

# **IPID4all Doctorate Research Exchange with the institute of physical chemistry in Warsaw**

## **Feedback report**

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*Exchange Period:  
05.02. – 04.03.17*

*Host Supervisor:  
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*Exchange topic:  
Nanoscale pH-mapping above nanostructural metallic catalysts*

### **Introduction**

The topic of my PhD studies is the imaging of various electrochemically active materials by using the scanning electrochemical cell microscopy (SECCM) and other related techniques. In order to do that I need to build a new setup for high resolution imaging and to develop special probes, which are sensitive for different properties of the sample. The group of Prof. Dr. Marcin Opałło for example developed a pH sensitive probe based on carbon nanoelectrodes (CNE). These probes are prepared by pyrolytic carbon deposition on the inside of a sharply pulled quartz capillary. Just by immersing the electrode into a solution of syringaldazine, this compound adsorbs to the electrode surface and the electrode becomes pH sensitive. This probe was used to image the pH distribution at different electrodes during the oxygen reduction reaction.

### **Research undertaken**

During my time in Warsaw we tried to expand the existing technique for pH-mapping with simultaneous topography mapping to ensure a constant distance to the sample and to assign the measured pH to different sites of the catalytic material. In Order to do so, we prepared probes from dual barrel quartz capillaries by laser pulling and we reached tip sizes of about 200 nm promising high possible resolutions in the imaging. Afterwards we deposited the pyrolytic carbon only in one of the two channels. The other one was filled with the electrolyte solution. With these probes we were able to perform scanning electrochemical microscopy (SECM) imaging for pH mapping on the carbon part and scanning ion conductance microscopy (SICM) imaging on the electrolyte part. The latter imaging technique is able to measure the probe to sample distance and can be applied for maintaining a constant distance and for topography imaging.

After probe preparation we performed a lot of test scans to improve the performance of our SICM measurements and encountered an intrinsic difficulty of the technique. The signal change in SICM between a distance far away from the sample and a position close to it is usually about 1 to 3% of the overall signal. This makes maintaining of a constant distance difficult. To solve this issue we extended the setup by a lock-in-amplifier and induced a small vibration to the probe. This changed the signal quality drastically to changes of about 70% during approach.

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### **Personal Experience**

From my personal experience it is always interesting and helpful in science to talk to lots of different people about plans, problems and ideas. This definitely accounts for my time in Warsaw, too. In my first week I had the opportunity to give a talk about my PhD topic and the setup I'm going to build and I got a lot of feedback and ideas for improvement. They also reminded me of problems I might encounter which I didn't think about so far. On the other hand I heard a lot of interesting talks from various research topics in the group seminar and in the additional PhD Seminar of the institute and I learned a lot about a wider field of physical chemistry.

Besides the scientific part of my exchange I had an interesting time in Warsaw and an informative insight into polish history and culture. Especially the old city center left a lasting impression in my memory.

### **Conclusions**

With this exchange IPID4All gave me a great opportunity to expand my horizon in the field of physical chemistry and to establish contacts with our polish coworkers. I learned a lot about the fabrication of carbon nanoelectrodes and scanning ion conductance microscopy. Both techniques are crucial for my future PhD studies and I will definitely benefit from the knowledge.

### **Outlook**

In the future we plan to build a setup for carbon nanoelectrode fabrication in our labs in Oldenburg and to use the dual function probes for combined SECM-SICM imaging.

In August we will meet the group of Prof. Dr. Marcin Opałło at their workshop on SECM techniques.

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