

COMMENTS

Super-resolution catalytic current imaging with interferometric electro-optical microscopy

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The dynamic evolution of electrochemical activity on the electrochemical interfaces at the nanoscale is of great importance for designing highly efficient electrocatalysts [1-3]. Although techniques such as scanning electrochemical imaging [4] and optical imaging [5, 6] have been employed for in-situ characterization of electrochemical interfaces, it remains challenging to simultaneously monitor the dynamic evolution of electrocatalytic activity on nanoscopic heterogeneous catalysts over a large spatial range [7]. Recently, the research group led by Rui Hao at Southern University of Science and Technology, in collaboration with the teams of Song Liu and Jiang Zeng from Hunan University, as well as the group of Yung-Chang Lin at the National Institute of Advanced Industrial Science and Technology (AIST) in Japan, has achieved breakthroughs in the field of super-resolution electrochemical interface imaging by developing Interferometric Electro-Optical Microscopy (IEOM) based on the electro-optic modulation effect and optical interference effect of electrochemical interfaces [8].

When electrochemical reactions occur at the electrode surface, Faradaic currents modulate the charge distribution across the electric double layer (EDL) and space charge layer (SCL) at the electrode-electrolyte interface. These charge redistributions induce refractive index changes via the Pockels effect in the EDL and the plasma dispersion effect in the SCL, which in turn alter the optical path of reflected light [9], as shown in **Figure 1a**. Through detecting the interference optical signal from the electrochemical interfaces, the technique can extract relative catalytic current information by subsequent temporal differentiation of the images [10], achieving relative current imaging, as shown in **Figure 1b-c**. The

subsequent temporal differentiation method suppresses static background and produces spatiotemporally discrete optical spots. Super-resolution localization via point spread function (PSF) Gaussian fitting is then performed on these discrete spots, allowing IEOM to achieve a spatial resolution of ~50 nm while maintaining millisecond-level temporal resolution, enabling parallel imaging of electrochemical activity across large sample areas, as shown in **Figure 1d-h**. Its reliability has been rigorously validated through benchmark experiments on standard Au and Pt electrocatalysts, and the activity mapping on Au shows excellent consistency with scanning electrochemical cell microscopy (SECCM) measurements.

The team further investigated bilayer MoS₂—a promising non-precious metal hydrogen evolution reaction (HER) electrocatalyst¹¹—and uncovered previously inaccessible dynamic phenomena. The technique resolved the heterogeneous activity distribution across bilayer MoS₂ flakes, demonstrating that small triangular domains in the top layer exhibit significantly higher HER activity than the underlying monolayer MoS₂ region, with lower onset potentials and higher maximum current densities, as shown in **Figure 2 a-f**. Most notably, IEOM enabled the first direct visualization of dynamic directional propagation of catalytic currents in a nano-strained MoS₂ system: HER activity initiates at localized sites and then propagates continuously along the zigzag or armchair crystallographic directions of MoS₂, with propagation distances ranging from 200 nm to 1 μm. The current trajectories even exhibit characteristic 60° deflections between two zigzag directions, revealing a strong correlation between electrocatalytic dynamics and crystal orientation, as shown in **Figure 2 g-h**.

Received: July 10, 2026. Revised: July 11, 2026. Accepted: July 11, 2026. Available online: July 11, 2026

