## Thursday Afternoon, May 15, 2025

Advanced Characterization, Modelling and Data Science for Coatings and Thin Films

Room Golden State Ballroom - Session CM-ThP

Advanced Characterization, Modelling and Data Science for Coatings and Thin Films Poster Session

CM-ThP-1 How to Predict the Deposition Rate During Reactive Sputtering Using an One-Volume Reference Resource?, Diederik Depla [Diederik.Depla@ugent.be], Ghent University, Belgium

A longstanding challenge in reactive magnetron sputtering is the quantitative prediction of the deposition rate, which is primarily determined by the partial metal sputtering yield from the oxide layer formed on the target surface during poisoning. The first step in addressing this issue is to determine the total sputtering yield of the oxide. This has been accomplished by refining a published semi-empirical model. This model has been applied to fit an extensive set of oxide sputtering yield data from the literature, comprising 65 datasets for 21 different materials. The fitting process establishes a relationship between the surface binding energies of metal and oxygen atoms and the cohesive energy of the oxide. The calculated partial sputtering yield of metal from a poisoned target is then compared with previously published experimental data on the metal sputtering yield during reactive magnetron sputtering. While both yields are linearly correlated, the magnetron-based sputtering yields are approximately eight times lower than the model predictions. This reduction in yield is attributed to the formation of an oxygen-rich surface layer, a hypothesis supported by binary collision approximation Monte Carlo simulations. However, these simulations do not fully capture the mechanism, as a more detailed description of the surface oxygen origin is needed. Despite this limitation, the experimental correlation provides a practical strategy for predicting deposition rates during reactive magnetron sputtering in fully poisoned mode. As demonstrated, the oxide sputtering yield can be calculated using standard data sources, and the empirical correlation between the sputtering yields enables a reliable estimate of the metal partial sputtering yield in poisoned mode, thus allowing for an accurate estimation of the deposition rate.

D. Depla, Note on the low deposition rate during reactive magnetron sputtering, Vacuum 228 (2024) 113546

D. Depla, J. Van Bever, Calculation of oxide sputter yields Vacuum 222 (2024) 112994

CM-ThP-2 Deep Insertion Induced Fracture in Soft Solids, MUTHUKUMAR MARIAPPAN [muthukumar.iitk@gmail.com], Department of Mechanical Engineering, IISc Bangalore, India

Deep insertion of sharp objects like a needle into soft tissues is a common procedure in the medical domain for delivering drugs, biopsies and other medical interventions. It is inevitable to avoid tissue damage during needle insertion which sometimes leads to catastrophic outcomes. Opaqueness and inhomogeneity of the tissues make it difficult to observe the underlying damage mechanisms. In this context, it is essential to understand the underlying mechanisms of the formation of various cracks, crack nucleation and crack propagation in soft tissue-mimicking materials during deep penetration to minimise tissue damage. In this talk, we discuss the fracture behaviour of soft tissue-mimicking gels during deep penetration of a sharp needle. For the first time, we observed nearly periodic, stable, and wellcontrolled 3-D cone cracks inside the soft gel during deep penetration. We show that the stress field around the needle tip is responsible for the symmetry and periodicity of the cone cracks. These results provide a better understanding of the fracture processes in soft and brittle materials and open a promising perspective in needle designs and the control of tissue damages during surgical operations.

