A NEW METHOD FOR QUANTIFYING LIGHT DELIVERY IN A CLOSED-CELL GAS-PHASE *IN SITU* TEM SYSTEM.

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Photocatalytic materials are of growing interest for applications including CO_2 reduction, water purification, and solar fuel generation, yet photocatalysis remains underrepresented in *in situ* TEM studies [1,2]. A major challenge is integrating controlled light delivery while maintaining realistic gas environments, pressures, and accurate temperature control. Existing approaches often rely on specialized environmental TEM systems operating at relatively low pressures and can suffer from uncertainties in optical power delivery and photothermal effects [1].

Closed-cell gas-phase TEM platforms provide an alternative, enabling pressures up to 1 bar and temperatures approaching 1000 °C using MEMS-based chips that maintain realistic reaction environments. However, introducing light into these systems introduces challenges related to optical losses and illumination-induced temperature changes.

In this poster, we present a new methodology for introducing and quantitatively characterizing illumination in closed-cell gas-phase TEM (Fig. 1). The approach enables wavelengths from 350–2000 nm and combines a unique *ex situ* characterization with software-assisted measurements to determine optical losses and sample power density. Specialized MEMS chips maximize light throughput while maintaining calibrated temperature accuracy under illumination, establishing a practical framework for expanding in situ TEM studies of photocatalysts and other light-sensitive materials under realistic reaction conditions.

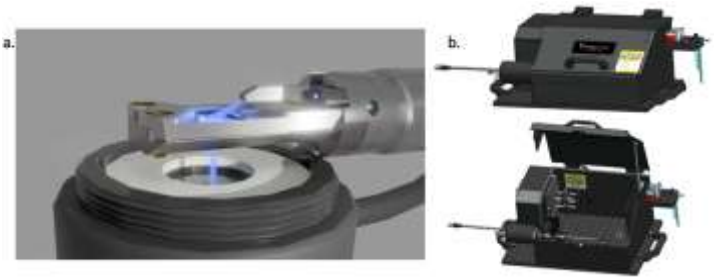

Fig. 1. a) Close up rendering of a new gas-phase holder (Protochips Sol for Atmosphere AX) with integrated optical fiber over a sensor for measuring power transmitting through the holder, which sits inside the *ex situ* characterization station and is performed before going into the TEM b) unique *ex situ* characterization station for measuring power density at the sample.

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UNRAVELING THE NANOSCALE SECRETS OF ELECTROLYSIS USING MULTIMODAL ELECTRON MICROSCOPY FOR A SUSTAINABLE HYDROGEN ECONOMY

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The transition to a sustainable energy future hinges on developing efficient and reliable hydrogen production technologies [1]. Electrolysis, whether it's the low-temperature methods like PEM (proton exchange membrane) and AEM (anion exchange membrane) electrolysis or the high-temperature approaches with solid oxide fuel cells (SOFCs) and solid oxide electrolysis cells (SOECs), is poised to play a pivotal role [2,3]. At the heart of these technologies are the electrochemical processes that ultimately determine the efficiency, durability, and overall performance of electrolysis systems. However, understanding the exact electrochemical mechanisms that govern the nanoscale processes is challenging and thus hinders the fast and widespread development.

We harness the power of advanced electron microscopy to delve into this nanoscale world. While transmission electron microscopy (TEM) is a cornerstone of materials science, its application to electrolysis materials presents unique hurdles. For example, the electron beam sensitive AEM and PEM electrolyte and catalyst layers demand low electron beam dose techniques as well as cryo sample preparation. Additionally, maintaining the critical hydration state of these materials during imaging is crucial for obtaining true information regarding the degradation mechanism. Furthermore, the limited field of view in TEM and the need for thin samples raises questions about the broader relevance of observations.

To overcome these challenges, we employ a correlative microscopy approach. By combining laser scanning microscopy (LSM) for large-scale context, cryo plasma focused ion beam (PFIB) for precise sample preparation, and TEM for high-resolution imaging, we construct a comprehensive picture of the material's structure and behavior. However, even these sophisticated ex-situ techniques cannot always capture the dynamic nature of electrochemical reactions. To truly understand these processes in action, we utilize in-situ TEM, allowing us to observe nanoscale phenomena in real time as they occur within the electrochemical environment.

This presentation will showcase examples from both low and high-temperature electrolysis, demonstrating how this integrated approach, combining LSM, cryo-PFIB, ex-situ TEM, and in-situ TEM, unveils the complex mechanisms underlying electrochemical performance and degradation. These insights are vital for guiding the development of next-generation electrolysis technologies that will drive the transition to a hydrogen-powered future.

