Ref. Ares(2020)7214663 - 30/11/2020

Cost-effective PROton Exchange MEmbrane WaTer Electrolyser for Efficient and Sustainable Power-to-H2 Technology

Grant No. 862253

***Start date**: 01.04.2020 – **Duration**: 36 months*
***Project Coordinator**: Daniel García-Sánchez - DLR*

D3.2 1st document of available testing protocols for electrolysers: additional needs and needs for PROMET

WP3 Bipolar Plates and Porous Transport Layers

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This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 862253

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Status: FD

(D: Draft, FD: Final Draft, F: Final)

Dissemination level: PU

(PU: Public, CO: Confidential,
only for Consortium members (including the Commission Services))

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 862253.

Document history

Version Number	Date of issue	Author(s)	Brief description of changes
01	13.11.2020	S. Stiber	First draft
02	18.11.2020	D.Garcia Sanchez	Revision
03	18.11.2020	Sh. Meital	Revision
04	26.11.2020	S. Siracusano	Revision
05	30.11.2020	A. Gago	Revision

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Executive Summary

Deliverable D3.2 “1st document of available testing protocols for electrolysers: additional needs and needs for PROMET-H2” will act as a pivot task for the definition and overseeing of the update of the needed harmonisation of test protocols throughout the project, from single cell to system. An overall review of the currently available harmonised testing protocols was collected and aspects that will be of application for the project purposes are identified. Specific publications at EU level, as part of the “Validated Methods, Reference Methods and Measurements” reports by JRC covering single cell testing, polarization curves and EIS of low temperature electrolysis cells. These existing protocols were analysed and adapted to the PROMET-H2 project, specifically in cases where the harmonised protocols are still under definition, and to include parameters, aspects directly linked with the novelties that are developed in the project.

1 Introduction

Harmonised test protocols, which are drawn up by a group consisting of a significant number of participants, are often based on compromises with regard to the different requirements of different programmes carried out. It is obvious that while such general protocols are extremely useful in ensuring fair comparison of results and performance across different organisations and programmes, they are also useful in ensuring that there is a high degree of flexibility in the use of such protocols. However, the specifications of a particular project may not be fully considered.
Therefore, the test protocols developed for this report have been based on those developed under the framework contract between the Joint Research Centre (JRC) and the Fuel Cells and Hydrogen 2 Joint Undertaking (FCH2JU) and further developed through annual rolling plans. These JRC harmonised test protocols have been established with the aim of defining protocols and tests to be used for the assessment of electrolysis equipment against the defined Key Performance Indicators (KPIs) described in the FCH JU (HARMONISED EU TEST PROTOCOL FOR WATER ELECTROLYSE APPLICA-TIONS) multi-annual work plan. The test protocols presented in this document have therefore been adapted to the operating conditions specified for the PROMET-H2 project.

The presented testing protocols will:

- facilitate the assessment of the single-cell performance and of the short stack/s, under specified operating conditions
- provide preliminary pre-normative results that can lead to recommended practices for the concept and the development of market-oriented specifications and pre-standards

The development of the Testing Protocol is based on partners' in-house testing protocols and is in accordance to the FCH JU harmonised testing procedures developed by the JRC in collaboration with European industry and researchers.

2 Existing standardized measurement protocols

2.1 Polarization Curves and Electrochemical Impedance Spectroscopy (EIS)

The JRC and FCH2JU published harmonised test methods for low temperature water electrolysis to perform polarization curves and electrochemical impedance spectroscopy.[1] The procedure is developed to characterize single cells and stacks at a specified operating condition for PEMWE, AWE and AEMWE.

A polarization curve plot either voltage U [V] or power density P_d [W cm^{-2}] vs. the current I [A] or current density j [A cm^{-2}] of the cell or stack. It provides information about cell / -stack processes:

1. Reaction kinetics
2. Ohmic losses
3. Mass transport limitation

The performance of the tested cell / stack is measured from the lowest to highest current density (ascending curve) followed by measuring the performance from highest to lowest current density (descending curve).
Testing protocols should begin and end with a polarization curve to compare the performance at beginning of the test (BoT) and end of test (EoT) in order to assess the degradation.[1]
For PEMWE the EU Harmonised Polarisation Curve Test Method for low temperature foresees the following steps shown in Table 1.

