

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, Martin Welters, KCS Europe GmbH, Fan-Bean Wu, National United University

1:40pm **TS1-2-MoA-1 The Effect of the Transition Metal Dopant on the Microstructure and Electrochemical Performance of Magnetron Sputtered Electrodes for Solid Oxide Fuel Cells Applications, Justyna Kulczyk-Malecka, Katharina Steier, David Shaw, Kleitos Panagi, Peter Kelly, Manchester Metropolitan University, UK**

The electrolytic energy conversion has become one of the main technologies considered to deliver actions on reducing CO₂ emissions in the energy, heavy-duty transportation and industrial processes sectors. The electrolytic cells (fuel cells and electrolysis cells) can be utilised in energy conversion, generation, and storage, which has been demonstrated at scale in many regions around the world already. Solid oxide cells are typically composed of porous ceramic matrix and Ni metal catalyst fuel electrodes, dense ceramic electrolytes, such as YSZ or GDC, and perovskite air electrodes. These cells operate at relatively high temperatures (typically 600-850°C) and, therefore do not require precious metal group catalysts to drive the reaction forward in both electrolysis (H₂ generation) and fuel cell (energy conversion) modes. Moreover, they are more versatile in terms of required fuel type allowing utilisation of hydrogen as well as alternative fuels, such as methanol, ammonia, or biogas.

In this work, thin (~3 µm) nanostructured cermet anodes consisting of YSZ-Ni and GDC-Ni were doped with transition metals to study their influence on coatings microstructure and electrochemical performance. The anodes were deposited onto commercial YSZ electrolyte support cells using oblique angle pulsed DC reactive magnetron sputtering. The coating microstructure was evaluated using FIB-SEM and TEM and focused on the triple-phase boundary evolution in relation to the amount of the added dopant (0-5 wt.%). The chemical composition of the coatings was assessed using EDX, XPS and XRD analysis. The polarization curves were obtained from SOFC single stack assemblies under hydrogen and air flows for anode and cathode, respectively, at operating temperatures of 850°C to evaluate the electrochemical performance of the deposited films.

2:00pm **TS1-2-MoA-2 Investigation of Ba_{0.5}Ce_{0.3}Zr_{0.18}Y_{0.01}Yb_{0.01}O_{3-δ} / Y_{0.2}Ce_{0.8}O_{2-δ} Composite Coatings for the Electrolyte of Solid Oxide Fuel Cell, Yen-Yu Chen, Ke-Hsing Wang, National Pingtung University of Science and Technology, Taiwan**

Solid oxide fuel cells (SOFCs) are one of the potential power generation devices for the sources of renewable energy. In this study, the composite coatings consisted of BaCe_{0.7}Zr_{0.12}Y_{0.1}Yb_{0.1}O_{3-δ} (BCZYYb) and Y_{0.2}Ce_{0.8}O_{2-δ} (YDC) were developed by a colloidal coating process as the electrolyte for SOFC. Both of the powders of BCZYYb and YDC were synthesized by the solid-state reaction methods. The well dispersed suspensions after refined by the planetary ball method were spin coated on the porous NiO/BaZr_{0.85}Y_{0.15}O_{3-δ} (BZY) substrates, which were prepared by a die pressing method after pre-sintering at 1200°C for 1 h. Several properties were analyzed including microstructures, crystallographic phases, and electrical performance. The BCZYYb/YDC composite coatings still showed dual-phases including perovskite-type BCZYYb and fluorite-type YDC after sintered at 1550°C for 24 h. The coating layer shows high density after sintered. And the thickness of the coating layers are around several µm. The composites show ionic conductive behaviors from the temperature between 500~800°C. The details will be reported in the presentation.

2:20pm **TS1-2-MoA-3 Unveiling the ORR Mechanism on Co Single-Atom Catalysts Using Operando Raman Spectroscopy with Catalyst-Coated Membrane (CCM) Methodology, Sun-Tang Chang, Yi-Qing Chu, Zih-Jhong Huang, Chen-Hao Wang, National Taiwan University of Science and Technology, Taiwan**

In this study, operando Raman spectroscopy was employed to investigate the mechanism of the oxygen reduction reaction (ORR) on a cobalt single-atom catalyst (Co-SAC). The Co-SAC was synthesized and utilized as a cathode catalyst for an alkaline anion exchange membrane fuel cell (AAEMFC). The results demonstrate excellent ORR activity, with an electron transfer number of 3.96 and J_{lim} is 5.5 mAcm⁻².

