

## Surface Engineering - Applied Research and Industrial Applications

### Room Palm 1-2 - Session IA1-TuM

#### Advances in Application Driven Research and Hybrid Systems, Processes and Coatings

**Moderators:** Vikram Bedekar, Timken Company, USA, Hana Barankova, Uppsala University, Sweden

**8:00am IA1-TuM-1 Advancing Correlative Microscopy: In-Situ Integration of AFM-SEM-EDS for Multi-Modal Analysis, Kerim T. Arat [karat@qdusa.com], William K. Neils, Stefano Spagna, Quantum Design Inc., USA**

There is a growing interest in in-situ correlation microscopy, which brings the complementary strengths of different imaging modalities without the inherent complications of sample transfer. These approaches ensure high confidence in correlation accuracy and eliminate the risk of sample contamination and alteration during the sample transfer.

We have developed a correlative microscopy platform based on AFM-SEM [1]. These techniques can map the surface in high resolution, and the trunnion stage, with up to 80° tilt capability, allows monitoring of tip quality and tip-sample interaction [2]. However, these methods fall short in identifying the elemental composition of the sample.

To address this issue, we have extended the capabilities of the correlative platform with an energy-dispersive X-ray spectrometer (EDS). The spectrometer is based on a state-of-the-art silicon drift detector [3], which provides high energy resolution. Its graphene window offers improved transmission performance, especially at the lower energy range, allowing elemental detection down to carbon. The elemental identification algorithm uses a background subtraction method to remove non-characteristic signals and compares the resulting spectra to reference datasets based on the NIST database for standardless quantification [4]. Both hardware and software integration allow the correlation of elemental information with the other imaging modalities that the tool can provide (see the supplementary document), where one can superimpose topography and elemental information.

Integration of the X-ray detector adds a comprehensive analysis capability to AFM-SEM techniques applicable to a diverse range of fields such as materials science, semiconductors and biosciences. With this option, researchers can obtain an in-situ correlation of high-resolution, localized elemental information with high-resolution lateral and vertical topographical information.

[1] A. Alipour et al., *Microscopy Today* 31 (2023), p. 17-22. doi: 10.1093/mictod/qaad083

[2] "FusionScope by Quantum Design," Open a world of easy-to-use correlative microscopy, 2022. <https://fusionscope.com/> (accessed Apr. 27, 2023).

[3] D. E. Newbury and N. W. M. Ritchie, *Journal of Materials Science* 50 (2015), p. 493-518. doi: 10.1007/s10853-014-8685-2

[4] D. E. Newbury and N. W. M. Ritchie, *Scanning Microscopies* 9236 (2014), p. 9236OH. doi: 10.1117/12.2065842

**8:20am IA1-TuM-2 Non-stick Hydrophobic and Superhydrophilic Metallic Coatings: Their PVD Fabrications and Applications, Jinn P. Chu [jpchu@mail.ntust.edu.tw], National Taiwan University of Science and Technology, Taiwan; Sea-Fue Wang, National Taipei University of Technology, Taiwan**

The presentation will begin with an introduction to a non-stick, low-friction hydrophobic metallic glass coating and its applications. This amorphous coating, fabricated using PVD techniques, has been successfully applied in various fields, including medical devices. For the superhydrophilic coating, a 316 stainless steel layer is sputtered onto the substrate, resulting in a water contact angle of approximately 10 degrees on the coated surface. This coating also demonstrates antifouling and underwater superoleophobic properties, which are advantageous for use in separation membranes for oil/water emulsions. Furthermore, it has proven highly effective in enhancing electrochemical responses in electrodes used as electrochemical sensors.

