Monday Morning, May 20, 2024

Topical Symposium on Sustainable Surface Engineering Room Town & Country B - Session TS1-1-MoM

Coatings for Batteries and Hydrogen Applications I

Moderator: Nazlim Bagcivan, Schaeffler Technologies GmbH & Co. KG, Germany

10:00am TS1-1-MoM-1 New Coating Methods for New Electrolyzer Technologies for PEM Electrolyzer and AEM Electrolyzer, Thomas Kolbusch (tkolbusch@coatema.de), Coatema, Germany INVITED

The author describes in his talk the scale up process for PEM electrolyzers regarding coating technologies. The first part of the talk shows the need for scale up in the green hydrogen market due to the huge amount of green hydrogen which has to be produced till 2030 and 2050 to reach net zero targets. For this industrial standardization of coating processes are needed as soon as possible. Here the upscale process of reproducible and reliable roll 2 roll equipment are shown and getting over the limiting factors like dimension instability of membrane materials and multi layer coating applications of rare materials like iridium.

The second part describes the boundaries of todays coating methods describing the parameters needed to be optimized. The background on one of the standard methods today, slot die coating is described here in detail with some theoretical background on slot die coating technology and an overview on developments for intermittent slot die coatings. The third part of the talk describes a new digital fabrication method for electrolyzers and shows the opportunity for the overall green hydrogen market using digital fabrication methods, reducing the carbon footprint of coating equipment for PEM electrolyzer.

10:40am TS1-1-MoM-3 Dual Doped Two-dimensional Carbon Supported Single Atomic Iron for Oxygen Reduction Reaction in Alkaline-Exchange Membrane Fuel Cells, *Afandi Yusuf (afandi2102@gmail.com), F. T. D. Wijaya, H. Hsin-Chih, C. Wang,* National Taiwan University of Science and Technology, Taiwan

Single atom catalysts (SAC) represent an intriguing option due to their ability to unlock the latent catalytic potential in oxygen reduction reaction (ORR). Nevertheless, the endurance of individual atomic entities is confronted by numerous impediments, encompassing the dissolution of metallic species, metal agglomeration, and the deactivation of catalytically active sites. A crucial factor in enhancing stability lies in the judicious selection of a compatible support catalyst capable of fostering strong metal-support interaction (SMSI).

Carbon is widely utilized as a catalyst support material owing to its favorable electron conductivity and the ability assume diverse dimensional configurations, ranging from 0D to 3D. Additionally, the carbon support facilitates additional customization, such as the introduction of Chalcogen or Pnictogen atom groups through doping, leading to enhanced catalytic activity.

In this work, we successfully fabricated a two-dimensional porous nanosheet electrocatalyst designed to enhance the Oxygen Reduction Reaction (ORR) in Anion-Exchange Membrane Fuel Cells (AEMFCs). This catalyst featured single atomic active sites of Iron supported by a Nitrogen-Phosporus co-doped Carbon material, aimed at reducing catalyst cost and thereby increasing the accessibility of the fuel cell for commercial applications. The electrochemical performance of the material was exemplified by the MDP-4-Fe-800 sample, demonstrating an onset potential of 0.97 V, an E_{1/2} of 0.86 V, and a J_{limiting} of 5.5 mA/cm² under alkaline conditions, surpassing that of commercial Pt/C under the same conditions. Moreover, the material exhibited notable stability after 30,000 cycles, experiencing only a marginal 0.39 mA/cm² in J_{limiting} and 30 mV decrease in both onset and E_{1/2}. While MDP-4-Fe-800 did not outperform the single-cell performance of commercial Pt/C, it displayed commendable activity, generating a power density of 244.8 mW/cm².

11:00am TS1-1-MoM-4 CO₂ Laser Processed Nickel Catalyzed Graphene Coating for Electrocatalytic Water Splitting and Energy Storage Applications, *Suparna Saha (suparna.saha@tcgcrest.org)*, TCG CREST (RISE), India; *S. Hiwase*, IISER PUNE, India; *S. Ogale*, IISER PUNE, TCG-CREST(RISE), India

Development of efficient, cost-effective, and environmentally friendly processes for the realization of high-quality graphene on metallic substrates is highly desirable for multiple energy applications as well as nextgeneration graphene-based green electronics. Several polymers including those derived from natural sources represent a rich source of carbons that can be converted into graphitic carbons by energy inputs in different forms. Lasers represent a form of energy input that is direct-write type and does not need the whole substrate to be heated at high temperatures for carbonization. Herein, we examine carbonization of natural product-derived polymer(s) into graphitic (few layers graphene) carbon using CO2 laserassisted direct-write process. Such a process is generally termed as Laser-Induced Graphene (LIG). In particular, we demonstrate the key and interesting role played by nickel in enhancing the degree of graphitization (Ni-LIG). Indeed, the unique advantage of this nickel-catalyzed scanning laser-induced transient pyrolysis process implemented under ambient conditions is that we are able to uniformly graphitize the thermoset polymer coating that would otherwise yield hard carbons or a mixture of ordered/disordered carbon in a furnace-based pyrolysis process. It was observed that upon optimized laser processing condition, the Ni-(CH₃COO)₂. 4H₂O salt added to the polymer gets reduced to Ni (111), which in turn catalyzes the nucleation process. We note the appearance of disordered carbon chains initially, which upon interacting precisely with the nickel surface lowers the activation barrier for graphene formation by annealing the defects. Moreover, due to the very low lattice mismatch between Ni (111) and graphene, a strong interphase is formed, facilitating efficient contact and charge transfer. As the surface coating is decorated with a mixture of Ni/NiO (as confirmed by XRD and XPS), water dissociation as well as adsorption of water oxidation intermediates is promoted, leading to an impressive value of overpotential for oxygen evolution reaction (OER) at 10 mA/cm² of only 330 mV in 1M KOH. We also examined the case of ureaincorporated material to induce N-doping so as to enhance the conductivity via the incorporation of the II-conjugated system. However, the incorporated pyrrolic N defects in the carbon layer were noted to hinder the nucleation of graphitization at the Ni atom, resulting in a low I_G/I_D ratio. This material was therefore studied for charge storage property by cyclic voltammetry and galvanostatic charge-discharge (GC) measurement. It was found that N-doped N-Ni-LIG has a higher specific capacitance compared to Ni-LIG.

11:20am TS1-1-MoM-5 Bimetal Phosphide (NiCoP)/Graphitic Carbon Nitride(g-C₃N₄) Composites for Hydrogen Evolution Reaction in Alkaline Electrolyte, Yu-Hsuan Kao (hsuan890411@gmail.com), National Cheng Kung University, Taiwan; S. Wang, Southern Taiwan University of Science and Technology, Taiwan; J. Huang, National Cheng Kung University, Taiwan; Y. Shen, Hierarchical Green-Energy Material (Hi-GEM) Research Center, Taiwan

In order to address the growing energy crisis and environmental concerns, the development of hydrogen energy through electrochemical water splitting into hydrogen represents a viable solution. The hydrogen evolution reaction (HER) during water splitting is multi-electron transfer process that requires catalysts to proceed at appreciable rates. Noble metals have been widely used for water splitting due to their low Gibbs free energy; however, their high cost limits their availability and hinders commercialization. To address this challenge, we conducted a study on HER in alkaline electrolytes with the aim of developing highly efficient and durable electrocatalysts. The efficiency of HER in an alkaline environment is determined by a delicate balance among three crucial factors: the energy required to dissociate water molecules, hydrogen adsorption (Had) on the catalyst's surface, and the prevention of hydroxyl adsorption (OHad), often referred to as the poisoning of active sites.

Graphitic carbon nitride (g-C₃N₄) has been extensively studied due to its two-dimensional layered structure and high nitrogen content. However, its poor conductivity limits its application in the field of HER. Therefore, we modified transition metal phosphide on g-C₃N₄ to enhance its conductivity and increase the number of active sites. Additionally, it has been mentioned in previous studies that nickel and cobalt atoms can promote the reaction kinetics of HER in the first and second steps, respectively, thus improving the efficiency of HER in an alkaline electrolyte. Therefore, we synthesized NiCoP/g-C₃N₄ composites in different weight percent to replace the platinum electrode in a 1.0 M KOH electrolyte.

We used Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), and transmission electron microscope (TEM) to determine the structure of NiCoP/g-C₃N₄. In addition, Linear Sweep Voltammetry (LSV) and Tafel slope were employed to confirm the electrochemical performance of NiCoP/g-C₃N₄ in HER. The results demostrate that we successfully synthesized g-C₃N₄ and NiCoP/g-C₃N₄ electrocatalysts using a wet chemical method and calcination. Furthermore, the electrochemical results indicate that the addition of 10 wt% NiCoP to g-

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 C_3N_4 significantly improves and exhibits excellent performance in HER in an alkaline electrolyte, reducing the overpotential from 560.7 mV to 338.9 mV and decreasing the Tafel slope from 197.2 mV/dec to 89.6 mV/dec. Then we will use in-situ TEM and in-situ Raman analysis to confirm the contribution and role of the NiCoP alloy on g-C₃N₄ in the hydrogen evolution reaction.

11:40am TS1-1-MoM-6 Hybrid Inorganic-Organic Nanolayered Thin Films Based on Zns-Ethylenediamine for the Photocatalytic Production of Hydrogen, L. Cerezo, Instituto de Investigaciones en Materiales, Universidad Nacional Autónoma de México; K. Valencia, Instituto de Ingenieria, Universidad Nacional Autónoma de México; M. Bizarro, Sandra E. Rodil (srodil@unam.mx), A. Hernández-Gordillo, Instituto de Investigaciones en Materiales, Universidad Nacional Autónoma de México

Hybrid ZnS-ethylenediamine nanomaterials (ZnS(en)_{0.5}) were produced in a mixed solvent of water, butanol, and ethylenediamine by solvothermal and precipitation methods. The effect of different molar ratios H₂O/Zn²⁺ between 15 and 91 have been investigated by diverse techniques; TG-DSC analysis, X-ray Diffraction, Scanning Electron Microscopy, Infrared, and Diffuse Reflectance spectroscopy. The material was then exfoliated using a combined sonication-illumination process to obtain stacked 2D ZnS nanolayers intercalated with the organic material. After optimization, the as-prepared and exfoliated ZnS(en)_{0.5} nanosheets were prepared as films by spin coating to evaluate the photocatalytic H₂ production reaction. The H₂ evolution reaction was performed in a homemade glass photoreactor containing 30 mL of ethanol-water solution (50:50 vol. %). Eight films were fixed around the internal walls of the glass reactor. The solution was magnetically stirred (at 600 rpm), irradiated with UV light provided by a Hg lamp Pen-ray (of λ =254 nm and I₀ = 4.4 mWcm⁻²), and placed in the center of the solution into the quartz tube. The system was bubbled with N_2 (to reduce the O₂ pressure), then it was sealed, and the lamp was turned on. The quantity of H₂ was measured using a Shimadzu GC-2014 gas chromatograph and N₂ as the carrier gas. The photocatalytic activity of all the as-prepared and sonicated-irradiated ZnS(en)0.5 samples was evaluated by 6 h and 6 cycles. The results showed an enhancement of 7 to 22 times in the H₂ production rate as a function of the synthesis and exfoliation conditions. Intrinsic hydrogen evolution rates up to 76 mmolg⁻¹h⁻¹ were achieved using the optimized exfoliated ZnS(en)_{0.5} hybrid material. This value constitutes a record in the community, which is more significant when the lamp's low power is considered. The increased photoactivity was correlated to the degree of exfoliation and the number of stacked ZnS layers in the structure.

12:00pm TS1-1-MoM-7 One-pot Synthesis of NiFeCo(OH)*@FeOOH@(NiFeCo)S* Electrocatalyst for Urea Oxidation Reaction, *Thi Xuyen Nguyen (nguyenxuyen1511@gmail.com), Z. Wei, J. Ting,* National Cheng Kung University, Taiwan

Urea oxidation reaction (UOR) is a promising energy-saving avenue for sustainable hydrogen production. However, the 6-electron transfer reaction lead the sluggish kinetic. In this work, an ultrafast, one-step method has been used to synthesize novel NiFeCo(OH)_x@FeOOH@(NiFeCo)S_x heterostructure supported on Ni foam as electrocatalyst toward UOR. We demonstrate that the NiFeCo(OH)_x@FeOOH@(NiFeCo)S_x exhibits outstanding UOR performance with a low potential of 1.36 V versus reversible hydrogen electrode at a current density of 100 mA cm⁻². The catalyst also shows great durability for 50 hours at 10 mA cm⁻². The outstanding electrochemical performance is attributed to the high surface area, the faster electron transfer, free of binder, and the synergistic effects of among metallic components.

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

Coatings for Batteries and Hydrogen Applications II

Moderator: Nazlim Bagcivan, Schaeffler Technologies GmbH & Co. KG, Germany

2:00pm TS1-2-MoA-2 Effect of Atomic Layer Deposited Films on Three-Dimensional Electrodes for Lithium-Ion Batteries, *P. Lin*, National Chung Hsing University, Taiwan; *Chih-Liang Wang (wangcl@mx.nthu.edu.tw)*, National Tsing Hua University, Taiwan

The great capability of atomic layer deposition (ALD) in precisely controlling the film quality, such as thickness, composition and comformality has been paid much attention. Herein, we presented a study of coating nanocomposite metal oxides via ALD on three-dimensional electrodes for lithium-ion batteries. The three-dimensional electrodes were prepared by the hydrothermal synthesis of TiO₂ nanorods on carbon cloths. The nanocomposite metal oxides of TiO2 and ZnO were deposited on TiO2 nanorods by ALD. The effect of metal oxides on electrochemical performance was systemically investigated by using different ALD cycles of TiO₂ and ZnO. The results indicated that the reversible capacity and rate performance of three-dimensional electrodes can be improved after ALD nanocomposite metal oxides. The improved performance can be attributed to the function of ALD TiO₂ to not only alleviate the volume change and the growth of solid electrolyte interphase but also improve electronic conductivity. More details related to battery performances and film properties, influenced by the ALD cycle of individual metal oxide, will be reported in the presentation.