CM-ThP-3 Temperature-Dependent Oxidation Mechanisms of Binary Nitride Compounds: A Molecular Dynamics Approach, Sara Fazeli, MS4ALL, France; Edern Menou, Marjorie Cavarroc [marjorie.cavarroc@safrangroup.com], SAFRAN, France; Pascal Brault, MS4ALL / GREMI, France

Binary nitride (XN) compounds represent an important class of advanced ceramic materials, increasingly recognized for their suitability in high-temperature applications such as aerospace components, turbine blades, and protective coatings. Transition metal nitrides such as titanium nitride (TiN) and zirconium nitride (ZrN) are especially noted for their outstanding hardness and resistance to corrosion. In addition, nitrides of non-transition metals, including carbon nitride (CN), silicon nitride (SiN), and boron nitride (BN), function as essential refractory materials due to their high stability

under extreme temperatures and durability in harsh environments. The oxidation behavior of binary nitride materials is often a crucial factor in selecting materials for high-temperature use, as the oxidation resistance of a given XN phase depends on its capacity to form a stable, passivating oxide layer. It is worth noting that a distinct change in the oxidation mechanism is observed at high temperatures, which is attributed to phase transformations in the oxidation products. The insights gained from the oxidation behavior will facilitate the more efficient design and rapid discovery of XN phases that maintain optimal performance in oxidizing environments at elevated temperatures. In this study, we perform ReaxFF and COMB3-molecular dynamics (MD) simulations of the oxidation of binary nitride compounds XN (X = B, C, Si, Ti, and Zr) at four different temperatures (900 K, 1300 K, 1500 K, and 1700 K) to elucidate the mechanism of the oxidation states in the oxide layer.

At the lowest temperature, oxygen chemisorption occurred on the binary compounds without significant surface oxidation. In contrast, at higher temperatures, the amount of  $\rm O_2$  adsorbed increased steadily, particularly for transition metal nitrides. High oxygen coverage at elevated temperatures may lead to structural reconstructions of the surface. This study provides valuable insights into the oxidation mechanisms, helping researchers identify strategies to form stable, protective oxide layers, which enhance corrosion resistance and broaden the industrial applications of high-temperature materials, paving the way for the development of other binary nitride compounds.

CM-ThP-4 Simulating Mode-I Crack Opening Process in Transition Metal Diborides via Machine-Learning Interatomic Potentials, Shuyao Lin [shuyao.lin@tuwien.ac.at], TU Wien, Institute of Materials Science and Technology, Austria; Zhuo Chen, Zaoli Zhang, Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Leoben, Austria; Lars Hultman, Linköping Univ., IFM, Thin Film Physics Div., Sweden; Paul Mayrhofer, Nikola Koutna, TU Wien, Institute of Materials Science and Technology, Austria; Davide Sangiovanni, Linköping Univ., IFM, Thin Film Physics Div., Sweden

The critical stress-intensity factor  $K_{IC}$  and fracture strength  $\sigma_f$  define the fracture resistance of brittle ceramics. However, their experimental measurement is challenging and provides limited atomic-scale insight into crack tip behavior. In this work, we overcome these limitations by offering atomic-scale information on crack growth while evaluating fracture toughnesses and fracture strengths via machine-learning-assisted simulations. Transition metal diborides (TMB2:s) serve as a case study, with a focus on understanding the Mode-I crack opening response across six distinct orientations within 2 different phases ( $\alpha$  and  $\omega$ ). Molecular statics and dynamics calculations were used to systematically test model sizes and thicknesses, ensuring efficient simulations and accurate extrapolation of macroscale mechanical properties via constitutive scaling laws. By incorporating the phase-dependent and anisotropic mechanical properties of the α-phase TMB<sub>2</sub>:s, the observed phenomena, as revealed through strain distribution and bond distances, align closely with those well-studied ceramics such as nitrides, offering insights into the fracture mechanisms within realistic deformation environments via atomistic level perspective. Furthermore, while  $\alpha\text{-}$  and  $\omega\text{-}WB_2$  exhibits minimal phase dependence in deformation plasticity strength, as supported by both theoretical and experimental results, the fracture strength, as determined through the defective model, demonstrates a significant variation. The results show that the K<sub>IC</sub> varies across different orientations and phases within the group IV, V, and VII TMB2:s, correlating with their respective tensile and shear strengths.

