StoRIES

Storage Research Infrastructure Eco-System

2nd IREP Call Documentation - User

StoRIES IREP1 Post Research Questionnaire (with satisfaction survey): User (s)

After the StoRIES International Research Exchange Programme stay, users are required to submit a post research questionnaire. This should be done within 4 weeks after the Access is completed unless otherwise agreed. The Post Research Questionnaire form will be given to the User(s) by the Project Management. The report contains sections related to the work performed, the main results and observations that were achieved.

[redacted]

Summary questionnaire for users who have been granted International Research Exchange Programme (IREP) under the StoRIES project Horizon 2020 IREP scheme.

General information about the project	
Project title (as used in application)	Electrode Lifecycle Enhancement through Computational Testing and Research Automation
Project acronym (max 15 characters)	ELECTRA
StoRIES IREP RI(s) accessed	*University of Toronto, Canada*
Keywords (up to ten, free text)	Catalyst development, lab automation, high throughput, data science, Bayesian optimization
Arrival date (in town where RI located)	29.09.2024
Departure date (from town where RI located)	07.11.2024
Starting date of access (first day at RI)	30.09.2024
Finishing date of access (last day at RI)	06.11.2024
Number of days not using the RI (during the above period)	11

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Reason for not using RI those days (describe)	Weekends + 1 public holiday
Number of days using the RI	27
Number of users granted access (group size)	1
Comments	
User	
User group leader or sole applicant (user group member 1)	
First name	[redacted]
Last name	
Affiliation / Employer	Fraunhofer IFAM Dresden
Country of Employer	Germany
E-mail	[redacted]
Comments	
User group member 2	
First name	
Last name	
Affiliation / Employer	
Country of Employer	
E-mail	
Comments	
User group member 3	
First name	
Last name	
Affiliation / Employer	
Country of Employer	
E-mail	
Comments	
User group member 4	
First name	
Last name	
Affiliation / Employer	

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Country of Employer	
E-mail	
Comments	
Please insert more fields if your groups had more than four members.	

Access Summary Report - work performed and initial results

Brief description of the objectives of your project (up to 200 words)

The development of catalysts for alkaline electrolysis (AEL), such as Raney-Nickel electrodes, is a time-consuming task which involves a lot of manual work and a large number of long experiments. To speed up the electrochemical experiments, this project aimed to explore both, lab automation techniques and machine learning. Lab automation is intended to decrease the time needed for the setup of one experiment. Machine Learning, for which the approach of Bayesian Optimization was used, is meant to reduce the measurement time, which is needed to characterize a certain catalyst material.

All in all, this project aims to reduce the effort of the characterization of one material regarding its suitability as a catalyst for alkaline electrolysis.

Activities performed (up to 600 words)

Lab automation – custom electrochemical cell

A custom, 3D printable electrochemical cell, in which material characterization in three electrode setup can be performed, was designed and printed. The cell is optimized for being low cost, allowing quick and automated setup of experiments and having a high degree of reproducibility. To develop this design, a team of three persons worked together: A technical designer, Sterling Baird and myself. In a public github issue, all relevant aspects of the first design were discussed and test results were shared. During the time of this project, 3 cell designs and 2 designs for the sample holder were printed. This way, numerous improvements could be implemented within the prototyping stage at high development speed.

The prints were carried out with two Stereolithography printers owned by the Acceleration Consortium and two printers owned by the National Research Council (NRC).

The material compatibility of the resin from which this cell and sample holder were printed, was tested within this project for the "clear v4" and "rigid 10k" resin provided by Formlabs. Cubes with a volume of 1cm³ each were printed of both materials and were exposed to concentrated KOH solution (30w%). These tests were carried out both, at room temperature and at 80°C. The mass of the test cubes was measured before, during and after the exposure to KOH solution to evaluate any mass loss due to degradation within KOH solution.

Machine Learning for electrolysis life cycle testing

A script was developed for creating an accelerated stress test protocol. By running multiple experiments with the same material with stress parameters selected by the Bayesian Optimization algorithm, the "worst" parameters for a given material are intended to be found. This way a

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protocol can be established, which is known to stress the electrode to the maximum so that within a short experiment the performance of a given material under maximal stress can be evaluated. To create this script, the search space was defined by specifying the parameters and their constraints. Once this was completed, the honegumi tool along with the Bayesian Optimization library "ax" were used to create the actual optimization script. The Bayesian Optimization script for developing this test protocol was completely written during this project. The actual experiments remain to be run in the labs of IFAM Dresden. If this approach turns out to be successful, it could greatly reduce the time for long term stability tests.

