UNIVERSITÀ
DEGLI STUDI
DI TRIESTE

Dipartimento di
Ingegneria
e Architettura

# ELECTROCHEMICAL INVESTIGATION TECHNIQUES AND ACCELERATED STRESS TESTS

## FOR FUEL CELLS AND WATER ELECTROLYSERS

*Prof. Marco Bogar*

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# OUTLINE

1. Electrochemical investigation techniques
2. The polarization curve
3. Electrochemical Impedance Spectroscopy
4. Total Harmonic Distortion
5. Cyclic Voltammetry
6. Linear Sweep Voltammetry
7. Current Mapping
8. Accelerated Stress Tests for FC and WE

# BIBLIOGRAPHY

| Reference | Paragraph/Pages |
|---|---|
| PEM fuel cells diagnostic tools, Haijiang Wang, Xiao-Zi Yuan, Hui Li, (2012), CRC Press | Ch. 1, 2, 3, 4, 5 |

| Insights | |
|---|---|
| R1 | Fuel Cell Engines, Mench M. M., (2008), Wiley, chapter 4 |
| R2 | Fuel Cell Engines, Mench M. M., (2008), Wiley, chapter 9 |
| R3 | Tang Z. et al., Recent progress in the use of electrochemical impedance spectroscopy for the measurement, monitoring, diagnosis and optimization of proton exchange membrane fuel cell performance, Journal of Power Sources, 468, (2020), 228361; DOI: https://doi.org/10.1016/j.jpowsour.2020.228361 |
| R4 | Ramschak E. et al., Detection of fuel cell critical status by stack voltage analysis, Journal of Power Sources, 157, 2, (2006), 837-840; DOI: https://doi.org/10.1016/j.jpowsour.2006.01.009 |
| R5 | Thomas S. et al., Online health monitoring of a fuel cell using total harmonic distortion analysis, International Journal of Hydrogen Energy, 39, 9, (2014), 4558-4565; DOI: https://doi.org/10.1016/j.ijhydene.2013.12.180 |

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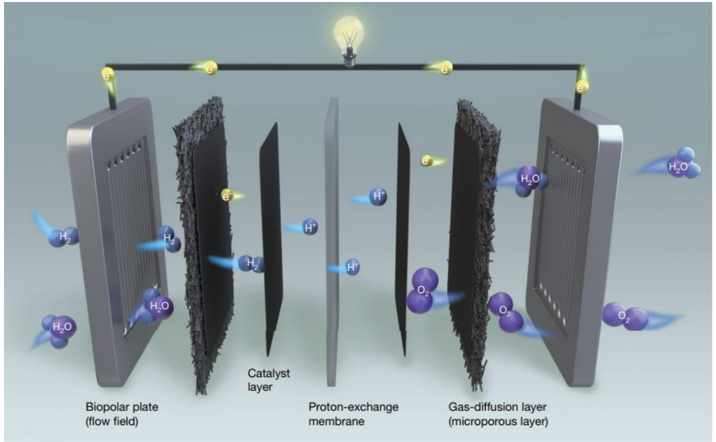
| Insights | |
|---|---|
| R6 | M. Heinzmann et al., Impedance modelling of porous electrode structures in polymer electrolyte membrane fuel cells, Journal of Power Sources, 444, (2019), 227279; DOI: https://doi.org/10.1016/j.jpowsour.2019.227279 |
| R7 | Perez L. C. et al., Segmented polymer electrolyte membrane fuel cells – a review, Renewable and Sustainable Energy Reviews, 15, (2011), 169-185; DOI: https://doi.org/10.1016/j.rser.2010.08.024 |
| R8 | Biswas I. et al., Advancement of segmented cell technology in low temperature hydrogen technologies, Energies, 13, (2020), 2301; DOI: https://doi.org/10.3390/en13092301 |
| R9 | R7. Pahon E. et al., A review of accelerated stress tests dedicated to proton exchange membrane fuel cells – Part I: Fuel cell component level, Journal of Power Sources, 546, (2022), 231895; DOI: https://doi.org/10.1016/j.jpowsour.2022.231895 |
| R10 | A review of testing procedures for PEMWE degradation, Journal of Power Sources, 557, (2023), 232569, DOI: https://doi.org/10.1016/j.jpowsour.2022.232569 |

# INTRODUCTION

Jiao K. et al., Nature, 595, (2021), 361–369



## Electrode degradation

- High temperatures
- High humidity
- High potentials
- High gas crossover
- Gas starvation

## Membrane degradation

- Mechanical degradation (stress and strain by alternating humidification temperatures)
- Chemical degradation (high temperatures and the presence of radicals)

- Is the fuel cell operating properly or are there any failures?
- Which degradation phenomena are taking place?
- How fast is degradation taking place?
- How the performances of a new material or component can be compared to the current standard?

# CLASSIFICATION OF CHARACTERIZATION TECHNIQUES

## Characterization techniques

**Ex situ**

Used to characterize some properties of individual components of the fuel cell once removed from the device in a non-operative form.

**In situ**

Used to characterize the performance of a single component of the cell in an environment with the same (or similar) characteristics found in a real device.

**In operando**

Used to characterize the performance of a single component or of a cell in real operative conditions.

## Characterization techniques

**Electrochemical**

Based on recording of the electrical parameters

**Micrographs**

Based on recording of microscopic images (using different probes)

**Optical**

Radiation at different wavelengths is used to investigate specific sample properties

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# 1. ELECTROCHEMICAL INVESTIGATION TECHNIQUES

# ELECTROCHEMICAL MEASUREMENTS

In any electrochemical experiment, the three fundamental variables to be monitored are voltage, current, and time. Moreover, the following distinction can be highlighted:

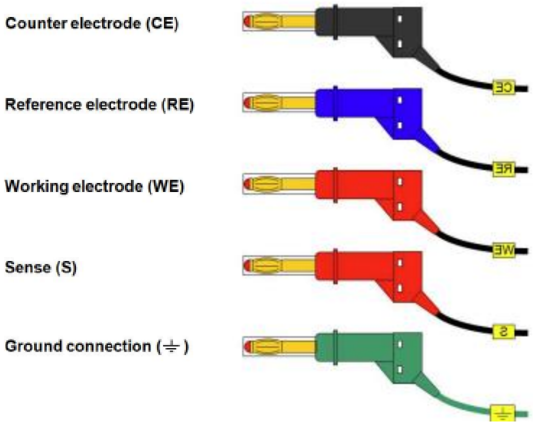
- **Voltage-controlled.** The voltage of a system is controlled by the user and the resulting current response is measured. Voltage can be either kept in steady state (*potentiostatic*, where the control voltage is constant in time) or dynamic (where the control voltage varies with time).
- **Current-controlled.** The current of a system is controlled by the user and the resulting voltage response is measured. Current can be either kept in steady state (*galvanostatic*, where the control current is constant in time) or dynamic (where the control current varies with time).

Measurements carried out in different dynamical ranges may lead to different outputs due to the fact that electrochemical kinetics could evolve differently depending on the frequency of the probe used.

# ELECTROCHEMICAL MEASUREMENTS – THE POTENTIOSTAT

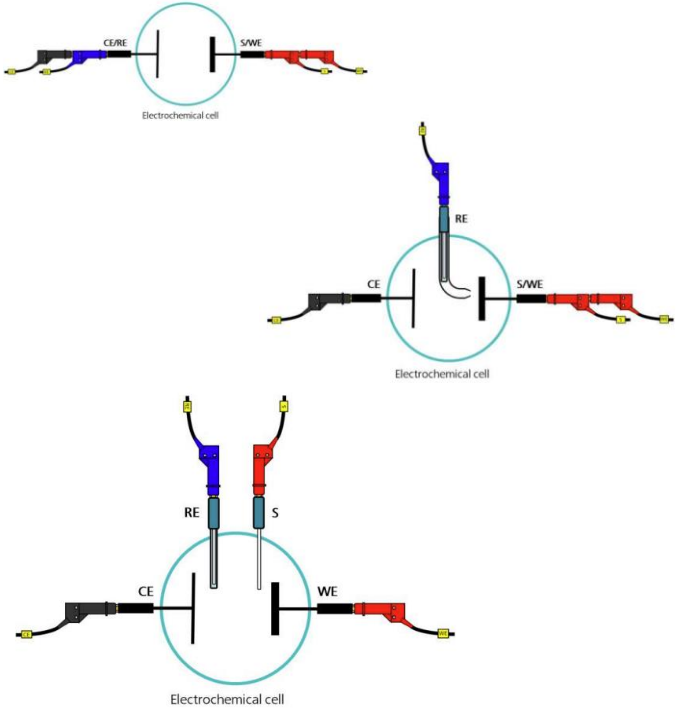
https://www.metrohm.com/it_it/prodotti/elettrochimica/modular-line.html

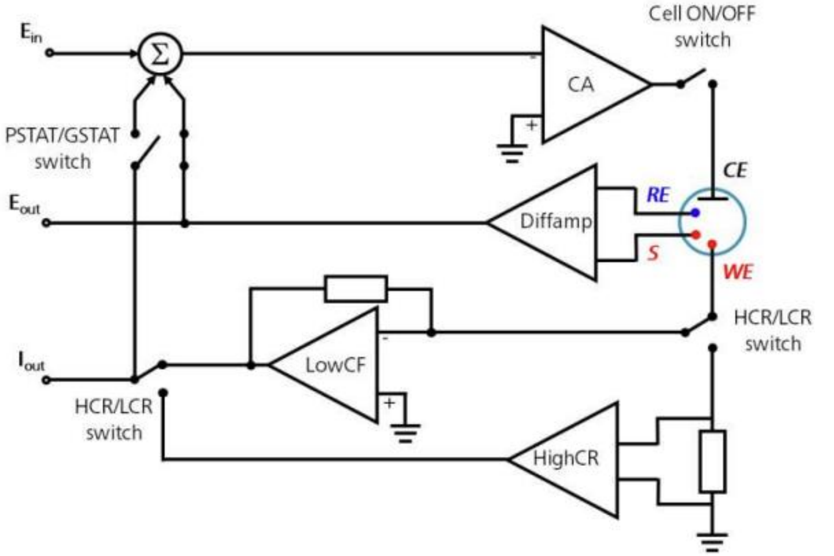
https://www.biologic.net/topics/what-is-a-potentiostat-how-potentiostats-work-and-their-use-in-science-and-industry/



Basic overview of the working principle of a potentiostat/galvanostat (PGSTAT) – Electrochemical cell setup
www.metrohm.com/en/products/electrochemistry

# ELECTROCHEMICAL MEASUREMENTS – THE POTENTIOSTAT





Basic overview of the working principle of a potentiostat/galvanostat (PGSTAT) – Electrochemical cell setup
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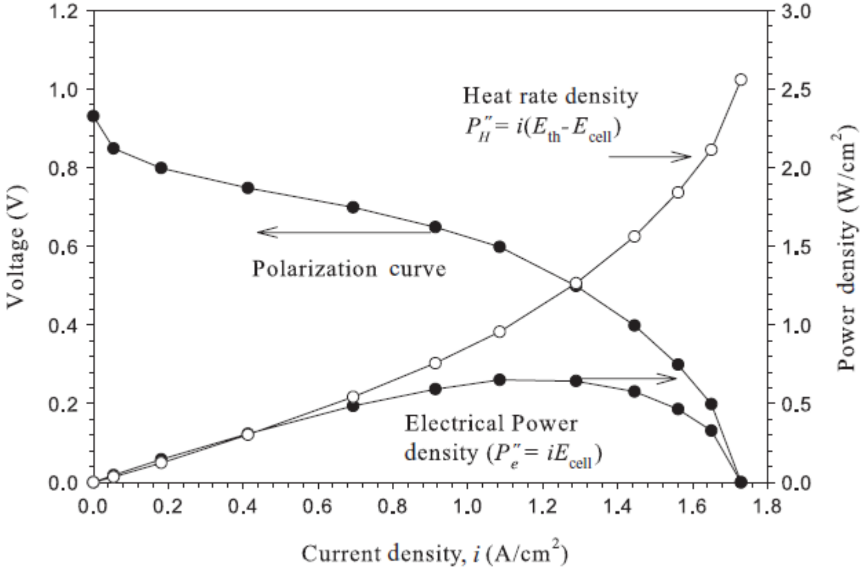
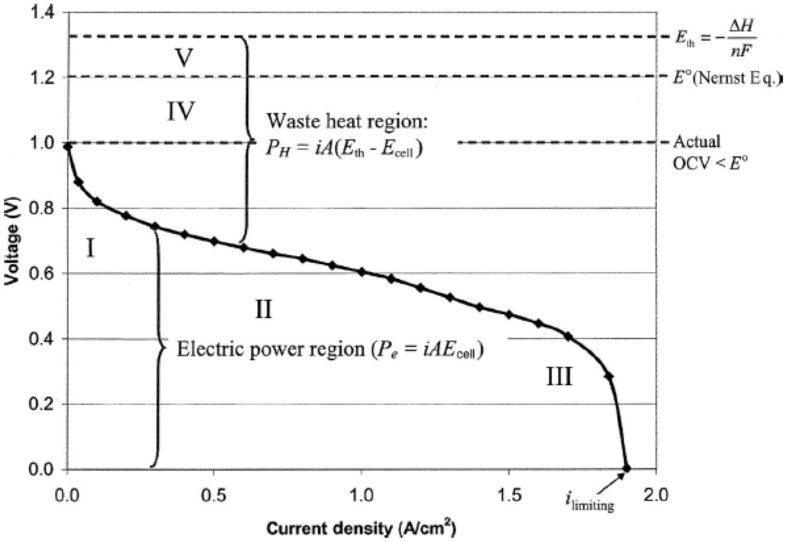
# 2. THE POLARIZATION CURVE

# POLARIZATION CURVE

The polarization curve represents the cell voltage–current relationship for a fuel cell (or electrolyser), where voltage (V) is plot versus current density (A cm$^{-2}$). It can be either expressed in terms of power density (W cm$^{-2}$).



# POLARIZATION CURVE



Thanks to the V/I curve, the main losses characterizing fuel cell operation can be identified in the different areas of the plot:

I. Activation (kinetic) overpotential at the electrodes.

II. Ohmic polarization losses (electrical and ionic conduction losses through the electrolyte, catalyst layers, interconnects, and contacts) .

III. Concentration polarization losses (mass transport).

IV. Departure from the Nernst thermodynamic equilibrium potential (undesired species crossover through the electrolyte, internal currents from electron leakage through the electrolyte, or other contamination or impurity).

V. Departure from the maximum thermal voltage, a result of entropy change.

# POLARIZATION CURVE

Polarization curves are measured after a conditioning stage in which the fuel cell is stabilised at the reference conditions. I/V can then be recorded in two ways, in steady-state (static) or in transient (dynamic) conditions. In steady-state conditions, at every new point of current density set, the corresponding voltage value is recorded once its rate of variation is below a specific threshold. In transient conditions the V/I curve is continuously recorded by setting a little slow rate.

# POLARIZATION CURVE – MODELLING

The difference of potential for a fuel cell can be expressed by accounting all of the polarization losses:

$$V = V^0(T, P) - \eta_{a,a} - \eta_{a,c} - \eta_x - \eta_\Omega - \eta_{mt,a} - \eta_{mt,c}$$

Moreover it was also show as the same equation can be written as:

$$V_c = V^0 - A\ln\left(\frac{i + i_n}{i_0}\right) - (i + i_n)r + B\ln\left(1 - \frac{i + i_n}{i_L}\right)$$

Where $V^0$ is the (reversible) open-circuit voltage, $i_n$ is the sum of equivalent current density from fuel crossover and the internal current density, A is the slope of the Tafel line, $i_o$ is a function of both exchange current densities (or the exchange-current density at the positive electrode if the overpotential is much greater than that of the negative electrode one), r is the resistance per unit area, B is the parameter in the mass-transfer overvoltage equation, and $i_L$ is the limiting current density at the electrode characterized by the lowest limiting current density.

# POLARIZATION CURVE – MODELLING

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# POLARIZATION CURVE – MODELLING

In order to understand the electrochemical behaviour of PEMFCs, several types of empirical modelling have been introduced to mimic polarization curves. In general the development of models is based on a first logarithmic term (deriving from Tafel approximation), a linear term, and a third term used to model the mass transport. In this case, different formulations were developed, some simpler, such as:

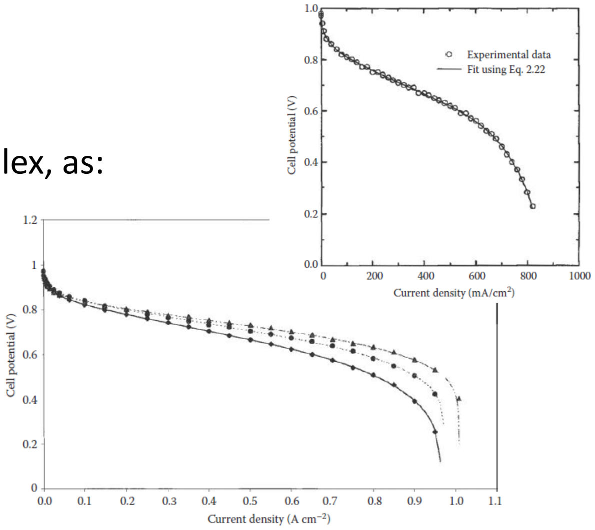
$$V_c = V + b\log(i_0) - b\log(i) - Ri - me^{\xi i}$$

Where m and ξ are used to model the mass transport limitation, some more complex, as:

$$V_c = V + b\log(i_0) - b\log(i) - Ri - t\ln\left(1 - \frac{i}{i_L} S^{-\mu\left(1-\frac{i}{i_L}\right)}\right)$$

Where S can be related to flooding, or specifically targeting one of the reactants:

$$V_c = V + b\log(i_0) - b\log(i) - Ri - b\log\left(1 - \frac{i}{i_{L,O_2}}\right)$$

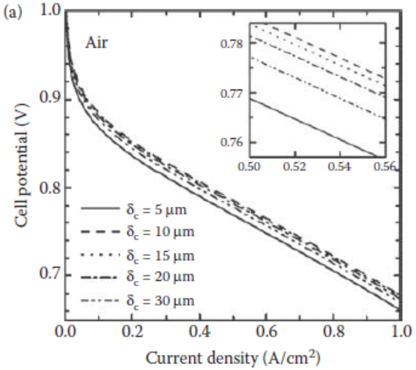


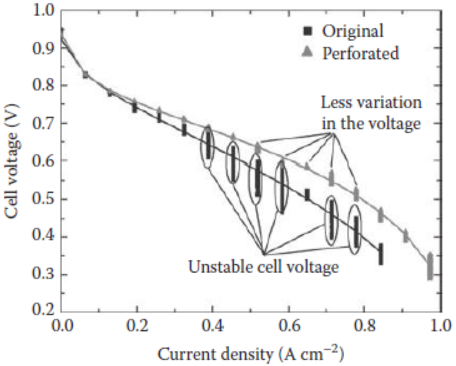
# POLARIZATION CURVE – APPLICATION

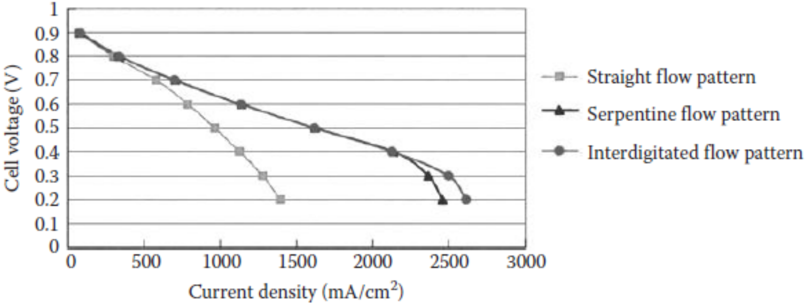
The polarization curve reflects an integral effect of all parameters of cell structure and operating conditions. It is a tool useful in studying both cell structure and operation.

**Cell structure optimization**

V/I curve comparison are useful for comparing different design choices, by either comparing the absolute values of data and their signal-to-noise ratio.



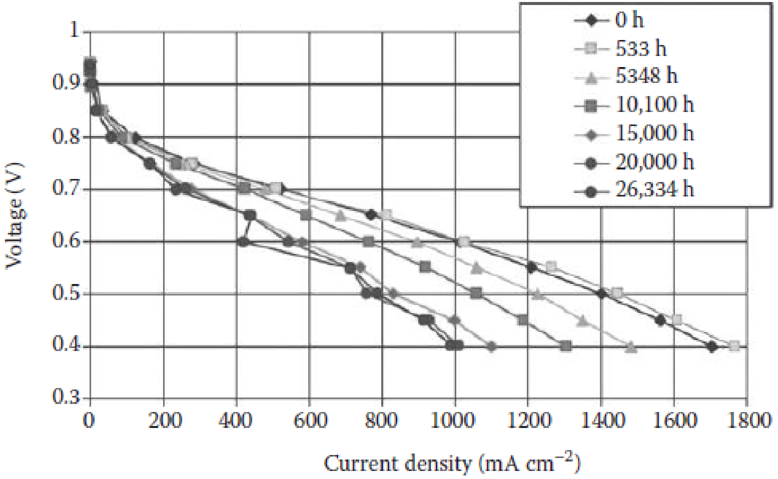




# POLARIZATION CURVE – APPLICATION

**Diagnosing degradation**

Periodic recording of V/I can be used to time-resolve loss of performances.



# 3. ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY

# ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY

EIS is a technique which is widely used in different fields dealing with electrochemistry due to its versatility and the depth of information which can provide. In fact, it allows to perform nondestructive measurements providing detailed diagnostic information about a wide range of electrochemical phenomena including charge transfer reaction at the interface electrode/electrolyte, reaction mechanisms, state of charge of batteries, electrode material properties, and state of health of fuel cells, and so on.

# ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY

Impedance can be viewed as a generalization of the resistance in the frequency domain:

$$Z(\omega) = R + jX(\omega) = |Z|\, e^{j\theta}$$

Where: $R = \mathcal{R}\{Z\}$ and $X = \mathcal{I}\{Z\}$, $Z_0 = |Z|\, e\, \theta = \arg(Z)$

In this extent, the ohm’s law can be expressed as

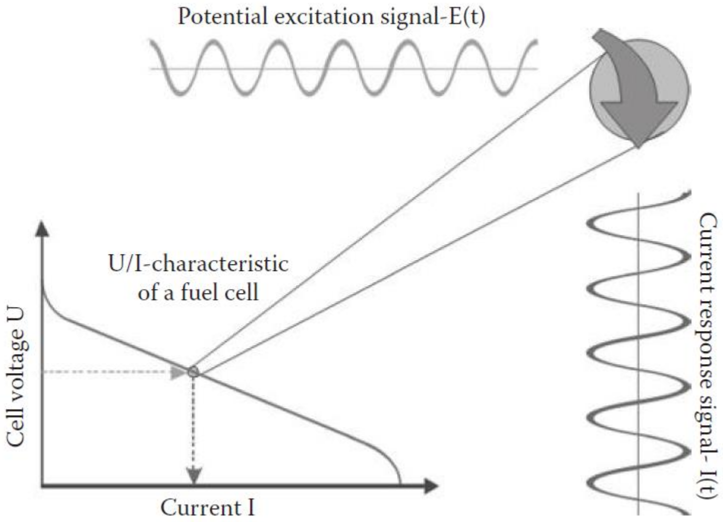
$$E = ZI$$

By considering to apply a sinusoidal voltage

$$Z = \frac{E(t)}{I(t)} = \frac{E_0 \sin(\omega t)}{I_0 \sin(\omega t + \theta)} = Z_0 \frac{\sin(\omega t)}{\sin(\omega t + \theta)}$$

With $\omega = 2\pi f$

# ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY



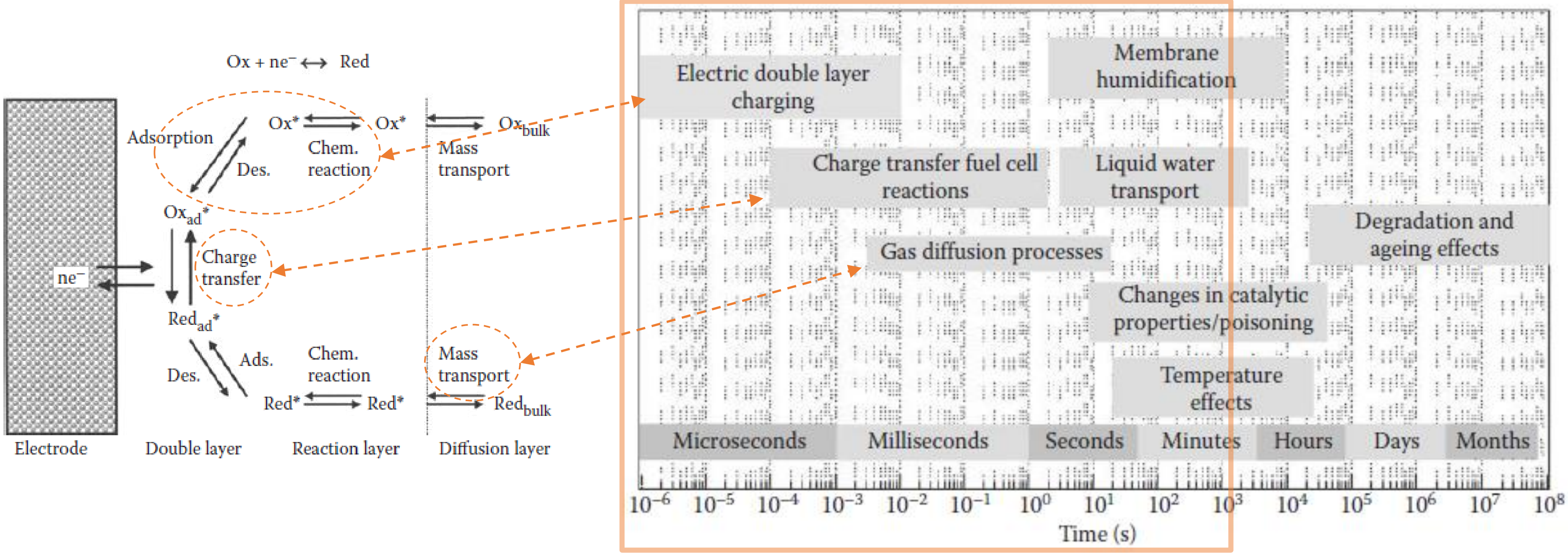
EIS is thus carried out by applying a small AC perturbation to a (running) system, sweeping in frequency usually within 10 kHz and 1 mHz. Frequency-dependant phenomena will start oscillate at the same frequency of the applied stimulus. EIS can either be potentiostatic (PEIS) or galvanostatic (GEIS). The system response (current for PEIS, voltage for GEIS) will be phase shifted according to the whole impedance of the object under analysis. In order to correctly carry out the measure, AC signal must be small that perturbation can be considered linear.

# ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY

Requirements:

- **Linearity** In general, electrochemical systems are non-linear *(e.g*. the current produced it follows the Butler–Volmer equation). Usually, when perturbations of a system from equilibrium are small, system response can be approximated with a linear model. Therefore, the amplitude of applied AC signal must be small enough to ensure a linear relation between perturbation and response during the entire measurement.
- **Causality** The response measured from the system must be directly originated from the applied AC perturbation.
- **Stability** The system must be returned to its original state without significant alteration after the applied AC perturbation is eliminated.
- **Finiteness** The measured impedance must be a finite value at the whole range of measurement frequency

# ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY



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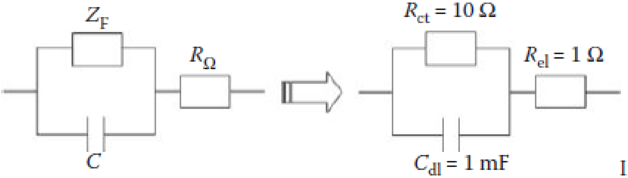
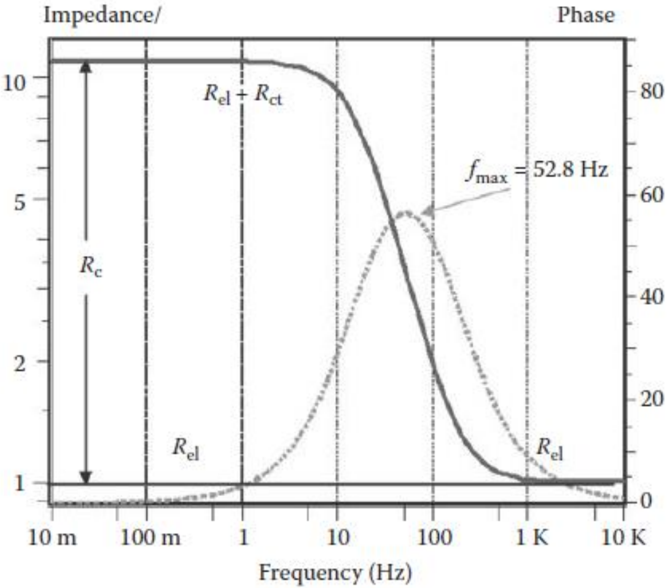
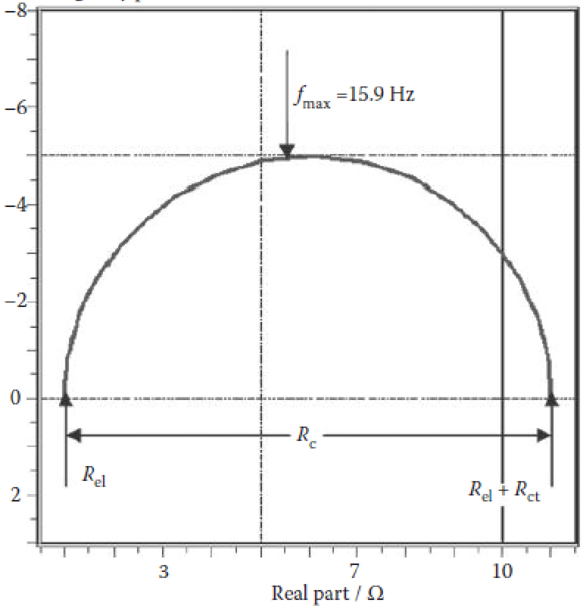


$R_{ct}$ – charge transfer resistance
$C_{dl}$ – double layer capacity
$R_{el}$ – electrolyte resistance





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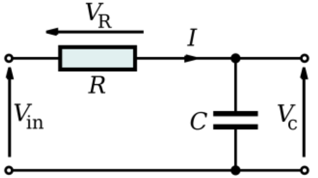


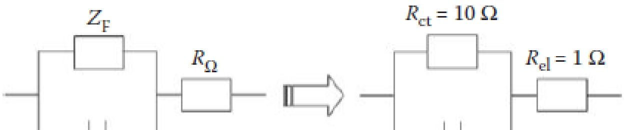
$$Z = R + X = R + \frac{1}{j\omega C} = R - j\frac{1}{\omega C}$$

$$V_C = \frac{X}{R+X} V_{in}$$

$$H(\omega) = \frac{V_C}{V_{in}} = \frac{1/j\omega C}{R + 1/j\omega C}$$

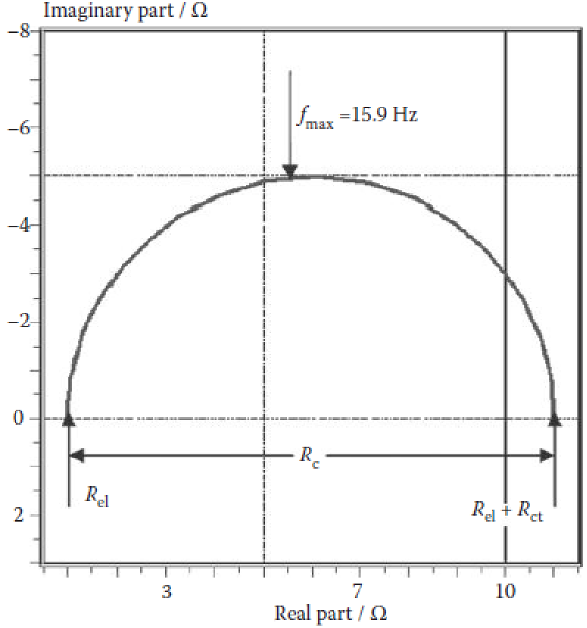
$$H(\omega) = \frac{1}{1 + j\omega RC} = \frac{1}{1 + j\omega/\omega_0}$$

$$\omega_0 = \frac{1}{RC} = 2\pi f_{max}$$



$$f_{max} = \frac{1}{2\pi R_{ct} C_{dl}}$$

$$R_{el} = Z(\omega = 0)$$



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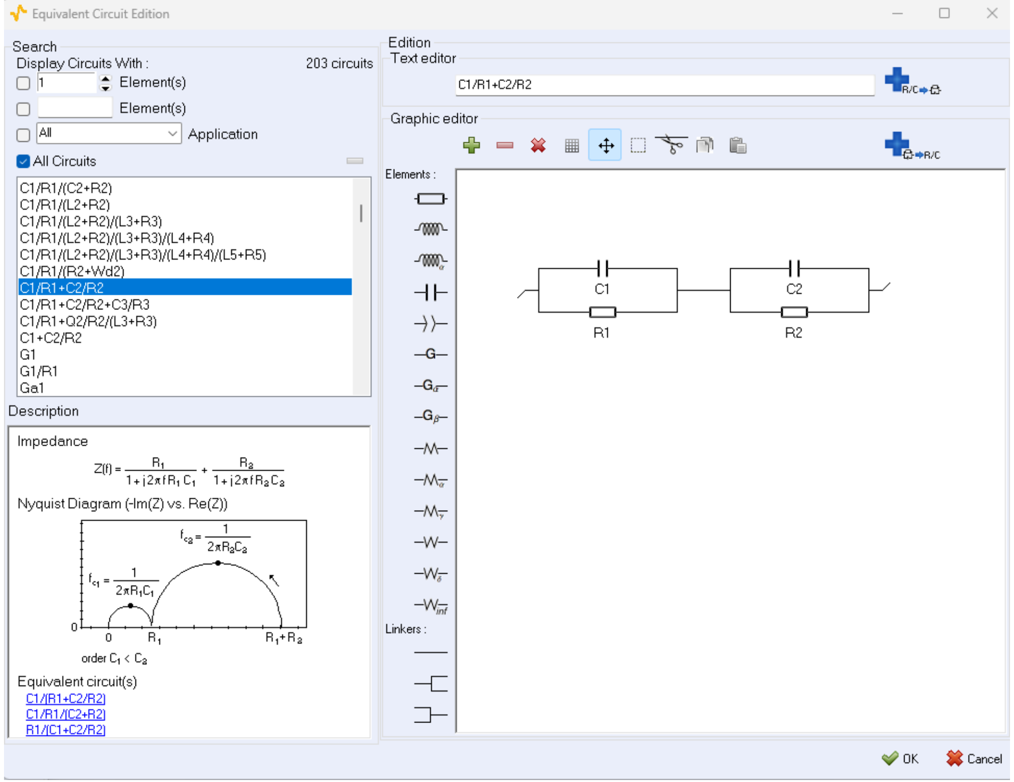


Display Circuits With : 203 circuits

☐ 1 Element(s)

☐ Element(s)

☐ All Application

☑ All Circuits

C1/R1/(C2+R2)
C1/R1/(L2+R2)
C1/R1/(L2+R2)/(L3+R3)
C1/R1/(L2+R2)/(L3+R3)/(L4+R4)
C1/R1/(L2+R2)/(L3+R3)/(L4+R4)/(L5+R5)
C1/R1/(R2+Wd2)
C1/R1+C2/R2
C1/R1+C2/R2+C3/R3
C1/R1+Q2/R2/(L3+R3)
C1+C2/R2
G1
G1/R1
Ga1

Text editor: C1/R1+C2/R2

Description

Impedance

$$Z(f) = \frac{R_1}{1 + j2\pi f R_1 C_1} + \frac{R_2}{1 + j2\pi f R_2 C_2}$$

Nyquist Diagram (-Im(Z) vs. Re(Z))

$f_{c_2} = \frac{1}{2\pi R_2 C_2}$

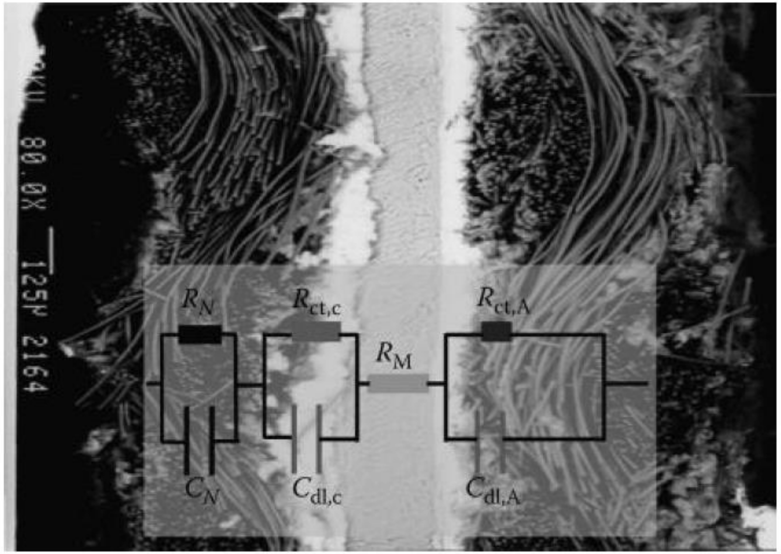
$f_{c_1} = \frac{1}{2\pi R_1 C_1}$

0 0 $R_1$ $R_1 + R_2$

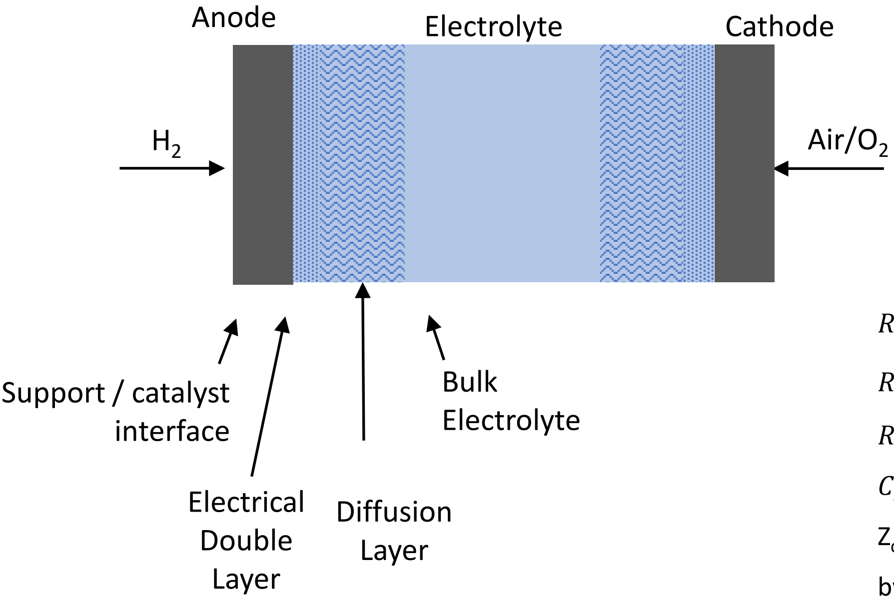
order $C_1 < C_2$

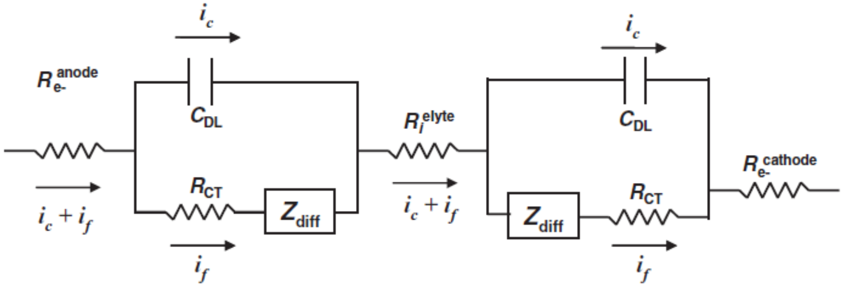
Equivalent circuit(s)

C1/(R1+C2/R2)
C1/R1/(C2+R2)
R1/(C1+C2/R2)



# ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY





$R_{e^-}^{x}$: Electrical resistance

$R_i^{elyte}$: Ionic resistance

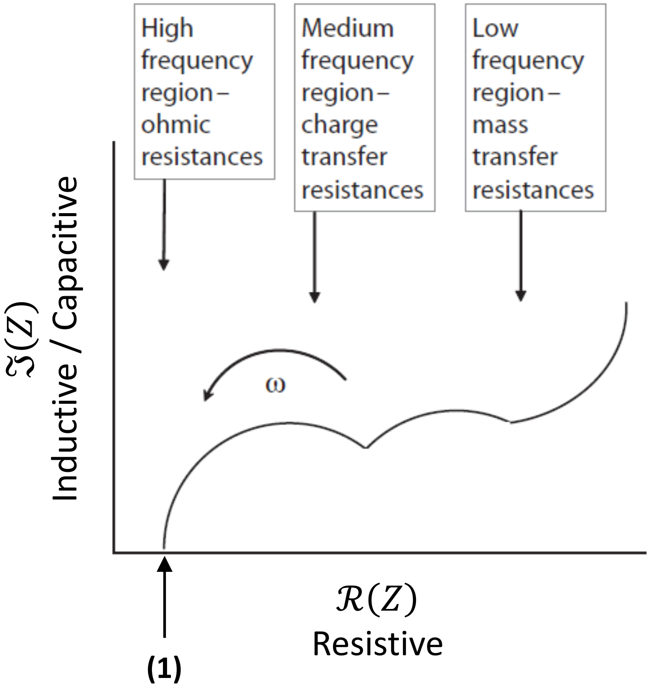
$R_{CT}$: Charge transfer resistance

$C_{DL}$: Double layer capacity (charge transfer processes across the double layer)

$Z_{diff}$: Diffusion-related impedance (also called Warburg resistance, W); it is characterized by a constant slope and $\theta = 45°$.

In some circumstances (such as to describe the porous structure of electrodes) the capacitance needs to be replaced by a Constant Phase Element, $Z_{CPE}$, is used to describe capacitive effects which have a strong non-ideal behaviour: $Z_{CPE} = 1/\left((j\omega)^{\beta} Q\right)$

# ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY



**High frequency region:**

$$Z_C = X_C = \frac{1}{2\pi f C} \quad \Rightarrow \quad X_C\Big|_{f\to\infty} \to 0$$

In (1) the High Frequency Resistance value is found: it is the sum of all of the ohmic resistances of the system (such as, for fuel cells: contact losses, ionic losses in thee electrolyte and electronic resistance).

**Medium frequency region:**

The presence of a (positive) semicircle indicates a capacitive process is taking place (such as: charge transfer across the double layer). The semicircle can be represented by a RC-parallel electric circuit, composed by the charge-transfer resistance and the charge-transfer capacity. The circle diameter is equal to the charge transfer resistance.

**Low frequency region:**

Mass transport phenomena are related to the evolution of the plot in this regime.

# ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY

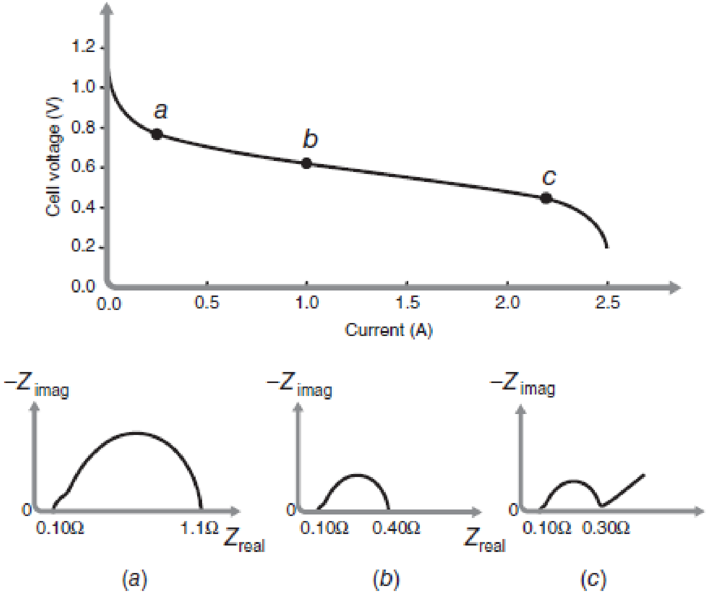
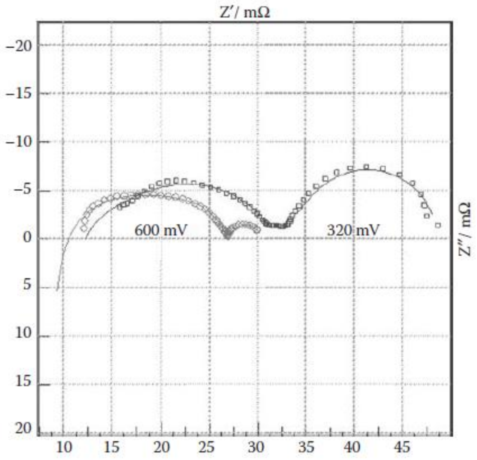
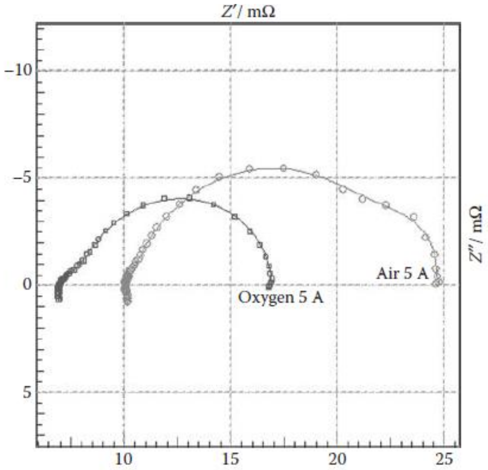


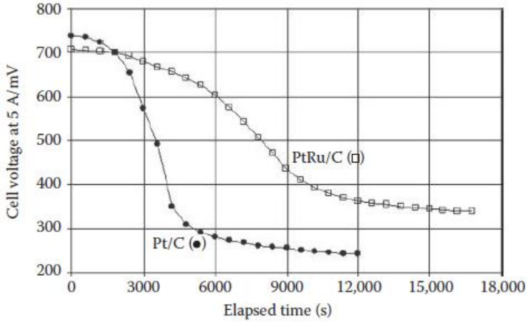
**Figure 7.14.** EIS characterization of a fuel cell requires impedance measurements at several different points along an *i*–*V* curve. The impedance response will change depending on the operating voltage. (*a*) At low current, the activation kinetics dominate and $R_f$ is large, while the mass transport effects can be neglected. (*b*) At intermediate current (higher activation overvoltages), the activation loops decrease since $R_f$ decreases with increasing $\eta_{act}$. (Refer to Equation 7.15.) (*c*) At high current, the activation loops may continue to decrease, but the mass transport effects begin to intercede, resulting in the diagonal Warburg response at low frequency.

