Sondalo, Italian Alps | 25 - 30 June 2026

Symposium on microscopic characterization of the solid/liquid interface

SELSIM

Vallesana • 25–30 June 2026

High-Resolution Microscopy & Spectroscopy at the Solid–Liquid Interface

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Book of abstracts

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Program overview

Time	Thu 25/06/2026	Fri 26/06/2026	Sat 27/06/2026	Sun 28/06/2026	Mon 29/06/2026	Tue 30/06/2026
08:45 - 9:00		**Opening**				
09:00 - 9:50		**Prof. Steven De Feyter (remote connection)** *The liquid-solid interface up close: seeing is believing*	**Prof. Christine Kranz** *Bifunctional Microsensors for SPM Studies of Light-Driven Catalysis*	**Prof. Emmanuel Maisonhaute** *Tip Enhanced Raman Spectroscopy to retrieve local chemical information about the electrochemic al interface*	**Social event**	**Prof. Tomasz Kosmala** *Electrochemical Interfaces Under Bias: Structure, Dynamics, and Reactivity by EC-STM*
09:50 - 10:30		**Prof. Markus Lackinger** *Monolayer polymorph selection at solid/liquid interfaces: The more expected, less obvious and surprising influences*	**Prof. Giampaolo Mistura** *Passive and active control of drop motion on engineered surfaces*	**Prof. Alessandra Serva** *Understanding electrolyte effects at solid/liquid interfaces: insights from molecular dynamics simulations*	**Social event**	**Dr. Kunal Mali** *It's Not A Bug, It's A Feature: Defects in (Moiré) 2D Polymer Bilayers*
10:30 - 10:55		**Jie Zhang** *Nanoscale Puzzle Games Disorder and Random Tiling of Naphthalene-Based 2D Polymers*	**Simone Buoso** *Sliding of water droplets across an optical force barrier*	**Dr. Marco Menegazzo** *Unveiling the spectroscopic properties of intercalated stratified*	**Social event**	**Matilde Padula** *New Insight into the Lifting of the Au(111) Reconstruction in Perchloric Acid: an Electrochemical*

Program overview

				systems: A focus on HOPG Blisters		*Scanning Tunneling Microscopy Investigation*
10:55 - 11:15		**Coffee break**	**Coffee break**	**Coffee break**	**Social event**	**Coffee break**
11:15 - 11:40		**Dr. Micaela Castellino** *From UHV to Operando NAP-XPS: Probing Electrochemical Solid/Liquid Interfaces*	**Dr. Kaigang Yang** *Dynamics of 2D microfluidics crystals in patterned Hele-Shaw cells* **Francesco Pierini** *Interpretation of time-resolved nanoscale calcite dissolution through multimodal probability distributions*	**Jessenia Villaverde Herrera** *Electrocatalytic Conversion of CO_2 to Ethanol on Multifunctional High-Entropy Oxide* **Erfan Afshar** *Graphene Membrane Electrochemical Cell for High-Vacuum SEM Observation of Electrocatalytic Reactions*	**Social event**	**Paulina Wira** *Watching Catalysis Unfold: Operando Insights into Oxygen Reduction Reaction Driven by a 2D Molecular Network* **Anna Futyma** *Uniaxial Electrocompression and Domain Wall Formation in Iodine Adlayers on Au(111): an EC-STM study*
11:40 - 12:05		**Ashraf Abdelrahman** *Probing Dynamic Surface Reconstruction at Solid-Liquid Interfaces Using Near-Ambient Pressure X-ray Photoelectron Spectroscopy*	**Sarah Horn** *In-Situ ec-AFM Investigation of Organic Polymer Composite Electrodes*	**Dr. Frederic Kanoufi** *Visualizing the Solid/Electrolyte Interface by Operando Optical Microscopy: From Batteries to Corrosion*	**Social event**	**Xingyu Li** *Tailoring the Interfacial Microenvironment: Impact of Hydrophilic/Hydrophobic Interface on CO_2 Reduction Selectivity*
12:05 - 12:30					**Social event**	

Program overview

12:30 – 12:55		**Dr. Guglielmo Albani** *Tracking Chlorine across the electrochemical interface: challenges and opportunities for operando NAP-XPS at solid-liquid boundaries*	**Prof. Gianlorenzo Bussetti** *Electrochemistry Under Confinement: From Sulfate Adlayers to Porphyrin Self-Assembly on Vicinal Gold Surfaces*		**Social event**	**Milda Tomy** *Beyond Catalyst Immobilization : Engineering Local Proton Donor Microenvironments for Molecular Electrocatalysis*
12:55		**Lunch**	**Lunch**	**Lunch**	**Lunch**	**Lunch**
15:00 – 15:50	**Registration**	**Prof. Marcello Campione** *Mechanistic aspects of the water-mediated CO_2 mineralization*	**Prof. Stefano Agnoli** *Responsive Solid/Electrolyte Interfaces: From Structural Dynamics to Catalytic Activity*	**Prof. Tobias Cramer** *Electrochemical Strain and Strain Waves in Mixed Ionic–Electronic Conductors*	**Social event**	**Prof. Andrea Auer** *Visualizing Solvent Structuring at Electrified Solid-Liquid Interfaces*
15:50 - 16:30		**Prof. Johanna Eichhorn** *Understanding Degradation of Catalyst–Semiconductor Interfaces via In-Situ Nanoscale Characterization*	**Prof. Shelley Claridge** *Polymerized Noncovalent Monolayers on 2D Materials: a Foundation for Nanostructured Material Design*	**Prof. Urban Bren** *Microwave Catalysis through the Lens of Atomistic Computer Simulations: Hydrolysis of Polymeric Materials*	**Social event**	**Dr. Arnaud Chovin** *Electrochemical probing of nanoscale bio-systems by AFM-SECM technique*
16:30 - 16:55		**Dr. Andrea Erroi** *Microwave-Assisted Spray*	**Dr. Marco Nalesso** *Point defects mediated activity and edge aging*	**Prof. Alessandro Podestà** *Atomic force microscopy for the nanoscale*	**Social event**	**Dr. Yivlialin** *Electron transfer kinetics of HOPG and GIC*

Program overview

16:55 - 17:20		*Carbonation of mineral slurries: Influence of Injection Mode and Reaction Parameters on Carbonate Formation* **Samantha Ancellotti** *Nanoscale AFM Imaging and Quantitative Dissolution Kinetics in Cadmium-Calcite Interactions at the Mineral-Liquid Interface*	*mechanisms on monolayer MoS_2 during acidic and alkaline HER investigated by EC-STM* **Marco Vigneri** *An integrated framework for AFM measurement and contact point detection in force-distance curves in chicken embryos fibrotic microenvironment*	*investigation of solid/liquid interfaces* **Lorenzo Dal Fabbro** *Mechanism of action of ionic liquids on biological membranes*	**Social event**	*investigated by cyclic voltammetry and scanning electrochemical microscopy* **Beatrice Morosini** *Towards a Process-Oriented Quality Control Protocol for Pt-Octaethylporphyrin Thin Films via Combined AFM–Raman Analysis*
17:20 - 18:00		**Sport - Volleyball**	**Coffee break**	**Coffee break**	**Coffee break**	**Coffee break**
18:00 - 18:40		**Sport - Volleyball**	**Prof. Claudio Conci** *New Technologies for merging Nonlinear Microscopy and Antimatter Spectroscopy for Intravital Imaging and Multimodal Characterization of wet life sciences*	**Dr. Louis Pacheco** – CSInstruments	**Prof. Magalì Lingenfelder** *A New Twist at Solid–Liquid Interfaces: Chiral-Induced Spin Selectivity for Energy Materials and Processes*	**Dr. Vitor Brasiliense** *Characterizing photoinduced reactions in the solid/liquid interface: control and mechanistic analysis with optical nanometrology*

Program overview

18:40 - 19:20	**Welcome-party and quiz game**	**Sport - Volleyball**	**free time**	**Prof. Victor Climent** *Use of electrochemical methods in combination with vibrational spectroscopy to probe the electrified interface*	**Dr. Cédric Leau** *Visualizing charge carrier transport at the photoelectrode–electrolyte interface under water oxidation conditions*	**Concluding remarcks + Awards**
19:30	**Welcome-party**	**Dinner**	**Dinner**	**Typical Dinner**	**Dinner**	**Dinner**
21:00	**Welcome-party** **Welcome-party**	**Table games - Chess** **Table games - Chess**	**Poster session** **Poster session**	**Party 80's** **Party 80's**	**Demo by CSInstruments** **Demo by CSInstruments**	**cinema** **cinema**
		lecture - 50 min invited - 40 min oral contribution - 25 min				

Social Event on the 29th Jun

Trip to VALGROSINA

Leaving Vallesana at around 9:00 a.m.

Lunch in a local montain shelter.

The liquid-solid interface up close: seeing is believing

Steven De Feyter

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Science is fun, especially at the liquid–solid interface. For many years, we have investigated the behavior of molecules at liquid–solid interfaces with the aim of creating new molecule-based nanoarchitectures and controlling the properties of molecularly modified substrates. To achieve this, we have developed and implemented a range of methodologies for surface modification and for the growth of molecule-thin films at liquid–solid interfaces.

Scanning probe microscopy, and in particular scanning tunneling microscopy, has proven to be an exceptionally powerful tool in satisfying our "seeing is believing" approach. Our ambition is to harness these modified substrates and thin films for a broad range of applications, including catalysis, sensing, spin-selective transport, and the doping of 2D materials.

In this presentation, I will introduce the methodologies we have developed for molecule-based nanostructuring at liquid–solid interfaces and highlight several representative applications. After all, seeing is believing. And the liquid-solid interface provides a lot of fun!

Monolayer polymorph selection at solid/liquid interfaces: The more expected, less obvious and surprising influences

Manuela Hocke[1], Oliver Ochs[1], Natalia Martsinovich[2], **Markus Lackinger[1, *]**

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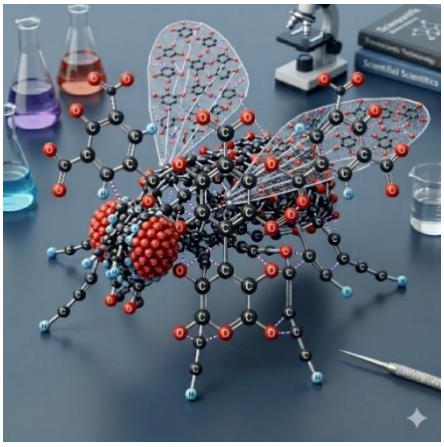
Polymorphism is an intriguing research topic of particular practical relevance in the pharmaceutical industry. Self-assembled monolayers at solid/liquid interfaces are a versatile model for fundamental studies of polymorphism, in which the surface adds specificity. Currently, it is not possible to predict the thermodynamically most stable monolayer polymorph under given conditions, notwithstanding additional kinetic influences. While identifying the polymorph with the highest free energy gain is key to solving this problem, accurately quantifying thermodynamic potentials experimentally remains challenging. Conversely, structures can be easily resolved in real space using Scanning Tunneling Microscopy. This provides access to a large database on polymorph selection under specific experimental conditions such as solvent, concentration, substrate and temperature. Additionally, valuable information can be obtained from variable temperature experiments on kinetic trapping and thermal stability as an indicator of thermodynamic stability.

Despite having been studied extensively, trimesic acid (TMA, benzene-1,3,5-tricarboxylic acid) monolayers continue to reveal new surprises, which is why TMA remains our model system of choice. By comparing surfaces with different interaction strengths, we discuss our current understanding of the roles of solvents and surfaces. Quantifying the free energies of the two main TMA polymorphs, chickenwire and flower, both of which are porous, indicates a significant thermodynamic contribution from solvent co-adsorption in the respective pores. Consequently, when exploring surfaces that interact more weakly, emerging differences in the relative adsorption strength of aromatic solute versus aliphatic solvent molecules can be crucial for polymorph selection.

While understanding the influences of reasonably well-controlled parameters remains challenging, cases of spontaneous polymorphism encountered time and again indicate additional, more concealed and less well understood influences. Pinpointing the underlying reason is worthwhile, as it provides unique insights. In the case of TMA, spontaneous polymorphism serves as a reminder that additional factors that are not actively controlled, such as solvent purity, must not be overlooked.

Keywords: polymorphism; trimesic acid; solvent co-adsorption;



Nanoscale Puzzle Games

Disorder and Random Tiling of Naphthalene-Based 2D Polymers

Jie Zhang,[1,2] Vipin Mishra,[1,2] Kunal S. Mali,[1,2] Steven De Feyter[1,2,*]

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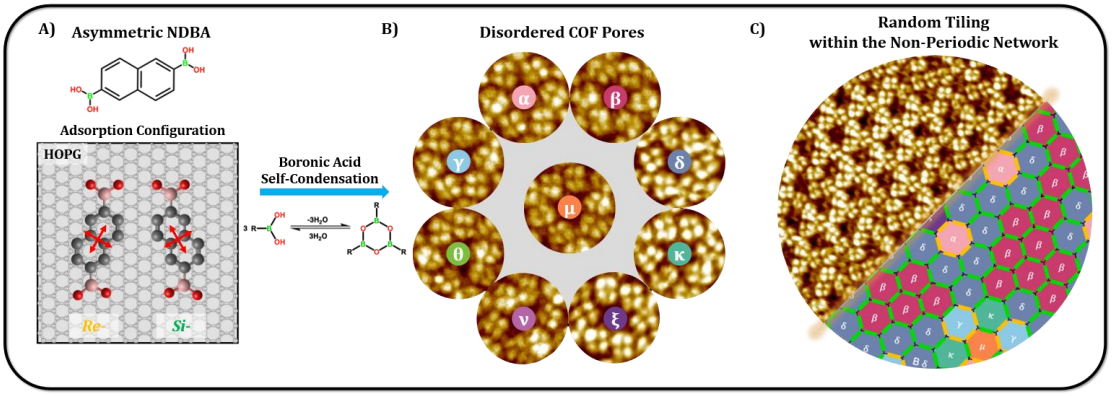
Two-dimensional covalent organic frameworks (2D-COFs) formed at solid-liquid interfaces provide a powerful platform for investigating how the order of polymer networks is influenced by monomer structures. Although 2D-COFs are commonly described as periodic crystalline networks, real structures can exhibit substantial nanoscale heterogeneity and defects that are difficult to access in bulk materials. Identifying this disorder and random tiling is essential for understanding the thermodynamic selection and kinetic evolution of 2D polymers. Here, naphthalene-2,6-diylboronic acid (NDBA), an asymmetric building block, was used to investigate how molecular adsorption configurations influence COF pore formation on graphite. STM imaging revealed that NDBA-COF forms an overall non-periodic polymer network composed of multiple pore types. Despite the absence of long-range periodicity, the tiling pattern exhibits local ordering tendencies. Different pore types show interdependent occurrence frequencies, connectivity preferences, and domain-dependent distributions, suggesting latent statistical order within the random tiling network. Notably, enantiomeric pores can coexist within the same COF domain, demonstrating that local chirality inversion can occur without complete domain separation. These results indicate that NDBA-COF is a thermodynamically selected non-periodic 2D polymer in which global disorder, local order, and nanoscale chirality inversion coexist. More broadly, this work highlights how molecular asymmetry and adsorption geometry can generate complex COF architectures beyond periodic crystalline frameworks with molecular-level resolution.

