

Wednesday Afternoon, October 21, 2015

Thin Film

Room: 114 - Session TF+AS+BI-WeA

Thin Films for Biological and Biomedical Applications

Moderator: Christophe Vallee, LTM, Univ. Grenoble Alpes, CEA-LETI, Angel Yanguas-Gil, Argonne National Lab

2:20pm **TF+AS+BI-WeA1 On-chip Characterization of Engineered Nanomaterial Surface Properties by Real-time Affinity Monitoring.** C. Desmet, A. Valsesia, P. Colpo, European Commission, Joint Research Centre (JRC), **Francois Rossi**, European Commission, Joint Research Centre (JRC), Italy **INVITED**

The exhaustive characterization of the physico-chemical properties of engineered nanomaterials (ENMs) is essential to understand their mode of action and potential impact on health and environment. The development of characterization methods has been the object of important work in the past years, and has led to a better understanding on the ENM interaction with cellular systems and living organisms. One of the important surface properties of ENMs is the surface energy, for which there is no standard characterization technique established. Here, we demonstrate the feasibility of a characterization method based on a disposable microfluidic chip connected to an optical reader. The detection platform is based on the use of a micropatterned surface with tuned surface properties to bind ENMs selectively by hydrophobic forces and electrostatic interactions. The real-time absorption of ENMs on the differently functionalized micro domains is monitored by a microscope-coupled camera and gives information on the kinetics of adsorption, related to the affinity of the ENMs for the different surfaces as a function of their sizes and shapes. Interpretation of the results within the extended DLVO theory allows retrieving the surface energy characteristics of the ENMs surfaces. The key advantage of the device is the increase of the characterization throughput thanks to the all-in-one characterization process and the multiplexing that is able to replace the use of different methods and expensive equipment. In this way, the full characterization of ENMs could be expanded in all the areas covering nanomaterial-related applications.

4:20pm **TF+AS+BI-WeA7 Titanium-Niobium Thin Films Deposited by Magnetron Sputtering on AISI 316L Stainless Steel Substrate.** D. Gonzalez, T.C. Niemeyer, C.R.M. Afonso, **Pedro Nascente**, Federal University of Sao Carlos, Brazil

Metallic biomaterials such as AISI 316L stainless steel (SS), chromium-cobalt alloys, titanium and its alloys are commonly used in medical implants due to their interesting mechanical properties and thermal stability. However, 316L SS and Cr-Co alloys have much higher elastic modulus than bone, causing the loss after some years of implantation [1]. The elastic modulus of Ti-based alloys ranges from 55 to 110 GPa, being significantly lower than those for 316L SS (210 GPa) and Cr-Co alloys (240 GPa), making them more suitable for use in dental and orthopedic applications. Also Ti alloys present high strength, low density, high corrosion resistance, and good biocompatibility [1]. Pure Ti has two allotropic forms: hexagonal closest-packed (hcp), known as α phase, and body centered cubic (bcc), known as β phase, structures. Studies have shown that the addition of alloying β -stabilizing elements such as V, Mo, Nb, Zr, Mo, and Ta causes the decreasing of the modulus of elasticity of the β -Ti alloys without compromising the strength [1]. In this study, thin films of Ti-Nb alloys were deposited on AISI 316L stainless steel substrate by magnetron sputtering, and the structure, morphology, and composition of the films were analyzed by means of X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), and transmission electron microscopy (TEM). Thin films of three compositions were produced: $Ti_{85}Nb_{15}$ (Ti-26wt% Nb), $Ti_{80}Nb_{20}$ (Ti-33wt% Nb), and $Ti_{70}Nb_{30}$ (Ti-45wt% Nb). Structural characterization by XRD indicated that only the β phase was present in the thin films. XPS analysis showed a predominance of oxidized Ti and Nb on the film surfaces. TEM analyses were carried out in the following image modes: bright field (BF) images, selected area diffraction (SAD), scanning mode (STEM) BF and in annular dark field (ADF), and X-ray mapping using energy dispersive spectroscopy (EDS). For the $Ti_{80}Nb_{20}$ alloy film, TEM analysis showed columnar grains (~100 nm width) of -Ti phase, with a Nb-rich transition layer ranging from finer grains (in contact with SS substrate) to a coarser columnar grains. For the $Ti_{75}Nb_{25}$ alloy film, TEM analysis showed columnar grains (~50 nm width) of β -Ti phase, with a transition layer away from the SS substrate.

