Coatings for Biomedical and Healthcare Applications Room Grand Hall - Session DP-ThP

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

DP-ThP-4 Development of Multilayer HA-Ag and TiN-HA-Ag Coatings Deposited by RF Magnetron Sputtering with Potential Application in the Biomedical Field, ***Julian Lenis***, *G Gaitán,* University of Antioquia, Medellín, Colombia; *P Rico, J Ribelles,* Universitat Politècnica de València, Spain; *F Bolívar,* University of Antioquia, Medellín, Colombia

The use of composite coatings emerges as a great alternative to induce superficially the combination of properties widely desired in surgical implants, such as: osteointegration and bactericidal character, which can not be conferred by a single material. In the present investigation, the effect of the incorporation of an intermediate layer of titanium nitride (TiN) on the chemical composition, structure, morphology, phases and adherence of a multi-layer Hydroxyapatite (HA) - silver (Ag) coating deposited on Ti-6Al-4V by magnetron RF sputtering was evaluated. The elemental composition analysis was performed by energy dispersive spectroscopy, while the techniques of micro-Raman spectroscopy, scanning electron microscopy and atomic force microscopy were used to determine the structure and morphology of the obtained coatings. A variation in the Ca/P ratio, the Ag content and the thickness in the HA-Ag coatings deposited on the TiN layer was found compared to the HA-Ag system deposited on the metallic alloy. In the same way, the roughness and structure of this coating was modified according to the surface where it was deposited.

Key words**:** Magnetron sputtering, Hydroxyapatite, Ca/P ratio, structure, multi-layer coating, intermediate layers, critical load.

DP-ThP-5 Electrochemical Activated Iridium Oxide Film as a Bio-interface Electrode for Neurostimulation Applications, *Y Chiu, P Chen,* National Taipei University of Technology, Taiwan; ***Chien-Ming Lei***, Chinese Culture University, Taiwan; *P Wu,* National Chiao Tung University, Taiwan

Electrode materials for neural stimulation have been widely investigated for implantable devices. Among them, iridium and iridium oxide are attractive materials for bio-interface applications due to their desirable stability, electrochemical performanc, and biocompatibility. In this study, metallic iridium thin film was deposited on a transparent conducting oxide substrate (ITO-coated glass) by radio-frequency (RF) magnetron sputtering, and we carried out an electrochemical activation to produce iridium oxide film through a repetitive biphasic pulsed current. The process parameters for sputtering of iridium film and electrochemical activation of iridium oxide film were optimized. The activated iridium oxide film exhibited superior electrochemical performance, including large charge storage capacity (CSC), high charge injection capability, low electrochemical impedance, and excellent stability. In addition, the biocompatibility of activated iridium oxide was evaluated by cytotoxicity, and the iridium oxide/iridium film showed high cell-viability. These findings suggest that the activated iridium oxide film is a promising candidate as an electrode material for the development of neurostimulating devices.

DP-ThP-6 HIPIMS Titanium Dioxide on Laser Roughened PEEK Surface for Biomedical Application, *P Hsieh,* Institute of Plasma, Department of Materials Science and Engineering, Feng Chia University, Taiwan; ***Chi-Jen Chung***, Central Taiwan University of Science and Technology, Taiwan; *H Tsou,* Taichung Veterans General Hospital, Taiwan; *H Chen,* China Medical University Hospital, Taiwan; *J He,* Institute of Plasma, Department of Materials Science and Engineering, Feng Chia University, Taiwan