Keywords: Two-Dimensional Covalent Organic Frameworks; Scanning Tunneling Microscope; Random Tiling; Solid-Liquid Interface.



From UHV to Operando NAP-XPS: Probing Electrochemical Solid/Liquid Interfaces

Micaela Castellino

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Understanding the chemistry and dynamics of solid/liquid interfaces is essential for advancing electrocatalysis, energy conversion, and heterogeneous catalytic processes. Historically, the microscopic and spectroscopic characterization of this interface has been severely hindered by the "vacuum gap," as traditional electron-based techniques require ultra-high vacuum conditions incompatible with volatile liquids. Over the past two decades, Near-Ambient Pressure X-ray Photoelectron Spectroscopy (NAP-XPS) has emerged as a revolutionary operando tool, successfully bridging this gap and enabling the direct investigation of surfaces in equilibrium with liquid phases or realistic vapor pressures.

This contribution will present an overview of the methodological transition from conventional UHV-XPS to NAP-XPS, highlighting the technological developments that enabled the investigation of electrochemical solid/liquid interfaces under working conditions. Attention will be devoted to the implementation of operando electrochemical cells based on three-electrode configurations, which allow simultaneous control of electrochemical parameters and spectroscopic monitoring of surface chemistry. After a concise introduction to the fundamental principles of XPS and the challenges associated with liquid environments — including electron attenuation, pressure limitations, and interface stability — the talk will discuss the strategies currently adopted to access buried or dynamic interfaces. The role of differential pumping systems, graphene and membrane approaches, meniscus configurations, and X-ray excitation schemes will be briefly addressed to provide a methodological framework for both expert and non-specialists. The main part of the presentation will focus on operando NAP-XPS studies of catalytic materials under electrochemical polarization. Examples will illustrate how the technique enables real-time monitoring of oxidation states, surface composition, adsorbates, and electrolyte-induced transformations during catalytic operation. Emphasis will be placed on the correlation between electrochemical response and spectroscopic signatures, demonstrating how operando XPS can reveal transient chemical states and interfacial processes often inaccessible through ex situ characterization.

Finally, the strengths and current limitations of operando NAP-XPS for the microscopic characterization of solid/liquid interfaces will be critically discussed, including spatial resolution, time resolution, beam effects, and quantitative analysis. Perspectives on future developments combining spectroscopy, microscopy, and electrochemical control will also be outlined. This work was supported by the European Research Council (ERC) under the European Union's ERC Starting Grant agreement "CO2CAP" (Grant No. 949916). The author also acknowledges financial support from the Italian National Recovery and Resilience Plan (PNRR) project iENTRANCE for the acquisition and development of the NAP-XPS instrumentation.

Keywords: Operando NAP-XPS, Electrochemical Interfaces, Surface Characterization.

Probing Dynamic Surface Reconstruction at Solid-Liquid Interfaces Using Near-Ambient Pressure X-ray Photoelectron Spectroscopy

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Understanding the solid–liquid interface is central to advancing catalysis, energy conversion, and functional materials. While scanning probe microscopy techniques provide real-space insights into interfacial structures, complementary spectroscopic approaches are required to resolve chemical composition and electronic structure under realistic conditions. In this context, near-ambient pressure X-ray photoelectron spectroscopy (NAP-XPS) has emerged as a powerful method for probing surface chemistry in the presence of water vapor and liquid-like environments, offering direct access to chemical states at the solid-liquid interface.
In this contribution, we present a NAP-XPS-based approach to investigate dynamic surface reconstruction processes in layered materials exposed to water vapor and acidic environments. By systematically controlling environmental conditions, we track the evolution of surface species associated with hydroxylation, oxidation, and dissolution processes. These transformations are reflected in core-level binding energy shifts and line-shape variations, enabling identification of chemically distinct surface states.
Our observations reveal that surface terminations in layered systems are highly sensitive to liquid-phase analog environments, undergoing rapid restructuring even in the absence of externally applied bias. In particular, water exposure promotes hydroxylation processes, while acidic conditions induce selective removal and reorganization of surface species, effectively modifying the interfacial chemistry. These findings highlight the importance of decoupling purely chemical effects from externally driven processes when interpreting interfacial behavior.
By bridging controlled gas-phase exposures with liquid-like conditions, NAP-XPS provides a unique platform for studying the chemical complexity of solid-liquid interfaces. The methodology presented here complements scanning probe microscopy by offering chemically resolved insights into surface transformations, thereby enabling a more comprehensive understanding of interfacial phenomena.
This work establishes a general framework for investigating dynamic surface reconstruction at solid-liquid interfaces and underscores the role of combined spectroscopic and microscopic approaches in advancing surface science.

Tracking Chlorine across the electrochemical interface: challenges and opportunities for operando NAP-XPS at solid-liquid boundaries

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The combination of electrochemistry and near-ambient pressure X-ray photoelectron spectroscopy (NAP-XPS) offers unique opportunities for probing electrochemical interfaces under realistic conditions. However, truly operando measurements at solid-liquid interfaces remain highly challenging. A key difficulty is the need to follow the evolution of electrolyte-derived elements, here chlorine, while they redistribute across different chemical environments. In particular, the Cl signal must be distinguished as it evolves from solvated perchlorate species in the electrolyte toward possible intercalated states within the electrode structure, despite strong signal attenuation and overlapping interfacial contributions.

Here, we present the recent implementation of an electrochemical NAP-XPS platform developed at the Physics Department at Politecnico di Milano and discuss its potential for in-operando investigations. Highly oriented pyrolytic graphite (HOPG) in perchloric acid solutions is used as a model system to explore the experimental limitations associated with operando measurements. Ex-situ and quasi-operando experiments, including partial immersion and electrochemical treatments, allow discrimination between immersed regions, dry surface areas and the meniscus region, while providing insight into wettability, defect-mediated reactivity and ion intercalation phenomena.

These results highlight the intrinsic complexity of operando NAP-XPS at electrochemical interfaces and establish a methodological framework for tracking ion transfer and intercalation processes under realistic electrochemical conditions, with meniscus-controlled measurements playing a central role in the development of reliable operando methodologies.

Keywords: NAP-XPS; solid-liquid interface; surface reconstruction; hydroxylation.

Mechanistic aspects of the water-mediated CO_2 mineralization

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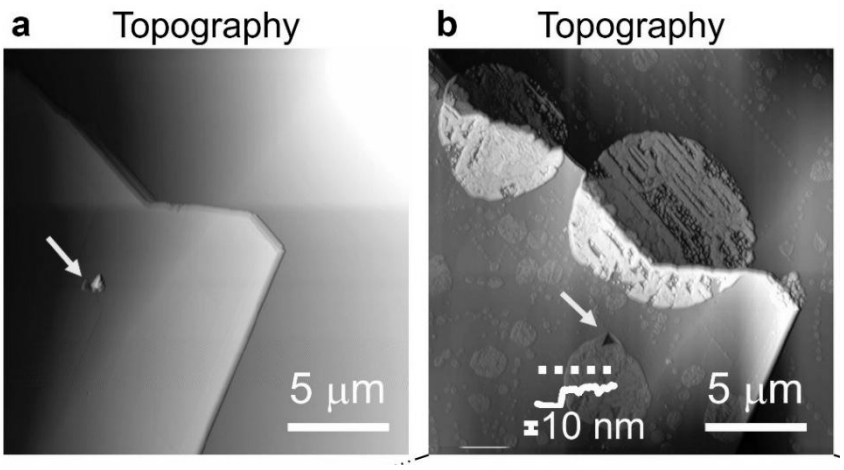
One of the pathways to CO_2 mitigation consists in accelerating the natural process of its mineralization. In this reaction, Mg- and Ca-bearing minerals (oxides, hydroxides and silicates) combine with CO_2 to form carbonates, which are very stable solids with the ability of permanently storing CO_2. This reaction, though thermodynamically spontaneous, occurs in nature with a very low rate, requiring millions of years to reach completion. One accelerating factor of this reaction is represented by the presence of water, assisting the release of metal ions and their precipitation as carbonates through processes occurring at the solid/liquid interface. The water-mediated mineralization opens some opportunities to enhance the reaction through physical-chemical methods. In a recent approach, the strong coupling of water and electrolytes with microwave radiation is exploited to efficiently heat the system to temperatures at which the reaction proceeds through the direct precipitation of anhydrous phases (magnesite and calcite). Additionally, the presence of proper electrolytes subjected to the energization of microwaves makes it possible to attain a catalysing effect, allowing for a strong reduction of the temperature at which precipitation of anhydrous phases occurs.

In this contribution, the results obtained in the field of the water-mediated mineral carbonation reaction, with particular emphasis on the prerogatives related to the use of microwave irradiation, will be illustrated. As reaction systems, mineral water slurries with defined concentration subjected to slight overpressures of carbon dioxide under mild hydrothermal conditions will be considered. To get insights into the microscopic mechanism of the reaction, scanning probe microscopy monitoring of the mineral surface subjected to carbonation treatments at the solid/vapour interface will be presented.



a Atomic force microscopy image of the surface of a freshly-cleaved single crystal of brucite; the arrow indicates a hole formed by nanoindentation for enabling its retrieval after *ex situ* successive treatments. **b** Same region as panel **a** after a carbonation treatment (10 min at 80 °C and RH ~ 5%, p-CO_2 = 5 bar + p-N_2 = 5 bar; the inset shows a cross-sectional profile (solid white line; vertical scale bar displayed) as taken along the white dotted segment.

Keywords: carbonation reaction; dissolution-precipitation; microwaves; atomic force microscopy.

Understanding Degradation of Catalyst–Semiconductor Interfaces via In-Situ Nanoscale Characterization

Sergej Levashov[1], Tim Rieth,[1,2] Ian D. Sharp,[1,2] and **Johanna Eichhorn[1]**

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The efficiency and stability of (photo)electrochemical materials are governed by processes at interfaces and surfaces, where atomic structure, electronic states, and chemical reactions meet. Disorder, local defects, and dynamic surface transformations strongly affect charge dynamics and transfer, often limiting performance under realistic operating conditions. However, material stability also strongly depends on nanoscale material imperfections, which often facilitate degradation processes. Despite the importance of nanoscale behavior, (photo)electrochemical performance and fundamental semiconductor characteristics are typically quantified by macroscopic techniques. While atomistic studies can provide insights into the fundamental reaction mechanism and enable the identification of electrocatalytically active sites, they often focus on idealized material systems and/or on idealized laboratory conditions.

To reveal nanoscale interfacial processes under realistic operation conditions, operando techniques with nano- to micrometer scale resolution are crucial. Here, we employ nanoscale in situ scanning probe microscopy to investigate the interfacial stability of photocathodes for hydrogen evolution by monitoring local transformation and delamination processes. We use sputtering and atomic layer deposition (ALD) for the deposition of Pt catalysts on conformal TiO_2 coatings with different nanoscale morphologies and interfacial characteristics. While sputtering produces a continuous Pt layer, ALD results in small Pt nano-islands, enabling the atom-efficient usage of precious metals. Under hydrogen evolution conditions, both systems exhibit similar onset potentials and saturation current densities, while chronoamperometry reveals a significant increase in the long-term stability of the nanostructured ALD-Pt films compared to the continuous Pt films. Specifically, the TiO_2/continuous Pt film experiences delamination from the underlying substrate, while the nanostructured ALD-Pt showed enhanced material durability. Direct imaging of the working surfaces allows us to identify different bubble formation processes, which can be assigned as the underlying cause of the variation in stability. Overall, the gained nanoscale understanding reveals the importance of optimizing material properties, while also highlighting the need for engineering interfacial processes to achieve durable material systems.

Keywords: Atomic force microscopy; photoelectrochemistry; degradation, thin films.

Microwave-Assisted Spray Carbonation of mineral slurries: Influence of Injection Mode and Reaction Parameters on Carbonate Formation

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Carbonation reactions at the solid–liquid interface are gaining increasing attention as promising pathways for carbon capture and utilization. In this study, we investigate a rapid and scalable approach to calcium carbonate synthesis through microwave-assisted spray carbonation of a model mineral slurry composed of portlandite [$Ca(OH)_2$] dispersed in water. The process is carried out in the resonant cavity of a modified microwave oven hosting an ultrasonic spray generator under controlled CO_2 injection.

Quantitative X-ray diffraction analysis, Raman spectroscopy and scanning electron microscopy imaging of the reaction products were carried out to investigate the effects of spray introduction into the resonant cavity relative to those of discrete droplets. These configurations were designed to modify the gas–liquid–solid contact area and mass transfer conditions during carbonation. The impacts of key operational parameters, including CO_2 injection time and microwave irradiation time, were systematically evaluated.

The results demonstrate that increasing the CO_2 injection time significantly enhances carbonation yield, indicating that prolonged exposure of the slurry to the reactive gas phase promotes more complete conversion of $Ca(OH)_2$ into calcium carbonates. Microwave activation further boosts this effect, likely due to enhanced reaction kinetics and improved heat and mass transfer at the microscale. Notably, the synergy between CO_2 availability and microwave energy input enables high conversion efficiencies within very short reaction times, with total process durations not exceeding 4 minutes. In addition to increasing overall conversion, microwave irradiation appears to influence phase formation, promoting the presence of aragonite among the carbonation products.

A major finding of this work is the strong influence of the slurry injection mode on the polymorphic outcome of calcium carbonate. While spray injection predominantly leads to the formation of calcite, the thermodynamically stable phase, droplet-based injection results in a relatively higher fraction of aragonite. This behaviour is attributed to differences in interfacial area and nucleation dynamics arising from the distinct dispersion regimes. In particular, the spray configuration enhances gas–liquid contact and promotes conditions closer to equilibrium, thus favouring calcite formation.

This study highlights the practical potential of microwave-assisted carbonation as a fast, energy-efficient, and scalable technology. The simplicity of the process and the short reaction times make it particularly attractive for industrial implementation. One envisioned application is the integration of this technique into flue gas treatment systems, where CO_2 emissions from industrial stacks could be directly converted into solid carbonates. In such a scenario, spraying calcium hydroxide slurry into exhaust streams under microwave activation could enable on-site carbon capture and mineralization, transforming a waste emission into a value-added material.

Overall, this work demonstrates that combining microwave irradiation with controlled slurry dispersion provides a powerful platform for tuning both the efficiency and selectivity of carbonation reactions. The approach opens new opportunities for sustainable CO_2 utilization.