Acknowledgments:
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NANOSCALE ELECTROCHEMICAL DYNAMICS OF COPPER NANOSTRUCTURES UNDER CONFINEMENT

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Understanding how copper behaves at the nanoscale during electrochemical reactions, especially within confined geometries such as nanopores or thin liquid films, is critical in the quest for miniaturizing electrochemical systems. Confinement can fundamentally reshape local ion transport and concentration gradients, thereby shifting pathways in processes such as nucleation and growth and dissolution/redeposition kinetics. This can, in turn, impact technologies from copper electrodeposition and corrosion to CO_2 electroreduction catalysts. In-situ liquid-cell TEM provides a unique route to directly visualize copper electrochemical behavior in confined conditions with nanometer-scale spatial resolution. By correlating real-time imaging with applied potential/current and, where possible, local structural/chemical contrast, liquid-cell TEM can reveal the dynamics of interfacial reconstruction, particle coarsening, and transport-limited instabilities that emerge under confinement.

In this work, we employed liquid-cell TEM to investigate copper growth and dissolution dynamics, inside nL-volumes of electrolyte and under electrochemical bias, to gain insights into the behavior of copper nanoparticles in the CO_2 electrochemical reduction reaction. We used a Cs-aberration-corrected JEM-ARM 200CF (JEOL, Japan), which was operated at 200 kV during the experiments. Experimental images and videos were recorded at the standard illumination conditions for real-time imaging. A Protochips Poseidon 500 liquid-cell holder was used for the in-situ experiments. Correlating the obtained electrochemical signals with time-resolved nanoscale images allowed us to resolve the interplay of electrochemical processes, including nucleation, growth, and dissolution, that cannot be inferred from electrochemical profiles alone [1].

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[1] The authors acknowledge support from the Fonds Wetenschappelijk Onderzoek in Vlaanderen (FWO, contract G0C3121N), the Slovenian Research Agency through the national program P2-0084, the Z2-50057 and J2-70084 ARIS projects, and the ARRS-FWO bilateral project N1-0196.

Investigating the Influence of Metal Ion Additives on the Electroplating Process of Zn using Liquid Cell Transmission Electron Microscopy

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Increasing energy demands in technological and industrial applications, from small-scale devices to automobiles, are driving the development of state-of-the-art rechargeable batteries that have improved durability and safety, and lower cost [1]. Alongside commerical Li ion batteries [2], batteries based on other metals such as Zn, Mg and Al have shown promise for meeting our growing energy needs [3]. From these possibilities, Zn has been identified as an attractive electrode material for rechargeable aqueous batteries because of its environmentally friendly nature and low cost. However, it can suffer from uncontrollable dendrite growth, surface corrosion and hydrogen evolution reactions [4]. Several strategies have been proposed to prevent dendrite formation, including electrolyte optimization by adding trace inorganic additives [5]. Nevertheless, the underlying mechanism of dendrite growth is not yet fully understood. Here, we use liquid-cell transmission electron microscopy to reveal real-time morphological changes during Zn plating from an aqueous $ZnSO_4$ solution, both with and without the addition of Cu^{2+} and Ni^{2+} ions. By combining these results with direction-resolved growth statistics, we present a holistic experimental framework for the study of additive-induced morphological variation at the nanoscale during Zn electroplating, which can be used to establish guidelines for the suppression of dendrite formation and for enhancing the stability and performance of Zn-based aqueous batteries.

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Correlating Atomic-Scale Dynamics and Reaction Products Using Aberration-Corrected ETEM with a Local Probe Mass Spectrometer

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Aberration-corrected transmission electron microscopy enables imaging of materials at sub-atomic resolution, providing insight into atomic structures and dynamic processes. When combined with environmental TEM (ETEM), aberration correction allows direct observation of reaction dynamics, such as the electrocatalyst–water interface during the oxygen evolution reaction (OER). In this context, correlating atomic-scale active catalyst states with reaction products detected by a mass spectrometer (MS) can reveal underlying catalytic mechanisms. However, achieving this combination remains challenging. Closed-cell ETEM setups integrated with MS often compromise spatial resolution and sensitivity due to diffuse scattering from SiN windows, while open-cell configurations in ETEM allow high-resolution imaging but suffer from dilution of reaction products as gases travel from the ETEM chamber to the MS. To address this, we aim to integrate aberration-corrected ETEM with a local probe MS (LPMS), enabling simultaneous high-resolution imaging and sensitive detection of reaction products.

