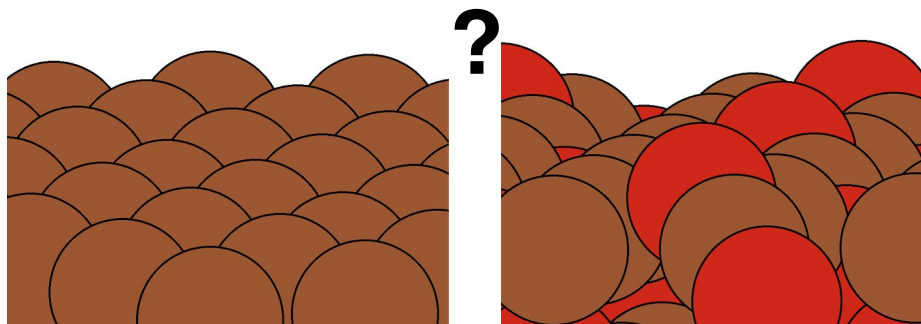


### ELECTROCHEMICAL CARBON MONOXIDE REDUCTION

## Oxygen prospecting

ACS Energy Lett. <https://go.nature.com/2EvposR> (2019).



The electrochemical reduction of  $\text{CO}_2$  or CO represents a valuable and environmental recycling strategy, especially when the supplied electricity is obtained from renewable energy sources. Copper is capable of producing multi-carbon products that can be used as fuels or chemical feedstocks. While many copper-based catalysts have achieved excellent performance for both reactions — including shape-controlled, polycrystalline, alloyed or in situ reduced oxides — many aspects regarding the actual active structure still remain elusive. In particular, it is unclear whether oxygen is present near the surface under reaction conditions. Also unclear is its potential role in promoting the activity and selectivity towards multi-carbon products.

Now, Ib Chorkendorff, Thomas Jaramillo and colleagues have demonstrated that the surface and near-surface regions of polycrystalline copper are fully reduced to the metallic phase under electrochemical CO reduction conditions. The researchers have performed in situ grazing incidence X-ray diffraction with synchrotron radiation in alkaline electrolyte at potentials varying from 0.65 V (close to the open-circuit

potential) to  $-0.4$  V (during CO reduction) versus the reversible hydrogen electrode.

A broad diffraction peak — suggesting low crystallinity — corresponding to the  $\text{Cu}_2\text{O}$  phase is observed at 0.65 V, likely formed upon exposure to air and/or at open-circuit potential. The oxide diffraction peak, however, vanishes between 0.4 and 0.3 V, correlating with the subsequent increase in intensity of the metallic Cu diffraction peaks. Significant presence of substoichiometric oxide in the surface or near-surface region under reaction conditions can be ruled out, since the diffractogram remains unaltered upon decreasing the potential from 0.3 to  $-0.4$  V. Furthermore, the researchers show, by comparing the  $\text{Cu}(200)/\text{Cu}(111)$  integrated peak intensity ratio in Ar- and CO-saturated electrolyte, that CO promotes restructuring of the surface region to the (100) termination.

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