

Supporting information

Bridging the gap between basic research and application: A half-cell setup for high current density measurements of Ir-based oxygen evolution reaction catalysts on porous transport electrodes.

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Hot pressing and cell assembly in the GDE and PTE setup configurations

Our laboratory-developed hot pressing station was constructed using a modified soldering iron installed in a small vertical lever press, see Figure S1A. The unit was set to a temperature of 130 °C, as measured with a type J thermocouple inserted in the hot tip. The sample was positioned on the hot end for 15 min before pressing to adjust it to the target temperature. In this study, the Nafion membrane (Nafion 117, 183 µm thick, Fuel Cell Store) was activated before pressing as described by Schröder et al. [1]. Then, the samples were pressed at 84 bar for 60 s. to achieve good interfacial contact between the coated substrates and the membrane

In the GDE setup configuration, see Figure S1B, the GDEs were prepared as described by Collantes et al. [2]. In this compartment, the CE and RE shared the same pool of electrolyte (HClO_4) and were separated only by independent porous glass frits, see Figure S1C. In the GDE setup measurements, the reactant was supplied through a humidified gas bubbler (Ar/O_2) and set to a constant flow rate of 100 sccm, see Figure S1D. The assembly details of the GDE setup appear below in Figure S1E.

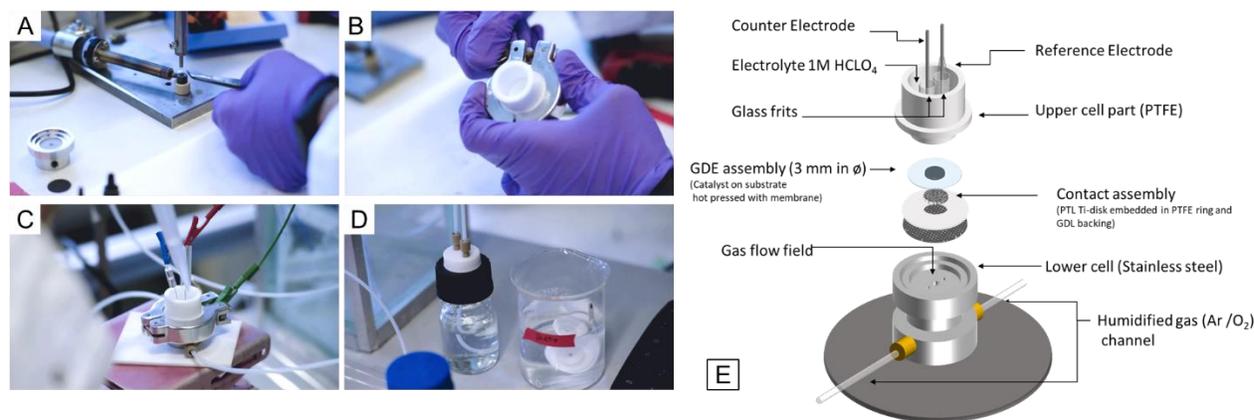


Figure S1. Snapshots of the sample manufacturing process and inner assembly of the GDE setup. A. Hot pressing procedure. B. Tightening of the top cell part after inserting the cell assembly. C. Electrodes connected and ready to test. D. Water bubbler for humidified gas. E. Schematic of the cell parts and the assembly used for GDL substrates.

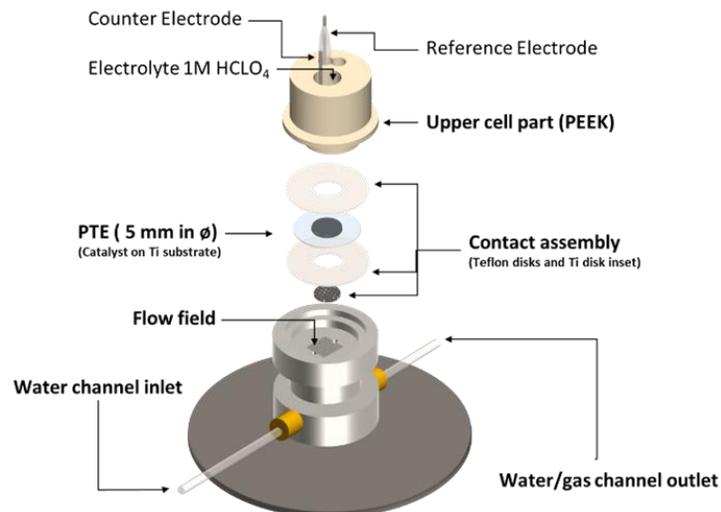


Figure S2: Schematic of the PTE cell setup and assembly used for Ti PTL substrates.