Polyetheretherketone (PEEK), known for its comparable elastic modulus to human cancellous bone characteristics and X-ray radiolucency, is greatly considered for spinal implant material. However, the bio-inertness and hydrophobic surface properties of PEEK results in poor osseointegration when implanted into human bodies. The aim of this study is to develop a combination method, *viz.*, laser roughening process and titanium dioxide (TiO_2) deposition, for modifying the biological properties of PEEK surface. A femtosecond pulse laser was utilized to avoid the thermal damage on PEEK, while high power impulse magnetron sputtering (HIPIMS) was employed to deposited high crystallinic TiO_2 coating at low temperature. The results showed that the hierarchically patterned PEEK surfaces composed of nano- and microstructures can be obtained by adjusting laser parameters. Such structure resulted in varied surface roughness and water wetting ability. On the other hand, the highest rank (5B) in adhesion tape test proved the superior adhesion of the HIPIMS prepared TiO_2 coating even on roughened PEEK surface. The strong adhesion is believed to arise from the advantage of high ion energy and high-density plasma characteristics of the HIPIMS discharge. Under proper laser roughening condition followed by HIPIMS-TiO_2, the *in vitro* osteoblast compatibility test performed a much higher level than bare PEEK.

DP-ThP-7 Corrosion Property and Biocompatibility Evaluation of Fe-Zr-Nb Thin Film Metallic Glasses, *B Lou,* Chang Gung University, Taiwan; *T Lin,* ***Jyh-Wei Lee***, Ming Chi University of Technology, Taiwan; *J Wang, Y Yang,* National Taipei University of Technology, Taiwan

The amorphous thin film metallic glasses (TFMGs) have drawn lots of attention by researchers due to their unique properties and ease of fabrication. Recently, the biocompatibility of Zr-based TFMG becomes an important issue because of its excellent corrosion resistance and bio safety . In this work, six Zr-Ti-Si TFMGs with different Si concentrations were fabricated on the bio-grade 316L stainless steel plates and Si wafer, respectively, by a hybrid bipolar high power impulse magnetron sputtering and radio frequency sputtering technique. The chemical composition of Si increased gradually from 3.3 to 34.7 at.%, respectively, as the Si target power increased from 25 to 250 W. The crystalline structure was observed for TFMG containing 3.3 at.% Si, whereas the amorphous phase was found for the TFMG containing higher than 9.6 at.% Si. The cross-sectional morphology changed from columnar to fine and featureless microstructure as more silicon contents were added into the thin film. Acceptable adhesion qualities, HF1 to HF 3, were obtained for all Zr-Ti-Si thin films. The maximum hardness, 15.7 GPa, and the highest H/E value around 0.088 were achieved for TFMG containing 34.7 at.% Si. The corrosion resistance of 316L stainless steel disk can be improved effectively by TFMGs. The lowest corrosion current density around 0.02 mA/cm^2, and the highest polarization resistance around 1042.1 $kWcm^2$, were achieved for TFMG containing 31.6 at.%. Six thin films had better biocompatibility than that of 316L stainless steel substrate. The hybrid HiPIMS-RF grown Zr-Ti-Si TFMGs with adequate hardness, good biocompatibility can be used as a promising candidate to improve the surface biocompatibility of biomaterials.

DP-ThP-9 Bone-like Nano-hydroxyapatite Coating on Low-modulus Ti-5Nb-5Mo Alloy Using Hydrothermal and Post-heat Treatments, *H Hsu, S Wu, S Hsu,* Central Taiwan University of Science and Technology, Taiwan; ***Wen-Fu Ho***, National University of Kaohsiung, Taiwan

Titanium and its alloys have been widely used as biomaterials for orthopedic and dental implants because of their excellent biocompatibility and mechanical properties. However, they are considered to be bioinert, such that when they are inserted into the human body these implants cannot bond directly to the surrounding living bone. This study aimed to improve the bioactivity of a low-modulus Ti-5Nb-5Mo alloy with a hydroxyapatite (HA) surface coating using eggshells as a Ca source through hydrothermal reaction and heat treatment. The results showed that the whole alkali-treated alloy surface was covered with amorphous calcium phosphate nanoparticles after hydrothermal reaction at 200 °C for 48 h. When sub sequently heat-treated at various temperatures (400, 500 or 600 °C) for 48 h, the surface coating of Ti-5Nb-5Mo alloy was transformed into crystalline rod-like HA nanoparticles. Also, heat treatment enhanced the adhesion between the HA coating and the Ti alloy substrate. Additionally, FTIR analysis confirmed the production of HA containing mixed AB-type carbonate substitutions. To evaluate bioactivity of the bone-like HA-coated Ti-5Nb-5Mo alloy, the capability of calcium phosphate apatite formation on the alloy surface was assessed by immersion in a simulated body fluid (SBF). Dune-like apatite layer was observed to densely deposit on the surface of HA-coated Ti alloy after 6 h of immersion in the SBF. Notably, the ability of Ti-5Nb-5Mo alloy subjected to sequential process with alkali, hydrothermal, and heat treatments to form bone-like HA nanoparticle coating was obviously greater than that of its counterpart without HA coating.

