Topical Symposium on Sustainable Surface Engineering
Room Town & Country A - Session TS1-ThP

Coatings for Batteries and Hydrogen Applications Poster Session

TS1-ThP-1 Hydrogen Permeation Testing: Electrochemical vs. Pressurized Methods, ***Phillip Rückeshäuser [phillip.rueckeshaeuser@tuwien.ac.at]***, TU Wien, Austria; *Szilard Kolozsvari, Peter Polcik,* Plansee Composite Materials GmbH, Germany; *Timea Stelzig,* Oerlikon AM Europe GmbH, Germany; *Konrad Fadenberger,* Oerlikon Balzers Coating Germany GmbH, Germany; *Klaus Boebel,* Oerlikon Balzers, Liechtenstein; *Tomasz Wojcik, Helmut Riedl,* TU Wien, Austria

The interactions between hydrogen and materials are subjects of significant interest in both research and industry. Consequently, the hydrogen charging of samples and following detection are crucial aspects of this field. Currently, there are two primary methods for hydrogen charging: electrochemical charging and pressurized hydrogen charging.

In electrochemical charging, atomic hydrogen is produced through the dissociation of water, while in pressurized charging, hydrogen is introduced in gaseous form and subsequently thermally dissociated. Although these two methods are based on fundamentally different principles, they ultimately achieve the same goal: the absorption and permeation of atomic hydrogen in the sample. However, most studies tend to focus on either one method or the other. Thus, it becomes essential to explore the correlation between these methods and identify equivalent charging conditions for specific hydrogen permeation characteristics.

In this study, we conducted hydrogen permeation tests using both electrochemical and pressurized setups. We applied nitride coatings such as TiN and CrN to steel substrates using physical vapor deposition (PVD) techniques and compared the permeation performance of the two charging methods. This comparison involved determining key parameters such as diffusion coefficients and permeation reduction factors. Additionally, we characterized the coating properties using secondary electron microscopy, X-ray diffraction, and linear sweep voltammetry.

TS1-ThP-2 Towards Defect-Free Laser-Induced Graphene Coating on Copper and Aluminum Foils for Anode-Free Li and Na Metal Batteries, ***Aarti Gunjal [aartitambe22@gmail.com]***, IISER PUNE, India; *Suparna Saha,* TCG-CREST Kolkata, India; *Swati Jadhav,* IISER PUNE, India; *Satishchandra Ogale,* TCG-CREST Kolkatta, India

Laser-induced graphene (LIG) is a very well-established process for realizing functional carbon coatings on different substrates. Depending on the laser processing parameters it yields structurally, morphologically and chemically interesting forms which are also adherent because of the thermal energy input. In most cases, however, a defect band (D band) is invariably observed in the Raman spectrum of such coatings, in addition to the so-called G-band corresponding to desired graphitic carbon. In this work we have applied CO2 laser processing to carbonize the coatings of the dried powder of aromatic eucalyptus (EU) leaves on metals. The effects of laser power density, scanning speed, substrate choice, and precursor coating thickness on the quality of carbon are carefully examined and optimized to obtain a unique almost defect-free few layers graphene coating, as reflected by a miniscule D-band and a significant 2D signature in the Raman spectra of few-layer graphene. Comparative studies on other aromatic leaves sample (e.g. lemon grass) and non-aromatic leaves suggest that aromaticity is important for realizing an enhanced 2D band, which could be due to internal turbulent cavitation upon laser-induced transient heating. In situ nitrogen doping is also achieved by applying the LIG process to a mixture of EU powder loaded with urea. Remarkably, the nitrogen incorporated coatings on copper and aluminum current collectors render far superior performance in anode-free Li and Na metal batteries, respectively, as against the defect-free few layers graphene coating. In fact, half cells with alkali metals show an impressive cycling stability of over 450 cycles for both Li and Na. Through thorough characterizations employing multiple techniques, an attempt is made to develop a mechanistic understanding of the issues involved.