Acknowledgements: A.L. Gobbi, C.A. Silva, S.R. Araujo, and J. Bettini from the Brazilian Nanotechnology National Laboratory, for their assistance

in the growth and characterization of the thin films; and CNPq and CNPEM (Brazil), for support.

[1] M. Geetha *et al.*, Prog. Mater. Sci. 54 (2009) 397-425.

4:40pm **TF+AS+BI-WeA8 SAM-based Models of Cell Surfaces to Study the Interactions with Lectins and Bacterial Fimbriae.** **Andreas Terfort**, University of Frankfurt, Germany, **K. Lindhorst**, University of Kiel, Germany

Biologically important events such as cell-cell adhesion or infection typically start by directed and selective interactions with the highly glycosylated layer surrounding most eukaryotic cells. This layer, called the glycocalyx, consists of intricate glycopolymers, which – although in apparent disorder – clearly identify the cells. It is therefore of paramount interest to understand, which structural elements are important for the cell identification.

Self-assembled monolayers (SAM) can be used to simulate the chemical and sterical environment within such a glycocalyx. For this, glycosides are attached to oligoethyleneglycol (OEG) chains, which simulate the hydrogel matrix for the respective receptor. In this talk, we will focus on mannose-derivatives, which can be selectively recognized either by a lectin, concanavalin A, or by the adhesive fimbriae (tiny protein extrusions) of E. coli cells.

We would like to present different strategies for the construction of such SAMs [1,2] and discuss the advantages and disadvantages of these approaches. In extension of the mostly static systems, we will also present an approach to dynamically reorient the glycoside at the interface to determine the influence of steric factors on surface recognition [3].

References

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- [2] Grabosch, C.; Kind, M.; Gies, Y.; Schweighöfer, F.; Terfort, A.; Lindhorst, T. K. *Org. Biomol. Chem.* **11**, 4006-4015 (2013).
- [3] Weber, T.; Chandrasekaran, V.; Stamer, I.; Thygesen, M.B.; Terfort, A.; Lindhorst, T.K. *Angew. Chemie Int. Ed.* **53**, 14583–14586 (2014).

5:00pm **TF+AS+BI-WeA9 Improving the Long-Term Stability of Thin-Film Contact and Electrode Metallizations for Implantable Silicon Neural Interfaces.** **Brian Baker**, R. Caldwell, University of Utah, **H. Mandal**, Blackrock Microsystems, **R. Sharma**, **P. Tathireddy**, **L.W. Rieth**, University of Utah

The Utah Electrode Array (UEA) is a penetrating multi-electrode interface designed to be implanted and communicate directly with the brain and peripheral nerves through recording and stimulation. These devices are used for treating neural disorders and controlling prosthetics.

The UEA is micromachined out of single crystal silicon and uses a Pt/Ir/IrOx thin film metallization stack as an electrical interface on the electrode tip and a Pt/Ir/Pt stack on the backside contacts. Delamination of these thin metal layers has been observed during fabrication processes, soak testing, and in vivo operation, and is the critical failure mode examined in this study.

Db-FIB and Cross-sectional STEM analysis were used to identify Kirkendall voids as the root cause of the adhesion failures. This investigation showed that these voids form during the platinum silicide annealing process at the interface between the PtSi and the Ir layers.

Typical thicknesses of the UEA metallization are 200 nm/500 nm/520 nm Pt/Ir/IrOx, and 200 nm/200 nm/325 nm Pt/Ir/Pt. We report the results of replacing the 200 nm base layer with 1) a 25 nm Pt base layer or 2) a 50 nm co-sputtered PtSi base layer. These layers were subjected to typical UEA annealing conditions of 375 °C in forming gas for 45 minutes, followed by a 475 °C, 30 minute oxygen anneal.