CM-ThP-5 Simulation Study on Color Modulation of Diamond Substrates via Localized Surface Plasmon Resonance Effects Induced by Metal Nanoparticles, Tsung-Jen Wu [d09224004@ntu.edu.tw], Sheng-Rong Song, Wen-Shan Chen, National Taiwan University, Taiwan; Wen Lin, National Taipei University of Technology, Taiwan; Shao-Chin Tseng, National Synchrotron Radiation Research Center, Taiwan

This study employs the Finite-Difference Time-Domain method to simulate the Localized Surface Plasmon Resonance effects induced by gold, silver, and copper nanoparticles on diamond substrates, aiming to recreate the rare pink, yellow, and blue hues observed in certain diamonds. The simulation results reveal that gold nanoparticles impart a pinkish hue to the diamond, silver nanoparticles produce a yellow tint, and copper nanoparticles create a blue shade. These color variations are significantly influenced by the size and arrangement of the nanoparticles, with optimized configurations enhancing the color effects in synergy with the diamond's crystalline structure. The findings of this study provide an

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innovative and cost-effective approach for the jewelry industry to manufacture colored diamond coatings and serve as a valuable reference for thin-film and coating technologies in applications involving optical components and sensors.

CM-ThP-6 Correlative XPS & SEM Analysis for NMC and Na-Ion Battery Cathode Material Surface Composition, James Lallo [james.lallo@thermofisher.com], Thermo Fisher Scientific, UK, USA; Nannan Shi, Albert Ge, Thermo Fisher Scientific, UK, China; Tim Nunney, Thermo Fisher Scientific, UK

Advanced energy storage has become increasingly vital in many fields, from transportation, to defence, to everyday connectivity. This has led to a growing market demand and development for lithium-ion battery storage solutions. High-tech products such as smartphones, tablets, drones, and electric vehicles all rely on compact, powerful energy storage, with lithiumion batteries being an essential component. Lithium battery primarily consist of cathode, anode, electrolyte, and separator materials. In lithium battery material research, how to comprehensively characterize and analyse battery materials, and how to use this characterization information to further improve battery material performance has become the focus of current researchers. This poster uses LiNixCoyMn(1-x-y)O2 (NCM)/LiCoO2 [NMC] composite cathode and Sodium Ion Fe/Mg cathode materials as examples. We employee a combination of Scanning Electron Microscopy (SEM) and X-ray Photoelectron Spectroscopy (XPS) characterization techniques to conduct a comprehensive analysis of the composite cathode materials. This approach yields rich sample information, helping researchers quickly evaluate and study any battery cathode materials.

The workflow combines scanning electron microscopy (SEM) [Thermo Scientific AXIA Chemisem] and X-ray photoelectron spectroscopy (XPS) [Thermo Scientific Nexsa G2 & ESCALAB QXi] into a correlated process, enabling the same regions of interest to be investigated; providing both high-resolution imaging and surface analysis from the same positions, even when collected using separate tools.

While SEM can easily visualize 2D materials, these layers are typically too thin to be easily characterized with the analytics commonly present on the microscope such as energy dispersive X-ray (EDX) analysis. XPS, meanwhile, cannot easily resolve surface structures at the required resolution, but can clearly detect what material is present at the surface, and quantify any chemical changes that might have occurred. XPS instrumentation typically also incorporates additional analytical techniques, such as an in-situ Raman spectrometer that is coincident with the XPS analysis position, which can be used to obtain further information.