Keywords: Microwave-assisted carbonation, microdrop reactions, CO_2 capture and utilizati

Nanoscale AFM Imaging and Quantitative Dissolution Kinetics in Cadmium-Calcite Interactions at the Mineral-Liquid Interface

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We investigate groundwater contamination by heavy metals at its fundamental level, focusing on the way mineral dissolution evolves during interactions taking place at mineral-fluid interfaces in the presence of flowing aqueous solutions. As a reference model, our work focuses on far-from-equilibrium dissolution of a crystal of calcite ($CaCO_3$) exposed to cadmium-contaminated water, tracking how surface morphology and reactivity change at the solid-liquid interface. Understanding these processes is relevant because metal adsorption and incorporation into mineral structures can alter the solid matrix, regulate metal mobility in subsurface systems, and ultimately influence water quality and development of preferential flow pathways. Carbonate minerals such as calcite are among the most abundant in the Earth crust, while cadmium (Cd) is a highly toxic and persistent contaminant. To address this, we combine high-resolution experimental observations with stochastic modeling. Dissolution experiments are performed upon relying on Atomic Force Microscopy (AFM), enabling *in situ* real-time imaging of calcite surfaces at the nanometric scale. From the ensuing topographic maps, spatially resolved dissolution rates are extracted and analyzed through a stochastic approach tailored to capture the heterogeneous nature of the reactive processes at the solid-liquid interface. A bimodal Gaussian mixture (GMIX) model is applied to characterize dissolution rates fluctuations under both ultrapure water and Cd-bearing solution flow. Our results reveal pronounced differences in calcite morphology and reactivity between the two environments. Even trace concentrations of cadmium fundamentally redirect reaction pathways by partially substituting Ca^{2+} within the crystal lattice, leading to the formation of otavite ($CdCO_3$), a less soluble secondary phase. This transformation enhances spatial heterogeneity, induces distinct localized behaviors across the surface, and progressively suppresses overall reactivity as the system evolves toward more stable configurations. Through integration of high-resolution morphological analysis with a stochastic modeling approach, our work establishes a quantitative link between nanoscale surface structure, fluid chemical composition, and dissolution kinetics. These methodological and operational advancements provide a robust and transferable framework for future investigations of mineral-metal interactions, with implications for modeling, remediation strategies, and metal recovery technologies.

Keywords: Atomic Force Microscopy; Calcite dissolution; Cadmium contamination; GMIX model.

Bifunctional Microsensors for SPM Studies of Light-Driven Catalysis

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The direct photocatalytic energy conversion of solar irradiation energy into chemical bond energy using molecular photosensitizers (PS) and catalysts (CAT) or non-toxic, sustainable polymeric carbon nitrides (PNCs) has gained attention as an environmentally friendly approach for hydrogen (H_2) or hydrogen peroxide (H_2O_2) production [1,2]. To stabilize the PS and CAT molecules, store photogenerated charges and mitigate degradation of the active components, they can be embedded in functional matrices for e.g., light driven water splitting [3]. Among the materials for light driven H_2O_2 generation, ionic poly(heptazine imides) (PHIs) are attractive, by reducing O_2 in the presence of alcohols [4]. The use of functionalized miniaturized electrochemical sensors e.g., H_2 [5] or H_2O_2 [6] detection in combination with scanning probe microscopy allows the localized electroanalytical determination of light-driven products under operando conditions. In this contribution, we present approaches for bifunctional microsensors such as the use of an opaque glass sheath covering a platinum-based microsensor, in combination with scanning electrochemical microscopy (SECM), to enable the localized illumination and simultaneous H_2 detection at PS/CAT modified Langmuir films. Thereby H_2 evolution rates and apparent quantum efficiencies (AQEs) can be determined. In addition, Prussian blue (PB)-based microsensors with integrated silver quasi-reference electrodes (QRE) as single-electrode configuration will be presented, which can determine H_2O_2 in a so-called short circuit mode e.g., at PHI-films. Information on light-driven activity, morphology along with nanomechanical properties of such films can be obtained via colloidal atomic force microscopy (AFM)-SECM probes modified with electrocatalytic layers. Challenges and advantages of the presented sensing schemes will be discussed.

Keywords: Bifunctional electrochemical microsensors; scanning electrochemical probe microscopy; light-driven catalysis.

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Passive and active control of drop motion on engineered surfaces

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The actuation and control of liquid droplets is a key function in many microfluidic and industrial applications. Over the years, several strategies have been employed to accomplish this task. Passive control can be achieved by patterning the surface with domains that have different wettability, morphology, or magnetic properties. Active control is usually accomplished through external physical stimuli, including electric and magnetic fields, acoustic vibrations, and, more recently, light beams because of the high versatility and reconfigurability of optofluidic devices. In this work, we present an optofluidic platform based on a z-cut lithium niobate crystal doped with iron (Fe: $LiNbO_3$), a material with remarkable photovoltaic, piezoelectric, and pyroelectric properties. The face of the crystal in contact with the droplets is coated with a lubricant-infused layer, which guarantees hydrophobicity and, more importantly, a very slippery surface. The resulting platform is highly flexible and reconfigurable and does not require moving parts. Sessile water droplets that have volumes of microlitres, corresponding to millimetres in size, can be easily actuated, guided, merged and split by projection on the lubricated Fe: LiNbO3 crystal of suitable static or dynamic light patterns. The actuated droplets can cover distances of centimetres on a timescale of a few seconds. If the light spot is sufficiently intense, it can induce the break-up of millimetric water droplets. Two quite distinct break-up mechanisms are found: droplet splitting in two fragments of similar volume or jetting of very small charged particles, which leaves a charged fragment having practically the same initial volume of the droplet. In either case, one charged fragment does not move randomly, but rather follows one of three well-defined trajectories separated by 120°, reflecting the anisotropic crystalline structure of Fe:$LiNbO_3$.

Sliding of water droplets across an optical force barrier

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We present the results of an ongoing experimental study of the statics and dynamics of water droplets deposited on a tilted lithium niobate crystal coated with a slippery lubricant film. On a homogeneous inclined surface, sessile water droplets will slide at speeds that are proportional to the volume of the droplet and the sine of the inclination angle[1]. Illuminating an area of the crystal with a laser generates surface charges because of the bulk photovoltaic effect. The resulting electric field produces an attractive dielectrophoretic force[2], which can be measured simply by determining, for a given angle of inclination, the maximum volume of the droplet pinned by the photovoltaic field. In this way, it is possible to study the forces produced by changing the illuminated area. Particular attention will be devoted to investigating how the droplet crosses this optical trap.

Keywords: Droplets; Lithium Niobate; Lubricated surfaces.

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Dynamics of 2D microfluidics crystals in patterned Hele-Shaw cells

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We present the results of an ongoing experimental study about the statics and dynamics of 2D microfluidic crystals confined in a patterned microchannel. More precisely, water droplets of uniform size are produced with a T-junction and inserted into a Hele-Shaw cell whose top is patterned with micrometric holes made by standard lithography techniques. A small hole etched in the top wall generates an attractive force that can be estimated from geometric arguments [1]. In the presence of external flow, the water droplet pushed by the oil continuous phase can remain trapped only if the pinning strength of the hole is sufficient to balance the drag force. An anchored drop can remain stationary indefinitely as long as the driving flow rate remains below a critical value that depends on the sizes of the well and droplet. The statics and dynamics of 2D droplet crystals in the patterned Hele-Shaw at different flow rates are recorded via a video camera and the time position of the crystal droplets is deduced by using machine learning visual methods.

Keywords: Droplets; Microfluidics; Interfacial pinning forces.

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Interpretation of time-resolved nanoscale calcite dissolution through multimodal probability distributions

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We rely on a unique data set stemming from Atomic Force Microscopy (AFM) imaging of a {104} calcite ($CaCO_3$) crystal surface subject to dissolution under continuous flow. Calcite is the most abundant carbonate mineral in the Earth system. Quantification of fundamental processes underpinning dissolution reactions at calcite-water interfaces is key for enhanced understanding of geochemical processes in natural geological systems. We integrate high spatial and temporal resolution AFM imaging to obtain an extended sequential record of nanoscale calcite dissolution patterns under far-from-equilibrium conditions. We track the evolution of transient morphological features together with more persistent topographic attributes, as driven by randomly distributed lattice defects and captured under a unique experimental framework. While a deterministic characterization of such heterogeneous surface features is effectively unattainable, we interpret the extensive time series of topographic maps as a temporal sequence of multi-modal, spatially correlated random fields. In this context, distinct modes correspond to diverse mechanistic processes driving the markedly heterogeneous retreat of the crystal surface. A growing body of empirical evidence suggests that many environmental and hydrological variables exhibit (i) scale-dependent stochastic behavior across multiple spatial ranges, and (ii) probability density functions (PDFs) with non-Gaussian traits. Accordingly, we analyze experimental data through a rigorous theoretical framework that embeds joint assessment of the PDF of a target variable and its associated spatial increments (taken between locations separated by any given distance or lag) through a Generalized sub-Gaussian Mixture model (GSG-MIX). This approach enables: (i) accurate assessment of detailed features (including tailing and extreme values) and (ii) exploration of the temporal evolution of model parameters. We further examine entropy-based metrics (e.g., Shannon entropy) to quantify the information content of the PDF of the target variable as well as of its spatial increments. Keywords: Calcite dissolution; Stochastic modeling; GSG-MIX

Keywords: Calcite dissolution; Stochastic modeling; GSG-MIX

In-Situ ec-AFM Investigation of Organic Polymer Composite Electrodes

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Sodium-ion batteries (SIBs) are emerging as a promising alternative to lithium-ion batteries (LIBs) largely due to the lower cost and natural abundance of sodium.[1] However, current SIB technologies still rely on inorganic transition-metal compounds derived from limited natural resources.[2] Organic active materials (OAMs) have emerged as promising cathode materials for post-lithium ion batteries due to sustainability aspects and potential availability from renewable raw materials. The tuneable redox potentials, low-density, structural versatility and low toxicity of e.g, organic polymers, based on carbonyl compounds have gained attention as promising cathode materials in SIBs and rechargeable aluminium batteries (AIBs).[3,4]
To date, little is known about dynamic structural and mechanical changes during ion insertion and extraction in redox-active organic active materials. Here, we report in situ electrochemical atomic force microscopy (ec-AFM) that enables the investigation of structural and morphological changes at OAM-based cathode electrodes during cycling. This includes processes such as cathode-electrolyte interface (CEI) formation [4] and volume expansion or swelling of the material during insertion and deinsertion of the shuttling ions.[5] A major challenge associated with organic cathode materials is the partial dissolution of the material into the electrolyte.[6] To address this issue, we employ embedded microelectrodes with tuneable cavity depth, enabling in-situ ec-AFM studies of (interfacial) process at organic electrodes.

Keywords: ec-AFM, Sodium-Ion Batteries, Organic Batteries, Aluminium Batteries

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Electrochemistry Under Confinement: From Sulfate Adlayers to Porphyrin Self-Assembly on Vicinal Gold Surfaces

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Vicinal surfaces offer a powerful model system for investigating the role of atomic-scale confinement at electrochemical interfaces. In this contribution, we present the evolution of our studies from sulfate adsorption on vicinal Cu electrodes to, in the next future, the assembly of porphyrin molecules deposited by underpotential deposition on vicinal Au(111) and Au(100) surfaces.

Our previous work on sulfate adlayers showed how surface steps and terrace width influence adsorption, ordering, and interfacial electrochemical processes, and viceversa. Building on these results, we are now exploring molecular systems in which the molecule–substrate interaction is significantly weaker. This makes porphyrin adlayers particularly suitable for isolating the effects of geometric confinement imposed by vicinal surfaces.

By comparing ionic and molecular adsorbates, we investigate how step density and terrace size affect self-organization and electrochemical behavior at the solid–liquid interface. These studies provide new insights into the interplay between confinement, intermolecular interactions, and interfacial charge-transfer processes, establishing vicinal surfaces as versatile platforms for probing electrochemical phenomena at the nanoscale.

Responsive Solid/Electrolyte Interfaces: From Structural Dynamics to Catalytic Activity

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The solid/electrolyte interface plays a critical role in determining the activity and stability of electrocatalytic systems, yet its dynamic evolution under operating conditions is often poorly understood. Here we will focus on the electrochemical behaviour of a surface metal–organic framework assembled on Au(111) through N-heterocyclic carbene coordination. Combining operando electrochemical scanning tunneling microscopy with theoretical calculations, we show that the interface undergoes a potential-driven structural reorganization during the hydrogen evolution reaction. Under cathodic polarization, the initially ordered molecular framework evolves into a highly dynamic interfacial phase: metal atoms are extracted from the surface selvedge and redistributed as freely moving adatoms, with possible nucleation of new clusters and islands. This dynamic process generates undercoordinated gold sites at the solid/electrolyte boundary, which display near-optimal hydrogen adsorption energetics and enhanced catalytic activity. These results highlight how adaptive restructuring at electrified interfaces can create transient active sites that are inaccessible in static materials, providing new insights into the design of responsive interfacial architectures for electrochemical energy conversion.

Polymerized Noncovalent Monolayers on 2D Materials: a Foundation for Nanostructured Material Design

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Diacetylene topochemical photopolymerization has been broadly studied since the original report by Wegner in 1969. Most diacetylene polymerizations have been studied in 3D crystals and Langmuir films. However, diacetylene monomers with flanking alkyl chain segments can also assemble on 2D materials such as graphite and graphene, with the alkyl chains lying parallel to the substrate. Such assembly generates repeating lamellar patterns of aligned diacetylenes, which can also be photopolymerized, generating precisely spaced arrays of polydiacetylenes with nanometer resolution and sub-10-nm pitch.

Because the polydiacetylenes are exposed at the interface, we have found that they can undergo further reactions, enabling the monolayer to be covalently transferred to the surfaces of other materials, including PDMS and hydrogels that are crosslinked in contact with the polydiacetylene layer.

Importantly, these transfer reactions retain key elements of the nanometer-scale chemical patterning within the polydiacetylene array, enabling us to use the functionalized soft interfaces for controlled multivalent binding and other processes including cell adhesion. We will discuss factors impacting efficiency of the on-surface precision polymerization, as well as the subsequent transfer reactions and applications.

Keywords: on-surface reactions; topochemical polymerization; soft matter; regenerative medicine

Point defects mediated activity and edge aging mechanisms on monolayer MoS_2 during acidic and alkaline HER investigated by EC-STM

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Monolayer MoS_2 is a promising electrocatalyst for the hydrogen evolution reaction (HER) for its low-cost, sustainability and activity on the edges comparable to that of platinum-group metals. However, its activity and long-term stability at the solid–liquid interface are governed by dynamic nanoscale processes that remain poorly understood. Via **operando electrochemical scanning tunneling microscopy (EC-STM)** it is possible to directly link the formation and evolution of active sites like point defects and edge structures to HER activity and to pH-dependent aging mechanisms.