The experimental setup combines a DENSsolution Stream gas/liquid flow biasing TEM holder with a MS (Fig. 1). To preserve spatial resolution, both the top and bottom membranes of the MEMS chip are removed, while the gas environment is controlled through the ETEM. Reaction products are transferred to the MS via a micro-scale capillary positioned near the sample and connected through one of the holder gas lines. Initial LPMS validation was performed in an environmental SEM (ESEM) using a custom flange for TEM holder insertion, demonstrating controlled gas delivery to the MS (Fig. 2).

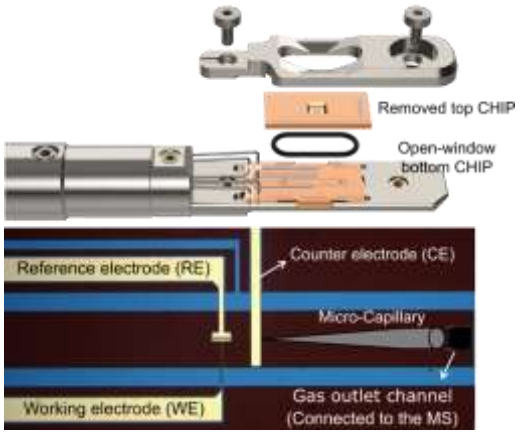

Fig. 1: TEM holder and CHIP design

Fig. 2: Demonstration of LPMS in an ESEM

Co_3O_4 nanoplates are investigated as a highly-efficient model catalyst during water splitting. Owing to their inherent electron transparency, atomic-resolution imaging can be achieved without TEM lamella preparation, thereby avoiding ion implantation and surface damage. In addition, a dedicated transfer approach is developed to position a defined number of nanoplates at the reaction site with controlled crystallographic orientation, enabling quantitative correlation between MS signals and atomic-scale ETEM observations.

IN-SITU ELECTRON MICROSCOPY INVESTIGATION OF PALLADIUM-ZIRCONIUM NANOPARTICLE CATALYSTS DURING DRY REFORMING OF METHANE

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The need to reduce greenhouse gas emissions due to the ongoing anthropogenic climate change has motivated research into methane reforming processes. One notable reforming process is the dry reforming of methane (DRM), during which two major greenhouse gases, CO_2 and methane, react to form a mixture of CO and hydrogen known as synthesis gas, an important feedstock for various chemical syntheses. A major challenge currently preventing widespread industrial application is catalyst deactivation through carbon deposition, commonly referred to as coking [1]. An example of filamentous coking of a spent catalyst is depicted in Fig. 1. Palladium nanoparticles supported on zirconia have been identified as a promising material system for inhibiting coking during DRM due to metal-support interactions as well as the carbon diffusion and storage properties of palladium [2,3]. In this work, in-situ scanning transmission electron microscopy (STEM) and scanning electron microscopy (SEM), combined with complementary materials characterization techniques such as Raman spectroscopy and energy-dispersive X-ray spectroscopy, are used to investigate coking phenomena during DRM at the nanoscale. The in-situ approach enables direct observation of structural and chemical transformations of individual palladium nanoparticles during reaction and deactivation. A palladium nanoparticle at an early stage of a DRM in-situ experiment is shown in Fig. 2. These microscopy-based observations provide insight into catalyst degradation mechanisms and are essential for the optimization of catalyst design and DRM operating conditions.

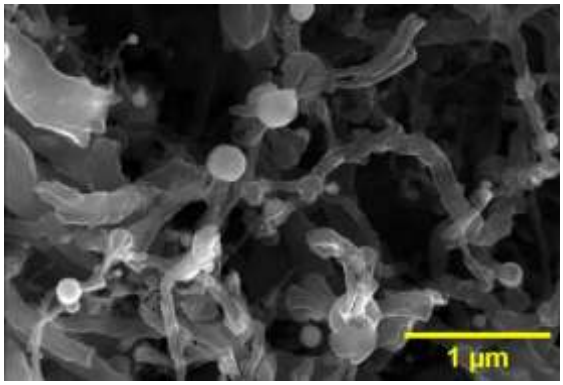

Fig. 1: Secondary electron SEM image of palladium nano-particles with filamentous coking

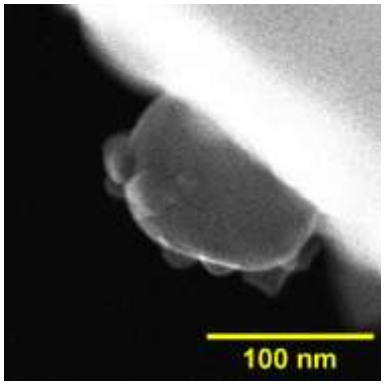

Fig. 2: Secondary electron STEM image of a growing palladium nanoparticle during an in-situ experiment