<u>Course – Autonomous Sytems for Discovery</u>
During the period of this project, I completed the first course of the "Autonomous Systems for Discovery" training program, which is created and maintained by the Acceleration Consortium. Within this course, I learned the basics of Bayesian Optimization, Device communication of a self-driving lab using the MQTT protocol and learned how to use MicroPython to set up a Rasperry Pi Pico W as an orchestrato. Especially the part of learning how to use MicroPython for laboratory applications was useful as it integrates perfectly into the next steps of automating the complete workflow of experiments which use the above-described electrochemical cell.

<u>Lab Setup</u>
The labs at the NRC are brand new and excellently built. As they were mostly empty when I arrived, part of my work was to set up the training lab to prepare it for electrochemical experiments.

Scientific results (up to 800 words)

<u>Lab automation – custom electrochemical cell</u>
Tests in 3 electrode setup for the characterization of a material as a catalyst for AEL need to be carried out in a certain electrolyte solution. This electrolyte needs to be exchanged between all experiments to provide the same conditions at the beginning of each experiment. The cell design developed allows to automate this step completely. The electrolyte can be removed with a pump via an inlet into the cell and a subsequent washing procedure can be applied via an integrated washing nozzle in the inside of the cell which is connected to external water supply. The water of this cleaning step can once again be removed with the previously described pump. Next, fresh electrolyte can be filled into the cell through another inlet.
Temperature control is implemented through double wall design, which allows an externally heated thermal fluid to be flowing between the inner and outer wall, thereby heating the electrolyte. Supply of inert gas into the electrolyte to purge solved oxygen is realised through a gas inlet tube which is connected to the cells tap. A Harber-Luggin-Capillary is also integrated into the tap, so that a HydroFlex Reversibel Hydrogen Electrode (Gaskatel) can be easily inserted into the setup.
The sample holder, into which the tested material is mounted was designed from scratch to optimize it for the given requirements. These are: Electrical contact between the sample and the external Potentiostat, no contact between any cable/wire and the electrolyte and mechanically reliable mounting of the sample. The design created in this project fulfils all these requirements by combining 3D printed parts with several seals. The sample holder is the only part, which needs to be removed between experiments. Thanks to it being printable with an SLA printer, multiple sample holders can be easily printed, so that it's easy to prepare several samples at once to reduce downtime between experiments.

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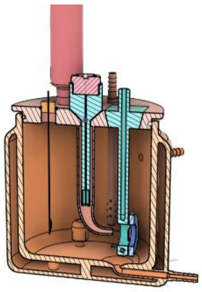
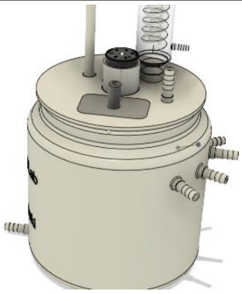

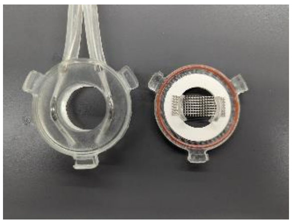

<u>Machine learning for electrolysis life cycle testing</u>

The general idea of this approach is to stress a sample during a fixed period of time under discontinuous conditions as it is known that rapid changes in load can contribute to the altering of an electrode material. The time over which a specific current density is applied effects the material as well as the changes in current density. E.g. forcing the Hydrogen Evolution Reaction to occur at the electrodes surface with a current density $j=0.5A/\text{-}cm^2$ for 2 hours will stress the electrode material differently than running the HER with $j=0.4A/cm^2$ for 1h and subsequently with $j=0.6A/cm^2$ for 1h, even though the net amount of produced hydrogen is the same. It is of interest to have a test protocol which is known to stress the electrode material significantly in a short period of time, which involves numerous load changes. However, developing such a protocol would be a highly time consuming task as it would involve running a great number of possible test protocols if the number of cycles and their corresponding current density would be tested. That's why Bayesian Optimization for the development of such a protocol was chosen.

The developed optimization protocol follows the following general structure:

1. ***Preconditioning** (1h) to get the sample into a constant electrochemical state*
2. ***Stress phase** (12h)*
3. ***Characterization phase** (ca. 2h)*

The stress phase will consist of a maximum of n cycles with varying time and current densities each. Current densities are allowed between -2 A and 0A, where 0A are effectively applied as OCP. The time of each cycle can be between 1s and the maximum stress phase time of 12h. The exact number of cycles, corresponding time intervals and current densities are chosen by the Bayesian Optimization Algorithm.