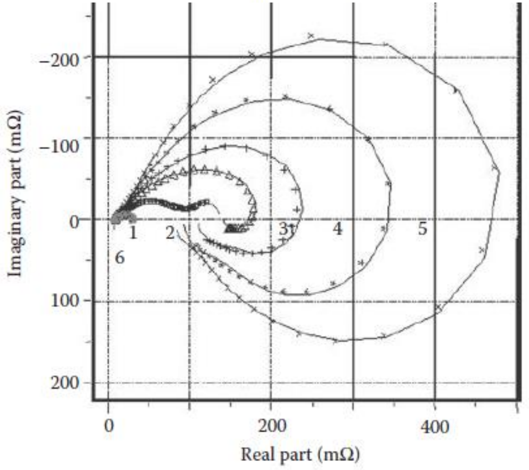
Fuel Cell Fundamentals, O'Hayre, Suk-Won, Colella, Fritz B. Prinz (2016) Wiley

# ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY

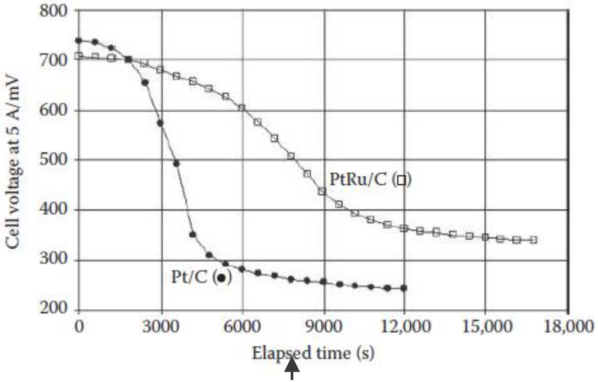


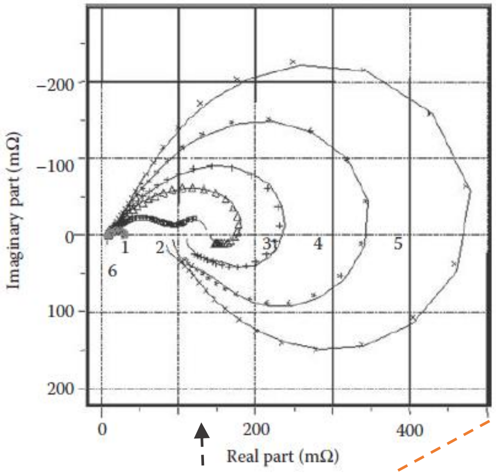


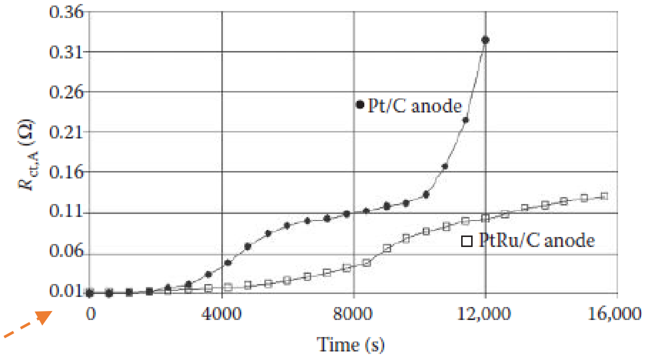


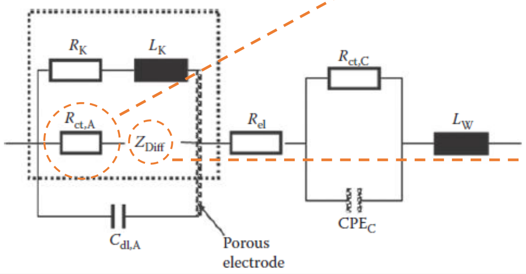


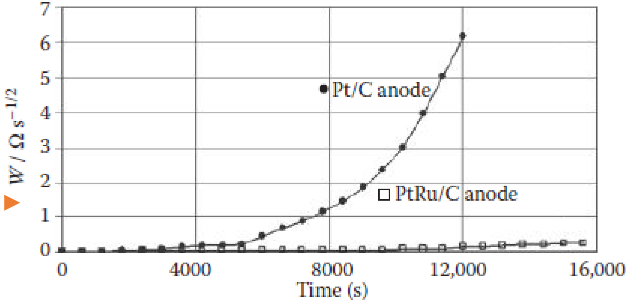
# ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY





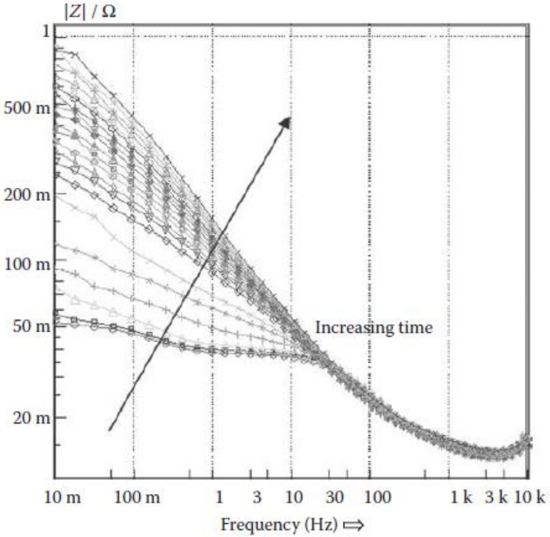


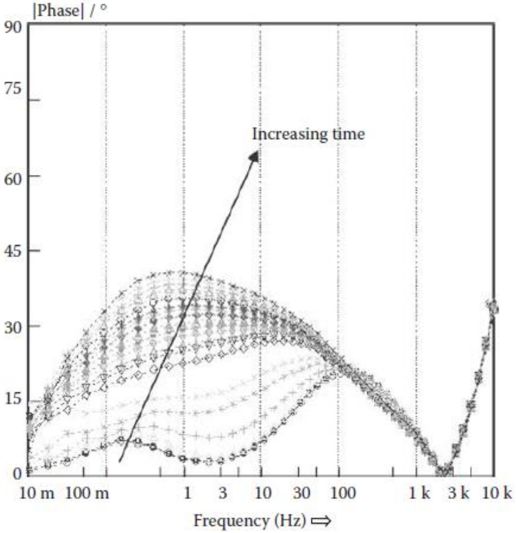


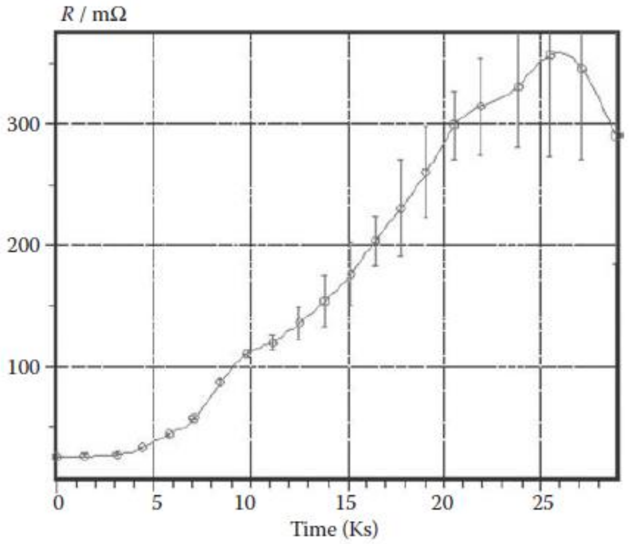


Example #1
Anodic CO poisoning

# ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY







Example #2
Cathode flooding

# ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY

No unique solution and / or model. Data analysis can be carried out following a model-based or a model-free approach.

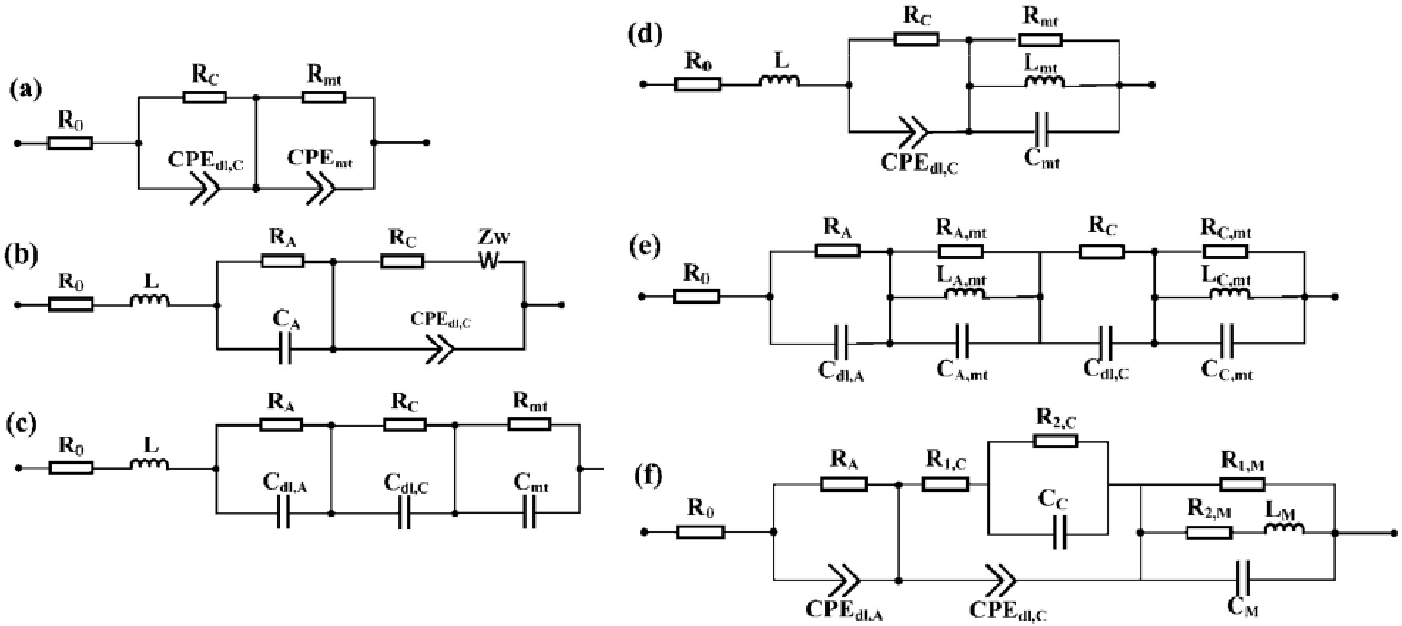
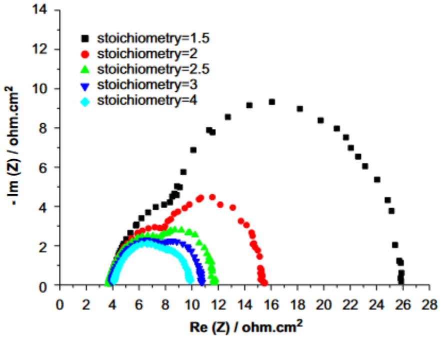


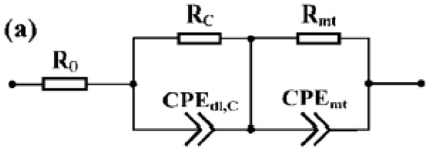
Fig. 2. Typical ECMs used to interpret the EIS data of PEMFCs under various conditions. (a) A 2 kW PEMFC stack [35], reproduced with permission from Ref. [35], Copyright 2007, Elsevier; (b) $SO_2$ contamination effects on ORR [36], reproduced with permission from Ref. [36], Copyright 2012, The Electrochemical Society; (c) electrode composition [37], reproduced with permission from Ref. [37], Copyright 2008, Elsevier; (d) water failure [38], reproduced with permission from Refs. [38], Copyright 2019, Elsevier; (e) inductive phenomena at LF [39] and (f) an ECM deduced from a complete process model [25,40], reproduced with permission from Ref. [25], Copyright 2016, Elsevier.

# ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY

**Model-based approach** consists in identifying the most suitable equivalent circuit model to analyse data.

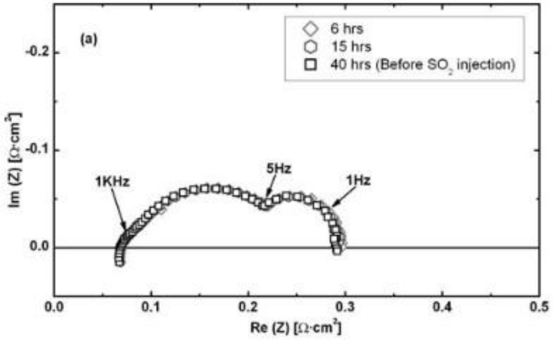


X. Yan et al., International Journal of Hydrogen Energy 32 (2007) 4358 – 4364

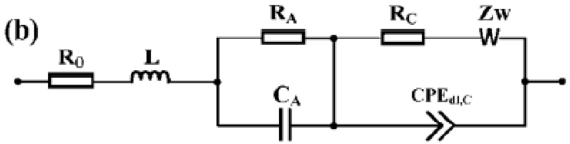


Recorded from a stack.

Anode features were not modelled as considered neglectable. $R_0$ includes both ionic and electronic conduction resistance of the electrolyte. Subscripts *C* and *mt* refer to charge transfer and mass transport respectively



Zhal Y. et al., Journal of The Electrochemical Society, 159, 5, (2012), B524-B530



Charge transfer at the anode is here considered, while mass transport at the cathode are modelled by means of a Warburg element.

# ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY

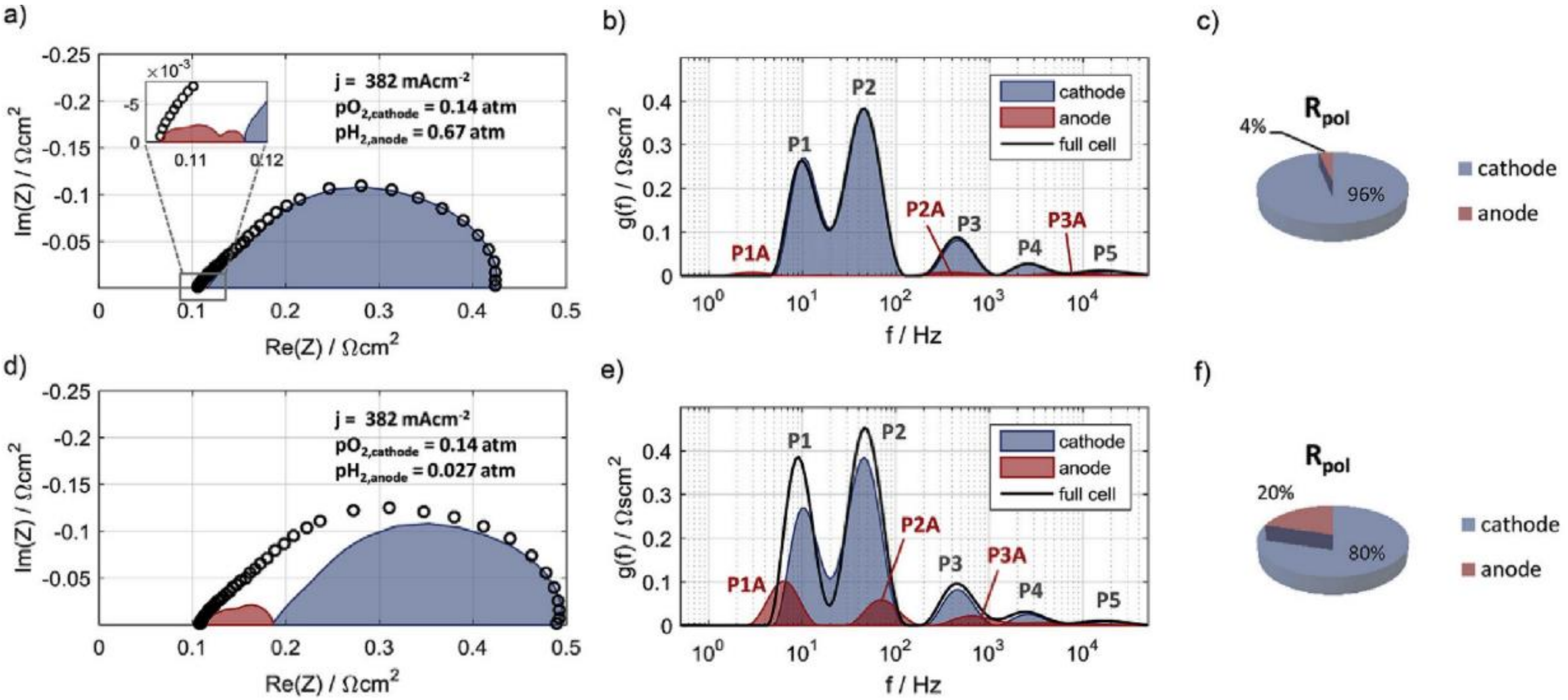
**Model-free approach** (also known with the name Distribution of Relaxation Times, DRT) does not require prior knowledge of impedance in the interpretation of EIS data. Here relaxing time are monitored, quantifying the time required for electrochemical systems to shift from one equilibrium state to another under external perturbation. It was theorised and firstly developed within the first two decades of the XX century. Here, impedance is described as:

$$Z = R_0 + R_P \int_0^{\infty} \frac{\gamma(\tau)}{1 + j\omega\tau} d\tau$$

$$\int_0^{\infty} \gamma(\tau) d\tau = 1$$

With $\gamma(\tau)$ being the probability distribution function of the relaxation times, $R_0$ the ohmic resistance, and $R_P$ the polarisation resistance.

# ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY



# ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY

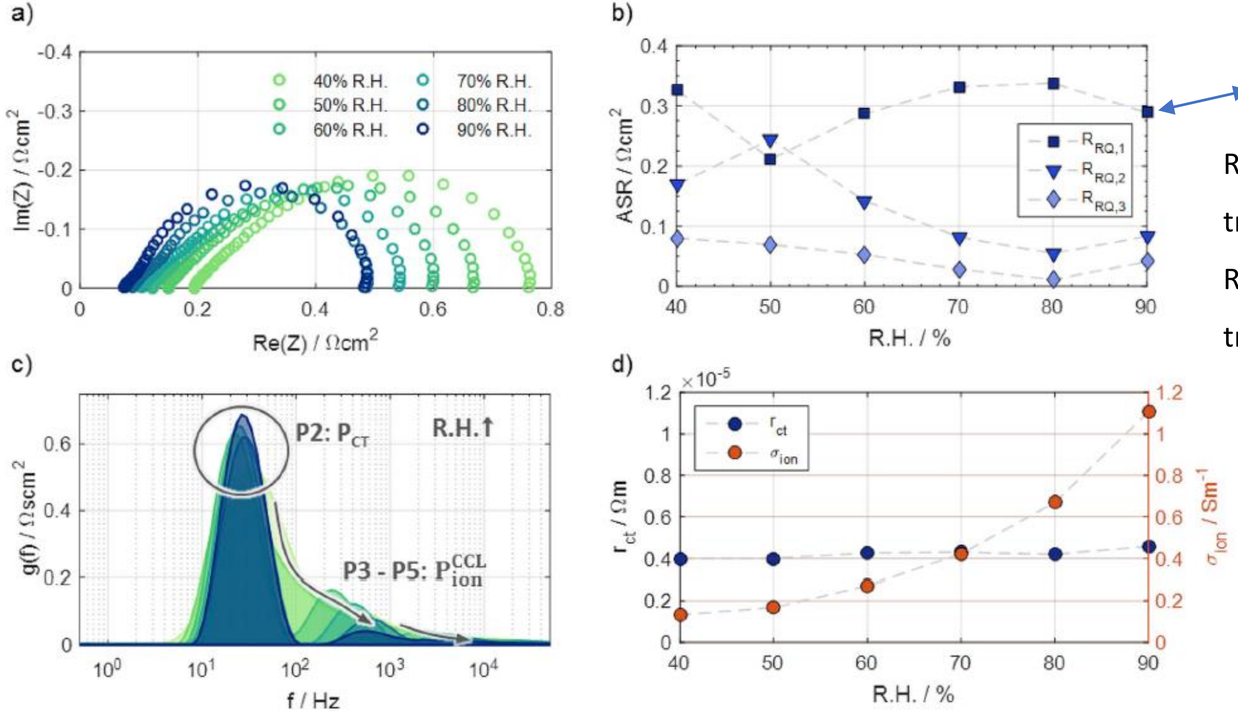
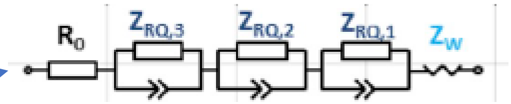


Fig. 4. Impedance spectra (a) and calculated DRTs (c) for a variation of relative humidity (from 40% R.H. to 90% R.H. at j = 146 mA cm$^{-2}$). (b) Obtained model parameters for application of ECM (3RQ). (d) Area specific charge transfer resistance and ionic conductivity determined by application of ECM (TLM).



$R_{RQ,1}$ describes the main peak (charge transfer) at about 25 Hz while $R_{RQ,2}$ and $R_{RQ,3}$ describe the secondary peaks (proton transport in the CCL) at higher frequencies

# ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY

EIS can be used to monitor:

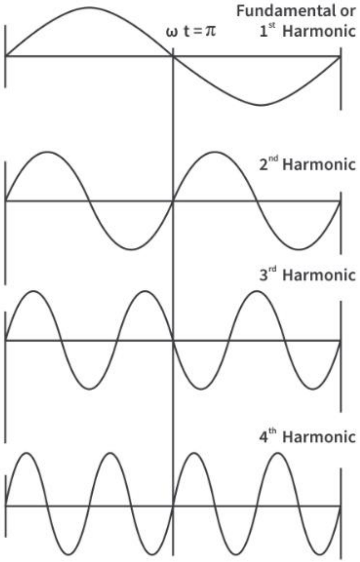
- Changes of performances due to stoichiometry variations, thus allowing to highlight the presence of reactant starvation.
- Contamination
- Presence of water content (drying, flooding)

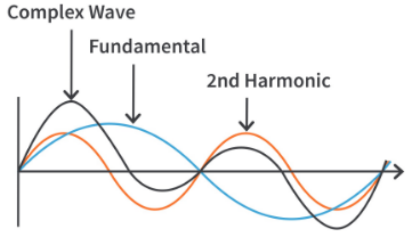
EIS can be a tool used in design optimisation:

- MEA composition
- Flow field design

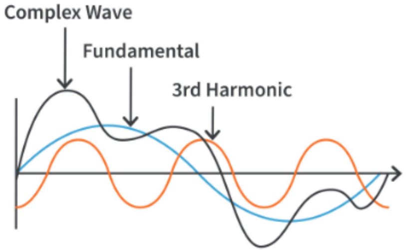
# 4. TOTAL HARMONIC DISTORTION

# TOTAL HARMONIC DISTORTION

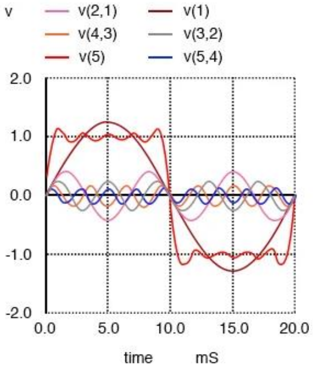




This waveform contains a 2nd harmonic.