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In Situ TEM Study of Size-Controlled Bi Quantum Dots in an Annealed GaAsBi/AlGaAs Multiple Quantum Well Structure

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In-situ transmission electron microscopy study of Bi quantum dots (QD) formation in annealed GaAsBi/AlAs multiple quantum well (MQW) structure is presented in this work. The investigated structure, containing two GaAsBi QWs and embedded in AlGaAs parabolic quantum barrier (PQB) was grown on semi-insulating GaAs(100) and was transferred onto an in-situ heating holder (DENS solutions) and heated up to 650 °C. Sample evolution was continuously recorded in-situ in Bright-Field STEM mode. Analysis revealed that QD formation occurs at lower annealing temperatures in case of in-situ heating of lamella than in bulk. In addition, we find that the mechanism governing Bi QD formation is different in the in-situ TEM experiment compared to bulk ex-situ annealing. Comparison of the ex-situ and in-situ annealed structures, as well as in-depth post-annealed structure TEM analysis is presented. It is shown, that lamella sample transformation occurs in much lower temperatures than in bulk sample, also high Bi QD's diffusion rate suggest different quantum dot formation mechanism than in wafer sample annealing. An obvious correlation between the size of the QDs and the width of the GaAsBi QW can be traced from the BF-STEM micrographs, providing technological route of formation of Bi quantum dots arrays of required QD size.

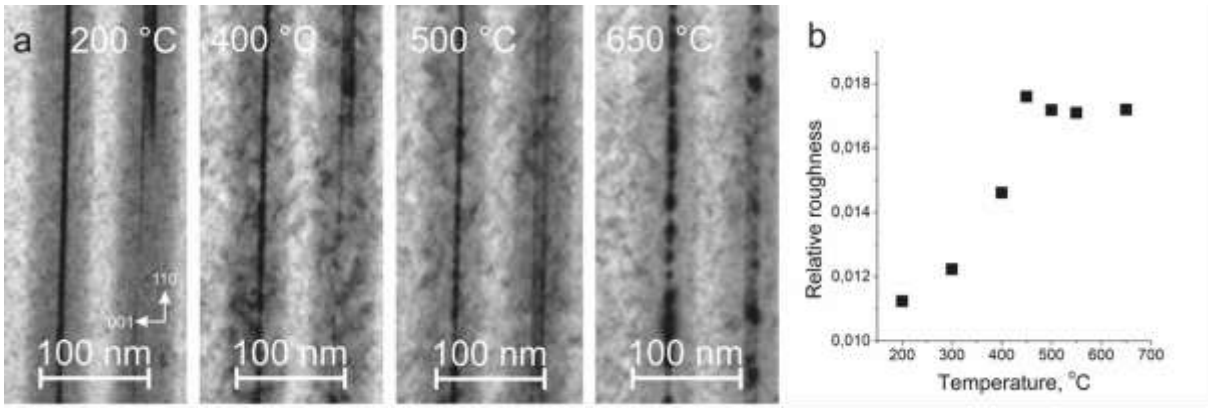

Fig. 1a) Sample evolution during heating experiment, and b) sample roughness dependence on heating temperature.

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Structural analysis of the 9R phase in Ir-Pd-Pt-Rh-Ru compositionally complex solid solution thin films by nano-beam electron diffraction

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Compositionally complex solid solutions (CCSSs) have emerged as promising electrocatalyst candidates [1], and their catalytic performance is closely linked to their (micro-)structural complexity arising from multielement mixing and associated crystallographic characteristics. In this study, we utilize nano-beam electron diffraction (NBED) to investigate 9R phase formation in Ir–Pd–Pt–Rh–Ru CCSS thin films, with a particular focus on the distribution of the 9R phase and the interface structure between the 9R phase and the CCSS matrix. The films were fabricated by magnetron sputtering onto a Pt buffer layer on a sapphire substrate. Based on X-ray diffraction and scanning transmission electron microscopy (STEM) with energy-dispersive X-ray spectroscopy, the equiatomic film exhibit a face-centered cubic (FCC) structure and a homogeneous elemental distribution throughout the film.

Despite this chemical homogeneity, a distinct type of planar defect is observed in the equiatomic film. The defect appears as finely spaced stacking faults and shows additional reflections at intervals of $\frac{1}{3}\mathbf{g}_{111}$ along the [011] zone axis of the FCC matrix in the fast Fourier transform pattern, indicating a more complex structure than conventional twins or stacking faults. Since stacking faults can disrupt local atomic arrangements more strongly than twins in CCSS [2], the presence of 9R phases is also expected to contribute to local structural complexity. The NBED data enable identification of the 9R phase, local crystal orientations, and their distribution throughout the film. In addition, high-resolution TEM is used to study the interface structure between the 9R phase and the matrix. Finally, we address how further Ru enrichment—given its preference for a hexagonal close-packed structure—may affect 9R phase formation in this system.