CM-ThP-7 Optimizing Combinatorial Materials Discovery with Active Learning: A Case Study in the Quaternary System Ni-Pd-Pt-Ru for the Oxygen Evolution Reaction, Felix Thelen [felix.thelen@ruhr-uni-bochum.de], Rico Zehl, Ridha Zerdoumi, Jan Lukas Bürgel, Wolfgang Schuhmann, Alfred Ludwig, Ruhr University Bochum, Germany Steering through the multidimensional search space of compositionally complex solid solutions towards desired materials properties makes the use of efficient research methods mandatory [1]. Combinatorial materials science offers rapid fabrication, e.g. magnetron sputtering, and high-throughput characterization methods. Still, improvements to materials exploration cycles are necessary, since combinatorial methods are also suffering from the curse of dimensionality. At the scale of multinary systems, planning follow-up experiments based on already acquired data is economically feasible only through the use of machine learning techniques

In this study, we comprehensively explored the quaternary composition space of Ni-Pd-Pt-Ru for electrocatalytic applications with a streamlined discovery workflow. Enabling a fast synthesis, the fabrication of the materials libraries was performed by magnetron co-sputtering, and all libraries were subsequently characterized by energy-dispersive X-ray spectroscopy and X-ray diffraction. Guiding through the composition space, an active learning algorithm was used in an optimization cycle, which balances exploration and exploitation through the expected improvement acquisition function. The libraries were characterized electrochemically by an automated electrochemical scanning droplet cell setup [3] for the oxygen evolution reaction.

Six materials libraries were enough to find the global activity optimum in the system. The findings of six additional libraries are used to validate the activity trend. Our approach illustrates the potential of ML-driven optimization frameworks in accelerating the identification of promising multinary materials and underscors the value of integrating ML with high-

throughput synthesis and characterization techniques in modern materials science.

#### References:

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CM-ThP-8 High-Throughput Aging Studies of Vapor-Deposited Perovskite Thin-Films Using Precise Automated Characterization and Machine Learning-Assisted Analysis, Alexander Wieczorek, Sebastian Siol [sebastian.siol@empa.ch], Empa, Swiss Federal Laboratories for Materials Science and Technology, Switzerland

High-throughput experimentation (HTE) is increasingly being employed to accelerate metal halide perovskite (MHP) semiconductor thin-film development. (11) As of now, most approaches focus on solution-based deposition methods. To address the need for scalable and fabrication approaches, vapor-based deposition methods are gaining popularity. (21) However, durability concerns remain a major obstacle for large-scale deployment. (31) This motivates high-throughput stability studies of vapor-deposited MHP thin films. Combinatorial materials science is perfectly suited to address this challenge, specifically for time-consuming degradation studies where parallelization of experiments is key. (41) Using vapor deposition techniques, large parameter spaces can be covered on single substrates, whereas automated characterization and data analysis facilitate rapid properties screening. (51)

In this work, we present a comprehensive workflow for the aging of thinfilm MHPs which includes structural, optical and chemical characterization. [6] To mitigate ambient degradation during characterization or transfers, we employ a complete inert-gas workflow. Furthermore, we perform a rapid in-situ screening of the transmission and reflectance under accelerated aging conditions. The samples are exposed to 85 °C and 1 kW m<sup>-2</sup> white light bias, probing intrinsic material degradation in an accelerated fashion. With a temperature variation of ±1 °C and light intensity variation of <2% across combinatorial libraries, meaningful combinatorial stability screening is enabled. Automated characterizations of the structural properties yield deep insights into the aging process, extending and validating insights from changes in the optical transmission. We further demonstrate how these data sets can be used to better understand changes in the optical properties for highly scattering thin-films using machine learning assisted analysis. Furthermore, the workflow can be combined with high-throughput surface characterization techniques that our group previously demonstrated as a novel tool for accelerated materials discovery and optimization.

As a case study, we investigate the effect of residual precursors on the stability of two-step deposited MHP thin films grown on vapor-deposited templates. This workflow further allows to screen compositional spaces of libraries grown from completely vapor-based deposition methods.