DP-ThP-10 Surface Characteristics and Structure of Porous TNM Alloy for Biomedical Applications, *W Ho,* National University of Kaohsiung, Taiwan; *S Wu, S Hsu, W Hsiao,* ***Hsueh-Chuan Hsu***, Central Taiwan University of Science and Technology, Taiwan

Titanium (Ti) and some of its alloys have been used widely as load-bearing implants because of their excellent mechanical properties, superior biocompatibility, and good corrosion resistance. Lately, there has been an increasing interest in studying porous alloy, which could imitate bone structures by altering the porosity of alloy. In this study, a biomedical porous Ti-5Nb-5Mo (wt.%) alloy was fabricated by mechanical alloying

(MA) process for different ball-milling times. The metal powders of Ti (99.9% pure), Nb (99.9% pure), Mo (99.95% pure) were milled in a planetary ball milling machine for 3 h, 15 h and 30 h. The ball milled Ti-5Nb-5Mo powders were compacted of 7 mm in diameter and 11 mm in height. The sintering process was carried out in two steps. The compacts were initially sintered at 175 °C for 2 h and then increased to 1100 °C for 5, 10 and 15 h respectively. The results found in this study are summarized as follows: The Ti–5Nb–5Mo particle size increased with the ball-milling time increased from 3 h (35 µm) to 15 h (72 µm). The ball milling produced alloy particles gather together, and caused a larger particle size. The ball milled Ti–5Nb–5Mo particles were significantly refined, and the Nb and Mo was integrated and uniformly distributed in the matrix. XRD analysis shown that the porous Ti–5Nb–5Mo was a α phase and has no obvious diffraction peaks of elemental Nb and Mo remained which confirmed Nb and Mo was integrated and uniformly distributed in the matrix . While the sintering of powder by ball milling enhances the homogeneity. The compressive strength and modulus of all the porous Ti–5Nb–5Mo match the necessary mechanical property of cancellous bones. Especially for the B3S15 specimen (Balling 3 h and sintering 15 h) which shows the highest strength. After soaking in a SBF solution for 7 days, the porous TNM alloy formed a dense apatite layer on the surface. It exhibited a better apatite-forming ability. The study reveals that the use of porous TNM satisfies the need of implants with an adequate mechanical strength and elastic modulus for the patients.

DP-ThP-11 In vitro Wear Tests of the Dual-layer Grid Blasting-plasma Polymerized Superhydrophobic Coatings on Substrates Made into Dental Stainless Archwires, ***Cheng-Wei Lin,*** Feng Chia University, Central Taiwan University of Science and Technology, 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

Dental stainless archwires, frequently used in orthodontics and dentofacial orthopedics, may accumulate food debris, promote bacterial overgrowth, and subsequently result in dental caries. A dual-layer grid-blasting plasma-polymerized (GB-PP) superhydrophobic coating was developed in a previous work by changing the micro- and nano-structured surface morphology on AISI 304 stainless substrates. In this study, in vitro wear tests were performed in artificial saliva that mimicked tooth brushing, peanut-chewing, and nougat-chewing modes to determine the durability of the superhydrophobic layer. Experimental results show that peanut-chewing causes more damage to the superhydrophobic surface than nougat-chewing because the carbohydrate, protein and oil ingredients in peanut and nougat might be transferred onto the surface, subsequently masking some of the fluorocarbon layer (also verified by SEM, EDS, and FTIR analyses). In conclusion, the GB-PP coatings deposited on medical-purposed stainless steel substrates exhibit good durability after tooth brushing and nougat-chewing wear tests.