2:20pm TS1-2-MoA-3 Effects of Additives on Electrochemical Performance of Sodium Ion Batteries, *Ting Ching Lin (tim881231@gmail.com), J. Huang,* National Cheng Kung University (NCKU), Taiwan; *C. Chang,* National University of Tainan, Taiwan

In recent years, the surge in electric vehicle popularity and the push for renewable energy development by various governments globally have enhanced energy security, mitigated the risk of fuel leaks, and lessened the dependence on imported fuels. This has been achieved by ensuring a steady supply of electricity and diversifying fuel sources. As the demand for lithium continues to rise with the increased adoption of lithium-ion batteries, attention is turning towards sodium ion batteries due to the limited availability of lithium resources. Sodium ion batteries are gaining traction, particularly in the electrical and electronic sectors. Their operational principle closely mirrors that of lithium-ion batteries, encompassing positive and negative electrodes, isolation membranes, and electrolytes. Notably, sodium ion batteries offer cost advantages over lithium batteries, with more readily available precursor materials for the negative electrode and a lower carbonization temperature during graphite negative electrode production. Despite these merits, sodium ion batteries currently encounter challenges, such as lower energy density and storage capacity compared to lithium batteries. The cycle life of existing sodium ion batteries also falls short of that seen in commercial lithium batteries. The primary hurdles stem from the lower energy density and suboptimal cycling performance, which heavily rely on the formation of solid electrolyte interface (SEI) films on the electrode surface.

To address these challenges, the research proposes a novel approach: the combination of different kinds of new ion additives to form a composite additive. The aim is to create a stable and dense SEI that exhibits thinness, electronic insulation, and ion conductivity. This approach seeks to overcome the limitations of sodium ion batteries, particularly their low energy density and poor cycling performance, by fostering the development of an enhanced SEI.

In this research, we used electrochemical impedance spectroscopy (EIS) to measure interface resistance, and used X-ray photoelectron spectroscopy (XPS), Scanning Electron Microscope (SEM), Fourier-transform infrared spectroscopy (FTIR) to observe the electrode surface and analyze the SEI film. The results indicate that the addition of the NaDFP ionic additive effectively improves the retention of battery capacity and establishes a durable SEI film.

Keywords: sodium ion batteries, new ion additives

2:40pm TS1-2-MoA-4 Effect of SiOx/RGO via Phosphorus Doping as Anode Materials for Lithium-Ion Batteries, Wen-Feng Lin (chanes40417@gmail.com), J. Huang, S. Brahma, National Cheng Kung University (NCKU), Taiwan; Y. Shen, Hierarchical Green-Energy Materials Research Center (Hi-GEM), Taiwan

Lithium-ion batteries has been one of the promising energy storage devices due to its high energy density, high capacity, and non-memory effect. Anode is one of the important component of the batteries. To meet the demand of electronic devices, scientists are dedicated to develop novel anode materials since the traditional graphite has the drawback of low theoretical capacity (approximately 372 mAhg⁻¹), which cannot achieve the requirement of the high-energy devices. There are different types of materials during lithiation/delithiation process, such as alloys, transition metals, and insertion/desertion materials. Among them, we choose nonstoichiometric silicon oxide (SiO_x, 0 < x < 2) because it has relatively low volume expansion as compared to pure silicon. However, the ratio of volume expansion is still a non-negligible phenomenon since it indirectly affects the capacity. Therefore, we utilize the characteristics of reduced graphene oxide (rGO) and phosphorus (P) to improve the disadvantages of SiO_x. The character of rGO is the carrier of lithium ions and electrons owing to the 2D-layered structure. Besides, rGO is a carbonaceous material which can enhance the conductivity of anode materials. For the purpose of improving initial coulombic efficiency (ICE), P acts as dopant that can create extra space for lithium-ion storage and transport because the chemical and physical structures of rGO may be changed. We first synthesize SiO_x/rGO, then modify the composite with P by using phosphoric acid as P source in the second step followed by post annealing as the last step. The final product is named as P-SiO_x/rGO (1P, 2P represent different concentration of phosphoric acid). From the results of XPS, P bond with O and EDS mapping shows the uniform distribution of P. Hence, the analyzed results ensure the doping of P. Further, the electric properties are verified through the testing of battery performance, charge/discharge, cycling and C-rate are included. Compared to the un-doped and doped samples, ICE of the latter (42.5 % for 1P-SiO_x/rGO and 56.2 % for 2P-SiO_x/rGO) are higher than the former (39.7 % for un-doped SiO_x/rGO), which means the effect of P is achieved. On the other hand, P-doping also stabilized the structure of electrode, the lithiation capacity of 1P-SiO_x/rGO and 2P-SiO_x/rGO retain a value of 340 and 424 mAhg⁻¹, respectively at 0.1 Ag⁻¹ after C-rate testing, while 291 mAhg⁻¹ for the un-doped electrode, indicating an enhancement of the cyclability and rate performance of the composite.

3:00pm TS1-2-MoA-5 The Research of Different Pre-Lithiation Methods to Enhance Coulombic Efficiency of SnO₂ Modified TiO₂ as Anode Material in Lithium-Ion Battery, *Cheng-Hsun Ho* (*n56124032@gs.ncku.edu.tw*), *J. Huang*, National Cheng Kung University (NCKU), Taiwan; Y. Shen, Hierarchical Green-Energy Materials Research Center (Hi-GEM), Taiwan

Lithium-ion batteries have been widely applied in our daily lives and there is an ongoing demand for LIB with higher energy density, lower selfdischarge, longer cycling life and better safety. Titanium dioxide (TiO2) has emerged as a highly promising anode material for lithium-ion batteries due to its remarkable cycling stability, impressive rate performance, costeffectiveness, and environmental friendliness. Nevertheless, the main obstacles associated with this material include its limited electronic/ionic conductivity and lower theoretical capacity. In order to overcome this issue, our research has been modifying high-capacity material (e.g., SnO₂) into TiO₂ with an eye to promoting its theoretical capacity. Modifying SnO₂ into TiO₂ reduces the impedance and increases the Li-ion diffusion rate. The SnO₂/TiO₂ composite is synthesized by the chemical bath with Sn(BF₄)₂, HBF4, Na₂S₂O₃ and TiO₂ (rutile and anatase mixed phase) followed by annealing at different temperature. From the TEM and XRD results, the SnO₂/TiO₂₋ composite had been successfully synthesized. It can be inferred from the figure that the structure of the composite might be the shell of the SnO_2 & TiO_2 solid solution while the core remains TiO_2 structure. However, the table below shows the serious fading problem of SnO₂/TiO₂ composite with different annealing temperature during the first and second cycle. Active lithium loss (ALL) in the initial lithiation process causes irreversible capacity of LIBs due to the formation of unstable solid electrolyte interface (SEI) layer on the electrode surface. To resolve the problem, pre-lithiation has been widely accepted as one of the most promising strategies to compensate for active lithium loss. Our group have been dedicated to trying various pre-lithiation techniques, such as electrochemical and thermal evaporation of lithium. Electrochemical prelithiation aims to generate stable SEI layers by applying constant voltage while lithium evaporation deposition involves depositing various thickness of lithium to achieve different levels of pre-lithiation. The ultimate goal

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would be giving out advantages and challenges of each one then finding out the optimal approach to improve cycling performance of tin-based anodes in lithium-ion batteries.

3:20pm TS1-2-MoA-6 Study on the Characteristics of Garnet-Type Solid Electrolytes in Lithium Metal Solid-State Batteries with Multilayer Interfaces, Hung-Ju Chen (m46111060@gs.ncku.edu.tw), J. Hung, S. Lin, National Cheng Kung University (NCKU), Taiwan

Lithium-ion batteries (LIB), which is a rechargeable battery, mainly used in related electronics industries, such as transportation vehicles, medium to large uninterruptible power systems (UPS), solar systems, energy storage systems, electric hand tools, aerospace equipment, power battery market and aviation battery. The LIB life would decrease with increasing in the number of charge and discharge cycles. Besides, the risk of LIB electrolyte is a liquid organic solvent, which is easy to burn and explode in case of fire. Recently, solid-state electrolytes have attracted attentions and show the many advantages including high safety, high energy density, and greater temperature tolerance. Among different types of solid-state electrolytes, garnet-type LLZTO (Li_{6.75}La₃Zr_{1.75}Ta_{0.25}O₁₂) electrolyte has high ionic conductivity ($10^{-3}to10^{-4}S$ / cm) and chemical stability towards lithium metal. However, lithium dendrites will be generated during the charge and discharge process of solid electrolyte batteries. The dendrites could lead to short circuit and failure of battery. Moreover, poor contact in the interface between solid electrolyte and lithium metal could cause the increased impedance and decreased conductivity. The aim in this study is to use different types and proportions of metal fluorides to modify the interface; meanwhile, the interface forms an electronically insulating and lithiumfriendly lithium fluoride layer to promote the chemical diffusion of lithium in lithium metal alloys and reduce interface impedance. After the project, we expect that a new generation of lithium metal solid-state batteries will be developed.

4:00pm TS1-2-MoA-8 Characterization Study of Sustainable Lithium Ion Battery with Cathode of Recycled LiNi_xMn_yCo_{1-x-y}O₂ (NMC), Yi-Chieh Tseng (e54101161@gs.ncku.edu.tw), National Cheng Kung University (NCKU), Taiwan; Y. Shen, Hierarchical Green-Energy Materials Research Center, Taiwan; J. Huang, National Cheng Kung University (NCKU), Taiwan

Nowadays, to meet the requirements of applying on portable electronic devices and electronic vehicles, energy storage system need to have high energy density and high power density. LiNixMnyCo1-x-yO2 (NMC) as the cathode for lithium-ion batteries (LIBs) could fulfil the demands. However, NMC is considered not friendly to the environment and harmful to man. To reduce the mining of poisonous, we had recycled the 622 and 811 NMC powder from the used battery of the electrical racing cars. By ultrasonic washing the electrodes in sodium chloride (NaCl) solution, the powder containing lithium (Li), nickel (Ni), magnesium (Mn), cobalt (Co), iron (Fe), aluminum (Al) and copper (Cu) is obtained. To remove the undesirable elements (Fe, Al and Cu), we washed the powder with various acids and used the Cyanex 272 to extract the target elements. From the X-ray diffraction (XRD) pattern, the recycled 622 and 811 NMC powder showed the characteristic peak of Ni, Mn and Co, proving the effectiveness of the extraction method. It was then assembled into coin cell (CR2032), still showing stable and relatively high capacity performance. In situ techniques are also used to examine the stability of the battery by recycled material, to be commercialized in the future. As the sustainability is the key requirement to be commercialized, the recycled battery with NMC as the cathode would have strong competitiveness.

4:20pm TS1-2-MoA-9 Investigation of Y-doped Li₇La₃Zr₂O₁₂(Y-LLZO) Coatings by Colloidal Coating Process for the Electrolyte of all Solid-state Battery, Yen-Yu Chen (yychen@mail.npust.edu.tw), G. Yao, National Pingtung University of Science and Technology, Taiwan; X. Yan, Chinese Culture University, Taiwan

Lithium ion batteries (LIBs) were widely applied on computer, communications, and consumer electronics for decades, as well as on the electric vehicles in recent years. Due to the risks of liquid electrolytes for current LIBs, solid state electrolytes for LIBs were investigated. In this study, Y-doped Li₇La₃Zr₂O₁₂ (Y-LLZO) materials were prepared by a solid-state reaction method. After well-dispersed, Y-LLZO coatings were deposited on the LiCoO₂/Y-LLZO composite anode from the Y-LLZO suspensions by a spin-coating method. The crystal phase analysis by the X-ray diffraction (XRD) method shows, the cubic perovskite phase of Y-LLZO can be obtained after calcined at 900°C for 12 h. The grain size of the Y-LLZO powders observed by the scanning Electron Microscopy are most within several µm. The crystal phase of the Y-LLZO samples after sintered at 950°C for 1 h is still mainly perovskite phase but become the tetragonal structure. A few of

minor La₂Zr₂O₇ phase can be found in the sample, that may due to the generation of the volatile lithium species during sintering. The Y-LLZO sample with Al₂O₃ sintering aid shows higher density and larger grain size after sintering. The thickness of the Y-LLZO coatings are around several μ m. The detail electrical properties will be shown in the following report.

Keywords: All solid-state lithium ion battery, LLZO, coatings, colloidal process, solid electrolyte

4:40pm TS1-2-MoA-10 Higher Power Density NVPF-CNT@Ni//CNF@Ni Configuration via Hydrothermal Route for Flexible Na-ion Capattery, Gagan Kumar Sharma (gkumarsharma@ph.iitr.ac.in), A. Pramanik, Rice University, USA; D. Kaur, Indian Institute of Technology (IIT) Roorkee, India; P. Ajayan, Rice University, USA

Considering the electrochemical performance and structural stability of nanohybrids, Na₃V₂(PO₄)₂F₃ (NVPF), multi-walled carbon nanotubes (MWCNTs), and carbon nanofibers (CNF) are emerging materials to promote the flexible sodium-ion capattery (FNIC) as a hybrid energy storage system. Direct incorporation of hydrothermally synthesized NVPF into MWCNT nanoarrays has been achieved on foldable nickel (Ni) foil via the slurry casting approach. We report an efficient utilization of the synergistic effect of ball-milled nanocomposites to design the FNIC cell from NVPF-MWCNT@Ni (battery-type) and CNF@Ni (capacitor-type) electrodes. The as-obtained FNIC device exhibits a remarkable specific capacitance of 136.17 F g⁻¹ with a corresponding specific capacity of 26.48 mAh g⁻¹at a potential scan rate of 1 mV s⁻¹. The charge storage ability is mainly viewed as the synergism stimulated across unique interfacial surface construction provided by NVPF and carbon-derived conductive host materials. Subsequently, the constructed asymmetric NVPF-MWCNT@Ni//CNF@Ni architecture exhibits a maximum working voltage of 0.70 V and a noticeable power density of 15.84 kW kg⁻¹ at 2.50 Wh kg⁻¹. The abundance conducting pathways in hybrid nanoarrays of facilitates intercalation/deintercalation of Na⁺/SO4⁻² ions into the electroactive sites, further endorsing pseudocapacitance. Besides, the flexibility test outcomes illustrate practically unperturbed electrochemical properties at a 160° bending angle, indicatingexcellent mechanical robustness. The FNIC cell retains90% of initial capacitance over 2,000 charge-discharge cycles, revealing a longer service lifespan attributed to advanced rate capability. The current study offers new avenues to develop energy storage systems for next-generation portable and wearable electronics at large-scale applications.