#### References:

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CM-ThP-9 Advanced Depth Profiling of Thin Films Using Angle-Resolved XPS/HAXPES, Jennifer Mann [jmann@phi.com], Norb Biderman, Kateryna Artyushkova, Physical Electronics, USA

X-ray photoelectron spectroscopy (XPS) is a powerful technique for non-destructive analysis of the chemical composition of thin layers and interfaces. Angle-resolved XPS (AR-XPS) has traditionally been used with Al K $\alpha$  (1486.6 eV) X-ray beams to determine non-destructively determine layer

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thicknesses up to 5-10 nm below the surface. Recent advancements in ARXPS, including the integration of Cr K $\alpha$  (5414.8 eV) hard X-ray photoelectron spectroscopy (HAXPES), have extended capability to 15-30 nm below the surface.

PHI's *Strata*PHI analysis software has been developed to reconstruct quantitative, non-destructive depth profiles from angle-dependent and single-angle photoelectron spectra. The latest version of StrataPHI combines Al K $\alpha$  and Cr K $\alpha$  XPS and HAXPES data within a single depth profile, enhancing the analytical information extracted from various depths.

Modern microelectronics devices contain thin films with different properties and purposes. Chips are often comprised of conducting films that form the interconnect layers as well as dielectric films that provide electrical insulation. In multilayer stacks, buried interfaces and subsurface layers are often beyond the analysis depth of traditional XPS. The information depth enabled by combined XPS and Cr K $\alpha$  HAXPES is particularly useful for analyzing these types of materials.

This poster will discuss the principles behind AR-XPS and HAXPES, the new features of *Strata*PHI, and show some recent applications of the combination of these advanced methods to non-destructively probe thin films relevant to microelectronics.

CM-ThP-10 Numerical Ellipsometry: Artificial Intelligence Based Real-Time, in Situ Process Control for Virtual Substrates Including Multiple Unknown Layers, Frank Urban [fk\_urban@yahoo.com], 7980 SW 144th St, USA; David Barton, Florida International University, USA

Ellipsometry can be used to determine the optical properties and thickness of a thin film depositing on a known substrate based on light reflecting from the surface. This approach has the advantage of being able to be used in situ during the growth of the film with commercially available equipment to pass the light in and out of the deposition chamber. Nevertheless, a serious challenge in practice is that the material structure underlying the growing film commonly is composed of multiple layers. In these cases, very accurate knowledge of all of the underlying structure is required in order to obtain accurate results. Another challenge is that it the computation takes significant time using pre-existing iterative solution methods such as Levenberg Marquardt. The work here demonstrates the use of an Artificial Intelligence (AI) method suitable for real-time growth in which the underlying structure is complicated. This method is based upon previous development using five separate reflections simultaneously to solve for the underlying reflection coefficients at the same time the film parameters are being determined. The method is sufficiently fast that multiple groups of five measurements can be analyzed during the growth to confirm results and to examine the vertical homogeneity of the film being deposited. Examples will be given using a single angle of incidence. Thin absorbing films (up to 45 nm) will be given using a multilayer perceptron configuration consisting of 10 input neurons and 10 output neurons with two hidden layers of 80 neurons each. Solutions are performed at each wavelength independently and do not rely on fitting functions. The design, training and use of a number of neural networks will be presented.

CM-ThP-11 A Computational DFT Investigation of γ-CuI as an HTM for Perovskite Solar Cells, *Salma Naimi* [eng.salma.naimi@gmail.com], Green Energy Park (IRESEN/UM6P), Benguerir, Morocco/ Mohammed V university, Rabat, Morocco

Perovskite solar cells (PSCs) are recognized for their high efficiency and potential for low-cost production. However, the use of organic Hole Transporting Materials (HTMs) in these cells poses challenges due to their high cost and tendency to degrade the perovskite layer over time, threatening the commercial viability of PSCs.

In this study, we employed first-principles calculations based on Density Functional Theory (DFT), utilizing both the Generalized Gradient Approximation (GGA) and GGA + Hubbard correction, to evaluate the potential of y-Cul as a cost-effective HTM. Initial investigations involved a comprehensive geometry optimization to ensure structural stability, followed by an analysis of elastic and mechanical properties, which confirmed the material's compatibility with flexible PSCs [1].

