Topical Symposium on Sustainable Surface Engineering
Room Town & Country B - Session TS1-2-MoA

Coatings for Batteries and Hydrogen Applications II

Moderators: Chen-Hao Wang, National Taiwan University of Science and Technology, Taiwan , **Martin Welters**, KCS Europe GmbH, Germany, **Fan-Bean Wu**, National United University, Taiwan

1:40pm TS1-2-MoA-1 Protective Coatings in Hydrogen Technologies: Degradation Mechanisms and Key Parameters for Standardized Testing, *Verena Lukassek [v.lukassek@zbt.de]*, The Hydrogen and Fuel Cell Center, Germany **INVITED**

Proton exchange membrane fuel cells (PEMFC) and proton exchange membrane water electrolysers (PEMWE) operate in harsh thermal, mechanical, and electrochemical environments. Their components, particularly metallic bipolar plates (BPP) and porous transport layers (PTL), are exposed to corrosive media, high electrical potentials, and cyclic thermal and mechanical stresses. Protective coatings are therefore essential to ensure the long-term stability, efficiency, and operational safety of these systems. In recent years, various coating strategies have been developed to improve corrosion resistance and electrical conductivity, thereby extending the service life of critical components. However, the lack of standardized testing protocols for evaluating coatings under relevant operating conditions still limits the comparability of results and the transfer of laboratory data to industrial practice.

This work provides an overview of the current state of protective coatings for hydrogen applications, focusing on the environmental and operational stresses they are exposed to and the associated degradation phenomena. Among them, precious metal-free coatings such as nitrides, carbides, and carbon-based films have been extensively investigated in recent literature for application in both fuel cell and electrolysis environments.

Experimental investigations include electrochemical testing as well as structural and chemical analyses to evaluate microstructure, interfacial adhesion, electrochemical stability, and degradation behaviour under relevant operating conditions. The results reveal that coating performance is strongly influenced by the adhesion between substrate and coating, the homogeneity of the microstructure, and the stability against oxidation. Moreover, the study identifies key parameters—such as coating architecture, surface pretreatment, and testing atmosphere—that critically affect corrosion resistance and contact resistance measurements. Understanding these interdependencies provides valuable input for the future development of harmonized testing protocols.

In conclusion, despite significant progress in material development and characterization, reproducible benchmarking remains challenging without harmonized testing procedures. The establishment of unified electrochemical test standards—analogous to existing PEMFC guidelines—combined with scalable coating processes and accelerated lifetime testing is crucial for reliable lifetime prediction and the advancement of next-generation hydrogen technologies.

2:20pm TS1-2-MoA-3 Parameter-Sensitive Ex-Situ Corrosion Testing of Coated and Uncoated PEMWE Bipolar Plates, *Julian Kapp [j.kapp@zbt.de]*, ZBT GmbH, Germany

Proton exchange membrane water electrolysis (PEMWE) is pivotal for future green-hydrogen production, yet bipolar plates (BPPs) remain major cost drivers in stack manufacturing. To accelerate validation of substrates and coatings efficiently, short, and representative ex-situ corrosion tests are needed. While targets and protocols exist for fuel cells, there is no clear, PEMWE-specific guidance on how to perform such tests.

Reliable lifetime extrapolation requires accurate, surface-proximal characterization of PEMWE operating conditions near the bipolar plate (temperature, applied potential history, local acidity, and halide/other additives) and a mechanistic understanding of how these factors govern corrosion in coated and uncoated BPP materials. These insights enable the definition of suitable short-term ex-situ test conditions.

To elucidate and decouple parameter effects, we run electrochemical corrosion tests—potentiodynamic scans, potentiostatic holds, and electrochemical impedance spectroscopy (EIS)—on coated and uncoated BPP materials while systematically varying electrolyte composition, temperature, applied potential, and setup factors (active area, gas bubbling). Pre- and post-exposure, we quantify electrical, chemical, and morphological changes via interfacial contact resistance (ICR), microscopy/SEM-EDX, X-ray diffractometer (XRD), contact-angle goniometry, and X-ray fluorescence (XRF), complemented by electrolyte analysis to track metal dissolution and impurities.

By decoupling and quantifying the effects of the studied parameters on dissolution, passivation stability, and coating integrity, we identify critical cross-effects and sensitivities. The results inform practical recommendations for test selection and configuration tailored to specific objectives (screening, mechanistic study, or qualification) and PEMWE environments (anode vs. cathode BPP, substrate/coating type, operating window).