DP-ThP-19 Obtaining of CVD Nanodiamonds and Evaluation of the Cytotoxicity in B16f10 Cells for Treatment of Melanoma, *C Wachesk,* Federal University of São Paulo (UNIFESP), Brasil*; C Hurtado,* Institute of Science and Technology, Federal University of São Paulo (UNIFESP), Brazil*; **Rebeca Falcão**,* Institute of Science and Technology, Federal University of São Paulo (UNIFESP), Brasil*; D Arruda,* University of Mogi das Cruzes, Brasil*; D Tada,* Institute of Science and Technology, Federal University of São Paulo (UNIFESP), Brasil*; V Airoldi,* National Institute for Space Research (INPE), Brasil

Recent studies have shown the potential use of nanodiamonds (NDs) as drug carriers for the therapy of cancer due to their high stability and small size. With the aim of obtaining a new system to be applied as drug delivery platform for the therapy of metastatic melanoma, a new technique of obtaining NDs from CVD diamond thin film was developed. The synthetic CVD-diamond film has similar physical and chemical properties to natural diamond: extreme hardness, excellent thermal conductivity, biological compatibility and chemical stability at temperatures below 800°C. Herein, CVD NDs were prepared by using laser ablation. The NDs were characterized by X-ray (XRD), (MEV-FEG), (TEM), energy dispersion spectroscopy (EDS), (XPS), Raman spectroscopy and dynamic light scattering. Furthermore, since cytocompatibility is one of the main features required for a drug delivery platform, the cytotoxicity of NDs was evaluated in B16-F10-Nex2 cells by MTT assay. The results showed that the laser ablation process reduced CVD particle size. The mean hydrodynamic diameter in aqueous suspension after the centrifugation changed from 54 nm. The high stability of aqueous suspension of CVD NDs was indicated by the low polidispersity index (0,2) and a small increase in the mean value of hydrodynamic diameter during the observed period (D = 215 nm). The high stability was provided by the high charge density on NDs surface as suggested by the high value of Zeta-potential (-36.39 and -30.94 mV). EDS analysis showed that NDs were composed of carbon (77.2%) and oxygen (22.2%). By X-ray diffraction analysis, it could be observed the characteristic peak of NDs at 43^0. Raman spectrum of CVD NDs showed three peaks at: 1332, 1500 and 1600 cm^{-1}, corresponding to D and G bands of diamond. Cytotoxicity assay showed 60% and 80% of cell viability after 24h 48h 72h and 96h of incubation with NDs. The high value of cell viability is an indicative of the cytocompatibility of NDs, indicating the potential use of NDs in biomedical applications such as drug delivery platforms.

DP-ThP-22 Tantalum Oxynitride PVD Coatings a Potential Candidate for Dental Implants Application, *O Banakh,* University of Applied Sciences (HES-SO), Switzerland*; **Pierre-Albert Steinmann**,* Positive Coating SA, Switzerland

Coating technology offers innovative solutions to improve the quality and durability of medical devices. Among new materials, titanium oxynitride coatings (TiOxNy) are considered promising for applications in implantology (cardiovascular stents) due to their high biocompatibility. The purpose of this study is to test tantalum oxynitride coatings (TaOxNy) as a potential candidate in dental implants application. Coatings with different nitrogen and oxygen contents were deposited by conventional reactive magnetron sputtering and by High Power Impulse Magnetron Sputtering (HIPIMS) in mixed Ar-O_2-N_2 atmosphere. The coatings were deposited onto titanium micro-rough substrates (Ti-SLA) and stainless steel substrates. In some experiments water vapor was used as a reactive gas, instead of oxygen. The Ti-SLA uncoated sample was chosen as control in cellular response biological tests. None of the specimens presented any signs of cytotoxicity. Their biological response was similar to that of Ti-SLA. The coatings produced with water vapor showed an improvement of the corrosion resistance as well as a slight enhancement of cell adhesion. Even though the tantalum oxynitride coatings didn't show a noticeable enhancement of biological response on Ti-SLA surfaces, their application looks promising on other substrates such as stainless steel.

