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A practical introduction to electrochemical atomic force microscopy and scanning tunneling microscopy

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Keywords: electrochemical atomic force microscopy, electrochemical scanning tunneling microscopy, in-operando scanning probe microscopy

Abstract

Directly monitoring the structure and evolution of an electrode surface during reaction provides uniquely powerful insight into interfacial processes. Electrochemical atomic force microscopy (EC-AFM) and Scanning Tunneling Microscopy (EC-STM) have therefore become essential tools for linking potential-controlled surface transformations to macroscopic current and product outcomes. In EC-AFM, surface features are measured from the deflection of a typically laser-tracked cantilever, enabling imaging of metals, semiconductors, and insulators in liquid electrolytes. By selecting appropriate probes and operating modes, EC-AFM maps topography together with complementary channels such as mechanical response, friction, and local electrical contrast, offering a multi-parametric view of the electrified interface during processes such as film formation, corrosion, gas evolution, and electrodeposition. By contrast, EC-STM measures the tunneling current between a conductive tip and a conductive substrate, providing access to atomic-scale surface order, reconstruction, and adsorbate structures under EC control. In liquid environments, STM performance is limited by parasitic faradaic currents and capacitive coupling, making careful EC configuration and robust tip insulation (coated everywhere except the apex) critical for stable imaging. This tutorial summarizes practical EC cell considerations, probe preparation, alignment and calibration steps, and data-handling best practices for acquiring reproducible in-operando SPM datasets. This tutorial seeks to make EC-AFM/EC-STM measurements more approachable for beginners by clearly explaining the fundamental principles as well as practical considerations, allowing practitioners to gain highly informative EC insights on their systems of interest. Throughout, we emphasize common EC artifacts and decision rules that help newcomers distinguish true potential-dependent surface dynamics from drift, bubble interference, leakage currents, and feedback-induced contrast, enabling reliable interpretation of AFM/STM observations in EC environments.

Glossary

HOPG: Highly oriented pyrolytic Graphite—This easy to prepare carbon material exhibits large atomically flat terraces and monatomic steps and thus represents a standard material for various test procedures.

In-operando: The imaging by AFM/STM is conducted simultaneously with electrochemical measurement, mimicking real-world electrochemical systems. This provides a direct correlation between electrochemical current/potential data and microscopic surface changes.

Photodiode detector: The reflected laser from the back of the cantilever is transferred to the photodiode detector. This is typically a four-quadrant detector, used to monitor cantilever deflection for topographic maps and cantilever twisting for lateral force maps.

Setpoint: The user-defined reference value used by the feedback loop to control the distance between the probe and the sample. The setpoint parameter corresponds to deflection force in contact mode AFM, amplitude in intermittent contact AFM, and tunneling current in STM.

Spring constant k : A measure of the cantilever's resistance to deformation. According to Hooke's Law $F = -kx$, it defines the relationship between the force (F) applied to the cantilever and the resulting vertical deflection (x). The unit is N m^{-1} .

Force-distance curve: A measure of the cantilever's deflection as the tip and sample are brought together and then separated. Due to the interaction between the tip and surface, the tip deflects either towards the substrate (attractive) or away from it (repulsive). Allows the determination of the adhesion force. Important: For an exact value of the separation between tip and sample, a force-distance curve must be converted to a force-separation curve.

(Deflection) sensitivity: A calibration factor used to convert the raw electrical signal from the photodiode (i.e. cantilever deflection) into a physical distance. It is given by the slope of a force-distance curve observed on a stiff surface (e.g. sapphire). The unit is nm V^{-1} .

Resonance frequency: The natural frequency at which the cantilever oscillates freely with maximum amplitude when excited by an external force (such as a shaker piezo) or thermal noise. The cantilever's stiffness and mass determine this value. This frequency decreases significantly when the cantilever is immersed in liquid due to hydrodynamic damping. The unit is kHz.

Potentiostat: An electronic instrument that controls the electrical potential at a working electrode referred to a reference electrode by injecting current through a counter electrode.

Thermal drift: One of common errors during AFM/STM measurements. The temperature change causes the inhomogeneous response from the components such as cantilever and scanner, resulting in shifted position of features in consecutive images.

