Application Note – Automated PEMFC Durability Testing with an 850e, 885 Fuel Cell PSTAT, Auto-Multi Gas Unit and Wet-Dry Humidifier By-Pass

Introduction

Durability is a key performance property of polymer electrolyte membrane fuel cells (PEMFCs). The U.S. Department of Energy along with USCAR's Fuel Cell Technical Team have developed accelerated stress tests (AST) to evaluate the durability of low temperature PEMFCs [1]. Test protocols exist that are intended to evaluate the mechanical durability of the membrane, the durability of the catalyst support, and the durability of the electrocatalyst (among others). Details of the U.S. DOE / USCAR AST protocols are shown in Figure 1 and Figure 2 and described in [1, 2].

It is easy to implement automated execution of the AST protocols using Scribner's 850e Fuel Cell Test System, 885 Fuel Cell Potentiostat (PSTAT) and 850 Auto-Multi Gas Unit.

FuelCell® software setup files for automated execution of electrocatalyst, catalyst support and membrane mechanical durability ASTs per the U.S. DOE / USCAR Fuel Cell Technical Team are available from Scribner's website:

***For ease of downloading, the file extension has been changed to '.zip'. Once downloaded, rename the file extension to '.fc3'*

FuelCell setup file for **Electrocatalyst Durability**

> *www.scribner.com/files/fuelcell/DOE Electrocatalyst Durability Protocol_25cm2.fc3*

FuelCell setup file for **Catalyst Support Durability**

> *www.scribner.com/files/fuelcell/DOE Catalyst Support Durability Protocol_25cm2.fc3*

FuelCell setup file for **Membrane Mechanical Durability**

> *www.scribner.com/files/fuelcell/DOE Membrane Mechanical Durability Protocol_25cm2.fc3*

If you find an error or an improved method of implementing these protocols in *FuelCell*, please let us know by emailing *fuelcellsupport@scribner.com*.

Notes:

- The *FuelCell* software setup files assume a 25 cm^2 cell. If testing a cell of different active area, the anode and cathode flow rates and current values used for the polarization curve must be modified.

- The *FuelCell* software setup files for the **Electrocatalyst** and **Catalyst Support Durability** test protocols assume the 850e unit is equipped with an 850 Auto-Multi Gas unit with H_2 plumbed (connected) to Anode Port A, Air plumbed to Cathode Port A and N_2 connected to Cathode Port B. All other ports are unused.

- The *FuelCell* software setup file for the **Membrane Mechanical Durability** test protocol assumes the unit is equipped with an 850 Auto-Multi Gas Unit with H_2 plumbed (connected) to Anode Port A, N_2 plumbed to Anode Port B, Air plumbed to Anode Port C, Air plumbed to Cathode Port A and N_2 plumbed to Cathode Port B. Cathode Port C is not used.

- The **Membrane Mechanical Durability** AST is based on cycling between super-saturated and dry gas and therefore requires that the 850e is equipped with the Wet-Dry Humidifier By-Pass Option. 850e units equipped with this option have "WD" listed under the Options section of the serial plate located on the rear panel of the unit. Contact Scribner for additional information or clarification if you are unsure if your 850e has this option.

- Under H_2 / N_2 conditions, the cell voltage ~ 0.1 V. To avoid a system shut down due to low cell voltage, in the Setup Cell menu, set the Minimum E (V) and Shut Down E (V) values to "-1". This effectively disables the low voltage alarms.

- Standard 885 PSTATs connect to the 850e via the Auxiliary Signals Connector have a maximum data acquisition rate of 0.1 s/point (10 points/s). High-speed 885 PSTATs use a USB connection to the host computer and have a maximum data acquisition rate = 0.01 s/point (100 points/s). The data sample rate in the 885 PSTAT Sweep Voltage experiments should be set accordingly.

- The *FuelCell* software setup files for the **Electrocatalyst** and **Catalyst Support Durability** tests consist of a series of experiment steps that execute:
    - Voltage sweeps under H_2 / N_2 conditions with the 885 PSTAT for the durability portion of test,
    - Setup Fuel Experiments that switch anode / cathode fuels between H_2 / N_2 and H_2 / Air depending on the experiment requirements,
    - Scan Current Experiments for the polarization curves with H_2 / Air,
    - Cyclic voltammograms with the 885 PSTAT under H_2 / N_2 for electrochemical surface area (ECSA) measurement,
    - Anode and cathode humidifier temperature changes for inlet gas relative humidity (RH) control as specified in the protocol,

    - Open Circuit Voltage and Constant Voltage Experiments for delays after gas switching and humidifier heat-up and cool-down periods.