X-ray absorption spectroscopy (XAS) revealed that the Co-SAC features a Co-N5 coordination structure, with cobalt in the +3 oxidation state. Furthermore, wavelet transform (WT) analysis confirmed the presence of isolated cobalt single atoms.

To minimize interference from the electrolyte during Raman laser measurements, the catalyst-coated membrane (CCM) method was adopted. This approach effectively prevents direct interaction between the laser and the electrolyte while ensuring efficient OH⁻ group transfer to the catalyst. Additionally, in the operando setup, electrochemical impedance spectroscopy (EIS) was integrated with Raman spectroscopy. This combination enabled a detailed observation of the ORR mechanism and the evolution of surface phenomena under different applied biases.

This study represents a significant breakthrough in unveiling the ORR mechanism, particularly for Co-based single-atom catalysts.

2:40pm **TS1-2-MoA-4 Study on Mo_xN Thin Films Deposited by HiPIMS and RF Sputtering with Heteroatom Doping for Hydrogen Evolution Reaction Catalysts, Hung-I Wu, National Yunlin University of Science and Technology, Taiwan; Ying-Hsiang Lin, National United University, Taiwan; Shih-Hung Lin, National Yunlin University of Science and Technology, Taiwan; Fan-Bean Wu, Chi-Yueh Chang, National United University, Taiwan; Thi Xuyen Nguyen, Chia-Ying Su, Rwei-Chi Lin, Jyh-Ming Ting, National Cheng Kung University (NCKU), Taiwan; Wan-Yu Wu, National United University, Taiwan**

In recent years, materials such as transition metal oxides and nitrides have been popular in catalyst research. Compared to scarce noble metals, molybdenum-based materials not only have more abundant resources but also exhibit excellent activity[1], making them highly suitable to replace the costly noble metal catalysts (Ru, Ir, RuO₂, IrO₂). Molybdenum nitride (MoN) possesses outstanding corrosion resistance and electronic conductivity[2], allowing it to perform the hydrogen evolution reaction (HER) in acidic media. If different elements like Ti, Co, Ni, and V are doped into Mo_xN as a substrate, a synergistic effect is expected to further enhance HER performance. Additionally, when attaching catalysts to electrodes, one must consider the uniformity of surface coverage and adhesion on electrodes of various shapes. As is well known, sputtering offers advantages such as uniform coating, easy control of film thickness, and excellent adhesion. Therefore, using this method to prepare catalyst thin films on electrodes is the optimal choice.

In this study, we employed RF sputtering and High Power Impulse Magnetron Sputtering (HiPIMS) techniques to deposit Mo_xN thin films for comparison. Initially, we deposited Mo_xN thin films using HiPIMS and found in preliminary results that the HiPIMS-deposited Mo_xN thin films exhibited an overpotential of 415 mV at η=10 during hydrogen evolution reaction (HER) tests in 0.5M H₂SO₄. Energy-dispersive X-ray spectroscopy (EDS) analysis revealed that the Mo and N contents in the Mo_xN thin films were 60 at.% and 40 at.%, respectively. Grazing-incidence X-ray diffraction (GIXRD) results indicated that the thin films have a face-centered cubic (FCC) structure similar to Mo₃N₂. Building on these Mo_xN results, we will further explore the impact of varying the N/Mo ratio on HER performance. Additionally, we plan to attempt doping other elements into Mo_xN to observe the changes induced by doping.

3:00pm **TS1-2-MoA-5 Ternary FeCoNi / Graphene Composites as Electrocatalysts for Highly Efficient Hydrogen Evolution Reaction, Yu Tsung Lin, Jow Lay Huang, Sheng Chang Wang, Yu Min Shen, National Cheng Kung University (NCKU), Taiwan**

As a consequence of the depletion of fossil fuels, the escalating energy crisis has driven researchers to explore innovative energy. To address this problem, exploring hydrogen energy generation via water splitting emerges as a promising solution. This process involves the hydrogen evolution reaction (HER), a multi-electron transfer process necessitating catalysts to facilitate efficient rates. Despite noble metals have conventionally served this purpose due to their favorable Gibbs free energy, their prohibitive costs pose challenges for widespread adoption and commercialization. In response, our investigation focuses on HER within alkaline electrolytes, aiming to engineer alternative electrocatalysts that are both cost-effective and efficient.