5:00pm TS1-2-MoA-11 Technological and Economical Aspects of Precious Metal Sputtering on Full-Size PEM Electrolyzer Components, Alexander Wemme (wemme.alexander@vonardenne.com), R. Stock, VON ARDENNE GmbH, Germany; C. Simons, Materion Advanced Materials, Germany; D. Fuller, Materion Corporation, USA

Sputter coating is a well-established process to produce thin layers with excellent properties, which can be used to replace electroplating of electrolyzer components with far less material consumption, hence minimizing cost of coating and thus the overall cost of the electrolyzer stack.

With precious metal material prices constituting up to 98% of the operating cost of a sputter coating equipment, the focus of production control shifts from minimizing downtimes and maximizing output to mitigate material losses as well as possible. Coating operations and the sputter target supply chain have to be evaluated under completely different aspects.

MATERION and VON ARDENNE have cooperated in implementing processes and technologies to drive this paradigm shift in cost structure by developing full-scale rotatable precious metal sputter targets.

The development of precious metal rotary targets will play a dominant role to achieve high utilization of target material thus keeping precious metal input and finance at low level in comparison to planar targets. The focus was to establish a target manufacturing process specifically designed for precious metals offering high homogeneity of target material, perfect sputtering behavior and layer formation. An additional objective was to maximize precious metal utilization by implementing an efficient recycling process.

In this presentation, the results of this recent work to make sputtering of precious metals economically advantageous will be discussed. Additionally, the practical implementation of such sputter targets in coating tools with hardware specifically designed to complement the precious metal life cycle from target fabrication over coatings at industrial scale to recycling is presented.

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Coatings for Batteries and Hydrogen Applications III

Moderators: Nazlim Bagcivan, Schaeffler Technologies GmbH & Co. KG, Germany, **Chen-Hao Wang**, National Taiwan University of Science and Technology, Taiwan

8:00am TS1-3-TuM-1 Oxygen Vacancy in Atomic Metal Oxide Clusters Demonstrate Outstanding Electrochemical Activity, Tsan-Yao Chen (chencaeser@gmail.com), National Tsing Hua University, Taiwan; K. Wang, National Central University, Taiwan INVITED Hierarchical structured heterogeneous catalyst comprising atomic metal oxide clusters with high contents of oxygen vacancy (O^{v}) and the carbon or Co oxide supported metal / oxide nanoparticle (NPs) is developed for electrocatalytic application. Such a catalyst processes a collaboration between $O^{v}s$ and the neighboring atoms in the electrochemical reaction. With this characteristic, the reaction kinetics of all steps are simultaneously operated consequent leading to a quantum leap on the current density and stability of the redox reaction. Apart from using noble metals, atomic scaled Co oxide clusters (CoO_x^a) were employed. Those clusters are decorated in surface defect regions of Co oxide supported Pd nanoparticles (CoOx-Pd) by using self-aligned nanocrystal growth followed by ultra-high-speed quench reaction with strong reduction agent. The decorated CoOx^a localize electrons from the neighboring atoms and thus boost the activity of CoOx-Pd in ORR. With a proper reaction time and loading control, the CoOx-Pd enhance its mass activity by 340 times as compared to that of commercial Pt catalysts in an alkaline electrolyte of 1.0M KOH.

8:40am TS1-3-TuM-3 Enabling Lightweight PEMFCs Based on PVD-Coated Aluminium Bipolar Plates for Aviation Applications, *Parnia Navabpour (parnia.navabpour@teercoatings.co.uk)*, *G. Sanzone, S. Field*, Teer Coatings Limited, UK; *K. Zhang*, University of Birmingham, UK; *H. Sun*, Teer Coatings Limited, UK

The aviation sector is a significant player in the global energy crisis and toward climate change. It currently accounts for 12% of transport-related CO₂ emissions and 2-3% of all anthropogenic emissions. To tackle this, targets have been set by various governments and organisations on aviation CO2 emissions and fuel efficiency. One tenable way to achieve these targets is through the use of hydrogen fuel cells for propulsion and/or auxiliary power units (APUs) in aircraft. The specific power of the fuel cell stack is a critical key performance indicator in the aviation industry, with bipolar plates being one of the most important components in a fuel cell stack, in terms of volume, weight and cost. Conventional PEMFCs utilise bipolar plates which are made from graphite or stainless steel. Hundreds of cells are needed within a multi-kW stack, hence a relatively small weight saving per plate will result in a large weight saving in the stack. The use of aluminium bipolar plates will enable the delivery of power densities double those of stainless steel bipolar plates. Challenges remain, however, with the corrosion of aluminium bipolar plates. This work focuses on the development of highly conducting and corrosion resistant coatings, aimed for PEMFC aluminium bipolar plates. The coatings were deposited using closed-field unbalanced magnetron sputtering technology and were evaluated for their adhesion and mechanical properties, as well as interfacial contact resistance and corrosion performance. Aluminium bipolar plates were coated and used in single cell fuel cells and tested under accelerated stress test conditions. The coated aluminium plates were compared with graphite plates and show the potential for using coated aluminium bipolar plates within PEMFCs.

9:00am TS1-3-TuM-4 Grazing Magnetron Sputtering of Cu_xO-MoS₂ Electrodes for Hydrogen Production, J. Castro, D. Cavaleiro, University of Coimbra, Portugal; M. Lima, University of Minho, Portugal; Albano Cavaleiro (albano.cavaleiro@dem.uc.pt), S. Carvalho, University of Coimbra, Portugal

The world energy grid faces a big issue in transit forward clean energy. Enlarging the possibilities to advance in cleaning the energy grid, humanity has made bids in several technologies, contemplating using Hydrogen as a sustainable and clean fuel. However, their production has important issues to overcome, such as the employment of expensive materials, which are non-abundant and challenging to obtain and process. Decreasing the manufacturing cost of electrodes used for producing Hydrogen could be a determinant to scale up this technology with competitive prices and, simultaneously, reduce the carbon footprint through affordable and environmentally friendly processes.

Copper is well known for its electrical properties; compared to other metals, it is cheaper and abundant. Recently, MoS2 has been demonstrated to favour the Hydrogen Evolution Reaction (HER). The present work presents the first insight into mixing these two materials using the grazing magnetron sputtering technique in a reactive atmosphere. C sheets and copper (sheets and foam) were used as substrates. SEM and EDS were used to determine the sample's morphologies and their chemical composition. Besides, several electrochemical techniques were employed to determine the electrochemically active surface area - ECSA, via linear sweep (LSV) and cyclic voltammetry (CV) and its electrochemical behaviour via electrochemical impedance spectroscopy (EIS). The results showed that including oxidised copper species together with MoS2 decreased the overpotential to start the HER at 10 mA/cm2 current density (~12% vs. the MoS2 overpotential). On the other hand, the sample with copper oxidised in a zig-zag configuration showed the highest double-layer capacitance and, hence, the highest ECSA, meaning that the obtained morphology during the deposition, influenced the electrochemical activity significantly.

Topical Symposium on Sustainable Surface Engineering Room Palm 5-6 - Session TS2-TuA

Sustainable Processing and Materials Selection for Surface Solutions

Moderators: Jörg Vetter, J.Vetter-S3-consulting, Germany, Fan-Bean Wu, National United University, Taiwan

1:40pm TS2-TuA-1 Microplasma-Enabled Upcycling for Nanomaterials Synthesis and Applications, Wei-Hung Chiang (whchiang0102@gmail.com), National Taiwan University of Science and Technology, Taiwan INVITED

Microplasmas are a special class of electrical discharges formed in geometries where at least one dimension is less than 1 mm. As a result of their unique scaling, microplasmas operate stably at atmospheric pressure and contain large concentrations of energetic electrons (1-10 eV). These properties are attractive for a range of nanomaterials synthesis and nanostructure engineering such as metal nanostructures and semiconductor nanomaterials [1]. Recently, we found that the energetic species including radicals, ions and electrons generated in the microplasmas were capable of initiating electrochemical-assisted reactions for the nucleation and growth of graphene quantum dots (GQDs), silicon quantum dots (SiQDs), and metal nanoclusters (MNCs). Moreover, we discover a simple and controlled synthesis of metal/metal, metal/QD heterostructures using our unique microplasma engineering. In this presentation, I will discuss these topics in detail, highlighting the advantages of microplasma-based system for the synthesis of well-defined nanomaterials for emearging appplications including detections of SARS-CoV-2 proteins [2], cancer and neurotransmitter biomarkers [3, 4], drug delivery [5] and environmental applications such as clean water production [6] and CO₂ adsorption [7]. These experiments will aid in the rational design and fabrication of nanomaterials for nanotechnology-enhanced biosensors and may also have significant impact in emerging applications for next generation biomedical applications.

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2:20pm TS2-TuA-3 Enhancing Hydrogen Production in 2D Materials via Surface Modifications: An Atomistic Study, N. Khossossi, S. Sagar, Poulumi Dey (p.dey@tudelft.nl), TU Delft, Netherlands

Hydrogen (H2) is one of the most potential candidates of sustainable energy produced in an eco-friendly manner. However, there are several challenges to be met before realization of H2 as an energy source. Known bottlenecks are slow kinetics and high overpotential associated with Hydrogen Evolution Reaction (HER), inefficient storage and H2 induced mechanical degradation of structural materials e.g., steels. To establish a viable 'H2-based economy', such bottlenecks could be addressed by designing materials with enhanced properties. To this end, the development of strategies for surface modification e.g., single-atom catalysts (SACs) supported on two-dimensional (2D) materials, are highly desirable. In this study, we perform Machine Learning (ML) assisted highthroughput screening of SACs supported on a 2D Ga-based system to expedite the prediction of HER overpotential. Firstly, Density Functional Theory (DFT) calculations are performed to investigate the catalytic properties of the system for HER. Our results reveal that, akin to many other 2D materials, the pristine Ga-based system is inert for HER due to its weak affinity towards hydrogen. However, the defective Ga-based system with surface sulfur-vacancy, exhibits highly desirable HER catalytic activity. Subsequently, we demonstrate the ML-accelerated prediction of HER overpotential for all transition metals on the system. By leveraging DFT calculations performed on 14 distinct SACs, we put forward a ML based model that maps the HER overpotentials to the atomic properties of the corresponding SACs. The trained ML model exhibits exceptional prediction accuracy and significantly reduces the prediction time compared to DFT calculations. Moreover, we identify an intrinsic descriptor that elucidates the relationship between the atomic properties of SACs and the overpotential. Our study thus provides valuable insights and a robust methodology for screening SACs on 2D materials, facilitating the design of high-performance catalysts for HER.

2:40pm TS2-TuA-4 Surface Wettability Modification of Polymers for Use in Electrocaloric Heat Pumps, Maria Barrera (maria.isabel.barrera.marin@fep.fraunhofer.de), Fraunhofer FEP, Germany; D. Pinkal, M. Wegener, Fraunhofer IAP, Germany; F. Fietzke, Fraunhofer FEP, Germany

Electrocaloric (EC) materials show a reversible temperature change in response to an external electric field, under adiabatic conditions [1]. In Germany, six Fraunhofer Institutes are working on the development of a refrigerant-free, energy-efficient electrocaloric heat pump, in which the heat transfer is done through latent heat when a fluid evaporates or condenses on the EC material. Due to their relatively high electrocaloric activity and mechanical flexibility, relaxor ferroelectric terpolymers such as P(VDF-TrFE-CFE) are being used as single thin films of a few micrometers thickness as well as multi-layer components to increase the thermal mass of the system [2].

In general, polymers have an intrinsic hydrophobic surface, which might have drawbacks for some applications. As an approach to overcome the poor surface wettability of the polymer components, there is a motivation to use superhydrophilic polyimide (PI) films for encapsulating the EC components, producing a complete wetting of the surface by the working fluid and hence improving the latent heat transfer for optimal performance of the electrocaloric heat pump. Additionally, PI also exhibits outstanding heat resistance and excellent chemical resistance [3], which makes it suitable as insulating material when the application of relatively high electric fields is required for inducing electrocaloric activity on the terpolymers.

For this purpose, the wettability of 12.5 μ m thick PI films (Upilex-S, Ube Industries) has been modified by pulsed magnetron sputtering under O₂ atmosphere, combining surface roughening and deposition of metal oxide layers. It has been found that the hydrophilic nature of the coatings together with the appropriate surface topography ensure a durable superhydrophilic performance of the flexible PI foils for (up to now) more than 290 days after treatment.

Results about the influence of composition variation, treatment time, material deposited, and surface roughening on the wetting properties of the PI foils will be presented. Finally, the integration of the encapsulating PI film with the EC polymer components is discussed.