Imaging the surface under both acidic and alkaline conditions, we find that **point defects act as transient active centers**: their local electronic contrast correlates with HER processes. Edges and edge-like sites display distinct aging pathways: under acidic HER conditions edges tend to undergo reconstruction and desulfurization via hydrogen etching, whereas in alkaline media we observe accelerated defect-mediated morphological smoothing and loss of edge-like contrast consistent with chemical etching. These divergent behaviors explain the commonly reported pH dependence of MoS_2 catalytic performance and highlight why engineered edge density alone does not guarantee sustained activity.

Our EC-STM data illustrate the need for operando nanoscale characterization, revealing a mechanistic picture in which defect activity, generation and migration, and edge restructuring are coupled to the local electrochemical environment and applied potential, producing an everchanging dynamic landscape of active and inactive sites that provides useful information for the rational design of MoS_2-based electrocatalysts.

Keywords: MoS_2; hydrogen; operando; microscopy.

An integrated framework for AFM measurement and contact point detection in force-distance curves in chicken embryos fibrotic microenvironment

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Characterizing soft, hydrated biological tissues via Atomic Force Microscopy (AFM) requires adapting standard protocols to the constraints of compliant samples in liquid [1]. To ensure physiological relevance, thin biological samples must be mechanically immobilized without compromising structural integrity, while preserving their native hydrated state during indentation measurements. Furthermore, their pronounced spatial heterogeneity and extreme compliance yield low signal-to-noise ratios [2]. Consequently, determining the tip-sample contact point (CP) becomes a major source of operator-dependent uncertainty [3], which ultimately compromises Young's modulus estimation [2]. To address this, we present an integrated framework for the micromechanical characterization of the chicken embryo Chorioallantoic Membrane (CAM). Our approach couples a custom-designed fluid holder with an automated hybrid pipeline that combines coarse CP identification and statistical refinement.

CAM patches were secured in the holder providing macroscopic stability via a non-perforating O-ring sealing mechanism. The holder, fabricated by stereolithography (SLA) using glass-loaded resists (Rigid 10K Resin, Formlabs), consists of two interlocking components forming a chamber that can be filled with 3 mL of saline solution or phosphate-buffered saline (PBS), ensuring continuous sample submersion. The patch housing region includes a glass coverslip coated with a 70 nm sputtered gold layer, providing a reflective surface for optical monitoring of the tissue with an AFM-integrated camera. Force-distance curves acquired via spherical indenters (MLCT-SPH-5UM, Bruker) were processed using a custom two-stage automated CP detection algorithm. A macroscopic geometric step first isolates the impact region by maximizing curve deviation from a linear baseline. Here, the local ratio of variance (RoV, $\sigma_{left}^2/\sigma_{right}^2$) between adjacent sliding windows mathematically differentiates a near-constant, minimally fluctuating fluid baseline from a high-amplitude oscillatory contact regime. The definitive CP is pinpointed via a backward-scanning statistical veto: contact is triggered when the leading half of the sliding window breaches a baseline-derived noise threshold and passes a Welch's t-test, while the trailing half remains within the noise floor. Finally, the Young's modulus is extracted by applying the Hertzian model to the identified contact region.

The holder ensured compatibility with commercial AFM systems by mechanically immobilizing tissue under continuous fluid immersion, preserving the sample's hydrated state while allowing optical accessibility for real-time monitoring, precise site selection and micromechanical mapping. This stability is essential for obtaining force-distance curves required by our automated CP detection algorithm, which successfully uses the local RoV as a marker to differentiate between fluid baseline and oscillatory contact regime. By replacing operator-dependent thresholding with a statistical approach, the algorithm ensures reproducibility across samples, while visual inspection of Hertzian fits provides definitive quality control for physical consistency. This integrated framework standardizes the mechanical profiling of compliant biological tissues.

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Keywords: Atomic Force Microscopy; Contact point detection; Soft tissue biomechanics.

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New Technologies for merging Nonlinear Microscopy and Antimatter Spectroscopy for Intravital Imaging and Multimodal Characterization of wet life sciences

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The investigation of the solid-liquid interface in biological systems represents one of the most significant challenges in bioengineering and more in general in the medical diagnostic scenario. To this end, critical phenomena such as inflammatory responses, fibrotic progression, and metabolic shifts have been investigated through advanced tissue engineering technologies like synthetic scaffolds, lab-on-a-chip etc. Traditionally, medical imaging techniques and fluorescence microscopy ones provided detail of interfaces from the meso- to the nano- scale in vitro and therefore allowed to retrieve structural and dynamic data of the connected biological processes. However, characterizing these interfaces and processes within complex, living organisms require a perspective that may bridge the gap between micro-scale morphology and subnanoscale [1]. This talk presents a novel methodological framework that integrates label-free nonlinear microscopy with Positron Annihilation Spectroscopy (PAS) to provide a complete view of biological phenomena in vivo and in vitro. We promote the application of Positron Annihilation Spectroscopy as a transformative tool for preventive diagnostics. PAS probes the "free volume" or local electron density within biological matters potentially providing data on cellular mechanisms in a nondestructive way [2]. We discuss the transition from bulk dry materials analysis to a refined wet approach, and the realization of a positron accelerated beam compatible with biological tissues, able to create, for the first time, a connection between high-vacuum spectroscopic techniques and wet life sciences. This involves the use of variable energy positron beams which can control the depth at which annihilation events occur in biological layers. Furthermore, we address the critical technological integration required for measuring wet life science samples: the development of a "wet fluidic chamber" design that maintains biological samples in their native hydrated state under atmospheric pressure while interfacing positron beamlines. This prototype utilizes a thin silicon nitride (SiNx) window as a crucial interface between the high-vacuum beamline and the atmospheric wet environment. To ensure the viability of this approach for living matter, we performed preliminary dosimetry calculations, estimating a dose rate that ensures cellular integrity during long term acquisitions. A central pillar of this work is the use of the avian chorioallantoic membrane (CAM) as a versatile, ethical living organism-on-a-chip to bridge the dimension gap in detection techniques [3]. By utilizing the "Eggs&Beacon" platform, a synthetic-eggshell system designed for longitudinal intravital fluorescence imaging, we demonstrated the ability to observe the solid-liquid interface between 3D-printed micro scaffolds and the embryonic vasculature. This integrated approach, merging the micro-scale contextual information of nonlinear imaging with the nanoscale sensitivity of PAS, paves the way for a new generation of non-invasive, preventive diagnostic tools capable of identifying pathological transitions long before they manifest gross morphological changes.

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Keywords: Intravital Imaging; Positronium; Nonlinear Microscopy; Time-Resolved Spectroscopy

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Abstracts Jun 27th – Poster Session

N° 1

Validation of contact mechanics models for atomic force microscopy *via* finite elements analysis and nanoindentation experiments

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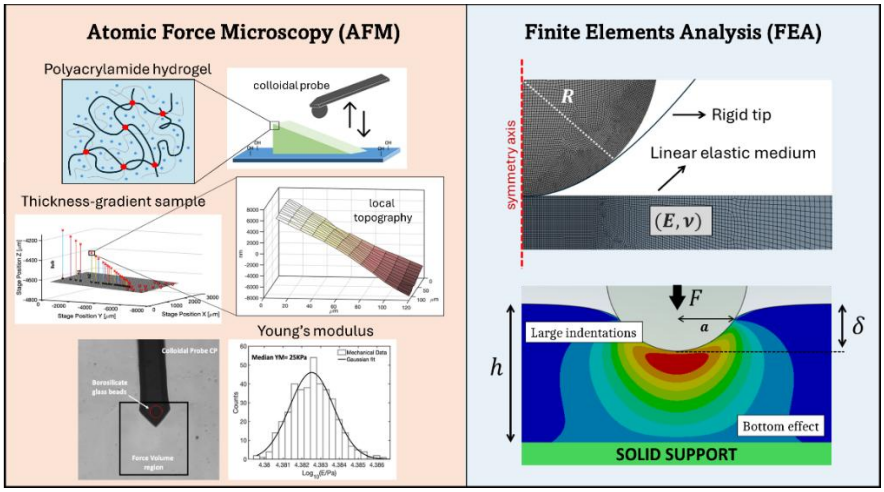
Atomic force microscopy (AFM) has emerged as a powerful experimental technique for investigating the nanomechanical properties of several systems through nanoindentation experiments, from cells and tissues to polymers. By measuring force-indentation responses, AFM allows the extraction of key mechanical parameters at the sub-micrometre scale. Among them, the Young's modulus (YM) quantifies the elastic response to compressive loads or, loosely speaking, to what extent something is "stiff" or "soft" with respect to compressions. In biological systems, the YM is strongly linked to the physio-pathological state of cells, including cancer progression, fibrosis, inflammation. However, the reliable estimation of the YM through nanoindentation experiments is generally affected by various artefacts, depending both on the sample under investigation (spatial confinement, elastic non-linearity) and on the tip used (sharp *vs* colloidal tips).



In the present work, for the first time we exploit the possibility to apply corrective factors to the force-indentations curves to simultaneously correct for the so-called "bottom effect", due to the vertical spatial confinement of the sample, and for the "large indentation effect", which is a consequence of the mathematical model used to fit force-indentation curves. We did so with a combined experimental/computational approach: on one side we performed nanoindentation measurements on polyacrylamide hydrogels possessing a thickness gradient, and on the other we performed finite elements analysis (FEA) simulations to validate the experimental results. Our results show, both numerically and experimentally, that the application of the proposed corrections allows to accurately quantify the YM of elasticity of linearly elastic samples using spherical tips in a wide range of indentation and thickness values that are relevant for the study of cellular systems.

Keywords: atomic force microscopy; finite elements analysis; nanomechanics; colloidal probes; polymers.

N° 2

Post-Mortem SECM Studies of the Solid Electrolyte Interphase (SEI) on Hard Carbon Electrodes for Sodium-Ion Batteries in Full-Cell Configuration

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Sodium-ion batteries (SIBs) represent an increasingly attractive and sustainable alternative to lithium-ion technology, specifically due to the high natural abundance, low cost, and global availability of sodium. Nonetheless, the commercial success of these systems depends on ensuring the long-term stability of the electrode/electrolyte interface [1]. Currently, hard carbon (HC) is regarded as the most promising negative electrode material, as it offers superior sodium storage capabilities compared to traditional graphite-based alternatives [2].

Recently, we characterized the solid electrolyte interphase (SEI) on spray-coated HC composite electrodes within half-cell configurations, specifically demonstrating stability of the SEI in 1M $NaPF_6$ in diglyme. Various factors, such as cathode-anode interactions, can significantly alter the evolution of SEI formation [3]. Post-mortem scanning electrochemical microscopy (SECM) provides a unique methodology for mapping local electron transfer kinetics and identifying heterogeneities in the SEI that contribute to cell degradation [4].

In this study, we present post-mortem SECM studies of the HC composite negative electrode cycled in a full-cell configuration using $Na_3V_2(PO_4)_3$ (NVP) as the positive electrode in 1M $NaPF_6$ in diglyme electrolyte. Utilizing SECM in feedback mode, we investigate the SEI formation after the cell was cycled for 40 cycles. We present the comparison of spatially resolved data on SEI formation observed in half-cells (vs Na metal) versus full-cells. By correlating electrochemical functionality with complementary surface-analytical techniques, including scanning electron microscopy/energy-dispersive X-ray spectroscopy (SEM/EDX), Raman spectroscopy, and X-ray photoelectron spectroscopy (XPS), comprehensive insights into the structure-property relationships of the SEI can be obtained, providing information on the influence of the cell configuration.

Keywords: Hard carbon composite electrode; solid electrolyte interphase (SEI); full cell configuration; post-mortem SECM.

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N° 3

Stiffness and mechanical manipulation of blisters grown on electrochemically intercalated graphite

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Electrochemical anion intercalation of highly oriented pyrolytic graphite in mineral acids (such as sulfuric, perchloric, etc.) represents a model system to study (i) the intercalation process of chemical compounds in stratified crystals; (ii) the evolution of gases at the surface and inside the sample and (iii) the evolution of swollen regions of graphite, called blisters. Although the explanation of the blister evolution mechanism can be dated back to the nineties of the last century, many chemical and physical properties have been explored only very recently. In particular, the mechanical properties of blisters on graphite are not widely discussed in the literature. Here, we employ in-situ atomic force microscopy (AFM) to characterize the blister stiffness. The same AFM tip is used to move mechanically the blister on the basal plane or even cut it. As a consequence of this operation, the entrapped gases (namely, CO, CO_2 and O_2) can deflate and the swollen graphite layers lay down to recompose the graphite basal plane.

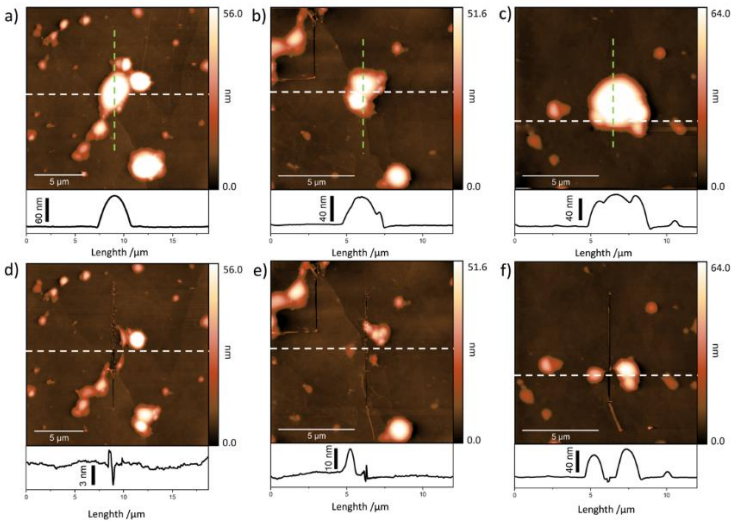

Figure 1. Topography images, acquired *in situ* and in non-contact mode, of three different blisters; respectively before (a, b, c) and after (d, e, f) their cut. The green vertical dash line evidences the path of the tip during the cut. The white dash line is in correspondence of the cross-section, reported at the bottom of each panel.

Keywords: Atomic force microscopy (AFM), nanoindentation, nanomanipulation, cyclic-voltammetry (CV)

N° 4

A modular platform to investigate calcite dissolution at near-freezing conditions via Atomic Force Microscopy

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We introduce a novel experimental platform enabling quantitative Atomic Force Microscopy (AFM) imaging of mineral-water interfaces under controlled liquid-flow conditions near water-ice phase transition. Freezing and thawing processes taking place at solid-liquid interfaces are key in driving a variety of environmental and engineered scenarios. These include, *e.g.,* (i) permafrost dynamics and (ii) degradation of mineral matrix composites in construction materials. Yet, major scientific and operational gaps are still persisting with reference to direct observation and quantitative interpretation of their impact on reactivity at mineral-water interfaces. Our approach circumvents the need of modifying the core AFM instrument upon resting on development of externally-integrated modules. Our novel platform integrates: (i) a thermal regulation system, (ii) a heat dissipation assembly designed through a customized sample holder, and (iii) an *ad-hoc* flow-control device enabling stable and well-defined chemical undersaturation conditions. Our study provides a robust framework for modular integration of external control units. We offer preliminary results of nanoscale time-resolved dissolution patterns emerging at the interface between water and a calcite crystal (i) in the presence of temperature conditions close to the water-ice phase transition boundary, and (ii) after detection of ice formation at the solid-liquid interface. This enhances our experimental capability to obtain AFM-based evidences of mineral dissolution processes under environmentally relevant low-temperature scenarios.

