VARIABLE TEMPERATURE ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY

Implementing an Experimental Setup for Future Science

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INTRODUCTION AND ABSTRACT

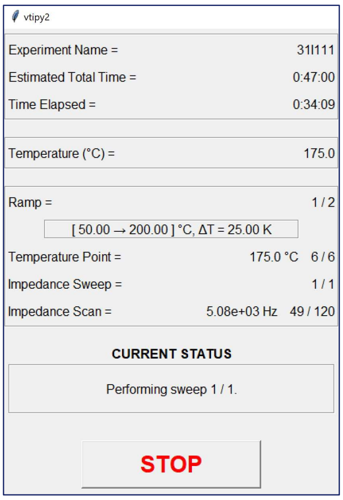
Electrochemical Impedance Spectroscopy (EIS) is the process of measuring the complex impedance of a sample over a spectrum of A.C frequencies. Data from EIS can be used to model the properties of the sample in the interest of use in batteries [1]. By utilizing this technique over a range of temperatures, other interests can be observed and measured. For example, activation energies and ionic conductivities can be measured following a temperature (Arrhenius) dependency, or crystalline phase changes can be observed occurring through temperature ranges [2].

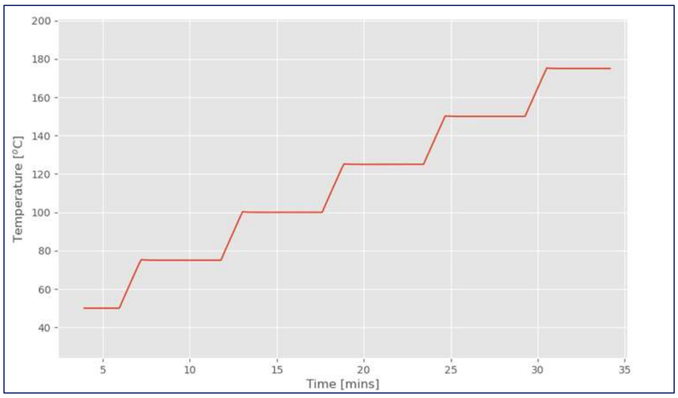
This work aims to abstract away hardware and software complexities to be able orchestrate a time-efficient experimental setup, used to perform variable temperature EIS. To validate the setup, a demonstration experiment was carried out.

SOFTWARE

To automate the hardware and hence experiments, a software control system was needed. In the interest of development speed and future adaptability, Python was used to leverage a range of Python, C and C++ libraries.

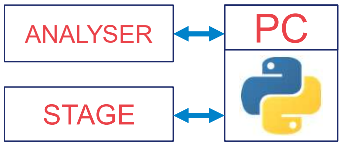
Given experiment parameters, the software automates hardware functionality and data collection via a range of communication protocols. Additionally, it displays live temperature data (below) and an informative dashboard of the current state of the experiment (right). The software also takes interest in safety measures and contains features to debug hardware and software issues – especially useful for future and external use.





HARDWARE

To perform variable temperature EIS, a method to heat and cool a sample is needed alongside means to measure the impedance. To this end, impedance analysers from *BioLogic* and *Solartron Analytical* were used in conjunction with temperature-controlled stages from *Linkam Scientific*. These recently developed state-of-the-art stages dramatically reduce experiment time when compared to typical EIS equipment, being box furnaces with slow heat transfer capability and large internal volumes.



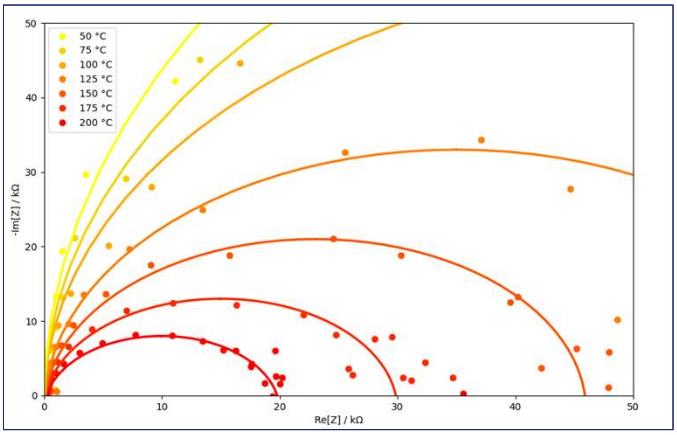
TESTING A SAMPLE

To evaluate the functionality and performance of the hardware and the software controlling it, an example measurement was carried out. Specifically, variable temperature EIS was carried on a Bismuth Oxide thin film sample grown on a Niobium doped $\mathrm{SrTiO_3}$ substrate. The sample was measured at temperatures from 50 °C to 200 °C in intervals of 25 K, with frequencies ranging from 0.01 to 10 Hz.

To analyse the data, semi-circles were fit to the expected semi-circular impedance profiles for the set of temperatures. Using this data to fit to the Arrhenius equation, an activation energy of $E_a = 0.25 \pm 0.02$ eV was found for the sample. This compares with a literature value of $E_a = 0.27 \pm 0.05$ [3].

There is a justification that doubt could be cast on this result, due to noisy data at low temperatures that could arise from a small known hardware issue, since addressed. Nonetheless, this result provides an argument for the case that the setup is behaving as intended, so therefore should be viewed as a success.

It is hoped that this demonstration will validate a wide range of impactful science soon to come.



CONCLUSION AND IMPACT

This project delivered a time-efficient control system for performing variable temperature EI, which has reduced the time to take these measurements down from the order of 100s of hours, to hours.

The software is soon to be published on GitHub for anyone in the scientific community to adapt and make use of in the future.

More immediately, it is hoped that this work will expedite the discovery of new electrodes Li-ion batteries and new solid-state electrolytes for solid-state batteries; aligning closely with the theme of the *FutureCat* project by *The Faraday Institution*.

REFERENCES

[1] Orazem, M.E. and Tribollet, B., (2008). Electrochemical impedance spectroscopy. *New Jersey*.

[2] Hodkinson, J. (2019). A study of the synthesis, structure, and ionic conductivity of sodium and lithium lanthanide pyrosilicates, $A_3Ln\mathrm{Si_2O_7}$. (Doctoral Thesis)

[3] Drożdż, Ewa and Kolezynski, Andrzej. (2017). The structure, electrical properties and chemical stability of porous Nb-doped $\mathrm{SrTiO_3}$– experimental and theoretical studies. RSC Adv.. 7.

INTERN BIO

Adam Alderton is studying for an MPhys Physics degree at the University of Exeter and has ambition for a career in research, in both academia and industry, in the field of semiconductor engineering or within the wider electrical engineering discipline.

This ambition stems from an interest in condensed matter theory, the optical properties of materials and the interplay of science and engineering for innovation.

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