This work establishes a parameter-sensitive framework for harmonized ex-situ corrosion testing of PEMWE bipolar plates, fostering convergence toward standardized testing protocols and improving cross-laboratory comparability. The resulting methodology accelerates the identification of cost-effective materials and supports more reliable lifetime predictions for PEMWE stack design.

2:40pm TS1-2-MoA-4 Advances in *Operando* and *In Situ* Cross-sectional Characterization of Thin Films for Battery and Hydrogen Applications, *Juraj Todt [juraj.todt@unileoben.ac.at]*, Montanuniversität Leoben, Austria; *Francois Lienard, Manfred Burghammer,* ESRF, Grenoble, France; *Tobias Huber,* Huber Scientific, Austria; *Henrik Bratlie, Daniel Rettenwander,* Norwegian University of Science and Technology (NTNU), Norway; *Rostislav Daniel, Markus Alfreider, Michael Tkadletz, Jozef Keckes,* Montanuniversität Leoben, Austria **INVITED**

As we are facing the worldwide need to reduce the carbon footprint of modern technologies, materials relevant to the fields of sustainable energy storage and transport have received widespread attention. A great deal of research effort has been devoted to their study and optimization, where current techniques often measure either bulk or aggregate properties such as specific capacities or power densities. However, to fully understand all the intricate and often localized mechanisms at play, a more detailed look is necessary. Methods such as impedance spectroscopy are able to capture the crucial role of interfaces with the help of some modeling, but direct observations for cross-sectional behavior are usually lacking.

This is where cross-sectional X-ray nano-diffraction (CSnanoXRD) could provide a very effective tool, as it offers the capability of comprehensive insights encompassing phase composition, preferred crystallite sizes and orientations, accumulated lattice defects and crucially also information on internal strains and stresses. This contribution aims to present our recent methodological advances in applying the CSnanoXRD concept to battery and hydrogen applications, showcasing the dedicated experimental set-ups that are necessary to make this possible.

For the study of zero-excess solid state batteries, a test platform capable to apply stacking pressures in the range of several 100 MPa on X-ray-transparent cross-sectional lamellae has been developed within the Horizon Europe project *OPERA*. First results on the role of anode interlayers in Li and Na deposition at the interface between solid electrolytes and current collectors will be shown. A focus will lie on the many technological challenges that had to be overcome until a fully working tool has been achieved, which is now available to the scientific community.

A further novel test platform will be presented, aimed at the study of hydrogen interaction with surface layers. It is based on the application of femtosecond laser ablation for sample patterning and two-photon lithography to create the appropriate microfluidic structures necessary for electrolytic H-charging of thin films at a CSnanoXRD experimental station. The behavior of various metallic layers including Ni and V, as well as Pd/Nb and Ti-V will be examined in detail, showing the formation of hydride phases and decoupling lattice expansion due to H uptake from the formation of residual stress due to mechanical constraints from the substrate and underlying material. Future possibilities for this approach will comprise the study of the role of interfaces and multilayered H-barrier structures.

3:20pm TS1-2-MoA-6 Atomic Layer Deposition for Enhancing Durability of Fuel Cell Catalysts, *Shao-Chuan Chang, Chih-Liang Wang [wangcl@mx.nthu.edu.tw]*, Department of Materials Science and Engineering, National Tsing Hua University, Taiwan

Fuel cells are promising clean energy devices for hydrogen conversion, yet their commercialization is hindered by the high cost and limited durability of catalysts. To address these challenges, developing low-cost alternatives with enhanced stability is essential. Surface coating has emerged as an effective strategy to improve catalyst durability by suppressing metal

particle agglomeration, dissolution, and carbon corrosion. In this study, Ag/C catalysts were prepared via a microwave-assisted method and subsequently coated with zinc oxide (ZnO) using atomic layer deposition (ALD) to evaluate their stability for anion exchange membrane fuel cell (AEMFC) applications. Ag/C catalysts obtained by conventional impregnation were also investigated for comparison. Structural (XRD, FTIR, TEM) and electrochemical (CV, ECSA) analyses demonstrated that the ALD ZnO-coated Ag/C prepared via the microwave-assisted route possessed markedly enhanced durability relative to the impregnated counterpart. Single-cell performance tests further confirmed the superior activity of the microwave-assisted ZnO-coated Ag/C catalyst, which achieved a higher peak power density than the impregnated sample. These results confirm that an optimally engineered ALD ZnO coating effectively mitigates Ag particle aggregation and dissolution, thereby stabilizing the catalyst structure and enhancing overall AEMFC performance.