Keywords: Mineral Dissolution; Atomic Force Microscopy; Modular AFM-Integrated Platform

N° 5

Preliminary *ex vivo* AFM assessment of doxorubicin-induced biomechanical alterations in the chicken embryo CAM

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Chemotherapeutic agents such as doxorubicin remodel the extracellular matrix (ECM) and modulate cellular behavior [1][2]. However, their biomechanical impact at the tissue level remains poorly defined [2]. Moreover, most nanomechanical data still derive from simplified 2D monolayer cultures that fail to recapitulate native tissue architecture, structural constraints, and mechanical homeostasis [3]. To overcome these limitations, high-resolution techniques such as Atomic Force Microscopy (AFM) must be extended to physiologically relevant *ex vivo* models, enabling analysis of drug-exposed tissues after harvest while preserving native architecture [2][3]. In this framework, the chicken embryo chorioallantoic membrane (CAM) represents a vascularized 3D tissue reproducing key aspects of ECM organization not achievable in conventional *in vitro* cultures [4], with a heterogeneous architecture posing significant challenges for biomechanical mapping [3]. Here, we evaluate the feasibility of resolving localized, doxorubicin-induced mechanical alterations in the CAM using AFM force-distance curve analysis. At embryonic incubation day (EID) 10, *ex ovo* cultured chicken embryos were either topically treated with 200 µL of 34 µM doxorubicin or maintained as untreated controls, and CAM patches were collected at EID 14. Directly exposed regions were compared with distal, non-treated areas within the same embryos, alongside untreated controls. Fresh CAM explants were maintained under liquid immersion and analyzed by contact-mode AFM equipped with 5 µm spherical indenters (MLCT-SPH-5UM, Bruker). The apparent Young's modulus was obtained by fitting the initial 350 nm of indentation to the Hertz model. Initial measurements revealed sensitivity in detecting biomechanical differences between treated, distal regions and untreated controls. Force-distance curves were highly consistent across samples, supporting the robustness of the approach. Preliminary data revealed a heterogeneous mechanical response, with Young's modulus values around 1-2 kPa, consistent with the order of magnitude reported for other multilayered soft tissues (e.g. retina) [5]. This observed response indicates that drug-induced biomechanical effects are closely coupled with the heterogeneous CAM's architecture. Thus, accurate interpretation of pharmacological impact necessitates further methodological refinement, especially for *ex vivo* responses that require rigorous spatial tracking, accounting for membrane polarity (i.e. ectoderm and endoderm) and local microvascular organization (i.e. vessel and inter-vessel regions). Overall, AFM emerges as a promising approach for probing native 3D tissue mechanics and drug-induced alterations.

Acknowledgements: ERC, project BEACONSANDEGG, G.A.01053122

Keywords: Atomic Force Microscopy; Chorioallantoic membrane; Doxorubicin-induced remodeling.

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N° 6

Electrocatalytic Conversion of CO_2 to Ethanol on Multifunctional High-Entropy Oxide

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Electrocatalytic CO_2 reduction offers a promising route to mitigate the greenhouse-gas emissions from our heavy reliance on fossil fuels by capturing CO_2 and converting it into e-fuels and e-chemicals. In recent decades, numerous metal-oxide electrocatalysts have been developed for the electrochemical conversion of CO_2, yet they still lack the selectivity and stability required to produce C_{2+} hydrocarbons efficiently. In this context, high entropy oxides (HEOs) emerge as superior catalyst, offering high structural stability and surface-site heterogeneity capable of breaking scaling relationships and thereby enhancing C_{2+} product selectivity. In the present work, an HEO composed of Cu, Co, Ni, Bi, and Sn was synthesized by hydrothermal method. X-ray diffraction results indicated characteristic peaks of a pyrochlore structure (Fm-3m) stabilized as the main phase, accompanied by a small amount of CoO as a secondary phase. Scanning electron microscopy images showed a pyramidal morphology with an average particle size of 2.821 µm. Furthermore, EDS analysis revealed a multimetallic system with a nearly equiatomic atomic composition, which gave a configurational entropy of 1.56R for the system. The high-entropy oxide achieved an ethanol Faradaic Efficiency of 64.4% at -0.5 V vs. Reversible hydrogen electrode (RHE) for electrocatalytic CO_2-to-ethanol conversion. Quasi in situ Raman spectroscopy shows that applying −0.5 V vs RHE intensifies all M–O lattice modes, with marked amplification of the 386 cm^{-1} Cu–OH and 598 cm^{-1} oxygen-vacancy bands signatures of interfacial motifs that respectively stabilize CO for C–C coupling and promote CO_2 activation. The persistence of oxide features under bias indicates HEO-driven stabilization of undercoordinated Cu^+ sites at the electrode–electrolyte interface, rather than full reduction to metallic Cu. Potential-dependent interfacial evolution and its correlation with emerging C_{2+} selectivity are currently being investigated.

Keywords: CO_2 electroreduction; High Entropy Oxides; Ethanol; Quasi-in-situ Raman

Tip Enhanced Raman Spectroscopy to retrieve local chemical information about the electrochemical interface

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Surface modification methodologies are continuously refined, which in parallel necessitates accurate analytical methods to probe their chemical composition at the nanoscale. To this end, we will present the advantages of Tip Enhanced Raman Spectroscopy. This approach furnishes an accurate view over electrochemical interfaces by providing simultaneously local topography and vibrational fingerprints at the nanoscale.[1] We will present our TERS results on various surfaces, in parallel with their electrochemical behaviour. First, giant rotaxanes present fast electronic communication, and can be easily self-assembled thanks to dithiolane entities. Second, apart from thiol, use of carbenes (and in our case mesoionic carbenes) is finding an increasing interest, one major argument being that a strong Au-C bond is formed.[2] Our electrochemical results however lead to relativise this potential advantage. Third, while the use of diazonium salts is widespread particularly in the French community, the overgrafting and multilayer formation needs to be scrutinized for each system. To this end, we will compare the TERS spectra obtained after electrochemical reduction versus spontaneous grafting for the system displayed in Figure 1, that is of interest for molecular electronics.[3] Finally, we will also show the interest of performing electrochemical TERS to scrutinize the intermediates formed during the complex electroreduction of 4-nitrobenzylmercaptan. Our recent results for operando *observations* by EC-TERS will also be described.

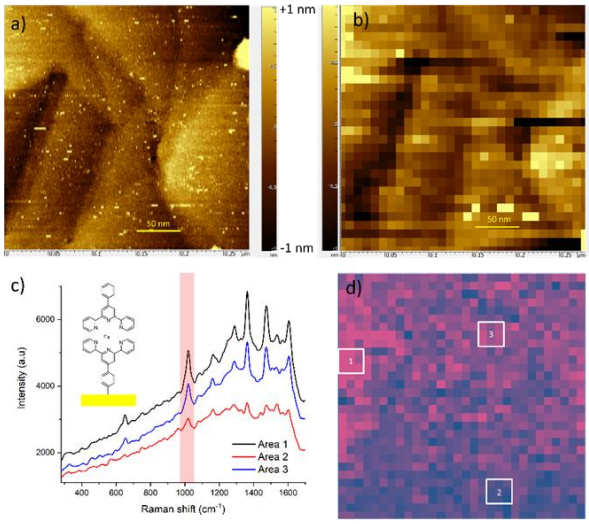

Figure 1 : a) Classical STM image obtained for a gold substrate modified with 1 by spontaneous adsorption. Bias voltage: 0.2 V. Setpoint current 200 pA. b) STM Topography obtained during TERS imaging. Bias voltage: 0.2 V. Setpoint current 200 pA. 30×30 pixels^2. Pixel size 9.33 nm. c) STM-TERS spectra obtained at positions 1, 2 and 3 d). Each spectrum was obtained by averaging a 3×3 pixels^2 area. d) TERS cartography corresponding to b) obtained after integration of the 1021 cm^{-1} band.

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Understanding electrolyte effects at solid/liquid interfaces: insights from molecular dynamics simulations

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In the optimization of energy storage devices and conversion applications, the electrolyte has emerged as a key component governing efficiency and performance. In this respect, understanding the behavior of the electrolytes at electrified interfaces, within the so-called electrochemical double layer, is essential for the rational design of improved systems. Despite its fundamental importance, many aspects about its structure and properties remain poorly understood at the molecular scale. Molecular dynamics (MD) simulations have proven to be a powerful tool for providing such microscopic insight and for complementing experimental investigations. In particular, over the past decade, a classical MD code has been developed in our group to simulate electrified solid/liquid interfaces, namely systems in which a liquid electrolyte is confined between two electrodes maintained at a constant potential.[1] In this talk, I will present some applications of this approach to the characterization of the structure of solid/liquid interfaces,[2,3] considering both planar and non-planar electrode geometries. I will also discuss the computation of key interfacial properties, such as the differential capacitance, and demonstrate the quantitative agreement achieved between simulation results and experiments. More generally, these studies highlight the capability of molecular simulations to provide detailed microscopic understanding of electrochemical interfaces relevant to next-generation energy storage technologies.

Keywords: molecular dynamics; solid/liquid interface; electrolyte.

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Unveiling the spectroscopic properties of intercalated stratified systems: A focus on HOPG Blisters

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Understanding the main steps but also the structural and physicochemical consequences involved in the **intercalation** of **stratified system** is an important subject in the energy storage industry and generally in surface science [1]. **Intercalation-induced blisters on highly oriented pyrolytic graphite (HOPG)** offer a powerful platform for exploring nanoscale mechanical deformation, electronic phenomena, and their broader technological relevance and these blisters, formed via electrochemical intercalation, provide valuable insight into interlayer dynamics [2,3].
In the recent years it has been shown the importance to use system that combines microscopy with spectroscopy to have a full comprehension on this intercalated system. This study combines **atomic force microscopy (AFM)**, **scanning tunneling microscopy (STM)**, and **Raman spectroscopy**, including **tip-enhanced Raman spectroscopy (TERS)**, to achieve high-resolution, in situ characterization of the physical and chemical properties of individual blisters and their intricated border regions. AFM and STM further elucidate blister morphology, mechanical stiffness, and atomic-scale surface features with exceptional precision. TERS enhances chemical sensitivity at the sub-nanometer scale, particularly useful for probing blister coalescence zones and edge dynamics.
The results shed light on the dynamic evolution and multifunctional character of these nanostructures, including the encapsulation of gases (e.g., CO, CO_2, O_2), drawing compelling parallels with **2D material bubbles**. Overall, this work highlights the transformative potential of combining scanning probe techniques with advanced Raman methodologies, bridging the gap between fundamental nanoscience and emerging applications.

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Electrocatalytic Conversion of CO_2 to Ethanol on Multifunctional High-Entropy Oxide

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Electrocatalytic CO_2 reduction offers a promising route to mitigate the greenhouse-gas emissions from our heavy reliance on fossil fuels by capturing CO_2 and converting it into e-fuels and e-chemicals. In recent decades, numerous metal-oxide electrocatalysts have been developed for the electrochemical conversion of CO_2, yet they still lack the selectivity and stability required to produce C_{2+} hydrocarbons efficiently. In this context, high entropy oxides (HEOs) emerge as superior catalyst, offering high structural stability and surface-site heterogeneity capable of breaking scaling relationships and thereby enhancing C_{2+} product selectivity. In the present work, an HEO composed of Cu, Co, Ni, Bi, and Sn was synthesized by hydrothermal method. X-ray diffraction results indicated characteristic peaks of a pyrochlore structure (Fm-3m) stabilized as the main phase, accompanied by a small amount of CoO as a secondary phase. Scanning electron microscopy images showed a pyramidal morphology with an average particle size of 2.821 µm. Furthermore, EDS analysis revealed a multimetallic system with a nearly equiatomic atomic composition, which gave a configurational entropy of 1.56R for the system. The high-entropy oxide achieved an ethanol Faradaic Efficiency of 64.4% at -0.5 V vs. Reversible hydrogen electrode (RHE) for electrocatalytic CO_2-to-ethanol conversion. Quasi in situ Raman spectroscopy shows that applying −0.5 V vs RHE intensifies all M–O lattice modes, with marked amplification of the 386 cm^{-1} Cu–OH and 598 cm^{-1} oxygen-vacancy bands signatures of interfacial motifs that respectively stabilize CO for C–C coupling and promote CO_2 activation. The persistence of oxide features under bias indicates HEO-driven stabilization of undercoordinated Cu^+ sites at the electrode–electrolyte interface, rather than full reduction to metallic Cu. Potential-dependent interfacial evolution and its correlation with emerging C_{2+} selectivity are currently being investigated.

Keywords: CO_2 electroreduction; High Entropy Oxides; Ethanol; Quasi-in-situ Raman

Graphene Membrane Electrochemical Cell for High-Vacuum SEM Observation of Electrocatalytic Reactions

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The integration of liquid or gaseous environments into electron microscopy (EM) remains a major challenge due to the high-vacuum requirements of these techniques. In recent years, graphene membranes have emerged as promising electron-transparent windows, enabling the separation of non-vacuum environments from the microscope chamber while preserving imaging capability. Literature reports highlight the exceptional properties of graphene, including its atomic thickness, high electron transparency at low primary beam energies, and outstanding mechanical strength, making it a strong candidate for in situ studies of electrochemical and catalytic processes.

Building on these developments, this work aims to explore the implementation of graphene windows for scanning electron microscopy (SEM)-compatible electrochemical cells. Graphene offers key advantages for SEM, particularly its transparency at low kinetic energies, which is critical for surface-sensitive imaging. However, several challenges remain, including the fragility of ultrathin membranes, limitations of current transfer processes, potential spectroscopic contributions of graphene itself (e.g., in reflection electron energy loss spectroscopy, REELS), and the controlled introduction of liquids or gases into sealed cells.

Mechanical and thermal considerations are also central to membrane performance. Graphene is known to exhibit a negative thermal expansion coefficient at low temperatures and an exceptionally high intrinsic strength (~130 GPa). Estimates from the literature suggest that a defect-free monolayer graphene membrane spanning a 10 µm aperture can sustain differential pressures on the order of 0.3–0.5 bar, with higher tolerances achievable using multilayer configurations. In parallel, Monte Carlo electron-transport simulations reported in the literature indicate that graphene membranes designed to withstand differential pressures exceeding several atmospheres can maintain high electron transparency at primary beam energies in the 10–30 keV range. These results underline the potential suitability of graphene membranes for SEM applications, although their practical implementation remains challenging.

