## Thin Films
## Room: Makai - Session TF-WeE

### Thin Film Synthesis and Characterization III
**Moderator:** HugoR. Navarro Contreras, Coordinación para la Innovación y la Aplicación de la Ciencia y la Tecnología (CIACyT-UASLP)

5:40pm **TF-WeE1 Static and Dynamic Magnetic Properties in FeCoN Thin Films Deposited Under Various Deposition Powers**, ***Yuping Wu***, National university of singapore, *Z.H. Yang*, National universisity of Singapore, *F.S. Ma, B.Y. Zong*, National University of Singapore

In this work, we deposited FeCoN thin films by reactive magnetron sputtering. The applied deposition power was changed from 150 W to 1000 W. The dynamic magnetic properties were measured with the shorted microstrip transmission-line perturbation method, which was developed in our lab. The damping coefficient was estimated by analyzing the measured permeability spectra based on Landau-Lifshitz-Gilbert (LLG) equation. Both the static magnetic properties and the damping coefficient in the magnetization dynamics can be conveniently and effectively tuned by varying the sputtering deposition power, which results in controllable and modified dynamic magnetic properties. The physical origin of the influences is related to the changed deposition rate, which is a critical factor determining the microstructure of films.

6:00pm **TF-WeE2 Substrate Heating during Reactive Magnetron Sputtering**, ***Julio Cruz***, *J. Restrepo, S. Muhl*, IIM-UNAM, Mexico

Substrate heating by the plasma during magnetron sputtering is known to occur, however, there have been very few detailed studies of this process which involves a combination of bombardment by ions, excited and neutral species and UV radiation incident on the substrate. We have studied the heating of the substrate during DC magnetron sputtering of a 4” titanium target as a function of the experimental conditions; plasma power, Ar gas pressure, floating, grounded and biased substrates. We have also studied the substrate heating during reactive sputtering mode by using a gas mixture of argon with nitrogen. On the other hand, it is known that the crystalline orientation of titanium nitride depends on the sputtering conditions. Here we report the effect of the plasma substrate heating, as a consequence of the plasma conditions, on the morphology and the crystalline structure of titanium nitride. The properties of the films were analyzed using SEM and X-ray Diffraction and the film thickness was measured using a stylus profilometer. The measurements of the non-reactive sputtering showed that the substrate temperature could reach temperatures higher than 200ºC with a plasma power of 200W and showed a non uniform temperature distribution over the substrate, with the highest temperature in front at the racetrack and the lowest temperatures in front of the edge of the target. Finally by using a Fluke Ti300 camera we show the temperature change in the substrate with time for both reactive and non-reactive processes.

6:20pm **TF-WeE3 The Hollow Cathode Discharges; How They Have Been used to Produce Thin Films and the Novel Toroidal Planar Hollow Cathode System**, ***Stephen Muhl***, Instituto de Investigaciones en Materiales, Universidad Nacional Autónoma de México, Mexico, *A. Perez*, Universidad Nacional Autónoma de México, Mexico, *A. Tenorio*, Instituto de Investigaciones en Materiales, Universidad Nacional Autónoma de México, Mexico, *E. Camps*, Instituto Nacional de Investigaciones Nucleares, Mexico

In 1916 F. Paschen first report the hollow cathode discharge he demonstrated that the system was capable of producing a high electron flux with relatively low ion and neutral temperatures. Approximately 40 years later Lidsky showed that hollow cathode arc discharges were one of the best plasmas sources available at that time. The term "hollow cathode discharges" has been used in reference to almost any discharge in a cathode with a cavity-like geometry, such that the plasma was enclosed by the walls which are at the cathode potential. Just as trapping of electrons in a magnetron cathode by the magnetic field results in an increase in the plasma density, in the hollow cathode the geometry of the cathode also produces a high plasma density. In general, three types of discharge can be established in a hollow cathode; at low power and / or at relatively low gas pressures the plasma is a “conventional” discharge characterized by low currents and medium to high voltages (a Discharge in a Hollow Cathode or D-HC). However, even this simple plasma has a higher density than a normal planar parallel electrode system because the hollow geometry reduces the loss of electrons. If the combination of gas pressure, applied power and hollow cathode diameter is correct, the negative glow of the plasma almost completely occupies the interior volume of the cathode. Under this condition the plasma current can, for the same voltage, be 100 to 1000 times the values for the “simple” D-HC discharge and the plasma density is very large (this is the Hollow Cathode Discharge or HCD). If the temperature of cathode can increase so that Thermal-Field electron emission becomes an important additional source of electrons the discharge can change into a dispersed arc (this is the Hollow Cathode Arc or HCA). The accepted explanation for the HCD phenomenon involves the existence of high energy “pendulum” electrons reflected from sheath to sheath on either side of the inside of the cathode; the long trajectory of these electron is thought to produce an increased number of secondary electrons, which produces the high plasma density and plasma current. We will discuss some of the problems associated with the well-accepted model and we will propose a new explanation which has some important implications.

