Utilising Electrochemical Impedance Spectroscopy on Thin Film Energy Materials

Developing techniques to analyse and assess future electrochemical devices

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ABSTRACT AND MOTIVATION

Electrochemical Impedance Spectroscopy (EIS) is a powerful technique used to observe and analyse electrochemical processes within materials or systems. In stable systems, it can enable the understanding of the key electrochemical mechanisms occurring at an interface, making it widely applicable in assessing the capabilities of many battery and fuel cell components [1].

Fuel cells are particularly efficient and energy dense, and there is a specific interest in solid oxide fuel cells (SOFC). However, SOFC performance is often limited by the cathode [2], benefitting greatly from analysis via EIS.

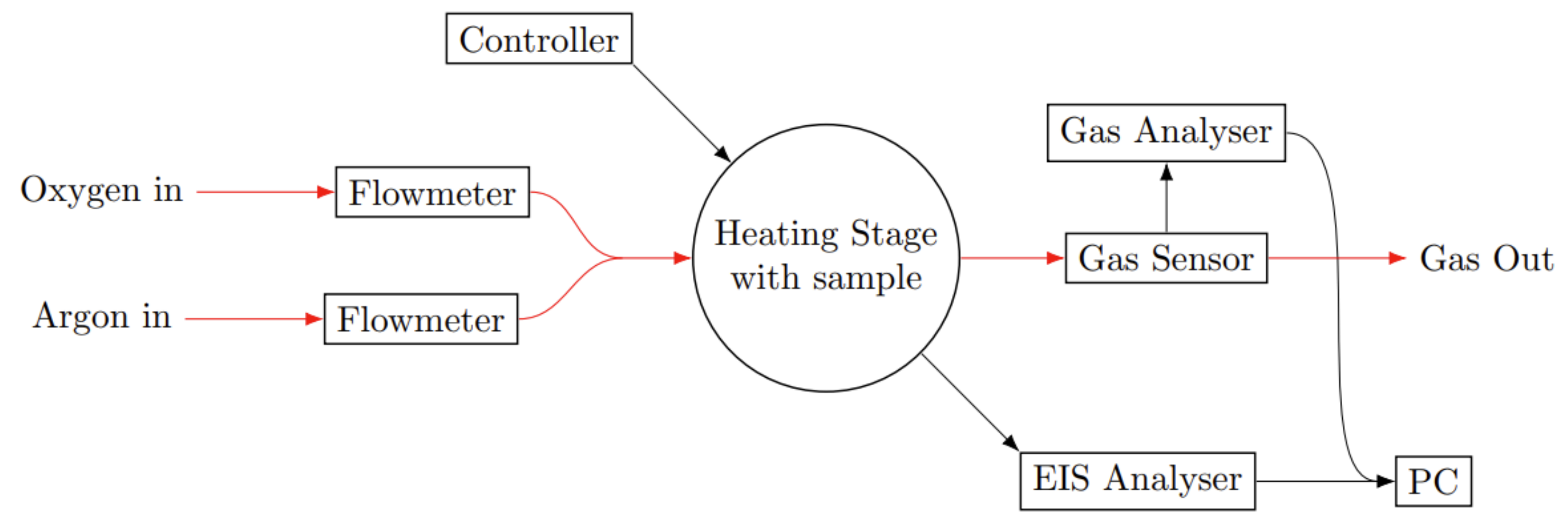
It is evident from the literature [3] that despite being widely important and reported, the nature of temperature dependent EIS measurements is often inconsistent. Challenges arise in the time-consuming process of ramping the temperature and making stable measurements manually. Also of significant importance for conduction is the effect on oxygen partial pressures [4].

The aim of this work is therefore to automate EIS experiments, varying these key parameters, vastly improving the ease of obtaining high quality data.

EQUIPMENT AND SETUP

Crucially, the setup relied on modern heating stages – the *Linkam HFS350* and *TS1000* – and their relative controllers to vastly improve the efficiency of heating. An impedance analyser, namely the *Biologic SP200*, was used to gather impedance data via the probes within the stage, which was processed on the connected lab PC.