**8:40am IA1-TuM-3 Novel CO<sub>2</sub> Laser Direct-Write Energy-Efficient Process for Functional Oxide-Carbon Composite Coatings and Their Energy Applications, Swati Jadhav [swatijadhav1602@gmail.com], Pratibha Jadhav, Ishwari Belle, Anuradha Ambalkar, Supriya Kadam, Satishchandra Ogale, Indian Institute of Science Education and Research, Pune, India**

The performance and operational longevity of several energy devices such as Batteries, Fuel Cells, and Electrolysers critically depend on the chemical and physical functionality, micro (nano) porosity, and robustness of the specialized coatings on metal current collectors. A large number of methods are available to obtain such coatings, but these are chemically complex and generally energy intensive. Moreover, several of these methods do not allow concurrent control of porosity and surface chemistry that drive the overall process efficiency, especially in surface catalytic phenomena. In this work we show that CO<sub>2</sub> laser (wavelength 10.6 mm) induced surface processing allows an excellent parametric control on achieving the desired results and that too with a dramatic reduction of energy inputs vis a vis the conventional methods. The key control parameters include laser power density, scanning speed, and coating constitution/thickness. The laser surface processing method is intrinsically direct-write type in scanning mode and as such allows in-plane micro-gradient patterning. We will show and discuss the results of several interesting cases wherein the effectiveness of this approach is demonstrated for composite oxide-carbon coatings obtained by using biomass (or biomass-derived) precursors and functional binary oxide systems. The biomass precursors include furfural alcohol, lemon grass, sugarcane bagasse while the oxide systems include NiO, CuO, TiO<sub>2</sub>. Use of urea and thiourea in the composite is also examined to achieve doping of nitrogen and Sulfur in carbon to enhance its conductivity. The resulting engineered coatings are studied for energy applications such as Anode-free (AF) Li and Na ion batteries, and Electrocatalysis for water splitting applications (oxygen evolution reaction, OER and Hydrogen evolution reaction (HER)). For AF batteries the laser processed coatings render low Li/Na nucleation overpotential, good columbic efficiency and cycling stability of up to 800 cycles limited by Li/Na inventory. In case of water splitting application as well superior properties are realized in terms of overpotential and stability.

**9:00am IA1-TuM-4 PVD Coatings for the Hydrogen economy - Applications, Testing and Production, Herbert Gabriel [h.gabriel@pvtvacuum.de], PVT Plasma und Vakuum Technik GmbH, Germany**

**INVITED**

Green hydrogen could be the fuel of the future. Generated by electrolysers powered by photovoltaics and used in fuel cells could be part of the solution to the human mankind's problems with the climate change.

The harsh environments in electrolysers and fuel cells require components to be coated for corrosion resistance, electrical conductivity and other related properties..

Most of the components are made of stainless steel or titanium, but still need for their performance and long lifetimes up to 100.000 hours coatings with high performance properties.

Depending on the application, whether PEMWE, PEMFC, AEM, SOFCs, SOECs or others, thin coatings made of materials such as C, Ti, Cr, Nb, Au, Pt, Ir, MCO, Al<sub>2</sub>O<sub>3</sub>..... are deposited in the nanometer to a couple of micron range.

Preferred coating processes are magnetron sputtering, respectively HIPIMS, high power impulse magnetron sputtering to deposit highly adherent and dense coatings.

Most components of fuel cells and electrolysers to be coated are thin 2-dimensional structures in high quantity. For this reason high productive so-called in-line systems with vertical orientation are the preferred coating systems for double-sided deposition.

Apart from a number of other QC – tests, adhesion, corrosion and ICR (interface contact resistance) prior and after corrosion testing are essential properties to continually be tested and monitored.

**9:40am IA1-TuM-6 Improving Doping Concentration for Shallow N<sup>+</sup>/P Substrate Germanium Pn Junction with Plasma-Immersion Ion Implantation Process, Bo-Syun Syu [brian2000713@gmail.com], National Tsing Hua University, Taiwan; Dun-Bao Ruan, Fuzhou University, China; Kuei-Shu Chang-Liao, Po-Chun Wu, National Tsing Hua University, Taiwan**

In recent years, due to the development of device technology nodes, the requirements for the mobility of channel materials have become increasingly demanding. Considering the compatibility with existing process

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equipment, the most suitable and high-mobility material is germanium (Ge) based channel material. However, unlike traditional silicon, Ge has a smaller band-gap, lower solubility for dopant impurities, a lower melting point, and other issues, making it very prone to phenomena such as Fermi-level pinning, small on/off junction current ratio, high reverse junction current, and poor reliability. Therefore, exploring an effective ion implantation method, which may achieve shallow high-density doping PN junction, is particularly important. This work successfully utilizes plasma immersion ion implantation technology to fabricate high-performance shallow N<sup>+</sup>/P junctions. It is believed that this work can provide an important technical path exploration for the application of Ge-based devices.