Table 1. Polarization curve set points for PEM electrolyser cell up to 2 Acm^{-2} proposed by JRC[1]

set point	j / Acm^{-2}	dwell time / s
1	0.001	30
2	0.005	30
3	0.01	30
4	0.025	30
5	0.05	30
6	0.075	30
7	0.1	30
8	0.15	30
9	0.2	30
10	0.25	30
11	0.3	30
12	0.35	30
13	0.4	30
14	0.45	30
15	0.5	30

[1] JRC, EU Harmonised Polarisation Curve Test Method for Low Temperature Water Electrolysis, European Commission, 2018.

16	0.6	30
17	0.7	30
18	0.8	30
19	0.9	30
20	1	30
21	1.1	30
22	1.2	30
23	1.3	30
24	1.4	30
25	1.5	30
26	1.6	30
27	1.7	30
28	1.75	30
29	1.9	30
30	2	30

Figure 1 show an example for polarisation curve recorded with a PEM electrolyser cell and explaining the contribution of each overpotential.

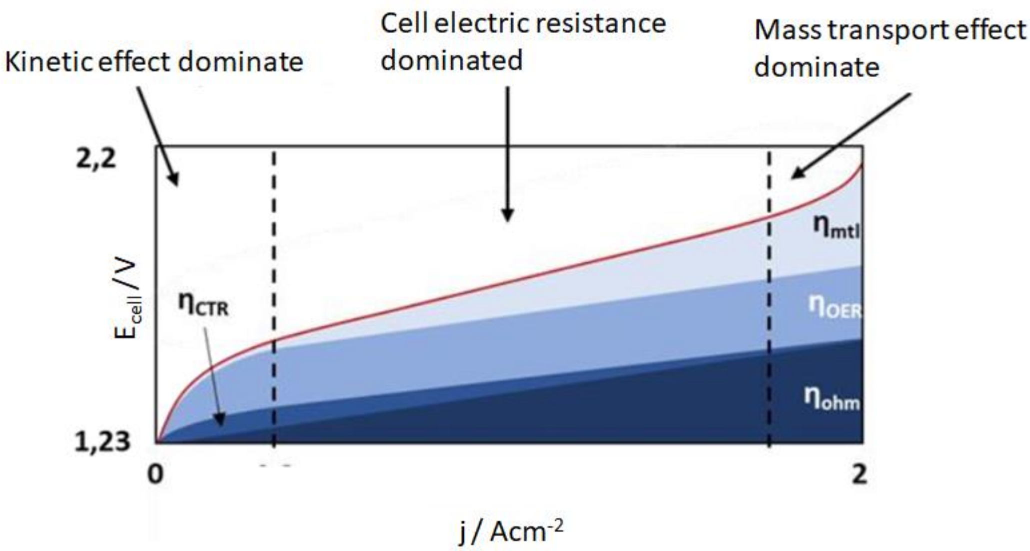

Figure 1. Overpotential for a typical PEM electrolyser polarization curve.

As a non-destructive measurement electrochemical impedance spectroscopy (EIS) provides information during operation on performance and cell degradation associated with the particular components not just in PEMWE. There are two different measurement mode:

1. Galvanostatic: the frequency dependence of impedance Z of a cell is measured by applying an alternating current (AC) as a perturbation signal while measuring the alternating voltage response
2. Potentiostatic: the frequency dependence of impedance Z of a cell is measured by applying an alternating voltage while measuring the AC response

The frequency range over several decades identifies the electrochemical and transport processes taking place in the water electrolyse cell over different time scales. This tool is used to optimize the structure of MEAs and to quantify changes in the parameters of element representatives of the cell components in an electrical equivalent circuit model to simulate cell impedances.[2]

Figure 2 presents a) an exemplary Nyquist plot for a PEM electrolyser cell and b) the electrical equivalent circuit diagram for simulation. This equivalent circuit consists of a serial connection of the following elements: (i) an inductivity L for the cables in the setup, (ii) an ohmic resistance R_{Ω} mainly dominated by the electrolyte resistance (membrane), (iii) RC element (parallel element of an ohmic resistance R_{HER} and a capacitor CPE_{HER} and (iv) RC element (parallel element of an ohmic resistance R_{OER} and a capacitor CPE_{OER} for the polarization reaction, and (v) an RNC element for mass transport.