This waveform contains a 3rd harmonic.



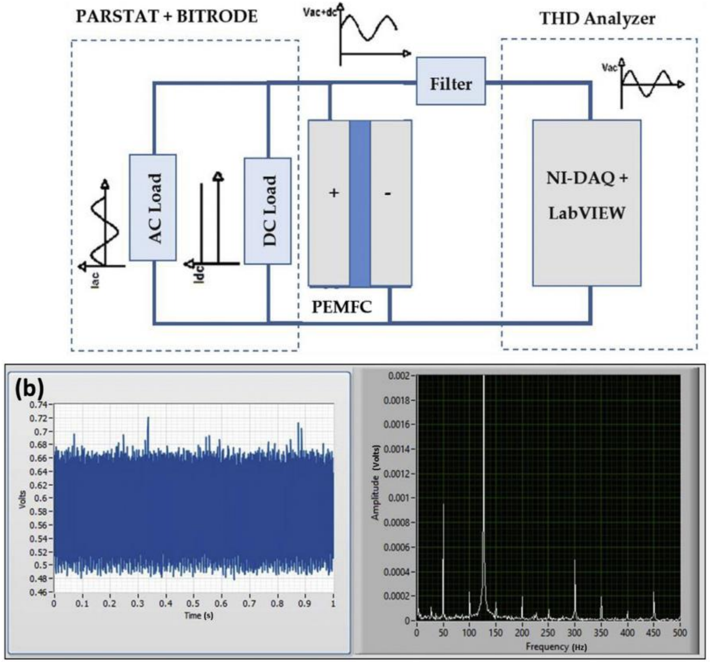
# TOTAL HARMONIC DISTORTION

Total harmonic distortion, is a measurement of the level of harmonic distortion present in power systems. It can be related to either current harmonics or voltage harmonics, and it is defined as the ratio of the RMS value of all harmonics to the RMS value of the fundamental component times 100%; the DC component is neglected.

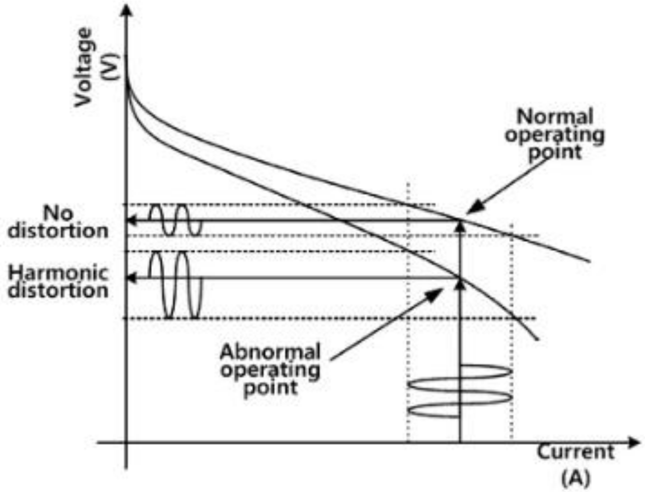
$$THD_V = \frac{\sqrt{\sum_{k=2}^{N} V_k^2}}{V_1}$$

where $V_k$ is the RMS voltage of the kth harmonic and k = 1 is the order of the fundamental component.

# TOTAL HARMONIC DISTORTION

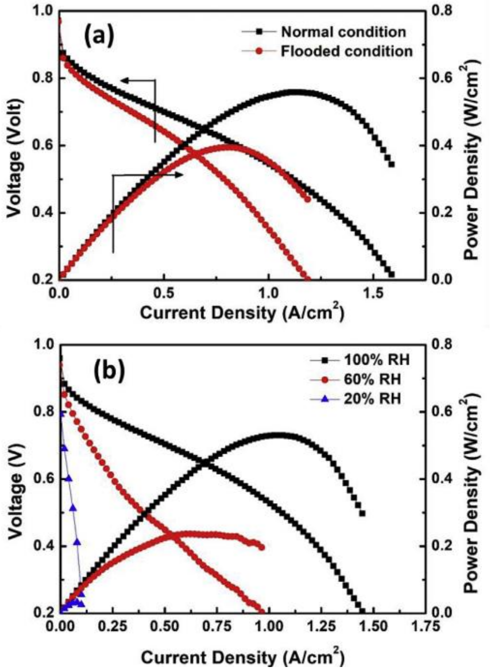


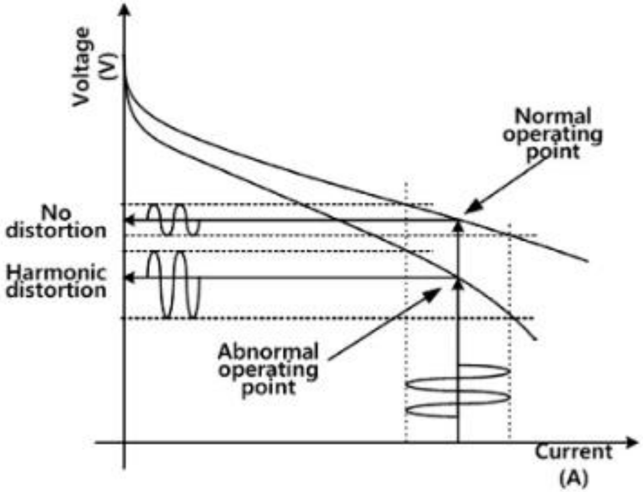
N.J. Steffy et al. Journal of Power Sources 404 (2018) 81–88



E. Ramschak et al. / Journal of Power Sources 157 (2006) 837–840

# TOTAL HARMONIC DISTORTION

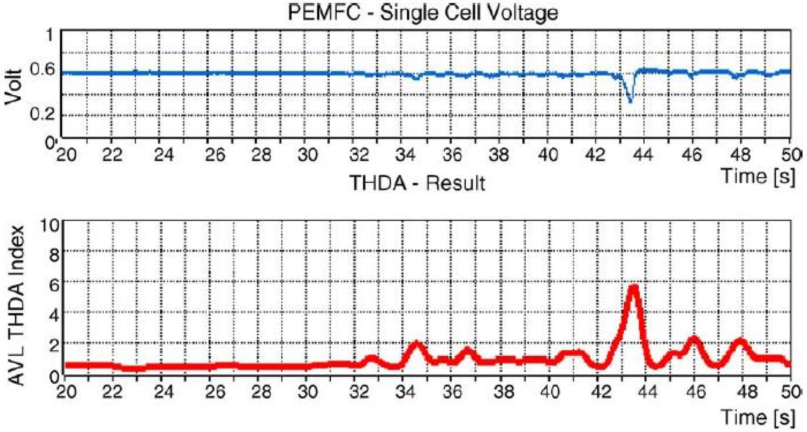




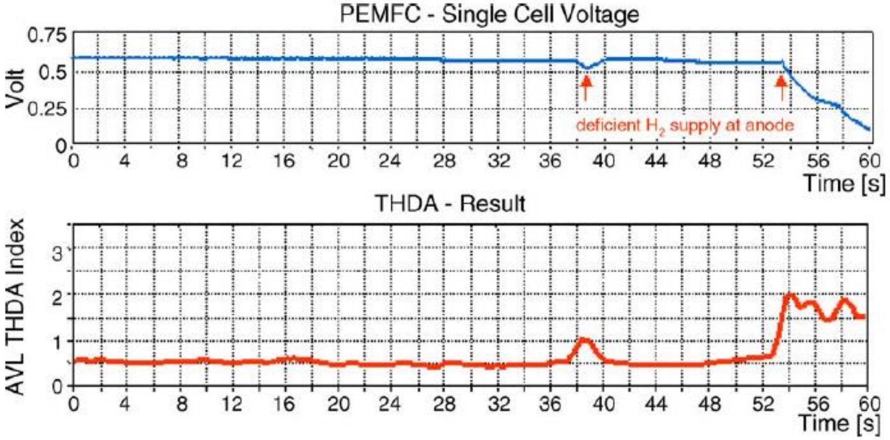
E. Ramschak et al. / Journal of Power Sources 157 (2006) 837–840

# TOTAL HARMONIC DISTORTION

## Air Starvation



## H2 supply deficiency



Stack-compatible

# 5. CYCLIC VOLTAMMETRY

# CYCLIC VOLTAMMETRY

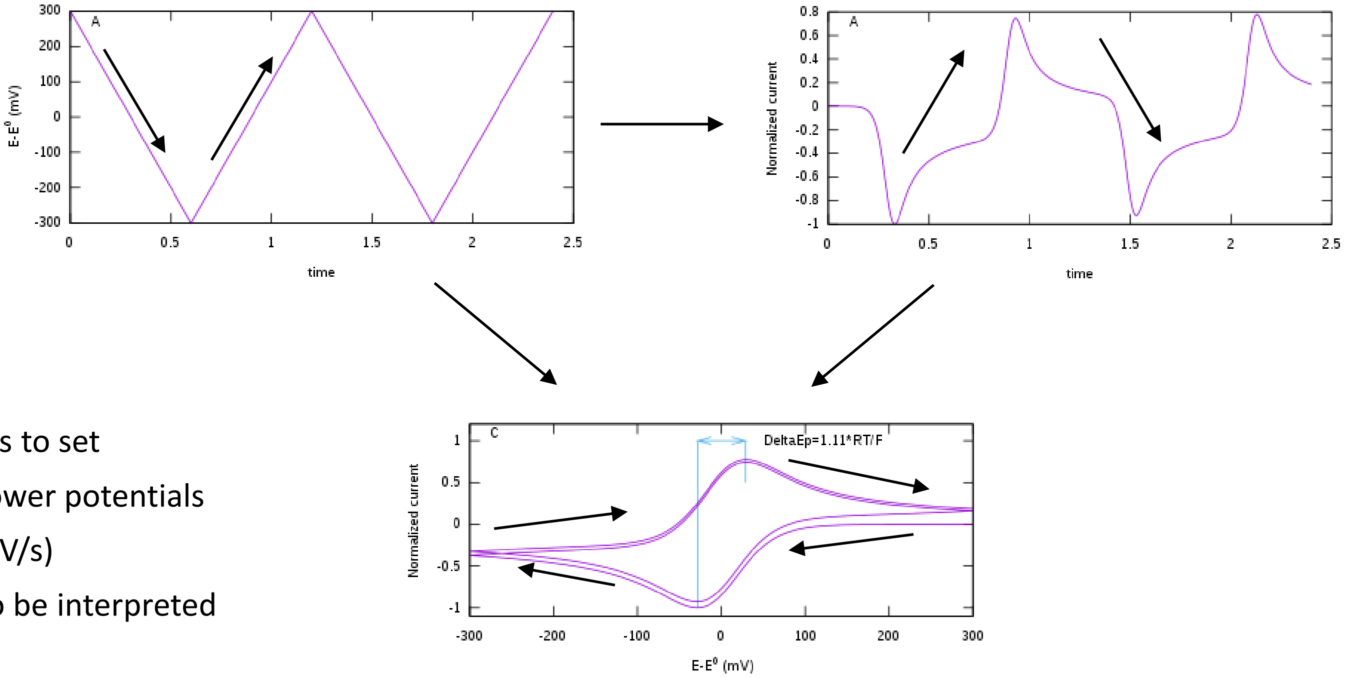
Cyclic voltammetry (CV) is one of the most widely used electrochemical techniques and it is frequently applied for the initial characterization of a redox system. It allows to quick analysis on the redox properties of the system under evaluation; more in depth, it allows to:

- Define the number of oxidation state of the specimen under investigation.
- Retrieve qualitative information about the oxidation state stability and the electron transfer kinetics.
- Retrieve quantitative information of redox potentials of the electroactive species

Moreover:

- It is non-destructive and does not require the device to be disassembled during operation
- It can be used on single electrodes (half-cells) or on the entire devices.

# CYCLIC VOLTAMMETRY



Main parameters to set

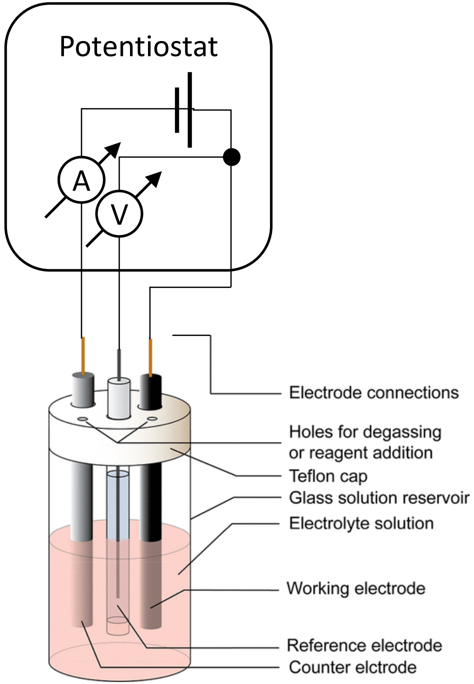
- Upper and lower potentials
- Scan rate, ν (V/s)

Main features to be interpreted

- Peaks
- Plateau regions

# CYCLIC VOLTAMMETRY

**Recorded from half cells / electrodes**

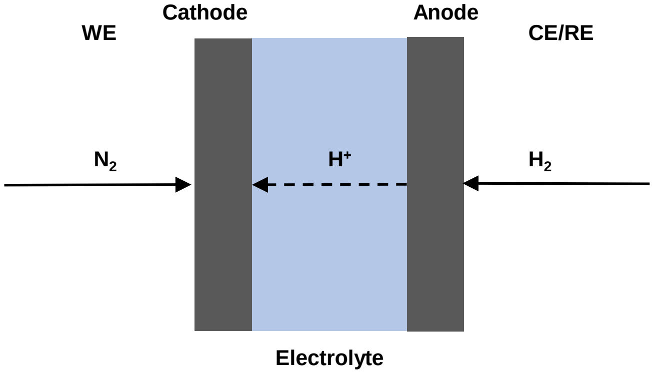


Elgrishi N. et al., J. Chem. Educ. 2018, 95, 2, 197–206

**Recorded from full cells**

For fuel cells, $N_2$ is used instead of $O_2$ for delating the background noise due to spurious hydrogen adsorption in addition to the hydrogen evolution/oxidation reaction.

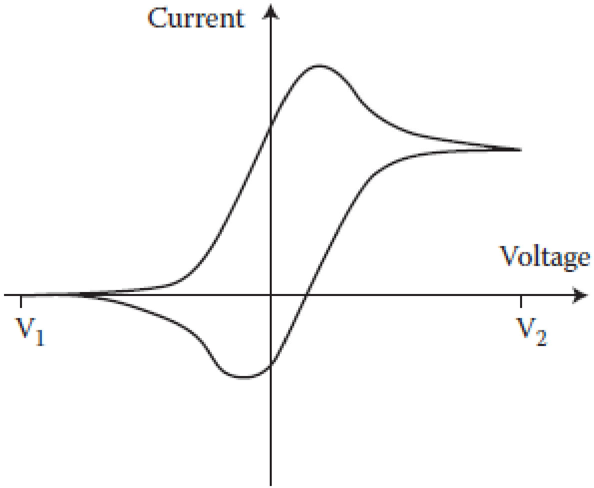
The potential reference is set at the hydrogen oxidation potential by connecting both RE and CE to the anode



# CYCLIC VOLTAMMETRY

The characteristics of the peaks in a cyclic voltammogram can be used to acquire qualitative information about the relative rates of reaction and reactant diffusion in a given electrochemical system.

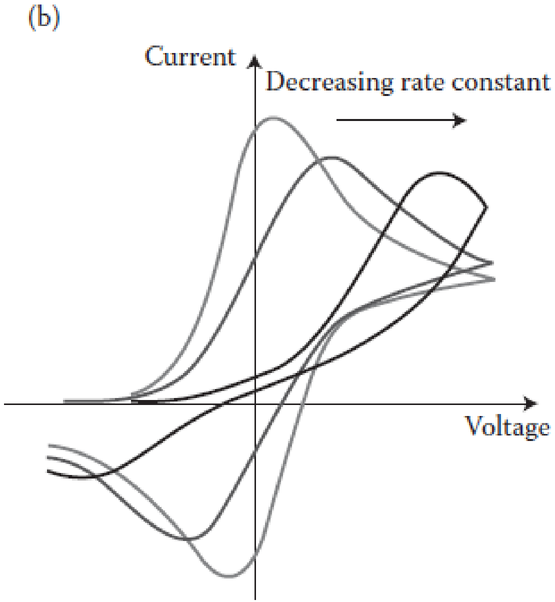
When the potential of the working electrode is more positive than that of a redox couple, the corresponding reactants may be oxidized and produce an anodic current. The peak current occurs when the potential reaches a value at which all the electrochemically active reactants at the electrode surface are completely consumed. them. After this peak, the current will decline because the double-layer thickness increases, resulting in a less steep concentration gradient for the active reactant.



# CYCLIC VOLTAMMETRY

A redox reaction is defined quasi-reversible or irreversible when the rate of electron transfer is slow compared to other processes. In this case, the peak position changes depending on the sweep rate used for recording the process.

The sweep rate thus becomes a crucial parameter in describing the meaning of the voltammogram, as some reactions can be reversible or irreversible at different scan rates, as the electron transfer process is constant. According to the selected sweep rate, it is thus possible to highlight different features of the redox reactions, such as oxidation and reduction potentials, as well as reaction kinetics.



# CYCLIC VOLTAMMETRY

In alkaline media

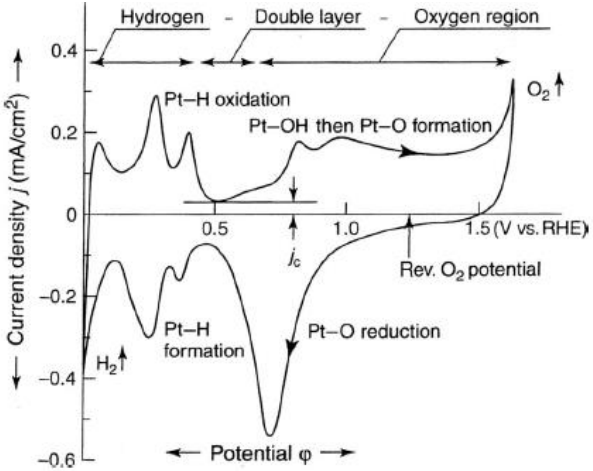


**Figure 9.8** Cyclic voltammogram of polycrystalline platinum in 1 M KOH alkaline electrolyte solution at 20°C with voltage sweep rate of 100 mV/s. (Reproduced with permission from Ref. [12].)

| | |
|---|---|
| HOR | $Pt - H + OH^- \rightarrow Pt + H_2O + e^-$ |
| Pt oxidation | $Pt + OH^- \rightarrow Pt - OH + e^-$<br>$Pt - OH + OH^- \rightarrow Pt - O + H_2O + e^-$ |
| OER | $4OH^- \rightarrow O_2 + 2H_2O + 4e^-$ |
| ORR | $Pt + H_2O + e^- \rightarrow Pt - H + OH^-$ |
| HER | $2H_2O + 2e^- \rightarrow 2OH^- + H_2$ |

# CYCLIC VOLTAMMETRY

## In acidic media

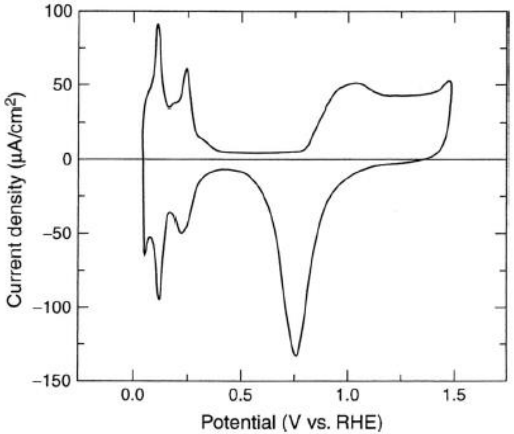
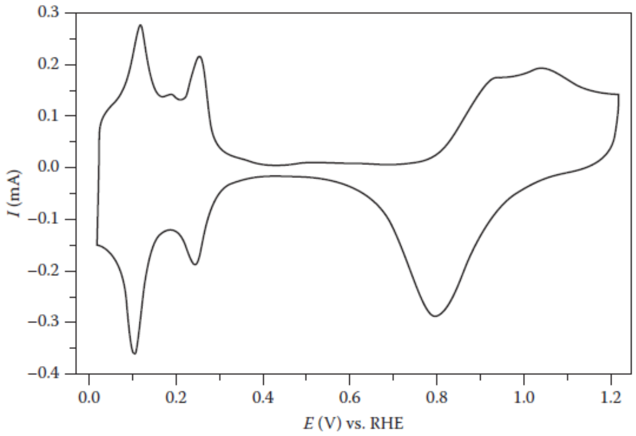


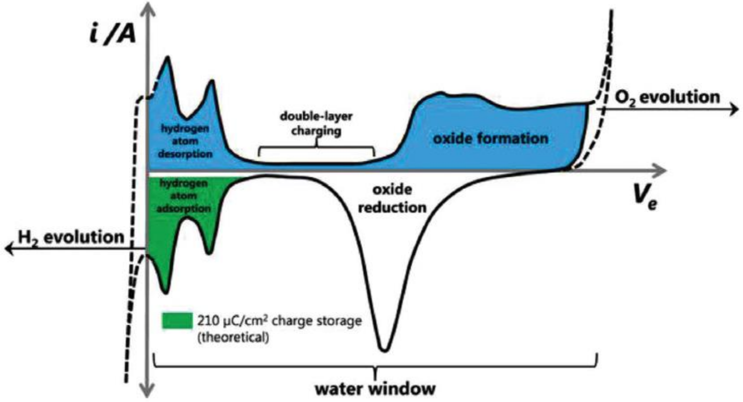
**Figure 9.9** CV of polycrystalline platinum in acid 0.5 M $H_2SO_4$ electrolyte solution with voltage sweep rate of 50 mV/s. (Reproduced with permission from Ref. [12].)

| | |
|---|---|
| HOR | $Pt - H + H_2O \rightarrow Pt + H_3O^+ + e^-$ |
| Pt oxidation | $Pt + H_2O \rightarrow Pt - OH + e^- + H^+$<br>$Pt - OH + H_2O \rightarrow Pt - O + H_3O^+ + e^-$ |
| OER | $2H_2O \rightarrow O_2 + 4H^+ + 4e^-$ |
| ORR | $Pt + H_3O^+ + e^- \rightarrow Pt - H + H_2O$ |
| HER | $2H_3O^+ + e^- \rightarrow 2H_2O + H_2$ |

# CYCLIC VOLTAMMETRY

During fuel cell operation, overall during the start-up and shutdown phases, potential spikes up to 1.5 V can be found. This is a big issue because, for potentials bigger than 0.85 – 1.0 V, PtO starts forming at the cathode electrode, slowing down the reaction rate, and consequently making the Pt reduction peak shift towards higher potentials (lowering Pt oxophilicity).