- The *FuelCell* software setup files for **Membrane Mechanical Durability** consist of a series of experiment steps within nested repeat loops that execute:
    - Inner Repeat Loop: 360 repetitions, 2 min wet + 2 min dry (24 hour total). Contains Setup Fuel Experiments that switch anode / cathode fuels between wet (super-saturated) and dry Air with 2 min delays between switching. High flow rates are used during the RH cycling to facilitate rapid hydration and dehydration of the cell and membrane.
    - Outer Repeat Loop: 56 repetitions of the inner repeat loop and diagnostics (below) for total of 20,000 wet-dry cycles,
    - Slow voltage sweep experiment with the 885 PSTAT under H_2 / N_2 for the Crossover measurement,
    - Constant voltage experiment with the 885 PSTAT under N_2 / N_2 for the shorting resistance measurement.
    - PSTAT Open Circuit Voltage (OCV) Experiments for delays after gas type and flow rate switching.

- The Fuel Cell Tech Team Polarization Protocol calls for the performance curve to be measured at 150 kPa_a while the remainder of the tests are done at ambient pressure (zero back pressure). This is possible to implement in an automated fashion with the use of Scribner's Auto Back Pressure Unit. However, the *FuelCell* setup files above assume that the Auto BP unit is not available and therefore all tests are conducted at ambient pressure.

Disclaimer

Scribner does not claim that the *FuelCell* setup files are error free and assumes no liability for damage that may occur as a result of their use. Users should carefully review the experiment list and have a clear understanding of the purpose and function of each experiment step. Please contact Scribner (*fuelcellsupport@scribner.com* or +1-910-695-8884) if you have any questions.

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	80°C	
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, 80°C, and 100% RH.

Table A-2. Catalyst Support Cycle and Metrics
Table Revised January 14, 2013

Cycle	Triangle sweep cycle: 500 mV/s between 1.0 V and 1.5 V; run polarization curve and ECSA; repeat for total 400 h. Single cell 25-50 cm^2	
Number	5,000 cycles	
Cycle time	2 seconds	
Temperature	80°C	
Relative humidity	Anode/cathode 100/100%	
Fuel/oxidant	Hydrogen/nitrogen	
Pressure	Atmospheric	
Metric	**Frequency**	**Target**
Catalytic 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, 10, 100, 200, 500, 1k, 2k, and 5k cycles	≤30 mV loss at 1.5 A/cm^2 or rated power
ECSA/cyclic voltammetry***	After 0, 10, 100, 200, 500, 1k, 2k, and 5k 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 0.6 V at 20 mV/s, 80°C, and 100% RH.

Figure 1. Test protocols, and diagnostic method and metrics for Electrocatalyst (Table A-1) and catalyst support (Table A-2) durability. Source: [1].

Table A-4. Membrane Mechanical Cycle and Metrics
(Test Using an MEA)
Table Revised December 10, 2009

Cycle	Cycle 0% RH (2 min) to 90°C dew point (2 min), single cell 25-50 cm^2	
Total time	Until crossover >2 mA/cm^2 or 20,000 cycles	
Temperature	80°C	
Relative humidity	Cycle from 0% RH (2 min) to 90°C dew point (2 min)	
Fuel/oxidant	Air/air at 2 SLPM on both sides	
Pressure	Ambient or no backpressure	
Metric	**Frequency**	**Target**
Crossover*	Every 24 hours	≤2 mA/cm^2
Shorting resistance**	Every 24 hours	>1,000 ohm cm^2

* Crossover current per USFCC "Single Cell Test Protocol" Section A3-2, electrochemical hydrogen crossover method.
** Measured at 0.5 V applied potential, 80°C, and 100% RH N_2/N_2. Compression to 20% strain on the GDL.

Figure 2. Test protocol, and diagnostic methods and metrics formembrane mechanical durability. Source: [1].

References

1. U.S DRIVE / USCAR, *Fuel Cell Technical Team Roadmap*, June 2013, http://energy.gov/sites/prod/files/2014/02/f8/fctt_roadmap_june2013.pdf, retrieved 23-02-2015.

2. S. Zhang, X. Yuan, H. Wang, W. Mérida, H. Zhu, J. Shen, S. Wu and J. Zhang (2009) "A review of accelerated stress tests of MEA durability in PEM fuel cells," *International Journal of Hydrogen Energy*, **34,** 388-404.