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Coatings for Biomedical and Healthcare Applications Room On Demand - Session DP

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

DP-1 Optimisation of Electrolytic Plasma Oxidation (PEO) Coatings Formed on Magnesium for Biological Applications*, Yue Guo (yue.guo-2@manchester.ac.uk),* *A. Rogov, B. Mingo, A. Matthews, A. Yerokhin,* The University of Manchester, UK

Magnesium has shown great potential for the next generation of resorbable implant materials. It has well biocompatibility and biodegradability, high strength-to-weight ratio and stiffness similar to that of the human bone. However, magnesium exhibits poor corrosion behaviour, leading to the early deterioration of the implant. Therefore, appropriate surface treatments have to be applied to improve the corrosion resistance of magnesium.

Electrolytic Plasma Oxidation (PEO) is a plasma-assisted technique to form ceramic-like coatings containing oxides comprising constituents of both the parent metal and the electrolyte. PEO coatings can increase corrosion resistance and mechanical properties of the metal substrate, improving the longevity and reliability of the implant. Furthermore, the coating properties can be adjusted by tailoring parameters of the PEO process, such as electrolyte composition and pulsed electrical regime. Previous studies have been mainly focused on the influence of current density, frequency and duty cycle of rectangular pulses, whereas very few works were dedicated to the influence of the pulse shape.

The objective of this work is to investigate a possibility of increasing the corrosion resistance of PEO coatings on Mg by tailoring the current pulse shape. Triangular pulses are given particular attention in comparison with the commonly used rectangular shapes. Two types of rectangular pulses are generated – a Slow ON pulse, where the applied current increases linearly at a certain rate followed by an instant drop of the current; and a Slow OFF pulse, where the applied current increases instantly before decreasing linearly. Characteristics and properties of the coatings produced under different waveform are thoroughly studied. Corrosion tests are performed to evaluate the corrosion resistance. The results have shown a positive effect of the Slow OFF pulse. A more uniform and defect-free coating surface morphology is obtained. The coating exhibits higher corrosion resistance correlated to the better morphology.

DP-2 The Property of Adhesion and Biocompatibility of Silicon and Fluorine Doped Amorphous Carbon Films*, Masafumi Toyonaga (m.tyng.keio@gmail.com),* Keio University, Japan*; T. Hasebe,* Keio University, Tokai University Hachioji Hospital, Japan*; S. Maegawa,* Tokai University Hachioji Hospital, Japan*; T. Matsumoto,* Keio University, Tokai University Hachioji Hospital, Japan*; A. Hotta, T. Suzuki,* Keio University, Japan

Application of nickel-titaniumu (NiTi) alloys to medical implant devices is increasing due to their unique characteristics. To ensure good biocompatibility in the human body, fluorine-doped amorphous carbon (a-C:H:F) coating is a promising candidate. Generally, a-C:H:F coating shows poor adhesion on metallic alloys, so that silicon-incorporated interlayer is introduced between a-C:H:F and metallic alloys. However, this membrane design has a risk of delamination at the outermost interface (a-C:H:F // interlayer), and also there is a practical problem that coating time becomes long because the deposition process in multiple stages is required. Here we develop silicon and fluorine doped amorphous carbon (a-C:H:Si:F) film which exhibits high adhesion and excellent biocompatibility.

The a-C:H:Si:F film and a-C:H:F film (control) were deposited on NiTi substrates using radio frequency plasma enhanced chemical vapor deposition (RF-PECVD) equipment. Chemical compositions and bonding states of the surfaces were determined by X-Ray photoelectron spectroscopy (XPS). Surface free energy was estimated based on Owens-Wendt method using the results of contact angle measurement. Nanoscratch tests were conducted in order to quantify the adhesion strength. Platelet adhesion test and leukocyte adhesion test were conducted in order to evaluate biocompatibility.

First of all, a-C:H:Si:F was deposited from a mixture of TMS ($Si(CH_3)_4$) and C_3F_8 at TMS flow rate of 6.0 sccm and C_3F_8 flow rate of 50 sccm. Although this shows the possibility of new film deposition from a mixture of TMS and C_3F_8, the adhesiveness and biocompatibility of a-C:H:Si:F were not higher than a-C:H:F with Si-interlayer. Therefore, "C_2H_2-doped" a-C:H:Si:F film, which was deposited using a mixcure of TMS, C_3F_8 and C_2H_2 at a TMS flow rate of gradually changed from 6.0 sccm to 0.0 sccm, C_2H_2 flow rate of gradually changed from 0.0 sccm to 3.0 sccm and C_3F_8 flow rate of gradually changed from 0.0 sccm to 50.0 sccm, was newly deposited and this film showed similar chemical composition, bonding state, surface free energy, and higher adhesive strength than a-C:H:F with Si-interlayer, and the same number of adhesive platelets and leukocytes as a-C:H:F.