TS1-ThP-3 HiPIMS Mo_xN and Cu-Mo_xN Thin Films for the Hydrogen Evolution Reaction, *Hung-I Wu,* Department of Electronic Engineering, National Yunlin University of Science and Technology, Taiwan; *Ying-Hsiang Lin,* Department of Materials Science and Engineering, National United University, Taiwan; *Shih-Hung Lin,* Department of Electronic Engineering, National Yunlin University of Science and Technology, Taiwan; *Fan-Bean Wu, Chi-Yueh Chang,* Department of Materials Science and Engineering, National United University, Taiwan; *Thi Xuyen Nguyen, Ruei-Chi Lin, Jyh-Ming Ting,* Department of Materials Science and Engineering, National Cheng Kung University, Taiwan; ***Wan-Yu Wu [wywu@nuu.edu.tw]***, Department of Materials Science and Engineering, National United University, Taiwan

Hydrogen produced by water electrolysis is a promising clean energy carrier, yet large-scale deployment is hindered by the high cost and limited durability of noble-metal catalysts. Transition-metal nitrides, particularly molybdenum nitride (Mo_xN), offer attractive corrosion resistance and electrical conductivity, enabling hydrogen evolution reaction (HER) catalysis in both acidic and alkaline media.

In this work, Mo_xN thin films were deposited and benchmarked using RF sputtering and high-power impulse magnetron sputtering (HiPIMS). In 0.5 M H_2SO_4, the HiPIMS-Mo_xN catalyst achieved an overpotential of 292 mV, corresponding to a ~61.2% improvement compared with pristine carbon paper (CP). The performance gain is attributed to the superior film adhesion achieved by HiPIMS, leading to a 9.7% reduction in charge-transfer resistance (R_{ct}) and the absence of large-area catalyst delamination after HER testing. Process optimization identified Cu addition is suggested to tune the Mo d-band center and improve overall conductivity, reducing the overpotential to 254 mV (~13.0% improvement vs. HiPIMS-Mo_xN) and decreasing the Tafel slope from 75.9 to 55.0 $mVdec^{-1}$, indicating accelerated HER kinetics via a synergistic effect. In 1.0 M KOH, HiPIMS-Mo_xN achieved the lowest overpotential (199 mV), while Cu addition deteriorated activity, suggesting Mo as the dominant active center in alkaline media and that secondary metals reduce accessible Mo sites.

TS1-ThP-4 Hydrogen Barrier Properties of Thin Oxide Films Prepared by Different Methods: Correlations of Thin Film Properties with Hydrogen Permeation Rates, *Dmitry Kalanov, Juergen W. Gerlach, Patrick C. With,* ***Yeliz Unutulmazsoy [yeliz.unutulmazsoy@iom-leipzig.de]***, *Ulrike Helmstedt,* Leibniz Inst. of Surface Eng. (IOM), Germany

Efficient hydrogen barriers are essential for the hydrogen economy, where minimizing hydrogen loss and ensuring material safety are critical. In the present study, we investigate thin oxide films, using TiO_2 as a model oxide system, deposited on PET substrates by UV photochemical conversion of metalorganic precursors under ambient conditions and by High-Power Impulse Magnetron Sputtering (HiPIMS) deposition without substrate heating. Using a dedicated gas-permeation measurement system, hydrogen permeation through the oxide films on PET was studied as a function of film thickness. Results demonstrate a strong thickness dependence of the barrier properties: 50 nm TiO_2 films exhibit a fivefold improvement, while increasing the thickness to 70 nm for HiPIMS grown thin films reduces permeation below the detection limit, highlighting the high barrier performance of dense, amorphous TiO_2 films. These findings are discussed in the context of current challenges in measuring hydrogen permeation in thin films on various substrates, compared to other oxide systems such as SiO_x [1], and in relation to differences in structural properties of the films arising from the distinct deposition methods.

[1] P.C. With, T. Pröhl, J.W. Gerlach, A. Prager, A. Konrad, F. Arena, U. Helmstedt, Hydrogen permeation through uniaxially strained SiO_x barrier thin films photochemically prepared on PET foil substrates, Int. J. Hydrog. Energy 81 (2024) 405-410

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