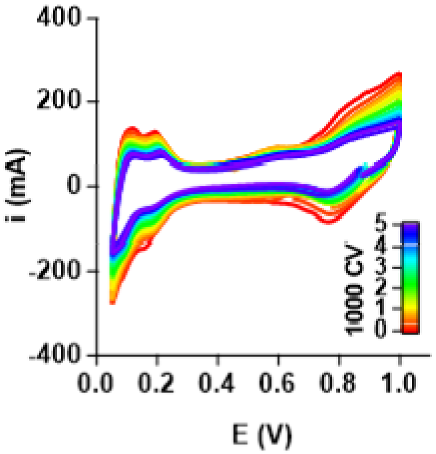




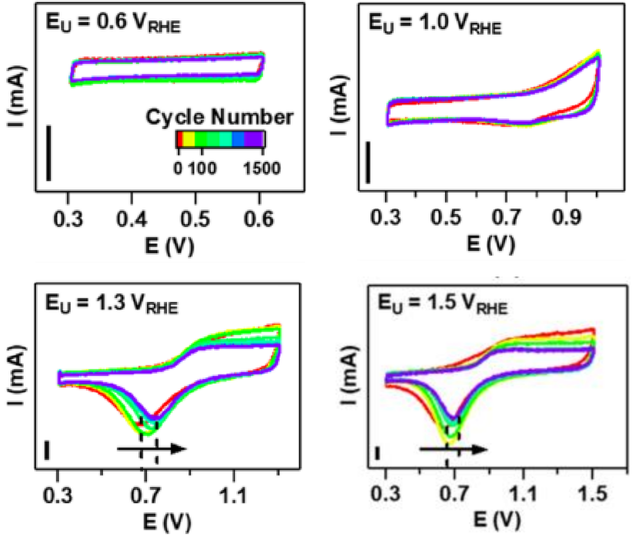
Suter T.A.M. et al., Adv. Energy Mater., 11, (2021), 2101025

# CYCLIC VOLTAMMETRY

In general, to evaluate catalyst activities, CV are recorded over time, searching for any shift of features towards higher potentials and/or of reduction of peak intensities, which are both a marker of activity reduction.



Bogar M. et al., Journal of Power Sources, submitted



Khalakhan I. et al., ACS Appl. Mater. Interfaces 2020, 12, 17602−17610

# CYCLIC VOLTAMMETRY

After the electrode fabrication process, not all of the deposited catalyst in the electrode is able to finally participate in the electrochemical reaction, due to either insufficient contact with the solid electrolyte or electrical isolation of the catalyst particles. In order to determine the effective area of catalyst available to participate into catalysis, the electrochemically active surface area (ESCA) is one of the main parameters which can be calculated by means of cyclic voltammetry.

More in dept, the ESCA is calculated by measuring the charge needed to remove (or deposit) a layer of hydrogen atoms adsorbed on the electrode surface.

# CYCLIC VOLTAMMETRY / ECSA

ECSA can be thus calculated basing on two assumptions:

1. Each platinum site can adsorb only one hydrogen proton.
2. Every available platinum site will be occupied with hydrogen during the transition from hydrogen adsorption to hydrogen evolution.

# CYCLIC VOLTAMMETRY

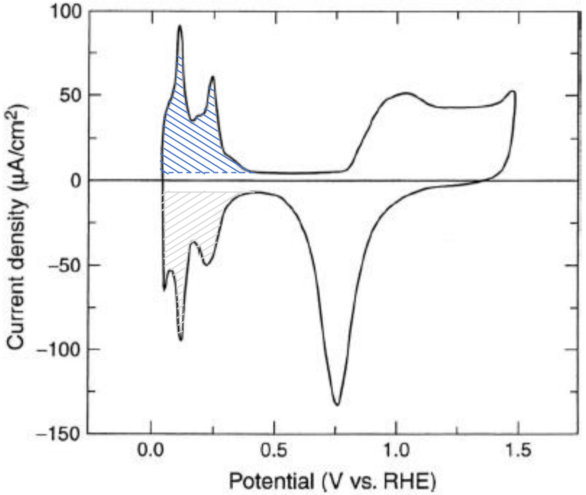
The charge is calculated as the integral of the voltammogram in the hydrogen adsorption (or desorption) region:

$$Q_{H,ad} = \int I(t)dt$$

Where the baseline is set from the low-potential-end-side of the formation of the double layer. Thus, the ECSA is calculated as:

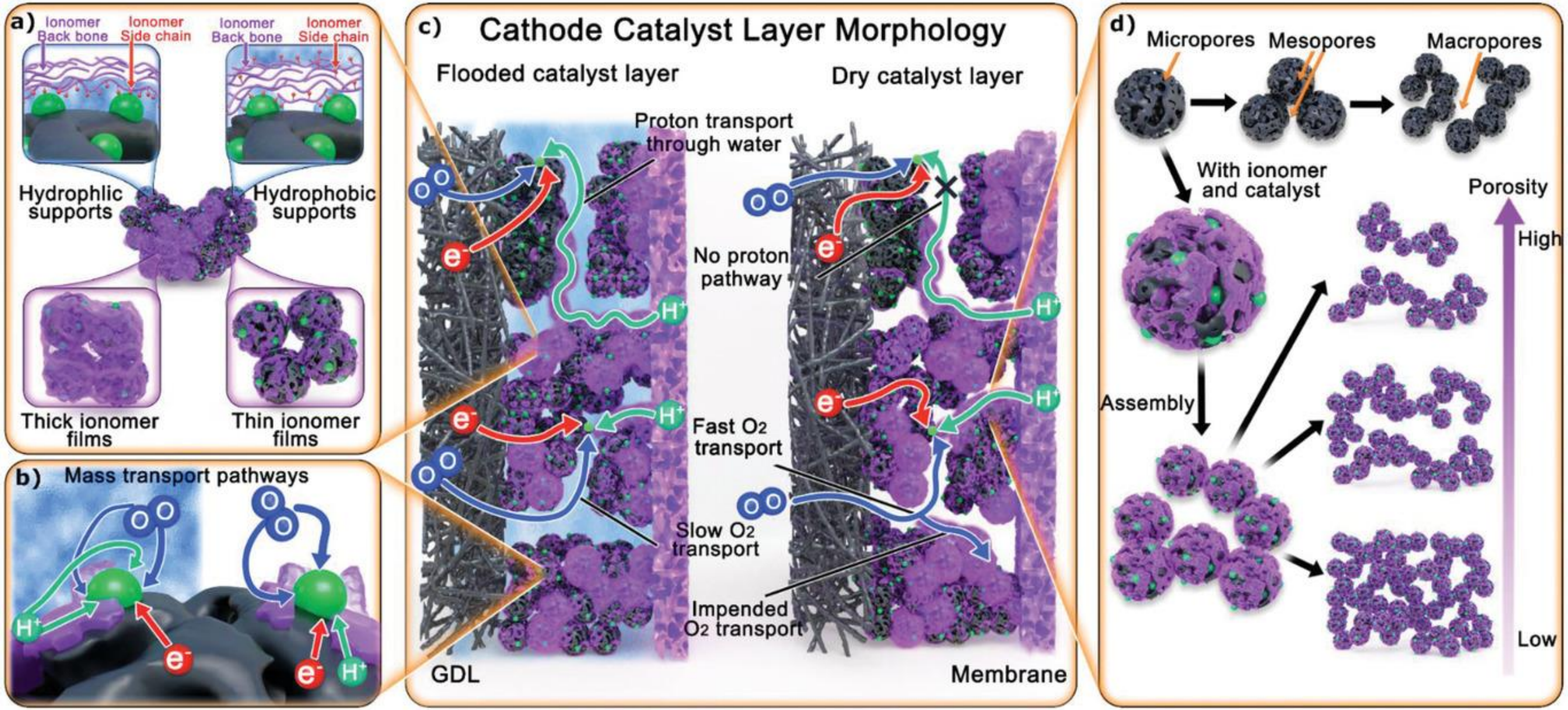
$$ECSA(cm^2/g) = \frac{Q_{H,ad}(C/cm^2)}{m(g/cm^2) * C(C/cm^2)}$$

Where m is the Pt mass loading (g/cm$^2$), and C is a proportionality constant relating charge and catalyst area. It is measured by stripping a layer of Pt atoms on a plane Pt surface, and is conventionally accepted to be equal to 210 μC/cm$^2$.



# CYCLIC VOLTAMMETRY

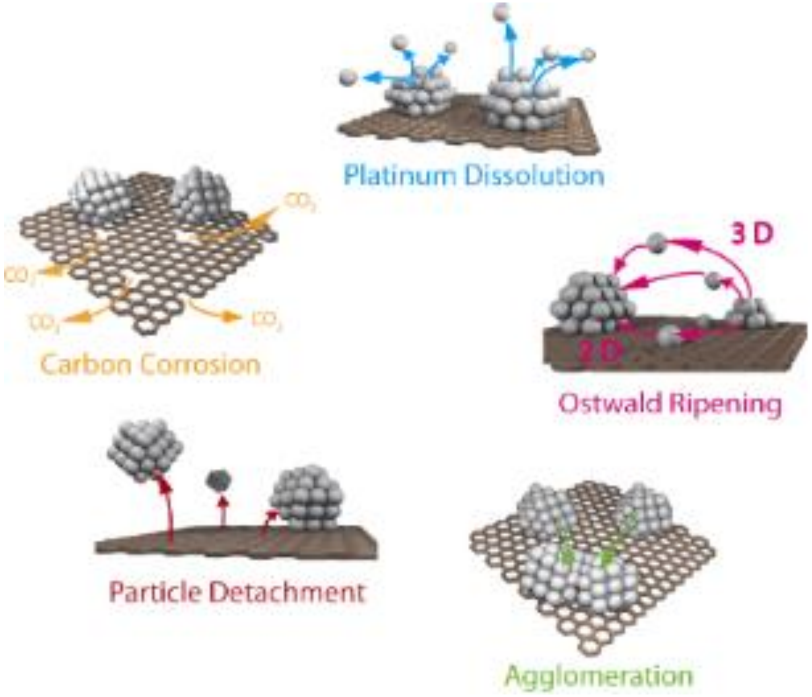
Investigation of conduction pathways



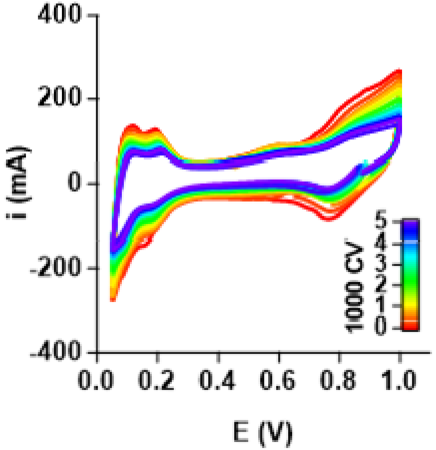
Suter A.M.T. et al., Adv. Energy Mater.2021, 11, 2101025, DOI: 10.1002/aenm.202101025

# CYCLIC VOLTAMMETRY

Investigation of degradation phenomena



Meier, Nanotechnol. 2014, 5, 44–67



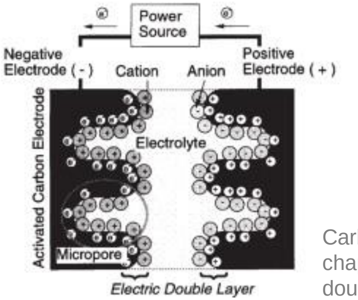
Bogar M. et al., Journal of Power Sources, submitted

# CYCLIC VOLTAMMETRY

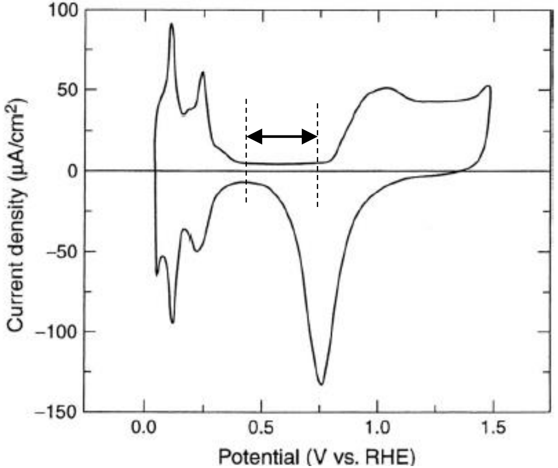
When an electrode surface is subjected to a voltage ramp, a steady-state capacitive current is observed in a short time ($i_C$) if the only process taking place within the voltage range is the charging of the double layer, that is, movement of ions on either side of an electrode/electrolyte interface. For ideal capacitors, $i_C$ is related to the capacitance ($C_{DL}$) and to the scan rate (ν):

$$i_C = \upsilon C_{DL}$$

A possible approach for determining the double layer capacitance consist in recording CVs at various scan rates within a potential region where no redox processes take place, extracting $i_C$ from the anodic and cathodic scans of the recorded voltammograms, and subsequently extracting $C_{DL}$ from the slope of the resulting $i_C$ vs ν plot



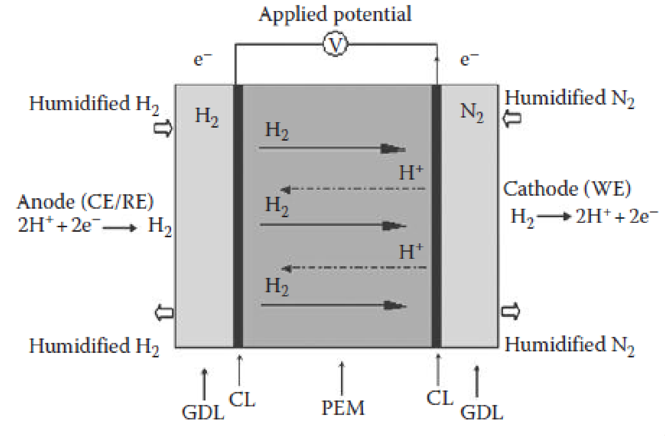
Carbon alloys (2003), chapter 27, electric double layer capacitors



# 6. LINEAR SWEEP VOLTAMMETRY

# LINEAR SWEEP VOLTAMMETRY

Linear sweep voltammetry is an investigation technique which is useful in estimating gas cross-over in operative conditions. A linear potential scan is applied to the fuel cell electrode to obtain a limiting current, which is useful for calculating the crossover rate of hydrogen. By repeating the test over time, membrane degradation can be monitored over time. The potential sweep is usually ended at a voltage below 0.8 V (to prevent irreversible Pt or carbon oxidation), at a low scan rate (such as 4 mV $s^{-1}$). The same approach can be also applied with DMFC for determining methanol crossover.

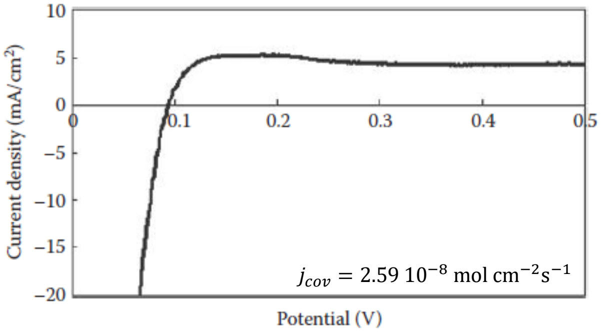


# LINEAR SWEEP VOLTAMMETRY

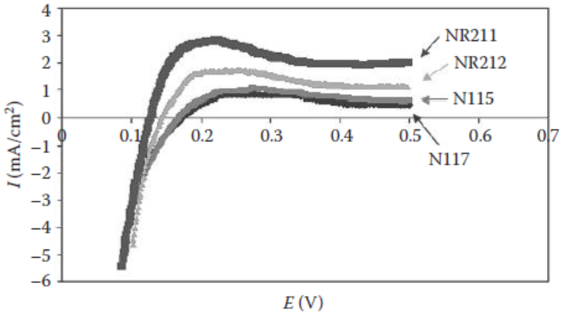
Being $N_2$ flow at the cathode, all of the current produced is solely attributable to the electrochemical oxidation. The voltage sweeps polarises the cathode until a value of limiting current is reached, which value is invariant to additional polarisation. Usually, the limiting current value is stabilised above 0.3 – 0.35 V, when no more effects related to hydrogen adsorption/desorption on platinum take place. The rate of fuel or oxidant crossover to the other electrode can be estimated using the maximum current measured during the polarization.

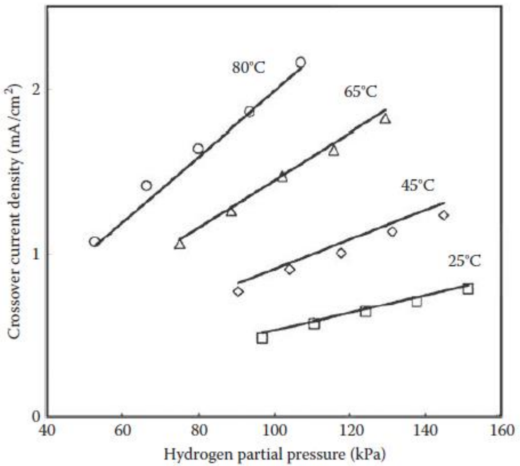
In this framework, the hydrogen crossover flux can be related to the limiting current density value via the Faraday's law:

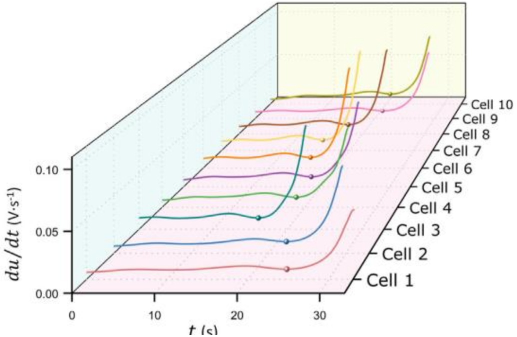
$$j_{cov} = \frac{i_{lim}}{nF}$$



# LINEAR SWEEP VOLTAMMETRY



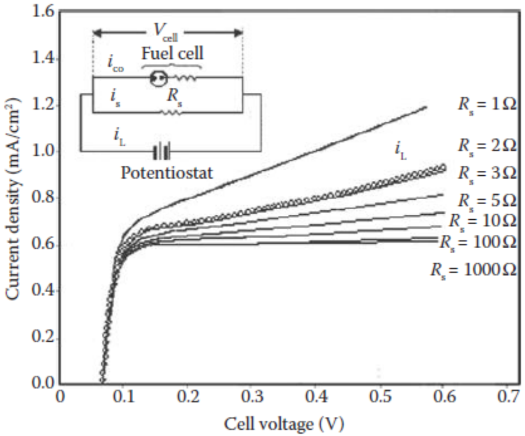




Li S. et al., International Journal of Hydrogen Energy, 47, 92, (2022), 39225-39238

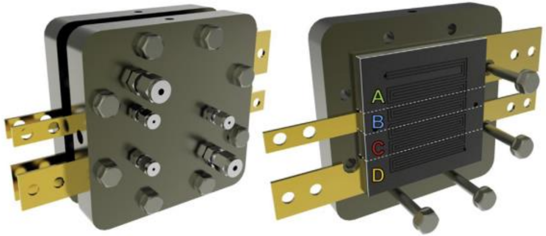
# LINEAR SWEEP VOLTAMMETRY

LSV can be also used to quantify internal short circuits over the electrolyte. Finite electrical shorts between anode and cathode may occur as a result of electrolyte thinning or pinhole formation. Short circuit current linearly increases with the applied voltage and is inversely proportional to the resistance of the short.

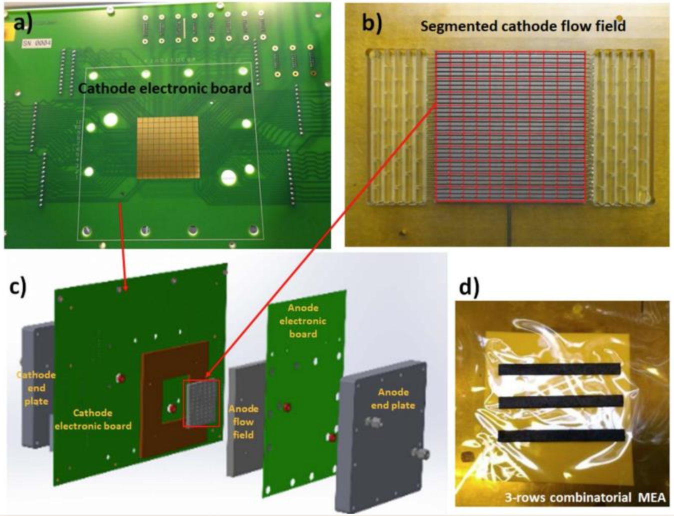


# 7. CURRENT MAPPING

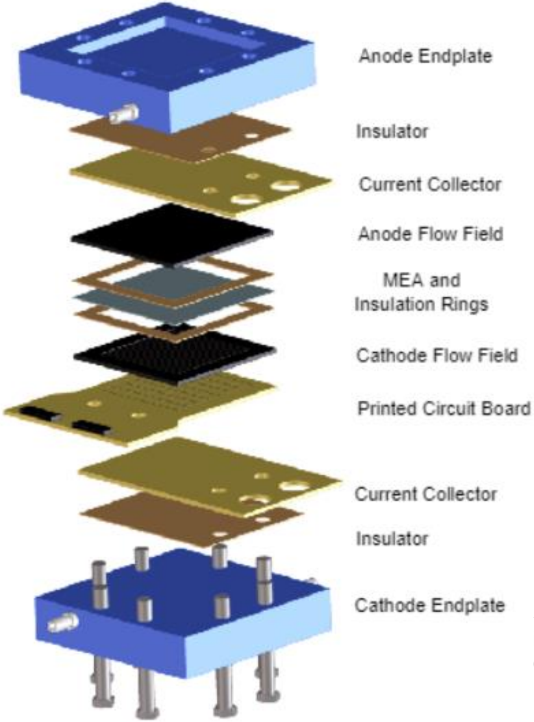
# SEGMENTED FUEL CELLS



Rabissi C. et al., Journal of Power Sources 397 (2018) 361–373



Osmieri S. et al., Journal of Power Sources 452 (2020), 227829



Sousa T. et al., (2011) Advances in Hydrogen Energy Technologies: Oportunities and Challenges in a Hydrogen Economy 4th International Seminar

# SEGMENTED FUEL CELLS

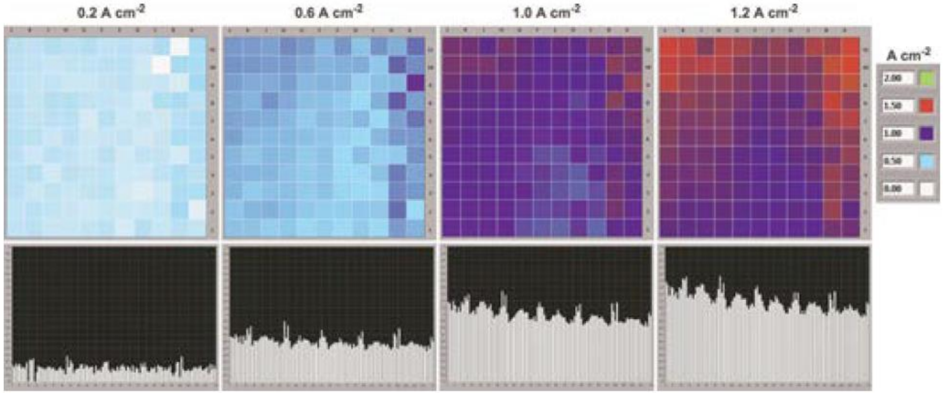


Fig. 6 SegCell wet current density distributions for pristine MEA at 0.2, 0.6, 1, and 1.2 A cm$^{-2}$ total cell current density.

