# Supporting Information For:

# Assessment of the Stability and Operability of Cobalt Phosphide Electrocatalyst for Hydrogen Evolution

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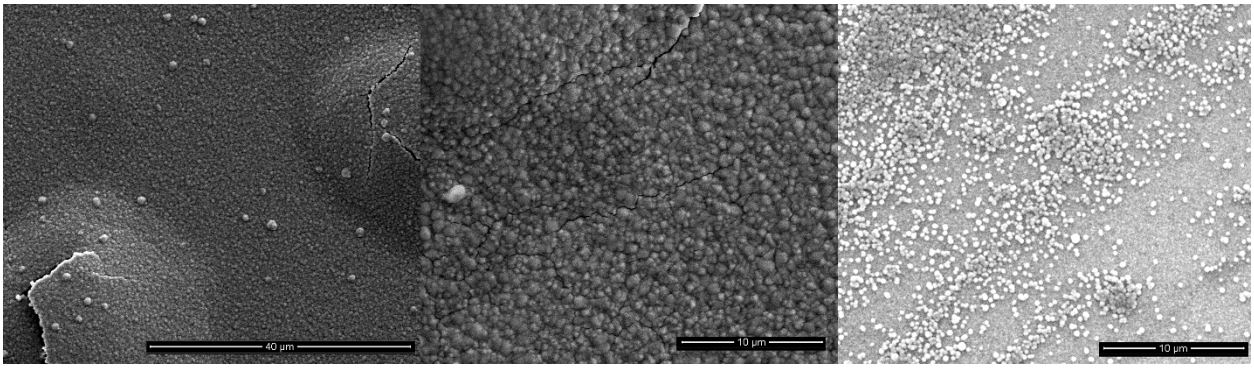


**Figure S1.** Scanning electron micrographs of the CoP HER catalyst on an FTO electrode. Left panel displays an image of a freshly prepared surface and the center panel that of an electrode after 20 minutes of operation. Little morphological change was observed. On the right panel of the image of the electrode after 60 minutes of use, and shown in the image is the largely exposed bare FTO surface due to the CoP film removal from oxidative stress.