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3:00pm TS2-TuA-5 High Volume Coating of Metallic Plates for Hydrogen Applications–A Challenge for Coating Machine Builders, *Philipp Immich* (*pimmich@hauzer.nl*), *R. Bosch, K. Fuchigami, R. Jacobs, T. Karla, P. Broekx,* IHI Hauzer Techno Coating B.V., Netherlands; *T. Hurkmans, J. Ummels, F. Schuivens*, IHI Ionbond AG, Netherlands

The hydrogen market is growing rapidly. The industry is developing for technical solutions for hydrogen generation and hydrogen-based electricity generation for mobile and stationary applications, and universities and institutes are investigating solutions for the long term. Today's challenge is to bridge the gap between current low to medium technology maturity level and market demand: how to be able to produce hydrogen on large scale and how to scale fuel cell production to high volumes? IHI Hauzer and IHI lonbond are working on this challenge for many years, developing low cost coatings to supply to the market either by machine solutions and

coating services. Key components of electrolyzers and fuel cell stacks like bipolar plates, PTL sheets and CCM's need high quality coatings to enable good catalyst performance, good electrical conductivity and good corrosion properties. For bipolar plates and PTL sheets, Hauzer and Ionbond have developed coatings based on PVD and DOT technology. In the presentation, both technologies will be addressed, including the current status of market introduction and our expected further roll-out within the next years. For PVD, the current main challenges related to machine and process solutions for high speed inline coating will also be addressed. For DOT technology the current main challenges are related to upscaling production capacity in the near future and optimizing precious metal use further.We will further address the requests from the market especially the electrolyzer business and give an outlook about possible solutions to serve these demands.

4:00pm **TS2-TuA-8** Iron Aluminide-Based Coatings as Sustainable Alternative for High Temperature Wear Protection, H. Rojacz, K. Pichelbauer, M. Rodriguez Ripoll, AC2T Research GmbH, Austria; G. Piringer, University of Applied Sciences Burgenland, Austria; P. Mayrhofer, TU Wien, Institute of Materials Science and Technology, Austria; Carsten Gachot (carsten.gachot@tuwien.ac.at), Vienna University of Technology, Austria

Strengthened iron aluminides show excellent mechanical properties up to 600°C. Therefore, coatings based on the intermetallic phase Fe₃Al are promising candidates to replace Co-, Cr- and Ni- rich coatings; critical raw materials with a high ecological impact. Different strengthening mechanisms can be used in order to increase the hardness of such coatings. Silicon can be used to for solid solution strengthening, whereas carbon as well as the combination Ti and B can be used to precipitate hardphases, intended to result in increased wear resistance. In this study, the influence of different amounts of alloying on the processing and moreover the wear resistance was evaluated. A thorough analysis of the materials and the present phases was conducted, using scanning electron microscopy, electron backscatter diffraction, hot hardness testing, nanoindentation as well as high temperature abrasion testing. Results show that the hardness can be significantly increased from ~260 HV10 to ~ 350 HV10 via solid solution strengthening with silicon or TiB₂ precipitations. Over 405 HV10 can be achieved by precipitating perovskite-type carbides Fe₃AlC_{0.6}. Hot hardness results show a good stability of the coatings >500°C. The wear results show a significant reduction of abrasive wear at high temperatures when strengthened, leading to lower wear rates at elevated temperatures due the increased formation of mechanically mixed layer. The obtained wear rates were used to estimate a lifetime utilised for ecological impact calculations from cradle to gate to compare the developed coatings with other wear protection coatings. Here, a reduction of the ecological impact of ~80% compared to cobalt based coatings can be assessed, showing the high potential of iron aluminide-based claddings as high temperature wear protection.

Wednesday Afternoon, May 22, 2024

Topical Symposium on Sustainable Surface Engineering Room Palm 5-6 - Session TS5-WeA

Circular Strategies for Surface Engineering

Moderators: Marcus Hans, RWTH Aachen University, Germany, Nina Schalk, Montanuniversität Leoben, Austria

2:00pm TS5-WeA-1 Coated Cemented Carbides – Tooling a Sustainable Future, Uwe Schleinkofer (uwe.schleinkofer@ceratizit.com), C. Czettl, CERATIZIT Austria GmbH, Austria INVITED

The demand for high performance manufacturing process in industries like aerospace, automotive and general engineering led to very productive and rapidly developing chip removal processes. Thuss coated cemented carbide cutting tools were developed during the last decades significantly to reduce costs and increase productivity. In addition to these needs, the sustainability aspects of such tools in terms of manufacturing and application got more and more in focus. One of the biggest contributors here is the raw material, mainly the element Tungsten for the Tungsten Carbide used in the cemented carbide. Examples of recycling technologies and closed loop scenarios to get back the worn-out tools will be summarized to show the state of the art. The influence of tailored microstructures and properties of coatings play a decisive role for the durability and performance of the tool. Furthermore, the metal cutting applications will be discussed to discuss the main factors influencing the sustainability in the chip removal process. The energy consumption of the machine is related to that of cooling pumps and additional aggregates. Examples are shown how innovative machining strategies can contribute to a more sustainable future.

2:40pm TS5-WeA-3 Designing Selective Stripping Processes for Al-Cr-N Hard Coatings on WC-Co Cemented Carbides, A. Kretschmer, V. Jaszfi, V. Dalbauer, TU Wien, Institute of Materials Science and Technology, Austria; V. Schott, S. Benedikt, A. Eriksson, Oerlikon Balzers, Liechtenstein; A. Limbeck, TU Wien, Austria; Paul Heinz Mayrhofer (paul.mayrhofer@tuwien.ac.at), TU Wien, Institute of Materials Science and Technology, Austria

Hard coatings like (AI,Cr)N are commonly used to protect cemented carbide cuttings tools from wear and corrosion. While such coatings increase the tool-lifetime, they also hinder the recycling effort of damaged tools. We report a new recycling strategy for coated cemented carbide tools by applying an interlayer between an arc evaporated Al_{0.7}Cr_{0.3}N coating and the WC-Co substrate. By selectively dissolving this interlayer in a concentrated basic solution, the coating spalls off the substrate, which is left intact. Four bases have been tested as saturated solutions: LiOH, NaOH, KOH, and CsOH, of which NaOH showed the highest reactivity. It was therefore used for subsequent investigations. Three different methods have been tested: (1) a metallic Al-doped interlayer region between substrate and coating can be removed in 3 h at 140 °C. Depositing an (2) Al_{0.7}Cr_{0.3} or (3) Al_{0.9}Cr_{0.1}N interlayer leads to a significantly faster removal compared to the first method. Hereby a substrate could be fully de-coated in 4 h at only 110 °C. The reaction time was also decreased by pre-treating the samples in concentrated HCl, HNO₃, or H₂SO₄ solutions at 90 °C for 10 min before subjecting the samples to the NaOH bath. Such treated substrates were fully de-coated in 3 h at only 90 °C. While HNO3 oxidized the substrates significantly, HCl and H₂SO₄ caused only negligible substrate damage.

3:00pm TS5-WeA-4 Perspectives on Sustainability of Coated Metal Cutting Tools, Lars Johnson (lars.johnson@sandvik.com), Sandvik Coromant R&D Materials and Processes, Sweden INVITED

Effects of climate change due to the emissions of greenhouse gases (GHG) from human activity is increasingly apparent, as predicted by climate science. Therefore, there is urgency in reducing the emission of GHG globally to meet the Paris Agreement goals of limiting the level of global warming. For the manufacturing industry, the last couple of years have been focused on the setting of targets in agreement with these goals, using frameworks as the Science Based Targets Initiative [1]. For example, Sandvik group has set targets of reduction of scope 1 and 2 GHG emissions to 50 % by 2030, 90 % by 2040, and net zero for scopes 1, 2, and 3 by 2050. To meet these ambitious yet necessary targets, improvements must be made across the full value chain; from suppliers and production, to customers' use of and the recycling of our products. Coated metal cutting tools are an interesting example to consider for the coating field, as the coating of tools have greatly enhanced their performance and service lifetime over the last half-century. Here, perspectives of the improvement in energy efficiency

and reduction of GHG emissions will be discussed. Starting from the deposition process itself, where recent reports on low-temperature deposition indicates that significant reductions in energy usage are possible [2]. Yet, as further improvements are needed, implications of how the coating equipment field needs to develop will also be considered. Another perspective is the impact of performance of the total GHG emissions over the lifetime of the tool at the user, where its performance and stability determine the total contribution to the emission when manufacturing a component. Finally, the need for reuse and recycling of used tools is also considered, which also greatly affects their sustainability. In closing the need for holistic approaches will be emphasized, to avoid suboptimizations, as it is our collective need and our collective improvements that will determine to what level we can mitigate climate change.

1. https://sciencebasedtargets.org/

2. Li, Xiao, Toward Energy-efficient Physical Vapor Deposition: Routes for Replacing Substrate Heating during Magnetron Sputtering by Employing Metal Ion Irradiation, Linköping University 2023, https://doi.org/10.3384/9789180752428

3:40pm **TS5-WeA-6 Towards Responsible Surface Engineering**, *Marcus Hans (hans@mch.rwth-aachen.de)*, *J. Schneider*, RWTH Aachen University, Germany; *A. Matthews*, University of Manchester, UK; *C. Mitterer*, Montanuniversität Leoben, Austria

Surface engineering comprises technologies, which enable improved structural and functional surface properties. Plasma-assisted physical vapour deposition (PVD) covers a set of advanced plasma-assisted surface engineering technologies, increasingly employed to address global challenges, such as reduction of CO₂ emissions. Despite the smaller volume and mass of thin films and coatings compared to bulk materials as well as the relatively low synthesis temperatures of PVD compared to other surface engineering technologies, PVD processes are often resource-intensive. In this review we critically evaluate two important questions in this context:

- 1. How sustainable are PVD processes and materials?
- Which pathways are needed for responsible surface engineering?

The consideration of energy and mass balances demonstrates that state-ofthe-art PVD processes and materials are not necessarily sustainable. Responsible surface engineering comprises pathways to enhance the sustainability of processes as well as materials and involves a change in mindset of materials scientists and process engineers.

4:00pm TS5-WeA-7 Reprocessing High Performance Cutting Tools -Performance Plus with Dedicated Coating Solutions, Dominik Blösch (d.bloesch@platit.com), C. Krieg, PLATIT AG, Switzerland; J. Kluson, PLATIT a.s., Czechia; H. Bolvardi, A. Lümkemann, PLATIT AG, Switzerland; B. Torp, PLATIT Inc., Switzerland INVITED In the last decades, the research and development within surface engineering has focused mainly on the enhancement of surface properties by the design of multifunctional coatings and surfaces, while the sustainability of such processes and products was typically neglected. The approach of a circular economy for surface engineering requires innovative rethinking along the lines of reducing, reusing, repairing, and recycling. In this contribution we would like to introduce circular economy strategies for surface engineering as follows:

 Sustainability with in-house PVD production: By integrating the coating process into the tool production plant, shipping between job coating centers and production places becomes superfluous, effectively reducing CO₂ production through optimized logistics. Furthermore, one major benefit of an vertically integrated coating center is having the PVD process including all aspects of the process chain on site. This permits functional coatings to be developed for specific applications, bringing us to the next point...

2. Extend usage by "Dedicated development": Using flexible inhouse PVD technology including pre- and post-treatment decoating and washing, the life of the coated part is maximized. Each step in the process chain is optimized individually. The lifetime of the coated parts for each specific application can thereby be extended rather than applying the typical job-coating business strategy where "one size fits all". The full potential of even the best PVD coatings cannot be exploited when making compromises in terms of coating type, coating thickness and plasma treatment.

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3. Resource conservation by reprocessing to restore original production quality: Worn tools are refurbished several times by resharpening just prior to reaching end-of-life, effectively preventing scrap. One challenge of reprocessing (de-coating, resharpening, cutting edge preparation, washing, coating, postpolishing) is removal of the worn and often partially oxidized PVD coating without damaging the base material below. Leveraging know-how for each individual process step, we achieve the original manufacturing quality in tools to enable multiple service lives.

Using this threefold approach of vertical integration, dedicated development, and reprocessing, substantial improvement to the sustainability of PVD-coatings is achieved without necessitating any compromise on quality.

Thursday Morning, May 23, 2024

Topical Symposium on Sustainable Surface Engineering Room Town & Country D - Session TS4-1-ThM

Coatings and Surfaces for Thermoelectrical Energy Conversion and (Photo)electrocatalysis I

Moderators: Clio Azina, RWTH Aachen University, Germany, Carlos Tavares, University of Minho, Portugal

8:00am TS4-1-ThM-1 Inorganic Thermoelectric Films for Harvesting Waste Heat Near Room Temperature: Opportunities and Challenges, *Rui Shu (rui.shu@liu.se),* Linkoping University, Sweden, USA INVITED The abundance of untapped low-grade heat presents a significant

opportunity for sustainable energy solutions. However, current recovery technologies often fall short due to their lack of cost-effectiveness. Effectively harnessing this waste heat holds the promise of fostering a more sustainable society. Notably, inorganic thermoelectric bulk materials have shown promise in capturing waste heat near room temperature, offering potential applications in energy harvesting and thermal cooling.

However, challenges persist, including limited thermoelectric performance when transitioning to thin films and hindered large-scale implementation due to the scarcity and costliness of elemental Te. Adapting highperformance inorganic bulk thermoelectric materials into consistent thin films remains a significant obstacle.

This presentation explores the opportunities and challenges associated with using inorganic thermoelectric films for waste heat recovery at room temperature. Recent research advancements focusing on materials like Mg₃Bi₂ and transition-metal-nitride-based thin films are highlighted. Notably, these materials circumvent the use of scarce and brittle tellurium, with Mg₃Bi₂-based substitutes showing promise as alternatives to commercially used Bi₂Te₃. Our study aims to pave the way for the practical utilization of highly efficient inorganic thermoelectric films in energy harvesting applications.

