Operationalization of Localized Electrochemical Impedance Spectroscopy

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Background

Localized electrochemical impedance spectroscopy (LEIS) is a technique that utilizes a scanning probe to measure electrochemical impedance down to the nanometer level. Using the same principles as electrochemical impedance spectroscopy (EIS), LEIS obtains the data for impedance by measuring the function of applied frequency. EIS provides a more well-rounded perspective compared to other electrochemical techniques as it gathers data not only from the specimen but also the surrounding electrolyte. This allows for the understanding of the changes on the surface of the material as well as its effect on the electrolyte solution it is exposed to. The changes in the specimen and the solution can be quantified to observe their relationship. The LEIS technique utilizes a bi-electrode robe to measure the current flowing in an electrolyte above the working electrode, also the specimen being studied. Figure 1 shows the basic LEIS set-up.

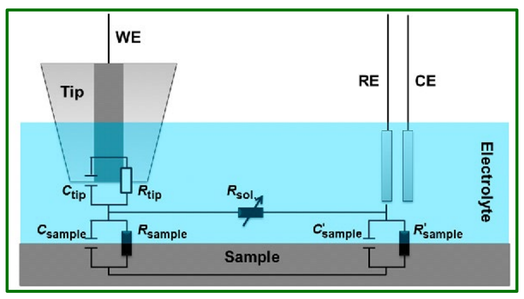

Figure 1: A Typical LEIS Set-Up

Being able to observe changes at a localized level is important as it provides a better understanding on how the microstructure of a material can affect its overall corrosion behavior. This is important for understanding various surface phenomena like phases, defects, precipitations, etc. whose properties are different from the base material. These differences in properties can influence the overall corrosion behavior of the material, and it is important to understand the effects of these various components to ensure that any detriment that can lead to corrosion is inhibited.

Objective

To operationalize the localized electrochemical impedance spectroscopy equipment and technique by writing a standard operating procedure (SOP) and developing an experimental design for prospective students.

Procedure

Literature Review

Existing literature on the LEIS technique was explored to gain a better understanding on its fundamentals as well as its potential applications. It is ensured that the literature cited showed connection to the technique and application, significant contribution to the field of materials science and engineering, and credibility. The compiled literature during this experiment will be passed on to prospective students who are potentially interested in working on this technique for their research project.

Writing the Standard Operating Procedure

To utilize the technique and equipment safely and effectively, a detailed SOP must be written first. An SOP consists of instructions how to assemble and set up the equipment, utilize the technique for experimentations, and troubleshoot if any problems arise. It also includes a job hazard analysis (JHA) that lists possible risks the user might encounter upon using the equipment and how it can be prevented. The completion of an SOP ensures that prospective students who want to utilize the technique and the equipment for their projects will feel prepared to operate it efficiently and safely.

Designing an Experimental Plan

Based on the literature review conducted, experimental plans are proposed as a suggestion for prospective students to consider. If laboratory access was permitted during the school year, one of the proposed experimental plans would be conducted in order to verify the validity of the SOP written and the quality of the equipment. A complete experimental plan consists of the required training that need to be completed before operating the equipment, the materials necessary to conduct the research, the methodology alongside the standards that need to be followed, and the analysis of results. This would ensure successful planning and execution of the experiment.

Figure 2: Versa Scan LEIS Equipment

Results and Discussion

Critical Issues

Due to the unexpected continued effects of COVID-19, this project had to be adapted to a virtual format. Laboratory access was prohibited, and there came a challenge of writing an SOP for an equipment that has not been seen or operated in-person. The challenge becomes greater with the limited laboratory access given to the graduate students. There had been plans to attempt to train how to operate the equipment virtually with a graduate student showing the students how the equipment looks like and how operation is conducted, but like all forms of education these days, the training had to adapt constantly according to the available resources.

Adapting to the Virtual Format

Despite these unfortunate circumstance, one cannot deny how fortunate the research group is for having a strong support network already established for future generations of students. Steps had to be taken to adapt to a virtual format, but the transition had been smooth for the most part. ZOOM meetings were held excessively to ensure constant communication was maintained throughout the process. Virtual lectures were prepared to relay the fundamentals of the topics of interest and literature sources were shared among the group to ensure that everybody is aware of any useful information and new developments on the topic. Laboratory work and experimentation have always been considered the backbone of engineering, but these times reminded everyone of the importance of collaborative effort and community in a research environment.

Deliverables

The Fall Semester was spent conducting literature review on LEIS and potential research interests. A comprehensive literature review had been compiled to ensure that the theory and application were fully understood. The compiled literature review will become the basis of a comprehensive report that will also be passed on to future generation of prospective students to ensure that they, too, will develop a well-rounded understanding of the topic.

The Spring Semester was spent writing the SOP using the knowledge gained during the Fall Semester as well as the virtual lectures from the graduate students. The effectiveness of the SOP in relaying instructions and ensuring safety is being reviewed by graduate students who have laboratory access and is currently being edited accordingly. The goal of the SOP is to be as clear and concise as possible to ensure that future experimentalists will be able to follow as smooth as possible.

Conclusions

Due to the lack of laboratory access, experimentation work was not able to be done. However, a standard operating procedure has been prepared for the next generation of prospective students. The remaining step that needs to be done is to prepare an experimental plan that lists potential project ideas with the different types of materials and corrosive environments given for a specified application.

The effectiveness of the SOP will be tested once laboratory access is more available to students. In the meantime, prospective students will be handed the comprehensive literature review not only as a form of training but also to receive feedback. The important aspect of this project is to ensure that the knowledge and interest on the technique and the equipment are passed on to continue obtaining data and bridging the gap in understanding the field of materials science and engineering.

Because of COVID-19, research as was understood and done in the past was forced to adapt to a more virtual and more collaborative environment.

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