Tunneling current (I_t): The current flowing by quantum tunneling between the conductive tip and sample across a nanometer-scale gap; it depends exponentially on tip-sample distance.

Bias voltage (V_{bias}): The potential difference between the STM tip and sample that drives tunneling; together with the current setpoint, it determines tip-sample separation and image contrast.

Scanning Tunneling Spectroscopy (STS): Measurement of I - V (sometimes dI/dV) curves at a fixed position to probe local electronic structure.

Local Density of States (LDOS): The local availability of electronic states near the Fermi level; STM/STS contrast can reflect both topography and LDOS.

1. Introduction

Scanning probe microscopy (SPM) techniques, particularly atomic force microscopy (AFM) and scanning tunneling microscopy (STM), resolve electrochemical (EC) interfaces down to the nano- and even atomic-scale under conditions that include ultra-high vacuum, ambient environments, and in-operando liquid electrolytes. Within this manuscript we strictly focus on aspects linked to ECSPM, providing a guide to start working with such techniques. Unlike optical or electron microscopy, which forms projection images from photon or electron interactions, AFM and STM reconstruct surface maps from local probe-substrate interactions measured as a sharp tip scan across the sample. In AFM, as shown in figure 1(a), a compliant cantilever bearing a nanometer-scale tip senses tip-sample forces. The resulting

deflection, typically tracked optically with a laser beam, is converted into topographic maps and complementary contrast channels such as phase or amplitude, enabling imaging of both conductive and insulating materials in electrolytes [1]. STM, by contrast, measures the tunneling current between a conductive tip and the substrate surface shown in figure 1(b). This current decays exponentially with distance and depends on electronic states available near the Fermi level. In constant-current STM, a feedback loop adjusts the tip position to maintain a constant tunneling current; consequently, the resulting map represents a convolution of geometric height and the local density of states, sampled over the applied bias window and effective tunneling barrier [2]. When paired with EC potential control, AFM and STM enable correlation of interfacial structure with macroscopic EC behavior at high spatial resolution [3–5]. An example of this is illustrated in figure 1(c), depicting the morphological evolution during Ag underpotential deposition (UPD) on iodine-modified Au(111) in 0.1 mM $\text{AgClO}_4/0.05 \text{ M HClO}_4$, recorded via in-operando EC-AFM. The cyclic voltammogram (CV) exhibits two distinct cathodic peaks at approximately 0.3 V and 0.1 V (vs Ag/Ag^+), corresponding to Ag monolayer formation. Shifting to more negative potentials (around -0.15 V) leads to Ag bilayer formation. As the potential is swept from 0.5 V to 0.13 V (highlighted in red), islands begin to emerge upon passing the monolayer deposition peak. Approaching -0.15 V (highlighted in blue), the island density increases significantly as bilayer formation occurs. Holding the potential at -0.15 V (highlighted in green) reveals that these newly formed islands progressively grow over time. Besides, in contrast to spectroscopic methods, direct visualization of processes at interfaces in EC energy storage and conversion helps to elucidate fundamental mechanisms in such devices. Thus, *in situ* and in-operando AFM has been used to monitor solid electrolyte interphase (SEI) nucleation and growth on battery anodes, linking film buildup, roughness, and mechanical evolution to electrolyte additives and formation conditions [6, 7]. In electrocatalysis, AFM has revealed potential-dependent restructuring of copper CO_2 reduction catalysts into nanoscale morphologies and correlated these changes with shifts in product selectivity [8]. Similarly, EC-STM has visualized potential-induced surface reconstructions and adsorbate ordering on Au(111) and Pt-group single crystals, revealing atomic-scale motifs and ordered surface phases relevant to catalytic function [9, 10]. For lithium metal solid-polymer electrolyte interfaces and silicon anodes, *in situ* AFM-based infrared near-field nanospectroscopy (nano-FTIR/s-SNOM) has resolved nanoscale chemical and structural heterogeneities during cycling, enabling links between interphase inhomogeneity and non-uniform deposition behavior [11–13].