The electronic and optical properties of γ-Cul were explored, revealing a low extinction coefficient and high refractive index across the infrared and visible spectra. Notably, γ-Cul demonstrated minimal reflectivity and absorption in key spectral regions, highlighting its potential to reduce optical losses in PSCs [1].

These findings position  $\gamma$ -Cul as a promising and economically viable HTM, offering significant advantages for the next generation of perovskite solar cells

Reference [1]

S. Naimi, S. Laalioui, E. Mehdi Salmani, K. Belrhiti Alaoui, and H. Ez-Zahraouy, "In-depth analysis of γ-Cul as an HTM for perovskite solar cells: A comprehensive DFT study of structural, elastic, mechanical, charge density, and optoelectronic properties," Solar Energy, vol. 276, p. 112680, Jul. 2024, doi: 10.1016/j.solener.2024.112680.

CM-ThP-12 Role of Gold-Doped Zno Nanoparticles to Degrade Dr-31 Dye as a Photocatalyst, *Manik Rakhra [rakhramanik786@gmail.com]*, Lovely Professional University, Jalandhar, India

Water contamination is a significant issue in the modern day, caused by the textile dyingbusiness, and it has a detrimental impact on living organisms. We report on the manufacture ofgold-doped ZnO nanospheres using a simple heat treatment approach, and the use of ZnOnanoparticles as photocatalysts for the degradation of methyl orange dye. To increase thisdegrading activity, Au was utilized as a modifier, and their temperature quenching effect wasnoticed. One of the most efficient electron grabbers in the conduction band is au ion. Thestructural, morphological, optical, electrical, and photo catalytic characteristics of thesynthesized nanocatalysts were determined. These nanoparticles have a grain size of 45-75 nm. Photocatalytic activity was investigated using UV Vis spectra, and a significant absorption peakabout 482 nm was discovered. With increasing frequency, the dielectric constant and frequencyof the produced nanoparticles drop. The kinetic analysis yields a rate constant of 0.0165 min -1 forNano sphere-like particles. At a concentration of 1% Au, the produced nanoparticles degrade thedye completely in 150 minutes when exposed to UV light.

CM-ThP-13 The Application of Environmentally Friendly and Sustainable Corrosion Inhibitor for Carbon Steel in Petroleum Fields, Omotayo Sanni [tayo.sanni@yahoo.com], University of Pretoria, South Africa; Ren Jianwei, university of pretoria, South Africa

In industrial sectors that deal with metallic materials, corrosion is a major problem. Steel corrosion causes significant economic losses in the oil and gas industry when oil wells are acidized. One common solution to this problem is the use of organic molecules as corrosion inhibitors. Therefore, the goal of this study was to determine the feasibility of using inexpensive, environmentally friendly, and organic compound from agricultural waste to reduce the rate of corrosion of carbon steel in an acidic environment that contains 1 M HCl. This research aims to investigate the potential use of agricultural waste as an inhibitory agent that can be reused for a variety of applications. Additionally, the extraction process in this work is done using water extraction. The compound was tested as a mitigator for the destruction of carbon steel in a 1 M HCl solution, and its composition was verified using a variety of spectroscopic techniques. Scanning electron microscopy-energy dispersive X-ray analysis (SEM-EDX) was used to investigate the surface of some corroded carbon steel samples in addition to electrochemical potentiodynamic polarization, impedance spectroscopy, and gravimetry studies. The data indicated that the addition of the waste compound inhibits the destruction of carbon steel by lowering the corrosion current density (iorr) and the double-layer capacitance (cdl). Tafel polarization data confirmed that the studied compound acted as a mixed inhibitor. The values of the cathodic Tafel slope (bc), are found to be near to each other demonstrating that the adsorbed chemicals did not modify the mechanism of hydrogen evolution. The spontaneity of the adsorption process is explained by the negative values of  $\Delta G^{\circ}$ ads.

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