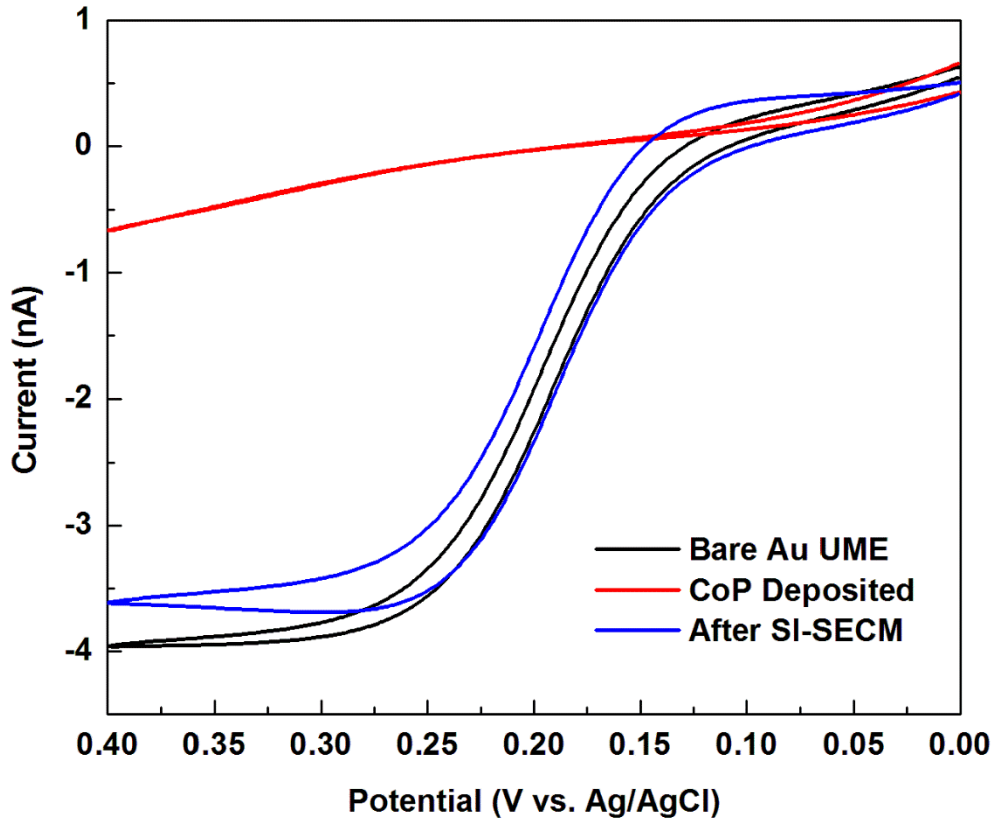


**Figure S2.** A cyclic voltammogram (CV) of 1,1'-ferrocenedimethanol (1.2 mM) oxidation in 50 mM phosphate buffered water at pH 5 (25 µm diameter gold UME, clean surface; black trace). Displayed in red trace is a CV of 1,1'-ferrocenedimethanol oxidation by a CoP deposited gold UME. Much of the Faradaic current arising from the ferrocene oxidation is subdued due to the conductive electrode area coverage by the resistive CoP material. The blue trace is a CV of the identical reaction by a CoP electrode after the SI-SECM experiments. Clearly, most of the conductive gold electrode surface was recovered, indicating that the CoP overcoat has been removed by the oxidative stressed imposed onto the electrode by the redox mediator during the SI-SECM experiments.

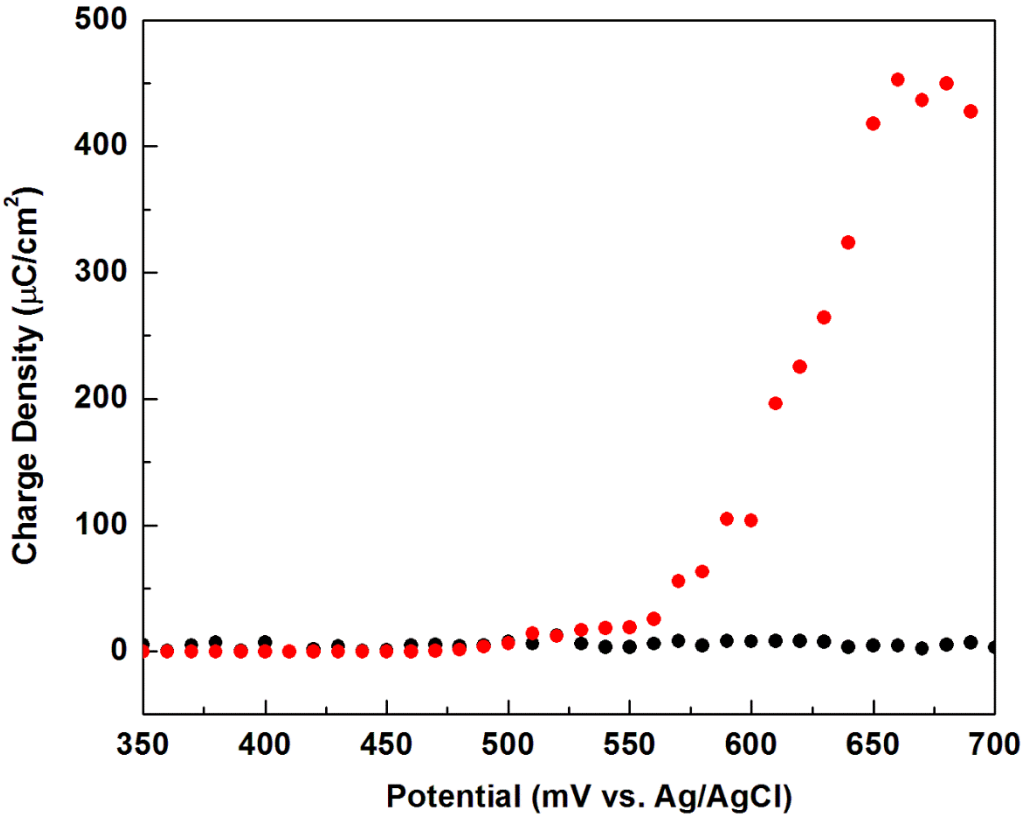


**Figure S3.** A SI-SECM redox titration curve of the CoP HER catalyst electrode obtained in a pH 2.8 solution using a $Ru(CN)_6^{4-}$ redox mediator (black dots). Similar to that shown in Figure 3 of the manuscript, little feedback was obtained due to rapid oxidative stripping of the catalyst film. Shown in red dots is a redox titration curve of a $MoS_2$ HER catalyst for reference.[1]

## References

(1) Ahn, H. S.; Bard, A. J. *J. Phys. Chem. Lett.* **2016**, *7*, 2748.