AFM and STM are distinctive among *in situ* probes because they link local morphological or electronic changes to EC function. AFM can quantify surface roughness, nucleation density, interphase growth, and gas bubble coverage across metals, semiconductors, and ceramics in relevant electrolytes, while STM provides atomic-scale lattice resolution on conductive surfaces and reveals potential-driven surface phases and bias-dependent electronic contrast [14]. Practical constraints require careful attention. In liquid electrolytes, EC-AFM cantilevers experience viscous damping that lowers quality factors and complicates dynamic-mode amplitude and phase contrast [14]. EC-STM requires conductive substrates, precise potential control, and insulated tips to suppress faradaic leakage currents. Both techniques are susceptible to artifacts from thermal drift, bubble interference, and local pH gradients [15, 16]. In some scenarios tip-sample interactions can induce local phenomena on the surface, masking true processes or generating false data. With rigorous controls and transparent reporting, however, in-operando SPM moves beyond topography to provide mechanistic insight into charge transfer, mass transfer, and interfacial stability in batteries, electrocatalysis, and corrosion.

2. Design

Utilizing SPM techniques effectively requires a comprehensive understanding of their inherent limitations. To guide the selection of appropriate techniques and methodologies, a decision-making framework is outlined in the flowchart in figure 2. For instance, while STM strictly necessitates conductive samples, AFM offers the versatility to characterize non-conductive or semi-conductive materials under ambient conditions. Conversely, under EC conditions, the sample functions as the working electrode (WE); consequently, the substrate must be electrically conductive.

Successful EC-SPM measurements begin with a properly designed electrochemical cell (EC-cell). While manufacturers offer ready-to-use setups, many practical formats exist, and most researchers eventually adapt or fabricate custom platforms. Beginners are advised to start with commercially available cells before transitioning to custom designs as specific experimental needs arise.

Chemically inert materials—such as PEEK, PTFE, Kel-F™ (PCTFE), and chemically resistant glass—are strongly recommended as EC-cell construction to prevent unwanted reactions, including corrosion,