8:40am TS4-1-ThM-3 Retaining Crystallinity of as-deposited Thermoelectric Fe₂VAI-based Thin Films Grown from DCMS and HiPIMS, *Ludwig Enzlberger (ludwig.enzlberger@tuwien.ac.at),* TU Wien, Institute of Materials Science and Technology, Austria; *S. Kolozsvari,* Plansee SE, Germany; *P. Mayrhofer,* TU Wien, Institute of Materials Science and Technology, Austria

Thermoelectric materials have gained much attention in recent years due to their ability to directly interconvert electrical and thermal energy via the Seebeck/Peltier effect. This can be used to convert waste heat back into usable electrical energy, making thermoelectrics very interesting materials in a world with increasing demand for renewable and efficient utilisation of energy. The efficiency of this process is generally dependent on three parameters - the thermopower *S*, the electrical conductivity σ and the thermal conductivity λ - which are represented together in the dimensionless Figure of Merit *ZT*.

Among thermoelectrics, Heusler and half-Heusler materials have shown to be promising candidates, due to their high Seebeck coefficients at room temperature and their high electrical conductivity, while generally higher thermal conductivity is often a drawback in thermoelectric performance.

In 2019, Hinterleitner *et al.* managed to produce magnetron sputtered thin films of bcc-Fe₂V_{0.8}W_{0.2}Al with an exceptionally high Seebeck coefficient, Power Factor and Figure of Merit, but the samples needed to be heat-treated for one week to crystallize from their initially amorphous state.

In this work, we present Fe₂VAl-based full-Heusler thin films retaining their crystallinity during sputter deposition. By tuning deposition temperature, bias potential and pulse on-time we managed to fabricate films of Fe₂V_{0.8}W_{0.2}Al in a W-type bcc-structure on silicon and austenite substrates. These films were analysed using XRD, EDX, electron microscopy and by measurement of transport data (resistivity, Seebeck coefficient). Thermal conductivity of the films was derived from measurements of thermoreflectance and specific heat capacity.

9:00am TS4-1-ThM-4 Thermoelectrical Investigations of TaC-Based Superlattice Protective Coatings, Barbara Schmid (barbara.schmid@tuwien.ac.at), S. Lin, T. Schöngruber, N. Koutná, TU Wien, Institute of Materials Science and Technology, Austria; S. Bühler-Paschen, TU Wien, Austria; L. Mitterhuber, Materials Center Leoben, Austria; D. Ingerle, TU Wien, Austria; S. Kolozsvari, Plansee SE, Germany; P. Mayrhofer, TU Wien, Institute of Materials Science and Technology, Austria Transition metal carbides (TMC) and nitrides (TMN) feature high melting points up to 4000 °C and superior thermal stability and are therefore regarded as ultra high temperature ceramics (UHTC). Those materials are well-established in the realm of protective coatings. Superlattice architecture can impact a plethora of different material characteristics such as mechanical, chemical and electric properties. Inspired by preliminary DFT high-throughput screenings, we developed TiC/TaC and TiN/TaC superlattice coatings exhibiting bilayer periods between 2 and 50 nm via non-reactive DC magnetron sputtering. Those materials exhibit high hardness and fracture toughness and are therefore immaculate choices as protective coatings. These superlattice materials also exhibit decent thermal stability and electrical conductivity, which motivated us to calculate their thermoelectrical properties via DFT using BoltzTraP and VASP. The experimentally determined Seebeck coefficients exhibit a significant bilayer-period-dependence. Ву conducting Time Domain Thermoreflectance investigations, we also derived their figure of merit (ZT) values.

9:20am TS4-1-ThM-5 Exploring the Potential and Challenges of Solution-Processed Inorganic Thermoelectric Materials, *M. Ibáñez*, Institute of Science and Technology Austria (ISTA), Austria; *Tobias Kleinhanns* (*Tobias.Kleinhanns@ist.ac.at*), Institute of Science and Technology, Austria INVITED

Over the past few years, there has been a significant surge in interest surrounding solution-based techniques due to their cost-effectiveness and scalability in the production of high-performance thermoelectric materials. This approach involves the synthesis of particles in a solution, followed by their purification and thermal processing to yield the desired dense polycrystalline material. In contrast to traditional methods, solution-based syntheses offer the ability to manipulate particle characteristics, including size, shape, crystal structure, composition, and surface chemistry, to an unprecedented degree. This fine-tuned control over powder properties opens up distinct opportunities for crafting thermoelectric materials with meticulously controlled microstructural attributes.

In this presentation, our primary focus will be on Ag2Se, an important thermoelectric material for harnessing thermoelectricity at or near room temperature, an area where the selection of high-performing materials is currently limited. While Ag2Se shows great promise, the main problems are the large discrepancy in the reported thermoelectric properties and difficulties in replicating its exceptional performance. These discrepancies often stem from the intricate control of defects within the material, such as vacancies, interstitial atoms, dislocations, grain boundaries, and precipitates.

We will show that our solution-based synthesis method enables precise defect control, especially avoiding fluctuations in stoichiometry. Additionally, we will illustrate how we can fine-tune microstructural defects, including strain, dislocations, and grain boundary density, leveraging the characteristic phase transition of Ag2Se during the sintering process. Our results will highlight that besides stoichiometry, the microstructure is crucial for tuning Ag2Se transport properties and how this control can be provided by our novel synthetic route. Furthermore, we will highlight the sustainability and scalability of our approach, where solvents can be recycled and energy consumption minimized, contributing to a more environmentally friendly production process.

10:20am TS4-1-ThM-8 3D Nanoscale Spatial Imaging of Doped ZnO and TiO₂ Transparent Thermoelectric Thin Films, J. Ribeiro, F. Correia, H. Faria, University of Minho, Portugal; A. Welle, T. Boll, Karlsruhe Institute of Technology (KIT), Germany; Carlos Jose Tavares (ctavares@fisica.uminho.pt), University of Minho, Portugal

Transparent thermoelectric materials are a promising technology for touchscreen displays and solar cell applications, rendering a sustainable powering of the device. In order to enhance the thermoelectric performance, the material must have a high Seebeck coefficient and high electrical but low thermal conductivity. This work focuses on the effect of

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doping on ZnO and TiO₂-based thin films deposited by DC magnetron sputtering. The properties of the films depend strongly on the dopant type and concentration. On the one hand, it has been documented that Al and Ga doping can improve the electrical properties in ZnO, as can Nb doping in TiO2. On the other hand, introducing heavier elements (such as Bi, Sb or Nb) into the metal-oxide matrix hinders phonon-mediated heat conduction, and consequently reduces the thermal conductivity, which is a promising approach. Atom Probe Tomography and Time-of-Flight Secondary Ion Mass Spectrometry are powerful tools to determine the composition and inherent homogeneity within the thin films, as well as to investigate the cation and anion segregations to interfaces and grain boundaries. For the ZnO-based films, Al and Ga dopants are homogeneously distributed within the crystals, with the exception of Bi, which is not incorporated in the ZnO wurtzite cell and segregates at the grain boundaries and at the triple junctions. Thus, Bi contributes to grain boundary scattering of phonons and contributes less to the reduction of the thermal conductivity, in comparison to Ga-, Al-, and Sb-doping in ZnO. For the Sb-doped ZnO thin films, a larger Zn content was registered at the triple junctions of the grain boundary. As for the Nb-doped TiO₂ thin films, Nb is homogeneously distributed into the TiO₂ matrix and no grain boundaries are visible. However, the composition varies depending on the deposition conditions, where the Nb content inside the film changes depending on the oxygen content controlled through the reactive O₂ flow during the sputtering depositions.

10:40am **TS4-1-ThM-9 Ni-B-based Polyalloy Electrocatalyst Coatings Deposited by MSPVD for Efficient Oxygen Evolution Reaction**, *Kubilay Sahin (ahink@uni.coventry.ac.uk)*, Institute for Clean Growth and Future Mobility, Coventry University, Department of Metallurgy, University of Mons (UMONS), UK; *V. Vitry*, Department of Metallurgy, University of Mons (UMONS), 23 Place du Parc, B-7000 Mons, Belgium., Belgium; *A. Cobley*, Institute for Clean Growth and Future Mobility, Coventry University, Priory St, Coventry, CV1 5FB, UK.; *J. Graves, G. Pourian Azar*, Institute for Clean Growth and Future Mobility, Coventry University, UK

Molecular hydrogen has been considered as one of the best green energy sources due to its high energy density ¹. Water splitting is a highly promising approach to generate molecular hydrogen without any damage to environmental health. However, electrocatalyst materials, which are generally expensive noble elements, are required to complete the reactions efficiently and sustainably. Due to their excellent features such as low cost, high abundance, high corrosion resistance and durability, catalytic activity, and good synergistic effect with other elements, Nickel-based electrocatalysts have been reported as one of the most valuable alternatives to expensive noble metals ². Electrodeposition, electroless plating, hydrothermal deposition, and physical vapour deposition are some of the used techniques to synthesize Nickel-based electrocatalysts. Electroless nickel-boron plating is a remarkably beneficial technique to produce Ni-B coatings with outstanding features such as hardness, wear resistance, and corrosion resistance. In recent studies, Ni-B coatings have also demonstrated encouraging catalytic activity with remarkable stability ³. While Ni-B coatings have been extensively studied for various applications, there is not enough research on their catalytic applications.Furthermore, there is no existing literature regarding the deposition of Ni-B coatings utilising a technique other than electrodeposition or electroless plating. However, the Magnetron Sputtering Physical Vapour Deposition (MSPVD) technique has the capability of producing Ni-B coatings with porous and tunable structures together with easy alloyability to further improve the electrochemical performance.

The current study is designed to investigate the electrocatalytic performance of Ni-B-based polyalloy coatings produced by MSPVD. Polyalloy coatings were co-deposited using a Ni-B and transition metal targets such as Fe, Co, and Mo. The coatings were deposited at different deposition parameters such as chamber pressure, substrate type and different chemical compositions. The electrocatalytic performance of the coatings was compared to see the effect of boron, alloying elements, morphology and crystal structure for the Oxygen Evolution Reaction. Superior features like low overpotentials, high stabilities, and high surface areas were obtained after the electrochemical analyses such as Linear sweep voltammetry, cyclic voltammetry, chronoamperometry and electrochemical impedance spectroscopy under alkaline conditions. The results showed promising efficiencies and stabilities for highly tunable, cost-effective Ni-B-based electrocatalyst coatings.

11:00am **TS4-1-ThM-10 Role of Grain Boundaries in the Stress Corrosion Cracking of Nanoporous Gold Thin Films,** *Aparna Saksena (a.saksena@mpie.de),* Max-Planck Institut für Eisenforschung GmbH, Germany; *A. El-Zoka,* Imperial College London, UK; *A. Saxena, E. Hatipoglu,* Max-Planck Institut für Eisenforschung GmbH, Germany; *J. Schneider,* RWTH Aachen University, Germany; *B. Gault,* Max-Planck Institut für Eisenforschung GmbH, Germany

For its potential as catalyst, nanoporous gold (NPG) prepared through dealloying of bulk alloys has been extensively investigated. NPG thin films can offer ease of handling and better tunability of the chemistry and microstructure of the nanoporous structure. They are however prone to intergranular cracking during dealloying, limiting their stability and potential applications. Here, we systematically investigate the grain boundaries in Au28Ag72 (± 2 at.%) thin films. We observe that a sample synthesized at 400 $^{\circ}\mathrm{C}$ is 2.5 times less prone to cracking than one synthesized at RT. This correlates with a higher density of coincident site lattice (CSL) grain boundaries, especially coherent Σ 3, increased, which appear resistant to cracking. Nanoscale compositional analysis of random high-angle grain boundaries reveals prominent Ag enrichment up to 77 at.%, whereas Σ 3 coherent twin boundaries show a Au enrichment of up to 30 at.%. The misorientation and the chemistry of grain boundaries have a crucial role in their dealloying behavior, which controls the cracking. Our results provide a target for optimizing the longevity application of NPG thin films for possible applications.

11:20am TS4-1-ThM-11 Metal/Oxide Heterostructure as Hydrogen Evolution Reaction Electrocatalyst, Thi Y Phung Nguyen (phungnguyen0398@gmail.com), National Cheng Kung University (NCKU), Taiwan, Viet Nam; J. Ting, National Cheng Kung University (NCKU), Taiwan In response to the growing demand for sustainable energy sources, there has been a concerted effort to develop efficient electrocatalysts for the hydrogen evolution reaction (HER). Previous researches indicate the need to improve consistent efficiency and stability at high current densities (\geq 500 mA/cm2) over 100 hours. In this study, we have investigated metal/oxide heterostructure HER electrocatalysts. The heterostructure is noble-metal free and synthesized using a hydrothermal method followed by thermal reduction for controlling the alloy/oxide ratio. Various characterization techniques, including scanning electron microscopy, transmission electron microscopy, X-ray diffraction, X-ray photoelectron spectroscopy, and in-situ Raman spectroscopy are used to examine the obtained heterostructures. Furthermore, the electrochemical performance is evaluated using linear sweep voltammetry, electrochemical impedance spectroscopy, cyclic voltammetry, and electrochemical specific surface area analyses. We demonstrate highly efficient and stable heterostructure HER electrocatalyst. This work contributes to cost-effective and sustainable hydrogen production, with significant implications for renewable energy integration.

11:40am **TS4-1-ThM-12 Copper-Based Porous Surfaces for Electrocatalytic CO₂ Reduction**, *Maria José Lima (mjlima@fisica.uminho.pt), S. Viana,* University of Minho, Portugal; *J. Castro, S. Carvalho,* University of Coimbra, Portugal

The United Nations (UN) has identified carbon dioxide (CO_2) as a greenhouse gas (GHG) that is present in the atmosphere as an environmental issue in Goal 13 for climate action.

