

Supporting Information:

Multi-spectroscopy study of electrochemically-formed oxide-derived gold electrodes

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Supplementary figures

Electrochemical cell representation with SFG output

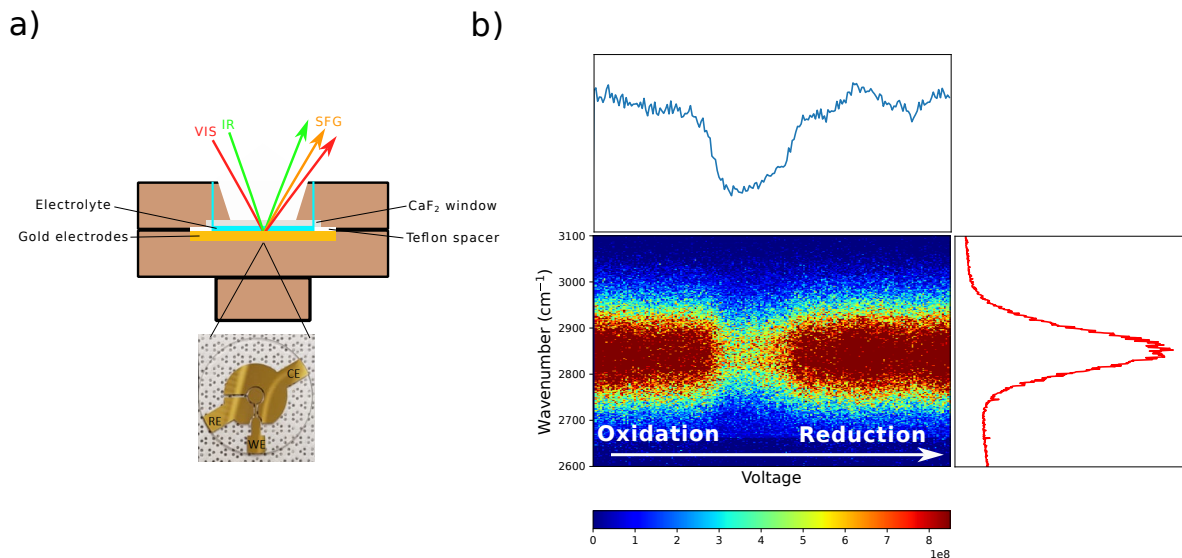


Figure S1: (a) Top - Schematic design of the EC highlighting the main parts. Bottom - Three polycrystalline gold electrodes (WE, CE, and RE) deposited on a quartz plate. (b) SFG spectrum of gold as a function of voltage. The side panels show the projected intensity on the axes.

Protocol 1: Electrochemical annealing before SFG measurements

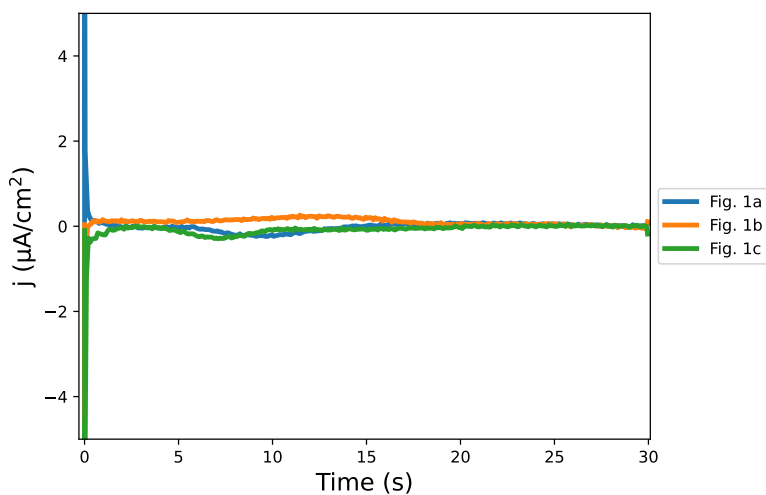


Figure S2: Preparation step (CA at 0.7 V vs. SHE) for the measurements in Figure 1 a-c.

