

# Review Report on PhD Thesis

Faculty: **Central European Institute of Technology  
Brno University of Technology in Brno**

Academic year: **2023/2024**

Student: **Bilal Bawab**

Doctoral study program: **Advanced Materials and Nanosciences**

Field of study: **Advanced materials**

Supervisor: **Dr. Ing. Jan Macák**

Reviewer: **Prof. Dr. Kornelius Nielsch**

**PhD thesis title:** Supported nanostructured Pd catalyst using atomic layer deposition: from methanol oxidation to hydrogen evolution reaction

## Topicality of doctoral thesis:

The conformal coatings of 3D nanostructures and high surface area materials for electrochemical energy conversion processes are highly demanded for the development of green energy technologies. Both example processes mentioned and selected in the thesis, hydrogen evolution reaction (HER) and methanol oxidation reaction (MOR), are very timely research topics and need further attention for performance improvement. The selection of saleable material synthesis approaches is ideal for technology transfer and the realization of future technologies.

## Meeting the goals set:

The PhD candidate, Mr. Bilal Bawab, has selected two template systems with high surface areas. He has systematically studied the coating of Pd with different ALD cycle numbers and the impact on these two different electrochemical conversion processes. For both processes (HER and MOR) he has managed to obtain a significant performance improvement. The analysis of the structural properties of these modified porous materials with Pd coating has been conducted with a wide set of methods. Especially, a very detailed electrochemical characterization has been performed for an extended period of time.

### **Problem solving and dissertation results:**

The candidate has developed two novel material systems for both electrochemical conversion processes based on electrocatalysis. The selection of the ALD processes for the successful coating with Palladium was properly done and yielded a mostly metallic coating. Partially, Mr. Bawab, has explained the different nucleation and growth behaviors of the ALD of crystalline Pd versus the ALD of an amorphous metal oxide layer. What remains unclear is how the nucleation density of Pd clusters and nanoparticles can be controlled and how the underlying substrate can be modified to optimize the nucleation. In this context, studies of the Pd growth by ALD on planar substrates with different metal oxides and metal coatings could provide a deeper understanding.

### **Importance for practice or development of the discipline:**

This thesis will have an important impact in the areas of materials science, electrochemistry and chemical engineering. This research work demonstrated performance improvement for the HER and MOR processes, but also shows results of the performance over time. Performance studies over very extended periods of time (as in weeks and months) will need to be performed in future. The approach is saleable and can be easily adjusted to other template systems or powder coating by ALD.

### **Formal adjustment of the thesis and language level:**

The English writing seems to be okay but should maybe be checked by a native speaker before the final publication of the thesis. The appendix sections in chapters two and three can maybe be dissolved and all-important images should be integrated in the main text. Maybe an appendix for the whole work at the end of the thesis should be considered.

### **Questions and comments:**

Q1: Explain in detail how the nucleation and growth behaviors of noble metals by ALD is different from the conventional ALD growth of metal oxides?

Q2: For the ALD growth on the porous TiO<sub>2</sub> templates, did you find evidence that the Pd growth is impacted by impurities from the electrochemical formation of the porous TiO<sub>2</sub>/Titania structure and its electrolyte?

Q3: The palladium precursor is very expensive and the efficiency of the ALD coating on the porous template is limited. How can the efficiency of the ALD coating be improved and is recycling the precursor after the ALD chamber possible?

Q4: What are the next characterization steps of real long-term conversation studies and the integration for application to reaching higher TRL levels?

## **Conclusion**

The PhD thesis is very well structured and has resulted in two first-author publications in well-known journal in the field of electrochemistry. Furthermore, he has been involved in the publication of another publication as a co-author and has presented a number of conference contributions. In general, the PhD thesis is written on a high scientific level and needs very minor improvements. In my opinion, the reviewed thesis fulfills all requirements posed on theses for obtaining a PhD degree. This thesis is ready to be defended orally, in front of the respective committee.

In Dresden, date April 20, 2024

**Prof. Kornelius Nielsch**