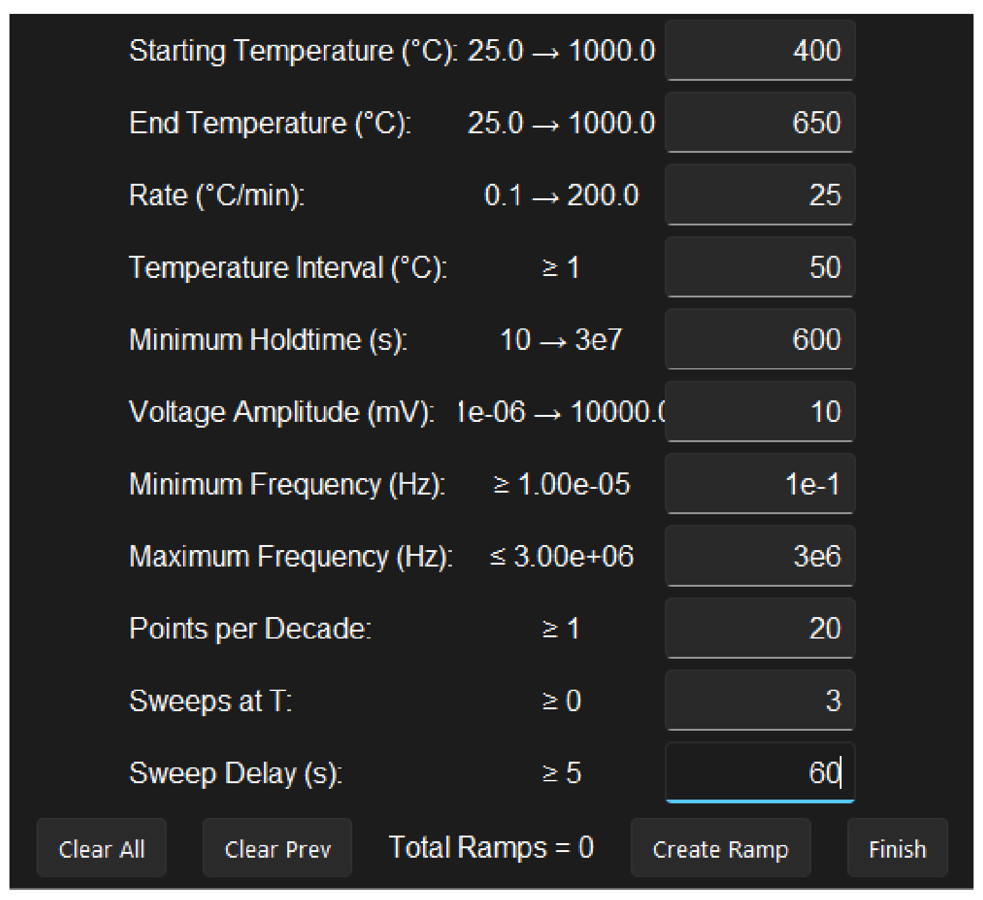
Simultaneously, atmospheric control over the sample was achieved via the use of oxygen and argon pumped into the stage through variable flowmeters under a fume hood. This is summarised in the diagram below, with red lines representing gas flow, whilst black lines indicate the passage of data via wires.