Protocol 2: Chronoamperometric measurements for steps 1 and 2

before SFG measurements

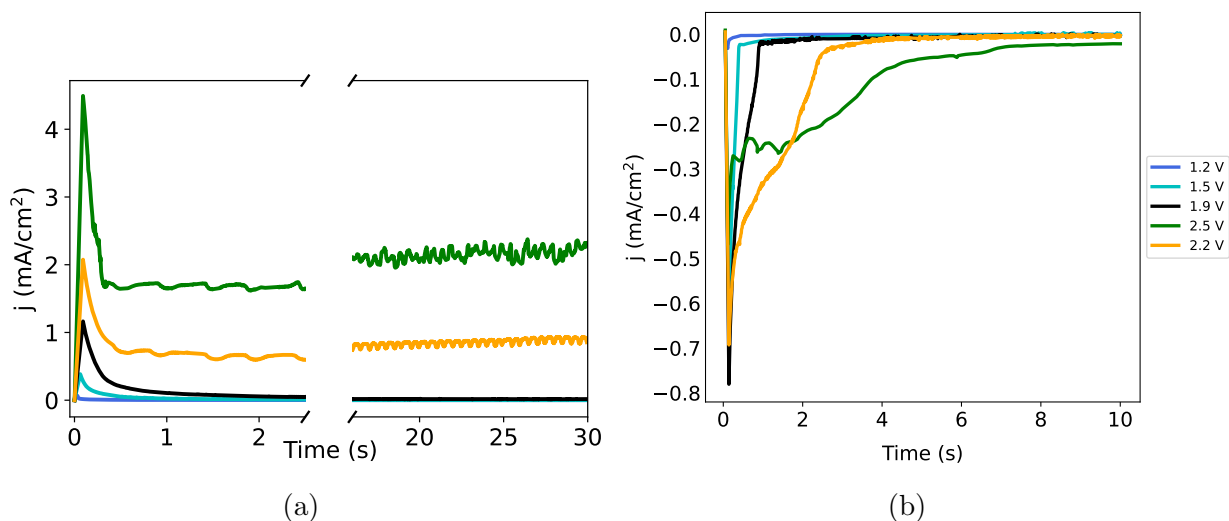


Figure S3: Additional points of the protocol summarized in figure 4 of the main article. a) Step 1: CAs vs. SHE in H₃PO₄ at various oxidation potentials from 1.2 V and up, where the increase in current density seen is due to the growth of gold oxide and the OER. b) Step 2: CA at 0.7 V after holding the positive potentials shown in (a).

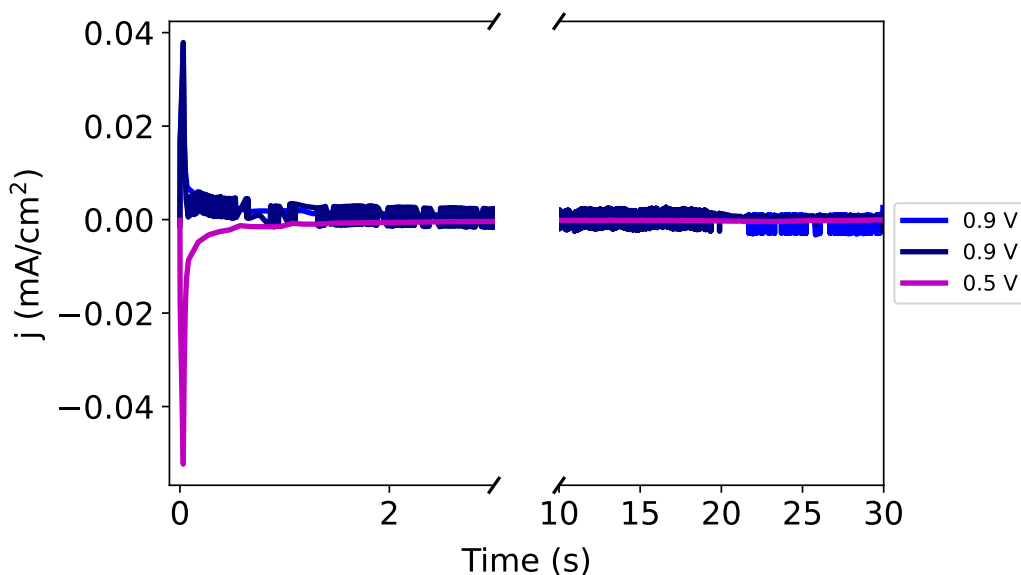


Figure S4: Additional points of the protocol summarized in figure 4 of the main article. Comparison of the CAs vs. SHE from step 1: 0.9 V held before (bright blue) and after (dark blue) the set of measurements for 2.2 V, showing the same trend. This is compared to the CA done at 0.5 V (violet), which matches the neat gold state shown by SFG.