DP-ThP-25 Influence of Ag-Cu Nanoparticles on the Microstructural and Bactericidal Properties of TiAlN- (Ag,Cu) Coatings Deposited by DC Magnetron Sputtering for Medical Applications, *H Mejía, G Bejarano, **Aida Echavarría**,* Universidad de Antioquia, Colombia

Most of the surgical and odontological instrumentation is manufactured using martensitic stainless steel AISI 420 and 440 due to their acceptable biocompatibility, good hardenability, proper hardness and resistance to corrosion. However, the resistance to wear of this type of steels is relatively low and may be susceptible to contamination with bacteria representing a potential risk to the health of patients. This research work focused on the development of a coating system of titanium-aluminum-nitride doped with silver and copper nanoparticles TiAlN (Ag, Cu) to provide it with an appropriate bactericidal effect for possible biomedical applications.

TiAlN coatings doped with four different contents of Ag and Cu nanoparticles (11 at.% to 20 at.%) were deposited onto 420 steel by means of self- manufactured DC unbalanced magnetron sputtering using two composited targets of Ti/Al and Ag-Cu (both 50/50 at.% and 99.9% purity), which were facing each other at 180 degrees. The diffusion of the Ag-Cu nanoparticles to the sample surface, as well as their quantity, size, shape and distribution was controlled by an appropriate adjustment of the power applied to the Ag/Cu- target, the temperature and time of the deposition process, since the mechanical, tribological and bactericidal properties of the compound depend, among others, on these characteristics of the nanoparticles. The microstructure, surface topography, chemical and phase composition were analyzed by scanning and transmission electron microscopy (SEM/TEM), energy dispersive X-ray spectroscopy (EDX) and X-ray difraction. To evaluate the bactericidal effect of steel and coated samples in vitro inhibition and adhesion test were carried out selected Staphylococcus aureus and Escherichia coli, two of the major pathogen frequently found in surgery and dentistry rooms and associated with infections . The coating without doping presents a structure of columnar growth, which becomes densified with the increase of the Ag-Cu content and takes a glassy appearance accompanied by an increased size of the Ag-Cu particles and consequently also in the surface roughness. All the coated samples exhibited a higher bactericidal effect in comparison with the uncoated and with TiAlN coated steel, however the greater inhibition (100%) and less adherence to both bacteria was showed by the sample coated with 17% at Ag-Cu . Based on the results obtained, the developed

nanostructured coating system might be considered for potential application in surgical and dental instrumentation.

DP-ThP-26 Antibacterial Activity of Conductive Thin Films Deposited on Water Filter Paper, *D Mihut, A Afshar, S Hill, L Khang,* ***Nicholas Cordista,*** Mercer University, USA

There is a high interest to investigate nanomaterials that can work effectively against different types of bacteria and provide alternative substitution for chemical substances and antibiotics. In this research, conductive materials nanoparticles in the form of thin films were deposited on water filter papers by using direct current (DC) high vacuum magnetron sputtering technique. The effectiveness of the nanoparticles to remove bacteria from polluted water was tested during the experiment. The morphology of the coatings and their adherence to the water filter paper was examined using the Scanning Electron Microscopy and their chemical composition was investigated using the X-ray diffraction technique. A ll thin films showed good adhesion to water filter fibers and ensured a high area of exposure to contaminated water. The antibacterial effect of different conductive thin films was characterized by using the standardized membrane filtering technique for water and wastewater examination. The testing media (i.e. contaminated water) containing bacterial samples were collected from local wastewater basins. Water was tested for the bacterial content before and after the exposure to conductive thin films coated filters.

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