Transition metals from the first row are active centers for the HER due to their 3d orbital, exhibiting excellent activity. Among them, nickel (Ni) and cobalt (Co) shows the most promising potential because the hydrogen adsorption energy of them approaches that of Pt. Furthermore, recent studies have indicated that the incorporation of third transition metals, such as iron (Fe), can further enhance the HER activity. This is attributed to the synergistic effect among the three metals. Additionally, it can be observed that a large number of studies use two-dimensional carbon

materials as supports. Graphene, a highly conductive 2D material, serves as an excellent supporting matrix due to its high surface area, facilitating efficient electron transfer during HER. Consequently, we synthesized ternary FeCoNi-LDH/graphene composite by hydrothermal method and measured the HER performance in 1.0 M KOH electrolyte. We used X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), and transmission electron microscope (TEM) to determine the crystal structure and morphology of the composite. The results demonstrate that we successfully synthesized FeCoNi-LDH/graphene. In addition, Linear Sweep Voltammetry (LSV) were employed to confirm the HER performance. The overpotential and Tafel slope is -386.6 mV and 85.8 mV/dec, respectively.

3:20pm TS1-2-MoA-6 Ti-Cr-N Nanopyramid/Nitrogen-Doped Carbon Quantum Dot/Stainless Steel Mesh as a Flexible Supercapacitor Electrode, Rajesh Kumar, Bhanu Ranjan, Krishan Kumar, Satyam Shankhdhar, Davinder Kaur, Indian Institute of Technology Roorkee, India

Nitrogen-doped carbon quantum dots (N-CQDs) incorporated into highly conductive transition metal nitride offer enhanced electrochemical performance, delivering high energy density and outstanding electrochemical stability. The present study reports a high-performance supercapacitor electrode consisting of electrophoretic anchored zero-dimensional N-CQDs with reactively co-sputtered titanium chromium nitride nanopyramids (Ti-Cr-N) thin film on flexible stainless-steel mesh (SSM) substrates. The nanopyramids of N-CQDs/Ti-Cr-N offer remarkable electrochemical performance through Li^+ storage, ascribed to the abundant electroactive sites and enhanced synergism between the high specific surface area of N-CQDs and higher conductivity of Ti-Cr-N. Subsequently, the N-CQDs/Ti-Cr-N/SSM electrode in 1M Li_2SO_4 aqueous electrolyte exhibits an excellent gravimetric capacitance of 393.8 F.g^{-1} at a specific current density of 0.32 A.g^{-1} . Further, the N-CQDs/Ti-Cr-N/SSM heterostructure outperforms other multi-cationic-based supercapacitors with a maximum energy density of 41.41 Wh.kg^{-1} and a superior power density of 7.0 kW.kg^{-1} . Impressive electrochemical stability of $\sim 88.6\%$ is retained by the heterostructure even after 5000 continuous charge-discharge cycles. Insights into charge-storage mechanisms highlight the dominance of surface-limited capacitive and pseudocapacitive kinetics, with fewer contributions from diffusion-controlled faradaic processes. Furthermore, an exemplary mechanical stability of $\sim 99.98\%$ over 1200 bending cycles demonstrates the N-CQDs/Ti-Cr-N/SSM heterojunction's excellent resilient structural strength, validating the present electrode potential for high-performance flexible supercapacitor application.

4:00pm TS1-2-MoA-8 Effects of Nb Content on the Water Splitting Performance of FeNiMoWNBx High Entropy Coating Grown by Magnetron Sputtering, Naveen Karuppusamy, Ming Chi University of Technology, Taiwan; Bih-Show Lou, Chang Gung University, Taoyuan City, Taiwan; Jyh-Wei Lee, Ming Chi University of Technology, Taiwan

Hydrogen fuel is an alternative green energy to neutralize the carbon emission and to fulfill the global energy demand. Electrochemical water splitting is a technique to produce hydrogen comprising of both oxygen evolution reaction (OER) and hydrogen evolution reaction (HER), where OER is a bottleneck reaction in water splitting for efficient performance. Thus, it is necessary to prepare a suitable catalyst with an excellent water splitting performance. Though RuO_2 is a benchmark catalyst for OER performance, scarcity and cost of resources are limited to the use of RuO_2 in water splitting performance. Transition-metal based electrocatalyst already garners attention due to its excellent redox properties, earth abundant and cost-effective, which makes it suitable for alternative catalyst.

