## Advanced Electroactive Materials for Sustainable Energy: Innovations in Electrocatalyst Design via the Nano Lab Concept

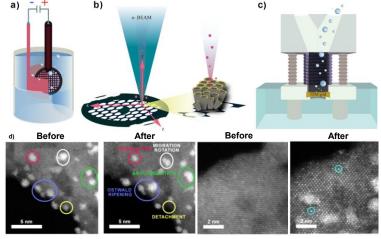
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As the global community faces the urgent global challenge of climate change, the search for sustainable energy solutions becomes critical. At the centre of this search is the innovation of technologies for energy storage and conversion. Our research introduces a novel approach to optimising catalysts for the oxygen evolution reaction (OER) using the **Nano-Lab concept**<sup>1,2</sup> (Fig. 1), an integrated experimental platform that provides insights into the relationships between synthesis, structureand performance at the atomic level. We focus on nanostructured titanium oxynitride ( $TiO_xN_y$ ) substrates, prepared via anodic oxidation (AO) and subsequent nitridation, which provide a high-surface-area and electrically conductive support for immobilized iridium nanoparticles. Leveraging strong metal—support interactions (SMSI), these catalysts demonstrate exceptional performance and stability while drastically reducing noble-metal loading<sup>1</sup>. A distinctive feature of our work is the implementation of identical-location electron microscopy (IL-TEM, IL-SEM) on dedicated TEM grids throughout all synthesis and electrochemical steps. Combined with floating

electrode electrochemical characterization, Raman spectroscopy, and XPS, this setup enables direct tracking of morphological, compositional, and structural changes at the nanoscale. We report dynamic transformations of both the Ir nanoparticles and the TiO<sub>x</sub>N<sub>y</sub> support during electrochemical operation, including the formation of atomically dispersed Ir species. Our results illustrate the path towards robust, cost-effective electrocatalysts that are essential for the transition to a carbon-neutral hydrogen economy.



**Fig. 1:** Nano Lab integrates multiple advanced characterization techniques: (a) AO of the TEM grid, (b) IL-TEM technique, (c) electrochemical characterization and d) two sequences of Identical location STEM—HAADF image of the TiOxNy—Ir sample before electrochemical degradation and after it - the left pair showing all the possible degradation processes and the right pair showing Ir nanoparticles as well as the formation of Ir single atoms<sup>2</sup>.

- 1. M. BELE, et al., ACS catalysis, 2020, 10, 13688-13700
- 2. M. BELE, et al., ACS applied nano materials, 2023, 6 (12), 10421–10430