Philipps A. *et al.*, Fuel Cell, 17, 3, (2017), 288-298

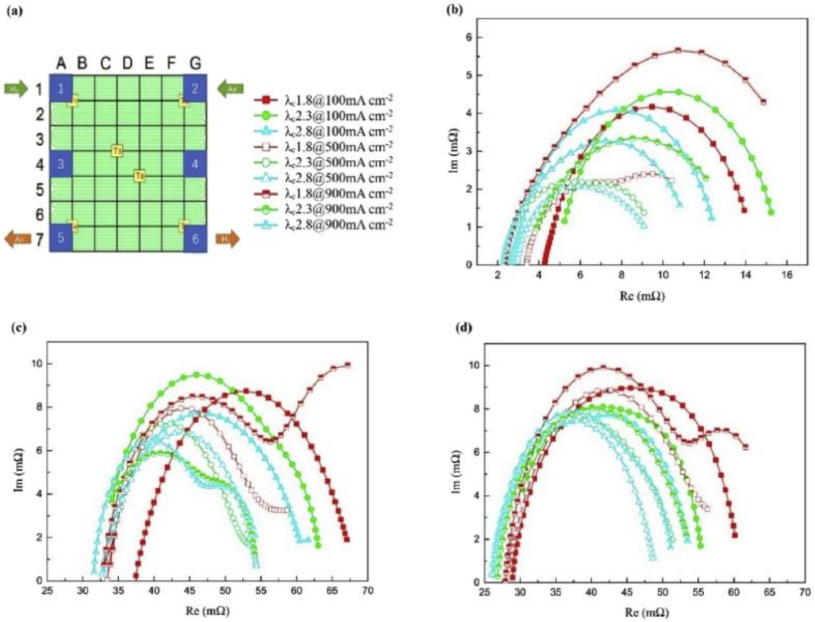
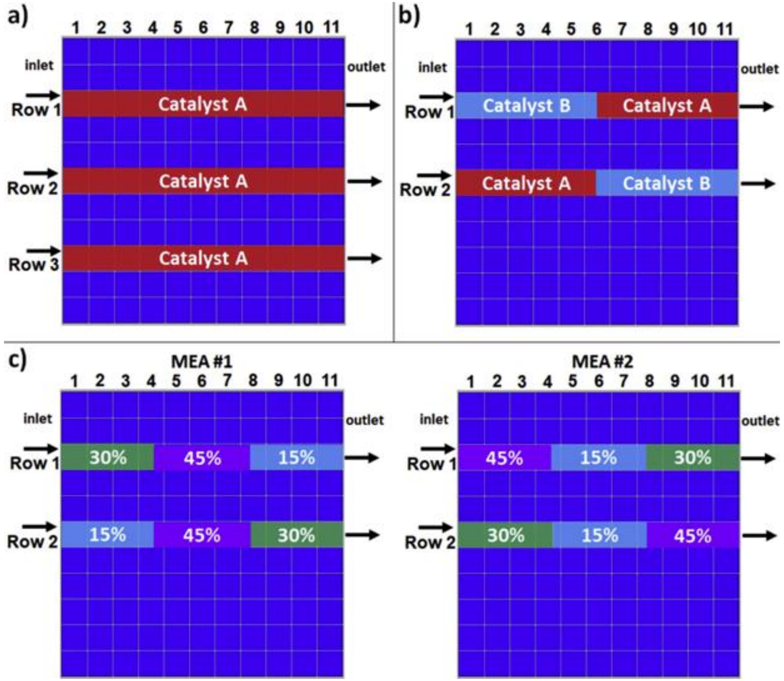


Fig. 5. (a) Distribution of the six special segments of the print circuit board; (b) Nyquist plot of the EIS results from the entire cell; (c) Nyquist plot of the local EIS results of Seg. 2 (cathode inlet); (d) Nyquist plot of the local EIS results of Seg. 5 (cathode outlet) [140]. Reproduced with permission from Ref. [140], Copyright 2019, Elsevier.

Z. Tang *et al.*, Journal of Power Sources 468 (2020) 228361

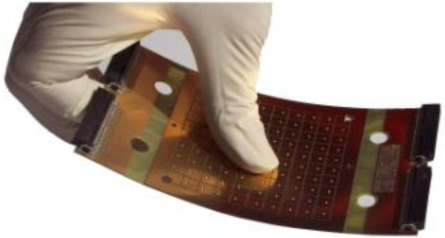
# SEGMENTED FUEL CELLS



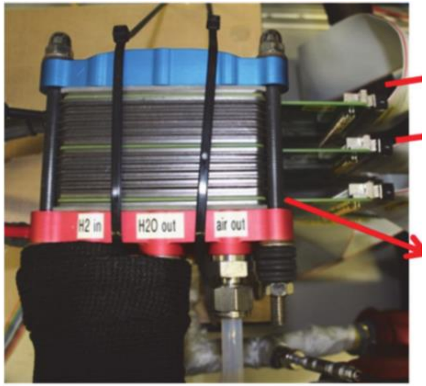
Osmieri S. et al., Journal of Power Sources 452 (2020), 227829

# CURRENT MAPPING

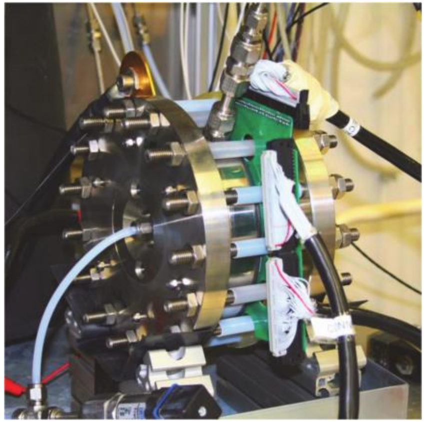
The shunt-resistance-based method offers simplicity and adaptability to different technologies



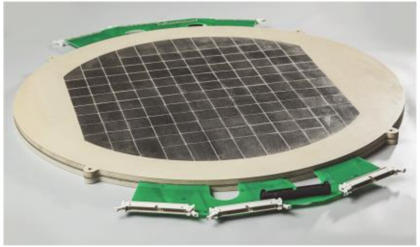
PEMFC stack



PEMWE stack



AWE



http://www.splusplus.com/

Biswas I. et al., Energies,12, (2020), 2301

# CURRENT MAPPING

R.7

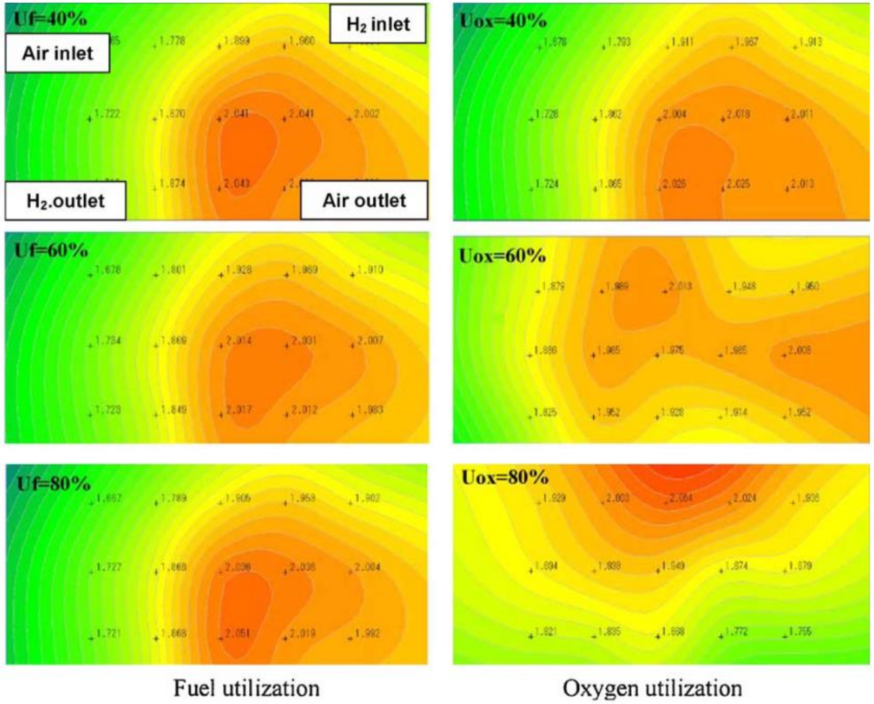


**Fig. 11.** Current distributions at different gas utilizations. Left figures—the influence of fuel utilization at a constant oxygen utilization of 40%. Right figures—the influence of oxygen utilization at a constant fuel utilization of 75%. Average current density is set at 0.25 A cm$^{-2}$. The temperatures are kept at a constant of 75 °C by circulation of coolant. Dew points of gases are kept at a constant of 65 °C.Adapted from Ref. [37] with permission.

# CURRENT MAPPING

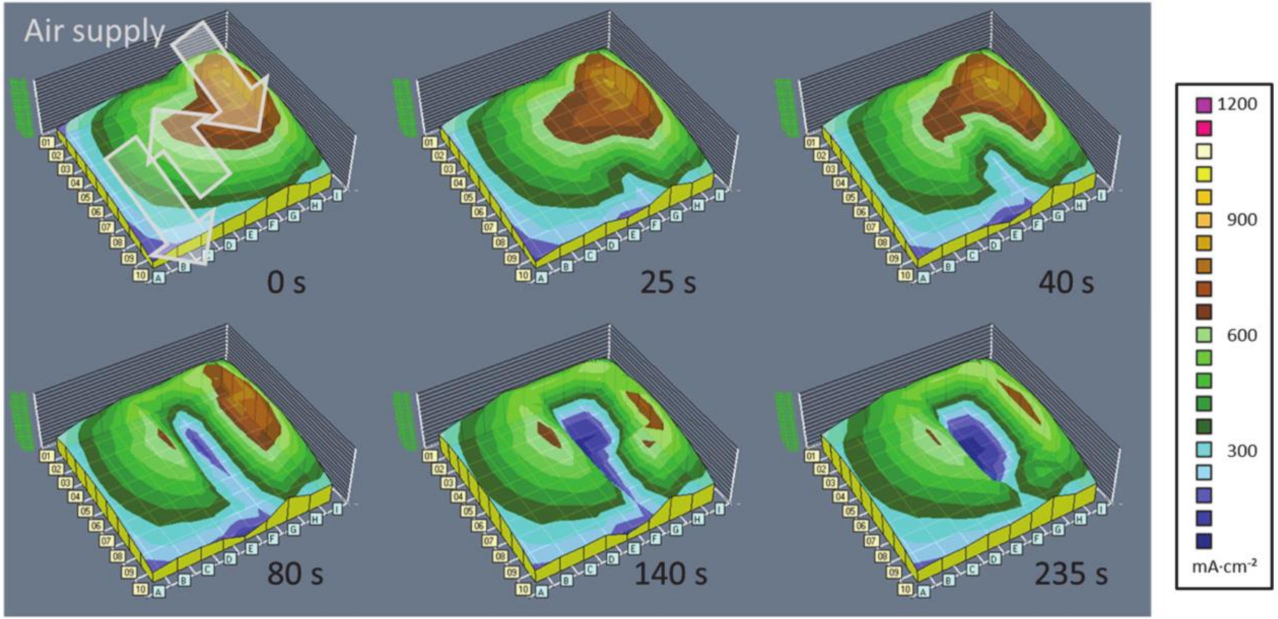


**Figure 2.** Series of current density mappings in a PEM fuel cell showing the local deactivation due to condensation of liquid water. (MEA: CCM by IonPower, 0.6 $mg_{Pt}/cm^2$, GDL: SGL Sigracet 25BC, 88 °C, 1.5 bar, 100% rH, stoichiometry: $H_2$: 1.5, air 2.0, constant voltage 680 mV).

# CURRENT MAPPING

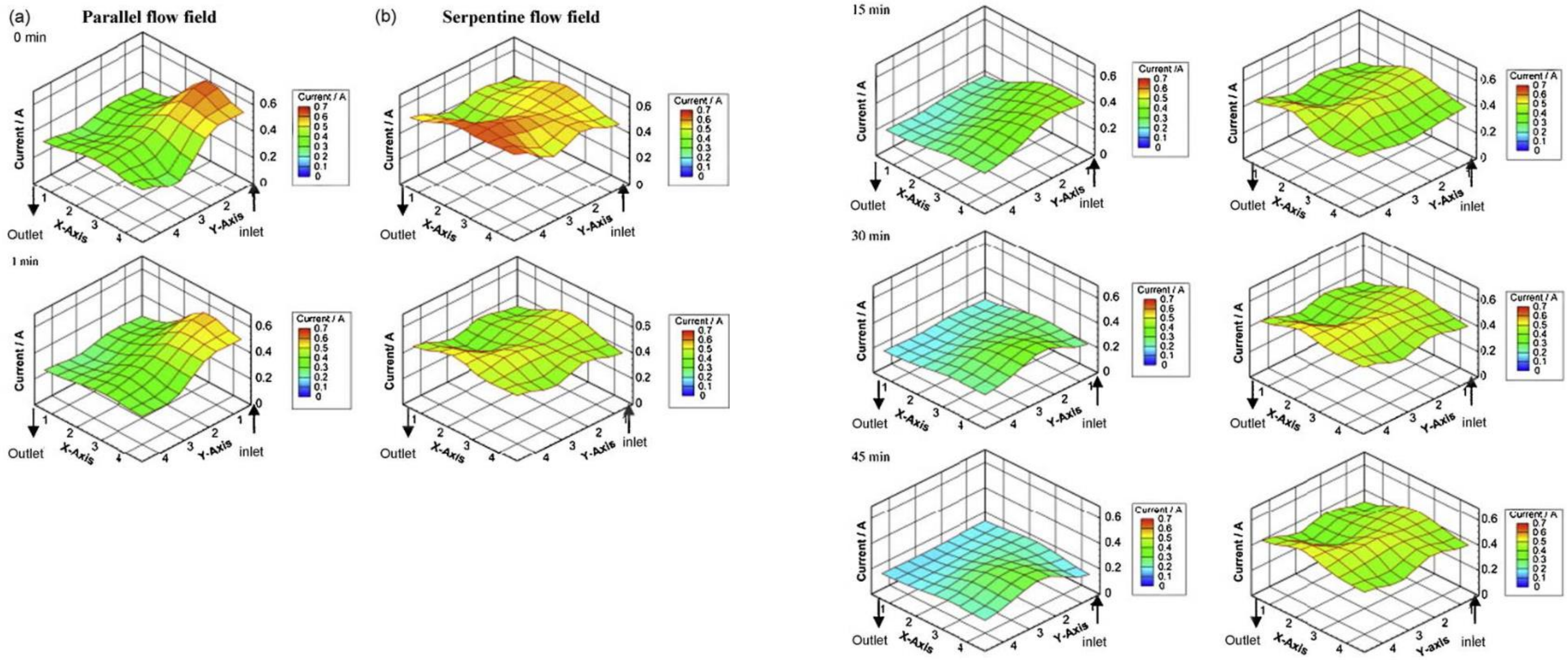


**Fig. 10.** Transient developments of the local current distribution, (a) parallel flow field and (b) serpentine flow field, $U$ = 0.5 V, $T_{cell}$ = 40 °C, $\lambda_{H_2} = 1.5$ and $\lambda_{O_2} = 2.5$.From Ref. [52] with permission.

# CURRENT MAPPING

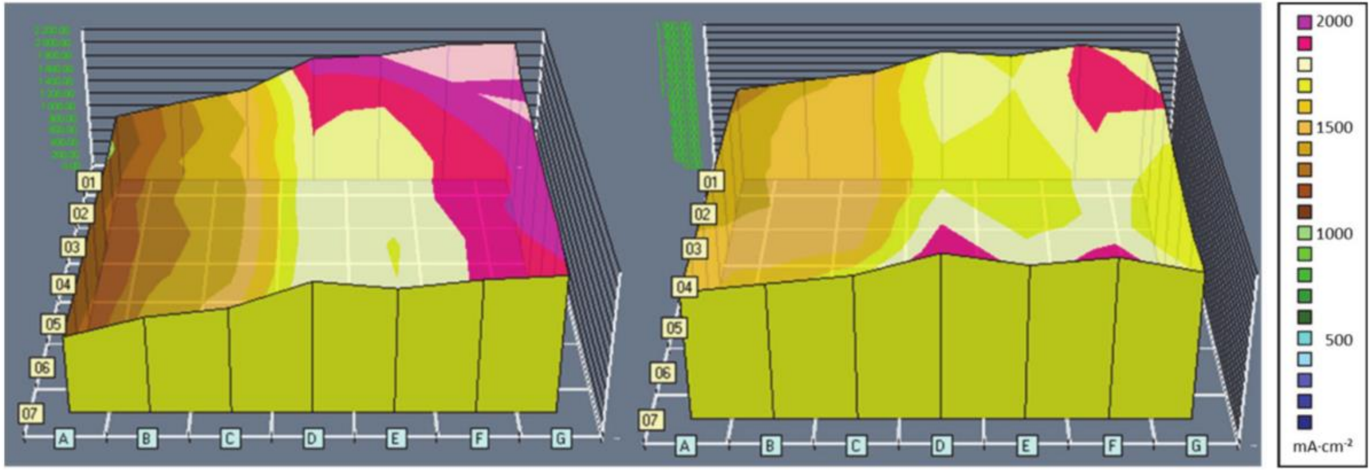


**Figure 5.** Current density distributions at an average of 1.6 A/cm$^2$ within a lab single cell, as recorded with segmented cell technology: The distribution with a laser-patterned GDL is more homogeneous (right) than without (left) (JM MEA02476, SGL Carbon 24BC, 80 °C, 50% rH, 1.5 bar, stoichiometry $H_2$: 1.5, air: 2, single serpentine flow field, co-flow configuration, gas inlet at upper right corner G1).

# CURRENT MAPPING

R.8

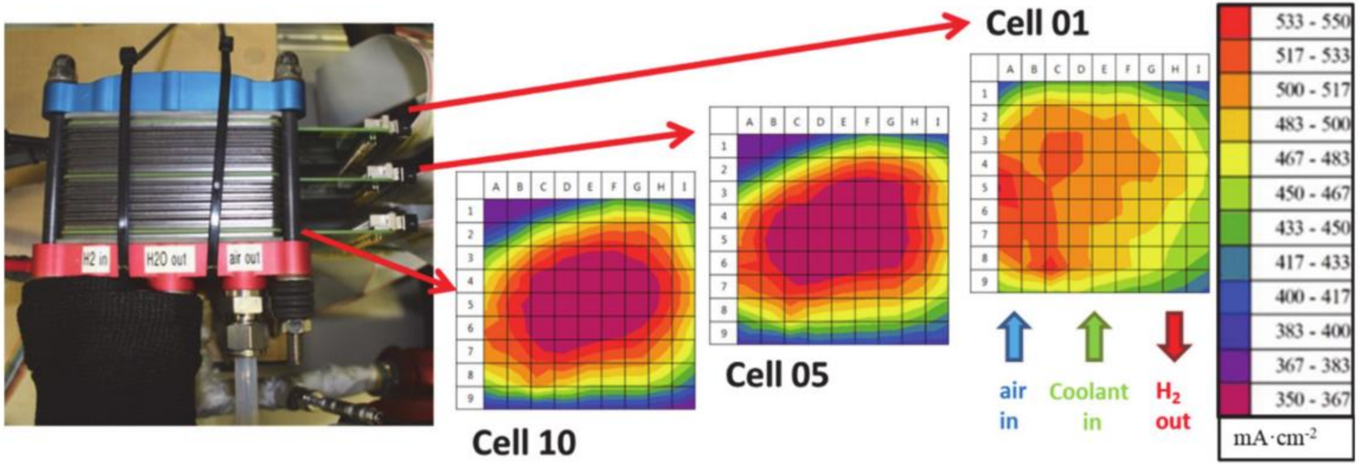
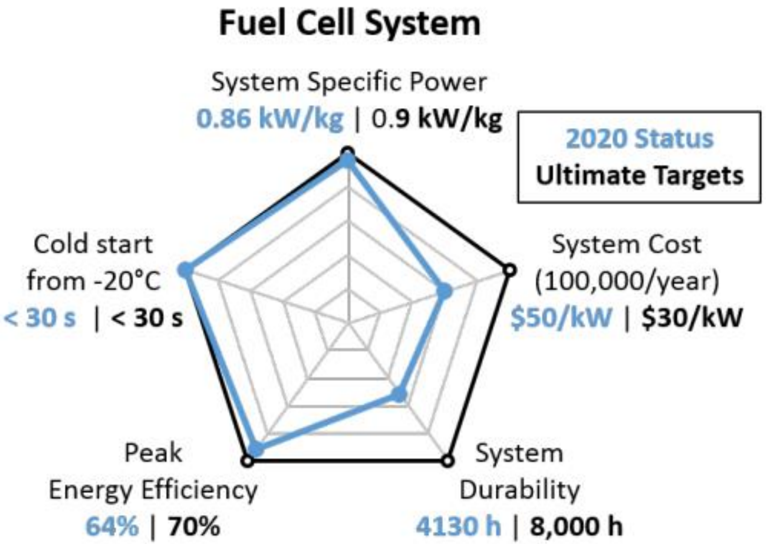


**Figure 9.** Stationary fuel cell stack (ten cells) with three integrated measurement boards for locally resolved current density measurements at the ends and in the centre. The highest difference between 350 mA/cm$^2$ (purple) and 550 mA/cm$^2$ (red) is observed in cell #10, while the other observed parts of the stack are more homogeneous.