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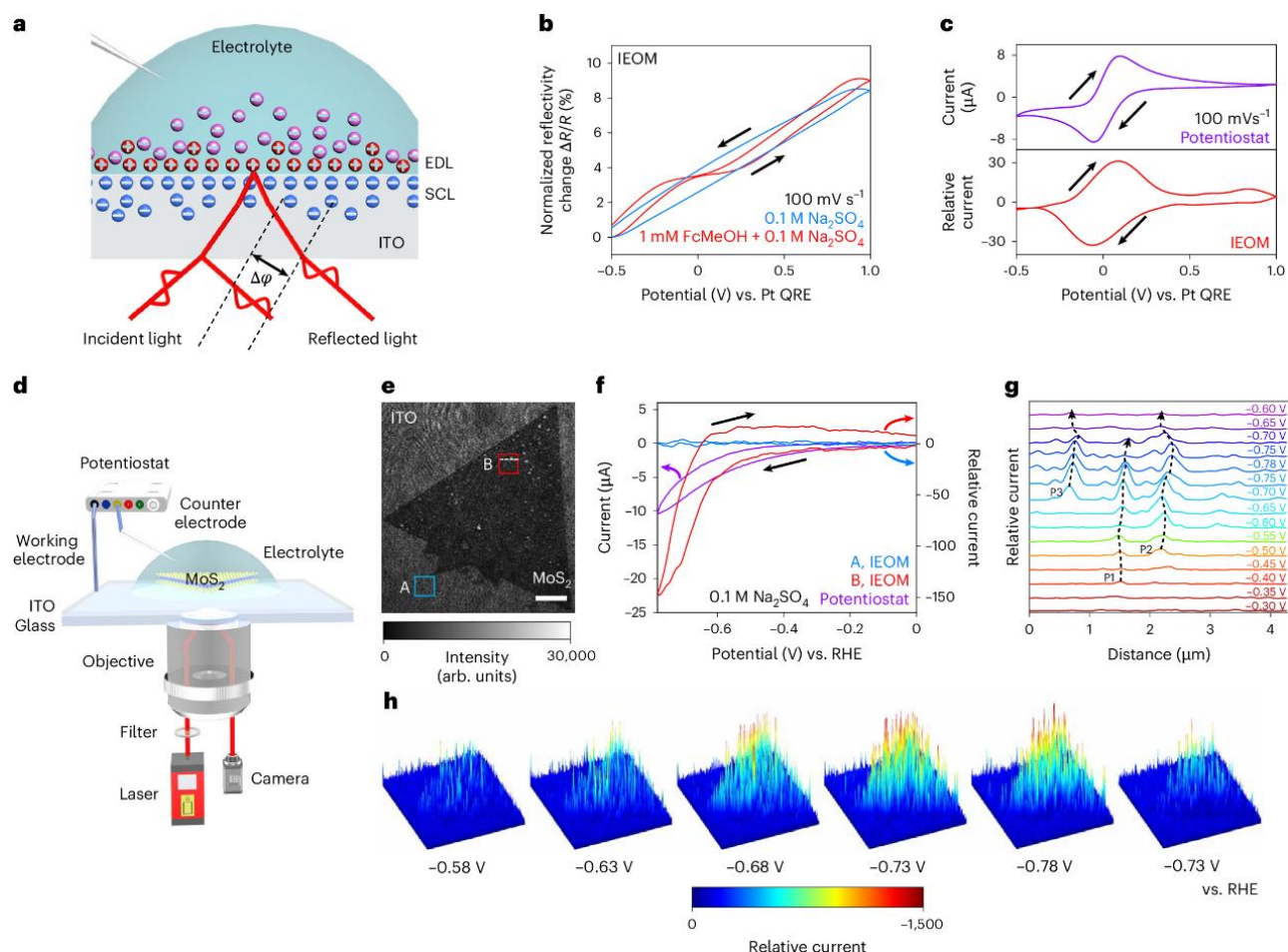


Figure 1. (a), Interference light path at the electrode-electrolyte boundary. (b), Normalized reflectivity changes measured via IEOM. (c), Comparison of CV profiles recorded simultaneously by a potentiostat and IEOM. (d), Schematic of the IEOM setup for mapping electrochemical reactions. (e), IEOM micrograph of a MoS₂ flake on an ITO electrode, 6.5 μ m. (f), Global and local CV responses extracted from regions A and B in (e). (g), Evolution of relative current relative to the applied potential across the white dashed transect shown in e. Evolution of relative current across the dashed transect in e, with active spots indicated (P1-P3). (h), 3D spatial distributions of relative currents at various applied potentials.

To elucidate the origin of this directional current propagation, the team combined aberration-corrected scanning transmission electron microscopy (STEM) and density functional theory (DFT) calculations, uncovering a nano-strain-induced directional chain HER mechanism. STEM imaging identified distinct nanoscale strain stripes extending along crystallographic directions across the top-layer MoS₂ triangles, which arise from the structural overlap of an unstrained bottom layer and a tensile- or shear-strained top layer. DFT calculations revealed that these strain stripes reduce the hydrogen adsorption free energy on the basal plane of MoS₂ by approximately 0.4 eV, significantly enhancing catalytic activity. Once the first hydrogen atom reacts at a strained site, it sequentially activates adjacent active

sites along the strain stripe—a chain activation effect. The "active hotspot" with the lowest hydrogen adsorption free energy consistently localizes at the boundary of the strain stripe and migrates along with it during hydrogenation, accounting for the observed directional current trajectories.

This work represents a major methodological advance in electrochemical imaging. IEOM provides high sensitivity and spatiotemporal resolution for super-resolution relative current imaging of electrochemical reaction kinetics. Moreover, it establishes a robust framework for quantitatively linking catalytic performance to nanoscale morphology, thereby enabling a rational pathway toward the development of advanced electrocatalysts.

