂/Ag Multilayer Antibacterial Coatings Using Magnetron Sputtering Technique for Potential Applications in Non-Permanent Implants, *Sebastián Rodríguez Maya, M. Restrepo Posada, F. Bolívar Osorio, G. Bejarano Gaitán, J. Lenis Rodas, Universidad de Antioquia, Colombia*

The development of ideal surfaces to be used in metal implants and osseous stabilization devices require the consideration of different phenomena such as the formation of a bacterial biofilm on the surface followed by the usual appearance of infections or the total rejection of the device. On the other hand, the overgrowth of bone material around these devices constitutes a barrier when it comes to removing them when their function has been fulfilled. Due to this, the development of multilayer antibacterial coatings of TiO₂/Ag on Ti6Al4V substrates by means of the Magnetron Sputtering technique for its potential use in non-permanent implants were studied. The coatings obtained using this technique exhibit a uniform and densely packed, columnar growth profile and it also offers significant control over the microstructural features. Consequently, the properties of the developed systems were characterized using scanning electron microscopy (SEM), X-ray diffraction (XRD), tribologic assays and surface properties such as rugosity and contact angle.

DP-ThP-7 Development of Multilayer SiO₂/Ag Coatings Fabricated by Magnetron Sputtering for Potential Applications in Removable Implants, *Magali Restrepo Posada, S. Rodríguez Maya, J. Lenis Rodas, F. Bolívar Osorio, Universidad de Antioquia, Colombia*

One of the main reasons for the success of ceramic coatings doped with soft metals is their high use in biomedical applications, where non-permanent implants stand out thanks to their good combination of mechanical, tribological and bactericidal properties.

Among the main complications associated with this type of devices is the bacterial colonization on the implant surface can result in: infection and inflammation of surrounding tissue followed by loosening of the implant, as a solution to these problems the study of SiO₂ coatings has been proposed, which have been widely investigated for their high mechanical strength, thermal stability and adequate biocompatibility, although they are not active against all types of bacterial species which restricts their wide range of applications in the implant industries, it is then suggested the use of antibacterial materials such as Ag, which improve this property of the coatings. The fabrication of these coatings was performed using the magnetron sputtering technique consolidating a SiO₂/Ag multilayer system with properties of high homogeneity, very good adhesion and compactness. The properties of the developed systems were characterized by scanning electron microscopy (SEM), X-ray diffraction (XRD), tribological tests and surface properties such as roughness and contact angle.

DP-ThP-8 Evaluation of Biocompatibility and Corrosion Resistance of Strontium-Calcium Phosphate Coated Magnesium by Electrodeposition, *Jung-Eun Park, J. Ji, Y. Kim, S. Byeon, M. Lee, Chonbuk National University, Korea*

This study aims to improve the initial corrosion and biocompatibility of biodegradable magnesium by coating the magnesium surface with calcium phosphate containing strontium by electrodeposition. In this study, calcium phosphate coatings doped with Sr of various concentrations were electrodeposited on the magnesium surface at an applied voltage of 4 V for 1 h. The corrosion behavior and biocompatibility of the modified magnesium surfaces were evaluated.

The surface of the magnesium coated with strontium-calcium phosphate showed a dense dendritic shape and became denser as the strontium content increased. strontium-calcium phosphate coated magnesium showed improved corrosion resistance and lower pH change than pure magnesium. In vitro strontium-calcium phosphate coated magnesium showed enhanced bioactivity. strontium-calcium phosphate coated magnesium showed excellent osteoblast proliferation and differentiation.

Therefore, the strontium-calcium phosphate developed in this study inhibited the initial corrosion of magnesium and effectively formed corrosion products and bioactive materials on the surface. In addition, excellent biocompatibility was confirmed.

"This research was supported by Basic Science Research Program through the National Research Foundation of Korea(NRF) funded by the Ministry of Education (No. 2021R11A1A01057579) and the National Research Foundation of Korea(NRF) grant funded by the Korea government(MSIT) (No. 2021R1A2C2005466)"

DP-ThP-9 Improvement in Corrosion Resistance and Biocompatibility of Biodegradable Mg Surface with Combination of Calcium Phosphate and Chitosan, *Seo-young Kim, Y. Jang, T. Bae, M. Lee, Jeonbuk National University, Republic of Korea*

Metallic implants for bone fixation have been used for healing a fractured bone. For success of implantation, the superior bearing capacity and biocompatibility is needed. Magnesium, which is biodegradable metal, has great mechanical properties than other biodegradable materials, and its biocompatibility can be improved by functional polymer or ceramic coating. In this study, Mg surface was modified with bio-ceramic (apatite) containing chitosan to improve its corrosion resistance and biocompatibility.