Decreased CO₂ emissions and participation in a circular economy are crucial to achieving these goals. To include CO₂ in a circular economy, capture and electroreduction of CO₂ into long-chain hydrocarbons or alcohols (C2+) can be the solution. From a material perspective, copper-based catalysts are active and selective cathodes capable of producing hydrocarbons, increasing the Faradaic efficiency of the CO₂RR to C2+ molecules.

Enhancing electrochemical active sites can be an additional strategy to improve CO_2RR . Different strategies can produce porous electrodes, thus increasing the electrochemical active surface area (ESCA) and the CO_2 -catalyst interaction. One technique that produces porous materials is the anodization process, an electrochemical process capable of producing a thin oxide layer at the metallic surface.

In this work, we systematically optimized the anodization parameters of metallic copper (Cu) to develop Cu_xO_γ porous electrodes. Different anodization parameters were studied, such as voltage, time, electrolyte concentration, and distance between the anode and the cathode.

Materials characterization was made by SEM, EDS, and XRD, where it was possible to observe a higher porosity and Cu_2O crystalline phase obtained using 0.1 M of K₂CO₃ and applying 25 V for 15 min. In fact, lower applied

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potentials show a compact surface with cracks. Increasing the potential to 25 V favors the formation of hollow tubes, whereas a further increase to 50 V results in structure compaction.

Electrochemical analyses, such as linear sweep voltammetry and cyclic voltammetry, will be shown to discuss the involved redox reactions of organic and inorganic species. The ESCA calculation of each electrode material will permit an understanding of how it correlates with morphology.

Topical Symposium on Sustainable Surface Engineering Room Town & Country D - Session TS4-2-ThA

Coatings and Surfaces for Thermoelectrical Energy Conversion and (Photo)electrocatalysis II

Moderators: Clio Azina, RWTH Aachen University, Germany, Carlos Tavares, University of Minho, Portugal

1:40pm TS4-2-ThA-2 Multifunctional Materials for Emerging Technologies, *Federico Rosei (federico.rosei@units.it)*, University of Trieste, Italy INVITED This presentation focuses on structure property/relationships in advanced materials, emphasizing multifunctional systems that exhibit multiple functionalities. Such systems are then used as building blocks for the fabrication of various emerging technologies. In particular, nanostructured materials synthesized via the bottom–up approach present an opportunity for future generation low cost manufacturing of devices [1]. We focus in particular on recent developments in solar technologies that aim to address the energy challenge, including third generation photovoltaics, solar hydrogen production, luminescent solar concentrators and other optoelectronic devices. [2-40].

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2:20pm TS4-2-ThA-4 Enhanced Photoelectrochemical Water Splitting on ZnCo₂O₄ Electrodes in Chloroplasts Driven by Spin Injection, Chien-Yu Lin (n56111453@gs.ncku.edu.tw), Y. Su, National Cheng Kung University (NCKU), Taiwan

This work demonstrated the photoelectrochemical water splitting efficiency of spinel-structured $ZnCo_2O_4$ on carbon paper substrate as photoelectrode and also coating on chloroplasts. $ZnCo_2O_4$ is p-type transition metal oxide semiconductor and could be synthesized by hydrothermal method and different annealing temperature, showing nanoparticles in morphology. Furthermore, we extracted the chloroplasts from chlorella to make it coat on $ZnCo_2O_4$ delectrodes as protection layer, which also could be boosting the photosynthesis reaction when the water splitting process goes on. We observed the applied bias photon-to-current efficiency (ABPE) by changing spin quantum states, and the chloroplasts photoelectrochemical water splitting cell shows a splendid efficiency of hydrogen production. Accordingly, the device can be successfully applied on energy storage and conversion, suggesting the great potential of the applications in electronic, catalysis, and solar applications.

2:40pm TS4-2-ThA-5 Piezoelectricity-Assisted Photocatalyst of BiOBr-Based Composites on a Flexible Substrate, *Thi Nghi Nhan Nguyen* (*nghinhan2410@gmail.com*), *K. Chang*, National Cheng Kung University (NCKU), Taiwan

A novel 3D network of BiOBr flakes was grown on carbon fiber (CF) substrates through a straightforward chemical deposition process. The BiOBr-based composites served as catalysts for photodegradation and as photoelectrodes for photoelectrochemical cells. The p-n junction formation was determined by Mott–Schottky measurements which was also confirmed through high-resolution transmission electron microscopy and X–ray photoelectron spectroscopy. The piezoelectric properties of BiOBr were verified using piezoresponse force microscopy. The

photoelectrochemical performance of samples was assessed through various techniques, including linear sweep voltammetry, chronoamperometry

[https://www.sciencedirect.com/topics/chemistry/chronoamperometry], amperometry andcyclic voltammetry. Under simultaneous illumination and mechanical pressure, the Ag₂O/BiOBr composite demonstrated a photocurrent of approximately 20.0 mA cm⁻² at 1.23V, showcasing a remarkable enhancement over 4 and 20 times compared to individual BiOBr and Ag₂O, respectively. The maximum applied bias photon to current efficiency values of Ag₂O/BiOBr composite with external stress was approximately 2.7 % at 0.9V. Additionally, a glucose sensor based on Ag₂O/BiOBr composite exhibited a high sensitivity of 400 μ A cm⁻² mM⁻¹, within a detection glucose range of 0.1–12 mM. The Ag₂O/BiOBr-based photolectrodes showed excellent stability and repeatability in glucose was attributed to the p-n junction formation, piezoelectric potentials, substantial active surface area and advantageous band positions.

3:00pm TS4-2-ThA-6 Hydrothermal Synthesis of (Ba,Sr)TiO₃/AgBr Films and Their Application for the Visible-light Piezo-photocatalysis, Yen-Lun Chiu (0953065268v@gmail.com), K. Chang, S. Han, National Cheng Kung University (NCKU), Taiwan

Perovskite-nanostructured films are attractive because of their excellent characteristics. Different kinds of properties can be obtained, e.g., dielectric properties, piezoelectricity, and thermoelectricity, from different materials. With the hydrothermal fabrication, the perovskite materials can be synthesized in a facile way with lower power consumption. However, studies on this topic directly through hydrothermal processes for the fabrication of perovskite-nanostructured films are still lacking. In this study, well-aligned (Ba,Sr)TiO₃ nanorod arrays composited with p-AgBr were synthesized through the hydrothermal reaction for visible-light piezophotocatalytic application. The hydrothermal parameters, including concentrations of precursor solutions, reaction time, temperatures. different types of ion species, and the surfactants used for the reaction, were manipulated. X-ray diffraction and transmission electron microscopy were employed to determine the phase and microstructure of the resultant samples. The amplitude of the piezoresponse (d₃₃) was measured through a piezoresponse force microscope for the materials. The photoelectrochemical activity of the samples were also studied for related applications. An energy band diagram was constructed to elucidate a potential mechanism for the remarkable activity.

Keywords: perovskite-nanostructured films, hydrothermal, (Ba,Sr)TiO₃ nanorod arrays, p-AgBr, piezo-photocatalysis

3:20pm TS4-2-ThA-7 Advances in Piezo-Photothermal Effect Enhanced Photocatalytic Activities of Heterostructure Composites, Van Ty Tran (tranvanty108@gmail.com), D. Chen, National Cheng Kung University (NCKU), Taiwan

This study focusses on developing heterostructure composites to enhance the efficiency of piezoelectric and photothermal-assisted photocatalytic processes for pollutant degradation and photoelectrochemical water splitting. The Ag₂O/BiFeO₃ and CuS/MoS₂ composites were fabricated through a hydrothermal method. The morphologies and microstructures of the samples are analyzed using scanning electron microscopy (SEM), transmission electron microscopy (TEM), X-ray diffraction (XRD), and X-ray photoelectron spectroscopy. The composites exhibit a low band gap, indicating their capacity to absorb light in the Vis-NIR range. The conductive type of the samples and p-n junction formation is determined through Mott Schottky (M-S) measurements. The formation of a p-n junction facilitates the separation of electron-hole pairs, thereby improving the efficiency of the photocatalyst. Additionally, the induced piezoelectric potential in the piezoelectric material promotes photocatalytic activity by reducing the recombination of photogenerated charges. Under irradiation, the generated heat further supplies kinetic energy to photogenerated carriers, enhancing reaction rates in photocatalytic processes. The piezoelectric composite demonstrates the ability to produce $\bullet O^{2^-}$, $\bullet OH$, and h^+ through photocatalysis, effectively degrading pollutants like tetracycline (TC) and Rhodamine B (Rh B) through oxidation. The degradation efficiency of the TC solution was further increased to 95% for CuS/MoS₂ composite in 30 min, which was higher than that of individual components. Moreover, The Ag₂O/BiFeO₃ heterostructure exhibited excellent photocatalytic degradation of Rhodamine B and TC, and photoelectrochemical water splitting activity.

4:00pm TS4-2-ThA-9 Photoelectrochemical Properties of Chlorophyll Coating on Cu₂O Photocatalyst by Mediating Charge Transfer Characteristic, Yu-Teng Wu (wuyuteng22@gmail.com), Y. Su, National Cheng Kung University (NCKU), Taiwan

Metal oxide semiconductors have impressive applications in the field of photo electrochemistry. This study utilizes electrochemical deposition to generate nano-thin films of cuprous oxide, applying them in green energy sources. During the photoelectrochemical (PEC) process, cuprous oxide faces issues of instability and insufficient durability due to photo-induced corrosion in aqueous solutions. To address this, the natural photosensitizing material chlorophyll is adhered to enhance charge transfer efficiency and provide a better surface electric field distribution. Additionally, the chlorophyll layer effectively isolates the aqueous solution from direct contact with cuprous oxide, enhancing sample stability. Detailed research results, including atomic force microscopy (AFM) and electrostatic force microscopy (EFM) surface electric field analyses, along with electrochemical methods, confirm that Chlorophyll/Cu2O exhibits superior stability and durability, enhancing the overall value of this PEC cell.

4:20pm TS4-2-ThA-10 Ligand Modified Bimetallic Metal-Organic Frameworks Electrocatalysts for Urea Oxidation Reaction, *Hui Chuan Chen (jace52112@gmail.com)*, National Cheng Kung University (NCKU), Taiwan; *T. Nguyen*, National Cheng Kung University (NCKU), Taiwan, Viet Nam; *J. Ting*, National Cheng Kung University (NCKU), Taiwan

In the quest for energy efficiency, electrocatalytic urea oxidation reaction (UOR) is a promising alternative to oxygen evolution reaction (OER) due to the favorable thermodynamics, meanwhile, it is also an environmentally friendly strategy.

In this regard, metal-organic framework (MOF) materials have the advantages of high specific surface area, high porosity, structural adjustability, etc., providing abundant metal active sites to achieve high efficiency electrocatalytic performance. However, due to the poor conductivity of MOF, the charge transfer ability is limited. In order to improve the shortcoming, ligand having redox activity is introduced. This ligand can not only adjust the synergistic effect of metal clusters and organic ligands to increase the charge transfer ability, but also can be an additional adsorption sites to promote the adsorption/desorption ability of intermediates. In this study, we report ligand modified bimetallic MOF synthesized via a low temperature hydrothermal method, this optimized bimetallic MOFs exhibits an outstanding UOR performance with high catalytic activity, low resistance and excellent electrochemical stability.

Keywords: MOFs, urea oxidation reaction (UOR), electrocatalyst

Topical Symposium on Sustainable Surface Engineering Room Golden State Ballroom - Session TS1-ThP

Coatings for Batteries and Hydrogen Applications - TS1 Poster Session

TS1-ThP-1 Formulating Advanced Materials for Energy Storage using Composite Solid- State Electrolyte Incorporating Al-Doped LLZO (Al-LLZO) in NCM811 for Solid State Lithium- Metal Batteries, C. Yang, Ming Chi University of Technology, Taiwan; Adere Tarekegne Habte (aderetarekegn@gmail.com), Ming Chi University of Technology, Ethiopia The development of advanced materials for energy storage devices has been of utmost importance for the transition towards clean and renewable sources of energy. In this context, solid-state lithium metal batteries have shown promising features such as high energy density, good ionic conductivity, and safer performance than conventional batteries. The primary aim of this research work is to develop a free-standing composite solid-state electrolyte (CSE) that incorporates Al-doped Lithium Lanthanum Zirconium Oxide (LLZO) into NCM811 for solid-state lithium-metal batteries followed by solution casting method. The researcher used various characterization techniques such as XRD, FT-IR, EIS, and SEM to analyze the structural and morphological properties as well as the impedance of the solid-state composite electrolyte. This material gives 4.9 x 10⁻⁴ Scm⁻¹ionic conductivity at room temperature. The NCM811/CSEs/Li exhibited after 100 cycles a specific capacity of 125.35 mAhg-1 and ⁻¹ and a capacity retention of 99.08 % at ambient temperature and with the rate of 0.2C.

TS1-ThP-2 Corrosion Stability and Electrical Conductivity of PVD Coated Electrolyzer Bipolar Plates, *Martin Welters (welters@kcs-europe.com)*, KCS Europe GmbH, Germany; *N. Kruppe*, Schaeffler Technologies AG & Co. KG, Germany; *R. Cremer*, KCS Europe GmbH, Germany; *M. Öte, N. Bagcivan*, Schaeffler Technologies AG & Co. KG, Germany

Bipolar plates (BPP) are one of the essential components of electrolyzer stacks for the generation of pure hydrogen by water electrolysis. In order to replace conventional and expensive titanium based BPP, stainless steel becomes more and more important against the background of increasing production volume due to cost and functional reasons. On the other hand, some requirements exist, which limit the use of stainless steel BPP in electrolyzers. Firstly, due to harsh corrosive conditions, the application of the stainless steel BPP requires the protection against corrosion. Secondly, the electrical conductivity of BPP must remain constantly low over the entire service life.

