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Scanning Electrochemical Microscopy (SECM): Brief Overview of the Technique and Application of its Surface Interrogation (SI-SECM) Variant in Probing Hydrogen Adsorption Affinity of Pt and Ir

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The principle (cross-talk between a microelectrode and a substrate via an electroactive species) and the main variants (feedback, generation/collection and surface interrogation modes) of Scanning Electrochemical Microscopy (SECM) are briefly overviewed. The application of SECM in the surface interrogation mode (SI-SECM) to probe the strength of metal-adsorbed H bonds at Pt and Ir substrates is presented in detail. Linear sweep voltammetry at a microelectrode positioned at a very small probe-substrate distance (2.5 μm) shows remarkable positive feedback in the presence of an oxidizable mediator (TMPD) when a full or partial H monolayer has been electrochemically pre-formed on Pt and Ir substrates. The magnitude and shapes of chronoamperometric responses at the tip are interpreted by means of H coverage variation with time and changes in the open circuit potential of the substrate, both related to H_{ads} and OH_{ads} affinity of the latter. A more complex behavior is observed at bimetallic Pt(Ni) and Ir(Ni) substrates. In all cases, the results of SI-SECM are correlated via the H adsorption strength to the activity of the substrate towards the hydrogen evolution reaction (HER) as typically studied by current-potential curves.

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