

Supporting Information

**Ultramicroelectrodes fabrication guidelines for multifunctional scanning
electrochemical microscopy**

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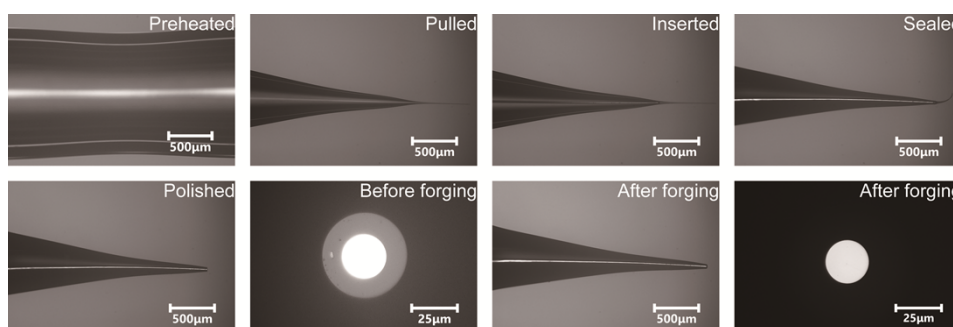


Figure S1. Fabrication and characterization of UME via a thick-wall nanopipette template and tip forging method, including the preheated and pulled capillary and the subsequent metal-inserted, sealed, and forged tip.

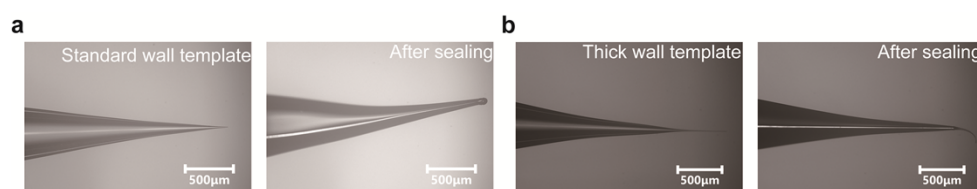


Figure S2. Bending effect during tip sealing process. **a**, Tip sealing using standard wall nanopipette template. **b**, Tip sealing using thick-wall nanopipette template.

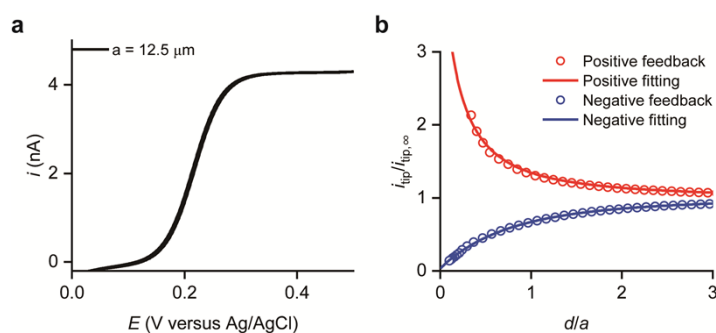


Figure S3. **a**, Steady-state voltammograms at a 12.5 μm -radius platinum disk UME. The scan rate was 10 mV s^{-1} . **b**, SECM approach curves recorded with the 5 μm -radius platinum disk UME toward an insulating and a conductive substrate. The approach velocity was 0.2 $\mu\text{m s}^{-1}$.

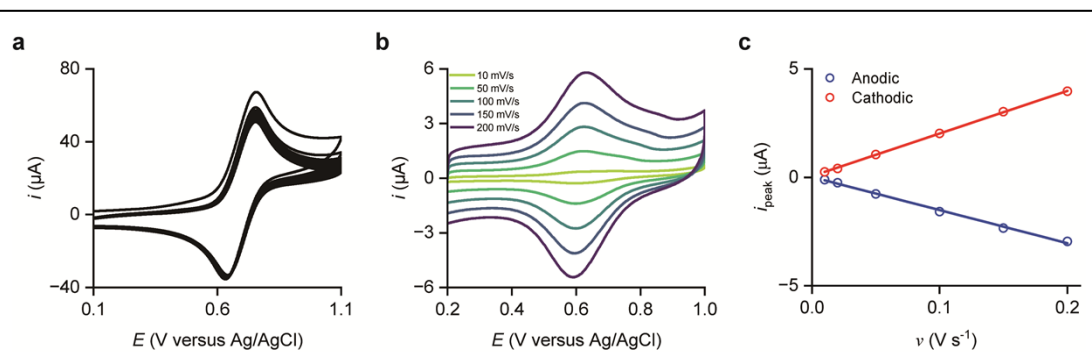


Figure S4. **a**, Electrografting of 4-amino-TEMPO molecule on GCE in PPB (pH = 7.5) solution. The scan rate was 0.1 V s⁻¹. **b**, CVs of TEMPO-modified GCE at different scan rates in PPB (pH = 7.5) solution. **c**, Scan rate dependence of the anodic and cathodic peak current.

