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In Situ AFM for Real-time Monitoring of Electrochemical Processes at the Nanoscale

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Introduction

Understanding electrochemical processes at the nanoscale is critical for the advancement of energy storage, corrosion science, and electrocatalysis. *In situ* Atomic Force Microscopy (AFM) enables real-time, high-resolution imaging of dynamic changes in electrode surfaces and electrochemical interfaces under operando conditions. This article reviews the principles and recent advances in *in situ* AFM techniques for monitoring electrochemical processes, including morphological changes, phase transitions, and charge-induced surface reconstructions. We explore applications in battery electrode analysis, electrocatalysis, and corrosion monitoring, and highlight challenges such as resolution limits in liquid environments and the need for robust electrochemical AFM cell designs. Future perspectives point toward integration with spectroscopy and multi-modal platforms for a comprehensive understanding of nanoscale electrochemistry.

Description

Electrochemical processes are inherently dynamic and interfacial, involving ion exchange, phase transformations, and charge redistribution at surfaces. Traditional characterization techniques, such as scanning electron microscopy (SEM) or X-Ray Diffraction (XRD), lack the spatial resolution or environmental compatibility to observe these events in real-time. *In situ* Atomic Force Microscopy (AFM), with its ability to operate in liquid environments and provide nanometer-scale resolution, fills this critical gap. AFM has long been used for topographic imaging, but when combined with electrochemical control, it becomes a powerful tool for investigating morphological and mechanical changes at the electrode–electrolyte interface. This is essential for understanding mechanisms in applications like lithium-ion batteries, fuel cells, and corrosion-resistant coatings. *In situ* AFM for electrochemical studies integrates a fluid-compatible AFM system with a three-electrode electrochemical cell (working, reference, and counter electrodes). The AFM tip may act as an observer (non-invasive imaging) or participant (biased to influence local reactions).

Contact Mode is useful for robust materials but can disturb soft or reactive surfaces. Tapping Mode reduces lateral forces and is preferred for sensitive electrochemical surfaces. Peak Force Tapping and Fast Force Mapping modes offer quantitative nanomechanical property mapping during electrochemical operation. Imaging in liquid introduces challenges such as cantilever deflection noise, reduced Q-factor, and tip–sample convolution due to ion layers. Nonetheless, advanced cantilevers and environmental control chambers have improved imaging stability and reproducibility. In lithium-ion and solid-state batteries, *in situ* AFM reveals real-time morphological changes in electrode materials during charge/discharge cycles. Volume expansion and contraction of silicon and tin anodes. SEI (Solid Electrolyte Interphase) layer formation and evolution. Dendrite growth in lithium-metal batteries, providing insight into failure mechanisms.

Surface reconstruction of platinum or gold catalysts during hydrogen evolution or oxygen reduction. Nanoparticle aggregation, dissolution, or detachment in catalyst layers. Real-time detection of active site degradation or poisoning. *In situ* AFM is a valuable tool for studying pitting corrosion, surface passivation, and coating breakdown. It allows localized mapping of degradation zones and quantifies pit growth kinetics under varying electrochemical conditions. ESM is a derivative of AFM used to map ionic motion and electrochemical activity through electromechanical coupling. It is particularly effective in characterizing battery materials and ion-conducting ceramics. Raman Spectroscopy for chemical mapping. Electrochemical Impedance Spectroscopy (EIS) for charge transport analysis. Scanning Electrochemical Microscopy (SECM) for faradaic current mapping. Recent advancements in high-speed AFM (HS-AFM) enable real-time observation of electrochemical reactions on the order of seconds to minutes, which is crucial for capturing transient phenomena.

Electrochemical reactions can alter or degrade AFM tips, affecting resolution and repeatability. Liquids reduce cantilever sensitivity, requiring optimized detection and low-noise electronics. While spatial resolution is high, capturing rapid electrochemical events remains difficult. Electrochemical cells must be precisely designed to allow stable imaging while maintaining realistic operating conditions. The field is advancing toward fully integrated operando AFM platforms capable of mapping not just morphology but also mechanical, electrical, and chemical properties in real time [1-5].

Conclusion

In situ AFM is a transformative tool for real-time nanoscale monitoring of electrochemical processes. Its ability to visualize dynamic surface changes and quantify mechanical properties under operational conditions provides unparalleled insight into energy storage, catalysis, and corrosion phenomena. Continued innovations in instrumentation, probe design, and multimodal integration will cement AFM's role at the forefront of electrochemical research.

Acknowledgment

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Conflict of Interest

None.

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