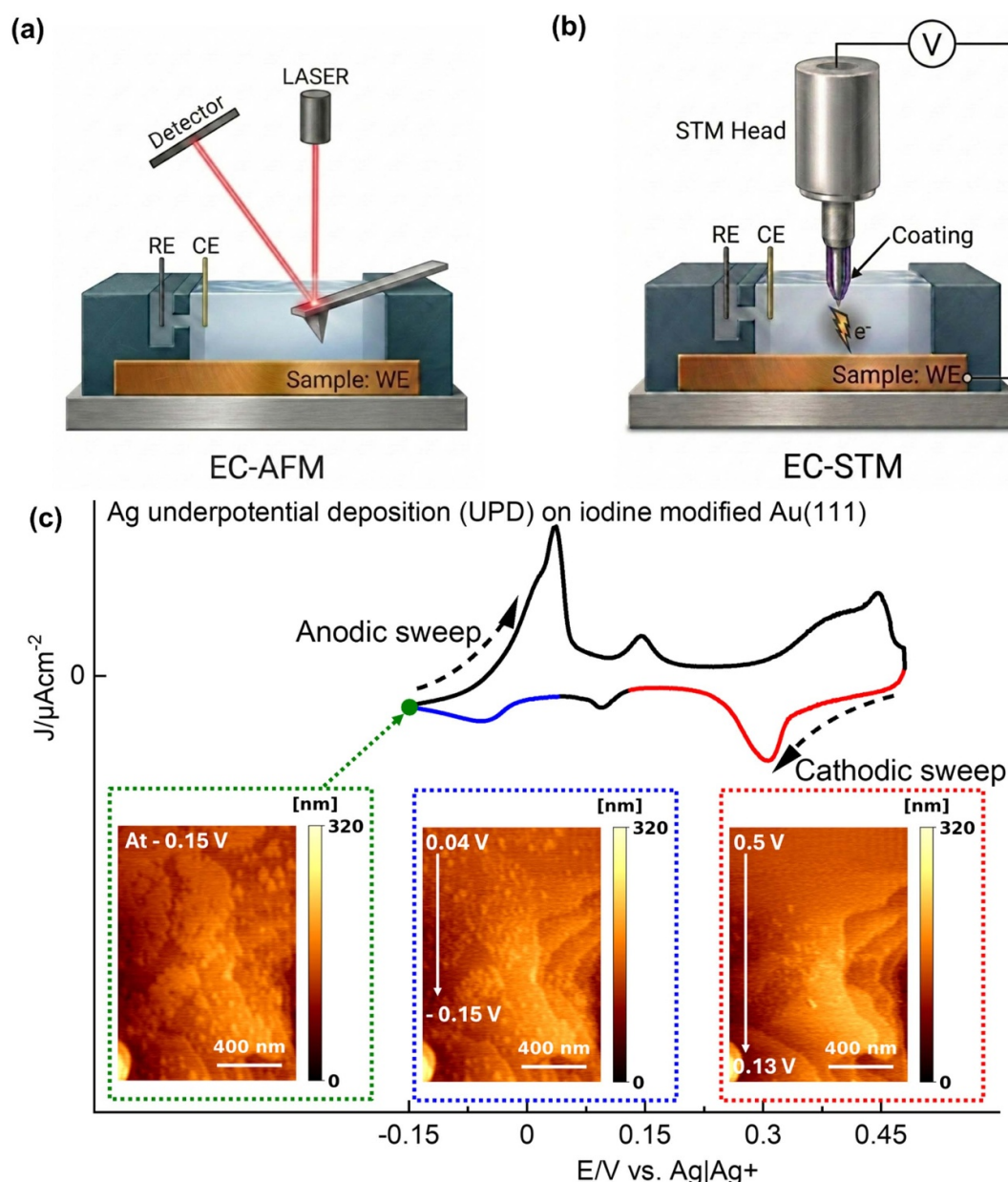
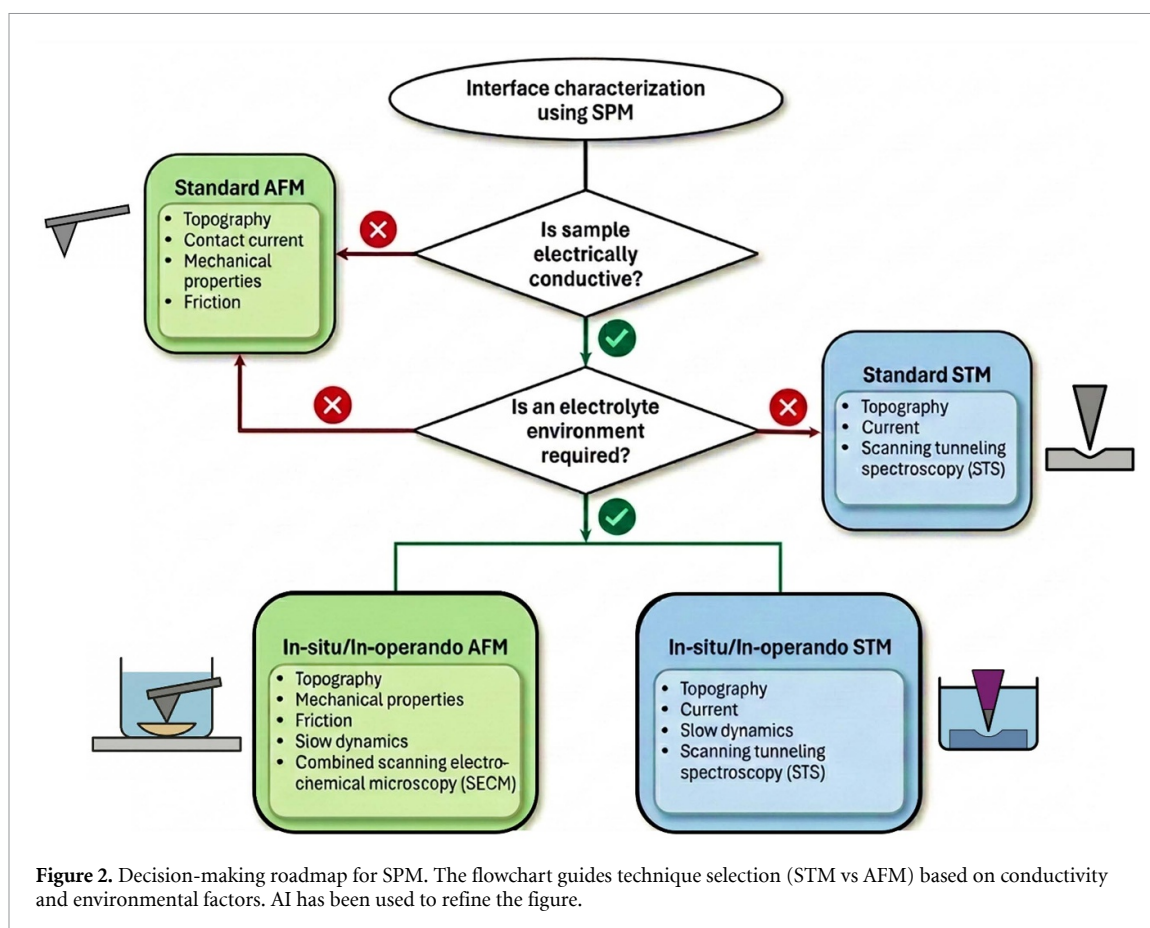