In this study, FeNiMoWNBx high entropy alloy (HEA) coatings with different Nb contents were fabricated on carbon cloth (CC) at high inclined angle using magnetron sputtering technique. The composition of Nb is altered with changing the RF power from 0 to 100 W. Chemical composition of FeNiMoWNBx was analyzed by the field emission electron probe microanalyzer (FE-EPMA). Phase analysis and crystallinity of FeNiMoWNBx were analyzed with glancing angle X-ray diffractometer (GA-XRD). Surface roughness and cross-section morphologies of FeNiMoWNBx films were examined using atomic force microscope (AFM) and field emission scanning electrochemical microscopy (FE-SEM). The OER performance of FeNiMoWNBx films was investigated with the polarization curve obtained in 1 M KOH and 1 M KOH + 3.5 wt.% NaCl aqueous solution. FeNiMoWNBx thin films exhibited a superior OER performance with low overpotential in 1 M KOH and 1 M KOH + 3.5 wt.% NaCl solution. The effects of Nb concentration in FeNiMoWNBx film and the sputtering inclined angle on the kinetics of OER performance was discussed. This work provides the

strategy for the fabrication of cost effective FeNiMoWNBx HEA thin film catalyst for efficient water splitting performance.

4:20pm TS1-2-MoA-9 Pseudocapacitive Storage in Molybdenum Oxynitride Nanostructures Reactively Sputtered on Stainless-Steel Mesh Towards an All-Solid-State Flexible Supercapacitor, Bhanu Ranjan, Davinder Kaur, Indian Institute of Technology Roorkee, India

Exploiting pseudocapacitance in rationally engineered nanomaterials offers greater energy storage capacities at faster rates. The present research reports a high-performance Molybdenum Oxynitride (MoON) nanostructured material deposited directly over stainless-steel mesh (SSM) via reactive magnetron sputtering technique for flexible symmetric supercapacitor (FSSC) application. The MoON/SSM flexible electrode manifests remarkable Na^+ -ion pseudocapacitive kinetics, delivering exceptional $\sim 881.83 \text{ F.g}^{-1}$ capacitance, thanks to the synergistically coupled interfaces and junctions between nanostructures of Mo_2N , MoO_2 , and MoO_3 co-existing phases, resulting in enhanced specific surface area, increased electroactive sites, improved ionic and electronic conductivity. Employing 3D Bode plots, b-value, and Dunn's analysis, a comprehensive insight into the charge-storage mechanism has been presented, revealing the superiority of surface-controlled capacitive and pseudocapacitive kinetics. Utilizing PVA- Na_2SO_4 gel electrolyte, the assembled all-solid-state FSSC ($\text{MoON/SSM} \parallel \text{MoON/SSM}$) exhibits impressive cell capacitance of 30.7 mF.cm^{-2} (438.59 F.g^{-1}) at 0.125 mA.cm^{-2} . Moreover, the FSSC device outputs superior energy density of $4.26 \text{ } \mu\text{Wh.cm}^{-2}$ (60.92 Wh.kg^{-1}) and high power density of 2.5 mW.cm^{-2} (35.71 kW.kg^{-1}). The device manifests remarkable flexibility and excellent electrochemical cyclability of $\sim 91.94\%$ over 10,000 continuous charge-discharge cycles. These intriguing pseudocapacitive performances combined with lightweight, cost-effective, industry-feasible, and environmentally sustainable attributes make the present MoON -based FSSC a potential candidate for energy-storage applications in flexible electronics.

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4:40pm TS1-2-MoA-10 Applicability of MoS_2 -aSiC Heterostructure for Durable Supercapacitance and NO_2 Gas Sensing in Harsh Environment, Habeebur Rahman, Indian Institute of Technology Roorkee (IIT Roorkee), India; Gagan Kumar Sharma, Indian Institute of Technology Roorkee, India; Preetam Singh, CSIR-National Physical Laboratory Delhi, India; Davinder Kaur, Indian Institute of Technology Roorkee, India

In the present work, the heterostructure of molybdenum disulfide (MoS_2) with amorphous silicon carbide (aSiC) on stainless steel (SS) and Si substrates was fabricated using a DC magnetron sputtering system. This unique heterostructure was examined for energy storage and NO_2 gas sensing applications suitable for harsh environmental conditions. The 2D MoS_2 nanostructured with dissolution resistive aSiC supercapacitor electrode delivers 1.5-fold enhancement in the gravimetric capacitance, a voltage window enlargement from 0.8V to 1.8 V, and an excellent stability of more than 4,000 charge-discharge cycles. Further, the high concentration NO_2 gas sensing performance of the MoS_2 -aSiC on Si substrate revealed the stable and recoverable response at high operating temperatures. Therefore, loading aSiC with 2D MoS_2 enables durable electrode material for energy storage and NO_2 gas sensing applications in adverse conditions. The as-

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fabricated heterostructure was systematically studied by various material and electrochemical characterizations.