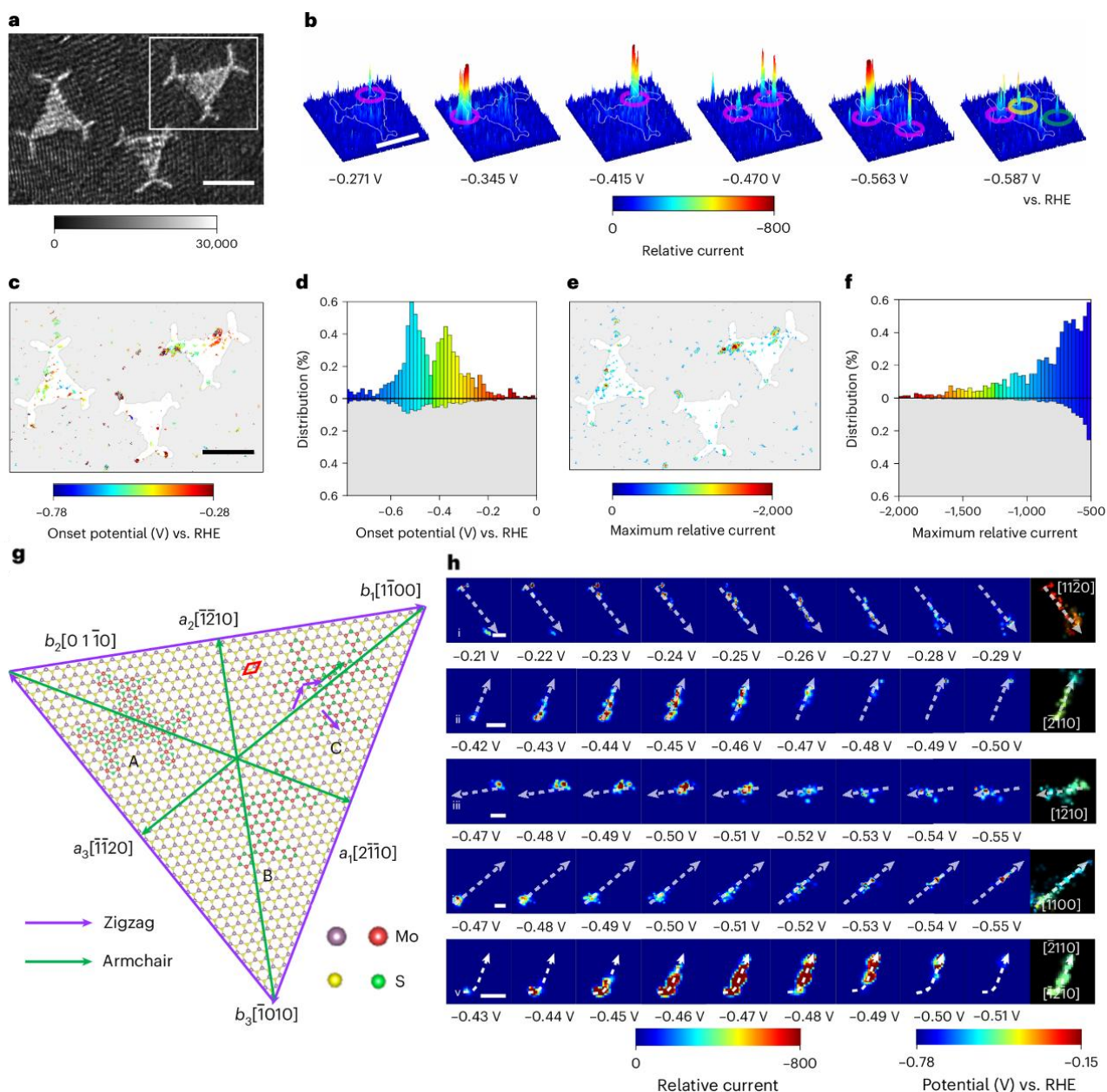


Figure 2. (a), IEOM micrograph of smaller MoS₂ flakes on a basal layer. Scale, 4 μm. (b), Spatial distribution of relative currents within the boxed area in a, highlighting distinct active peaks. Scale, 4 μm. (c), Spatial map of local onset potentials across the MoS₂ flakes. Scale, 4 μm. (d), Frequency distribution histograms of local onset potentials. (e), Spatial mapping showing the peak relative currents achieved across the MoS₂ flakes. (f), Distribution histograms of peak relative currents. (g), Diagram of crystal lattice orientation outlining zigzag and armchair propagation paths. (h), Snapshots of localized current peaks and their propagation paths during a CV sweep.

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