Application Note – Automated Implementation of U.S. DOE PEM Fuel Cell Durability Protocols using Scribner Fuel Cell Test Systems

Introduction

Durability is a key performance property of polymer electrolyte membrane fuel cells (PEMFCs). The U.S. Department of Energy's (DOE) Fuel Cell Technology Office (FCTO) has developed accelerated stress tests (AST) to evaluate the durability of low temperature PEMFCs [1]. Test protocols exist that are intended to evaluate the mechanical and chemical durability of the membrane, the durability of the catalyst support, and the durability of the electrocatalyst, among others.

Scribner has developed the hardware, *FuelCell®* software setups and scripts for automated execution of DOE PEMFC Durability Protocols. The protocols are defined in the 2017 FCTO Multi-Year Research, Development, and Demonstration Plan (MYRDDP) [1].

Test protocols given in Appendix of the Fuel Cells Section of the MYRDDP include:

- Electrocatalyst Cycling for Cathode Electrocatalyst Durability – Table P.1
- Catalyst Support Cycling for Catalyst Support Durability – Table P.2
- MEA Chemical Stability for Membrane Chemical Durability – Table P.3
- MEA Mechanical Cycling for Membrane Mechanical Durability – Table P.4
- Combined Chemical/Mechanical Cycling for MEA Durability – Table P.5
- Polarization Curve – Table P.6
- Unmitigated Start-Up / Shutdown Durability Protocol – Table P.8
- MEA Recovery Protocol – Table P.9

This document covers fully- or partially-automated implementation of the fuel cell testing protocols using Scribner's fuel cell test systems and software. Hardware requirements, *FuelCell®* setups and Arbitrary Control files (scripts) are discussed, as are precautions and notes.

Required Hardware Requirements (specific hardware requirements depend on the durability protocol to be implemented)

1. 850C/e Fuel Cell Test System with Auto Water Fill
2. Humidifier By-Pass Option is required to implement the Membrane Mechanical Durability Protocol
3. Auto Multi Gas (AMG) Unit – Specific gas configuration depends on the protocol to be implemented
    a. H_2 connected to Anode Port A
    b. Air connected to Cathode Port A, N_2 connected to Cathode Port B
4. 885 Fuel Cell Potentiostat for ECSA by CV and H_2 Crossover by LSV as well as for Electrocatalyst and Catalyst Support Durability Protocols

[1] https://www.energy.gov/sites/prod/files/2017/05/f34/fcto_myrdd_fuel_cells.pdf

5. Automated Humidifier Drain / Fill (AHDF) Option (factory installation required) for rapid cooling of humidifiers – see Note 11 if your system is not equipped with the AHDF Option
6. Temperature Controlled Fan for Cell Cooling for rapid cooling of cell fixture
7. 5 or 25 cm^2 PEMFC fixture with MEA (*FuelCell* setups are for a 25 cm^2 cell)
8. Automated or manual back pressure unit
9. Pressurized supply of DI water

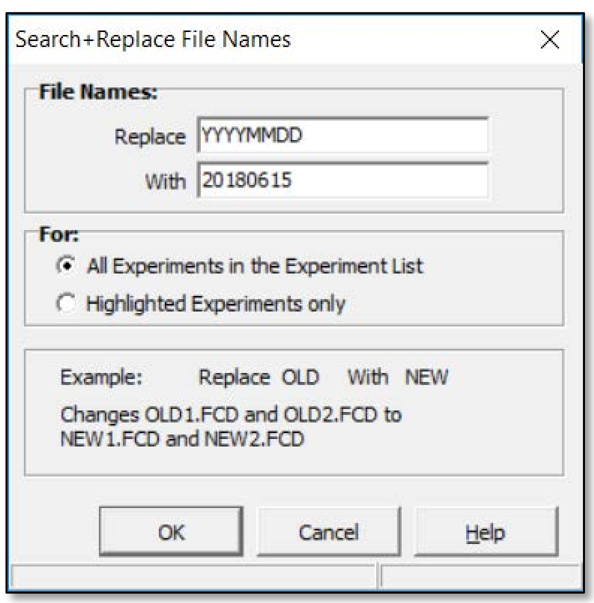
- 850R External Interface with at least one MFC (5 SLM Air or N_2 recommended) – required for implementation of Unmitigated Start-Up / Shutdown Durability Protocol – Table P.8

Notes

1. Users of the setups and script files provided by Scribner should fully understand the procedure and verify that their instrument configuration matches that used for the provided setups.
   - *e.g.*, H_2 connected to AMG[§] Anode Port A, Air connected to AMG Cathode Port A, and N_2 connected to AMG Cathode Port B
   - *e.g.*, 25 cm^2 active area
   - Alternatively, the provided setups can be modified to match the user's instrument configuration

2. The *FuelCell* software setup files (.fc3) and the Arbitrary Control files (.fcc) should be located in *c:\FuelCell\Setups*. The folder \Setups\ must be created by the user.