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Electron Holography of Light-induced Charge Transfer

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Engineering internal electrostatic landscapes in nanoscale devices enables control over photo-excited charge carriers and underpins applications ranging from photocatalysis and photovoltaics to memristive computing and biological energy conversion [1]. Characterizing the interplay between photo-excited charge and internal electrostatic landscapes is therefore essential for understanding both natural and engineered energy-conversion systems.

Electron holography provides direct access to electrostatic potentials with nanometer resolution and has famously mapped built-in fields in semiconductor devices [2]. With the advent of ultrafast transmission electron microscopy and laser wavefront shaping, the use of laser has become standard practice in electron microscopy [3]. Combining laser excitation with off-axis holography is therefore a promising new method, offering sensitivity to light-induced potential changes. Recent laser-assisted holography has already visualized light-induced potential variations in nanoparticles and PN junctions [4,5], but spatially resolving the charge redistribution response inside a semiconductor device remains outstanding.

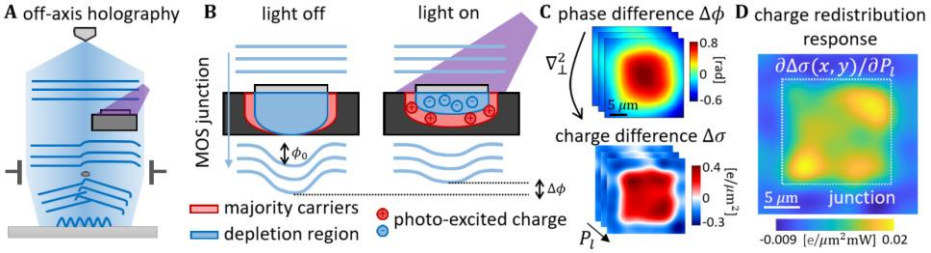

Figure 1: Laser-assisted electron holography of photo-induced screening in an MOS junction. (A) Off-axis holography records the electron phase. (B) Illumination generates photo-excited carriers that screen the junction field, producing a phase difference $\Delta\phi$ between light-off and light-on conditions. (C) The transverse Laplacian of $\Delta\phi$ (for a range of laser powers P_l) yields the projected charge density difference $\Delta\sigma$ (Eq. (1)). (D) Laser power-dependent measurements provide a spatial map of the local photo-response $\partial\Delta\sigma/\partial P_l$ within the junction.

Here we perform continuous-wave laser-assisted off-axis electron holography to spatially resolve the charge redistribution response of a 60 nm thick metal–oxide–semiconductor (MOS) junction (Si/SiO_2/Ti/Pt). Illumination generates photo-excited carriers that screen and confine the junction field, producing a measurable phase difference $\Delta\phi = \phi_{\mathrm{on}} - \phi_{\mathrm{off}}$ between laser-on and laser-off conditions (Fig. 1B). Wide-field holograms (~ 30 μm field of view) are recorded and reconstructed to obtain $\Delta\phi(x,y)$ (Figs. 1A,1C). The transverse Laplacian yields the projected charge density difference

$$\nabla_{\perp}^2 \Delta\phi(x,y) = -(C_E/\varepsilon_0)\,\Delta\sigma(x,y), \qquad (1)$$

where $C_E = 6.53 \times 10^6\ \mathrm{rad\,V^{-1}m^{-1}}$ for 300 keV electrons is the relativistic interaction constant and ε_0 is the vacuum permittivity (Fig. 1C). By measuring $\Delta\sigma$ over a range of laser powers P_l we extract the local photo-response using a slope map $\partial\Delta\sigma/\partial P_l$ (Fig. 1D), obtaining a mean response $\langle\partial\Delta\sigma/\partial P_l\rangle_{\mathrm{junc}} = 0.013\ \mathrm{e}/(\mu\mathrm{m}^2\cdot\mathrm{mW})$ with a standard deviation of $\sim 0.003\ \mathrm{e}/(\mu\mathrm{m}^2\cdot\mathrm{mW})$, demonstrating pronounced spatial variation in screening efficiency. The variation likely reflects non-uniform coverage of the ultrathin SiO_2/Ti/Pt layers that locally modifies the contact potential. These measurements establish a practical method to probe the interplay between the internal electrostatic landscape and photo-excited charge carriers in nanoscale devices.

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