SOFTWARE

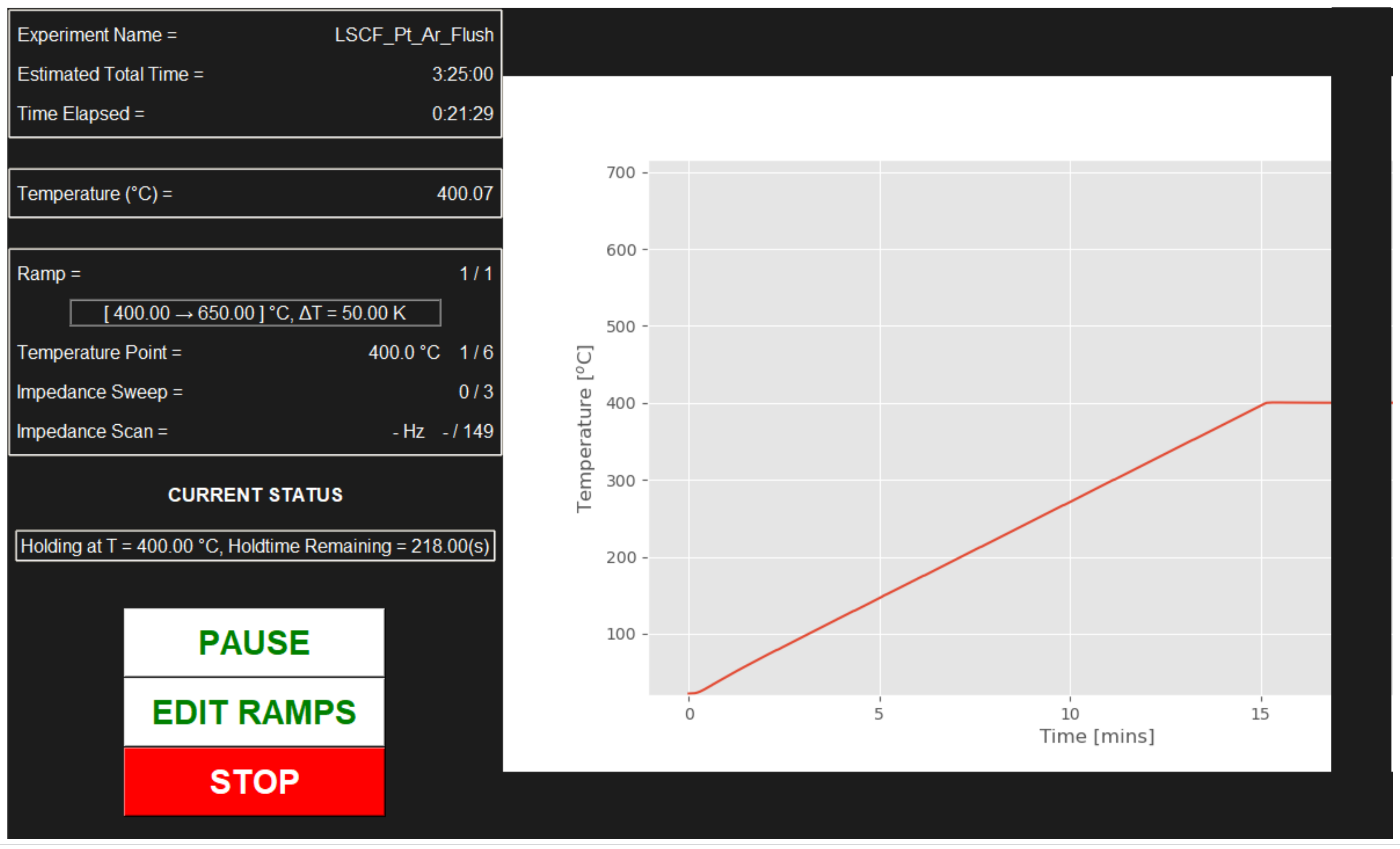
This project was centralised upon creating a graphical user interface (GUI) with which the temperature ramping and EIS sweeps could be controlled and automated. To this end, various Python scripts were used to achieve the level of functionality needed in order to aid EIS measurements.

A variety of libraries in C++ and similar utilities were provided by the hardware manufacturers for direct device communication. These were accessed and managed via the Python scripts in question in order to provide full temperature and impedance data to the GUI, and allow precise control of temperature intervals and voltage/ frequency sweeps from user inputs.