Cross-sectional STEM elemental mapping of each film stack showed complete transformation of the platinum layer to PtSi, with a 40 nm layer of iridium silicide formed at the PtSi/Ir interface. In addition, a reduction in the nanogaps caused by Kirkendall voiding was demonstrated by STEM analysis in the two new film stacks.

Both the 25 nm Pt base layer stack and the 50 nm co-sputtered PtSi base layer stack demonstrate low-resistance Ohmic contacts and wire bondability after annealing. Further electrical characterization of these thinner base layer stacks used on tip metal demonstrated impedances of 5-10 kOhms and charge injection capacities of 1-2 mC/cm² for typical electrode tip surface areas. Cross-sectional STEM analysis of the reactively sputtered iridium oxide film reveals a three dimensional morphology whose nanostructures provide a large augmentation of electrode surface area and a corresponding

increase in charge injection capacity. In vitro stimulation and accelerated lifetime tests are ongoing and electrical measurements and thin film adhesion stability will be reported.

5:20pm **TF+AS+BI-WeA10 On-Surface Synthesis of Organic Nanostructures on Copper Surfaces**, *Q.T. Fan*, University of Science and Technology of China, *J.M. Gottfried*, Philipps-Universität Marburg, Germany, *Junfa Zhu*, University of Science and Technology of China

The on-surface synthesis of organic nanostructures known as bottom-up approach paves a new way for surface structuring, which plays a vital role in catalysis, sensor systems, or organic electronics. In this presentation, we will report our recent studies on the on-surface synthesis of 2D organic nanostructures on Cu(111) and Cu(110) surfaces using a specially designed bromo-terphenyl precursor, namely 4,4'-dibromo-*meta*-terphenyl (DMTP). The study was performed under ultra-high vacuum conditions using a combination of scanning tunneling microscopy (STM) and X-ray photoelectron spectroscopy (XPS). The results indicate that the two different surface structures of Cu drive the precursor molecule to form different nanostructures on the surface. We will show temperature-dependent organic nanostructures formed after DMTP adsorbed on Cu(111) and Cu(110). These organic nanostructures include large-area, defect-free 2D ordered nanostructures of intact DMTP on Cu(111), 1- or 2D polymeric zigzag organometallic intermediates formed on Cu(111) and Cu(110), and the macromolecular nanostructures including hexagonal close-packed arrays of cyclo-octadecaphenylene (hyperbenzene), oligophenylene nanowires formed through Ullmann reaction mechanism. *This work is supported by the National Natural Science Foundation of China (21173200, 21473178) and National Basic Research Program of China (2013CB834605)*

5:40pm **TF+AS+BI-WeA11 Carbon Nanotube-Templated, Porous Films for Thermal Isolation**, *J.M. Lund*, *D.B. Syme*, *R. Vanfleet*, *R.C. Davis*, *B.D. Jensen*, *Brian Iverson*, Brigham Young University

Sensor usage has increased dramatically in detection applications due to miniaturization of components through micro and nanofabrication. These fabrication methods have also greatly increased production rates, as several sensors can be constructed in parallel. Reduction in feature size of sensors has resulted in an increase in sensor component proximity, making thermal diffusion or cross talk detrimental to proper function. This work investigates the use of carbon nanotube-templated manufacturing (CNT-M) to create thin-film, isolation layers for use in thermal sensors. CNT-M is a process wherein carbon nanotubes are used as a scaffold and coated with insulating materials (e.g. SiO₂) to create porous insulating films. Carbon nanotubes are removed in a post-deposition, burn out process rendering a porous matrix of insulating material. Thin-films are characterized using scanning electron microscopy, nanoindentation and the 3-omega method to determine mechanical and thermal properties. Thermal conductivity on the order of air has been observed while still maintaining a rigid structure that is compatible with subsequent MEMS processing.

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