4:00pm TS1-2-MoA-8 Electrocatalytic Performance Analysis of FeNixMoWCu High Entropy Alloy Thin Films: Effects of Ni Content, *Yen-Chin Lai [lai.juju0120@gmail.com], Po-Chun Chen,* National Taipei University of Technology, Taiwan*; Bih-Show Lou,* Chang Gung University, Taiwan*; Jyh-Wei Lee,* Ming Chi University of Technology, Taiwan

Hydrogen energy has attracted significant attention due to its cleanliness, non-polluting and carbon-free characteristics [1]. Among various hydrogen production methods, water splitting is considered one of the most promising. However, both the hydrogen evolution reaction (HER) and oxygen evolution reaction (OER) require catalysts to reduce energy losses caused by overpotential. Currently, the most widely used electrocatalysts are based on expensive noble metals, such as platinum and palladium [2], which limits their large-scale application. Therefore, this study aims to develop low-cost, stable, and highly efficient FeNixMoWCu high entropy alloy (HEA) thin films as potential electrocatalyst materials for water-splitting applications [3].

In this research, equimolar FeNiMoWCu targets and pure Ni targets were employed to deposit FeNixMoWCu high entropy alloy (HEA) thin films with different nickel contents onto 304 stainless steel, silicon wafers, and nickel foam (NF) substrates using a co-sputtering system. Grazing-incidence X-ray diffraction (XRD) analysis revealed that all films exhibited a typical amorphous structure. The nickel content has no influence on the crystal structure of thin films. The cross-sectional morphologies indicated that all films possessed dense microstructures without noticeable defects. Mechanical property measurements demonstrated stable hardness values, while scratch testing confirmed excellent adhesion, with all films showing a critical load (L_{C3}) exceeding 100 N.

Lower overpotential results than untreated NF for the evolution reaction (HER) were obtained for the FeNixMoWCu thin films deposited on NF after the electrochemical test in 1 M KOH aqueous solution. The effect of Ni content on the electrocatalysis performance of HEA thin films was explored. We can conclude that the HEA film prepared at a Ni target power of 75 W exhibited the best HER performance, with an overpotential of 121 mV and a Tafel slope of 177.7 mV/dec at a current density of 10 mA/cm^2, indicating excellent electrocatalytic activity for hydrogen evolution. This research develops the novel FeNixMoWCu electrocatalyst thin film materials for water-splitting hydrogen production. The results will provide insights into the potential of FeNixMoWCu HEA films for green energy applications with enhanced electrocatalytic performance.

Keywords: High entropy alloy thin films, Hydrogen evolution reaction, FeNi$_x$MoWCu, electrocatalytic property

4:20pm TS1-2-MoA-9 Development of FeNiMoWCuN and FeNiMoWCuC High Entropy Alloy Thin Film as Efficient Electrocatalysts for Water-splitting Applications, *TAI Kao Cheng [dylan37859631@gmail.com], Lee Jyh-Wei,* Ming Chi University of Technology, Taiwan*; Lou Bih-Show,* Chang Gung University, Taoyuan, Taiwan*; Li Chia-Lin,* Ming Chi University of Technology, Taiwan

This study investigates the microstructure and electrocatalytic properties of FeNiMoWCuN and FeNiMoWCuC high entropy alloy (HEA) thin films deposited by high power impulse magnetron sputtering (HiPIMS) anddirect current (DC) magnetron sputtering techniques, respectively. The HEA films were fabricated at various argon-to-nitrogen and argon-to-acetylene gas flow ratios to assess the impact of nitrogen and carbon contents, respectively, on the phase, microstructure, and electrocatalytic properties of the thin films. A proper nitrogen content was found to promote the formation of the metal nitride phase, thereby enhancing the electrocatalytic activity of the films. Notably, improved performance was observed for the HEA films with different N contents in the hydrogen

evolution reaction (HER) and oxygen evolution reaction (OER), characterized by lower overpotentials and smaller Tafel slopes. Similarly, a proper carbon content was achieved for FeNiMoWCuC, resulting in improved electrocatalytic performance. Overall, FeNiMoWCuN and FeNiMoWCuC thin films deposited using HiPIMS and DC with optimized nitrogen content exhibited superior electrocatalytic properties, highlighting their potential for applications in water splitting.