Starting Temperature (°C):	25.0 → 1000.0	400
End Temperature (°C):	25.0 → 1000.0	650
Rate (°C/min):	0.1 → 200.0	25
Temperature Interval (°C):	≥ 1	50
Minimum Holdtime (s):	10 → 3e7	600
Voltage Amplitude (mV):	1e-06 → 10000.(	10
Minimum Frequency (Hz):	≥ 1.00e-05	1e-1
Maximum Frequency (Hz):	≤ 3.00e+06	3e6
Points per Decade:	≥ 1	20
Sweeps at T:	≥ 0	3
Sweep Delay (s):	≥ 5	60

Total Ramps = 0



Experiment Name = LSCF_Pt_Ar_Flush
Estimated Total Time = 3:25:00
Time Elapsed = 0:21:29

Temperature (°C) = 400.07

Ramp = 1 / 1
[400.00 → 650.00] °C, ΔT = 50.00 K
Temperature Point = 400.0 °C 1 / 6
Impedance Sweep = 0 / 3
Impedance Scan = - Hz - / 149

CURRENT STATUS

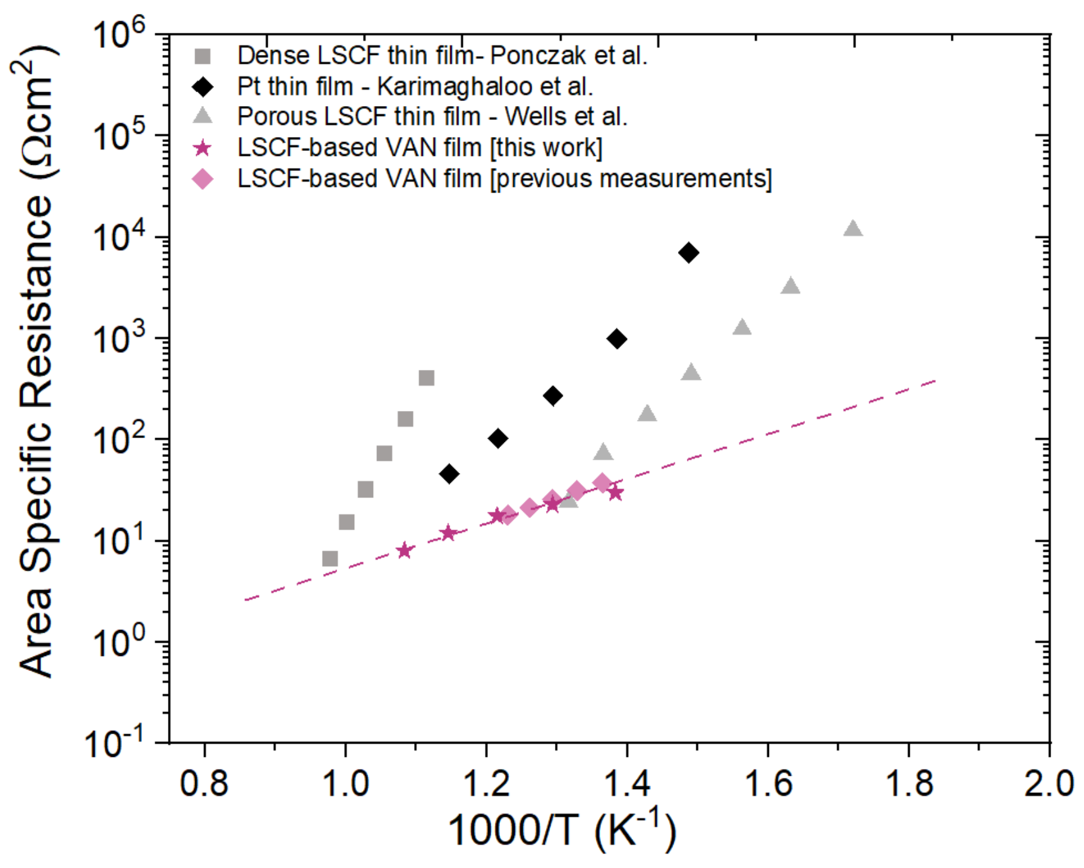
Holding at T = 400.00 °C, Holdtime Remaining = 218.00(s)