Figure 1. Overview of in-operando EC-SPM methodologies. (a) Schematic of an EC-AFM system illustrating optical lever detection within a three-electrode cell. (b) Schematic of an EC-STM system highlighting the insulated tip, piezo scanner, and bipotentiostat configuration. (c) Cyclic voltammogram (CV) profile for Ag UPD in 0.1 mM $\text{AgClO}_4/0.05$ M HClO_4 on iodine modified Au(111). Gold and Ag wires were used as counter electrode and reference electrode, respectively. Insets show AFM height maps recorded at different potentials during cathodic sweep, with the border colors of each inset corresponding to the marked potentials on the CV curve. AI has been used to refine (a) and (b).

trace-metal leaching, and swelling [17]. Furthermore, because the cantilever holder is submerged in the electrolyte, the cell dimensions must be large enough to accommodate it. Careful consideration of cell depth is essential: an excessive electrolyte volume can contact the metal components of the scanner, creating an unintended conductive pathway that leads to electrical short-circuits and potential hardware damage. When maintaining a constant reactant concentration is required, flow cells featuring integrated inlet and outlet channels can be utilized [17].

In AFM, actuation and imaging mode strongly influence stability in liquid. Conventional acoustic (piezo) drive for dynamic modes can excite spurious resonances from the holder and fluid cell, complicating amplitude and phase interpretation and increasing sensitivity to small changes in liquid level or boundary conditions. Photothermal and magnetic excitation often yield cleaner frequency responses and more stable feedback in liquids, improving force control and reducing artifacts during extended in-operando measurements [18, 19]. In EC environments where gas evolution, viscoelastic interphases, or strong hydrodynamic damping degrade dynamic-mode contrast, contact-mode can be advantageous



in some highly damp conditions by providing robust tracking of height and frictional responses under potentiostatic control. Typical tips are made of doped silicon and are a good choice to start with. Since the AFM tip only acts as observer and is not part of the EC setup, short circuiting is not a severe issue in comparison to STM, where a good insulation of the tip is necessary. However, depending on individual conditions, e.g. when using metallic lithium that interacts with silicon, other cantilever materials such as diamond are commercially available.

Electrode choice defines chemical relevance. Au(111) and Pt(111) single crystals provide easy to prepare (by flame annealing), atomically flat, structurally well-defined benchmarks for identifying adsorbate ordering, step dynamics, and potential-driven reconstruction without ambiguity from roughness or heterogeneous grain orientation [9, 10].

In certain conditions, AFM is preferred over STM because non-conductive regions on the sample surface, such as the SEI, disrupt the tunneling current. For lithium-metal and anode-free battery studies, polished and PVD copper substrates are widely used as controlled deposition hosts for AFM measurements of nucleation and early growth. Battery-grade Li foil has also been interrogated directly to capture mechanically evolving interphases under realistic surface chemistries, including *in situ* AFM quantification of SEI mechanical evolution on metallic lithium as a function of EC history [4, 5, 7]. Such sensitive measurements often require a glovebox or environmental chamber to prevent parasitic reactions. Graphene-based platforms further expand electrode design space by combining chemical inertness and electronic conductivity with unique interfacial access: graphene can act as an ultrathin WE that seals a liquid reservoir while enabling scanning-probe interrogation of a buried electrified interface, reducing meniscus- and evaporation-related artifacts [13, 19]. Oxides and solid-state electrolytes can also be probed by AFM, though their insulating nature imposes additional constraints on charge injection and electrostatic crosstalk that must be managed through careful biasing and grounding strategies [3, 4].