The present work focuses on the experimental development of graphene membrane platforms. Graphene transfer onto Cu and Ni support grids has been investigated, with emphasis on process optimization and membrane integrity. These initial studies aim to establish reliable fabrication routes and to assess the robustness of graphene membranes under handling conditions relevant to SEM integration.

Future work will extend these efforts toward graphene transfer onto Si and SiNx supports, enabling more controlled and scalable device architectures. Additional investigations will include REELS measurements on both free-standing and supported graphene to evaluate its spectroscopic contribution, as well as the development of sealed cell structures using focused ion beam (FIB) techniques.

Overall, this work represents a step toward the development of graphene-based platforms for in situ SEM studies of electrochemical systems, with current efforts focused on fabrication and fundamental assessment of membrane behaviour.

Keywords: Wet cell, Electron microscopy, Electrochemistry, Focus ion beam

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Visualizing the Solid/Electrolyte Interface by Operando Optical Microscopy: From Batteries to Corrosion

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Electrochemical interfaces rarely operate homogeneously. During corrosion, electrocatalysis, or battery cycling, the global current measured at an electrode often operates through local events such as nucleation, dissolution, ion insertion, phase-front propagation, gas formation, or electrolyte redistribution. Operando optical microscopy offers a simple and powerful way to recover this missing spatial information by converting electrochemical activity into optical contrast.

In this presentation, I will discuss how wide-field, reflection-based, optical microscopy can be coupled to electrochemical measurements to image dynamic reactivity at the level of individual particles and local interfaces. In corrosion-related systems, optical reflectivity provides access to the spatiotemporal evolution of active sites, surface transformations, and chemical communication between reactive regions. When combined with image analysis or machine-learning-assisted classification, these movies can reveal reactivity patterns that are difficult to extract from electrochemical signals alone. In battery materials, optical contrast can similarly report on changes in oxidation state, ion insertion, electrochromism, phase-front motion, and particle-to-particle heterogeneity during operation. Examples will include operando optical imaging of electrochromic cation insertion materials and local probing of charge-storage.

The take home message of this communication is that optical microscopy should not be viewed only as an illustrative tool, but as a quantitative operando probe of materials conversion which can be further combined with scanning electrochemical probes for deciphering novel conrtributions in electrochemical processes. This correlative strategy provides a versatile framework for studying corrosion, electrocatalysis, and battery operation under realistic conditions, while remaining compatible with accessible laboratory instrumentation.

Keywords: operando optical microscopy; corrosion; electrochemical imaging; battery particles.

Electrochemical Strain and Strain Waves in Mixed Ionic–Electronic Conductors

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Materials exhibiting mixed ionic–electronic conductivity play a central role in emerging technologies, including energy storage, bioelectronic interfaces, and neuromorphic computing. In this tutorial lecture, I will discuss advanced atomic force microscopy (AFM) techniques for probing coupled ionic and electronic transport processes in these materials under operando conditions.
A key phenomenon in mixed conductors is the generation of local electrochemical strain arising from changes in ionic and electronic concentrations. AFM-based methods are uniquely suited to detect these minute strain responses with high spatial sensitivity, as demonstrated by electrochemical strain microscopy (ESM). I will present representative examples spanning conducting polymers as well as battery cathode and anode materials. Beyond conventional ESM, I will introduce advanced approaches that combine nanoscale strain measurements with electrochemical impedance spectroscopy, enabling direct access to transport kinetics and dynamic material responses. Finally, I will present the concept of strain wave microscopy, which allows visualization of ionic and electronic transport along mixed-conducting thin films and wires. Together, these methods provide powerful tools for investigating transport phenomena in functional materials across multiple length and time scales. [1–3]

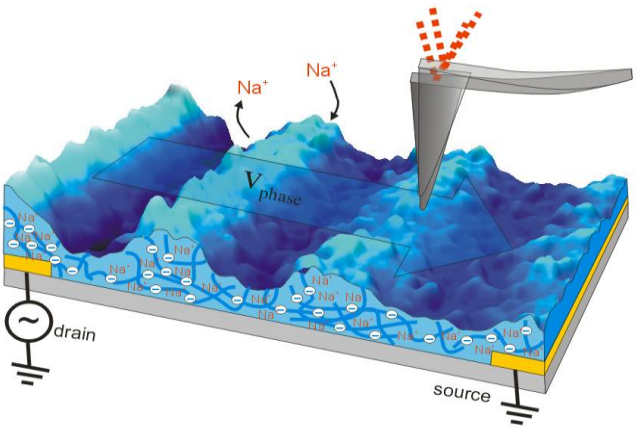

Figure: Schematic illustration of electrochemical strain waves (ESWs) generated in the channel of an electrochemical transistor. ESWs propagate into the device channel through combined charge transport and swelling.

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Microwave Catalysis through the Lens of Atomistic Computer Simulations: Hydrolysis of Polymeric Materials

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We propose a novel physical mechanism for microwave catalysis based on rotationally excited reactive species and verify its validity through computer simulations of realistic chemical reactions – neutral hydrolyses of polyester and polyamide materials. This nonequilibrium system is formally described by introducing rotational temperature, which is higher than the translational temperature. A Born-Oppenheimer surface was constructed on the density functional theory level and applied to derive an analytical expression for microwave catalysis. The simulations gave a reduced activation free energy when the rotational temperature was higher than the translational temperature, which constitutes a catalytic effect. For example, our calculation predicts that with rotational and translational temperatures of 310 and 300 K, respectively, the polyester hydrolysis should proceed 4.5 times faster than when both temperatures are 300 K. Moreover, this microwave catalytic effect is less pronounced at higher temperatures, which may have serious implications for the interaction of microwaves with living organisms in the context of widespread mobile telephony and wireless networks.

Keywords: microwave catalysis; polyester; polyamide; quantum-mechanical simulations.

Atomic force microscopy for the nanoscale investigation of solid/liquid interfaces

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Atomic force microscopy (AFM) allows to investigate solid/liquid interfaces thanks to its ability to combine truly nanoscale high-resolution imaging with the mapping of several interfacial properties, like wettability, electrostatic surface charge distribution, friction, (visco)elasticity, etc. The production of custom probes allows to adapt size and shape of the tip to the peculiar length scales of the system under study and to the analytical models used to fit the experimental data, while dedicated tip and surface functionalisation protocols permit to target specific molecular interactions. In this talk, I will present some examples of application of AFM-based techniques to the study of solid/liquid interfaces that are of relevance in different scientific fields, from materials science to biophysics and nanomedicine; these examples highlight the importance of nanostructure and nanoscale morphology on surface wettability as well as on the charging mechanisms of surfaces in electrolytic solutions.

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Mechanism of action of ionic liquids on biological membranes

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Ionic liquids (ILs) are peculiar chemical compounds possessing superior physio-chemical properties, such as negligible volatility, high ionic conductance, thermal stability, and the tendency to be in a liquid state at room temperature. This, combined with the ease of synthesis, makes them ubiquitous in the energetic and chemical industry. However, two decades ago it was found that they may exert an important cytotoxic effect toward a plethora of organisms, from eukaryotic cells, bacteria and plants. This, added to their stability, rises concerns about their persistence in the environment.

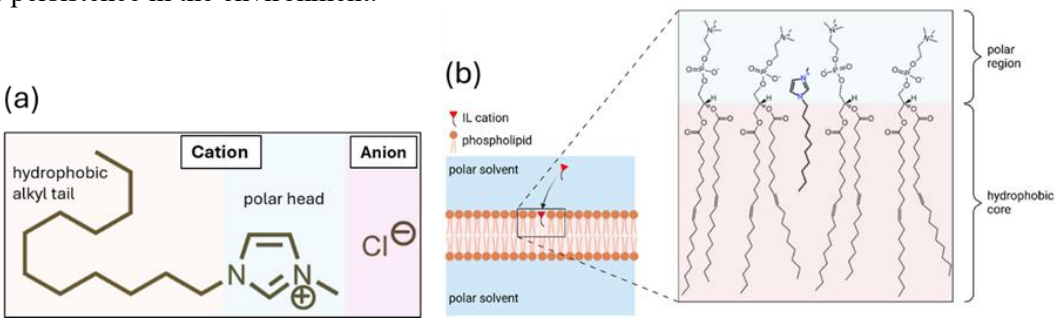

Figure 1: (**a**) structure of an Im-IL (**b**) Im-IL cation intercalation in a phospholipid membrane.

Among the different ILs which exert a cytotoxic effect, imidazolium-based ionic liquids (Im-ILs) are one of the most widely used. They are composed of an anion and a cation, the former generally being a simple monoatomic species (such as chloride, Cl^-), while the latter is an amphiphilic molecule with an alkyl chain of varying length and a polar head (Figure 1a). Because the structure is akin to that of phospholipids found in the plasma membrane of eukaryotic cells, the cation tends to intercalate into the membrane, affecting its fluidity and lateral cohesion (Figure 1b). This mechanism, which has been extensively studied in the past years, was attributed to the toxic effect toward eukaryotic cells. However, experimental observations have revealed that the cells' death may occur even after exposing the cells to extremely low concentrations of Im-ILs, at which the membrane's mechanical properties are not affected appreciably. This suggests that the mechanism of action of Im-ILs on cellular systems is more complex. Indeed, the functionality of protein-bound membranes, is expected to be affected by the presence of exogenous molecules in the membrane. Among the different protein, ionic channels are trans-membrane proteins responsible for the exchange of ionic species across the plasma membrane. However, literature lacks a systematic investigation of the effect of Im-ILs on ionic channels.

In my research project, I investigate the interaction of Im-ILs with real and artificial model membranes, mimicking to some extent the real ones of eukaryotic cells. Specifically, the goal is to characterize the interaction of ionic channels with Im-ILs. On one side, the change in the membrane's mechanical properties is studied by means Atomic Force Microscopy (AFM) on supported lipid bilayers. On the other, changes in ionic channels' functionality are studied with electrophysiology techniques, that is, single-channel current recordings and patch clamp.

Keywords: ionic liquids; lipid membranes; atomic force microscopy; ionic channels.

Use of electrochemical methods in combination with vibrational spectroscopy to probe the electrified interface

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This communication will revise the strengths offered by combining pure electrochemical methods, such as cyclic voltammetry and coulometry, with thermodynamic analysis, temperature variation, and spectroscopic measurements to examine the electrode-electrolyte interface. A novel methodology will be described for temperature variation, involving the use of a high-power pulsed laser to rapidly alter the interface temperature. The change in open circuit potential in response to this temperature shift can be analyzed using thermodynamic relationships to determine the entropy of formation of the double layer. This magnitude is closely linked to the orientation of water dipoles at the interface, depending on the electric field's sign. Finally, the vibrational signature of interfacial water complements the information from the laser-induced temperature jump, providing a comprehensive view of the microscopic structure of the interface.

Keywords: single crystal surfaces, surface enhance infrared reflection absorption spectroscopy (SEIRAS), laser induced temperature jump (LITJ)

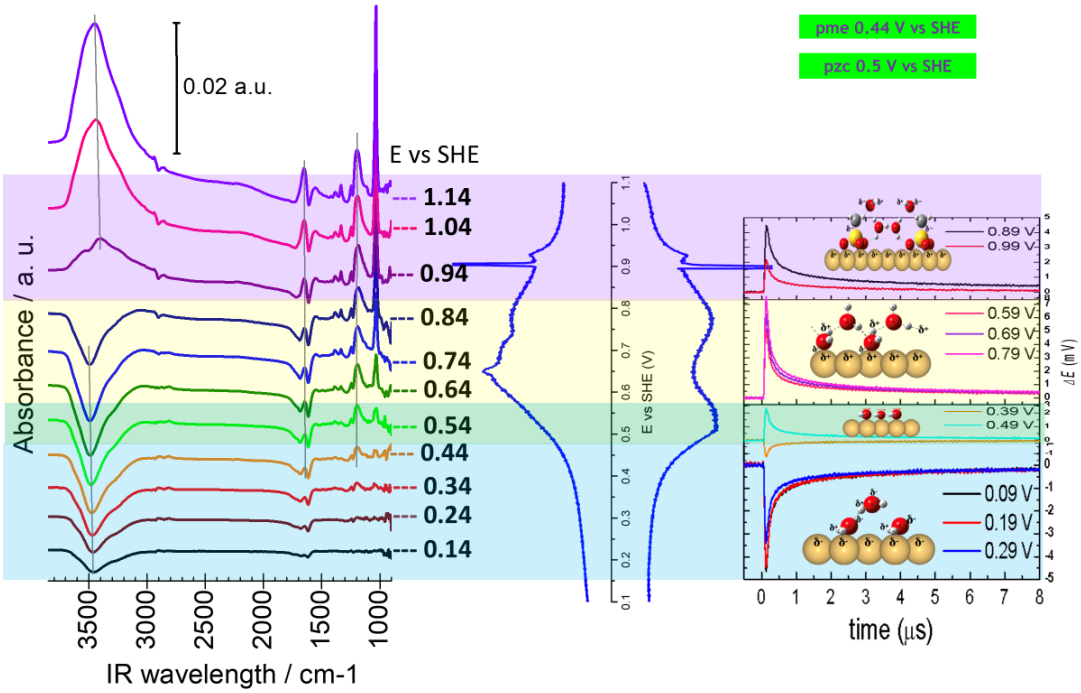

Figure: Multidimensional interrogation of the interfase between 0.1 M methanesulphonic acid solution and Au(111) electrode surface using SEIRSAS (left), Cyclic voltammetry (center) and LITJ (right)

A New Twist at Solid–Liquid Interfaces: Chiral-Induced Spin Selectivity for Energy Materials and Processes

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At solid-liquid interfaces, molecular chirality can imprint spin polarization on interfacial charge transfer and redefine how energy-relevant reactions are controlled. Yet these interfaces are dynamic and heterogeneous, so understanding chiral-induced spin selectivity (CISS) in catalysis requires methods that resolve structural and electronic evolution under operating conditions. In this talk, I discuss how scanning probe microscopy and operando nanoscale imaging provide this missing window into spin-selective energy materials, linking local interfacial restructuring to reactivity, selectivity and kinetic losses.

A broader framework has recently emerged in energy science in which chiral-induced spin selectivity (CISS) positions spin polarization as a new design variable for electrocatalysis, charge transport, and interfacial materials engineering [1]. Within this perspective, solid-liquid interfaces are the key arena: they host electric fields, adsorbates, solvent organization and catalytic intermediates, all of which can couple to spin-polarized transport. Early operando nanoscale studies of CO_2 electroreduction showed how Cu surfaces dynamically reconstruct under bias into nanocuboid motifs, illustrating both the complexity and the opportunity of following catalytic interfaces in real space [2]. Extending this interfacial viewpoint to water oxidation, operando imaging of Ni oxide catalysts revealed Fe incorporation, fragmentation and the emergence of dual active sites during oxygen evolution, clarifying how local restructuring governs performance [3].