The characterization phase will consist of three steps, which analyse the electrochemical behaviour of the electrode material after the stress phase. These steps contain a galvanostatic step with $-0.5A/cm^2$ for 30 min, a Tafel-Analysis with measurements between 0 and $-1A/cm^2$ and a cyclovoltammetric step for the measurement of the double layer capacity. The results of this analysis phase are hence the overpotential, Tafel-slope, exchange current density and double layer capacity. These results will then be reported back to the optimization algorithm, which in turn will calculate the stress parameters of the next test protocol.

<u>Course – Autonomous Systems for Discovery</u>

The first course of the "Autonomous Systems for Discovery" educational program was successfully completed and the corresponding certificate was achieved. Feedback about the concept of the course and ideas for some improvements were also reported.

Interpretation of the results (up to 400 words)

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Lab automation – custom electrochemical cell

It was possible to print a prototype of the custom designed electrochemical cell which proves the general applicability of SDL printing to this task. Also, the cell fulfilled all the specified requirements such as in- and outlet of fluids and gases, mechanical stability and precision as well as water/gas tightness. Therefore, the selected approach of redesigning standard electrochemical cells is considered to be generally successful. A first test with a standard Nickel mesh as working electrode showed the expected results, which again confirms the general functionality of the built setup. Next, a direct comparison of the proven test setup at Fraunhofer IFAM and the custom design will be carried out to compare the electrochemical data obtained from both setups at the same conditions. If the results of these measurements match closely and show only the expected deviations the custom setup can be considered suitable for the specified task.

The material compatibility tests described above showed that there was no change in mass or shape for the cube printed from clear v4 resin over almost two weeks in 30w% KOH at room temperature. Only a slight mass loss could be detected for the same material at 80°C, which makes further analysis at this temperature necessary. At IFAM Dresden ICP-OES measurements of the electrolyte before and after a 24h experiment at 80°C will be done to detect any degradation issues of the clear v4 resin, if they exist.

Cubes printed from rigid 10k resin showed mass loss already over several hours both at room temperature and at 80°C. This material is therefore not considered suitable for the given application.

Once the design if fully optimized and proven to work as expected, it will be a great chance for research groups all around the globe to get access to a cheap but fully functional 3 electrode setup cell. This is especially relevant for groups in countries with little public funding. E.g. this is the case for a collaboration partner at the University of Cordoba, Argentina, who is already interested in the cell design.

Machine Learning for electrolysis life cycle testing

There are no actual results yet, as the work focused on the preparation of the Bayesian Optimization script. However, the developed approach is likely to be well implemented as it was discussed in detail with several experts in the field of Bayesian Optimization

Main achievements during the IREP related work (up to 250 words)

- *Prototype of a fully equipped and automatable custom electrochemical cell*
- *Many lessons learned from the design process of the custom cell. This experience is highly valuable for the next iterations of the design and is already speeding up the development significantly*
- *Script for the development of an accelerated stress testing protocol for electrochemical analysis of electrode materials in a 3-electrode setup*
- *Successful completion of a course from the Acceleration Consortiums educational program*
- *Many discussions with scientists form University of Toronto, the Acceleration Consortium and the National Research Council. Several ideas for collaborations have already grown from these encounters.*

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Difficulties during the IREP related work (up to 250 words)
One of the SLA printers of the Acceleration consortium had technical issues which we were not able to solve. This delayed the first print of the electrochemical cell, as it was the Acceleration Consortiums only printer, which can create such a large structure. Help was offered by scientists at the National Research Council, where the same printer was available. There we could successfully print two versions of the cell.
Intended publications
If the custom cell proves to be a reliable tool for the intended electrochemical measurements and several further improvements are incorporated, a publication about this design is planned. As the whole design is already publicly available and will be released under an open-source license once finished nothing stands against a publication with test results and further explanation. If more automation features of this setup, like controlling the pumps with a microcontroller, automating the sample exchange, etc. are implemented, these will also be published.
Data management
All data created within this project is available on github within the AccelerationConsortium/ac-training-lab repository. Data about the electrochemical tests in the custom cell will be published in the corresponding issues, too. *Custom electrochemical cell (v1): https://github.com/AccelerationConsortium/ac-training-lab/issues/62#issuecomment-2462873229* *Custom electrochemical cell (v2): https://github.com/AccelerationConsortium/ac-training-lab/issues/104#issuecomment-2474112589* *Machine learning for life cycle testing: https://github.com/AccelerationConsortium/ac-training-lab/issues/98#issuecomment-2452563216*
Conclusions / additional comments
The StoRIES Project is a great way to organize a research stay abroad relatively easy with great outcome. For me, it was a beautiful opportunity to stay in Toronto for six weeks during which my time was dedicated a hundred percent to learning new skills. This is something, which often falls too short in the daily business at home.
Did you complete the EC user questionnaire available at https://ec.europa.eu/eusurvey/runner/RIsurveyUSERS ? (necessary)
☒ *Yes* ☐ *No*
Feedback – HSE, Ethics and Satisfaction