ANALYSING A SAMPLE

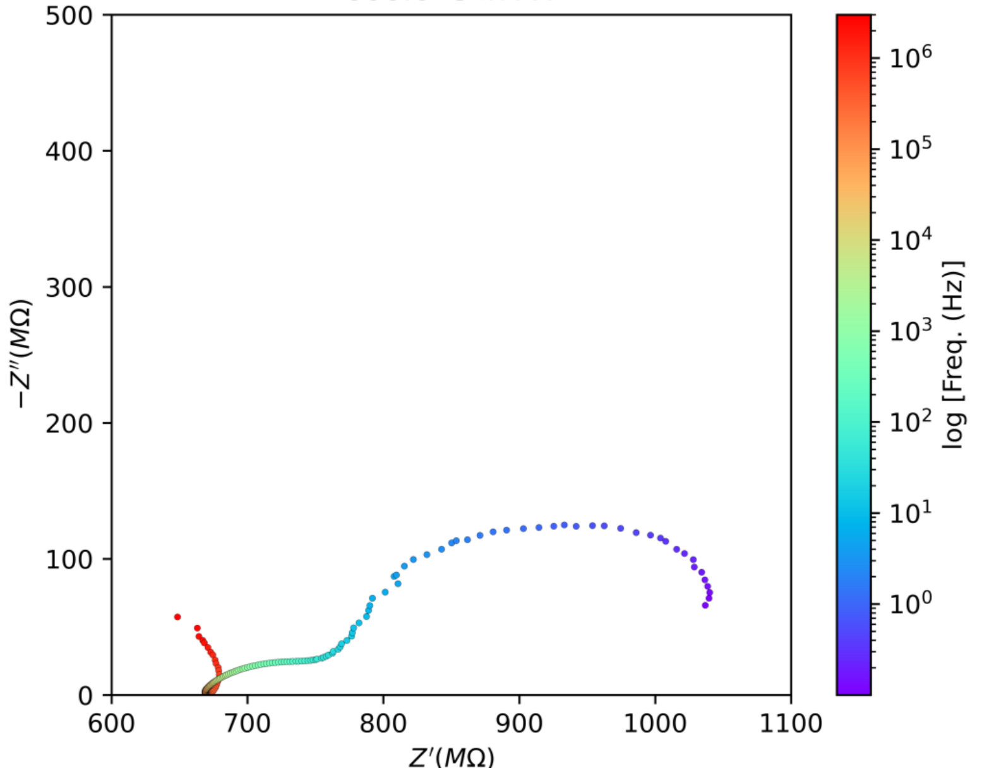
Using this approach, a novel nanocomposite thin film cathode comprising of LSCF and a secondary oxygen vacancy supplying phase was analysed at various temperatures and partial pressures. The cathode was placed upon a YSZ substrate, forming part of a hypothetical SOFC fuel cell.

As is often the case, the data below is presented via Nyquist plots, as it can easily be modelled and interpreted. Here, it is reasonable to assign the leftmost feature to the YSZ, with the larger feature on the right representing the effect of the cathode, demonstrating the limiting effect of the cell's efficiency. Taking this and plotting the area specific resistance (shown top left) for the cathode only, we can see that at lower, and therefore more realistic, temperatures, this sample outperforms current leading materials in the field, agreeing with previous measurements from within the department [5,6,7].

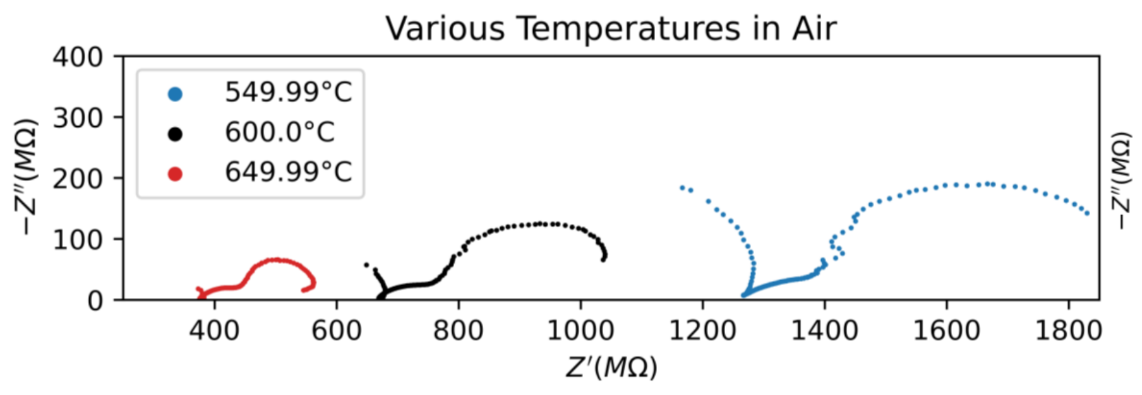
Also shown are the noticeable effects of temperature and atmosphere.



