## **Abstract**

Hydrogen technologies present a promising alternative to internal combustion engines in the transportation sector and serve as a viable option for stationary power generation. Water electrolysis produces hydrogen, which can later be used in a fuel cell to generate clean electricity.

The commercialization of hydrogen technologies faces challenges due to the high costs of proton exchange membrane water electrolyzers and fuel cells, which rely on expensive noble metals like platinum and iridium. Advanced functional nanomaterials with supported metal-based nanoparticles can be utilized to reduce these costs while maintaining good catalytic properties. The structural characteristics of an electrocatalyst significantly influence its catalytic activity and stability. Therefore, it is crucial to gain a deeper understanding of how its structure forms and evolves to interpret measurements and plan future syntheses.

This dissertation examines the diversity of crystal structures in electrocatalysts that contain platinum or iridium-based nanoparticles on high-surface-area supports. It aims to clarify several specific aspects of the structure-property relationships of the materials studied. A key focus of this work is to develop a reliable methodology for analyzing experimental data.

First, we examine the presence of anti-phase boundaries in carbon-supported PtCu<sub>3</sub> nanoparticles. In the second part, we use 4D-STEM to determine and track the local crystal structure of carbon-supported PtCu<sub>3</sub> nanoparticles through identical location imaging. Then, we track and quantify individual degradation mechanisms using STEM images of a Pt-Co/C electrocatalyst. Lastly, we examine how metallic nanoparticles on ceramic support are influenced by metal-support interactions.

**Keywords**: electrocatalysis, nanoparticles, structure-property relationship, electron microscopy, data analysis