These results demonstrated that a single film with both adhesion of Si-interlayer and biocompatibility of a-C:H:F was fabricated. Furthermore, this a-C:H:Si:F coating can be anticipated as an effective film coating method in a practical point of view, because the film deposition is completed in one process.

DP-3 *In Vitro* Study of Very Thin Gold Film Deposited Collagen Fabric*, Sheng-Yang Huang (huangmochiqqegg@gmail.com), . Hsieh,* Feng Chia University, Taichung Veterans General Hospital, Taiwan*; R. Chang,* Feng Chia University, Taiwan*; C. Chou,* Taichung Veterans General Hospital, National Yang-Ming University, Taiwan*; C. Chung,* Central Taiwan University of Science and Technology, Taiwan*; J. He,* Feng Chia University, Taiwan

The goal of this study is to test a novel collagen fabric biomedical material with very thin gold film successfully deposited by using high-power impulse magnetron sputtering (HIPIMS). Previous study show that the gold layer is morphologically tunable from island distribution to continuous layer by manipulating deposition time. Here, this study aims to explore the *in vitro* response of the specimens deposited for 0, 3, 6, 51, and 81 seconds, representing the gold layer coverage percentage from 3.07% to 51.22%, respectively. Cell attachment test based on Alamar Blueassay using WS1 fibroblast and antibacterial test based on Kirby-Bauer disk diffusion method were carried out. Experimental results reveal that the gold layer prohibits fibroblast attachment, regardless of the gold layer coverage percentage. Microscopic observation disclosed the fibroblasts inactivation on the gold layer surface. For pseudomonas aeruginosa pathogen, randomly selected specimens with 3 and 6 seconds gold deposition showed inhibition zone of 13 and 11 mm, respectively. The results of this work support the use of this biomedical material in early phase of wound healing for its evidence of fibroblast attenuation and antibacterial effects.

DP-4 e-Poster Presentation: Metallization of Polymers for Medical Applications using HiPIMS*, Aarati Chacko (aarati.chacko@empa.ch), K. Thorwarth, R. Crockett, U. Müller, H. Hug,* Empa, Swiss Federal Laboratories for Materials Science and Technology, Switzerland

In contrast to wet processes, which require toxic precursors, High Power Impulse Magnetron Sputtering (HiPIMS) is a relatively clean method to achieve polymer metallization. This makes it especially interesting for medical applications such as coating polymer implants. The large proportion of metal ions in the coating discharge, characteristic of HiPIMS, allows a high level of control over film-forming species. This physical vapor deposition method is therefore our method of choice to tailor and study the substrate-film interphase region responsible for adherent and durable coatings.

This study aims to understand the effect of oxygen plasma activation on the surface of a polymer using AFM and XPS. We then relate this to HiPIMS-metallized surfaces and interfaces using ATR-FTIR. The metal-polymer system for this study is titanium on PEEK (Polyetheretherketone), which has shown exemplary adhesion in the case of orthopedic implants for use in spinal fusion surgery. We aim to understand the interactions that lead to this good adhesion to bring further improvements, and also, translate our understanding to other metal-polymer systems.

DP-5 TiZrSiN Coatings, Structural Characterization, and Corrosion Resistance in Ringer's Lactate*, Claudia Patricia Mejía Villagrán (clapamevi21@gmail.com),* Universidad Nacional de Colombia*; M. Chellali,* Karlsruhe Institute of Technology (KIT), Libya*; C. Garzón Ospina,* Universidad Nacional de Colombia*; H. Hahn,* Karlsruhe Institute of Technology (KIT), Germany*; J. Olaya Flórez,* Universidad Nacional de Colombia*; L. Velasco Estrada,* Karlsruhe Institute of Technology (KIT), Germany

TiZrSiN coatings produced by Physical Vapor Deposition in dual condition are characterized. Power density in Zr target was adjusted at three different values (2.0 Wcm^{-2}, 2.9 Wcm^{-2}, and 3.5 Wcm^{-2}), while power density on the TiSi target was fixed (0.55 Wcm^{-2}). As a result, three types of coating were obtained; one with a mostly amorphous structure (2.0 Wcm^{-2}), one with crystalline structure with some amorphous structure (2.9 Wcm^{-2}), and a third one with mostly crystalline structure (3.5 Wcm^{-2}).