To push this platform further, we combine operando nanoscale imaging with spin-polarized electrodes [4]. At the same time, we recently showed that maintaining spin alignment across the electrode-electrolyte interface promotes the oxygen reduction reaction [5].

Together, these results show that scanning probe and operando nanoscale methods do more than visualize catalytic surfaces: they reveal how spin, structure and dynamics become intertwined at solid-liquid interfaces. This insight opens a route toward chiral and spin-selective strategies for designing energy materials and processes with improved efficiency, selectivity and reduced dependence on critical elements.

Keywords: chiral-induced spin selectivity; solid-liquid interface; scanning probe microscopy; electrocatalysis

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Visualizing charge carrier transport at the photoelectrode–electrolyte interface under water oxidation conditions

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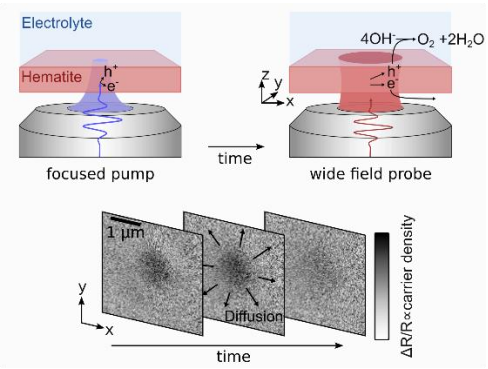
A major challenge in solar fuels is to identify and control the multiple factors that determine the efficiency of the light-driven molecular transformation under realistic operating conditions. In semiconductor photoelectrodes such as hematite, recombination and trapping of photogenerated charge carriers compete with their transport to the electrode-electrolyte interface, thereby limiting overall conversion efficiency. While these processes have been studied at microscopic spatial scales [1] or ultrafast temporal timescales [2], their simultaneous investigation remains largely underexplored. In particular, direct characterization of carrier transport is missing. Despite carrier diffusion length being a critical parameter in the calculation of photo(electro)chemical efficiency, it is rarely known.

Transient reflectance microscopy [3, 4] has emerged as a powerful method to spatiotemporally resolve the lifetime, transport, and diffusion length of photogenerated charges in a wide range of photoactive materials. This presentation will describe the development of this method to study hematite photoelectrodes under water oxidation conditions (Figure 1). I will discuss recent results that quantify how these carrier transport metrics are affected under applied bias.



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Keywords: Electrochemistry; Optical Microscopy; Energy Materials; Pump-Probe

Electrochemical Interfaces Under Bias: Structure, Dynamics, and Reactivity by EC-STM

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Electrochemical interfaces play a central role in electrocatalysis, energy conversion, corrosion, sensing, and materials synthesis. Their microscopic characterization remains challenging because the solid/liquid interface is inherently dynamic: its structure, composition, and reactivity depend on electrode potential, electrolyte environment, adsorbate coverage, and faradaic processes. Electrochemical scanning tunneling microscopy (EC-STM) is one of the few techniques capable of visualizing such interfaces directly in real space under electrochemical potential control, providing access to local atomic- and nanoscale phenomena that are hidden in spatially averaged measurements.

This talk will provide a broad and practically oriented introduction to EC-STM for the study of electrochemical interfaces under bias. The first part will focus on experimental aspects that are often decisive for successful measurements, including STM tip preparation and insulation, minimization of leakage currents, electrolyte cleanliness, potential control, tunnelling conditions, drift, image stability, and reproducibility. Particular attention will be given to practical strategies for obtaining reliable EC-STM data for stable in situ imaging and operando measurements.

The second part will combine selected scientific examples with approaches for qualitative and quantitative analysis of EC-STM data. Representative cases may include specifically adsorbed ions on metal single-crystal electrodes, potential-induced surface restructuring, two-dimensional materials supported on metals, molecular networks, nanoparticles, and catalytic interfaces under reaction-relevant conditions. These examples will also illustrate how Fourier-space analysis, lattice calibration, periodicity tracking, and image comparison can be used to extract quantitative information from complex datasets. In this context, I will introduce software tools developed in our group, including lattice Fourier analysis approaches, as well as the emerging role of machine learning and deep learning in feature recognition, segmentation, and automated extraction of structural descriptors.

The overall aim is to show how EC-STM, supported by quantitative analysis and modern computational tools, can bridge surface science, electrochemistry, and material design by enabling electrochemical interfaces to be studied directly, locally, and under bias.

Keywords: EC-STM; in-situ; operando; solid/liquid interface.

It's Not A Bug, It's A Feature: Defects in (Moiré) 2D Polymer Bilayers

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A 2D moiré superlattice forms when two or more atomically thin layers, such as graphene or transition metal dichalcogenides (TMDs), are stacked with a slight twist or lattice mismatch. This results in a moiré pattern with a periodicity much larger than the original lattice constants, giving rise to novel quantum properties. Moiré superlattices have revealed a range of exotic quantum phenomena, including flat electronic bands, the anomalous quantum Hall effect, unconventional superconductivity, and ferromagnetism. These discoveries underscore their transformative potential for future quantum technologies, as they provide a tunable platform bridging superconducting and insulating states.

Despite significant scientific progress, a major challenge remains: the lack of a general and efficient method for producing high-quality 2D moiré materials. In my talk, I will explore the potential of synthetic 2D polymer bilayers, created via on-surface synthesis, as alternatives to graphene- or TMD-based moiré superlattices. I will first discuss how on-surface synthesis, combined with scanning tunneling microscopy (STM), enables the characterization of the structural and dynamic properties of synthetic 2D polymers. Finally, I will present our recent findings on the formation of moiré superlattices in synthetic 2D polymers, especially the formation of defects within such moiré bilayers.

Keywords: 2D polymers; on-surface synthesis; moire superlattices; STM

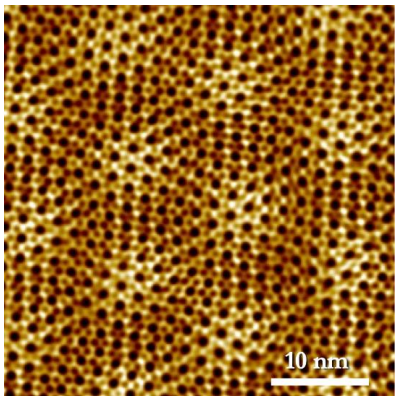

Figure 1. A moiré superlattice formed by a synthetic 2D-polymer bilayer

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New Insight into the Lifting of the Au(111) Reconstruction in Perchloric Acid: an Electrochemical Scanning Tunneling Microscopy Investigation

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The dynamic stability of metal surfaces under electrochemical conditions remains a central challenge in understanding electrocatalysis and interface-driven nanostructuring. In this work, we present an in situ electrochemical scanning tunnelling microscopy (EC-STM) investigation of the Au(111) surface during cyclic voltammetry in 0.1 M perchloric acid, revealing a restructuring pathway that goes far beyond the conventional picture of the reversible lifting of the herringbone structure (HBS). We show that gold islands nucleate within an exceptionally narrow anodic potential window, within which the HBS becomes unstable. Moreover, the island formation is not random but strongly aligned along the original reconstruction ridges, demonstrating that the anisotropic surface stress landscape templates their nucleation. Analyses confirm that the islands are mono-atomically high and form via lateral incorporation of mobile adatoms. A quantitative atomic balance provides a key mechanistic insight: the number of excess atoms that are released by lifting of the HBS are insufficient to account for the observed island size. They are thought to form local nuclei that subsequently capture at least 75% additional material, establishing step edges as active atomic reservoirs and demonstrating that the restructuring process is intrinsically non-local. Thus, electrochemical cycling induces long-range, stress-driven mass transport coupling terrace reconstruction and step-edge dynamics. Upon reversing the potential, island dissolution is slow, diffusion-limited, and strongly anisotropic. The herringbone reconstruction reappears only in elongated domains aligned along the former ridge direction, while residual islands act as pinning centers, introducing pronounced structural hysteresis. The state of the surface at a given potential therefore depends on electrochemical history, not solely on thermodynamic equilibrium. These findings redefine the response of Au(111) to electrochemical perturbation as a directionally biased, stress-mediated redistribution of matter rather than simple reversible reconstruction lifting. The results have broad implications for the long-term morphological stability of gold electrodes under repeated polarization and provide a model framework for understanding electrochemically driven evolution of noble metal surfaces.

Keywords: EC-STM; Au (111); cyclic voltammetry; herringbone.

Watching Catalysis Unfold: *Operando* Insights into Oxygen Reduction Reaction Driven by a 2D Molecular Network

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Electrocatalysis is emerging as a promising strategy for decentralized, sustainable chemical energy conversion and the synthesis of essential chemicals such as hydrogen peroxide [1]. Central to these processes is the oxygen reduction reaction (ORR), whose sluggish kinetics limit overall efficiency [2]. Although platinum-group metal (PGM)-based catalysts deliver state-of-the-art performance, their scarcity and cost hinder large-scale implementation, motivating the search for earth-abundant alternatives. To date, catalyst development has largely relied on empirical trial-and-error methodologies, often lacking the resolution to rationalize structure–performance relationships dictating catalytic outcomes.

To move beyond these empirical limits, we adopt a knowledge-based approach centred on cobalt tetrakis (N-methyl-4-pyridyl)porphyrin (CoTMPyP). As a representative single-atom catalyst (SAC), CoTMPyP features an unambiguously defined active site at the central cobalt atom. Here, we leverage its self-assembly on HOPG and Au(111) surfaces to form highly ordered 2D molecular networks. These model systems provide the structural definition needed to disentangle interfacial complexity under catalytic conditions. Electrochemical measurements reveal that CoTMPyP/Au(111) exhibits enhanced catalytic performance compared to the HOPG system. This improvement is attributed to more efficient electron transfer and higher surface coverage, arising from stronger electrostatic and π–π interactions with the gold substrate. Notably, CoTMPyP promotes the ORR at potentials more negative than the formal $Co^{III/II}$ redox couple, indicating that the catalytic cycle operates independently of the metal centre reduction and instead reflects the intrinsic electrochemical properties of the O_2-adduct [3]. To access the interfacial dynamics underlying these macroscopic responses, we employ *operando* EC-STM to visualize ORR in real time at the single-molecule level. This approach reveals distinct molecular species at the interface (***Fig.1***), corresponding to reaction intermediates. Time resolution of forward/backward offers a direct window into their evolution during ORR, enabling correlation with macroscopic performance.

Together, these findings highlight the capability of *operando* EC-STM to deliver mechanistic insight into the ORR, identifying key intermediates and providing a basis for assessing the rate-limiting step (RDS) under operating conditions.

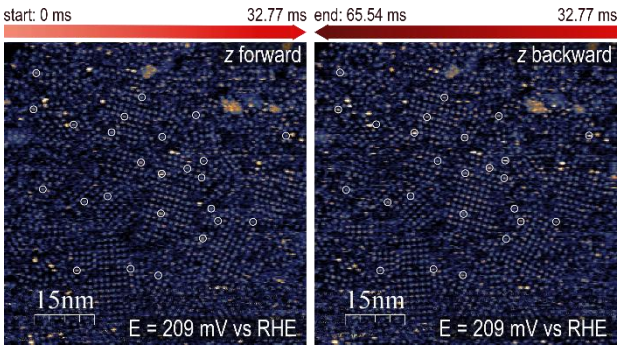

Fig.1 Forward/backward EC-STM images of CoTMPyP/Au(111) in 0.1 M $HClO_4$. Molecules undergoing transitions are marked (white circles); single-line scan time shown on top. Tunneling conditions: $It = 0.22\ nA$, $Ub = 57\ mV$.

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Keywords: oxygen reduction reaction (ORR); 2D organic network, metalloporphyrins, electrochemical scanning tunneling microscopy (EC-STM).

Uniaxial Electrocompression and Domain Wall Formation in Iodine Adlayers on Au(111): an EC-STM study

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Specific adsorption of halides at electrified metal–electrolyte interfaces enables controlled tuning of two-dimensional surface order under electrochemical conditions. However, atomically resolved relationships between electrode potential, adlayer structure, and strain-relief mechanisms remain incompletely understood. In this work, in situ electrochemical scanning tunnelling microscopy (EC-STM), together with substrate-anchored affine calibration and reciprocal-space analysis, is used to investigate the potential-dependent structure of iodine adlayers on Au(111), with a focus on the potential range in which the first stable ordered phase is observed and assigned to a centered-rectangular $c(p \times \sqrt{3})R30°$ structure exhibiting striped surface modulation.

With increasing electrode potential, the system undergoes continuous uniaxial electrocompression, reflected in a gradual reduction of the nearest-neighbour distance and a corresponding decrease in stripe periodicity, before transitioning towards a rotated-hexagonal phase at higher potentials. Fourier analysis of the STM data reveals characteristic satellite splitting consistent with the formation of a striped superheavy domain wall (SHDW) structure [1]. The observed relationship between stripe spacing and coverage is consistent with potential-driven soliton insertion as the dominant strain-relief mechanism.

Local real-space analysis further shows that the iodine atoms occupy multiple adsorption geometries, with significant variations in interatomic distances along the compression direction. With increasing electrochemical compression, the distribution of iodine–iodine distances broadens and the minimum spacing decreases, indicating enhanced structural heterogeneity within the adlayer. The results demonstrate that the iodine/Au(111) interface under electrochemical control is best described as a continuously evolving system with potential-dependent structural states driven by strain accommodation in strongly relaxed SHDW lattice.

Keywords: EC-STM; iodine adsorption; electrocompression; domain walls

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Tailoring the Interfacial Microenvironment: Impact of Hydrophilic/Hydrophobic Interface on CO_2 Reduction Selectivity

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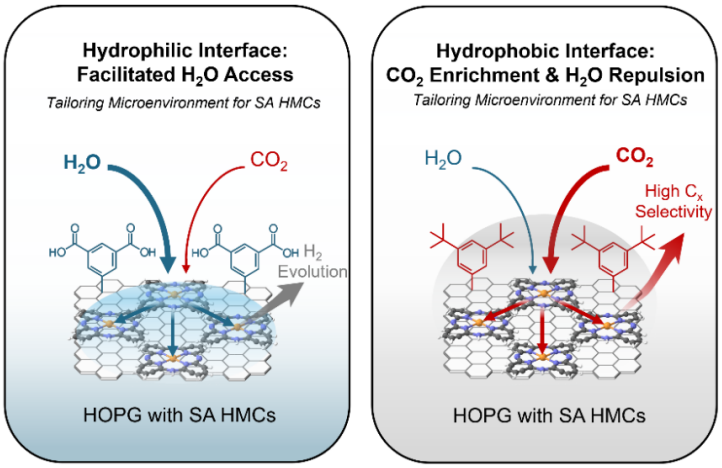
Contemporary catalysis research seeks a rational correlation between atomic-scale structures and catalytic performance. For heterogeneous molecular catalysts (HMCs), the interfacial microenvironment—specifically the cationic effect within the electrochemical double layer—plays a pivotal role in governing activity and selectivity by stabilizing key intermediates through electrostatic interactions. However, the effectiveness of these cations is heavily modulated by the local interface's chemical nature.