3. Select "Replace Files..." or **Background | Search + Replace File Names...** to change the placeholder text "YYYYMMDD" in the file names with a unique identifier, such as the date or sample ID. Refer to the "Replace Names..." feature in the *FuelCell* software manual.



4. The default location for data files are is *c:\FuelCell\Data*. This can be changed by first creating a folder that the data files will be saved to, and the using "Replace Files..." feature to change the path name.

5. 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 (and other current-controlled steps) must be modified.

6. 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.

7. 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 gas plumbed as shown in the table:

AMG Port	Anode	Cathode
A	H_2	Air
B	N_2	N_2
C	Air	Unused

8. 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.

9. Under H_2 / N_2 conditions, the cell voltage is ~ 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" and "-2", respectively. This effectively disables the low voltage alarms. This can also be done with a Change Cell experiment step as part of the experiment list.

10. Older 885 PSTATs that connect to the 850e via the Auxiliary Signals Connector have a maximum data acquisition rate of 0.1 s/point (10 points/s). Current 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.

11. If your test system is not equipped with the Automated Humidifier Drain / Fill (AHDF) Option please refer to ***Application Note – Manual Method for Accelerated Humidifier Cooling***.

12. The *FuelCell* software setup files for the **Electrocatalyst** and **Catalyst Support Durability** tests consist of a series of experiment steps that execute:
    - Voltage sweeps and/or potentiostatic holds 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.

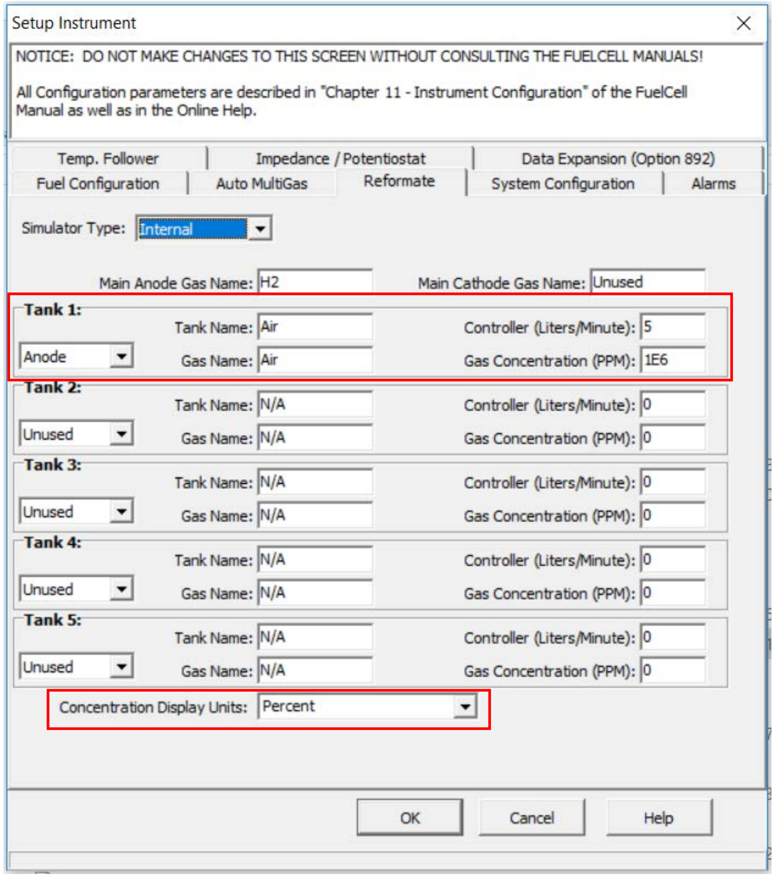
13. The *FuelCell* software setup files for **Membrane Mechanical Durability** consist of a series of experiment steps within nested repeat loops that execute:
    - Inner Repeat Loop: 360 repetitions, 2 min wet + 2 min dry (24 hour total). Contains Setup Fuel Experiments that switch anode / cathode 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.
    - PSTAT Voltage Sweep experiment with the 885 PSTAT under H_2 / N_2 for the H_2 Crossover measurement.
    - PSTAT 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.

14. The **Polarization Protocol** calls for the performance curve to be measured at 150 kPa_a whereas durability protocols are conducted at a different back pressure or ambient pressure (zer0 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 or under fixed back pressure such as 100 kPa_g (15 PSIG).