# 8. ACCELERATED STRESS TESTS FOR FUEL CELLS AND WATER ELECTROLYSERS

# AST FOR PEMFC



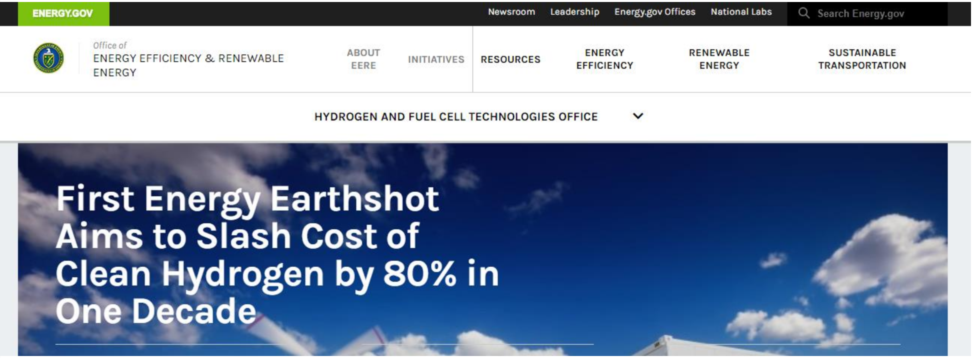
https://www.hydrogen.energy.gov/pdfs/20005-automotive-fuel-cell-targets-status.pdf

Accelerated Stress Tests (AST) consists in protocols defining fuel cell key operational parameters such as pressure, temperature, contamination concentration, contamination exposure, test duration, and the loading conditions which should be measured and applied to fairly compare fuel cell devices.

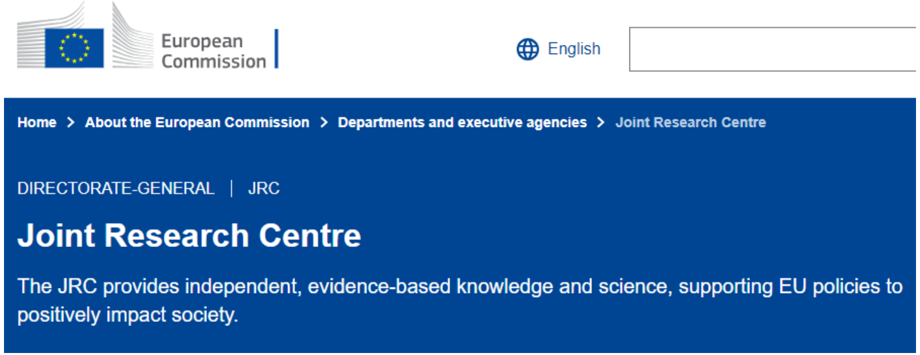
# AST FOR PEMFC

They are structured to ensure that the data generated and supplied are:

- Applicable: test results are relevant to the needs of the industry.
- Repeatable: test results could be duplicated under the same conditions, with the same type of equipment
- Reproducible: test results could be duplicated under the same conditions, with a different type of equipment.
- Scalable and serviceable: sufficient information is provided such that single cell test data can be scaled up to represent a full-size stack.



First Energy Earthshot Aims to Slash Cost of Clean Hydrogen by 80% in One Decade



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Joint Research Centre

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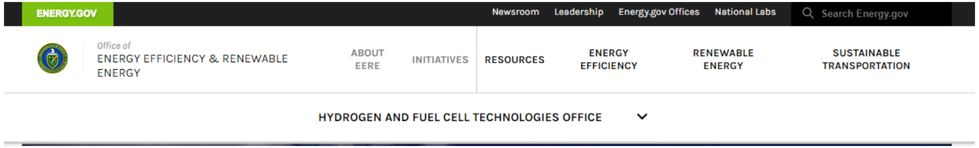
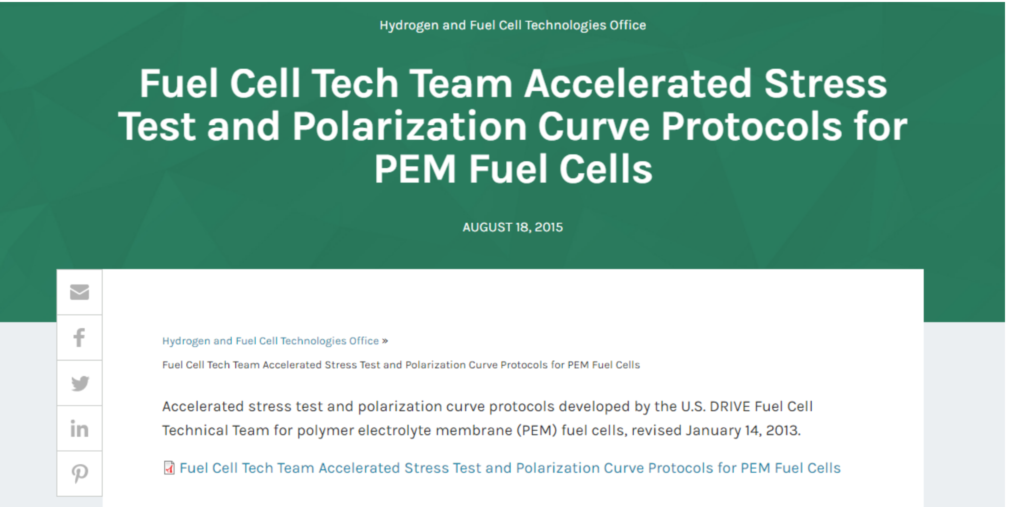
# AST FOR PEMFC




Hydrogen and Fuel Cell Technologies Office

## Fuel Cell Tech Team Accelerated Stress Test and Polarization Curve Protocols for PEM Fuel Cells

AUGUST 18, 2015

Hydrogen and Fuel Cell Technologies Office »
Fuel Cell Tech Team Accelerated Stress Test and Polarization Curve Protocols for PEM Fuel Cells

Accelerated stress test and polarization curve protocols developed by the U.S. DRIVE Fuel Cell Technical Team for polymer electrolyte membrane (PEM) fuel cells, revised January 14, 2013.

Fuel Cell Tech Team Accelerated Stress Test and Polarization Curve Protocols for PEM Fuel Cells

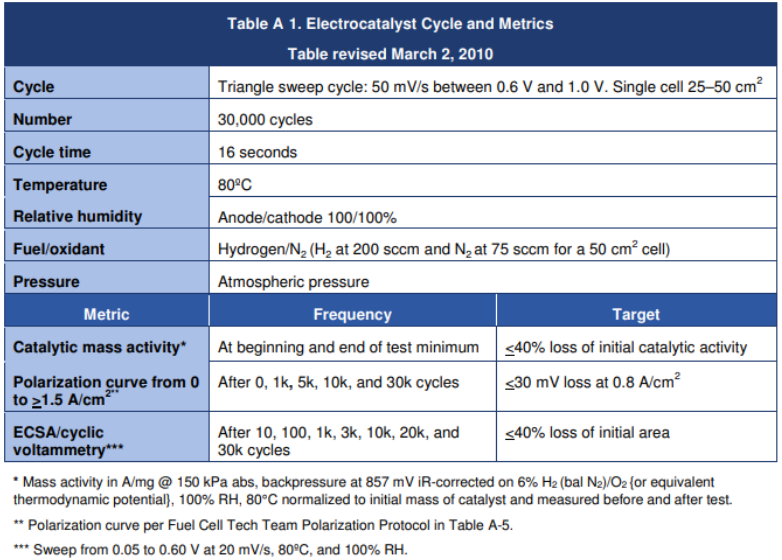


**Table A 1. Electrocatalyst Cycle and Metrics**
**Table revised March 2, 2010**

| | | |
|---|---|---|
| **Cycle** | Triangle sweep cycle: 50 mV/s between 0.6 V and 1.0 V. Single cell 25–50 $cm^2$ | |
| **Number** | 30,000 cycles | |
| **Cycle time** | 16 seconds | |
| **Temperature** | 80ºC | |
| **Relative humidity** | Anode/cathode 100/100% | |
| **Fuel/oxidant** | Hydrogen/$N_2$ ($H_2$ at 200 sccm and $N_2$ at 75 sccm for a 50 $cm^2$ cell) | |
| **Pressure** | Atmospheric pressure | |
| **Metric** | **Frequency** | **Target** |
| **Catalytic mass activity*** | At beginning and end of test minimum | ≤40% loss of initial catalytic activity |
| **Polarization curve from 0 to ≥1.5 $A/cm^2$**** | After 0, 1k, 5k, 10k, and 30k cycles | ≤30 mV loss at 0.8 $A/cm^2$ |
| **ECSA/cyclic voltammetry***** | After 10, 100, 1k, 3k, 10k, 20k, and 30k cycles | ≤40% loss of initial area |

* Mass activity in A/mg @ 150 kPa abs, backpressure at 857 mV iR-corrected on 6% $H_2$ (bal $N_2$)/$O_2$ {or equivalent thermodynamic potential}, 100% RH, 80°C normalized to initial mass of catalyst and measured before and after test.

** Polarization curve per Fuel Cell Tech Team Polarization Protocol in Table A-5.

*** Sweep from 0.05 to 0.60 V at 20 mV/s, 80ºC, and 100% RH.

# AST FOR PEMFC

**Home** > **About the European Commission** > **Departments and executive agencies** > Joint Research Centre

DIRECTORATE-GENERAL | JRC

## Joint Research Centre

The JRC provides independent, evidence-based knowledge and science, supporting EU policies to positively impact society.

JRC SCIENCE FOR POLICY REPORT

EU HARMONISED TEST PROTOCOLS FOR PEMFC MEA TESTING IN SINGLE CELL CONFIGURATION FOR AUTOMOTIVE APPLICATIONS

Georgios Tsotridis, Alberto Pilenga, Giancarlo De Marco, Thomas Malkow

2015

EUR 27632 EN

JRC TECHNICAL REPORT

Development of reference hardware for harmonised testing of PEM single cell fuel cells

Bednarek, T, Tsotridis, G

2021

EUR 30592 EN

# AST FOR PEMFC

**Verification**
- Visual inspection
- Electrical insulation test
- Leak test

→ **Break-in (Activation)** → **Conditioning** → **Beginning of Test (V/I )** → **AST**
- **Membrane**
- **Catalyst**
- **GDL**
- **Bipolar plates**

→ **End of Test (V/I )**

Objectives

Measure leakage rate by pressure decrease in anode and cathode compartment and assure gas tightness of cell and test station.

Objectives

Activate the catalyst layer according to manufacturer's specifications.

Objectives

Assure that the cell voltage will be stable before the test initiation.

Objectives

Measure and test durability of FC components for defining its lifetime.

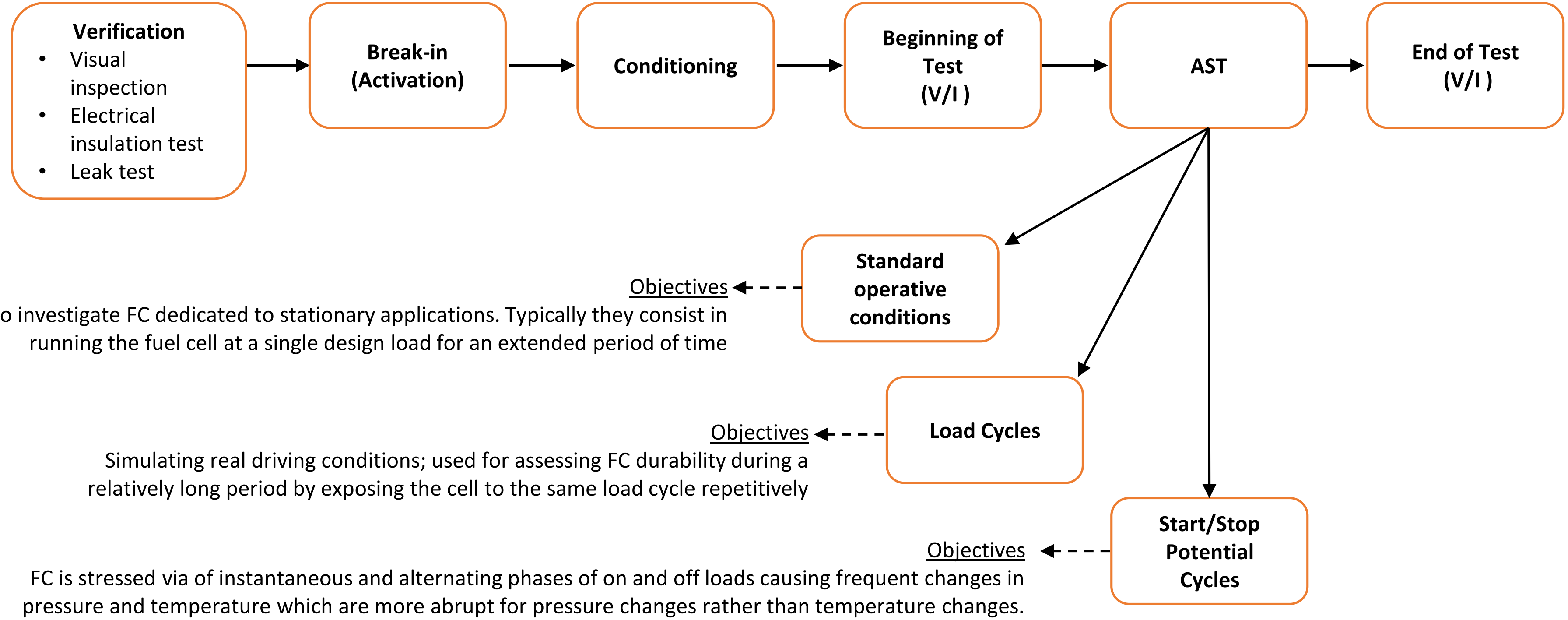
Durability is defined as the capability of the fuel cell to maintain its performance over a period of time, without significant irreversible changes to the initial recorded performance being detrimental.

Stability: ability to recover power loss during continuous operation. It is impacted by the evolution of operating conditions such as temperature or humidity.

Reliability: ability of a cell or stack to supply the wanted power under stated conditions for a period. That includes failure modes that can lead to performance below an acceptable level or to failures

R.9

# AST FOR PEMFC



**Verification**
- Visual inspection
- Electrical insulation test
- Leak test

**Break-in (Activation)**

**Conditioning**

**Beginning of Test (V/I )**

**AST**

**End of Test (V/I )**

**Standard operative conditions**

Objectives

To investigate FC dedicated to stationary applications. Typically they consist in running the fuel cell at a single design load for an extended period of time

**Load Cycles**

Objectives

Simulating real driving conditions; used for assessing FC durability during a relatively long period by exposing the cell to the same load cycle repetitively

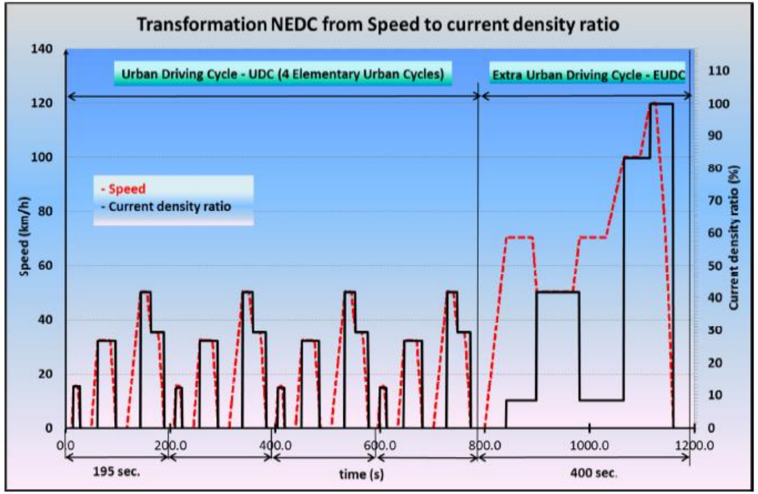
**Start/Stop Potential Cycles**

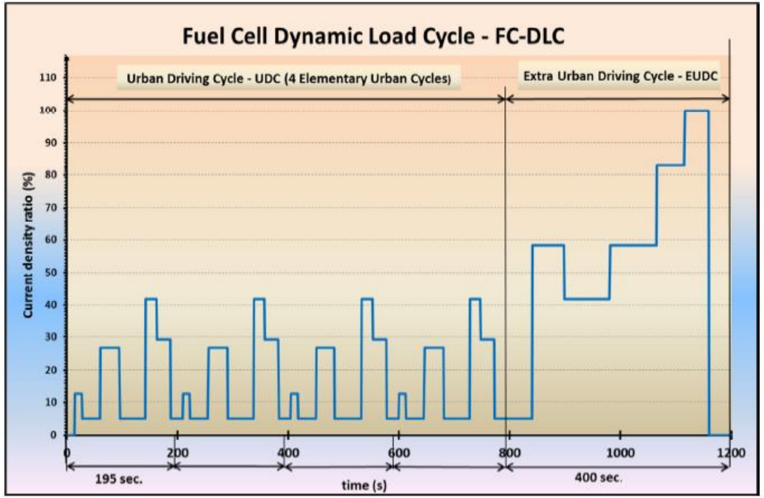
Objectives

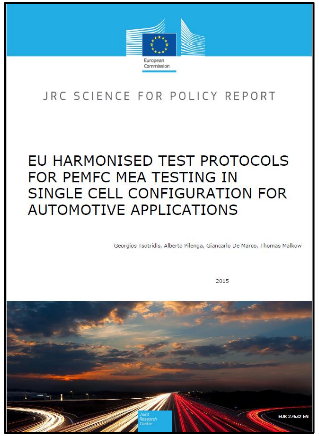
FC is stressed via of instantaneous and alternating phases of on and off loads causing frequent changes in pressure and temperature which are more abrupt for pressure changes rather than temperature changes.

# AST FOR PEMFC

## AST for catalyst durability: Load Cycling







JRC SCIENCE FOR POLICY REPORT

EU HARMONISED TEST PROTOCOLS FOR PEMFC MEA TESTING IN SINGLE CELL CONFIGURATION FOR AUTOMOTIVE APPLICATIONS

Georgios Tsotridis, Alberto Pilenga, Giancarlo De Marco, Thomas Malkow

2015

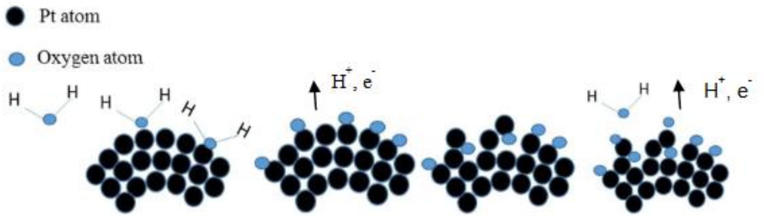
EUR 27632 EN

# AST FOR PEMFC

## AST for catalyst durability: Start/Stop Potential Cycles

It is known as the main mechanisms involved in catalyst degradation are:

- Pt dissolution, agglomeration, and/or detachment
- Carbon corrosion (oxidation) from the support
- Mechanical stress
- Pt catalyst contamination
- Pt oxidation



(a) Pt to Pt-$H_2O$ (b) Pt-$H_2O$ to PtO (c) PtO to OPt (d) OPt to $PtO_2$

Figure 1: Summary of the processes occurring during Pt oxidation

Arisetty S. et al., ECS Transactions, 69, 17, (2015), 273-289

**Table 1**

Fuel cell operating conditions affecting catalyst layer degradation.

| Catalyst layer degradation | Operating conditions |
|---|---|
| Carbon support corrosion | Cold start-up<br>Fuel starvation<br>Start/stop cycling<br>Potential cycling |
| Pt dissolution/agglomeration/detachment | Load cycling<br>Potential cycling<br>Temperature/Relative humidity (RH) cycling |
| Catalyst contamination | Impurities from supply reactants |
| Pt oxidation | RH cycling<br>Fuel starvation<br>Start/stop cycling |

# AST FOR PEMFC

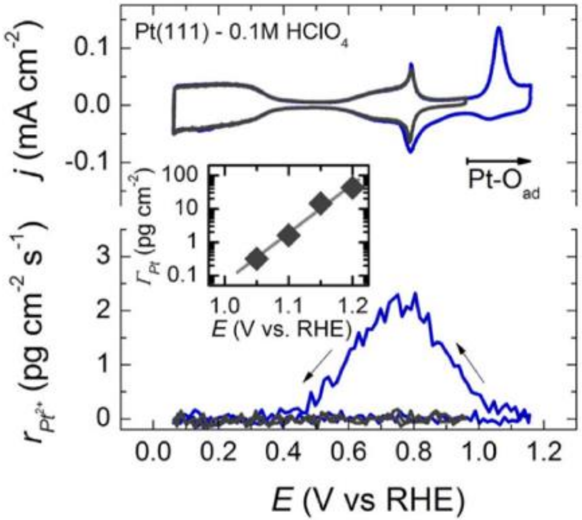


**Fig. 1.** Cyclic voltammetry and corresponding dissolution rate curves for Pt (111) in 0.1 M $HClO_4$ at 50 mV $s^{-1}$ at two potentials: 0.95 V (gray) and 1.15 V (blue). Inset curve shows the exponential relationship between the total amount of Pt dissolution and the potential where Pt oxide formation begins (Pt-Oad) [49]. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

Pt degradation can develop in two conditions. During OCV, when no current is drawn from the FC, being the cell potential around 1 V, in the region where Pt-O starts to form. Once switching to the operating condition (where the voltage reduces to about 0.6 V), Pt reduction can take place following three mechanisms:

$$Pt \rightarrow Pt^{+2} + 2e^-$$

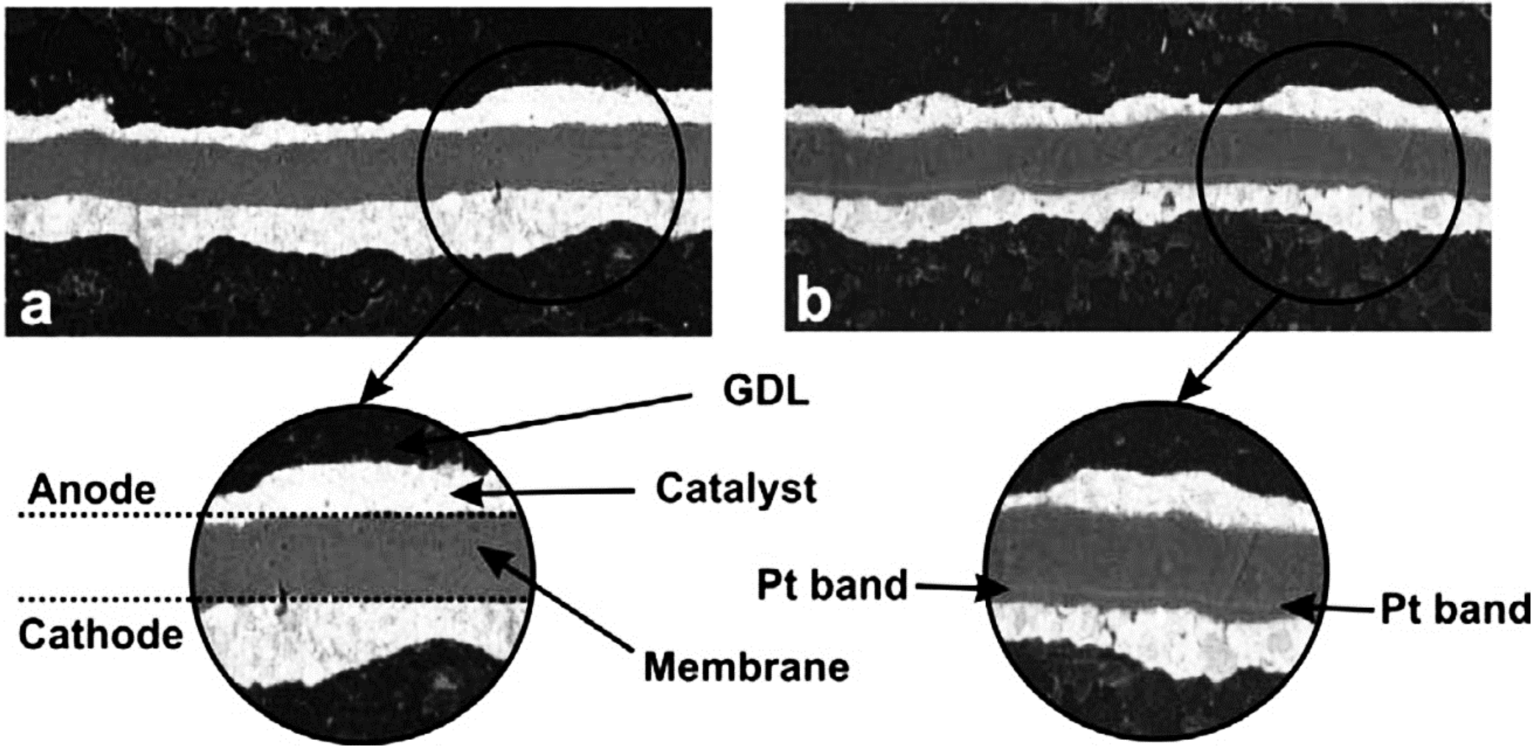
$$PtO + 2H^+ + 2e^- \rightarrow Pt^0 + H_2O$$

$$PtO^* + 2H^+ \rightarrow Pt^{+2} + H_2O$$

Leading to Pt dissolution. On the long term, from ex situ analysis, Pt-band formation can be found within the electrolyte.