**10:00am IA1-TuM-7 Molecular Layer Deposition – Versatile Tool for High Performance CNT-Polymer Composites, Roie Yerushalmi [roie.yerushalmi@mail.huji.ac.il]**, Edmond J Safra Campus, Givat Ram, Israel

Composite materials, particularly those reinforced with carbon nanotubes (CNTs), are gaining significant attention due to their remarkable strength-to-weight ratio compared to traditional materials. Additionally, these composites exhibit exceptional thermal and electrical properties. However, a primary challenge hindering the widespread application of CNT-reinforced composites arises from the very properties that make them desirable—the CNT Csp<sup>2</sup>-Csp<sup>2</sup> network. To enhance the compatibility of CNTs with polymer matrices, modifications to the surface properties of CNTs are necessary. Unfortunately, these modifications often compromise the integrity of the CNT network, creating a significant barrier to progress. To address this challenge, we have developed an innovative vapor-phase approach that utilizes combined Atomic & Molecular Layer Deposition (M/ALD). This method allows for molecular-level precision in tailoring CNT interfaces while minimizing negative impacts on the CNT network. By integrating surface engineering with M/ALD vapor-phase chemistry, we achieve a balanced interaction between non-covalent and covalent bonds with the polymer matrix in a single streamlined process.

This approach facilitates the fine-tuning of physical properties, enabling the design of high-performance CNT-reinforced polymer composites. The combined M/ALD methodology is broadly applicable for engineering the CNT-polymer interphase, providing precise control over surface interactions. Ultimately, this paves the way for the systematic development of high CNT loading composites and other nano-reinforced systems, exhibiting enhanced strength, toughness, and a range of additional desirable properties typical of nanomaterials composites.

**10:20am IA1-TuM-8 Energy Bandgap Engineering for Gate-All-Around Poly-Ge Charge Trapping Flash Memory by Using Stacking Tunneling Layer, Kuei-Shu Chang-Liao [kschang@ess.nthu.edu.tw]**, National Tsing Hua University, Taiwan; *Dun-Bao Ruan*, Fuzhou University, China; *Chu-Chun Su*, National Tsing Hua University, Taiwan

A high-performance junction-less charge-trapping flash memory device based on gate-all-around structure with a poly-Ge channel was successfully fabricated in this work. By leveraging the high carrier mobility of Ge, the use of a low-temperature poly-Ge channel enhances the operational speed of the flash memory device. However, under stringent thermal budget limitation, the reliability of the Poly-Ge flash device may degrade without enough thermal processing. This degradation is likely attributed to the narrow energy bandgap of Ge material and Ge out-diffusion phenomena. By integrating post plasma process and stacked tunneling layer engineering, the memory device incorporating an aluminum oxynitride (AlON) tunneling layer demonstrates significant improvements, including high programming/erasing speeds, excellent endurance cycles, and long data retention time. These enhancements can be primarily attributed to the superior thermal stability and interface quality of AlON, which may mitigate interface defects and improve robustness during thermal cycles. Besides, compared to traditional silicon nitride charge-trapping layers, AlON exhibits shallower trap energy levels. This property enables faster charge injection/ejection during programming/erasing operations without compromising data retention or endurance performance.

**10:40am IA1-TuM-9 Analysis of White Strip Defects in the Galvannealed Coating Surface of Hot -Dip Galvannealed DP Steel, Guang Chen [CHENGUANG@BAOSTEEL.COM]**, Baoshan Iron & Steel Co., Ltd., Shanghai, China

The microstructure and characteristics of two types of white stripe defects in hot -dip galvannealed DP steel were studied, and the relevant processes were analyzed and discussed. The results show that both types of white stripe defects originate from the surface defects of the substrate before

galvanizing, and one originates from iron scale of finishing roller, with its microstructure characterized by characteristic elements Mo, V, Cr, Ni, etc; Another type originates from microcracks in the casting slab, whose microscopic characteristics are mainly secondary oxides. Based on the above analysis, the paper clarifies the impact of hot rolling and slab casting processes on white strip defects.

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