Finally, we will describe how hollow cathodes can be used to deposit thin films and nanostructured coatings, including the use of our novel toroidal planar hollow cathode to produce bismuth thin films, nanoparticles and bismuth/a-C:H nanocomposites.

6:40pm **TF-WeE4 Depth Profiling Organic Thin Films with Argon Cluster Beam**, ***Jean-Jacques Pireaux***, *Nittler, Noël, Houssiau*, University of Namur, Belgium

Many modern devices are based on multilayers of different materials, combined to reach a specific application. Monoatomic ion beam depth profiling did – and still do- immensely contribute to the learning of such multilayered structures containing metallic and oxide films; but one couldn’t study (in ToF-SIMS, AES or XPS) an organic/other material interface, or an organic multilayered structure because of the damages induced by the ion beam. For the profiling of organic layers, the development and commercial availability of sputtering sources based on poly-atomic ions (SF6, C60, coronene) came as a significant and promising evolution; the still more recent Argon cluster-ion source appears now as a revolution for a true quantitative depth analysis of organic films. Numerous applications of gas cluster ion beams have been presented at international conferences; publications study the influence of cluster size, of cluster energy on sputter rates on series of different polymers, and on depth resolution in sputter depth profiles [1]. The present work aims to study by XPS the sputter yield, and the depth resolution in samples consisting of multilayered amino-acid films while profiling with different Ar cluster ions (Thermo Fisher Scientific Escalab 250Xi spectrometer, MAGSIS source). The materials were chosen for their ease to be reproducibly deposited by sequential thermal evaporation in high vacuum under quartz-crystal balance monitoring ; the method revealed successful for the study of delta layers in dual beam Cs+/Bi3+ profiling in ToF-SIMS [2]. Phenylalanine (Phe) and tyrosine (Tyr) were used for this study. Although both amino acids differ only by an OH- group, their characteristic chemical fingerprints could be differentiated throughout the whole depth profile. Both materials surprisingly present a very different sputter yield: the erosion yield of Phe is larger (almost twice the value, depending on the Ar cluster size and energy) than for Tyr – a trend that is completely reversed in ToF-SIMS using Cs+[2,3] ; this strongly suggests different sputtering mechanisms, that will be reviewed during the presentation. Depth resolution at the interface between two layers is found to be better when sputtering from the high sputter yield material to the low one. It is worth to note in addition that, as suggested by Laser scanning confocal microscopy, the gas cluster ion beam profiling does not increase significantly the sample surface roughness.

[1] P.J. Cumpson et al, J.V.S.T. A31 (2013)020605 ; SIA 45 (2013)601

[2] C. Noël, University of Namur, Master Thesis, 2013

[3]C. Noël, L. Houssiau. SIMS Europe 2014 Conf., Münster.

7:00pm **TF-WeE5 Adaptive Functional Surfaces: Ni-Ti(-Cu) Shape Memory Thin Films**, *M. Callisti*, University of Southampton, ***Tomas Polcar***, University of Southampton, UK

Shape memory alloys could be effectively used as thin films acting either as active/passive actuators or superelastic interlayers. In this study s series of NiTiCu coatings with increasing was fabricated by magnetron sputtering with a thickness of 2 µm. In order to obtain superelastic properties, the films were isothermally annealed for 1 hour at 500°C in a high vacuum environment. Subsequently the superelastic layers were coated by magnetron sputtering with a functional tribological coating (DLC-W and self-lubricant WSC film).

The chemical composition of every single layer was measured by Energy-dispersive X-ray spectroscopy (EDS), while the structure was evaluated by grazing-incidence X-ray diffraction (GIXRD) and transmission electron microscopy (TEM). The mechanical properties of the single layers as well as those of the bilayers were measured by nanoindentation. Finally, the tribological behaviour of the bilayers and of the single layers were characterised by pin-on-disc.

We will demonstrate that superelestic interlayer could significantly increase coating adhesion. Pure NiTi interlayers underwent progressive irreversible martensitic transformation during the sliding and lost superelasticity; on the other hand, transformation of NiTiCu film was fully reversible. We will show that we can control compressive and shear stress in functional coating during sliding by selection of an optimum superelastic interlayer.