5:00pm TS1-2-MoA-11 One Step Fabrication of Highly Ordered Binder Free Vanadium Oxide Thin Film Cathode for Next Generation Micro Batteries, Ananya Bansal, Indian Institute of technology Roorkee, India; *Ramesh Chandra*, Indian Institute of Technology Roorkee, India

The increasing demand for microelectronics has significantly driven the advancement of thin film energy storage devices, specifically batteries. Till now, a range of materials have been investigated for lithium-ion batteries, with vanadium oxide emerging as a promising material. Vanadium oxides are known for multiple oxidation-reduction states during electrochemical reaction, hence, can promote multiple diffusion of Lithium ions resulting in high energy and power density. In this work, binder-free Vanadium oxide (V_2O_5) has been synthesized by reactive DC magnetron sputtering on aluminium foil substrate. Vanadium target (99.99% pure) is bombarded with high-energy Ar^+ ions which dislodge atoms from the vanadium surface. These ejected atoms then react with oxygen ions to form highly pure V_2O_5 and deposit onto a substrate, forming a thin film. The deposited layers were analyzed for their structural and surface morphology using XRD and SEM techniques. Highly ordered brick like nanostructures were observed during SEM analysis. X-ray photoelectron spectroscopy measurements were carried out to understand the chemical bonding of the cathode. The surface of vanadium oxide obtained from this binder-free approach helps us to create a high-quality cathode-electrolyte interface with high wettability (33.3°). Cyclic voltammetry (CV), galvanostatic charge-discharge (GCD) cycling, and electrochemical impedance measurements were used to investigate the capacity and cycling stability of the V_2O_5 cathode in 1 M lithium hexa-fluoro-phosphate. As a result, it can be interpreted that this binder-free technology can be used to fabricate efficient lithium free cathode for new generation thin film batteries.

5:20pm TS1-2-MoA-12 Research Coating Conductive Material on $SiO_x@rGO$ Composite Materials as Anode Material in Lithium-Ion Batteries, Yi-Ling Chen, National Cheng Kung University (NCKU), Taiwan

With the advancement of technology, lithium-ion batteries have emerged as a future energy storage technology with the gradual development of electric vehicles. Silicon-based materials, due to their high theoretical capacity, energy density ($\sim 4200\text{mAh/g}$), and natural abundance, are considered as candidates for negative electrode materials in lithium-ion batteries.

In this study, our research team successfully prepared reduced graphene oxide (rGO) using the Hummer method and incorporated commercial SiO_x micron-sized powder to synthesize $SiO_x@rGO$ composite material as an anode for lithium-ion batteries. The initial charge capacity was measured at 1487mAh/g , with a discharge capacity of 1060mAh/g , yielding an initial coulombic efficiency of 71%. After 40 cycles, the capacity retention remained at 91%. However, there are currently no theoretical studies addressing the lithiation process and lithium ion insertion/extraction mechanisms in SiO_x materials.

Therefore, in our study, we not only explore the use of $SiO_x@rGO$ composite material as an anode to improve the theoretical capacity and energy density of lithium-ion batteries but also aim to enhance the electrical conductivity and electrochemical performance of the battery. Conductive materials such as copper, gold, and platinum will be deposited on the prepared $SiO_x@rGO$ anode material. These conductive coatings will provide additional electrons, creating a driving force for lithium ion diffusion into the anode material during discharge to achieve charge conservation. This process is expected to enhance the capacity and cycling stability of lithium-ion batteries. We will characterize the materials using X-ray diffraction (XRD), scanning electron microscopy (SEM), energy-dispersive spectroscopy (EDS), and transmission electron microscopy (TEM). Furthermore, charge-discharge and cycling performance tests will be conducted on the lithium-ion batteries to investigate the effect of the conductive materials on their performance. Cyclic voltammetry will be employed to observe the electrochemical reactions during charge and discharge cycles.

This study conducts a theoretical analysis of $SiO_x@rGO$ composite material as an anode for lithium-ion batteries by coating it with conductive materials, aiming to provide valuable reference data for both commercial and academic purposes.

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