In the PTE setup, a disk of $\text{\O} 5 \text{ mm}$ was punched from the sputtered Ti PTL - the PTE - and hot pressed with a $\text{\O} 16 \text{ mm}$ Nafion membrane at $130 \text{ }^\circ\text{C}$ for 60 s. The resulting half-cell assembly, see Figure S2, was immediately immersed in DI water for one minute to rehydrate the membrane and avoid detachment due to differential strain in the interface with the substrate, and then dried before assembly. For the assembly, $\text{\O} 20 \text{ mm}$ disks with concentric holes were punched using a concentric circular steel punch (BOEHM, Germany) from a 0.25 mm and 0.50 mm Teflon sheet respectively, and also from a $\text{\O} 5 \text{ mm}$ PTL blank. The thinner Teflon disk was positioned directly over the flow field, with the PTL blank as an inset. Then, the half-cell assembly was dried lightly on a lab tissue (Rotizell, Roth) and sandwiched with the thicker Teflon disk. The latter disk also featured a small eccentric $\text{\O} 1.5 \text{ mm}$ hole centered 5 mm aside from the main aperture to communicate the tip of the Luggin capillary in the RE chamber directly to the membrane. After mounting the PTE and the contact assembly into the lower stainless steel cell, the PEEK part was carefully positioned on top, ensuring an unobstructed path for the Luggin capillary to the membrane. The two parts were manually clamped, and water tightness was checked filling the Luggin the capillary while checking that the main chamber was dry.

Setup reproducibility:

Starting from a sputtered IrOx $0.250 \text{ mg cm}^{-2}_{\text{geo}}$ catalyst deposition on a carbon substrate, two GDE catalyst assemblies were hot pressed at 84 bar and the OER activity was measured using humidified oxygen gas in the GDE setup (hollow black circles) and the PTE setup (hollow red circles), see Figure S3. The assembly measured in the PTE setup showed an improvement of $15 \text{ mA cm}^{-2}_{\text{geo}}$ measured at $1.55 \text{ V}_{\text{RHE}}$ and consistently larger improvements at higher potentials over the GDE setup, which indicates a better iR correction from the Luggin capillary design ending on the membrane. Additionally, the lower Tafel slopes up to higher current densities show that more favorable mass transport conditions are achieved in the PTE setup, see Table 2. However, it is also seen that both setups exhibit limitations at $100 \text{ mA cm}^{-2}_{\text{geo}}$, which as it was mentioned earlier indicates the degradation onset in carbon substrates operating with potentials exceeding $1.6 \text{ V}_{\text{RHE}}$.

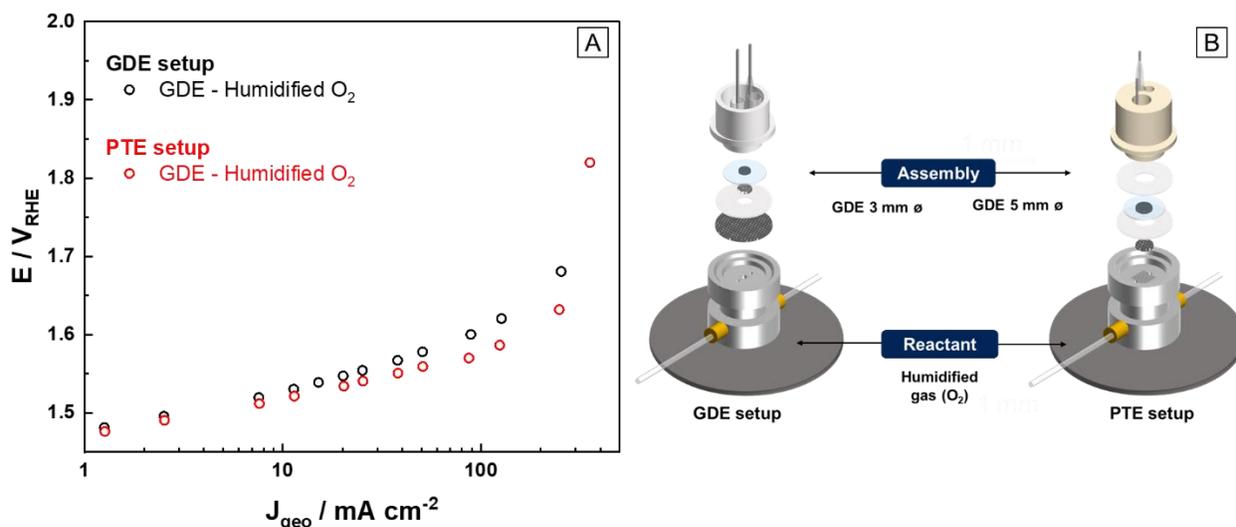


Figure S3. A. Influence of the setup configuration (GDE vs PTE) in the OER activity of a sputtered IrOx $0.250 \text{ mg cm}^{-2}_{\text{geo}}$ catalyst measured in punched samples of 5 mm in diameter on GDL substrates. B. Schematic figure of the assembly and experimental conditions in both setup configurations.

References

- [1] J. Schröder, V.A. Mints, A. Bornet, E. Berner, M. Fathi Tovini, J. Quinson, G.K.H. Wiberg, F. Bizzotto, H.A. El-Sayed, M. Arenz, M.F. Tovini, J. Quinson, G.K.H. Wiberg, F. Bizzotto, H.A. El-Sayed, M. Arenz, Supporting Information - The Gas Diffusion Electrode Setup as Straightforward Testing Device for Proton Exchange Membrane Water Electrolyzer Catalysts, *JACS Au*. 1 (2021) 247–251. <https://doi.org/10.1021/jacsau.1c00015>.
- [2] P. Collantes Jiménez, G. Sievers, A. Quade, V. Brüser, R.K. Pittkowski, M. Arenz, Gas diffusion electrode activity measurements of iridium-based self-supported catalysts produced by alternated physical vapour deposition, *J Power Sources*. 569 (2023). <https://doi.org/10.1016/j.jpowsour.2023.232990>.