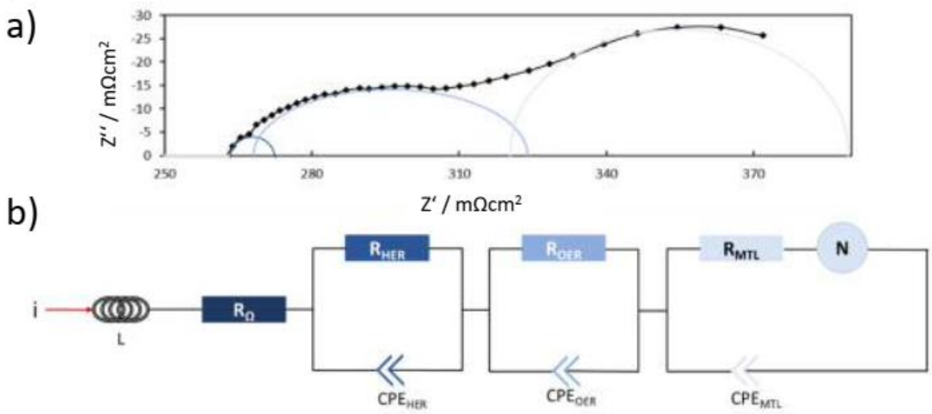

Figure 2. Exemplary of a) EIS measurement in the Nyquist diagram; b) electrical equivalent circuit diagram for simulation

2.2 Accelerated Stress Test (AST)

Stack component development for commercial application of these innovations is currently very challenging because durability needs to be demonstrated for > 50,000 h. Therefore, it is clear that accelerated stress tests (AST) are urgently needed based on a deep understanding of degradation mechanisms.
The stressors of the particular application need to be identified first, in order to develop ASTs that reflect their influence on the tested components. Currently, the main stressors are[3]:

1. High current density (> 1 A cm^{-2})
2. Dynamic operation
3. Shutdown process

Based on the two AST protocols proposed by P. Aßmann et al. with the goal to correlate the degradation rates R1 and R3 under non-stressing conditions with the degradation rates R2 and R4 under stressing conditions with high current densities and dynamic operation which are plotted in Figure 3. Thereby it is important to ensure that the AST is linked to real operation scenarios, including:

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[2] JRC, EU Harmonised test procedure: electrochemical impedance spectroscopy for water electrolysis cells, European Commission, 2018.

[3] Aßmann, P., Gago, A. S., Gazdzicki, P., Friedrich, K. A. & Wark, M. Toward developing accelerated stress tests for proton exchange membrane electrolyzers. Current Opinion in Electrochem., 21, 225-233 (2020). https://doi.org/10.1016/j.coelec.2020.02.024.

i. Nominal current density operation
ii. High current density operation
iii. Load cycling
iv. OCV or shut-down process

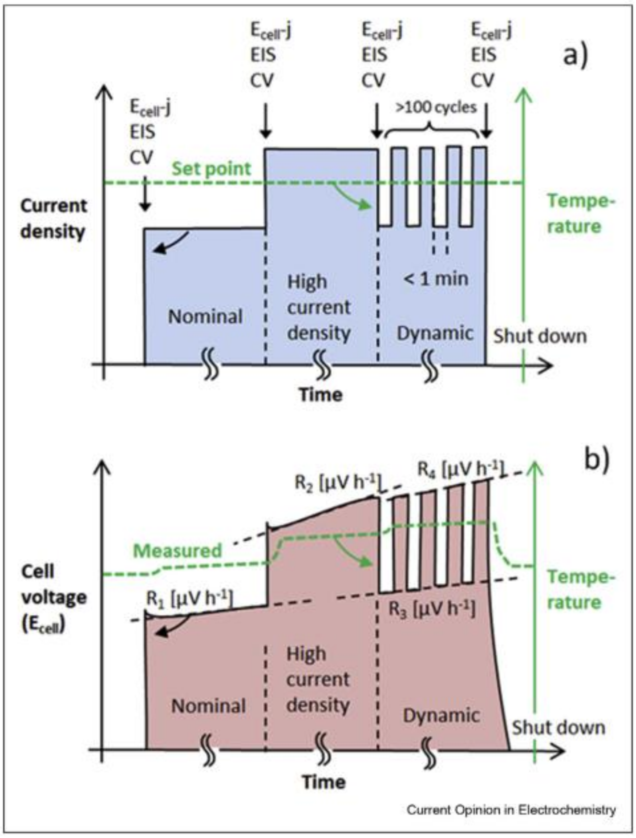


Figure 3. Proposed AST: (a) current density vs. time profile for accelerating degradation including all the main stressors PEMWE and the expected (b) E_{cell} profile with degradation rates (R).[4]