In this work, we regulate the local environment of HMCs by engineering vertical hydrophilic and hydrophobic interfaces on Highly Oriented Pyrolytic Graphite (HOPG) via molecular grafting, followed by the construction of self-assembled HMC networks. The morphology is characterized using STM, AFM, and Raman spectroscopy. Our results demonstrate that the hydrophilic interface facilitates H2O access, promoting proton-coupled electron transfer and favoring the competing H2 evolution reaction. Conversely, the hydrophobic interface creates a local microenvironment characterized by CO2 enrichment and H2O repulsion. This spatial control not only suppresses HER but also optimizes the local cation distribution to stabilize CO2 reduction intermediates, leading to high selectivity for carbon-based products (Cx). This study offers fundamental insights into tailoring interfacial wettability and ion distribution to dictate CO2RR selectivity.

Keywords: CO2RR Catalysis; Grafting; Self-assembly

Beyond Catalyst Immobilization : Engineering Local Proton Donor Microenvironments for Molecular Electrocatalysis

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Conversion of CO_2 into value-added products is crucial for sustainable energy storage, but requires catalysts with high activity, selectivity, and stability. Heterogeneous molecular catalysts (HMCs) address this by combining the robustness of heterogeneous systems with the tunability of molecular catalysts through immobilization on conductive supports.

We present a design concept in this work that extends beyond conventional catalyst immobilization. 2,3,7,8,12,13,17,18-octaethyl-21H,23H-porphine iron (III) chloride (Fe(OEP)Cl) is self-assembled on highly oriented pyrolytic graphite (HOPG) that has been functionalized by covalently grafted benzene carboxylic acid groups (Fig. 1). The grafted carboxylic acid groups are expected to act as local proton donors, mimicking enzymatic active sites and enabling control over proton availability near the catalyst.

Local proton donors placed close to the catalytic centre provide key insight into how local proton availability influences the catalysis and guides the design of more efficient heterogeneous molecular catalysts. Thus, this work lays framework for investigating the fundamentals of CO_2 electroreduction while precisely controlling the active sites and their surrounding chemical environment.

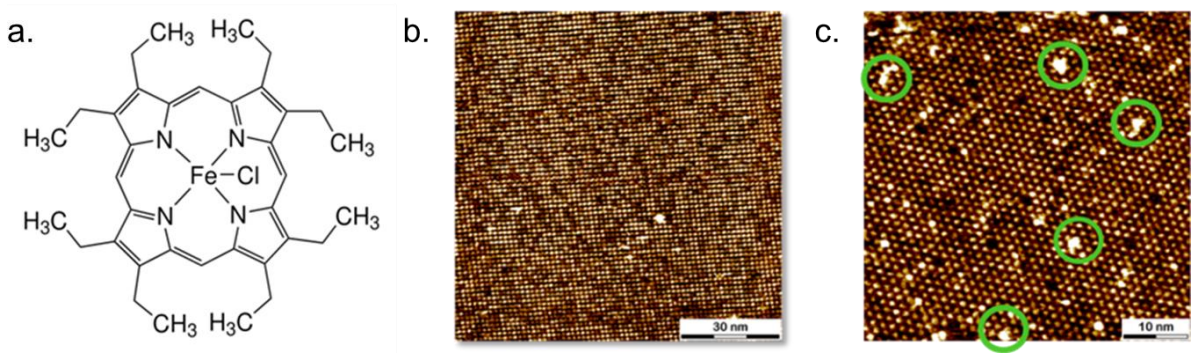

Fig. 1: (a) Chemical structure of Fe(OEP)Cl. Scanning tunneling microscopy (STM) images showing the self-assembly of Fe(OEP)Cl on (b) pristine HOPG and (c) HOPG functionalized with benzene-1,2,3-tricarboxylic acid. Green circles highlight the grafted molecules.

Keywords: Heterogeneous molecular catalysis (HMCs); CO_2RR; HER; STM

Visualizing Solvent Structuring at Electrified Solid-Liquid Interfaces

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Electrochemical and electrocatalytic processes are governed by the intricate interplay at the electrodeelectrolyte interface. Gaining deeper insights into these complex solid–liquid boundaries is key to advancing electrochemistry, making them a compelling subject for in situ surface-sensitive techniques such as scanning probe microscopy. While electrochemical scanning probe methods effectively track structural transformations of electrode surfaces–even under reaction conditions–they typically lack sensitivity to solvent organization on the liquid side. In contrast, high-resolution atomic force microscopy (AFM) has demonstrated the ability to resolve the vertical arrangement of solvent molecules relative to the surface, as exemplified in Ref. [2]. However, only a few studies have explored this capability under potential control within an operational electrochemical environment. In this talk, I will discuss how the simultaneous operation of electrochemical STM and AFM with qPlus sensors [2-4] provides new opportunities to probe the molecular-scale structure of electrified interfaces. I will present recent findings from our studies on well-defined single-crystal electrodes, particularly Au(111) in aqueous electrolytes, where we observe distinct, potential-dependent oscillations in the frequency shift signal along the surface-normal direction. These oscillations, which vary with the applied potential, electrode charge, and ion type, provide valuable insights into water and ion structuring at electrochemical interfaces. By correlating these observations with atomistic molecular dynamics simulations, we enhance our understanding of the interfacial organization in electrochemical systems.

Keywords: EC-AFM/STM; interfacial solvent structure; Au(111).

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Electrochemical probing of nanoscale bio-systems by AFM-SECM technique

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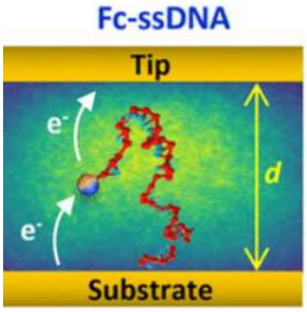
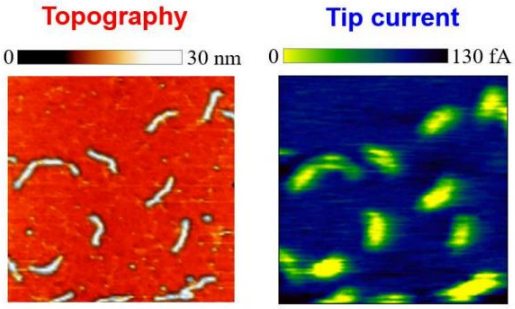
We present a cutting-edge electrochemical imaging approach delivering true nanoscale resolution for probing electron transfer (ET) at bio-interfaces. Our unique local probe strategy merges scanning electrochemical microscopy (SECM) with atomic force microscopy (AFM), featuring an original design where the redox "mediator" is flexibly tethered directly to the nanoscale object under investigation. Using a custom lever-shaped gold nanoelectrode tip, we simultaneously capture high-resolution topography (AFM tapping mode) and correlated electrochemical tip-current maps.[1]



Mt/AFM-SECM

At the core of this method is a "mediator-tethered" AFM-SECM platform based on DNA or polyethylene glycol (PEG) chains attached to the surface of an electrode, and carrying a ferrocene (Fc) redox head at their free end. Once confined and driven by the elastic motion of the chains, Fc units undergo rapid redox cycling, enabling highly efficient electron shuttling within the "nano-gap" between the tip and the substrate. This amplifies the tip current signal sufficiently that only a few tens of Fc-chains are required. Coupled with advanced molecular dynamics simulations (QBIOL, LIMMS partner, University of Tokyo), this approach provides new insights into fundamental ET mechanisms in Fc-DNA systems, a key system that pioneered the groundwork for DNA-based electrochemical sensors.[2-3]



We further extend this concept to single-protein electrochemical imaging via redox immunolabeling, using FcPEG-functionalized antibodies (IgG).4 This strategy enabled the creation of fully integrated enzymatic nanosystems, consisting of a glucose-oxidizing enzyme and its elastically attached Fc mediator onto virus capsids. With AFM-SECM, we resolve and quantify the enhanced enzymatic activity of the resulting electrocatalytic virions at the single entity level.5-6 revealing how the catalytic current depends on viral scaffold structure and topology. Notably, we achieved a state-of-the-art electrochemical measurement of enzymatic activity in clusters containing fewer than ten enzyme molecules.[7]

Current efforts are focusing on the development of integrated enzymatic systems, spatially organized with true nanoscale precision, using DNA origami as a scaffold. Finally, we address emerging challenges in AFM-SECM bio-imaging, particularly the functional investigation of "soft", biologically relevant objects such as redox-active liposomes and mitochondria.

Keywords: Electrochemical Imaging, AFM-SECM Hybrid Microscopy,Bio-Nano-Electrochemistry

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Electron transfer kinetics of HOPG and GIC investigated by cyclic voltammetry and scanning electrochemical microscopy

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Highly oriented pyrolytic graphite (HOPG) is one of the most commonly used host materials for obtaining and studying graphite intercalated compounds, due to the high structural order of this polycrystal. Experiments on electrochemically intercalated HOPG in sulfuric acid have a model character, as the results obtained can be effectively generalized, not only to other graphite compounds but also to the intercalation of other layered host lattices. Additionally, the HOPG/H_2SO_4 system has an appealing potential for the electrochemical production of graphite oxide, ideally through reversible oxidation/reduction cycles, which is of interest for energy storage and large-scale graphene production. However, the oxidation/reduction cycles in this electrochemical intercalation process are neither reversible nor topotactic, leading to significant alterations in the HOPG structure. This alteration may impact, for instance, the quality of the electrochemically produced graphene. In particular, the effect of electrochemical intercalation on the conductivity of the basal planes of HOPG, and consequently on graphene sheets, is still a topic of debate. In this study, we investigated both the macroscopic and microscopic electron transfer (ET) kinetics of the HOPG surface, before and after intercalation in 1 M H_2SO_4, using cyclic voltammetry (CV) and scanning electrochemical microscopy (SECM), respectively. The heterogeneous kinetic constant (k_0) of the HOPG was quantitatively evaluated using the redox systems $[Fe(CN)_6]^{3-/4-}$ and $[Ru(NH_3)_6]^{3+/2+}$. The morphology of the samples was also examined using atomic force microscopy (AFM), which revealed widespread formation of blisters and precipitates during the HOPG intercalation process. The CV and SECM results indicate that, upon intercalation, the electrochemical behavior of the HOPG changes significantly, and the ET decreases notably. However, this effect depends on the redox mediators used and is more pronounced for the $[Fe(CN)_6]^{3-/4-}$ system, for which a decrease in k_0 by several orders of magnitude was observed. The reduction in ET can be correlated with the blisters and precipitates that occur during the HOPG intercalation, as observed by AFM [1].

Keywords: electrochemical intercalation; HOPG; AFM; SECM

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Towards a Process-Oriented Quality Control Protocol for Pt-Octaethylporphyrin Thin Films via Combined AFM–Raman Analysis

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Inkjet printing has emerged as a versatile and digitally controlled deposition technique for the additive and maskless fabrication of functional thin films, offering high spatial precision and scalability. However, achieving reproducible film quality remains challenging because the final morphology strongly depends on the interplay between substrate properties and solvent-mediated growth dynamics occurring during film formation.

In this work, a systematic process-oriented protocol is proposed to assess and optimize inkjet-printed films through comparison with established deposition techniques, namely Organic Molecular Beam Epitaxy (OMBE), molecular spray, spin coating, and drop casting, which provide different growth conditions and film formation dynamics.

Platinum(II) octaethylporphyrin (PtOEP) thin films were deposited on highly oriented pyrolytic graphite (HOPG), standard quartz, and ultraflat quartz substrates, selected for their technological relevance and distinct surface characteristics. Film quality was investigated through a combined Atomic Force Microscopy (AFM) and Raman spectroscopy approach, enabling the correlation of morphological characteristics with molecular organization and vibrational response.

The comparative analysis identifies substrate morphology and solvent evaporation dynamics as the dominant factors governing film formation. In particular, substrate morphology was observed to play a dominant role in influencing final film architecture even beyond the initial growth stages, while solvent-mediated processes determine aggregation behavior and film continuity.

Overall, the proposed AFM–Raman methodology provides a reproducible framework for identifying similarities and deviations among deposition routes and for guiding the optimization of inkjet printing processes. Although the present work relies on *ex situ* characterization of deposited films, the identified correlations provide indirect insight into the interfacial processes likely involved in film evolution and establish a basis for future developments toward integrated and time-resolved approaches aimed at directly probing film formation at the solid–liquid interface. Such developments could further extend the present correlative framework toward *in situ* investigations and potentially include higher-spatial-resolution techniques, such as tip-enhanced spectroscopies, for probing local molecular organization and interfacial dynamics.

Keywords: Inkjet printing protocol, PtOEP films, Atomic Force Microscopy (AFM), Raman spectroscopy

Characterizing photoinduced reactions in the solid/liquid interface: control and mechanistic analysis with optical nanometrology

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Decoration of surfaces with chemically precise layers and high spatial resolution is a desirable goal towards several applications, including nanosensors and nanoactuators. However, reliable and scalable strategies for preparing spatially resolved surface patterns with controlled thickness are realtively scarce. In this communication, I will show how optical nanometrology can be an interesting alternative for characterizing reactivity in the solid-liquid interface in operando conditions.

I will describe how optical nanometrology approaches enable careful control over a wide range of photoinduced phenomena. Starting from quantification of photoinduced grafting reactions, examples of photoinduced polymerization and plasmonically induced redox phenomena will also be presented. By combining controled excitation of chemical reactions with in situ quantification, I aim to show that photochemical strategies can be used add a variety of chemical fucntions to interfaces at lengthscales that span several orders of magnitude, leading to chemically complex patterned surfaces.

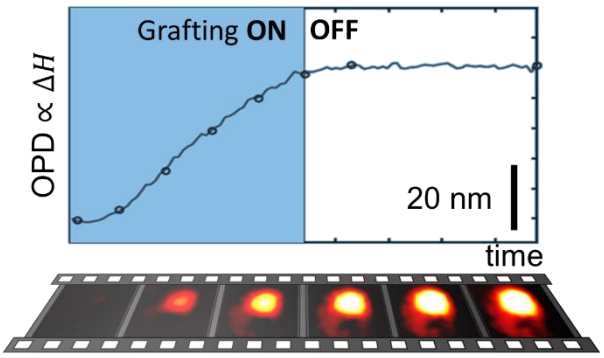

Figure 1. Operando monitoring of photoinduced polymerization on glass-water interfaces through quantitative phase imaging.

Keywords: Surface photochemistry, operando monitoring, optical nanometrology, photopolymerization.

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