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Please rate on a scale from 1 (excellent) to 5 (poor). Feel free to provide additional comments					
Practical information on how to apply for IREP and the overall application process	1 (excellent)	2	3 (neutral)	4	5 (poor)
	☒	☐	☐	☐	☐
Information provided, once your project was accepted, on how to proceed	1 (excellent)	2	3 (neutral)	4	5 (poor)
	☒	☐	☐	☐	☐
Support received at the site(s) regarding technical/scientific matters and logistics	Have you got sufficient support from the RI staff during the project? If not, please, specify the problems. ☒ *Yes* ☐ *No*				
RI extension / upgrades required	In your opinion, is the RI needed to be upgraded? If yes, please give an explanation. ☐ *Yes* ☒ *No*				
Problems with local regulations	Have you had any problems with regulations of the visited RI owner (HSE, lab working hours, etc.)? If yes, please, specify ☐ *Yes* ☒ *No*				
Not exactly a problem but the amount of bureaucracy which I was faced in the process of getting access to the labs of the National Research Council was quite extensive and time consuming. I believe that the efficiency of this process could be improved.					
Health and safety issues	Did you encounter any health or safety issue during your research? Please provide details. ☐ *Yes* ☒ *No*				
Environment & Ethics	Did your research involve the use of elements that may cause harm to the environment, to animals or plants? Please provide details. ☒ *Yes* ☐ *No*				
Potassium hydroxide (KOH) was used as electrolyte for experiments about alkaline electrolysis and can harm the environment if not discarded properly. All precautions were taken and no actual harm is to be expected.					
Environment & Ethics	Did your research deal with endangered fauna and/or flora and/or protected areas? Please provide details. ☐ *Yes* ☒ *No*				

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Environment & Ethics	Did your research involve the use of elements that may cause harm to humans, including research staff? Please provide details. ☒ *Yes* ☐ *No*
Concentrated potassium hydroxide solution (30w%) is highly basic and can cause serious harm to skin and eyes if in direct contact. All necessary safety precautions were taken (gloves, goggles, face shield, fumehood) and no hazards have occurred.	
Environment & Ethics – Dual use	Does your research have the potential for military applications? Please provide details. ☐ *Yes* ☒ *No*
Environment & Ethics – Misuse	Does your research have the potential for malevolent /criminal/terrorist abuse? Please provide details. ☐ *Yes* ☒ *No*
Environmental issues	Were any potentially dangerous substances (materials / gases etc.) released into the environment (atmosphere, water, or land)? Please provide details. ☐ *Yes* ☒ *No*
Ethics issues	Are there any other ethics issues that should be taken into consideration? Please specify ☐ *Yes* ☒ *No*
Overall impression of communication and interaction after finishing your IREP related work	1 (excellent): ☒ / 2: ☐ / 3 (neutral): ☐ / 4: ☐ / 5 (poor): ☐
Suggestions for other international and EU facilities not included in StoRIES which you would use for your research	
None so far.	
Suggestions how StoRIES can improve future IREP programme, how to make the IREP more impactful on the energy storage technologies and how to enable the achievement of high TRL levels.	
More advertisement directly in the institutes, e.g. via the Fraunhofer newsletter or within specific research groups at universities.	

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A meeting of alumni of this program could be a good event to connect young researchers and to joint the different experiences they collected during their research journeys.	

Feedback – Pro-active Innovation Support

Awareness	Did you know about the pro-active innovation support of StoRIES. ☐ Yes ☒ No
I'm not sure, what the pro-active innovation support is	
Personal experience	Have you taken advantage of or benefited from the pro-active innovation support? ☐ Yes ☒ No

Information/service provided by the pro-active innovation support?	1 (excellent)	2	3 (neutral)	4	5 (poor)
	☐	☐	☐	☐	☐

I declare that the above provided information and especially that information on the number of days visited the RI is correct.

☒ *I have read the [StoRIES privacy policy](#) for participation in the StoRIES Accesses and consent to participation and the associated data processing.*

[redacted]

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