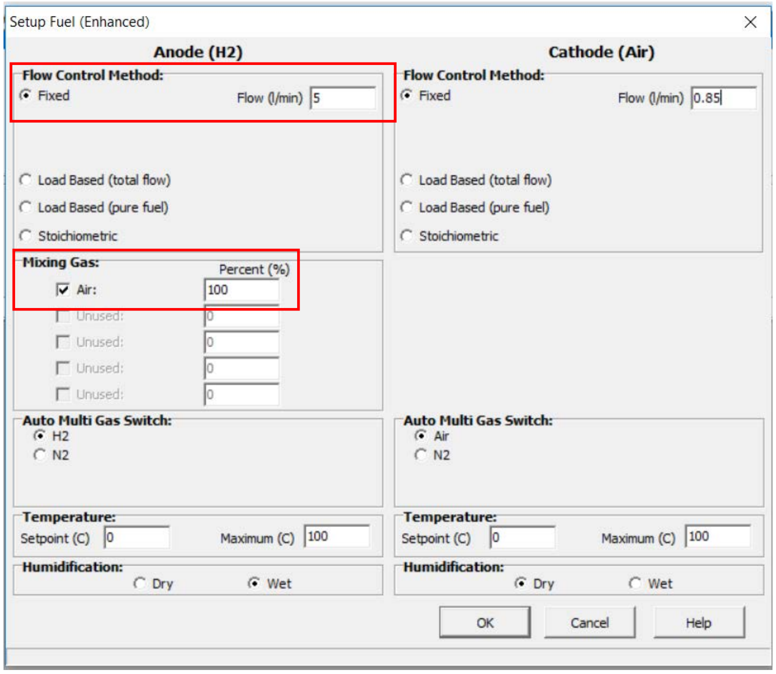
15. The **Unmitigated Start-Up / Shutdown Durability Protocol** requires rapid purging of H_2 from the cell's anode compartment with dry Air (refer to the "Step 3 – Shutdown in Table P.8). This is facilitated using an 850R External Interface Accessory with a 5 SLM mass flow controller (MFC) plumbed with Air. The outlet of this external MFC is plumbed directly to the cell anode inlet using a T-fitting.

    An example configuration of the external MFC in *FuelCell* is shown in below. In this example, the external MFC is connected to Reform Channel #1 on the 850R External Interface (labelled "R1") and has a full scale flow rate of 5 SLM. The

MFC is plumbed to the Anode, therefore it is assigned to the “Anode”. Display units are set to “Percent”.



The Setup Fuel and Change Fuel Experiment windows then appear as shown in the figure below. Rapid purging of the anode compartment with dry Air during the Shutdown Step of the Start/Stop protocol is executed with a Change Fuel Experiment configured as shown, followed by a 5 second Open Circuit Experiment.



Arbitrary Control Files (scripts)

Automated Humidifier Drain / Fill Arbitrary Control Script

The Automated Humidifier Drain / Fill (AHDF) procedure is used to rapidly cool the 850 humidifiers for reduced dew point testing. This is accomplished using the required Auto Humidifier Drain / Fill Valve Assembly Option as well as Arbitrary Control Experiment Steps that execute the AHDF script. Refer to the Auto Humidifier / Fill Valve Assembly Instructions. The AHDF script parameters – drain time and fill time - must be determined by the user.

The Arbitrary Control file "*arb ctrl - humidifier drain+fill x1.fcc*" will drain and fill each humidifier once. This is sufficient to reduce the humidifier temperature from 80 ^{o}C to ~ 60 ^{o}C, which is the desired humidifier temperature (*i.e.*, dew point) for the DOE polarization curve protocol.

The same Arbitrary Control file can be used in a Repeat Loop with 2 or 3 repeats to reduce the humidifier temperature to ~30 ^{o}C, depending on the source water temperature.

DOE MEA Recovery Protocol with Arbitrary Control Script

Some durability protocols call for an MEA recovery procedure after performing the durability portion of the protocol and prior to performing the respective diagnostic experiments. The Arbitrary Control file "*DOE MEA Recovery Protocol.fcc*" performs the recovery method described in Table P.9 of the MYRDDP. It can be inserted in

The MEA recovery procedure consists of a series of anode / cathode gas types, flow rates and durations. The Arbitrary Control file is based on gases connected to the AMG as described above and that the tests stand's mass flow controllers are sized to meet the flow rates called for in the protocol, *i.e.*, 2 SLM on anode and 4 SLM on cathode.

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.

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

References

1. Fuel Cell technologies Office, U.S. DOE, Multi-Year, Research, Development and Demonstration Pan (MYRDDP), 2016 Fuel Cells Section, https://www.energy.gov/sites/prod/files/2017/05/f34/fcto_myrdd_fuel_cells.pdf (last retrieved 2018-11-19).

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.