As part of the H2Giga StaclE joint project, KCS Europe developed titaniumbased PVD protection layers for stainless steel BPP in order to fulfill the demanding requirements of water electrolysis. In addition to the functional suitability of the coatings, the economic viability and suitability for largescale production was kept in mind. Therefore, the coatings were deposited by means of a tailored inline coating system, applying PVD technology. The coating system enables an easy scale up to larger production quantities in future. Measurement of Interfacial Contact Resistance at 10 bar and Linear Sweep Voltammetrie of the coatings revealed promising results regarding corrosion stability and electrical conductivity prior and after electrochemical cell tests on laboratory scale adapting electrolyzer cell conditions.

TS1-ThP-3 PVD Core-Shell-Catalysts for Water Electrolysis, Jan-Ole Achenbach (achenbach@kcs-europe.com), KCS Europe GmbH, Germany; M. Berger, Institute of Technical and Macromolecular Chemistry, Germany; M. Pilaski, The Hydrogen and Fuel Cell Center - ZBT, Germany; R. Cremer, KCS Europe GmbH, Germany

The expected increasing demand of hydrogen in the upcoming years requires adequate electrolysis capacities in order to be able to serve the market. One technology for mass production of hydrogen is the alkaline polymer membrane water electrolysis (AEM-WE). Heart of AEM-WE are the catalysts, which enable the decomposition of water at low energy input. Therefore, the availability of sufficient and suitable catalysts is a limiting factor for high H₂ production volumes.

Within the research project H2Giga AlFaKat, KCS Europe follows up the approach of replacing the usual expensive catalysts containing precious metals by cheaper core-shell particles. Therefore, particles of a suitable low-cost material are activated by the deposition of a more expensive catalyst material by means of physical vapor deposition (PVD). For the realization, a coating demonstrator was developed and validated in the

project. Thereby, the basic suitability, the efficiency and useability for mass production of the concept as well as the coating growth, homogeneity and properties were investigated. Analyses of the core-shell-particles showed a homogeneous coating of the core particles after processing by PVD coating demonstrator. Further investigations regarding catalytic activity revealed high activity of new PVD catalyst. In this context, a comparison confirmed similar activity of PVD core-shell-catalyst and pure catalyst. Thereby, only the top layer of the core-shell-catalyst is active, meaning the same performance at significantly less material input. The results prove, that a reduction of catalyst material by Core-Shell-concept is possible.

TS1-ThP-4 Production of Cost-Effective Precious Metal Free Bipolar Plates for Future High Demand, *Sijia Yang (yang@kcs-europe.de)*, KCS Europe GmbH, Germany; *J. Kapp, V. Lukassek*, The hydrogen and fuel cell center ZBT GmbH, Germany; *R. Cremer*, KCS Europe GmbH, Germany

Objective of the R&D project "BipolarPilot" was the economic mass production of bipolar plates, which form the heart of fuel cells and represent around 30% of the total system costs. Together with its research partner The hydrogen and fuel cell center ZBT GmbH, KCS Europe has both developed appropriate PVD coatings as well as an inline PVD pilot coating system in order to enable mass production of cheap and corrosion stable stainless steel Bipolar plates.

While batch coaters are coating units with only one cycle-time limiting coating chamber, inline systems are coating units that enable a continuous processing and coating with short cycle times. Thus, the pilot coating unit has been designed with a future high demand for bipolar plates in mind.

KCS Europe's coatings for bipolar plates have been tested based on DoE specifications. The deposited coatings were investigated in detail by ZBT. Both, the electrical properties and the corrosion behavior under application conditions were investigated. KCS Europe's coatings for bipolar plates are homogenous precious metal free coatings. Investigations at ZBT proved, that the coatings beat the various limitations given by the DOE (Department of Energy) of the United States to meet the stack conditions. The contact resistance of all investigated coatings was determined below 0.6 m Ω cm² and thus well below the DOE limit of 10 m Ω cm². Moreover, the corrosion current identified by polarization test of all coatings meet the target values for bipolar plates defined by the DOE (lower than 1 μ A/cm²).

TS1-ThP-5 Hydrogen Diffusion in Protective Coating Materials, P. Rückeshäuser, A. Bahr, W. Zhao, R. Hahn, T. Wojcik, Christian Doppler Laboratory for Surface Engineering of high-performance Components, TU Wien, Austria; S. Kolozsvári, P. Polcik, Plansee Composite Materials GmbH, D-86983 Lechbruck am See, Germany; T. Stelzig, F. Rovere, Oerlikon Balzers, Oerlikon Surface Solutions AG, 9496 Balzers, Liechtenstein; Helmut Riedl (helmut.riedl@tuwien.ac.at), Institute of Materials Science and Technology, TU Wien, Austria

Introducing hydrogen-based energy production, storage, and conversion technologies implies materials withstanding hydrogen's specific, reactive behavior. Durability-related issues such as hydrogen embrittlement in structural components or corrosion-induced phenomena in fuel cell or electrolyzer technologies are of significant interest in transitioning to green and sustainable energy supplies. In more detail, the stimulated interaction of hydrogen with the topmost micrometers and the microstructural features of materials will play a predominant role. Therefore, using physical vapor deposited (PVD) coating materials to protect and functionalize material surfaces will be critical in diverse future applications.

Degradation mechanisms related to hydrogen occur at multiple length scales, involving different strategies for exposure or treatments. These strategies are typically divided into the two hydrogen-related material research worlds: (i) electrochemical degradation setups and (ii) and nonelectrochemical treatments as a collective term for pressure/gas-related setups. Nevertheless, the broad field of hydrogen-related applications is increasingly merging. Consequently, the different degradation strategies primarily focus on electrochemical test setups as they are highly versatile.

Therefore, in this study, the interaction of hydrogen with well-known protective coating materials such as TiN, CrN, ZrN, or TiAlN and AlCrN is described by an electrochemical Devanathan-Stachurski permeation setup. Different sets of these ceramic coating materials have been deposited on ferritic steel sheets through sputter and arc-evaporation technologies with varying deposition parameters. Subsequently, the coatings have been electrochemically loaded, and parameters such as diffusion coefficients, permeability, or permeation reduction factors are estimated. These results are correlated with the coatings' microstructural appearance before and

after hydrogen testing using a set of diverse high-resolution techniques such as SEM, TEM, XRD, and micro-mechanical testing methods.

Keywords: Protective coatings; Hydrogen; Electrochemical testing; PVD; Nitrides;

Topical Symposium on Sustainable Surface Engineering Room Golden State Ballroom - Session TS3-ThP

Solar Thermal Conversion - TS3 Poster Session

TS3-ThP-1 Tailoring the Structural, Optical and Electrical Properties in Perovskite Nickelates Through the Tilt Control of Nd_{1-x}Sm_xNiO₃ Thin Films, *Zil Fernández-Gutiérrez (zil.fernandez-gutierrez@univ-lorraine.fr), T. Easwarakhanthan, S. Bruyère, D. Pilloud, S. Barrat, F. Capon,* Institut Jean Lamour - Université de Lorraine, France

Rare-earth nickelates (RNiO₃) are functional oxides with a vast landscape of properties, with the metal-insulator transition (MIT) being the most attractive one. This latter can be modified by varying the rare-earth atomic composition $(R'_{1-x}R_xNiO_3)$ and the consequent tilt of the crystalline structure. However, obtaining solid solutions of this perovskite family is even more complex due to the specific composition control and the bottleneck that has been their synthesis over the years. Even so, this work aims to customize the thermochromic behavior of the layers to enhance infrared thermal regulation in solar collectors between 60 and 80 °C. Therefore, Nd_{1-x}Sm_xNiO₃ thin films were synthesized using reactive magnetron sputtering and air-annealing. Comprehensive XRD and TEM analysis confirms the efficient synthesis of various crystallized nickelates within the SmNiO3 to NdNiO3 range. This methodology facilitates the tunability of MIT temperatures between -80 and 120 °C as Sm atoms progressively replace Nd atoms. Optical performance, assessed through FTIR spectroscopy, aligns with literature-reported MIT temperatures. Additionally, a detailed examination of the structural, electrical, and electronic properties of the Nd_{0.2}Sm_{0.8}NiO₃ combination is presented. Lastly, ellipsometry measurements reveal a metal-like to dielectric-like phase change in the imaginary part of the dielectric function with photon energy, while the real part indicates oxidation in the upper film volume. These findings advance the understanding of Nd_{1-x}Sm_xNiO₃ nickelate thin films and their potential applications in thermochromic devices for solar energy utilization.

TS3-ThP-2 Trigeneration Plants Based on Solar Selective Surfaces of Carbon, Jose L. Endrino Armenteros (jlendrino@uloyola.es), E. Valbuena Niño, Universidad Loyola Andalucia, Spain; F. Montero-Chacón, Universidad Loyola Andalucía, Spain; A. Sandoval, M. Zurita, Universidad Loyola Andalucia, Spain

The SSC material is one of the major components in solar-driven trigeneration technologies. The optical properties determine the efficiency of the energy conversion from the concentrated sun irradiation. Solar receivers are usually maintained under vacuum as to limit convective thermal losses, the absorber coating is typically designed to operate without oxidative atmosphere. The development of a solar receiver, able to operate under air, is a challenge to reduce some of the costs. It is also a way to lower the complexity of existing linear CSP technologies. Although standard solar troughs receivers operate inside an annulus with a pressure of less than 10-4 mbar, the proposed trigeneration technology will require the SSC material to operate in an oxidizing (ambient air) atmosphere and avoid critical troubleshoots due to accidental glass breakage and an unexpected vacuum loss.

Solar absorber layers based on nanocomposite materials (a-C:MeC) are here proposed. These nanocomposites are suitable candidates for mediumtemperature solar absorber applications. Pure a-C thin films in combination with group 4, 5, and 6 transition metals form a number of exceptionally stable interstitial carbides. These carbides are characterized by high melting points, high thermal and electrical conductivity, and high reflectivity in the entire UV-Vis-IR spectral range. The presence of these carbides stabilizes the nanocomposite microstructure at this range of temperatures. Different microstructures and morphologies nanocomposite layers are here explored in combination with the Infrared (IR) layer made of titanium nitride (TiN) and а defect free alumina antireflective (AR) laver.

In addition, a simulation platform which we can run several scenarios that include, but are not limited to, hospitals, supermarkets, schools, residences, etc. will provide information on the trigeneration plant (i.e., heating,

cooling, and electricity loads) for given scenarios using carbon-based coating materials.

Topical Symposium on Sustainable Surface Engineering Room Golden State Ballroom - Session TS4-ThP

Coatings and Surfaces for Thermoelectrical Energy Conversion and (Photo)electrocatalysis - TS4 Poster Session

TS4-ThP-1 Dopant-defect Engineering in SnS₂ Thin Films for Improved Gasphase Photocatalytic CO₂ Reduction, *Tadios Tesfaye Mamo* (*tadios.tesfaye@aau.edu.et*), Department of Chemistry, National Taiwan University, Taiwan; *M. Qorbani*, Center for Condensed Matter Sciences, National Taiwan University, Taiwan; *A. Hailemariam*, Department of Applied Chemistry, National Yang-Ming Chiao Tung University, Taiwan; *A. Sabbah*, Center for Condensed Matter Sciences, National Taiwan University, Taiwan; *S. Kholimatussadiah*, Department of Physics, National Taiwan University, Taiwan; *C. Huang*, Institute of Atomic and Molecular Sciences, National Taiwan University, Taiwan; *K. Chen*, Institute of Atomic and Molecular Sciences, Academia Sinica, Taiwan

To address the issue of CO₂ amount increment in the Earth's atmosphere, various semiconductor photocatalysts have been employed to convert CO₂ into valuable products. Designing an efficient photocatalyst that can activate the CO₂ molecule with the least amount of activation energy is one of the challenging problem. In this regard, we report a combined experimental and computational analysis on a thin film of SnS2 doped with phosphorous ions at various ion doses. Thermal evaporation followed by sulfurization and ion implantation processes were used to prepare the regulated amount of phosphorous ion-implanted 20-nm SnS2 thick thin films. Our findings reveal that phosphorous doping synergistically enhances light harvesting by lowering the band gap and energetically stable CO2 binding sites with the lowest activation energy. The optimized P-SnS₂ photocatalyst has a three times higher CO2 conversion rate than the pristine one, with a high selectivity of about 92% towards CH4 formation. Because Phosporous play a vital role in the activation of CO_2 by serving as an active site and due to its low electronegativity, it increases the charge density of the Sn atom adjacent to it. Also, P-doping affects the charge density of the neighboring S atom by serving as a bridge to improve the charge distribution between Sn and S. This degree of electron density alteration would facilitate the electron transfer in the photocatalytic reaction.NAP- Insitu XPS and XAS results with formation energy, Bader charge, and Gibbs free energy calculations are used to carefully assess the overall impact of phosphorus in the SnS₂ sample. DFT calculations accord well with the experimental findings and help us to know the reaction pathway. We anticipate that our result will motivate additional ion implantation research to modify the material's active site for CO2 reduction and examine its CO2 conversion capability and related optical and charge transfer behavior.

TS4-ThP-2 e-Poster Presentation: Copper-Based Coatings on Polylactic Acid for Electrocatalytic CO₂ Reduction, *M. Lima*, University of Minho, Portugal; *J. Castro, Sandra Carvalho (sandra.carvalho@dem.uc.pt)*, University of Coimbra, Portugal

The climate crisis caused by global warming is recognized by the United Nations (UN) as a trigger for catastrophic effects such as weather extremes and natural disasters. Carbon dioxide (CO₂) emissions constitute about three-quarters of the total greenhouse gases (GHG) released and have gathered global attention due to their significant contribution to global warming.

Developing new catalytic processes can accelerate the transition to a more sustainable Earth. Electrocatalytic methods are the most promising of all the catalytic processes because they are energy efficient, selective, easy to control, and flexible. They are also known as the most viable solution for the CO_2 reduction reaction ($CO_2 RR$).