Corrosion tests using electrochemical impedance while dipping the coatings in ringer's lactate, showed that coatings with TiZrSiN demonstrate better corrosion resistance than their ZrN peers. From all studied conditions, the one with the best resistance to corrosion (2.9 Wcm^{-2}), also demonstrated the highest hardness and the best performance and stability at the tests of electrochemical impedance spectroscopy with time. The improved properties in 2.9 Wcm^{-2} condition/case are attributed to the lattice stabilization for solid solution and adequate portion of silicon content.

DP-6 A Novel Synthesis Method of Carbide Derived Carbon (CDC) Surface Modification for Hip Implants, ***Yani Sun (ysun98@uic.edu)***,** University of Illinois at Chicago, USA*; K. Cheng, M. Mathew,* UIC College of Medicine at Rockford, USA*; M. McNallan,* University of Illinois at Chicago, USA

The inferior tribocorrosion behavior of commonly used biomedical alloys has led to the early failure of total hip replacements (THRs) and serious complications. In 2011, the tribolayer comprising graphitic carbon was found from the retrieved implant, and it was reported as solid lubrication which can reduce the friction between the femoral head and cup. Inspired by this interesting discovery, we have proposed a surface modification method, carbide-derived carbon (CDC), to mimic the tribolayer to improve the tribocorrosion resistance. The results have shown that CDC produced by direct chlorination can provide excellent protection to Ti6Al4V and has high durability.

In this study, a novel method has been developed to synthesize CDC on Ti6Al4V substrates by electrolysis from a low melting point halide salt. Compared to previous preparation processes, this newly developed approach eliminates the exposure to chlorine gas and the requirement of the inert gas environment, which makes the synthesis process more controllable and the CDC layer more uniform. X-ray diffraction (XRD), Raman spectroscopy and scanning electron microscopy (SEM) were utilized for characterization. Based on the results, the produced CDC has a porous structure which may contain nanocrystalline graphite (NCG) and amorphous carbon (a-C). The theoretical thickness of the sample is estimated by a calculation which is approximately 1.44 µm. In addition, the performance of the new coating was tested in a tribocorrosion hip simulator. A system has a pin on ball contact and immersed in bovine calf serum of 30g/L protein concentration, with a pH of 7.6 and a temperature of 37˚C. We applied a normal force of 16N to obtain a contact pressure of around 10MPa and ran the test for 3600 cycles with 1Hz. A Gamry made Potentiostat is connected to the test system, monitoring the electrochemical responses induced by the tribological activity. According to the recordings of normal and tangential forces, the evolution of the friction coefficient is deducted and reported in the results.

The findings have shown that the CDC samples prepared by the electrolysis method exhibit smaller friction coefficient (approximately 0.1), wear loss and potential drop (less than 100 mV compared to 600mV for substrate). Therefore, it is promising that the CDC prepared by the novel electrolysis approach can protect Ti6Al4V substrates from the tribocorrosive damages. For future work, we propose to conduct the adhesion test, the tribocorrosion experiments under potentiostatic mode and the biocompatibility test to fully evaluate CDC's value as a novel material for hip implants.

DP-7 Enhancing Osseoointegration on PEEK Spinal Implant by Using Laser Surface Roughening and HIPIMS Titanium Coating, ***Ping-Yen Hsieh (pyhsieh@fcu.edu.tw)***,** Feng Chia University, Taiwan*; H. Tsou,* Taichung Veterans General Hospital, Taiwan*; C. Chung,* Central Taiwan University of Science and Technology, Taiwan*; J. He,* Feng Chia University, Taiwan