Comprehensive reporting is indispensable; parameters including cell dimensions, electrode placement, electrolyte identity, flow conditions, reference electrode configuration, imaging mode and recorded data channels, as well as tip preparation should be documented to ensure reproducibility and meaningful comparisons across studies.

3. Experiment

Acquiring high quality data requires careful calibration of the system prior to conducting EC measurements. In AFM, the laser reflected from the back of the cantilever maps the topography of the sample surface. Therefore, accurately positioning the laser on the cantilever—specifically near the tip—and centering the reflected beam on the photodiode maximizes the initial signal intensity [1, 14]. This initial intensity varies slightly depending on the cantilever type and the presence of a reflective coating. Additionally, when the cantilever is submerged in an electrolyte, the photodiode position must be readjusted to compensate for the change in light refraction. In STM, high quality data depends primarily on the sharpness, cleanliness, and insulation quality of the tip. A constant bias voltage is applied between the tip and the sample, prompting the STM tip to approach the surface until it reached the position where the target tunneling current (the current setpoint) is established. However, if the tip is poorly insulated, the highly conductive electrolyte can generate a large EC background current. The feedback loop may mistake this for the tunneling current halting the tip before it actually reaches the surface. While users can attempt to outpace this leakage by increasing the tunneling current setpoint, this approach is limited by the maximum current threshold of the instrument (typically 10 nA). Furthermore, achieving high spatial resolution requires maintaining a low tunneling current.

Following the assembly of the EC-cell, a thorough leakage test must be performed. Tiny leaks can be exceptionally difficult to detect initially, often manifesting overtime and compromising the integrity of subsequent AFM experiments. A key indicator of a tiny leak is a progressive decrease in the laser's sum signal, caused by localized changes in light refraction as electrolyte escapes. Electrochemically, leaks typically induce anomalous CV behaviors, such as sudden current drops or potential instability at the working electrode (WE). Due to their lower surface tension, organic electrolytes are significantly more prone to these problematic tiny leaks than their aqueous counterparts.

With a properly designed EC-cell, a commercial Ag/AgCl reference electrode (RE) can be utilized in aqueous electrolytes [17]. Alternatively, pseudo-reference electrodes, such as Pt or Ag wires, can be employed in both aqueous and aprotic electrolytes. When using pseudo-REs, users must verify their stability and potential windows. The potential range can be readily estimated by comparing the open-circuit potential against a standard reference electrode.

When EC reactions on the WE lead to gas evolution, or when using a gas-saturated electrolyte, understanding gas solubility is crucial. Low solubility often leads to bubble formation on the SPM probes. These bubbles disrupt laser reflection during EC-AFM measurements and block tunneling currents during EC-STM experiments. To mitigate this issue, users can utilize flow cells to maintain the dissolved gas concentration below saturation point. However, the flow rate must be carefully optimized to avoid disrupting the EC reactions occurring at the electrode surface [17]. Another approach is to investigate the surface near the onset potential, where gas evolution is minimal [8].

During the experiment, a potentiostat controls the WE potential while the AFM or STM records local height or deflection changes; in conventional EC-AFM topography imaging, the tip is not part of the EC circuit and acts as an observer only. In EC-STM, a bipotentiostat controls the potential of two electrodes, i.e. WE and STM tip, simultaneously versus the reference electrode. Therefore, the actual potential of the STM-tip will change when the potential of the WE (sample) changes, ensuring a constant bias voltage between the tip and the WE (sample), with typically small values (e.g. tens to hundreds of mV). Tunneling currents between tip and WE (sample) are in the order of pico- to nanoampere, while EC currents between the WE (sample) and the counter electrode (CE) are significantly higher. This underscores why proper insulation of the STM tip is critical for reliable EC-STM measurements as no larger faradaic currents should occur between tip and counter electrode. However, the presence of such currents makes any EC leakage immediately noticeable. For example, when investigating monatomic depositions, such as UPD, lowering the bias voltage can enhance spatial resolution by positioning the STM tip in very close proximity to the surface. However, this configuration increases the risk of a tip crash if drastic topographic changes occur on the surface during the experiment.