7:40pm **TF-WeE7 Metal Oxide thin Films for Medical Applications**, ***Sandra Rodil***, *A. Almaguer-Flores*, Universidad Nacional Autonoma de Mexico, *P. Silva-Bermudez*, Instituto Nacional de Rehabilitación **INVITED**

Nowadays, it is generally accepted that surfaces are critically important to nearly all aspects of biomedical technology since most of the biological reactions occur at the interfaces. In vitro studies have demonstrated that the surface properties are directly related to important biological events, such as protein adsorption, bacterial attachment and cell growth. In the case of medical implants, during the last years the research has evolved from the improvement of bulk properties and design of the implants to the development of a variety of bio-functional surface modifications, such as surface topography at the nanoscale, adhesion of growth factors or coating deposition. There is an extensive research to find methods of designing tailored surfaces, which might act as stimuli to guide specific cell responses according to the specific medical application.

This presentation explores one of the many surfaces modifications that have been proposed; plasma deposited coatings. The talk is then divided into two parts. Firstly, a short review about the specific needs to improve odontological implants. Secondly, the results of the physical, chemical and biological characterization of metal oxide thin films ($TiO_2$, $ZrO_2$, $Nb_2O_5$ and $Ta_2O_5$) deposited by magnetron sputtering are presented. The factors considered of biological relevance in order to understand the surface interaction and that will be presented include: a) protein adsorption on the metal oxides studied by Ellipsometric Spectroscopy, Atomic Force Microscopy and X-ray photoelectron Spectroscopy, b) Corrosion behaviour of the oxides immersed in simulated biological solutions, c) Bacterial attachment and d) Cell adhesion, proliferation and differentiation.

8:20pm **TF-WeE9 Synthesis and Characterization of $TiO_2$ and $Bi_2O_3$ Thin Films for Photocatalytic Applications**, ***JuanCarlos Medina***, *M. Bizarro*, IIM-UNAM, Mexico, *M. Giorcelli*, *A. Tagliaferro*, Politecnico di Torino, Italy, *P. Silva-Bermudez*, Instituto Nacional de Rehabilitación, Mexico, *S.E. Rodil*, IIM-UNAM, Mexico

The magnetron sputtering technique was used to obtain bismuth oxide ($Bi_2O_3$) and titania ($TiO_2$) thin films. The films were characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM), X-ray Photoelectron Spectroscopy (XPS) and contact angle measurements. The results indicated that the $Bi_2O_3$ thin films presented the cubic delta phase and the $TiO_2$ thin films showed a combination of rutile-anatase. The photocatalytic activity for both films was evaluated testing the degradation of methyl orange dye ($C_{14}H_4N_3SO_3Na$) under ultraviolet light and a solution of pH 3.5. The dye degradation and the kinetic of the reaction were estimated using the variation of the corresponding absorption band as a function of the irradiation time. The results pointed out that the photocatalytic activity was always larger for $Bi_2O_3$ films than for $TiO_2$ films. Moreover the activity was also larger for $Bi_2O_3$ in comparison to equivalent mass-amounts of $TiO_2$ powders (P25) under the same experimental conditions. However XPS tests showed that after a degradation cycle bismuth oxide transforms to Bismuth Oxychloride (BiOCl) due to the interaction with Cl ions from the HCl solution used to decrease the pH, and as a consequence the photocatalytic effect was reduced. After calculating and comparing the reaction kinetic constants for both oxide films, it is concluded that under UV light, the $Bi_2O_3$ reaction rate is three-fold larger than $TiO_2$ reaction rate constant. These results suggest that the $Bi_2O_3$ films are a new promising photocatalytic material for water treatment application. Moreover, studies of photoinduced changes in the wettability demonstrated a similar behavior between $Bi_2O_3$ and $TiO_2$ thin films.

8:40pm **TF-WeE10 Preparation of Ce-doped Hafnium Oxide Thin Films by Sol-Gel Method**, ***Luis Garcia-Cerda***, *A. Puente*, Research Center on Applied Chemistry, Mexico, *S. Galvez-Barboza*, *L.A. Gonzalez*, Center for Research and Advanced Studies of the National Polytechnic Institute

Mono and multilayer Ce-doped hafnium oxide thin films were deposited on silicon wafers and quartz by spin-coating technique using a solution prepared by solgel with hafnium chloride, cerium nitrate, citric acid and ethylene glycol as starting materials. Ce-doped $HfO_2$ thin films with 1, 3 and 5 layers were annealed in air for 2 h at 500, 700 and 900 °C. The thin films were then characterized for structural, surface morphological and optical properties by means of X-ray diffraction (XRD), Atomic force microscopy (AFM), scanning electron microscopy (SEM) and optical absorption. X-ray diffraction analysis indicated that the cubic $HfO_2$ films could be obtained by annealing at 500 °C. AFM and SEM images revealed well defined particles which are highly influenced by annealing temperatures.

# Authors Index

## Bold page numbers indicate the presenter