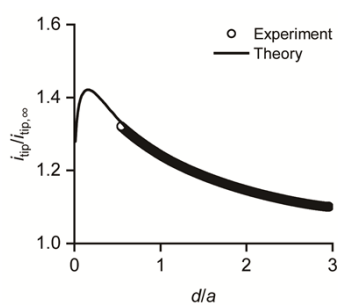


Figure S5. Approach curve of 12.5 μ m-radius platinum/gold DDE at the TEMPO-modified GCE. The approach curve was recorded at a velocity of 0.2 μ m·s⁻¹ using FcMeOH as the redox mediator.

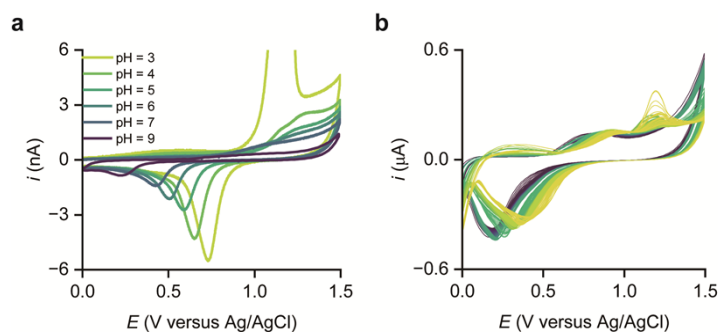


Figure S6. **a**, pH calibration of the gold pH sensor in PPB solution. **b**, In situ pH measurements of the gold pH sensor positioned 12 μ m above the substrate surface in 0.1 M KOH. The scan rate was 0.5 V s⁻¹.

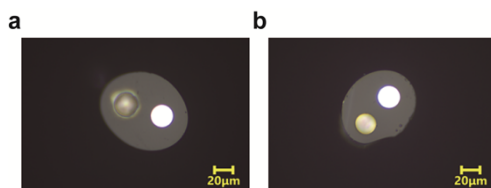


Figure S7. **a**, Optical micrograph of 12.5 μm-radius platinum disk/mercury sphere electrode. **b**, Optical micrograph of 12.5 μm-radius platinum disk/mercury disk electrode.

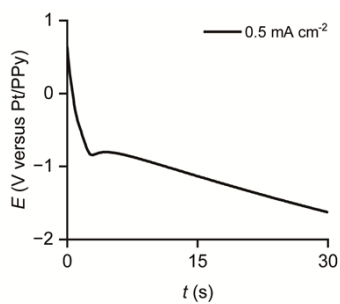


Figure S8. Lithium plating at copper using chronopotentiometry.

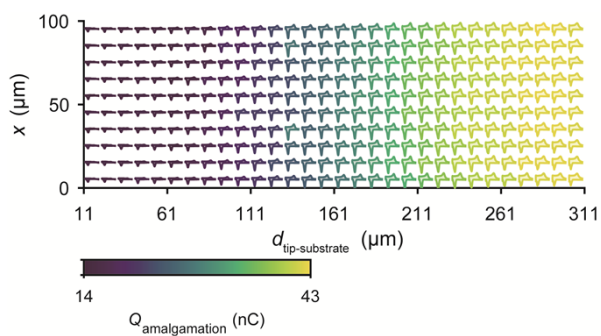


Figure S9. CV-SECM data of mercury disk during lithium plating at 0.5 mA cm^{-2} .

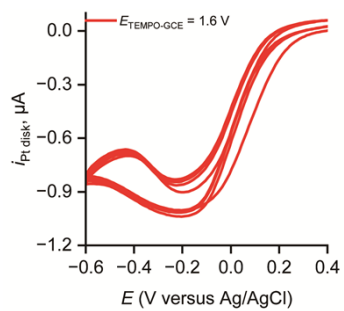


Figure S10. Voltammetric response of the platinum disk for O_2 detection in 0.1 M KOH over five consecutive cycles at a tip-to-substrate distance of 12 μm . The scan rate was 0.5 V s^{-1} . The substrate potential was 1.6 V versus Ag/AgCl.