For coating, chitosan_(sol) was prepared by dissolving 1% chitosan powder in 1% acetic acid (pH 6.0). Calcium phosphate_(sol) was prepared by mixing 0.25mol/L Ca-EDTA and 0.25mol/L KH₂PO₄ in DW (pH 5.9). The surface of pure Mg was thermally treated in the different ratio of mixture [Calcium phosphate_(sol) : chitosan_(sol)] for 1 h at 90°C. The morphology and composition of the surface were examined by SEM with EDS. The precipitated crystal structure and chemical bonding was analyzed using XRD and FT-IR. The electrochemical corrosion resistance of surface was measured by potentiodynamic polarization. The cytocompatibility on MC3T3-E1 osteoblastic cells was conducted.

After thermal treatment, porous and fine particles were formed on the Mg surface. The particles were uniformly covered on the surface. Increasing the mixing ratio of chitosan contributed to miniaturization of the particles and improvement of the density of the coating layer. The particles in coating layer were composed of Mg, C, O, P, and Ca components, and it was an apatite-based compounds with octacalcium phosphate (OCP) and hydroxyapatite (HA) crystal phase. FT-IR analysis results demonstrated the presence of chitosan in the coating layer. The bio-ceramic (apatite) layer increased the potential of the Mg surface, which improved corrosion resistance. It was effective on the increase of cell's proliferation.

The surface coating technique can form the uniform film consisted with chitosan-apatite particles. The dense apatite layer was effective in improving the corrosion resistance of magnesium and enhancing cytocompatibility.

"This work was supported by the National Research Foundation of Korea (NRF) grant funded by the Korea government (MSIP) (No. 2021R1C1C2006915 & 2021R1A2C1008740)."

DP-ThP-10 Influence of Plasma-Enhanced Chemical Vapor Deposition Associated with Different Finishing Procedure on the Degradation of CAD/CAM Dental Ceramic, *Aldiéris Alves Pesqueira, L. Scaion Silva, São Paulo State University (Unesp), School of Dentistry, Araçatuba, Brazil; V. Adelino Ricardo Barão, (University of Campinas (UNICAMP), Piracicaba Dental School, Brazil; K. Henrique Cruz, V. Alves Nascimento, J. Pedro Justino de Oliveira Limírio, São Paulo State University (Unesp), School of Dentistry, Araçatuba, Brazil; B. Egumi Nagay, University of Campinas (UNICAMP), Piracicaba Dental School, Brazil; E. Cipriano Rangel, Sao Paulo State University (UNESP), Laboratory of Technological Plasmas (LaPTec), Engineering College, Sorocaba, Brazil*

Acid erosion is common in patients with gastroesophageal reflux disease or eating disorders (nervous bulimia and/or anorexia). Such condition can degrade the mechanical strength of oral restorative materials such as ceramics. Surface treatments have been used to protect or improve the mechanical properties of ceramic materials. In this context, plasma-enhanced chemical vapor deposition (PECVD) is a promising technique for depositing a thin film on ceramics. Therefore, we aimed to evaluate the structural and mechanical characteristics of zirconia/silica ceramic in a CAD-CAM interpenetrating resin matrix (Shofu Block HC) as a function of different finishing procedures associated or not with PECVD after erosive challenge. A total of 120 specimens were divided into 4 groups of surface finishing: only mechanical polishing (MP), only application of light-curing sealant (S), association of MP or S with PECVD (MP+PECVD and S+PECVD). A steel reactor was used for PECVD application and a thin-film was deposited under an atmosphere of 85% hexamethyldisiloxane monomer (HMDSO) and 15% argon (Ar). The erosive challenge was performed with 5% HCl (pH = 2.0) for 273 h. The surface roughness (Ra), surface free energy, Vickers microhardness, flexural strength, and elastic modulus of

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ceramic material as a function of surface finishing were investigated before and after the erosive challenge. A homogeneous silicon-based thin film was successfully deposited on ceramic surface. The PECVD thin film was able to reduce the surface roughness and increase flexural strength of ceramic when associated with mechanical polishing, even after erosive challenge, while the other groups had an increase in roughness ($p < 0.05$). In general, PECVD increased the ceramic surface hardness, and reduced the surface free energy when associated with mechanical polishing or sealant application before and after erosive challenge ($p < 0.05$). In conclusion, the PECVD thin film effectively enhanced the roughness, hardness, surface energy, flexural strength, and elastic modulus of zirconia/silica ceramic in an interpenetrating resin matrix, mainly when associated with mechanical polishing. The creating of silicon-based thin films might be a good strategy to reduce ceramic degradation in the oral environmental.

Funding from São Paulo Research Foundation (FAPESP) (Grants # 2021/08529-7 and 2021/07251-5).

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