Current spinal interbody fusion cages are most widely adopted from polyetheretherketone (PEEK) due to its favorable biomechanical properties and X-ray radiolucency characteristics. Unfortunately, the smooth and bioinert surface of PEEK may limit the osseointegration and inhibit bone fusion. Plasma spraying, providing porous and rough titanium layer over the PEEK spinal implant, has been commercialized in clinical application though, this study aims to develop an alternative approach by firstly laser roughening PEEK surface, followed by high power impulse magnetron sputtering (HIPIMS) to deposit a strongly adhered titanium layer for improving osteointegration of PEEK spinal implant. The experimental results showed that properly controlled laser condition gives micrometer-scale topography over the PEEK surface as opposed to the smooth bare PEEK. After HIPIMS deposition, the obtained titanium film presented an adhesion of 5B grade even after immersion in simulated body fluid (SBF) environment for 28 days based on the Scotch-tape adhesion test. Such excellent film adhesion performance is ascribed to the advantage of high ion energy and high-density plasma characteristics of the HIPIMS discharge. In addition, the titanium film on roughened PEEK presented better osteoblast compatibility and osseointegration than the commercial product, so as to provide the high spine stability after implantation. Finally, the long-term assessment results revealed the high stability and no degradation concern for the modified PEEK, which can avoid the malignant reaction between implant and host to ensure the safety after implantation in the human body. In summary, the two-step surface modification on PEEK satisfy the requirements for enhancing osseointegration, suggesting clinical application consideration.

DP-8 Superamphiphobic Stainless Steel Surface Prepared by Femtosecond Laser Patterning and Pulsed Plasma-Polymerization, *C. Lin,* Central Taiwan University of Science and Technology, Taiwan*; C. Chou,* Taichung Veterans General Hospital; National Yang-Ming University, Taiwan*;* ***Chi-Jen Chung (cjchung@seed.net.tw)***, Central Taiwan University of Science and Technology, Taiwan*; J. He,* Feng Chia University, Taiwan

Superamphiphobic surfaces, being super-repellent either water or oil, show various applications in self-cleaning, antifouling, non-staining surfaces, spill-resistant, corrosion prevention, and liquid separation. By employing femtosecond laser patterning and pulsed plasma polymerization, this study developed a dual-technique of surface modification to obtain superamphiphobic surfaces on the AISI 304 stainless steel substrates, usually made into dental archwires in orthodontics and dentofacial orthopedics. The characteristics of the superamphiphobic surfaces and *in vitro* wear tests in artificial saliva that mimicked tooth brushing, peanut-chewing, and nougat-chewing modes were performed to determine the durability of the superamphiphobic layer.

The experimental results showed that the water and oil contact angle (WCA and OCA) for bare stainless steel is 65° and 18°, respectively. After dual-technique treatment, the WCA and OCA were 160° and 146°, respectively; namely, both hydrophobicity and oleophobicity were enhanced significantly. It remains WCA and OCA to be 137° and 120°, respectively after 500 times toothbrush wear test. On the other hand, for simulating the food chewing circumstances, the WCA and OCA were, respectively, 129° and 26° for peanut, and 133° and 80° for nougat after 500 times. The peanut-chewing causes much disappearing superamphiphobic behavior than nougat-chewing because the carbohydrate, protein and oil ingredients in peanut transferred onto the surface. This has been verified by SEM, EDS, and FTIR analyses. As a whole, the superamphiphobic surface prepared on the dental stainless steel substrate exhibits good durability, demonstrating the promising applications in dental archwires for orthodontics and dentofacial orthopedics.

DP-9 Light-activated High Efficiency Antimicrobial and Antiviral Coatings, ***Victor Bellido-Gonzalez (victor@gencoa.com)***,** *P. Killen, T. Sgrilli, D. Monaghan,* Gencoa Ltd, UK*; O. Hernandez-Rodriguez,* IK4-TEKNIKER, Spain

Antimicrobial resistance (AMR) is one of the major global challenges facing healthcare. Prevention of infections acquired in hospitals is the most effective way to fight AMR. Bacteria and other pathogens could be transferred via shared touch surfaces and instrumentation, and unfortunately health centres like hospitals present a breathing ground opportunity for some of the more resistant strains of pathogens. Maintaining an sterile environment is not always easy. Some of the complex instrumentation and equipment in hospitals, like robotics surgery instrumentation, are difficult to undergo through regular complete sterile conditioning protocols as they require complex and expensive cleaning procedure. In some cases the standard sterilasition autoclaving is not possible due to the nature of the instrumentation itself.

An approach which would offer a lower risk of cross contamination in such environments is the use of surfaces which can be "activated" and rapidly kill pathogens. In this paper we will present solutions based on surface coating technology which by light-activation becomes a very effective self-sanitizing surface, able to kill to levels of >99.99% of bacteria.

Recent developments by the authors have provided new analytical techniques for quantifying the ligh-activated antimicrobial efficiency of these coatings. Some of the coatings developed have been able to achieve high sterilisation performance even under "standard office"visible light conditions. Results will be presented.

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