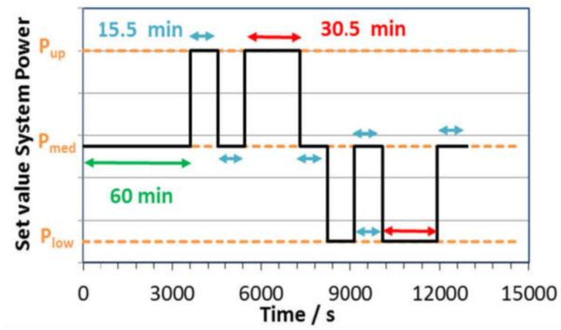
2.3 Grid Balancing

The EU funded project QualyGridS had the objective to establish standardized testing protocols for electrolysers to perform electricity grid services. The QualyGridS protocols developed will be applied to both alkaline and PEM electrolyzers systems, respectively, using electrolyser sizes from 50 kW to 300 kW.

A dynamic operation protocol studies performance, response time and degradation under operating conditions. The chosen testing protocol is an evaluation of the electrolyzer performance to frequency control reverse (FCR) service grid and its steps are illustrated in Figure 4.

[4] Aßmann, P., Gago, A. S., Gazdzicki, P., Friedrich, K. A. & Wark, M. Toward developing accelerated stress tests for proton exchange membrane electrolyzers. Current Opinion in Electrochem., 21, 225-233 (2020). https://doi.org/10.1016/j.coelec.2020.02.024.

This selected protocol could be also tested within PROMET-H2 project to show the ability of the components for their dynamic operability.



Ramp duration	t_{full}	18 sec	≤ 30 sec
Stability: maximum deviation	Δ_{max}	30 kW	≤ 5%* (P_{med}-P_{low})= 26.2 kW
Initial response time	t_{init}	2±1 sec	≤ 1.5 sec
with P_{low}= 444 kW P_{med}= 968 kW P_{up}= 1492 kW			

Figure 4. Frequency containment reserve (FCR) – symmetric service with fast variations[5]

2.4 Protocols from other EU projects

Furthermore, EU-funded projects (i.e. PRETZEL, NEPTUNE) developed harmonised protocols to test electrolyzer components operated in a PEM cell under extreme conditions of current density, temperature and pressure. These test protocols are also addressed to the assessment of performance, efficiency and durability of a PEM water electrolyser (PEMWE) stack operating up to 8 A cm^{-2}, 140 °C and 100 bar.[6,7]

[5] EU-project QualyGridS, https://www.qualygrids.eu/
[6] EU-project NEPTUNE, https://cordis.europa.eu/project/id/779540/results/de
[7] EU-project PRETZEL, https://cordis.europa.eu/project/id/779478/de

3 Additional needs and needs for PROMET-H2

3.1 Break-In Procedure

In terms of the break-in procedure of the MEA the manufacturer instructions are normally followed. Therefore, it is recommended to operate the cell / stack for a certain time to ensure the MEA(s) have reached a steady state. The cell / stack current density should be kept at 0.1 A cm^{-2} for 5 minutes to ensure the catalysts are in the proper oxidation state. By setting lowest current value in steps of 25 mA cm^{-2} per minute the aforementioned steady state conditions should be reached. Keeping the cell / stack at these conditions for one hour, the variation in cell voltage at the lowest current should not be more than 1%.

4 Conclusion

For project PROMET-H2 already existing protocols on performing polarization curves and impedance spectroscopy developed by JRC were selected as the baseline for the measurements required in the project. These are the basic characterization tools for evaluating the component characteristics at single cell and stack level. To be able to validate the stability and durability of components without excessive and long testing procedures an AST was proposed as well. The 25 kW system will be operated following the described protocols to evaluate the components properties. Also, a possible protocol for gird service was selected which was already tested primarily in a laboratory cell and can be applied to the 25 kW system.