# *In situ* Conductive Atomic Force Microscopy for Localized Analysis of CO2 Electrocatalysts

Martin Munz, Maël Brulé, Jeffrey Poon, Wiebke Frandsen, Christopher Kley, Beatriz Roldan Cuenya

A key challenge in catalysis and energy conversion research remains probing, at the nanoscale and under liquid phase reaction conditions, a material's local electric conductivity, catalytic activity, product selectivity or the chemical nature of its reaction environment. Particularly, *in situ* derived structure-property relationships are required to rationally innovate (photo-) electrochemical interfaces that dictate major catalytic reactions including CO2 electroreduction (CO2RR) and water splitting.

In this work, we introduce *in situ* correlative atomic force microscopy (AFM) to simultaneously image an electrode's conductivity, chemical-frictional and morphological properties. By combining functional imaging with the high spatial resolution afforded by AFM, we address the challenge of resolving nanoscale heterogeneities of electrode surfaces and associated spatial variations in the interfacial electron transfer. Here, we demonstrate the feasibility of using conductive AFM (c-AFM) for electric current imaging1 in relevant aqueous electrolytes, beyond the common operation in air2 or nonpolar liquids3.

For the case of CO2RR, we show that *in situ* c-AFM allows laterally resolved analysis of variations in the electric current occurring across the electrode/electrolyte/probe interface, where the electrode is exemplified by bimetallic copper-gold CO2RR electrocatalysts in air, water and bicarbonate electrolyte4. In line with contrasts of current images, current-voltage curves reveal resistive CuOx islands, while frictional imaging indicates qualitative variations in the hydration layer molecular ordering upon change from water to electrolyte. *In situ* c-AFM imaging of nanocrystalline Au, at the granular length scale, suggests that concave intergranular regions are more resistive than convex intragranular regions. Furthermore, we report a correlation indicating that higher friction forces are accompanied by reduced interfacial currents, thus highlighting variations in the interfacial molecular ordering with the electrolyte composition and ionic species present, characterized by their chaotropic or kosmotropic effects.

Our approach facilitates disentangling the effects of electrochemical micro-environments onto interfacial charge transfer or adsorption processes and sets the basis for addressing fundamental challenges in photo-electrocatalysis, battery technology, corrosion science and tribology.

References

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