Protocol 2: Chronoamperometric measurements for steps 1 and 2

before HP-XPS measurements

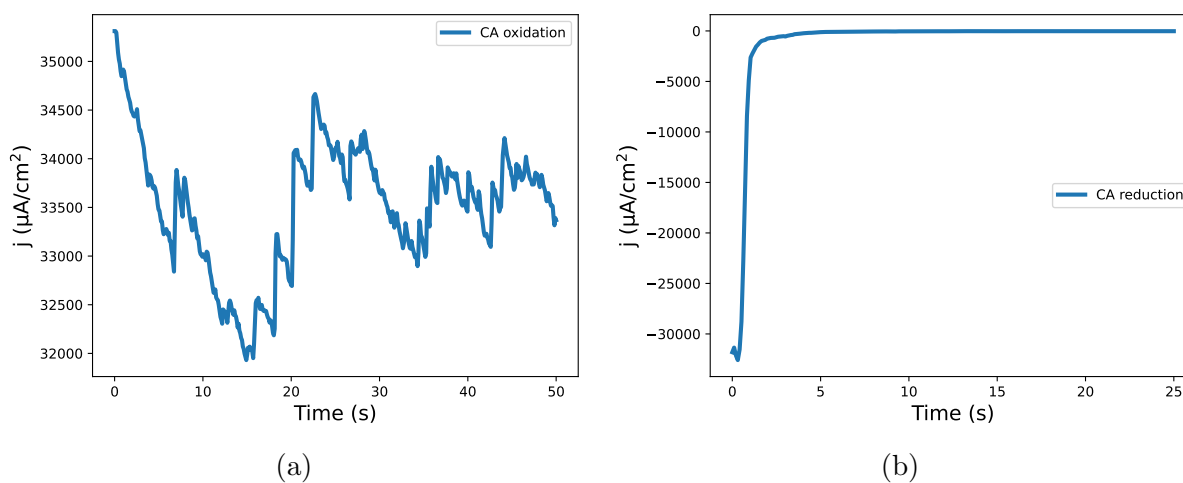


Figure S5: Electrochemical treatment done before collecting the HP-XPS spectra in Figure 6. a) CA at 2.5 V vs. SHE to oxidize the gold surface. b) CA at 0.7 V vs. SHE to reduce the oxide formed in a).

Additional HP-XPS spectra

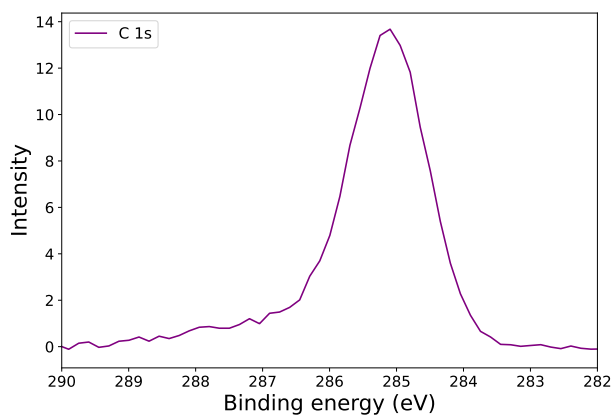


Figure S6: C 1s spectrum collected to check the possible presence of contaminants on the gold surface.

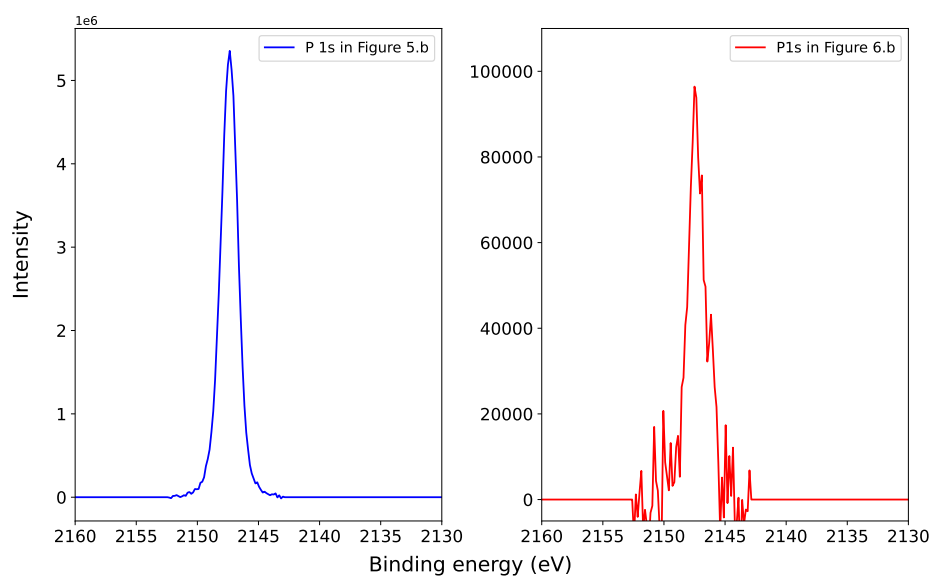


Figure S7: P 1s spectra collected together with Au 4*f* and O 1s spectra. The integrated intensity of the only peak in P 1s core-level region was used to calculate the contribution of H_2PO_4^- in the O 1s spectra (more details in the main text, Figs. 5.b and 6.b). A Shirley background was subtracted.