Metallic copper has notable electrical conductivity, making it suitable for many electrode-based applications. Additionally, copper-based materials were reported to be active and selective electrocatalysts capable of producing hydrocarbons from the CO₂RR. However, the updated version of the Element Scarcity—EuChemS Periodic Table by the European Chemical Society brings attention to the limited abundance of copper. Ensuring a sustained supply of this element is a significant challenge. Utilizing thin film coatings to produce electrodes is a potentially practical approach to mitigating element shortages. Furthermore, Cu-based electrodes using a

polymeric skeleton can provide several benefits concerning cost, material accessibility, and weight.

This work used polylactic acid (PLA) as the substrate for Cu-based electrodes. PLA is widely used in additive manufacturing, a low-cost technique that enables the fabrication of 3D-structured electrodes. Magnetron sputtering (PVD technique) was applied to develop copper metallic surfaces on PLA. Different coatings with Cu/CuO_x/Cu layers were produced. Anodization was a secondary technique applied to enhance the electrochemical active surface area. The CuO_x in the middle of the coating might act as a barrier material to stop the oxidation reaction during the anodization process while maintaining film adhesion. The chemical and morphological characterization of the resulting films will be discussed, as well as the electrochemical properties for CO_2RR applications.

TS4-ThP-3 Two-Dimensional Ruddlesden–Popper Phase of B-site substituted Can-1Mnn-3Nb3O_{3n+12}. (n=4,5,6) Perovskite Nanosheets Integration with Chlorella vulgaris for Electrochemical Water Splitting, *Yao-Yuan Chang (m56111135@gs.ncku.edu.tw), C. Chang, Y. Su,* National Cheng Kung University (NCKU), Taiwan

Two-dimensional (2D) perovskite nanosheets have emerged as potential candidates for hydrogen production and spintronic devices due to their large surface area, special optical, electric, magnetic, and structural properties. In this study, we synthesized 2D Ruddlesden–Popper (RP) phase perovskite nanosheets $Ca_{n-1}Mn_{n-3}Nb_3O_{3n+1}$ (n=4,5,6) to tune their physicochemical properties and catalytic performances via soft chemistry process. In this configuration, manganese (Mn) partially substitutes niobium (Nb) at the B-site within the niobate perovskite lattice structure exhibited positive influences in water splitting applications. The combination of CMNO nanosheets with Chlorella on the photoelectrode surface has demonstrated improved photoelectrochemical performance, especially for CMNO (n=6) nanosheet. This research contributes to the future outlook for sustainable energy solutions by unique properties of 2D perovskite oxide nanomaterials in conjunction with bio-inspired components.

Keywords: two-dimensional, Ruddlesden–Popper phase perovskites, Chlorella vulgaris, water splitting, magnetic

Friday Morning, May 24, 2024

Topical Symposium on Sustainable Surface Engineering Room Town & Country C - Session TS3-FrM

Solar Thermal Conversion

Moderators: Telmo Echániz, University of the Basque Country, Spain, Marcus Hans, RWTH Aachen University, Germany

8:00am TS3-FrM-1 Application of Surface Engineering Solutions in Concentrating Solar Power Key Components, Ramón Escobar-Galindo (rescobar1@us.es), University of Seville, Spain; J. Sanchez-Lopez, T. Rojas, CSIC-University Sevilla, Spain; H. Barshilia, CSIR-National Aerospace Laboratories, India; M. Krause, Helmholtz Zentrum Dresden-Rossendorf, Germany INVITED

Thermal conversion of solar energy into electricity is one of the most efficient methods of harvesting renewable energy. In this regard, the development of new materials is crucial to improve the performance of concentrating solar power (CSP) plants. The future developments of thermosolar plants demand, among others, (1) mirrors with higher reflectivity, better protection, and lower costs, (2) absorber receiver components operating at higher temperatures, with wavelength selective capabilities, or (3) more stable materials under corrosive environments (i.e. molten salts) (see Figure 1). In this presentation, a review of surface engineering concepts (i.e. tailoring of multilayer materials, control of the interface design) applied to thermosolar energy will be discussed. Examples from our own research on the design, characterization, and thermal testing of ultra-reflective dielectric multilaver mirrors¹ and solar selective coatings (SSCs) for high-temperature applications,^{2,3,4} will be presented. In the former, we have demonstrated the design, using a genetic algorithm, and manufacture, using Physical Vapour Deposition (PVD) techniques, of metaldielectric multilavered solar mirrors that can outperform silver reflectors commonly employed in thermosolar and photovoltaic systems. In the latter, the thermal stability of SSCs based on metal oxynitrides prepared by PVD was studied after high-temperature annealing of the samples performed in air (ex-situ) and in vacuum (in-situ).

(1) A. Jiménez-Solano et al. *Aperiodic Metal-Dielectric Multilayers as Highly Efficient Sunlight Reflectors.* Advanced Optical Materials 5,9 (2017) 1600833

(2) T.C, Rojas Ruiz et al. *High-temperature solar-selective coatings based on Cr(Al)N. Part 2: Design, spectral properties and thermal stability of multilayer stacks.* Solar Energy Materials & Solar Cells. 218 (2020) 110812

(3) R. Escobar-Galindo et al. *Solar selective coatings and materials for hightemperature solar thermal applications*, Chapter 13 in "Sustainable Material Solutions for Solar Energy Technologies", Elsevier, 2021

(4) K. Niranjan et al. WAISIN-based solar-selective coating stability-study under heating and cooling cycles in vacuum up to 800 °C using in situ Rutherford backscattering spectrometry and spectroscopic ellipsometry. Solar Energy Materials & Solar Cells. 255 (2023) 112305

8:40am TS3-FrM-3 Development and Thermal Characterization of High-Temperature Coating Materials for Solar Thermal Energy Conversion, Renkun Chen (rkchen@ucsd.edu), University of California, San Diego, USA INVITED

Coating is fundamentally important and ubiquitous for solar thermal energy conversion. From low temperature solar water heaters to intermediate parabolic trough solar collectors to high temperature solar towers, solar absorbing coatings play an important role. In this presentation, I will introduce our work over the past few years. Specifically, we have developed coatings based on high-temperature stable spinel oxide nanoparticles with ultra-high solar absorptance. The spinel oxide nanoparticles are synthesized with a scalable hydrothermal process. In the first application, the particles are mixed with silica resin to form a slurry for coating. The slurries can be spray coated onto substrates with a scalable process followed by standard curing and sintering processes. The coating on steel and Inconel substrates shows high solar absorptance (with solar to thermal conversion efficiency over 90% for 1000-sun at 800 oC absorber temperature). The coatings also exhibit superior thermal stability after long thermal isothermal ageing tests at 800 oC and thermal cycling tests from room temperature to 800 oC. This scalable coating process can be applied to cylindrical solar collector tubes with liquid heat transfer fluid (e.g., molten salts) in solar towers. In the second application, the black spinel nanoparticles are coated onto particulate heat transfer media (HTM) based on sand or ceramic particles of a few hundred microns in diameter. The black coating can greatly enhance

the solar absorptance of sand HTM, and can also lead to stable solar absorptance of Carbo ceramic particles. We also developed high-entropy version of black nanoparticles which show remarkable stability against grain growth and can be used to coat thermochemically active particles to simultaneously increase the solar absorptance and suppress particle sintering, which is important to sustain high reaction kinetics of the thermochemical materials.

9:20am TS3-FrM-5 Smart Coatings for Concentrated Solar Thermal: from Optical Design and Plasma Synthesis to Performance and Durability Assessment, Audrey Soum-Glaude (Audrey.Soum-Glaude@promes.cnrs.fr), A. Diop, PROMES-CNRS, France; A. Mahammou, D. Ngoue, PROMES-CNRS, Perpignan University, France; A. Grosjean, EPF Montpellier, France; B. Plujat, S. Quoizola, PROMES-CNRS, Perpignan University, France; A. Bousquet, E. Tomasella, University Clermont Auvergne, France; L. Thomas, PROMES-CNRS, Perpignan University, France INVITED

Half of our energy consumption is heat for industrial processes and buildings. Contrary to solar PV and wind where electricity is directly produced, in solar thermal technologies, solar radiation is harvested by a solar receiver and converted into heat. In concentrated solar thermal (CST) technologies, solar radiation is concentrated by mirrors on the receiver to increase temperature. The generated heat can be stored more efficiently and at lower cost than electricity, providing great dispatchability and mitigating the intermittency of the solar resource. Solar thermal is thus a major solution for sustainable energy production, complementary to PV and other renewables. Additionally, the hybridization of PV and CST solar technologies can take advantage of the low cost of PV electrical production and the thermal storage of CST to produce both electricity and heat on demand.

To increase solar-to-heat conversion efficiency, the metallic surface of CST receivers can be covered with Solar Selective Absorber Coatings (SSACs) with high absorptance (i.e., low spectral reflectance) in the solar range, and low thermal emittance (i.e., high spectral reflectance) in the infrared range to limit radiative thermal losses. These coatings must also be resistant to high temperatures in air and high thermomechanical stresses, particularly for CST where they are exposed to harsh working conditions for long durations.

Similarly, for PV/CST hybridization, compact systems where PV cells are installed on solar concentrators to produce electricity, and CST thermal absorbers placed at their focus to produce heat, require spectrally selective mirror coatings. The latter must transmit part of the solar radiation to the underlying PV cells (typically from 400 to 1100 nm) and reflect the rest towards the CST thermal solar receiver.

In both types of surfaces, such spectrally selective behavior is obtained by combining advanced optical design and optimization of the coatings with their experimental synthesis, microstructural and thermo-optical characterization, and aging studies including aging under representative solar irradiation. In this paper, this complete development strategy will be illustrated with two examples of coating developments carried out at PROMES-CNRS laboratory: [W/SiCH]_n and W/WSiCH/TaO_xN_y multilayer stacks for CST high temperature SSACs and [SiO₂/TiO₂]_n for selective "PV mirrors" for PV/CST hybridization, deposited by PVD and PECVD techniques.

10:20am TS3-FrM-8 Controlling Infrared Emissivity of Thermochromic VO₂ Films via V₂N Precursor Thickness for Enhanced Solar Thermal Regulation, A. Garcia-Wong, D. Pilloud, S. Bruyère, S. Migot, S. Hupont, F. Capon, Jean-François Pierson (jean-francois.pierson@univ-lorraine.fr), Institut Jean Lamour - Université de Lorraine, France

The control of infrared (IR) radiation is crucial for applications related to solar radiation. Thanks to the IR properties modification during its metal-insulator transition, monoclinic vanadium dioxide $(m-VO_2)$ is an excellent material for solar thermal regulation device development. Yet, these applications are linked to substrate IR properties on which the VO₂ is deposited.

In this study, we propose a novel method for tuning the sign of the VO₂ IRemissivity without changing the substrate. Thermochromic VO₂ films have been synthesized by air oxidation of reactively sputtered V₂N films deposited on silicon substrates. As-deposited vanadium nitride films have been oxidized in air at 550 °C. The structure of the VO₂ films has been characterized using X-ray diffraction and Raman spectrometry. Transmission electronic microscopy combined with electron energy loss spectroscopy (EELS) bring relevant information to describe the oxidation mechanism of V₂N. At the interface between the remaining V₂N film and the formed VO₂

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one, a thin layer of VN of 20 nm thick has been evidenced. At this oxidation temperature, the annealing duration to obtain efficient thermochromic VO₂ films is fixed to 3 minutes. The initial precursor (V₂N) thickness is the key parameter for tuning the IR properties. V₂N films with low thickness allow the formation of thermochromic VO₂ films with negative emissivity switch (approx. -0.2) while thick V₂N films allow positive emissivity switch (approx. 0.2). Our findings introduce a different strategy for IR emissivity control on thermochromic devices.

10:40am TS3-FrM-9 Emissivity and Reflectivity Measurements of Coatings for Solar Applications, Telmo Echaniz (telmo.echaniz@ehu.eus), I. Gonzalez de Arrieta, M. Sainz-Menchon, J. Gabirondo-Lopez, G. Lopez, University of the Basque Country, Spain

Widespread adoption of concentrated solar power relies on lowering its operational costs, which requires efficient absorbing materials capable of withstanding high temperatures. This motivates the development of coating materials based on oxides. Unfortunately, these materials do not always feature appropriate optical properties, which means that strategies to increase their solar absorptances must be carried out. This work reports on the optical characterization of high-absorbing coatings. For that, directional spectral infrared emissivity measurements up to 600 °C, integrating sphere and bidirectional reflectance measurements in the UV-Vis-NIR range at room temperature have been performed. Spectral directional emissivity measurements were performed in the HAIRL emissometer using using a DTLaGS detector between 1.43 and 25 μm and the sample was placed between 10 and 80° every 10° for each directional measurement [1]. This emissometer is equipped with a Bruker Vertex 80v FTIR spectrometer that possesses an IR integrating sphere. Once the spectra were obtained, the emissivities were integrated along the electromagnetic spectrum and the solid angle to obtain total hemispherical emissivity values. Bidirectional reflectance measurements were performed between 350 and 2500 nm in a Cary UMA device. They were measured in both s and p polarizations, from which the unpolarized reflectance function was reconstructed. Measurements in the off-specular directions were also measured to complement these data and allow estimation of the total reflectance, weighted by the solar spectrum. Both the infrared emissivity and the BRDF measurements allowed obtaining the conversion efficiency.

Acknowledgements: This work was funded by the University of the Basque Country, Spain (GIU19/019) and the Basque Government, Spain (IT-1714-22 and PIBA-2021-1-0022). J. Gabirondo-López and I. González de Arrieta also acknowledge financial support from pre- and post-doctoral fellowships by these institutions (University of the Basque Country, Spain PIF 21/06; Basque Government, Spain: POS-2021-2-0022).

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