4. Data

Even though it is not directly used for data interpretation, it is imperative to also archive the calibration data of AFM, including deflection sensitivity, cantilever spring constant and resonance parameters in the measurement environment. In both AFM and STM, setpoint or gain settings used during imaging should be archived as well so that observed values can be reported on an absolute and reproducible basis. Most of this data can be extracted from the header of individual images but should be

verified before storing the data. Metadata should comprehensively record electrode type and preparation, reference electrode placement, electrolyte identity, and flow conditions. Additionally, noting the minimum tunneling current setpoint during the tip approach procedure is highly recommended for STM measurements.

CVs are recorded simultaneously with SPM data, providing quantitative EC insights. In AFM, numerous data channels—such as topography, stiffness, adhesion, surface potential, amplitude, and phase—can be captured concurrently, though the available channels vary by AFM mode. For instance, amplitude and phase channels are unique to dynamic modes, while mechanical properties like adhesion and stiffness are accessible during force-distance mapping. Furthermore, the raw detector deflection and the commanded scanner motion (Z-piezo) signals should be recorded so that the changes in contrast can be separated from feedback artifacts, drift, and true interfacial evolution [1, 14]. In contrast, EC-STM typically captures two primary channels: topography and local spectroscopy (I - V curves).

Image post-processing should remain minimal yet rigorous, and all processing steps should be explicitly reported in the experimental section to preserve quantitative interpretability. Typically, for topography observed from AFM and STM, planar leveling or line-by-line flattening and low-order background subtraction are applied to correct scanner bow, sample tilt, and slow drift, while mild stripe removal can be appropriate when it targets known scan-direction artifacts rather than real features [1]. However, aggressive filtering, excessive smoothing, or contrast enhancement can distort observed features so such operations should be justified. Quantitative outputs of topography can include roughness evolution, feature and nucleation density, areal coverage of molecules or deposits, step or pit density, and interphase morphology, thickness proxies [3, 4]. Additionally, mechanical-property or surface potential contrast can be qualitatively or quantitatively acquired for AFM measurements.

5. Practical considerations

Using AFM, diverse information about the surface, including topography, conductivity, mechanical properties, and friction, can be obtained. For reliable measurements, the spring constant of the cantilever should be chosen based on the sample stiffness and the property of interest, because it sets the usable force range and the sensitivity to small changes in tip-sample interaction. For example, when measuring mechanical properties such as Young's modulus on soft materials like polymers, the cantilever should be compliant enough to resolve small indentations without driving large, uncontrolled deformation. For friction or lateral force measurements, it is generally best to begin at very low normal loads (often on the order of a few nN, depending on the tip radius and environment) to minimize wear and reduce tip-induced modification, then increase the load in a controlled manner only as needed to improve signal-to-noise and map trends. Crucially, there is no universal 'good' load threshold, since the effective contact pressure depends on tip geometry, surface roughness, electrolyte lubrication, and whether the surface film is viscoelastic or brittle, so loads should be reported and justified for the specific system.

In EC environments, the stability of the laser signal is a crucial factor. If the electrolyte is volatile, evaporation limits the measurement window and induces drift. Furthermore, if gas evolution occurs during EC reactions and the solubility of the gas is low, bubbles often adhere to the AFM cantilever, causing significant laser instability and signal.

In STM, although the probe does not physically contact the sample, tip contamination remains a significant risk that directly degrades resolution. HOPG is a standard benchmark sample due to its ease of preparation (just peel-off the top layer by tape) and large atomically flat terraces; however, it is susceptible to exfoliating onto the tip during scanning, causing artifacts [20]. Additionally, the EC-STM tip must be well insulated, leaving only the very apex exposed. The primary purpose of coating the STM tip is to suppress Faradaic leakage from the electrolyte when a DC bias is applied between the tip and the WE (sample). Ideally, the coating material should be an effective insulator with a low dielectric constant ($\epsilon_r \approx 2.5$). However, many coating materials are prone to developing microscopic cracks—often invisible to the naked eye—that allow electrolyte penetration and prevent a successful tip approach. Consequently, selecting coating material that forms a robust, impermeable physical barrier is critical for reliable EC-STM measurements. Several coating materials are commonly used, including Apiezon wax [15], nail polish [16], and molten polymers. Apiezon wax can become brittle upon solidification, potentially allowing faradaic leakage through cracks in the coating. Nail polish is accessible but may dissolve depending on the electrolyte solvent. Molten polymers (e.g. polyethylene) offer chemical resistance and flexibility, and tips can be recoated easily; however, some electrolytes may still permeate the coating over time. Therefore, a primary task for EC-STM operators is to identify the coating material best suited for their specific chemical environment. Crucially, the tip apex must remain uninsulated to allow tunneling. With

a high-quality, well-coated tip, measurements can be performed with very low leakage currents, leading to highly resolved images.

