Advanced Characterization, Modelling and Data Science for Coatings and Thin Films
Room Palm 1-2 - Session CM1-1-ThM

Spatially-resolved and in situ Characterization of Thin Films, Coating and Engineered Surfaces I
Moderator: Barbara Putz, Empa Thun, Switzerland

8:40am **CM1-1-ThM-3 Analysis of Deuterium by Atom Probe Tomography (Apt) - D in V Films and Fe/V Multi-Layered Films***, **Ryota Gemma [goettingenma@googlemail.com]**,* Tokai University, Japan*; Talaat Al-Kassab, Astrid Pundt,* University of Göttingen, Germany **INVITED**

In this presentation, we will present the results of deuterium distribution and quantitative analysis by atom probe tomography (APT) in single-layered V or Fe/V multi-layered films. While V is a hydrogen-absorbing metal, Fe hardly dissolves hydrogen (H). Therefore, in Fe/V multi-layered films, almost all of the H atoms are supposed to be in the V layer, and the H distribution should show a clear contrast at the Fe/V interface. This is also the case for deuterium (D). D has a lower diffusion coefficient than H. Hence, a surface segregation of D during the APT analysis can be suppressed, enabling to visualize original D position in the host metal lattice. Furthermore, D can be distinguished from background hydrogen in the APT analysis chamber. By using a portable chamber to prevent the sample from being exposed to oxygen, we were able to measure the D concentration in V or Fe/V films over a wide concentration range. We compared the D concentration measured by APT with the compared with the results of measurements of the hydrogen concentration dependence of electromotive force (EMF) for similar samples, it was found that the average D concentration in the sample could be correctly evaluated using APT.

9:20am **CM1-1-ThM-5 Monitoring Thin Film Battery Electrodes via in-Situ/in-Operando Ellipsometry***, **Máté Füredi [mate.furedi@semilab.hu]**,* Semilab Semiconductor Physics Laboratory Co. Ltd., Hungary*; Jialin Gu, Adam Lovett,* University College London, UK*; Bálint Fodor, András Marton,* Semilab Semiconductor Physics Laboratory Co. Ltd., Hungary*; Stefan Guldin,* Technical University of Munich, Germany*; Thomas Miller,* University College London, UK

The electrochemical energy storage behavior of nano- and microscale (thin- and thick-film) electrodes displays unique characteristics that provide crucial insights into various charge storage mechanisms, essential for the optimal design of commercial battery applications. Additionally, these films are applicable for constructing microbatteries for miniature electronic devices (such as sensors). Critically, material chemistry, crystallinity, and nanostructure significantly influence active charge transfer mechanisms in these systems, generally classified as electrochemical double layer capacitive, pseudocapacitive, or battery-type behaviors. In lithium-ion batteries specifically, the charge storage mechanism involves the (de)/intercalation of lithium ions in active electrode materials, such as silicon, graphite, or transition-metal oxides.

By optically monitoring thin-film electrodes under electrochemical charge/discharge, a range of time-resolved structural data can be obtained. This work elaborates on this by integrating operando spectroscopic ellipsometric data acquisition. Ellipsometry, highly sensitive to thin films, offers an advantage by effectively excluding any electrolyte side-reactions from measurement, thus providing accurate, real-time data on the evolving structure of lithiated electrodes across charging states. Additionally, ellipsometry tracks thickness changes, enabling precise monitoring of degradation mechanisms.

This work demonstrates (on the example of transition-metal oxide thin-film electrodes) how ellipsometry can reveal intercalation processes, diffusion limitations, and pseudocapacitive contributions. This is further correlated with the complementing electrochemical data. The considerations of this work are furthermore broadly applicable to other thin-film electrode materials.

9:40am **CM1-1-ThM-6 Exploring the Benefits of Automated, Redox Reactions in XPS Analysis***, **James Lallo [james.lallo@thermofisher.com]**,* Thermo Fisher Scientific, UK, USA*; Robin Simpson, Paul Mack, Tim Nunney,* Thermo Fisher Scientific, UK

This presentation investigates the benefits of automated, in-situ redox reactions for the purpose of producing well controlled oxide growth on the surface of various sample types. The driving force behind using such a procedure is in the potential for generating a sequence of spectra from a progressively chemically-modified surface to remove ambiguities that can lead to misinterpretation, thus aiding in faster understanding of the unmodified surface. Our study presents XPS results from coupled stepwise oxidation/reduction of surfaces, to aid in resolving such ambiguities across a wide array of materials. We use gas-phase oxidation agents to control the redox states of a specimen, leveraging the logarithmic growth of oxide thickness. This oxidation is implemented using vacuum ultraviolet light (VUV) and the generation of ozone and gas-phase hydroxide free radicals close to the surface of the specimens within the entry-lock of the Thermo Scientific Nexsa surface analysis instrument. This work focusses on the benefits of automating this process to ascertain the potential merits of including it into a standard operating procedure for XPS analysis.

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