

Atom Probe Tomography of Catalyst Nanoparticles

Nora Vorlaufer^{1*}, Jan Josten¹, Chandra Macauley¹, Nemanja Martić², Andreas Hutzler³, Nicola Taccardi⁴, Karl Mayrhofer³ and Peter Felfel¹

¹ Institute I, General Materials Properties, Friedrich-Alexander-University Erlangen-Nürnberg, Erlangen, Bavaria Germany.

² Siemens Gas and Power GmbH & Co. KG, Günther-Scharowsky-Str. 1, Erlangen, Bavaria, Germany.

³ Helmholtz Institute Erlangen-Nürnberg for Renewable Energy, Forschungszentrum Jülich GmbH, Erlangen, Bavaria, Germany.

⁴ Chair of Chemical Engineering I, Reaction Engineering, Friedrich-Alexander-University Erlangen-Nürnberg, Erlangen, Bavaria Germany.

* Corresponding author: nora.vorlaufer@fau.de

Nanoparticles are among the most important catalytic materials, especially in electrochemistry, where they enable e.g. the conversion of CO₂ to high value fine chemicals [1] or dehydrogenation reactions [2]. To achieve an understanding of their chemical makeup, atom probe tomography (APT) is a uniquely capable measurement technique, as it can provide single atom sensitive analysis with three-dimensional chemical information. To enable analysis of nanoparticles down to ca. 50 nm, a ‘pick and coat’ approach [3] was recently introduced.

With this method it is possible to select a specific nanoparticle for analysis in an SEM and subsequently prepare an atom probe specimen. The proposed preparation technique avoids damaging the particle by the FIB beam. Additionally very delicate nanoparticles can be handled without damaging them.

In this study we demonstrate the usage of the ‘pick and coat’ technique to characterize two different types of nanoparticles that are highly-relevant in catalytic reactions.

Firstly, SCALMS nanoparticles for dehydrogenation reactions [2] are analyzed in the atom probe. This new kind of catalyst nanoparticles have the special limitation that the matrix consists of gallium. Thus they exhibit a low melting point and tend to melt using conventional preparation methods. The ‘pick and coat’ method enables the preparation of this delicate type of nanoparticles without damaging them.

The second example covers the analysis of nanorods that were designed for deployment in CO₂ electrolysis reactions [1]. The nanorods were measured in their pristine form. The acquired APT data were directly correlated with STEM EDX measurements. The results demonstrate that the nanorods are thermodynamically not stable and tend to change their chemical composition easily when energy is introduced into the system.

Additionally, it was possible to analyze two different morphologies that evolve during the catalytical process (fig. 1). Not only an evolution of the chemistry of the rods can be observed, but also the composition of a newly evolving morphology was analyzed. For the first time we reveal the incorporation of the electrolyte into the surface of the catalyst and thereby contribute to understand the evolution of the catalytic behavior [4].

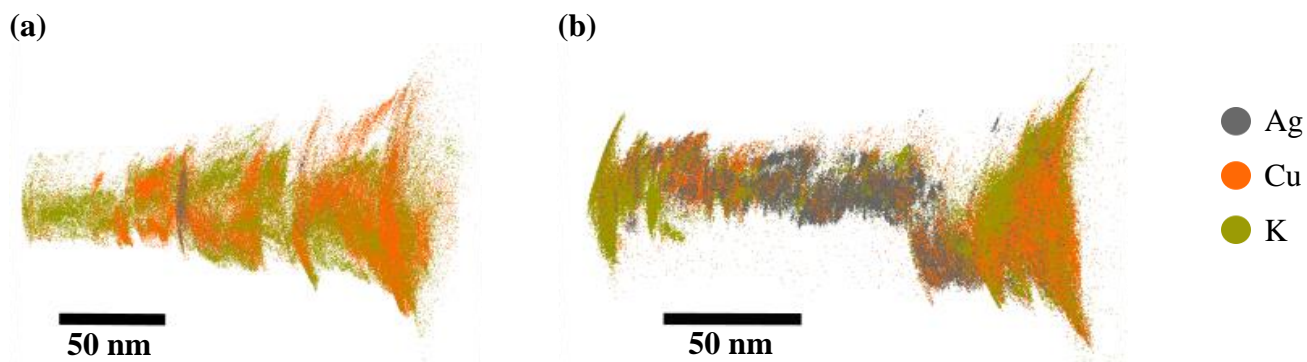


Figure 1. (a) and (b) show two kinds of microstructures that evolve during CO₂ electrolysis reactions. (a) is rich of potassium, while (b) is silver rich.

References:

[1] N. Martić, et al., *Energy & Environmental Science* **13** (2020), p. 2993-3006. doi: 10.1039/D0EE01100B

[2] N. Taccardi, et al, *Nature Chem* **9** (2017), p. 862-867

[3] J. Josten, P. Felfer, *Microscopy and Microanalysis* (2021), p. 1-10. doi:10.1017/S1431927621000465

[4] This work was funded by the Deutsche Forschungsgemeinschaft (DFG - German Research Foundation) under project FE 1593/2-1, the Sonderforschungsbereich 1452 „Katalyse an flüssigen Grenzflächen“ (CLINT – Catalysis at Liquid Interfaces) and the research training group GRK 1896 “*In Situ* Microscopy with Electrons, X-rays and Scanning Probes.”