6. Summary

EC-AFM and EC-STM have become indispensable tools for characterizing the complex interfaces found in energy storage and conversion devices, such as batteries, fuel cells, and electrolyzers. By visualizing surface dynamics in real-time, these techniques have fundamentally advanced our understanding of critical processes like SEI formation, catalyst degradation, and surface reconstruction. While modern instruments offer accessible liquid cells for standard measurements, the full potential of these methods is realized through advanced operating modes that probe mechanical, electrical, and frictional properties simultaneously. A key advantage of in-operando SPM is its ability to bridge the gap between macroscopic electrochemistry and nanoscale structure. While potentiostats measure the global response of an entire electrode, AFM and STM reveal the local phenomena that drive those macroscopic dynamics. This connection allows researchers to distinguish between average performance and site-specific activity or failure mechanisms. Ultimately, by providing multi-parametric data from the nanometer to the micrometer scale, these techniques enable a comprehensive view of the electrified interface. When combined with correlative spectroscopy, EC-AFM and EC-STM provide the structural insight necessary to guide the rational design of next-generation energy materials.

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Data availability statement

The data that support the findings of this study will be openly available at the following URL/DOI: <https://doi.org/10.26165/JUELICH-DATA/EK6OJS> [21].

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Conceptualization (equal), Supervision (lead), Visualization (equal), Writing – original draft (equal), Writing – review & editing (equal)

Highly recommended/further reading

The interest in electrochemical AFM and STM experiments towards energy systems increased over the last years and the following articles are recommended to gain an overview about recent research, cell design and image analysis:

Basic introduction to AFM and typical image processing and artefacts:

Eaton P, West 2010 *Atomic Force Microscopy* (Oxford University Press)

Electrochemical AFM:

Wang W-W, Yan H, Gu Y, Yan J and Mao B-W 2024 *In Situ* Electrochemical Atomic Force Microscopy: From Interfaces to Interphases *Annu. Rev. Anal. Chem.* **17** 103–126 <https://doi.org/10.1146/annurev-anchem-061422-020428>

EC-AFM in battery research:

Zhang Z, Said S, Smith K, Jervis R, Howard C A, Shearing P R, Brett D J L and Miller T S 2021 Characterizing batteries by *in situ* electrochemical atomic force microscopy: a critical review *Adv. Energy Mater.* **11** 2101 518 <https://doi.org/10.1002/aenm.202101>

Gao Q, Tsai W-Y and Balke N 2022 *In situ* and operando force-based atomic force microscopy for probing local functionality in energy storage materials *Electrochem. Sci. Adv.* **2** e2100038, <https://doi.org/10.1002/elsa.202100038>

EC-SPM in Electrocatalysis:

Wang Y, Skaavik S A, Xiong X, Wang S, Dong M, 2021 Scanning probe microscopy for electrocatalysis, *Matter* **4**(11) 3483 <https://doi.org/10.1016/j.matt.2021.09.024>.

Liang Y, Pfisterer, J H K, McLaughlin D, Csoklich C, Seidl L, Bandarenka A S, Schneider O 2019 Electrochemical Scanning Probe Microscopies in Electrocatalysis *Small Methods* **3** 1800 387 <https://doi.org/10.1002/smtd.201800387>.

Cell-Design for electrochemical AFM/STM:

Valtiner M, Ankah G N, Bashir A and Renner F U 2011 Atomic force microscope imaging and force measurements at electrified and actively corroding interfaces: challenges and novel cell design *Rev. Sci. Instrum.* **82** 023703 <https://doi.org/10.1063/1.3541650>.

Freeware for image analysis:

Nečas D, Klapetek P 2012 Gwyddion: an open-source software for SPM data analysis *Cent. Eur. J. Phys.* **10** 181–188 <https://doi.org/10.2478/s11534-011-0096-2>.

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