4:40pm TS1-2-MoA-10 Fluorine-Doped Tin Oxide Aerogel Support for Pt Catalysts: Enhancing Hydrogen Evolution Reaction via Sustainable Interface Engineering, *Hyung-Ho Park [hhpark@yonsei.ac.kr],* Yonsei University, Korea

Electrochemical water splitting, especially the hydrogen evolution reaction (HER), plays a pivotal role in sustainable hydrogen production. To overcome the cost and scarcity limitations of Pt-based catalysts, the design of advanced catalyst supports with high surface area, electrical conductivity, and corrosion resistance is essential. In this work, we introduce a fluorine-doped tin oxide (F-SnO$_2$) aerogel as a novel, ultra-lightweight, and porous metal oxide support for Pt catalysts.[1] The aerogel framework was synthesized via a sol-gel method, achieving a high surface area (321 m$^2 \cdot$g^{-1}), interconnected meso-/macroporosity for efficient ion transport, and strong mechanical and chemical stability.[2] Atomic-level fluorine doping significantly enhanced the electrical conductivity by modulating the SnO$_2$ band structure and reducing charge transfer resistance. A thin layer of Pt was deposited via sputtering onto the F-SnO$_2$ surface, resulting in a robust hybrid catalyst (F-SnO$_2$@Pt) that exhibited superior HER performance in acidic media—requiring only 42 mV overpotential at 10 mA·cm^{-2}, outperforming commercial Pt/C in mass activity and turnover frequency. The catalyst also maintained remarkable long-term stability over 10,000 cycles. These enhancements are attributed to: (1) optimal Pt dispersion over a high-surface-area aerogel matrix, (2) ion-permeable porosity facilitating mass transfer, (3) improved conductivity via F$^-$ substitution in the oxide lattice, and (4) strong metal-support interactions altering Pt's electronic structure and water dissociation kinetics. The results demonstrate that F-doped SnO$_2$ aerogels can serve as sustainable catalyst supports for efficient hydrogen evolution, aligning with the goals of green hydrogen generation and surface/interface engineering for clean energy systems.

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[2] V. G. Parale, T. Kim, V. D. Phadtare, W. Han, K.-Y. Lee, H.-N.-R. Jung, H. Choi, Y. Kim, H. M. Yadav, H.-H. Park, J. Mol. Liq. **287**, 110990 (2019).

[3] T. Kim, S. B. Roy, S. Moon, S. Yoo, H. Choi, V. G. Parale, Y. Kim, J. Lee, S. C. Jun, K. Kang, S. Chun, K. Kanamori, H.-H. Park, ACS Nano. **16**, 1625 (2022)

5:00pm TS1-2-MoA-11 High-Entropy Oxide Thin Films for Sustainable Battery Applications, *Pavel Soucek [soucek@physics.muni.cz], Tatiana Pitonakova, Tomas Rada,* Masaryk University, Czechia*; Tomas Kazda, Antonin Simek,* Brno University of Technology, Czechia*; Petr Vasina,* Masaryk University, Czechia **INVITED**

High-entropy materials represent a rapidly growing frontier in materials science, offering new routes toward multifunctional and resource-efficient technologies. While high-entropy alloys (HEAs) have gained considerable attention, the concept has now been successfully extended to ceramics, including oxides, nitrides, borides, and carbides. These materials derive their unique properties from four synergistic effects: high configurational entropy, severe lattice distortion, sluggish diffusion, and the cocktail effect. The resulting structural stability, chemical resilience, and tunable electronic properties make high-entropy oxides (HEOs) highly promising candidates for advanced electrochemical applications such as lithium-ion and sodium-ion batteries.

In this contribution, we present a study of the Mg-Co-Ni-Cu-Zn-O system, crystallizing in a rock salt-type (MgCoNiCuZn)O structure, and Cr–Mn–Fe–Ni–Cu–O system, crystallizing in a spinel-type (CrMnFeNiCu)$_3$O$_4$ structure. While the first system represents a "conservative" choice close to current materials used in batteries, the second is entirely free of critical raw materials while offering the potential for improved electrochemical performance and long-term stability, aligning with the vision of sustainable and circular energy storage technologies. The thin films are deposited by reactive DC and High Power Impulse Magnetron Sputtering (HiPIMS), allowing precise control of elemental composition and plasma energy input. The influence of deposition temperature, stoichiometry, and process parameters on the morphology and crystalline structure of the as-deposited coatings is systematically investigated.

Special emphasis is placed on the fabrication of porous thin films, achieved by adjusting the deposition pressure, target-to-substrate distance, and substrate tilt angle—from normal to glancing-angle configurations. The resulting morphological variations strongly affect ion transport and electrochemical activity. Finally, the electrochemical behavior of selected coatings against lithium and sodium is evaluated and correlated with their structural and compositional features. The findings open new pathways for designing high-entropy oxide electrodes that combine sustainability, structural tunability, and superior performance for next-generation battery systems.

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