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Adverse X-ray Beam Effects in Electrochemical Nano-Focused Synchrotron Studies

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The palladium hydrogen system is a useful model system in the study of both hydrogen absorption for energy storage, and 3D lattice gas systems for basic thermodynamic models. Here, we will show how nano-focused x-ray diffraction and Bragg coherent x-ray diffraction imaging carried out at the newly upgraded ESRF, a 4th generation x-ray light source, give us insight into the strain evolution in palladium nanoparticles during hydrogen absorption and desorption cycling.

In these measurements the control parameter is the quantity of hydrogen available for insertion, which is normally determined by the electrochemical potential, or partial pressure in a gas system. However, the x-ray nanobeam itself, used to characterize the nanoparticles, interacts with the electrolyte. X-ray absorption by water, and possibly the sulfuric acid, produce radicals that can effectively modify the local electrochemical potential. During the nano-focused diffraction experiments this was accounted for by using the lowest photon flux possible. However, a model can be used to estimate the photo-generation of various reactants, their inter-reactant dynamics, and their spatial diffusion. This model based on Bjorling et al. [1] is expanded to include the x-ray effects on the sulfuric acid. Our results confirm the findings of Bjorling et al. that there is a complicated interplay between the photo-production of reactants and their corresponding reactions, and that the diffusion process partially compensates for the accumulation of reactants. However, the inclusion of x-ray effects on sulfuric acid could be critical if one wishes to calculate the local electrochemical potential.

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[1] Björling, A., Marcal, L. A. B., Arán-Ais, R. M. & Solla-Gullón, J. *Chemical limits on X-ray nanobeam studies in water*. (2023) doi:[10.26434/chemrxiv-2023-mf0p2](https://doi.org/10.26434/chemrxiv-2023-mf0p2).