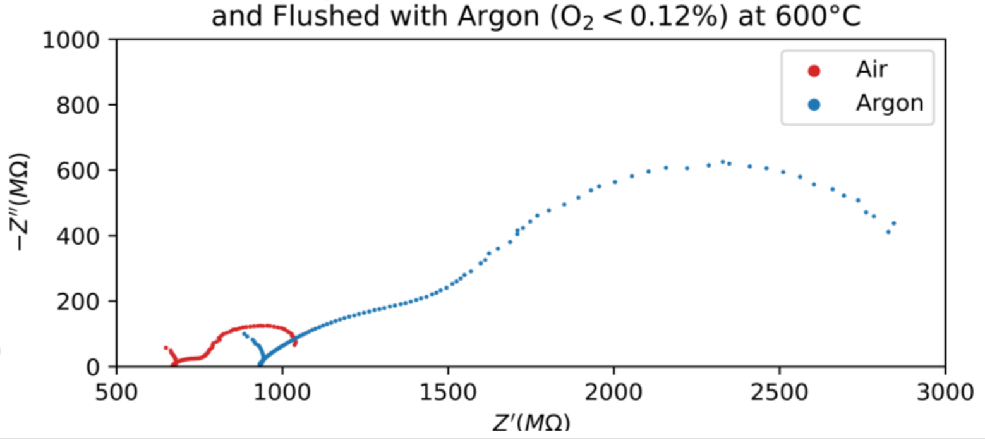
600.0°C in Air



Various Temperatures in Air



Comparison between the sample in Air and Flushed with Argon ($O_2 < 0.12\%$) at 600°C



CONCLUSION, IMPACT AND NEXT STEPS

From the sample analysis, it has been shown that this setup can successfully perform under various conditions. It appears that the data produced are accurate and possess the ability to highlight key advances in the field.

In total, the automated scripts ran for over 6 hours, demonstrating their vast utility. Manually, this process would have been less efficient and incredibly time consuming. It is hoped that this setup will greatly aid future research.

Further improvement could be attained from the introduction of electronic flowmeters, integrated into the Python script, for more advanced and efficient automation.

LINK TO CODE ON GITHUB

SCAN ME

REFERENCES

[1] Wang, S. *et al. Nat Rev Methods Primers* **1**, 41 (2021).
[2] Liu, Z., *et al. Journal of Energy Chemistry, 22*(4), pp.555-559 (2013).
[3] Deleebeeck, L. *et al. International Journal of Energy Research, 44*(9), pp. 7158-7182 (2020).
[4] Mueller, M.P. *et al. Journal of Applied Physics* **129**, 025104 (2021).
[5] Plonczak, P. *et al. Journal of The Electrochemical Society* **159**, B471–B482 (2012).
[6] Karimaghaloo, A. *et al. ACS Omega* **2**, (2017).
[7] Wells, M.P. *et al. ACS applied materials & interfaces, 13*(3), pp.4117-4125 (2021).

INTERN BIO

Arun Atwal is studying Natural Sciences, specialising in Physics, at the University of Cambridge. With an interest in multidisciplinary research, especially at the interface between Physics and Materials Science, this project has been ideal in his ambition to explore the connection between research and potential implementations.

Specific areas of interest also include computational and condensed matter physics, particularly in theory and modelling, but with an awareness of practical applications.

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