Abstract:

The design, assembly, and operational considerations of a multi-technique interfacial physics instrument are described, highlighting the integration of traditional electrochemical methods with modern surface spectroscopic and preparative techniques. The instrument, referred to as ECSSA (Electrochemical Surface Science Apparatus), is a central tool in the experimental approach of the surface science laboratory at JCAP-Caltech. The principal function of the ECSSA is to provide a platform for the non-traditional, atomic-level approach to the study of CO₂R heterogeneous electrocatalysts. The interrogation protocol is based on the detailed examination of well-defined model catalyst systems before, during, and after precisely controlled reaction conditions. The complementarity of EC-CSA with the seriatim module of operando tools developed separately bridges the pressure and materials gap in the investigation of model and “real world” electrocatalysts.

Research Activities: Highlights

- **Approach: Emission Potential/Structure/Composition Correlates**
- **Example Work: Electrochemical Deposition of Fe/WC(pc) Thin-Films**
- **Dependence of Stripping Charge on Potential and Charge-limits**
- **Calibration of As-Deposited Fe-Adlayer Thickness**
- **Surface Characterization and Product Analysis**

Team 2012-2020

- **Apparatus for Surface Science Investigations of CO₂R Catalysts**

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Conclusions

1. Electrochemical surface science protocols have been developed for the reproducible electrodeposition and characterization of Fe films, with monolayer to bulk coverages, on WC(pc).
2. The surface packing density of Fe adatoms on WC may be controlled by a reproducible electrodeposition and characterization of Fe films, with monolayer to bulk coverages, on WC(pc).
3. Monolayer coverages of Fe on WC(pc) enhance the selectivity of CO₂R in bicarbonate solution toward CH₄ production.