Pt dissolution can also lead to catalyst coarsening and to particle detachment too once the potential is risen above 1.2 V, while C corrosion is always present and harsher, the higher the potential is.

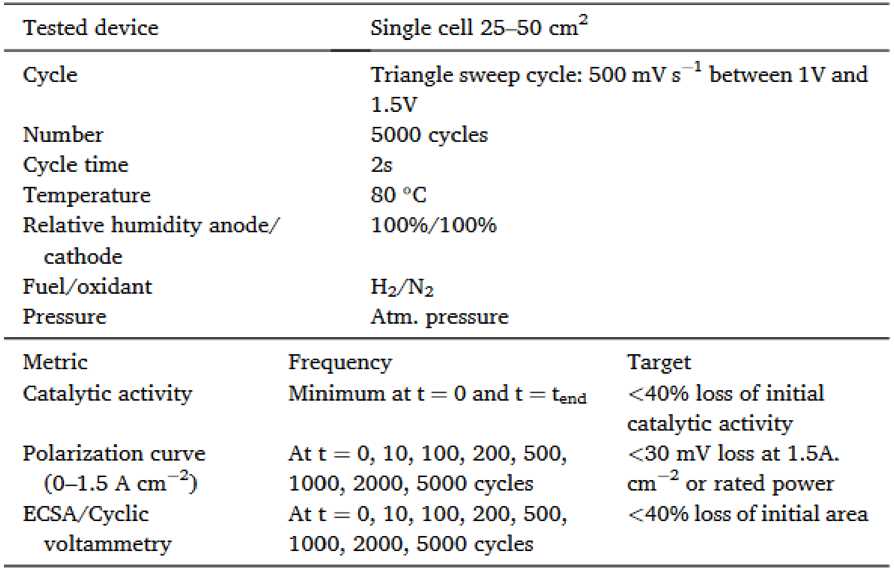
# AST FOR PEMFC



Macauley N. et al., ECS Electrochem. Lett. 2, (2013), F33

# AST FOR PEMFC

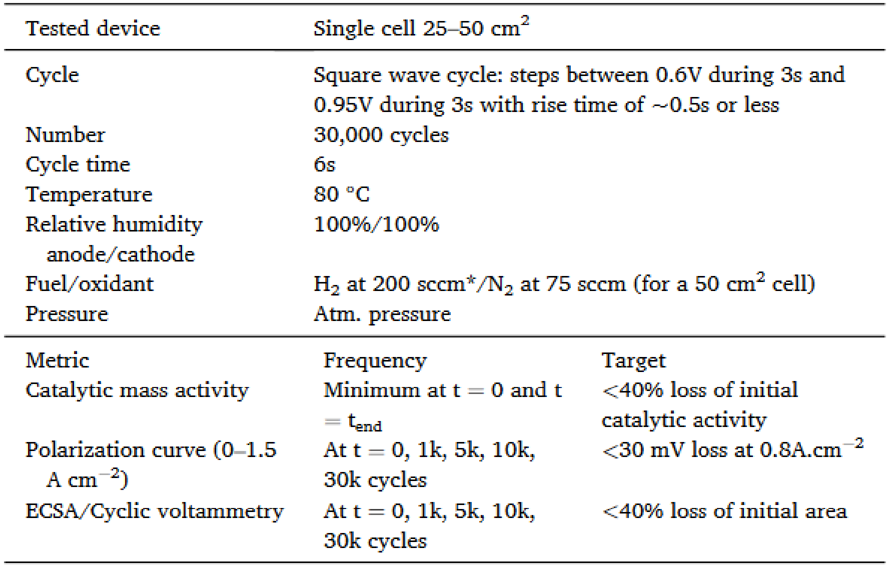
2017 US DoE catalyst support AST and metrics (adapted from Ref. [74]).



| Tested device | Single cell 25–50 cm$^2$ |
|---|---|
| Cycle | Triangle sweep cycle: 500 mV s$^{-1}$ between 1V and 1.5V |
| Number | 5000 cycles |
| Cycle time | 2s |
| Temperature | 80 °C |
| Relative humidity anode/ cathode | 100%/100% |
| Fuel/oxidant | $H_2/N_2$ |
| Pressure | Atm. pressure |

| Metric | Frequency | Target |
|---|---|---|
| Catalytic activity | Minimum at $t = 0$ and $t = t_{end}$ | <40% loss of initial catalytic activity |
| Polarization curve (0–1.5 A cm$^{-2}$) | At $t = 0$, 10, 100, 200, 500, 1000, 2000, 5000 cycles | <30 mV loss at 1.5A. cm$^{-2}$ or rated power |
| ECSA/Cyclic voltammetry | At $t = 0$, 10, 100, 200, 500, 1000, 2000, 5000 cycles | <40% loss of initial area |

2017 US DoE electrocatalyst AST and metrics (adapted from Ref. [74]).



| Tested device | Single cell 25–50 cm$^2$ |
|---|---|
| Cycle | Square wave cycle: steps between 0.6V during 3s and 0.95V during 3s with rise time of ~0.5s or less |
| Number | 30,000 cycles |
| Cycle time | 6s |
| Temperature | 80 °C |
| Relative humidity anode/cathode | 100%/100% |
| Fuel/oxidant | $H_2$ at 200 sccm*/$N_2$ at 75 sccm (for a 50 cm$^2$ cell) |
| Pressure | Atm. pressure |

| Metric | Frequency | Target |
|---|---|---|
| Catalytic mass activity | Minimum at $t = 0$ and $t = t_{end}$ | <40% loss of initial catalytic activity |
| Polarization curve (0–1.5 A cm$^{-2}$) | At $t = 0$, 1k, 5k, 10k, 30k cycles | <30 mV loss at 0.8A.cm$^{-2}$ |
| ECSA/Cyclic voltammetry | At $t = 0$, 1k, 5k, 10k, 30k cycles | <40% loss of initial area |

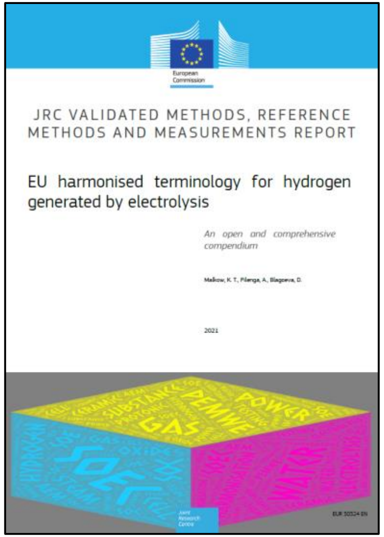
# AST FOR PEMFC

- High potentials imply Pt particle size growing.
- High temperature and high relative humidity induce Pt particle size rate of growth increasing.
- High potentials and low relative humidity imply fast carbon corrosion.

- The longer the OCV holding time is, the higher is the degradation, unlike a long-term idling that do not largely affect the fuel cell performance. The load cycling enhances the Pt aggregation.
- The degradation of the electrocatalyst under load cycling with long OCV period should be reduced, when running at low current density and low humidity
- A high potential operation (0.73V) enhances the Pt oxidation and the Pt dissolution by using an oxide film.
- Low RH condition suppresses both the Pt oxidation and the Pt dissolution.

# AST FOR PEMWE

No official standards or regulations have been issued yet; the JRC has started drafting the first guidelines, basing on what has been already done with FC.



European Commission

JRC VALIDATED METHODS, REFERENCE METHODS AND MEASUREMENTS REPORT

EU harmonised terminology for hydrogen generated by electrolysis

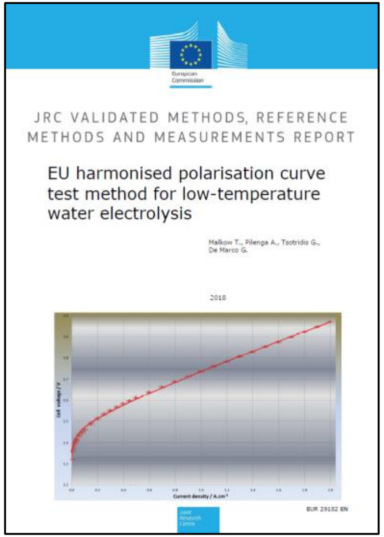
*An open and comprehensive compendium*

Malkow, K. T., Pilenga, A., Blagoeva, D.

2021

Joint Research Centre

EUR 30324 EN



European Commission

JRC VALIDATED METHODS, REFERENCE METHODS AND MEASUREMENTS REPORT

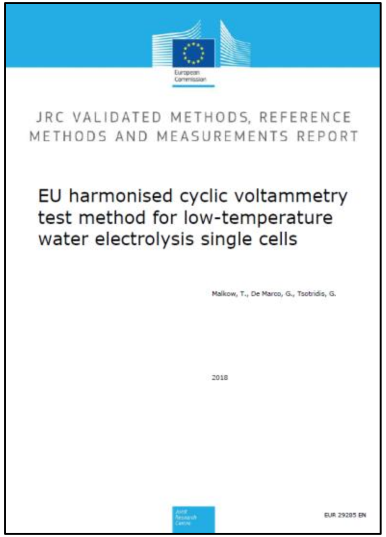
EU harmonised polarisation curve test method for low-temperature water electrolysis

Malkow T., Pilenga A., Tsotridis G., De Marco G.

2018

Joint Research Centre

EUR 29182 EN



European Commission

JRC VALIDATED METHODS, REFERENCE METHODS AND MEASUREMENTS REPORT

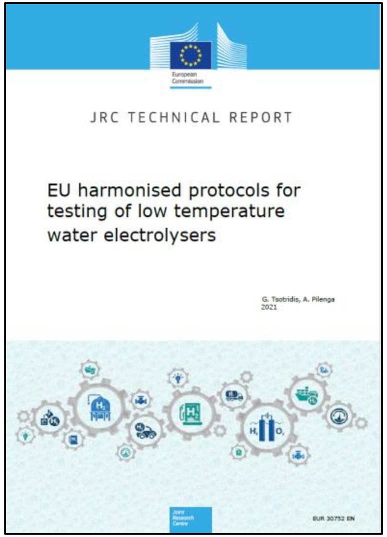
EU harmonised cyclic voltammetry test method for low-temperature water electrolysis single cells

Malkow, T., De Marco, G., Tsotridis, G.

2018

Joint Research Centre

EUR 29285 EN



European Commission

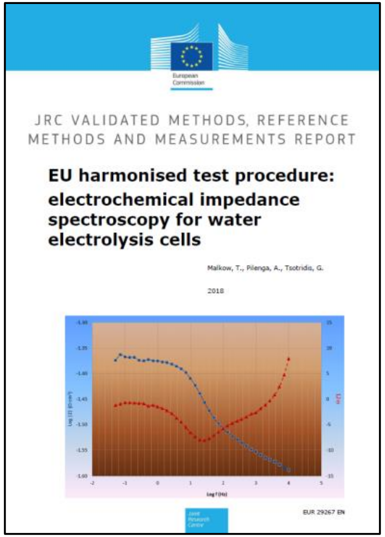
JRC TECHNICAL REPORT

EU harmonised protocols for testing of low temperature water electrolysers

G. Tsotridis, A. Pilenga
2021

Joint Research Centre

EUR 30752 EN



European Commission

JRC VALIDATED METHODS, REFERENCE METHODS AND MEASUREMENTS REPORT

**EU harmonised test procedure: electrochemical impedance spectroscopy for water electrolysis cells**

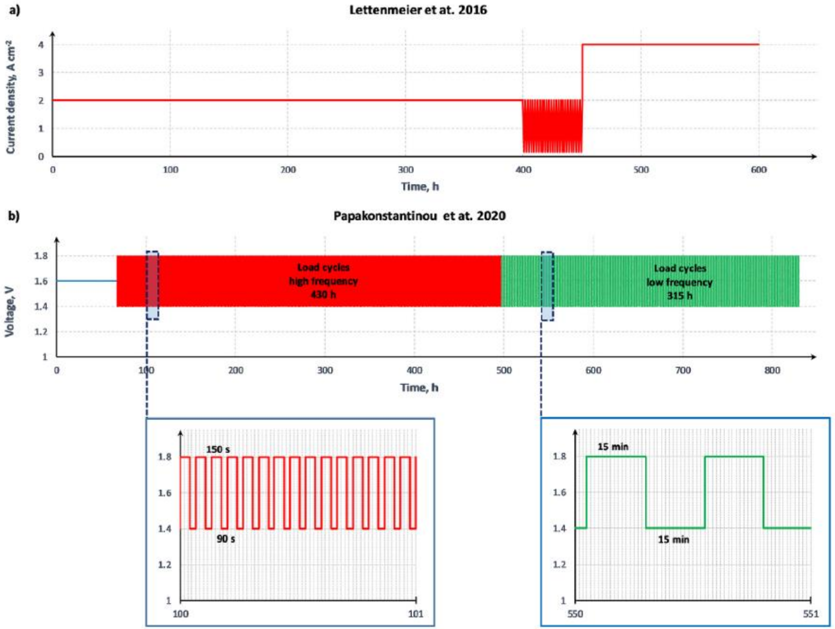
Malkow, T., Pilenga, A., Tsotridis, G.

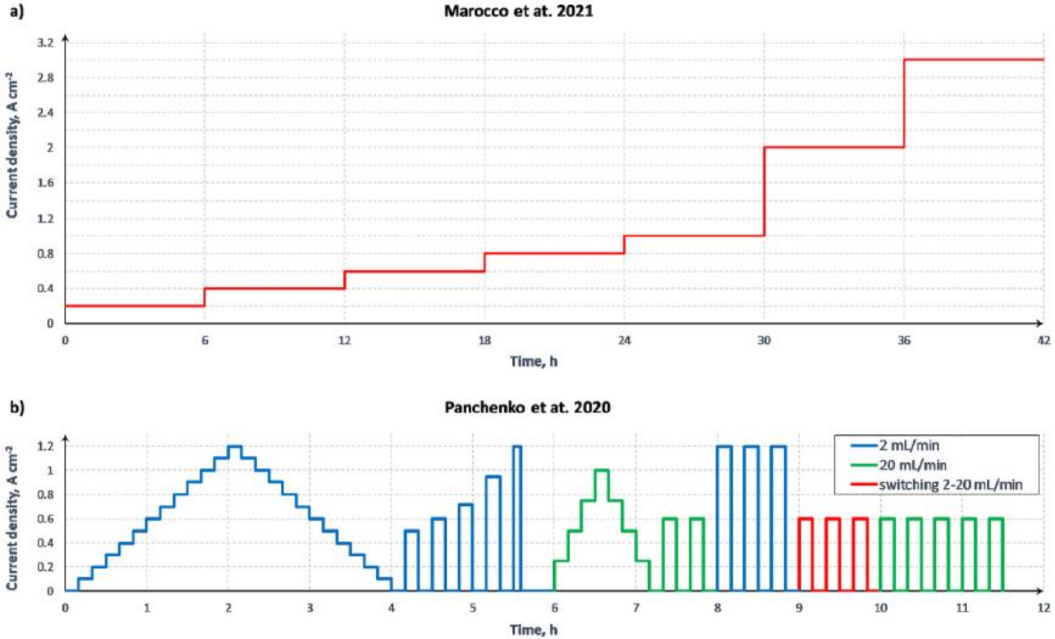
2018

Joint Research Centre

EUR 29267 EN

# AST FOR PEMWE





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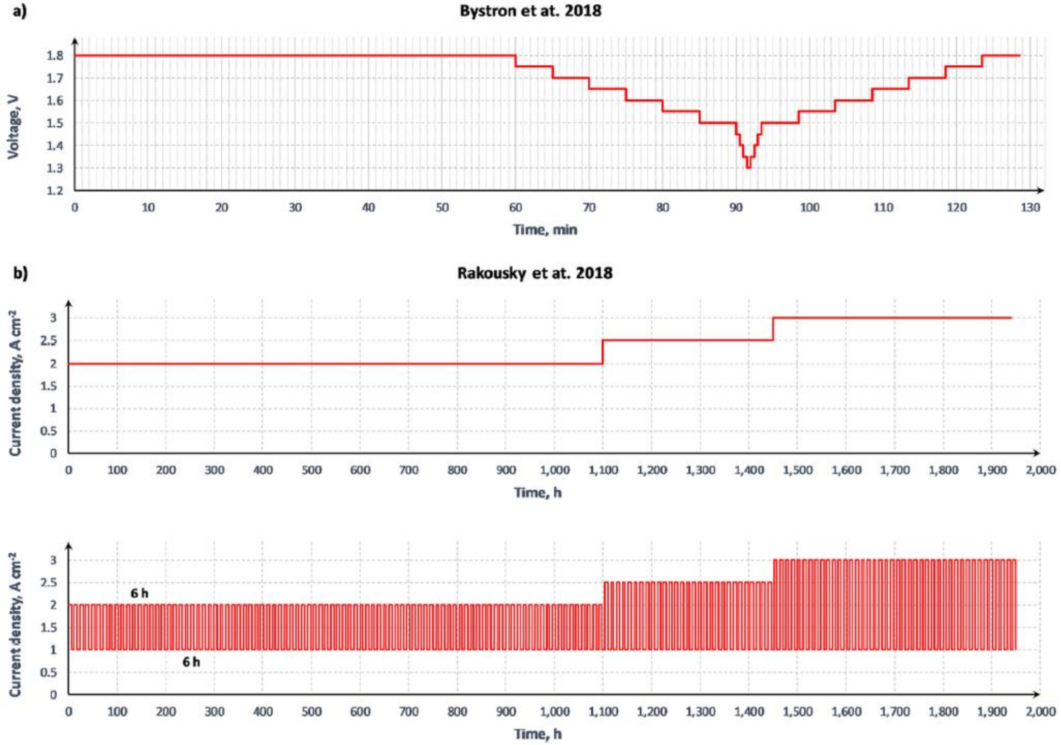

# AST FOR PEMWE

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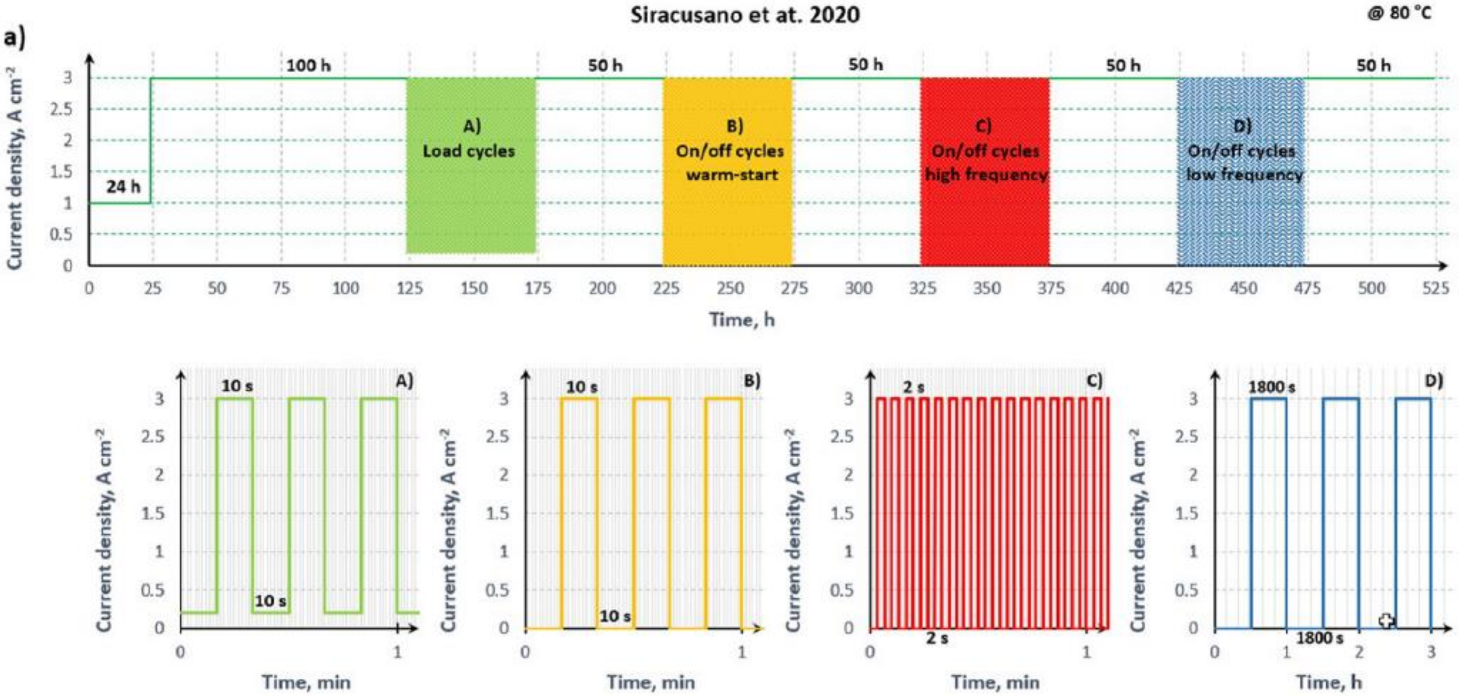

# AST FOR PEMWE

In general, to date it was reported:

- Higher temperature causes faster degradation, but also higher performance
- Dissolution of the catalyst layer, thinning of the membrane, growth of the oxide layer on the metal components of the electrolyzer and contamination of the membrane can be extracted as the most important degradation mechanisms.
- However, voltage cycling affects this degradation process more than current cycling.

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